# PRISM



# USER MANUAL

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# Contents

# Section 1



# Introduction

This manual provides the operation and maintenance instructions for the PRISM Mass Spectrometer with Dual Inlet. It will discuss all the options available with the PRISM, however if some of these have not been purchased, then those sections of the manual covering them should be ignored. The accessories (e.g. GC, Manifold, EA, etc.) available with the PRISM are covered in their own manuals, which you will receive if they have been purchased.

We have endeavoured to include all operational aspects within this manual, however if you feel more information is required or that you can add some more details, then please contact the Customer Service Department at Micromass or your local representative.

# Section 2



# Safety Notices

Important safety information is highlighted as WARNING and CAUTION instructions. The use of  WARNINGS and CAUTIONS is defined below:

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**WARNING**

**Warnings are given where failure to observe the instruction could result in injury or death to persons.**

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**CAUTION**

**Cautions are given where failure to observe the instruction could result in damage to the equipment, associated equipment or process.**

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Ensure that maintenance is carried out by a suitably qualified technician. Comply with all local and National requirements for electrical and mechanical safety. Please contact the Customer Service Department at Micromass or your local representative if you require any further information.

# Section 3



# Isotope Ratio Mass Spectrometers

# Introduction

This section of the manual is aimed at those users who are new to isotope ratio mass spectrometers (IRMS) or are unfamiliar with some of the basic concepts of these instruments. This section only touches the surface of the material it could cover. However we feel that it is sufficient in depth to enable the new user to move on to use the instrument with confidence and understanding. Most experienced users will probably ignore this section, however the 'Calculations and Corrections' pages contain definitions of formulae for calculations and corrections generally used and thus may be of interest as a reference section.

This is a generalised section and is used in all the user manuals, so please ignore any parts which are not relevant to your system.

# Basic mass spectrometry concepts

## Mass Selection

If an ion of mass M and charge Z is accelerated in a potential V and injected into a uniform magnetic field B then the ion experiences a force and moves in a circular orbit of radius R. The motion is defined by;

$$\frac{M}{Z} = \frac{B^2 R^2}{2V} \tag{1}$$

and for singly-charged ions the radius is determined by the choice of magnetic and electric field. The combination of fields selects ions of particular mass and forms a mass filter. This principle is the basis of all magnetic-sector mass spectrometers and equation (1) is frequently termed the mass spectrometer equation.

# Mass Spectrometers

To analyse a sample gas its molecules must be ionised, formed into a beam, accelerated by an electric field, deflected in a magnetic field, and finally detected. These five processes take place in the analyser of a mass spectrometer, which consists of three separate sections: the source, flight tube, and collector. Ionisation, beam formation and acceleration all occur in the source, magnetic deflection takes place in the flight tube and detection takes place in the collector.

The ionisation is commonly achieved by passing a beam of electrons through the gas sample. Collision or close approach of an electron and sample molecule can cause one or more electrons either to adhere to the molecule and form a negative ion, or to detach from the molecule and leave a positive ion. Except in special cases, it is the singly-charged positive ions (molecules that have lost one electron) which are used in mass analysis. These positive ions are accelerated and formed into a well-defined beam by raising the ionisation chamber to a positive potential and accelerating the ions out through a slit towards a second defining slit at ground potential. The two slits are known as the source and alpha slit respectively.

The flight tube forms an arc of a circle and passes between the poles of a magnet. As the ion beam travels down the tube, it is separated into beams of different radii corresponding to different masses. A particular radius, and hence mass, is selected by a slit at either end of the flight tube; the alpha slit in the source and the resolving slit in the collector.

In the collector, ions of the chosen mass are transmitted through a resolving slit and detected by a Faraday cup. The ion current from the cup is proportional to the number of incident ions and hence to the partial pressure of the corresponding isotopic molecular species in the sample gas. Multiple slits and Faraday cups are frequently used to obtain simultaneous detection of different masses. This method is used in isotope ratio instruments to measure a major beam, due to the most abundant isotopic species of a molecule, and minor beams from the less abundant species.
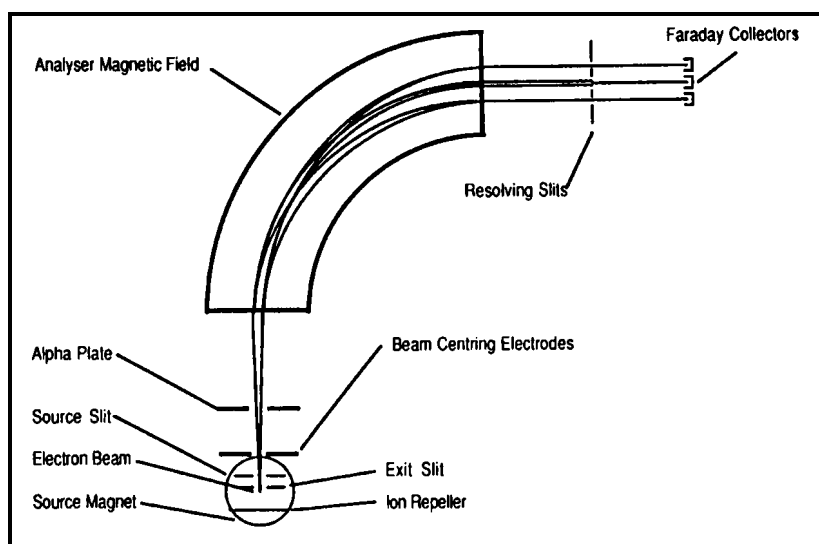


**Mass Separation Diagram**

# Analytical and Isotope Ratio Analysers

The most pronounced difference between analytical and isotope ratio instruments lies in the peak shape observed by scanning the magnetic or electric fields. In analytical work a mass range is scanned to obtain a spectrum of mass peaks which are characteristic of chemical composition. In isotope ratio work the chemical composition of the sample is known and the fields are held constant so that the variations of isotopes in one chemical species may be measured with high precision. Thus an analytical instrument requires a very narrow peak to distinguish closely spaced masses, whereas a very broad peak is required for high stability in the amplitude measurements of isotope ratio instruments.

# Peak Shape

Peak shape is defined by the source and resolving slits because the analyser is constructed such that the image of the source slit is focused at the resolving slit. (The alpha slit merely eliminates unwanted ions.)

For a resolving slit (at the collector) much narrower than the source slit, a triangular peak shape would be observed as the image of the source slit was swept across the resolving slit (see Narrow Collector Slit figure below). Thus the source slit determines the minimum peak width.

For a resolving slit much wider than the source slit, the image of the source slit would be wholly contained within the resolving slit, and there would be a broad range of electric and magnetic fields over which the image of the same mass could be observed. Although the slopes of the triangular peak would remain, they would be separated by a large mass range over which the signal did not change and a broad flat-topped peak would be observed (see Wide Collector Slit figure below). Thus the resolving slit determines the width of a flat-topped peak, which is limited simply by the encroachment of the adjacent mass peaks.

A narrow source and resolving slit suit analytical measurements, but a narrow source and broad resolving slit best accomplish isotope ratio measurements
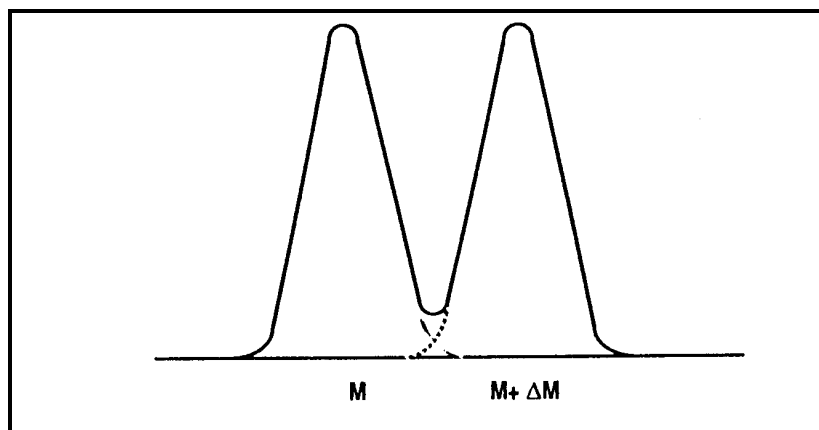


**Narrow Collector Slit**
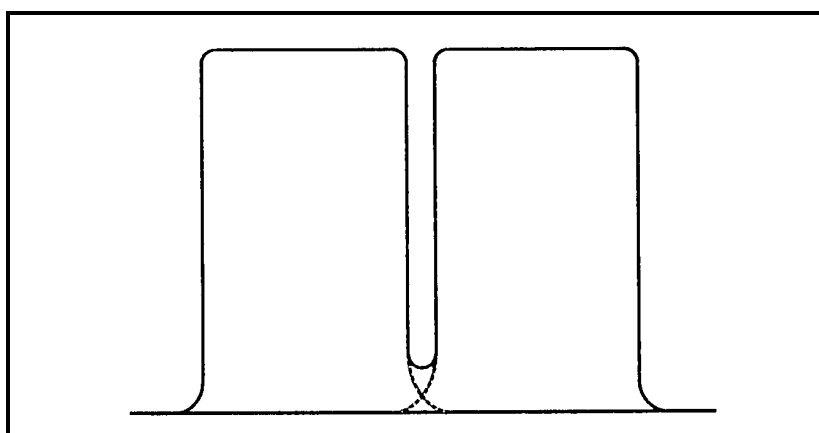


**Wide Collector Slit**

# Dispersion

The mass dispersion determines the separation between adjacent mass peaks.

For an analyser of radius r, source slit-to-magnet exit distance $x_s$ and resolving slit-to-magnet exit distance $x_r$, the mass dispersion measured perpendicular to the ion trajectory is expressed as:-

$$D = \Delta r \left( 1 + \sqrt{\frac{\left(r^2 + x_r^2\right)}{\left(r^2 + x_s^2\right)}} \right) \tag{2}$$

However, differentiating the mass spectrometer equation ("Mass Selection" above, equation (1) gives:-

$$\frac{\Delta M}{M} = \frac{2\Delta r}{r} \tag{3}$$

and therefore the mass dispersion may be expressed as:-

$$D = \frac{r\Delta M}{2M} \left( 1 + \sqrt{\frac{\left(r^2 + x_r^2\right)}{\left(r^2 + x_s^2\right)}} \right) \tag{4}$$

In a symmetrical analyser where $x_s = x_r$, the mass dispersion reduces to:-

$$D = 2\Delta r = \frac{r\Delta M}{M} \qquad \text{or}$$

$$D = \frac{r\Delta M}{M} \tag{5}$$

in isotope ratio instruments where peaks are separated by one mass unit. It is clearly seen that dispersion is specified by the radius of a symmetrical instrument and this is frequently quoted as the dispersion itself.

For an asymmetrical analyser, the dispersion is frequently compared to an effective radius

$$r_{eff} = \frac{r}{2} \left( 1 + \sqrt{\frac{\left(r^2 + x_r^2\right)}{\left(r^2 + x_s^2\right)}} \right) \tag{6}$$

which combines all the necessary geometric information.

# Resolution

Two peaks are completely separated if (neglecting aberrations):

$$D > \left(W_s + W_r\right)$$

where $W_s$ is the source slit width
and $W_r$ is the resolving slit width

Mass resolution is defined as

$$R = \frac{M}{\Delta M}$$

from 5

$$R_{max} = \frac{r_{eff}}{D} \qquad (7)$$

which (from 6) is a maximum when $D = W_s + W_r$

i.e. $\qquad R = \dfrac{r_{eff}}{W_s + W_r} \qquad \left(W_s > W_r\right) \qquad (8)$

The resolving power of a mass spectrometer is the highest mass number at which peaks of adjacent molecular weight and equal heights have a valley between them of 10% of the peak height. This is known as the 10% valley definition.



The figure shows the appearance of two peaks at masses M and M+1 from an instrument with the resolving power of M. That is, if the resolving power is 100, then the peaks at mass 100 and mass 101 would be as shown.
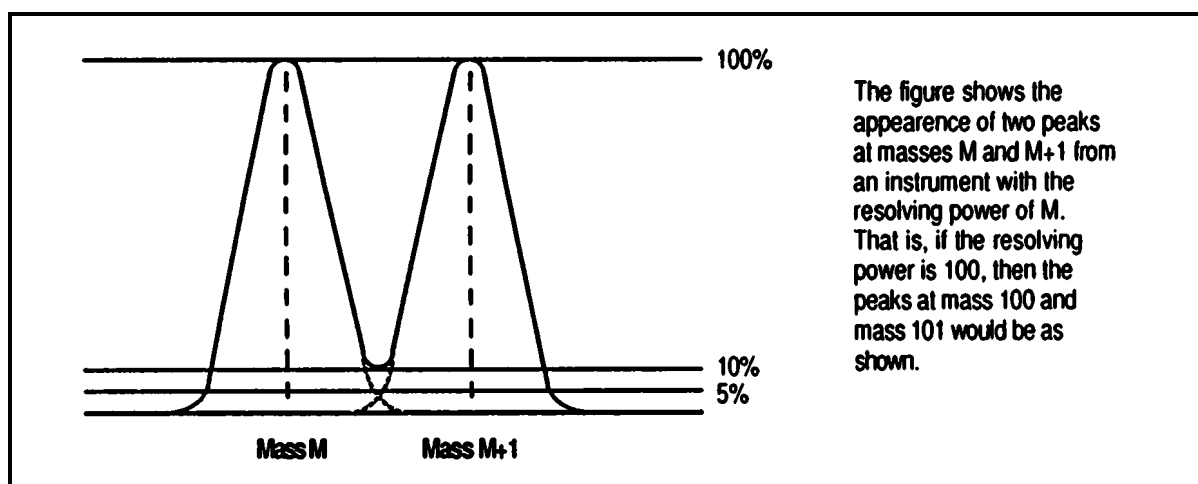
**10% Valley Resolution**

As an alternative 5% definition is identical to the 10% valley. In this case $\Delta M$ is defined as the width of the peak at the 5% height espoused as a proportion of the distance between adjacent masses.

The '5% peak height' definition is an alternative interpretation of the '10% valley' definition.
The resolution of the peak

shown as $\dfrac{M}{\Delta M}$ where $\Delta M$ is the distance between the 5%

points expressed as a proportion of the distance between adjacent mass numbers.

If $\Delta M$ is less than unity, then resolving power is greater than M.
If $\Delta M$ is greater than unity, then resolving power is less than M.

**5% Peak Height Resolution**

The separation between the peaks is determined by the image size, ions extracted from the source on the wrong trajectory and by aberrations in the optics.

In principle the maximum resolution is directly calculable from the radius and slit width equation (8).

However, the design of the high sensitivity source used in Isotope machines results in a high intensity image which is narrower than the source defining slit and so increases the actual available resolution of the instrument.

# Vacuum System

## Why is a Vacuum required?

In mass spectrometry we are particularly interested in vacuums for two reasons:-

1. Ion scattering:- If the ions collide with any residual gas molecules their trajectory will be modified resulting in peak broadening.

2. Contamination:- Residual gases in the ionising chamber are also ionised together with the sample material giving rise to an instrument background.

# How is a Vacuum measured?

Gases are composed of small particles which are in constant motion. As these particles move around in space they collide with other objects and exert a force. If we can take a unit of area and measure the number and intensity of particle impacts then the resultant is a pressure measurement.

The pressure per unit area P is

$$P = \frac{1}{3} nmv^2_{rms}$$

where

$$v = \left(\frac{3KT}{m}\right)^{-\frac{1}{2}}_{rms}$$

m is the mass of the molecule,
n the number of molecules in unit volume.
v is the root mean square of all the possible molecular velocities.
K is Boltzmans constant $\left(1.38 \times 10^{-23}\, Pa.M^2.K^{-1}\right)$
and T the temperature of the gas in degrees Kelvin.

# Units of Pressure

Atmospheric pressure is equivalent to

| | |
|---|---|
| 14.7 | lbs per sq inch |
| 760 | mm of Hg |
| 760 | Torr |
| 760000 | millitorr or microns |
| 101325 | Pascal |
| 1.01325 | Bar |
| 1013.25 | millibar |

Common terms for pressure regimes are

| | |
|---|---|
| Rough vacuum | $10^3$ to $10^{-3}$ millibar |
| High vacuum | $10^{-3}$ to $10^{-8}$ millibar |
| UHV | $< 10^{-8}$ millibar |

# Gas Flow

The process of evacuation involves the removal of gas from a vacuum vessel. The rate of removal (i.e. the gas flow) determines the rate at which the pressure decreases. It is therefore important to understand viscous and molecular flow regimes.

## Viscous Flow

In general gas molecules occupying a space at pressures of greater than $10^{-2}$ millibar act very much as a fluid. In this viscous flow range the molecules are constantly bumping into each other and are so closely packed together that as the vacuum pump moves some of them out of the chamber others rush to fill that empty space. In viscous flow ranges molecular movement is predictable and we can use smaller diameter hoses and tubes for rough pumping. Viscous flow allows great quantities of molecules to be moved per unit time from one place to another.

## Molecular Flow

At lower pressures molecules are so far apart that they no longer exert any influence on each other and motion is strictly random. Depending on pressure a gas molecule may move mm, cm, meters, or even kilometres before it strikes another molecule. This means we cannot depend on molecular movement to push or start a flow pattern. This is why we need to have such large diameters on high vacuum pumping systems, as then we increase the probability that one of these randomly moving molecules will move into the pump.

The difference between the flow regimes does not depend solely on pressure but also on the dimensions of the vacuum container, pipes, etc. It basically depends on the mean free path of the gas molecules.

## Pumps

The purpose of a pump is to provide a means for removing gas. Its pumping speed S is rated in litres per second. Most practical pumps operate continuously so that if a constant flow of gas Q is introduced into the pump then a steady state is set up with an associated constant pressure within the pump. The pumping SPEED is defined as

$$S = \frac{Q}{P}$$

and is clearly an indication of pumping ability.

Air is easily removed at high pressures but after a short while very little gas remains so that pumping speed does not indicate how many molecules we are removing from the system. The amount of gas present in the system is determined by multiplying the pressure (in torr) by the volume (in litres) which tells us the number of molecules in the system or the gas LOAD. Apart from back streaming of pump fluids and influx from real leaks, there are four main sources of gas load in a high vacuum system: (1) desorption of absorbed gases, (2) volatilisation, (3) diffusion of gases from inside solids and (4) gas permeation.

In vacuum systems we are interested to know how much work has to be done to transfer a mass of gas from one place to another.

## The Rotary Vane Pump

The rotary vane pump removes gases by compressing them to a point slightly above atmospheric pressure by means of an offset rotor with spring loaded vanes. It then expels the gas to the outside world, approximately 99.999% of the air being removed. It is used to produce rough or foreline pressures. The pump is immersed in an oil bath and the oil is purified to remove high vapour pressure contaminants. It serves to cool and lubricate the pump and provides the seal against atmospheric pressure. The pump motor may be directly coupled or belt driven. Most pumps have two stages to produce a better vacuum. As the film of oil makes the final seal the ultimate pressure achievable is partly determined by the vapour pressure of the oil. If the oil becomes too loaded with water or other impurities the pump's performance will be drastically reduced. Clean oil in the rotary pump is therefore very important. When operating at low pressures the rotary vane pump tends to back stream oil vapour to the roughing line which may even migrate to the vacuum chamber. This can be controlled by means of suitable precautions such as foreline traps in the pumping line.
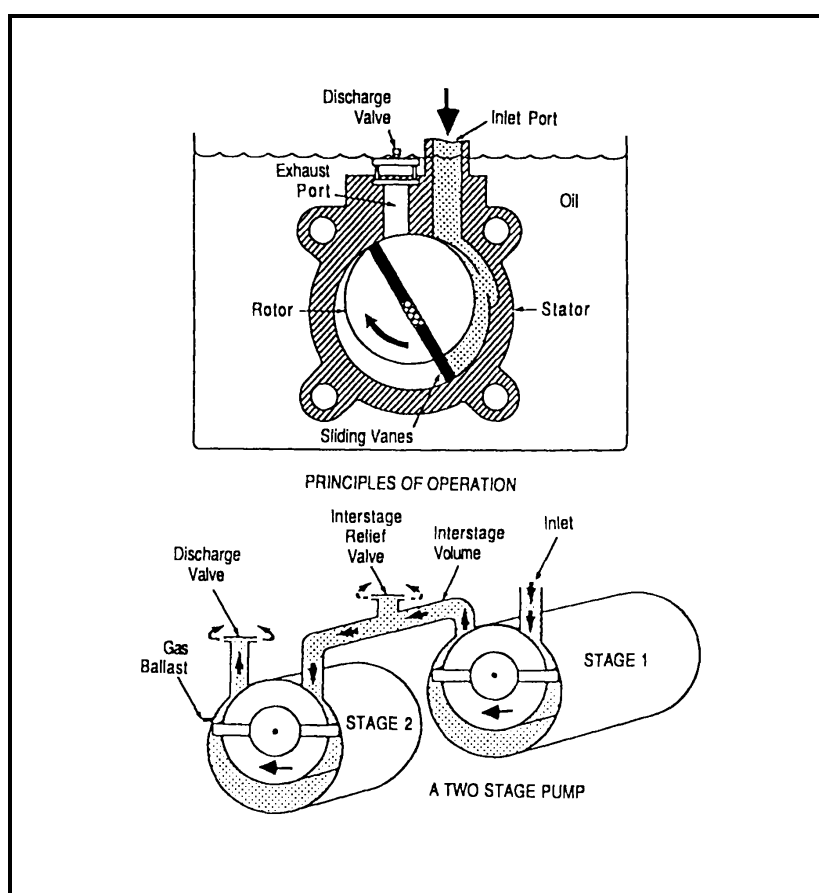


**Rotary Vane Pump**

An oil-sealed mechanical pump includes a housing, or stator, an offset rotor with spring loaded vanes, an intake port and an exhaust port equipped with a discharge valve. They may also have a ballast valve. Direct drive pumps pop.
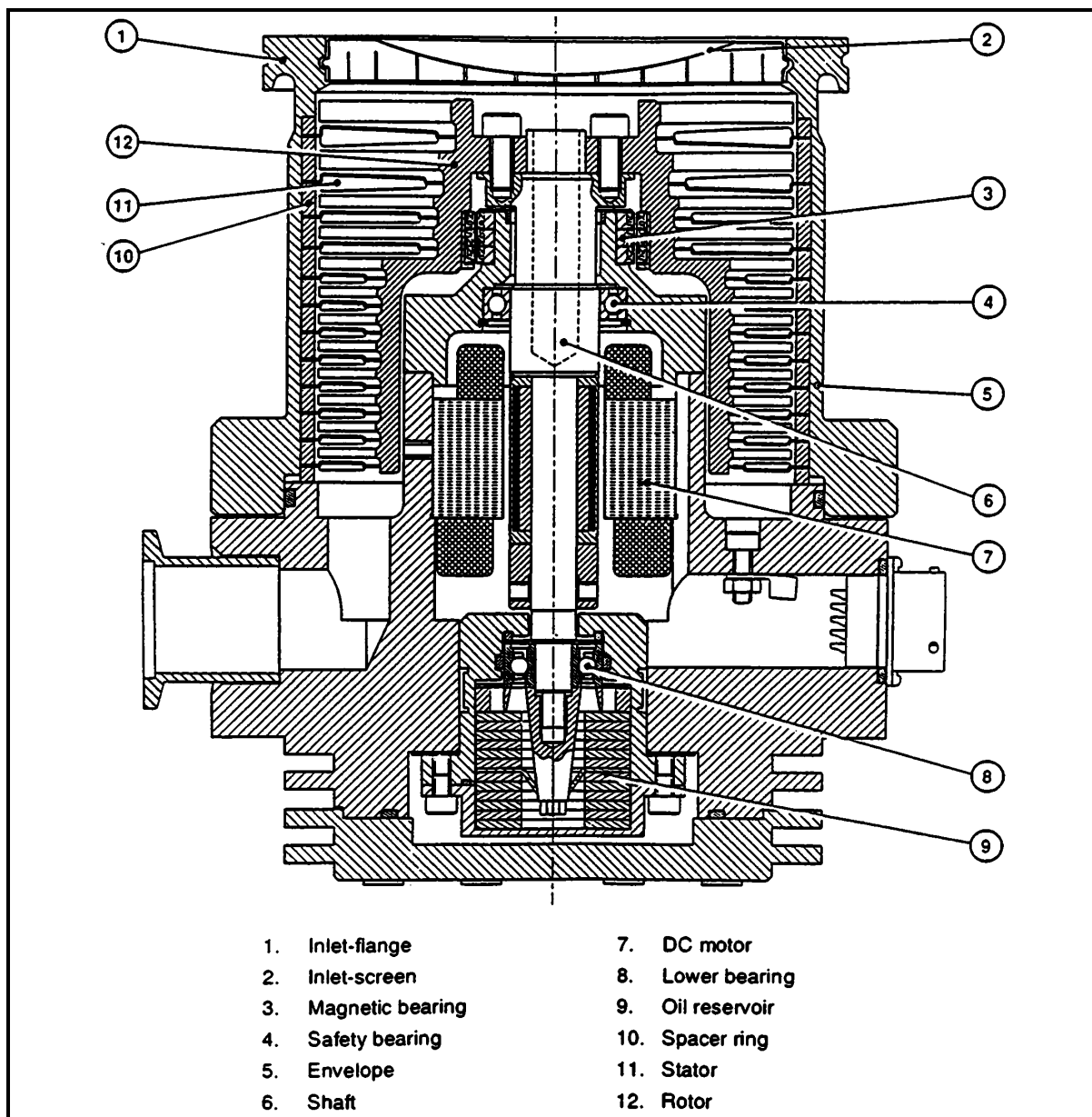
For a more detailed description please refer to the pump manufacturers manual.

## The Turbo Molecular Pump

A Turbo Molecular pump (TMP) is essentially a high speed axial compressor. Gas is taken from the low vacuum side, compressed and exhausted to the vacuum foreline to be pumped away by the rotary pump. If the TMP is turned off while open to the vacuum system, hydrocarbons from the foreline and motor bearing will diffuse through the rotor/stator interspaces to the high vacuum part of the pump. A vent valve which admits vent gas to the TMP is opened when the pumping system is turned off and the pump speed has dropped below 50% of the full rotational speed to prevent this back diffusion.

The TMP will only operate if the rotary pump is working. The TMP is self regulating and accelerates as the pressure drops.



| | | | |
|---|---|---|---|
| 1. | Inlet-flange | 7. | DC motor |
| 2. | Inlet-screen | 8. | Lower bearing |
| 3. | Magnetic bearing | 9. | Oil reservoir |
| 4. | Safety bearing | 10. | Spacer ring |
| 5. | Envelope | 11. | Stator |
| 6. | Shaft | 12. | Rotor |

**Figure : Turbo Molecular Pump**

For a more detailed description please refer to the pump manufacturers manual

# Pressure Gauges

## Total Pressure Transducer

This consists of a stainless steel pressure chamber isolated by a stainless steel diaphragm which resistive to corrosive materials. The inner face of the metal diaphragm has high gain strain gauges bonded to it. When the diaphragm is deflected resulting from a change in pressure in the vacuum system, the strain gauge output changes. This 1-6V output signal is proportional to system pressure and is monitored to give a measure of pressure in mBar.

## The Pirani Gauge

This is very similar to the thermocouple gauge in operation in that it relies on gas molecules colliding with a filament conducting heat away. However in this case the filament resistance rather than the temperature is measured. The filament forms part of a bridge circuit. In the balanced bridge circuit the current flow through path one and two is equal and a meter placed at the centre as shown reads zero. When the filament changes resistance the balance is upset and a voltage difference is developed at the meter terminals and a current flows through it. This is calibrated in terms of pressure. The compensator is very similar to the filament and is held at a constant temperature and pressure. The Pirani gauge generally operates in the range $10^3$ mBar to $10^{-3}$ mBar.
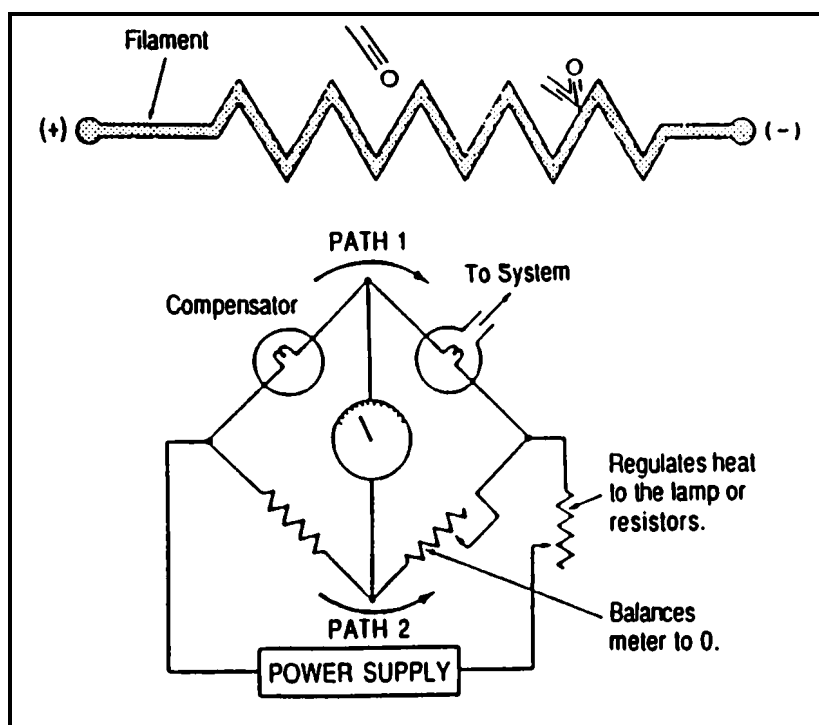


**Figure : Pirani Gauge**

## The Ionisation Gauge

The ionisation gauge works on a very similar principle to an ion pump and is unique in that it can operate over 12 orders of magnitude from $10^{-2}$ mBar to $10^{-14}$ mBar. It is more commonly used over 6 orders of magnitude from $10^{-4}$ mBar to $10^{-10}$ mBar and is expected to be within 30-50% of the correct pressure for high vacuum work.

It consists of a hot filament, a grid, an ion collector and a control unit to provide power, amplification and metering. Electrons from the hot filament are emitted and attracted towards the grid. However many electrons miss the grid wires and swing around it several times before striking it. Because of the large number of electrons emitted from the filament a fairly constant electron cloud is formed about the grid. Many electrons collide with gas molecules ionising them and releasing more electrons. The positively ionised molecules are attracted towards the collector where they produce an ion current proportional to the number of gas molecules in the chamber and hence the pressure. To get meaningful pressure readings the sensitivity of the gauge must be known and the emission current well regulated.
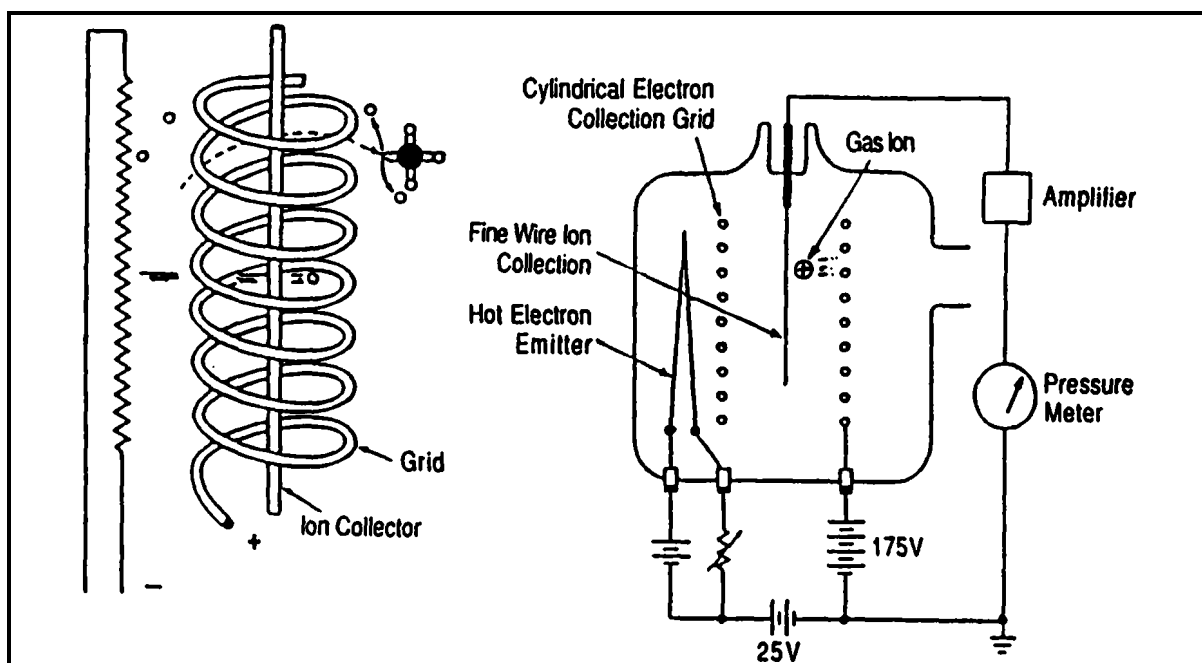


**The Ionisation Gauge**

It should be noted that sensitivity varies with different gas species. Ion gauges are usually air calibrated and therefore most accurate when analysing nitrogen

The ionisation gauge like any other has its limitation. Low energy X-rays are created when the electrons strike the grid which causes the releases of photo-electrons and hence a constant error signal. Above $10^{-10}$ mBar this is insignificant but at lower pressures, corrections must be made for this X-ray effect.

# Sample Inlet Principles

## Dual Inlet Operation

The Dual Inlet comprises two identical halves each containing a variable gas reservoir. One half is used to hold the sample gas whilst the other is used to store a gas of known isotopic composition.

Sequentially these two gases are fed to the analyser via a viscous leak formed by a crimped capillary. The two measurements obtained are then used to calculate the difference in isotopic abundance between the two gases. This difference is expressed in delta notation, details of which can be found in the Calculation and Corrections section below.

## ContinuousFlowOperation

The Continuous Flow Inlet comprises of two vitreous silica capillaries which continuously carry Helium gas into the ion source of the mass spectrometer. These two capillaries draw Helium at two open splits, one fed from a reference gas injector system, the other from a sample preparation system (e.g. gas chromatograph, elemental analyser or μgas injector).

At user defined times the reference gas injector pulses rectangular peaks of pure reference gas, (pre-calibrated against an international standard) into the ion source. The sample gases exiting from a gas chromatograhic column are carried by a stream of Helium towards the sample open split, from here the sample gases also enter the ion source.

The areas under the sample and reference peaks are calculated, from which the difference in isotopic composition between the two is evaluated. This difference is expressed in delta notation as with the dual inlet, details of which can be found in the Calculation and Corrections section below.

# Calculations and Corrections

## Enrichment Delta, $\delta$

Enrichment is the excess of any particular isotope species in the sample compared to the same isotopic species in the reference. The enrichment delta expresses this excess as a fraction of the particular isotopic species in the reference and is almost universally quoted in parts per thousand and written as ‰ or per mil. Thus the enrichment delta is;

$$\delta = \left( \frac{R_{sam} - R_{ref}}{R_{ref}} \times 1000 \right) ‰$$

Where $R_{sam}$ and $R_{ref}$ are the sample and reference ratios of minor to major beam as measured on the isotope ratio instrument.

Enrichment delta notation can be used to express results for all species of gases measured.

## IsotopicAbundance(Atom%)

This expresses the number of atoms of a particular isotope of an element in the sample as a fraction of the total number of atoms of that element present. It is usually expressed as a percentage and notated as Atom% or At%.

This notation is particularly popular amongst the biological users and for nitrogen measurement can be defined as:-

$$Atom\% = \frac{number\_of\_^{15}N\_atoms}{\left(number\_of\_^{15}N\_atoms\right) + \left(number\_of\_^{14}N\_atoms\right)}$$

Some users prefer to express their results as atom% excess (i.e. atom% enrichment in excess of the reference gas) which is defined as

Atom% excess = sample atom% - reference atom%

## Internal Reproducibility

In a set of n measurements of a quantity x whose mean is $x_{mean}$ there are two commonly used and related quantities for the precision.

Standard deviations of a measurement, $\sigma$

This is also referred to as the standard deviation and defines the precision of one individual measurement from the complete set of measurements. It is estimated from the data by

$$\sigma = \sqrt{\frac{\sum \left(x - x_{mean}\right)^2}{(n-1)}}$$

Standard deviation of the mean, $\sigma_{mean}$

This is also referred to as the standard error and defines the precision of the average over the complete set of measurements. It is rigorously related to the standard deviation by

$$\sigma_{mean} = \frac{\sigma}{\sqrt{n}} = \sqrt{\frac{\sum \left(x - x_{mean}\right)^2}{n(n-1)}}$$

The internal reproducibility of an instrument is defined as its standard error, where n is the number of values of delta produced by one set of sample/reference comparisons. For example a set of 6 pairs of sample/reference comparisons producing 10 values of delta would give

$$\sigma_{mean} = \sqrt{\frac{\sum \left(x - x_{mean}\right)^2}{90}}$$

# Instrument Corrections

In isotope ratio instruments there are three correction factors which may be of concern

1. A tail contribution from major to minor isotope peak.(abundance sensitivity correction)

2. A background gas peak at the minor isotope mass.

3. The cross mixing of sample and reference gasses due to seat leakage in the changeover valve.

In normal dual inlet mode the abundance sensitivity corrections and the changeover valve cross mixing corrections are negligible, and are ignored.

If the vacuum system is clean, background corrections also should be ignored, although software facilities are available to subtract background readings from all acquired data if required. If the sample has been prepared incorrectly, there may be problems due to contaminants at the masses of interest (eg. CO peaks overlapping with nitrogen peaks in a dirty nitrogen sample). This type of interference may cause small errors in delta values. The peak jump facilities in the software are provided in order to assess the nature of possible contaminants.

# Carbon Dioxide Corrections (Craig)

When analysing $CO_2$ different isotopic species of the same element can produce a contribution at certain masses and a correction must be made. For example the international $CO_2$ standard PDB has a contribution (approximately 6%) at mass 45 due to the $^{17}O$ isotope. Similarly , 0.2% of mass 46 is derived from isotopic species containing $^{13}C$ and $^{17}O$ but not $^{18}O$ . Masses higher than 46 have negligible abundance's.

For a triple collector instrument measuring a sample close to PDB, the correction formulae are

$$delta\,^{13}C = 1.0676 delta\left(45/44\right) - 0.0338 delta\,^{18}O$$

and

$$delta\,^{18}O = 1.0010 delta\left(46/44\right) - 0.0021 delta\,^{13}C$$

# Nitrogen Corrections

When analysing nitrogen a problem can, at low enrichments, arise owing to trace nitrous oxide (NO) causing an interfering peak at mass 30. This, if not correctly taken into consideration, can adversely affect the resultant calculation of $^{15}N$ atom%.

To overcome this problem it is common to use a formula modified from the standard formulae when the enrichments are low (less than 5%). The two formulae commonly used are:

Standard formula for high enrichments

$$^{15}N\_atom\% = \frac{R_{29} + 2R_{30}}{2\left(1 + R_{29} + R_{30}\right)} \times 100$$

(1)

modified formula for low enrichment

$$^{15}N\_atom\% = \frac{R_{29}}{2 + R_{29}} \times 100 \tag{2}$$

where $R_{29} = 29/28$ mass ratio and $R_{30} = 30/28$ mass ratio

Derivation of formula used

Given the probabilities of $^{15}N = \alpha$ and $^{14}N = 1 - \alpha$, then assuming the nitrogen sample is in equilibrium the $N_2$ probabilities can be expressed as:-

$$mass\_28\left(denoted\_I_{28}\right) = (1 - \alpha)^2$$

$$mass\_29\left(denoted\_I_{29}\right) = 2\alpha(1 - \alpha)$$

$$mass\_30\left(denoted\_I_{30}\right) = \alpha^2$$

From the following 29/28 and 30/28 ratios can be expressed as:

$$R_{30} = \frac{\alpha^2}{(1 - \alpha)^2} \qquad and \qquad R_{29} = \frac{2\alpha}{1 - \alpha}$$

hence giving

$$R_{30} = \left(\frac{R_{29}}{2}\right)^2 \tag{3}$$

Atom% can be written as

$$\frac{2\left(^{15}N^{15}N\right) + \left(^{15}N^{14}N\right)}{2\left(^{14}N^{14}N\right) + 2\left(^{14}N^{15}N\right) + 2\left(^{15}N^{15}N\right)} \tag{4}$$

$$Atom\% = \frac{2I_{30} + I_{29}}{2I_{28} + 2I_{29} + 2I_{30}} \times 100 \tag{5}$$

Giving the standard high enrichment formulae (1)

$$Atom\% = \frac{R_{29} + 2R_{30}}{2\left(1 + R_{29} + R_{30}\right)} \times 100 \tag{1}$$

Substituting (3) in (1)

$$Atom\% = \frac{2\left(R_{29}/2\right)^2 + R_{29}}{2\left(1 + R_{29} + \left(R_{29}/2\right)^2\right)} \times 100 \tag{6}$$

$$Atom\% = \frac{R_{29}^2 + 2R_{29}}{4\left(1 + R_{29} + R_{29}^2/4\right)} \times 100 \tag{7}$$

$$Atom\% = \frac{R_{29}\left(R_{29} + 2\right)}{\left(R_{29} + 2\right)\left(R_{29} + 2\right)} \times 100 \tag{8}$$

which gives the modified low enrichment formulae

$$Atom\% = \frac{R_{29}}{2 + R_{29}} \times 100 \qquad (2)$$

Both formulae (1) and (2) have limitations:-

formula (1) although accurate at high enrichments can cause errors at low enrichments due to the presence of Nitrous Oxide.

formula (2) overcomes the NO constitution problem at low enrichments by substitution of $R_{29}$ ratios, however it relies on equilibrium of sample nitrogen, thus causing possible errors at high enrichments.

The normal method is to switch between the two formulae at a particular enrichment. The level of enrichment that is decided is dependant on the sample nitrogen. i.e. how well it is equilibrated and its NO concentration.

# Hydrogen Calculations

The deuterium to hydrogen ratio is measured by the intensity ratio of the $H_2^+$ and $HD^+$ peaks at masses 2 and 3 respectively. However there is also an interfering peak at mass 3 due to $H_3^+$ formed by an ion molecule reaction in the source. The intensities $\left[HD^+\right]$ and $\left[H_3^+\right]$ combine to give the measured mass 3 intensity. Therefore the mass 3 to mass 2 ratio can be expressed.

$$\frac{\left[HD^+\right]+\left[H_3^+\right]}{\left[H_2^+\right]} = \frac{\left[HD^+\right]}{\left[H_2^+\right]} \times \frac{\left[H_3^+\right]}{\left[H_2^+\right]}$$

which is simply the sum of the desired deuterium to hydrogen ratio and the undesired $H_3^+$ correction ratio.

The intensity of the mass 2 peak is a direct measure of the hydrogen pressure and the effect of the interfering peak may be determined by its variation with the hydrogen pressure, measured by the intensity at mass 2. As the $HD^+$ ion is formed directly from the input gas,

$$\left[HD^+\right]\alpha\left[H_2^+\right]$$

whereas the $H_3^+$ ion is formed by binary collision, making it doubly dependent on hydrogen density, thus

$$\left[H_3^+\right]\alpha\left[H_2^+\right]^2$$

Therefore the desired deuterium to hydrogen ratio is constant, whilst the undesired $H_3^+$ correction ratio varies linearly with hydrogen density. The plot of measured mass 3 to mass 2 ratio versus the intensity at mass 2 is linear with the deuterium to hydrogen ratio as the intercept and the $H_3^+$ correction factor

$$\frac{\left[H_3^+\right]}{\left[H_2^+\right]^2}$$

as the gradient.

The $H_3^+$ correction factor is quoted in ppm/nA and the calibration is performed once prior to the series of sample measurements. This calibration is used to extrapolate to the true deuterium to hydrogen enrichment, as if the sample had been measured theoretically at zero pressure, where no $H_3^+$ ions would have been formed.

# Other Analysis Considerations

**Note:** The following sections are provided to allow users to fully understand the data reduction algorithms used in the program. Users may wish to skip this section on first reading.

# Sequence of data reduction (General case)

The following calculations are performed where necessary for any gas:

1. Convert the reference gas deltas to the standard format for further calculation.
   For example if $CO_2$ is being analysed and the reference deltas are input as delta(13) and delta(18), it is necessary to convert these values to delta(45) and delta(46) for further use. This is done automatically if required using the Craig formulae.

2. Convert the sample delta, measured with respect to the laboratory standard, to the delta w.r.t. the international standard.
   This is done using the following formula:

   δ(sam w.r.t. international) = δ(sample) + δ(ref w.r.t. international) + δ(sample) * δ(ref w.r.t. international) / 1000

3. Convert the observed delta values of a gas comprising more than one element to the delta values for the pure element.

The most familiar example is probably the Craig correction for $CO_2$ mentioned above. The other common case is for sulphur analyses using the 64 and 66 peaks of $SO_2$. To allow these calculations to be undertaken in the general case a matrix is set up using constants from the parameter file (only accessible at supervisor level) such that:

$$\delta1(corrected) = C1 * \delta1 \ - \ C2 * \delta2(corrected)$$

and

$$\delta2(corrected) = C3 * \delta2 \ - \ C4 * \delta1(corrected) \ .$$

This format has been chosen to closely represent the original Craig formulae. A little thought shows :

|    | $CO_2$ | CO | $SO_2$ | $N_2$ | HD |
|----|--------|--------|--------|-----|-----|
| C1 | 1.0676 | 1.0378 | 1.09 | 1 | 1 |
| C2 | 0.0338 | 0.0169 | 0 | 0 | 0 |
| C3 | 1.0010 | 1.0010 | 0 | 1 | 0 |
| C4 | 0.0021 | 0.0021 | 0 | 0 | 0 |

This general approach allows most gas species to be analysed by determining the values of the four constants. It also allows for non standard collector arrangements to be utilised by careful choice of constants.

A number of preparation systems rely on equilibrium states being reached, rather than reactions proceeding to completion. Examples include the carbonate reaction with acid, the carbon dioxide equilibration over water for oxygen analysis and the hydrogen equilibration over water for hydrogen analysis. The equilibration can be treated in a general way as

$$\delta(true) \ = \frac{(\delta \ (measured) \ -1000 * (E - 1))}{E} \ + K * ( \ T(reaction) \ -T(standard))$$

By specifying E as 1 and K as 0 this formula can be applied to any delta as it will not change the result. **THIS IS THE DEFAULT AND SHOULD NOT BE CHANGED UNLESS A CORRECTION IS REQUIRED.**

For the determination of the oxygen isotope ratio in waters by equilibrium the constants are :

$$E = 1.04115 \text{ and } K = 0.17 \text{ (approx)}$$

whilst in the carbonate reaction using phosphoric acid:

$$E = 1.01025 \text{ and } K = 0.04.$$

The value of T(standard) is normally 25°C. These constants are input in the gas parameter files.

## A Simple Example for Carbon Dioxide

If the delta 13 and delta 18 of the reference gas are input these are converted to delta 45 and delta 46 using:

$$\delta 45 \ = \ (\ \delta 13 + (C2 * \delta 18\ ))\ /\ C1$$

$$\delta 46 \ = \ (\ \delta 18 + (C4 * \delta 13\ ))\ /\ C3$$

The sample deltas measured with respect to the lab gas are then modified to be w.r.t. the international standard using the formula given in (2) above.

We can then use the Craig constants again to calculate the delta 13 and 18 of the sample gas relative to the international standard by:

$$\delta 13 \qquad = ((\ C1 * \delta 45\ ) - (\ C2 * C3 * \delta 46\ ))\ /\ (\ 1 - C2 * C4)$$

$$\text{and} \quad \delta 18 \qquad = ((\ C3 * \delta 46\ ) - (\ C1 * C4 * \delta 46\ ))\ /\ (\ 1 - C2 * C4)$$

If the corrections are done in any other order the Craig constants (which apply for PDB only) would have to be re-calculated, a point often missed.

## Calculations specific to Carbon Dioxide

### Santrock and Hayes

These authors have suggested an alternative algorithm for converting delta 45 and 46 to delta 13 and 18 which has yet (at the time of writing) not achieved international recognition. They start from the general formula relating the oxygen 17 to oxygen 18 ratios of:

$$^{17}R \ = \ K(\ ^{18}R\ )^{\alpha}$$

The derivation of Craig which is used by most users assumes that the constant ($\alpha$) has the value of 0.5 and permits an exact derivation of the corresponding equations to determine delta 13 and 18. In the general case considered by the above authors an iteration approach is required. The values of the constants K and a also are not well determined. At the time of writing the "best" values appear to be:

$$\alpha \qquad = \ 0.516 \text{ and}$$

$$K \qquad = 0.0099235.$$

Two other constants are required for this analysis, namely the carbon 13 to carbon 12 ratio and the oxygen 18 to oxygen 16 ratio in the international standard PDB.(The oxygen 17 ratios are obviously determined in the calculation using the above formula). These values are:

$$^{13}R_{PDB} \ = 0.0112372$$

$$^{18}R_{PDB} \ = 0.002079$$

**If the Santrock method is required the user is responsible for determining that the constants employed are correct, otherwise the final reported data will be in error**.

These constants can be inserted in the parameter file, accessible only at supervisor level. For further discussion of this technique the reader is referred to the original papers:

Santrock, Studley and Hayes: Anal. Chem **57** 1444-8 (1985)  and

Bakke, Beaty and Hayes - Paper presented at GSA 1991.

Finally it should be noted that (obviously for consistency) if this approach is to be used the reference gas  deltas should be converted to a delta 45 and delta 46 value by the same technique off-line, and these values loaded into the appropriate parameter file. If the delta 13 and 18 values are input the Craig method is automatically employed to do this calculation.

## SMOW

Since two international standards for oxygen isotopes exist, PDB and SMOW, it is necessary to be able to report the analyses relative to both standards. The formula used is:

$$\delta 18(SMOW) = \delta18 * E + 1000 * (E\text{-}1)$$

Where the equilibrium constant E has the value 1.03086.

## Atom %  Carbon

Once the enrichment  of the sample has been determined relative to a reference, it is straightforward to calculate the carbon atom % composition of this sample, using the known composition of the reference. The three constants used in this calculation (13R, 18R and 17R) specified in the parameter file supplied (accessible at supervisor level only) refer to the international standard PDB. **If an alternative standard is required it is left to the user to determine the required constant values.**

# Calculations Specific to Nitrogen

## Atom %  Nitrogen

Once the enrichment  of the sample has been determined relative to a reference, it is straightforward to calculate the nitrogen atom % composition of this sample, using the known composition of the reference. The single constant used in this calculation ($^{15}$R) specified in the parameter file supplied (accessible at supervisor level only) refers to the international standard of the nitrogen in the air. **If an alternative standard is required it is left to the user to determine the required constant value.**

## Enriched Nitrogen Analysis

It is sometimes necessary to determine the nitrogen isotopic composition of a sample which is close to natural but where the $^{15,15}N_2$ is not in thermodynamic equilibrium with the $^{14,14}N_2$ and $^{15,14}N_2$. In this case the simple formulae given above may not be used and the analysis must take account of the 28, 29 and mass 30 signals. A problem is often encountered with trying to use the mass 30 signal in that the recorded signal is due not only to a contribution of nitrogen (which is the signal of interest), but also a large contribution from NO is often seen, especially if even a <u>minute</u> trace of air is present in the samples being analysed.

To permit this analysis to be undertaken the following approach is used:

- The delta 29 and delta 30 of the sample is calculated in the normal manner.

- From the calculated atom % of the reference gas (obtained from the input atom percent of the international standard and the reference gas delta value) the expected 30 amu signal is calculated and the <u>difference</u> between this value and the observed is assumed to be the unwanted contribution from NO. This calculation assumes that the mass 30 of the reference gas **is** in thermodynamic equilibrium (otherwise no method exists to determine the NO signal in the general case).

- The assumption is also made that this contribution is also present in the sample gas (i.e. that they have both similar oxygen impurities or that the major source of oxygen is elsewhere, e.g. from the filament ) and the mass 30 amu signal modified accordingly. The correction used is weighed so that at natural abundance the full correction is used whilst at 100% enrichment the correction falls to zero.

- This corrected 30 signal is then the value used in the subsequent atom % calculation.

  **If it is felt this approach is inappropriate for the samples being analysed it is left to the user to convert the measured deltas to atom % off line.** The above method has proved quite successful at a number of sites where labelling with $^{15,15}N_2$ is performed. Obviously these difficult experiments in the general case may render the above approach inappropriate, but the user should be aware of the problems which may result from this NO contamination.

# Atom Percent Formulae

We now list , for reference, the atom percent formulae use in the code. Note that these equations assume equilibrium thermodynamics in all cases except for enriched Nitrogen analyses where the 30 amu intensity is monitored. Even here the Reference gas must be in equilibrium, and this specific case is dealt with above.

## Carbon Dioxide

$$APC(^{13}C) = \frac{100\ R13}{1 + R13}$$

$$R13 = (\delta 1/1000 + 1)\ R1$$

$$R18 = (\delta 2/1000 + 1)\ R3$$

$$R17 = (0.5\ \delta 2/1000 + 1)\ R2$$

and

$$APC(^{18}O) = \frac{100\ R18}{1 + R17 + R18}$$

R1,R2 and R3 are the three reference ratios input into the constants page and refer to 13/12, 17/16 and 18/16 ratios of the international standard.

The same formulae apply to carbon monoxide since we have deconvoluted the $\delta^{29}$ and $\delta^{30}$ to $\delta^{13}$ and $\delta^{18}$.

## Nitrogen

The calculation method employed for enriched nitrogen (and samples where the 30amu signal must be considered) is given above. Here we only consider the case where the 29amu beam is used.

$$APC(^{15}N) = \frac{100\ R29}{2 + R29}$$

Thus for hypothetical gas consisting of the international standard:

$$^{ref}R29_{calc} = \frac{2\ APC^{input}}{100 - APC^{input}}$$

From the observed  sample delta (with respect to the international standard used, normally air), a calculated sample ratio $^{sam}R29_{calc}$ is obtained, from which the sample atom percent is calculated.

The APC of the international reference is input into the R1 field in the constants page.

## Oxygen

We assume that beams 32,33 and 34amu are measured and that the data is deconvoluted such that $\delta1$ refers to delta 17 and $\delta2$ refers to delta 18. Obviously the deconvolution constants must be calculated to refer to the standard gas being used. The definition of the deconvolution constants used in the program is given above.

$$R17_{sample} = ( \delta1 / 1000 + 1) \; R17_{ref}$$

$$R18_{sample} = ( \delta2 / 1000 + 1) \; R18_{ref}$$

Therefore:

$$APC(^{17}O) = \frac{100 \; * \; R17}{1 + R17 + R18}$$

$$APC(^{18}O) = \frac{100 \; * \; R18}{1 + R17 + R18}$$

The value of $R17_{ref}$ is input into the R1 field and the value of $R18_{ref}$ into the R2 field of the constants page.

## Hydrogen

The formulae given above for normal Nitrogen calculations apply here, replacing R29 with R3 in the above formulae.

## Sulphur Compounds

Since not all the sulphur peaks (due to S32, S33, S34 and S36) are monitored and there does not appear to be a recommended relationship to correlate the variations of one ratio with another, a general formula for the atom percents cannot be given.

## Nitrous Oxide (N$_2$0)

Here again we assume the observed R45 and R46 ratios have been deconvoluted by the user, calculating the constants for the laboratory standard employed and using the general deconvolution parameters as defined above. We assume further that the data is deconvoluted such that $\delta1$ refers to delta 15 and $\delta2$ refers to delta 18.

Then:

$$R15_{sample} = ( \delta1 /1000 + 1 ) * R15_{ref}$$

Where:

$$R15 = \frac{APC15}{100 - APC15}$$

With the APC15 of the reference gas input into the R1 field of the constants page the value of $R15_{sample}$ is calculated, and the APC of the sample evaluated using:

$$APC = \frac{100 \; * \; R15}{1 + \quad R15}$$

The Oxygen APC is calculated using the same method as with Carbon Dioxide above.

**NOTE:** The different definitions for nitrogen and oxygen in these fields.

# Section 4



# Equipment Description

*Equipment Description*

# Introduction

The PRISM has been designed for modular construction so that all options and accessories can be added to the basic instrument. An important benefit for the user is that this makes it easy to upgrade the system, so that it can grow to meet your changing requirements. This section describes the core design of the system, including all the components of the basic instrument, as well as the HD option and Differential Pumping option.

The PRISM has a space efficient design with low level bench tops, which give good ergonomics for a pleasant laboratory environment. All the main components are located for ease of access and maintenance.

Above bench top height the major modules are:

- Ion Optics.

- Dual Inlet system (if fitted).

- Manifold (if fitted)

- HD option (if fitted)

- Differential Pumping option (if fitted)

  Below bench are located:

- Electronic Units.

- High vacuum pumping system.

- Electrical wiring assembly, mains switch and circuit breaker panel.

- Utility inputs and output panels

  The low vacuum pumps (rotary pumps) are in a separate assembly outside the bench.

  When fitted, sample preparation systems join on to either side of the main bench as bolt on modules (please refer to the appropriate section of this manual for details).
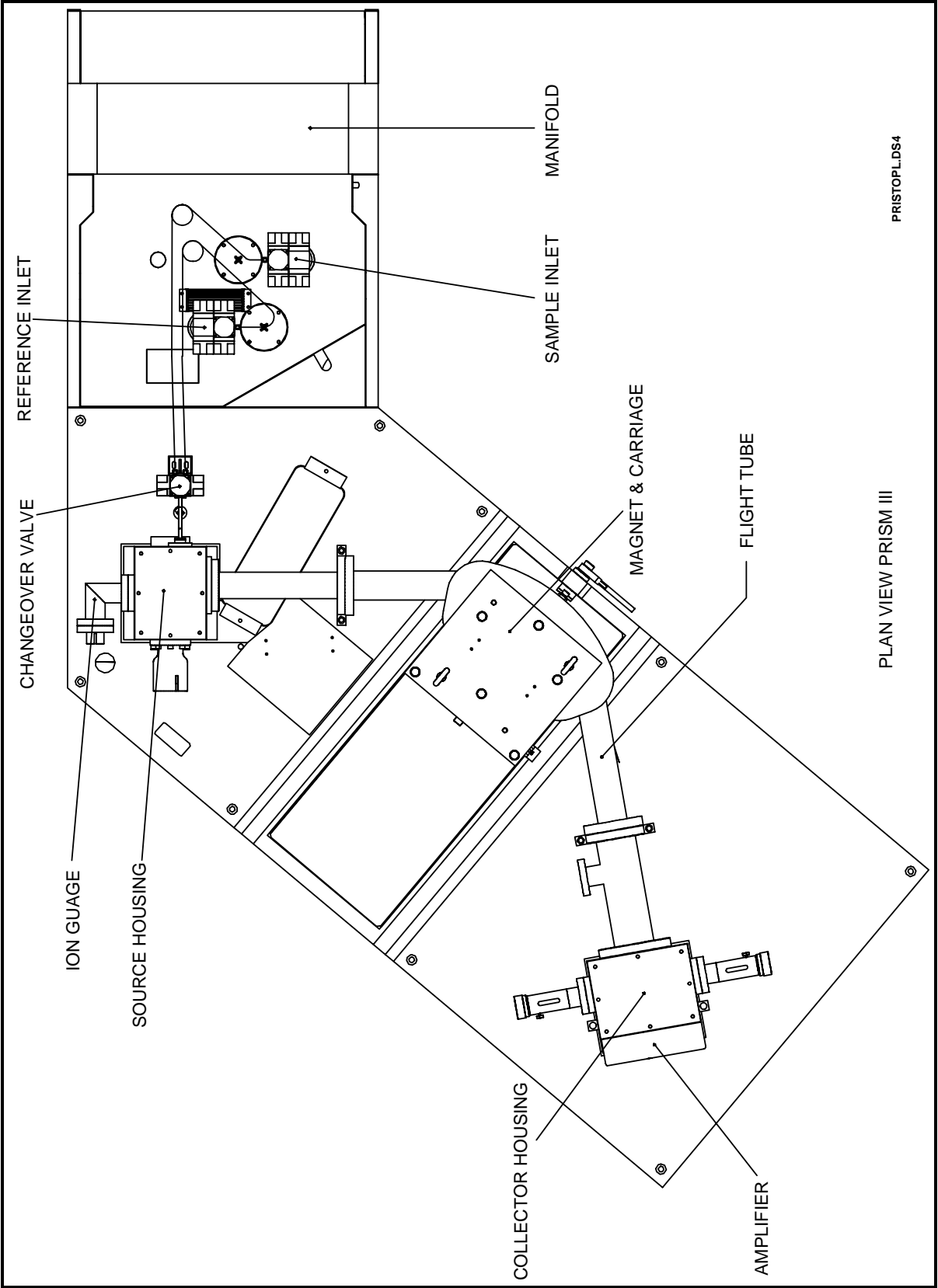
**PRISM Plan View**

# Ion Optics

## Overview

The ion optics of the PRISM have many new and unique features which we will discuss in detail in this section of the manual.

The first major feature of the manufacture is that the ion optics are mounted on a single cast table top which has been machined using the latest computer controlled techniques. The cast table top is then mounted on a steel frame to provide a mechanically rigid base, which ensures optical alignment of all the ion optics, even when adding options at later time.

The manufacture of the housings follow the successful concepts of the OPTIMA and are again machined using computer controlled tools so again optical alignment is ensured. The stainless steel optics housings therefore require an absolute minimum of flanges, which has great benefits in vacuum integrity.

The ion optics have a horizontal geometry, and the design allows individual access to the source and collectors through the lids of the housings. Routine maintenance is therefore extremely easy: it is possible to change filament without removing the ion source. The turbomolecular pumps are mounted directly to the base of the housings to give totally unrestricted high vacuum pumping, which produces very good abundance sensitivity, very low backgrounds, and minimum inter-sample memory.

## Ion Source

The ion source is an evolution of previous versions of a field proven source. It features improved accessibility and ease of maintenance, a new rapid mounting system, and many detail changes for improved performance. It retains all the benefits of low $H_3^+$ contribution, high sensitivity, and linearity which are characteristic of Stable Isotope Ion Sources.
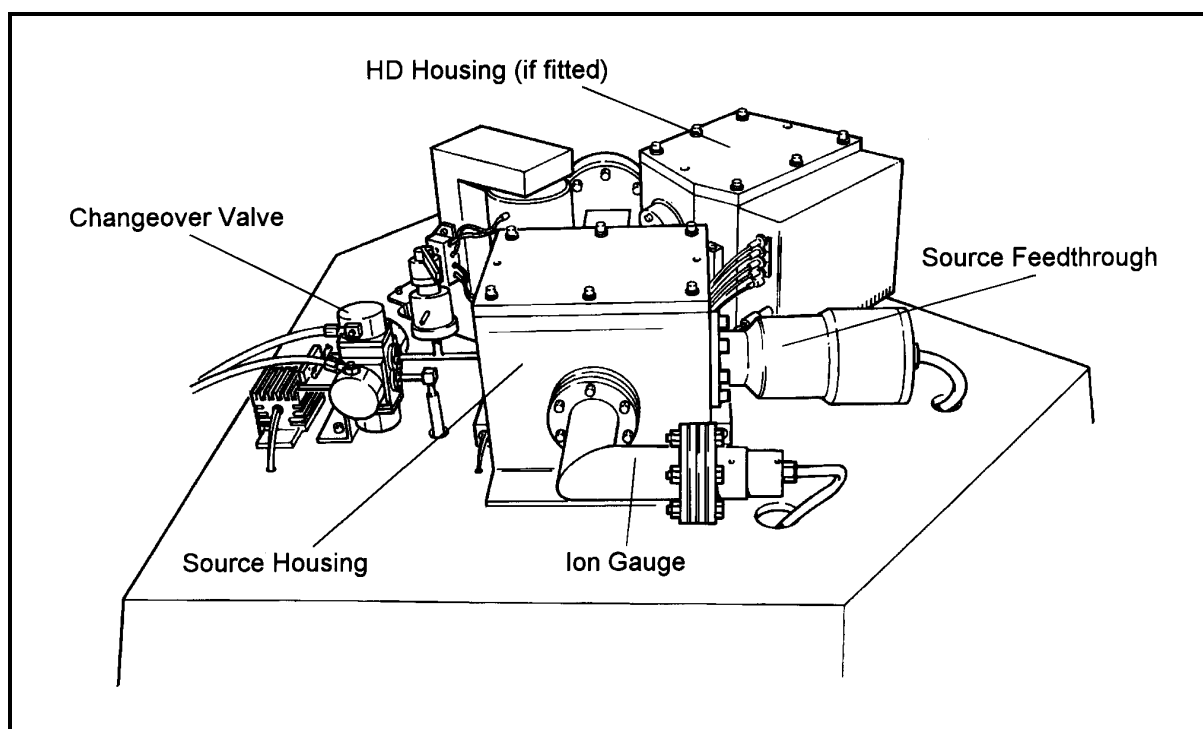


**Source Housing**
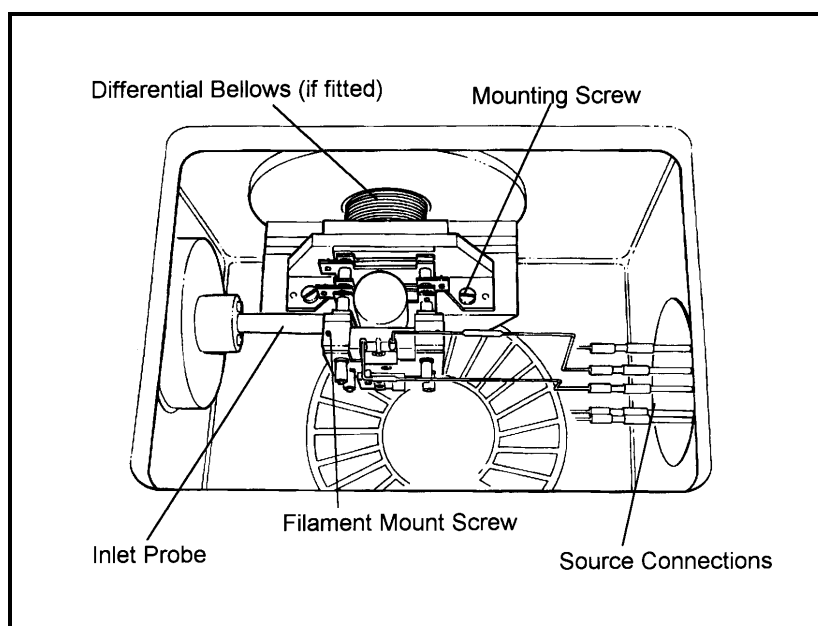
**Ion Source mounted in the Source Housing**

The ion source is a small chamber in which gas molecules are ionised by collisions with electrons. This is why it is known as an Electron Impact (EI) ion source. The electrons are emitted by thermal excitation from an incandescent wire filament, and are accelerated through the source by a small voltage between the filament and source (electron volts) of 50 -100 volts. The electrons are collimated by a narrow electron entrance slit opposite the filament, and follow a helical path through the source under the influence of the magnetic field produced by two small permanent magnets (the source magnets). This raises the ionisation efficiency by increasing the probability of collisions between gas molecules and electrons.

The majority of electrons which do not cause ionisation's are collected at the Trap (to produce the Trap current). The current through the trap is used in a feedback loop to control the emission from the filament via the filament current. This is known as a trap-regulated system, and provides a constant flow of electrons through the source.

The source block is a machined component which forms the base and two sides of the ion source block. In it is machined the gas inlet aperture, where the ceramic inlet probe admits gas to the source. Adding the "Wrap-around" to the source block forms the source 'box'.

The "Wrap-around" is a photo etched sheet which forms the top surface and two of the sides of the source box. It contains the electron entrance (filament side of the source) and exit (trap side) slits on opposite side faces, and the ion exit slit on its top face.

The ion repeller is a flat electrode at the base of the source box, opposite the ion exit slit, which is used to move the region of ionisation with respect to the slit.

The half plates are two electrodes which provide a focusing effect on the ions as they emerge from the ion exit slit, and also x-y beam steering.

The defining (source) slit ensures that the ion beam is well defined as it leaves the source and is held at earth potential to collect any scattered ions.

The Z plates are two electrodes which provide beam steering in the Z plane.

The final alpha plate defines the maximum beam width at that point prior to the ion beam entering the flight tube on its way to the collectors.

The source voltages and currents are variable and depend on the gas species to be analysed. These voltages and currents are supplied from the electronics units and controlled via the computer (please see later sections of this manual for details of tuning the source and electronic units). The connections to the source are made via a single feedthrough flange located in the source housing.
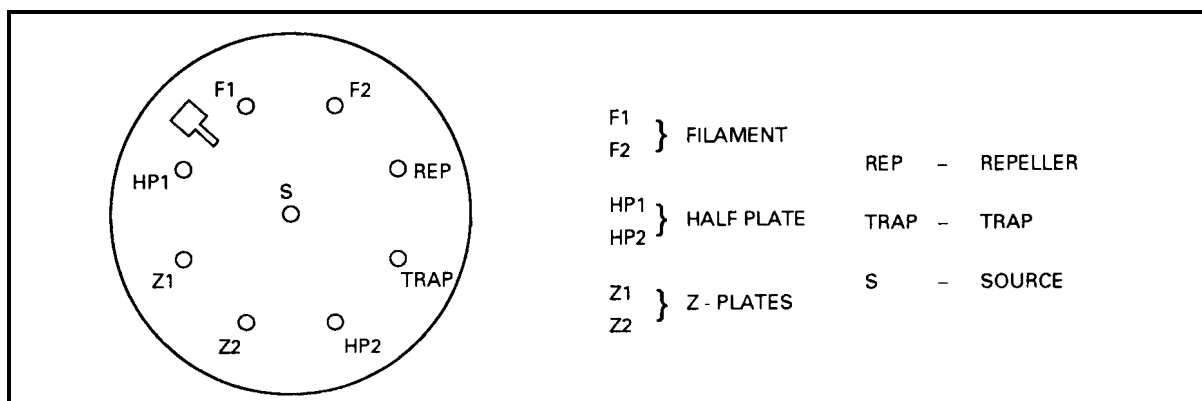


**Source Feedthrough Connections**

# Gas Inlet Probe

The changeover valve of the Dual Inlet is connected to the inlet probe flange by a short stainless steel pipe. All connections are made with gold gaskets. The inlet probe consists of a tube made from machinable ceramic to insulate against the high acceleration voltage of the source. The probe is spring loaded as a piston assembly within the stainless steel connecting flange. This means the probe has positive contact with the source, ensuring the gas path from inlet to source through the probe is continuous and gas tight. This is further helped by the flat face at the source end of the probe which mates to the source 'Wrap-around'.
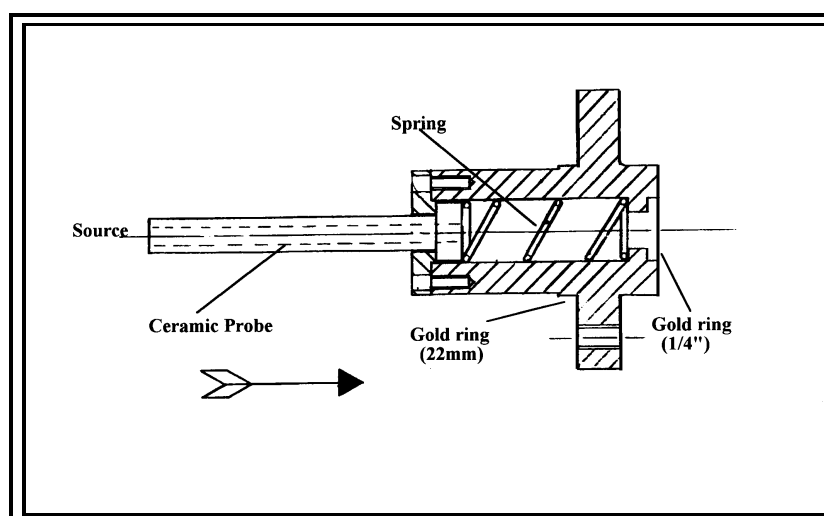


**Inlet Probe Assembly**

# Electromagnet

The Electromagnet is mounted on a carriage which moves the magnet on precision bearings from front to back to position it over the flight tube. When the magnet is in approximately the correct position the carriage is fitted with a ratchet drive for final adjustment. A screw fitted in the front of the carriage provides a stop so that the magnet can be returned to the same position, for example after bakeout of the system. Movement from side to side is restricted as the carriage runs in a trough machined in the table casting. The magnet carriage also allows the magnet to move up and down around the flight tube as the magnet sits on three adjustable screws. Accurate adjustment of the magnet position is therefore provided by the magnet carriage, providing control of x-y-z axes.
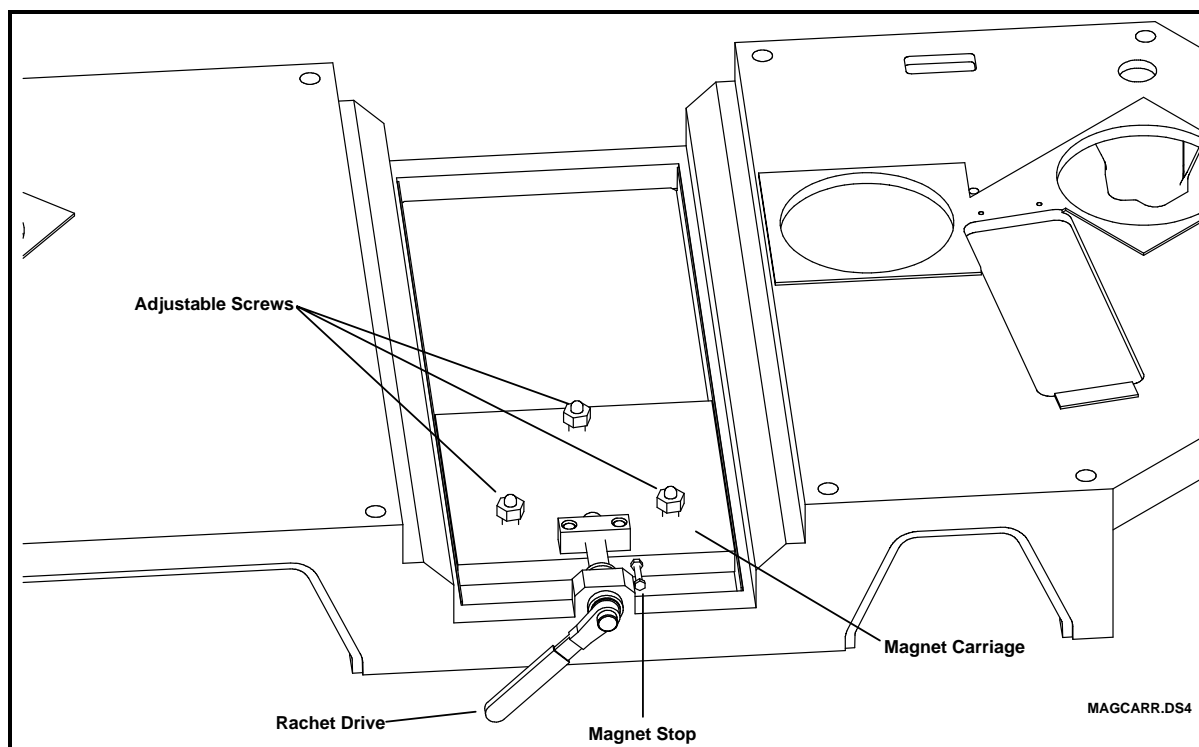


**Magnet Carriage**

The Magnet also has an adjustable exit pole angle which allows for rotation of the ion beam to give the best available peak shapes (please see later section of this manual for details). The exit pole is of the patented curved, design which results in the image of the ions being normal to the central ion trajectory, so the collectors lie in the plane normal to the ion trajectory.

As with the source supplies the magnet current is variable and depends on the gas species. This current is supplied from the Magnet Supply unit and magnet card in the system controller, and is fully variable via the computer (please see later sections of this manual for details of changing the magnet current and Magnet Supply). The connection to the magnet are made via a single cable running through the bench.
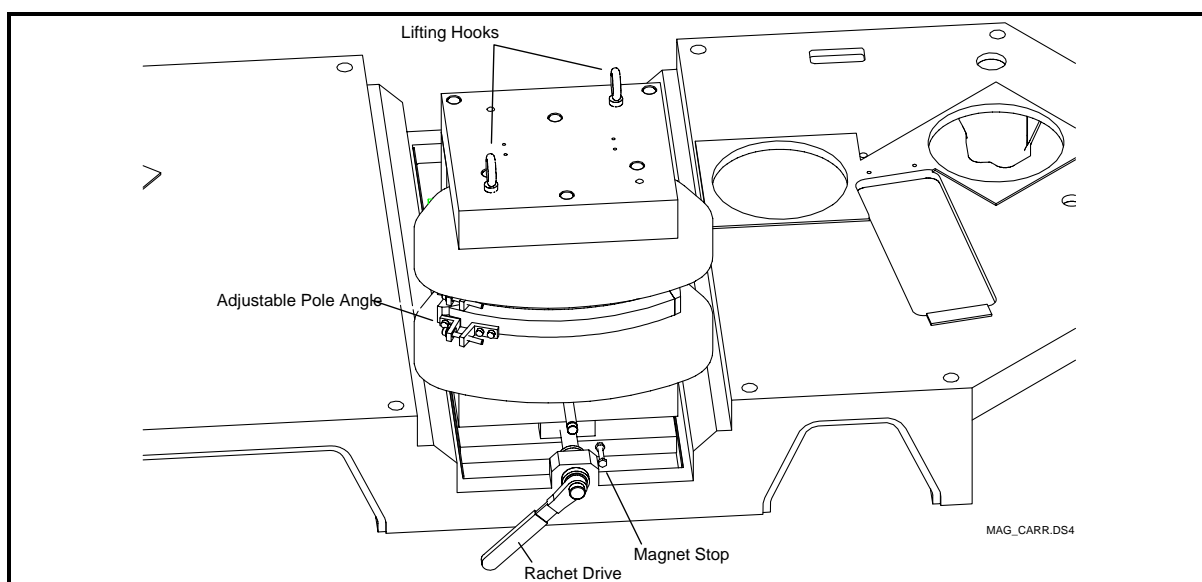
**Magnet (magnet and carriage)**

**Note:** The magnet can also be fitted with a Hall Probe, to provide feedback to the magnet supply (see later sections of this manual for details).

# Flight Tube (Analyser Tube)

The Flight Tube is made from stainless steel to the latest vacuum specifications and incorporates a horizontally mounted flight tube, with 80° symmetric geometry, giving an effective analyser dispersion of 50cm. The analyser tube is made up of three sections which allow for alterations to the system when adding the various options (i.e. HD and/or Differential Pumping - see later sections of this manual).
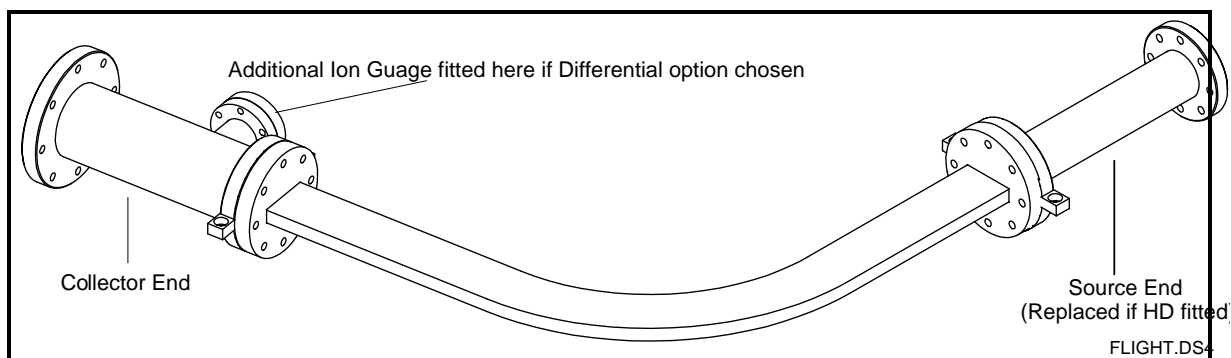


**Flight Tube (Analyser Tube)**

The centre section of the Flight Tube is the part which sits between the pole pieces of the magnet and is connected at each end to the other two sections via FC64 copper flanges. The source end section is made from a straight stainless steel tube which connects to the source housing. This section is replaced with the HD spur if the HD option is chosen. The collector end section is again made from a straight tube connecting to the collector housing. This has an additional flange on the side of the tube to allow for an analyser Ion Gauge in the case of Differential Pumping.

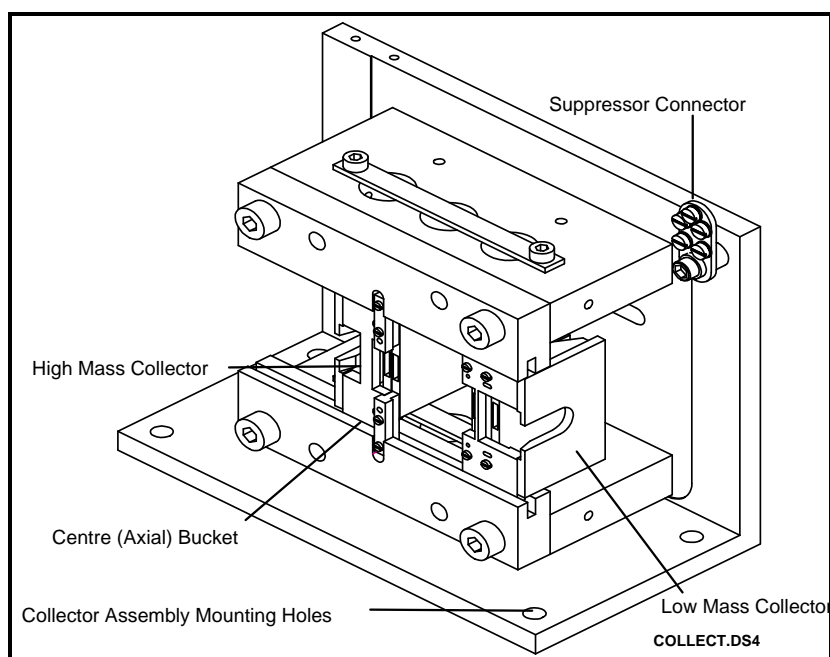**Note:** The end sections are as large as possible to give the best pumping speed to the centre section.

# 'Multi' Collector

The PRISM uses an adjustable quadruple collector arrangement. The collectors are mounted into a housing similar in design to the source housing, access is therefore gained via the top, making adjustment and maintenance easy.
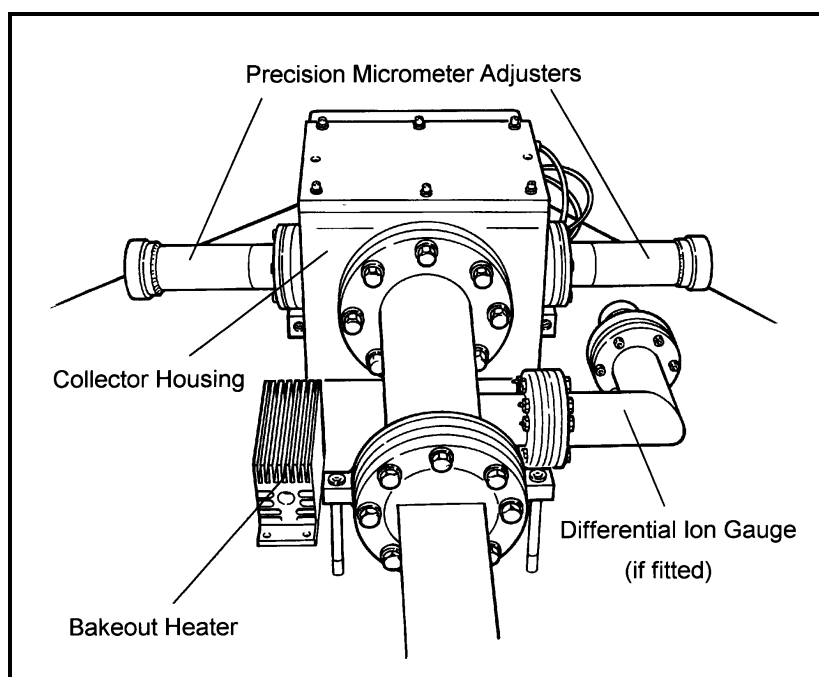


**Collector Housing**



**PRISM Collector**

The 'Multi' collector has four identical deep Faraday buckets with integral suppressers. The centre bucket is fixed (i.e. it is not adjustable) and the outer buckets are externally movable using precision micrometer adjusters. The two low mass collectors are mechanically strapped together (but electrically isolated from each other) and therefore move as a pair when using the external adjuster. This is to enable both $CO_2$ and $N_2$ to be measured without adjusting the collectors, with mass 28 measured in the low 2 collector and mass 44 in the low 1 collector (see diagram below for details of which mass is measured in which collector).The two low mass collectors can be moved in relation to each other with the screws provided, but this requires the vacuum to be broken (these collectors are factory set and should not need to be readjusted).
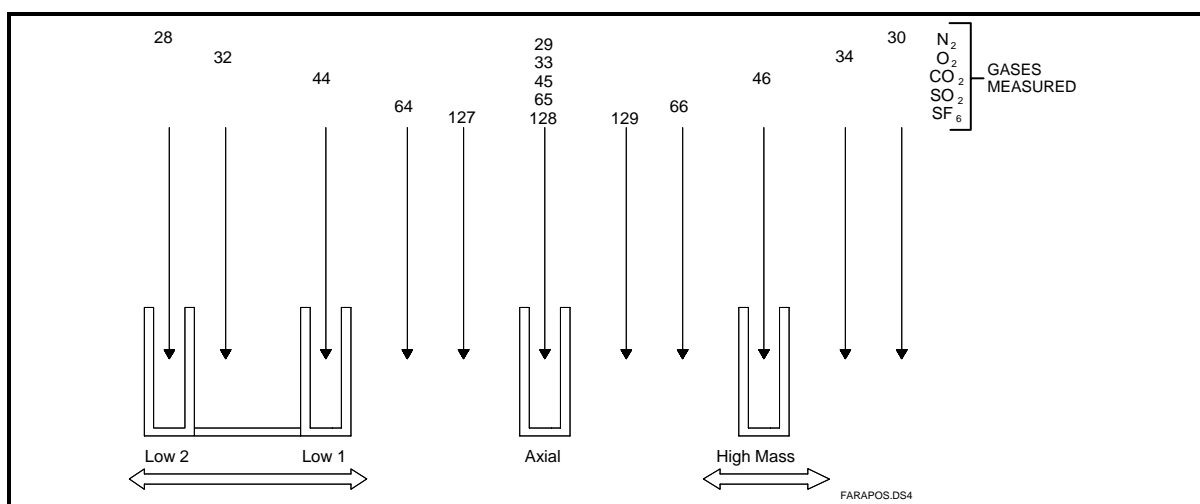


## Collector Schematic (Beam Mask)

This arrangement permits any combination of masses to be studied without the need to break vacuum. The software decides which collector signal is to be measured depending on the mass selected (see later sections for details).

## Faraday Buckets

Each Faraday bucket is individually shielded by a stainless steel jacket to ensure unwanted charged particles are not recorded. The electron suppresser plate at approximately -43V is mounted immediately after the resolving slit to reject secondary electrons generated by ion bombardment. An earth shield is placed between the suppresser electrode and the Faraday cup to prevent charge leakage down the mounting ceramic, which could lead to spurious offset signals. External magnets are also fitted above and below the bucket assembly to further minimise secondary electrons.
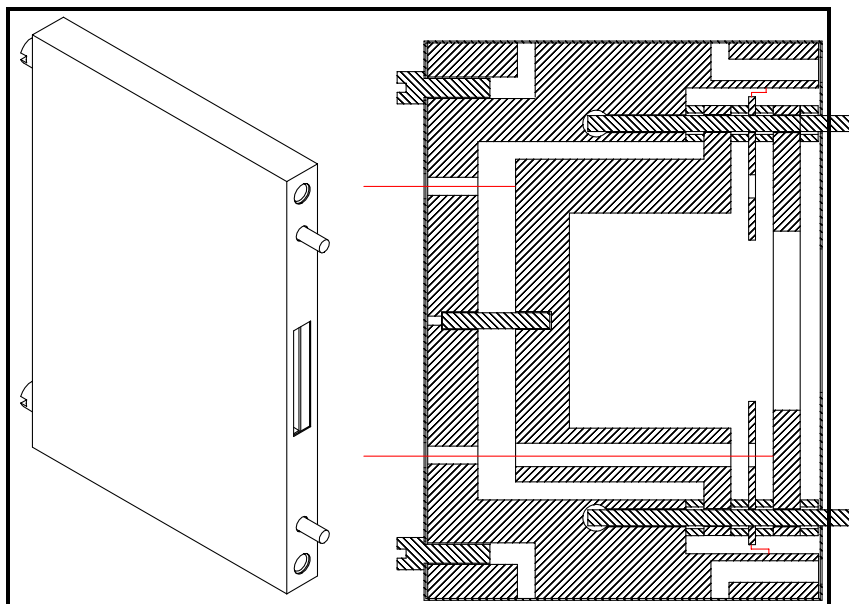


**Faraday Bucket**

The Faraday buckets are connected to the amplifier electronics via a five pin feedthrough flange (the extra feedthrough is for the suppresser) mounted in the housing and shielded to prevent spurious signals.
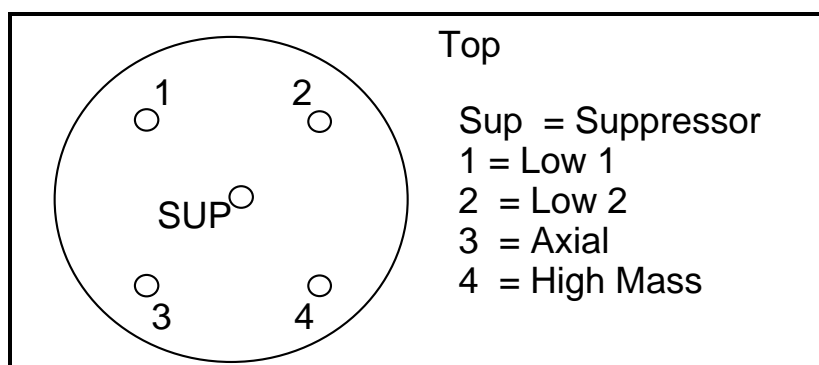


**Multi-Collector Five Pin Feedthrough Connections Outside View**

# Bake-out Heaters

Each of the housings incorporate integral sleeves for the insertion of cartridge heaters to directly heat the housings. The Flight tube section is heated using a heater tray which mounts under the flight tube when the main magnet is moved to its back position (see diagram below). There are also additional heater blocks mounted at the collector and source end of the bench, although the source heater block is removed if the HD option is fitted.



Magnet in the Rear Position

Heater

Changeover Valve Heater

Heater Tray
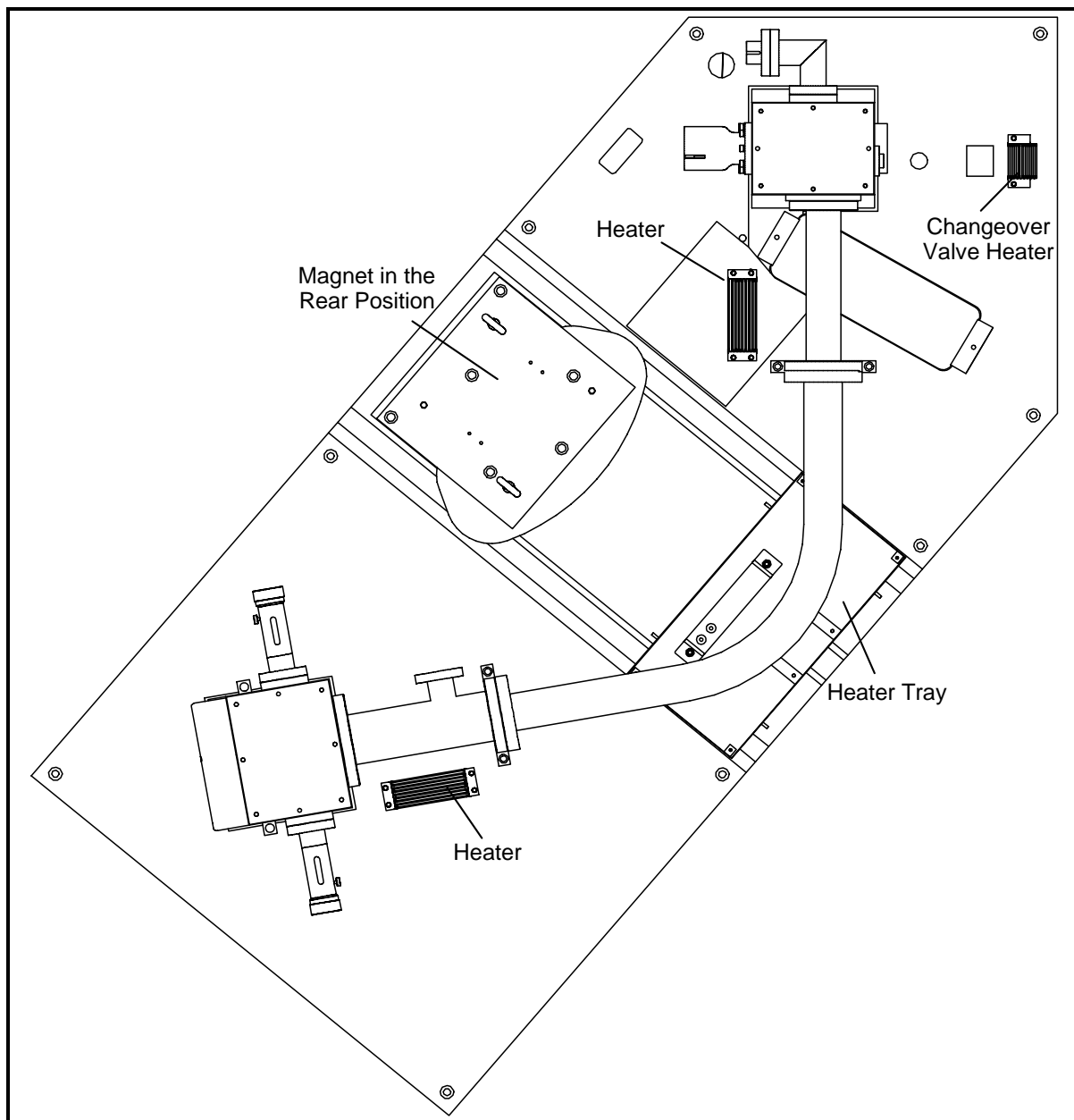
Heater

**Heater Positions**

When the system is then covered with the fabric insulating bake-out cover an even bake-out temperature is obtained. The bake-out is controlled via the software (see later sections of this manual for details) with the temperature monitored by a thermistor mounted at the source.

---

**CAUTION**

**It is essential to remove the head amp prior to baking out the analyser. See section 4 page 37.**

---

# HD Option

For Hydrogen, the ion beam separation is much greater due to the greater relative mass difference (3/2 for HD compared with 45/44 for $CO_2$), necessitating a separate analyser assembly for Hydrogen analysis. This consists of flight tube spur, magnet, magnet carriage and collector assembly.
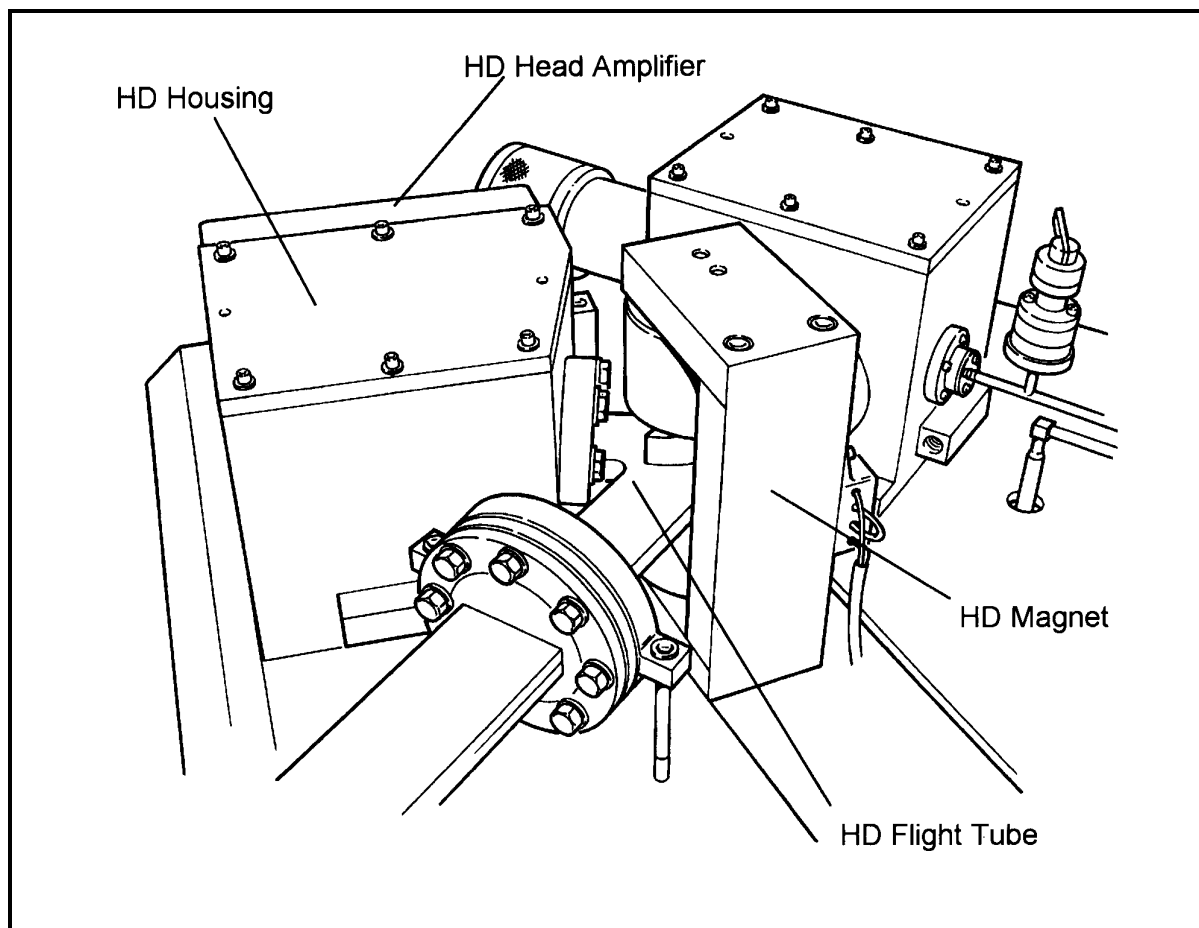


**HD Option**

## HD Spur

This is the flight tube for the Hydrogen analyser and replaces the source end section of the main flight tube. It uses the same construction techniques used for the main flight tube. The ion beam can be directed to either of the two collector assemblies depending on the position of the HD magnet.

## HD Magnet and Carriage Assembly

The HD magnet is mounted on a pneumatically operated magnet carriage which allows the HD magnet to be moved into position via the computer. Stops are provided so the magnet returns to the same position each time. The magnet carriage allows the magnet to be adjusted in all planes prior to fixing the stops, so the best peak shape can be obtained (see later for details).
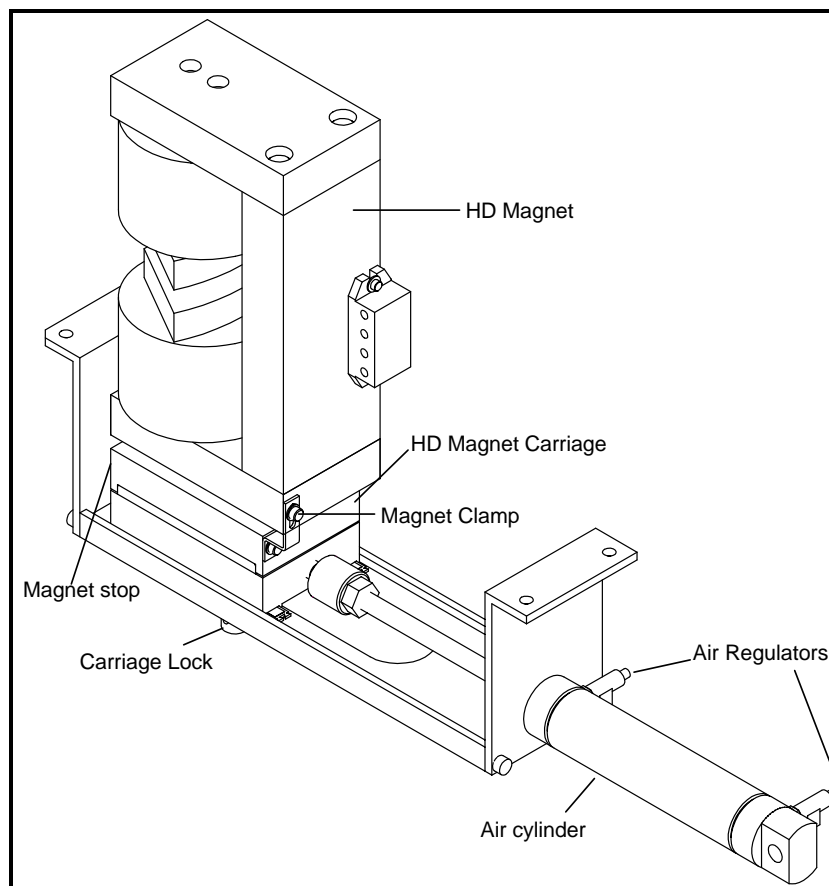


**HD Magnet and Carriage**

## HD Collector Assembly

The HD collector assembly contains two deep Faraday buckets, (similar in design to the main collector Faraday buckets) one for each Hydrogen mass to be measured. The collectors are mounted in a housing similar in design to the source and collector housings. Access is gained via the top, making adjustment and maintenance easy.

The high mass (mass 3) collector is fixed in position and has a narrower collector slit than the low mass (mass 2) collector which is adjustable. The mass 2 collector is factory set (and should not need to be altered) to allow mass 3 to be measured simultaneously with mass 2, (i.e. mass 3 peak shape is in the centre of the mass 2 peak). The Faraday buckets are connected to the electronics via a five pin feedthrough flange mounted in the housing and shielded to prevent spurious signals.
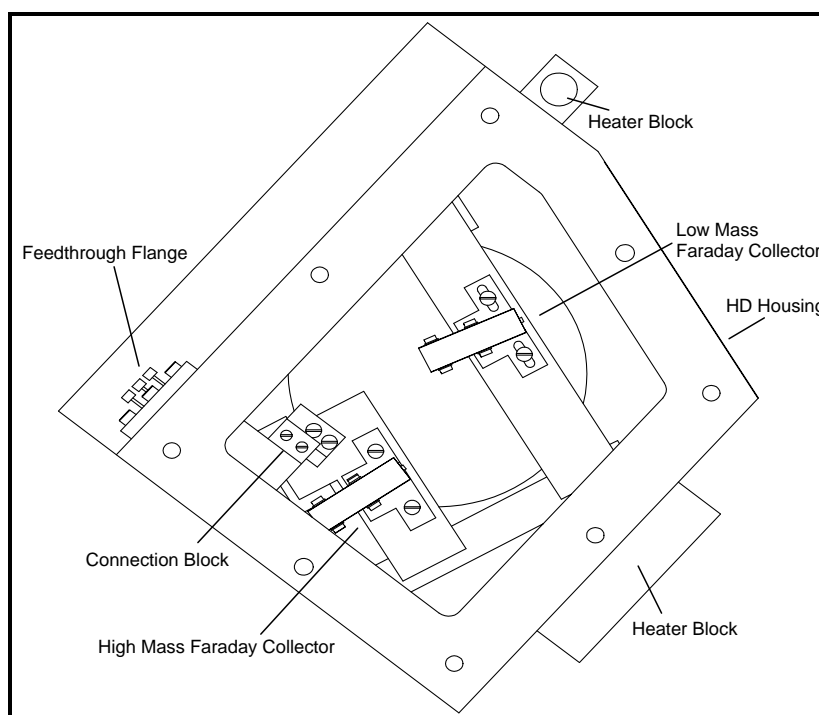


**HD Collector Assembly**
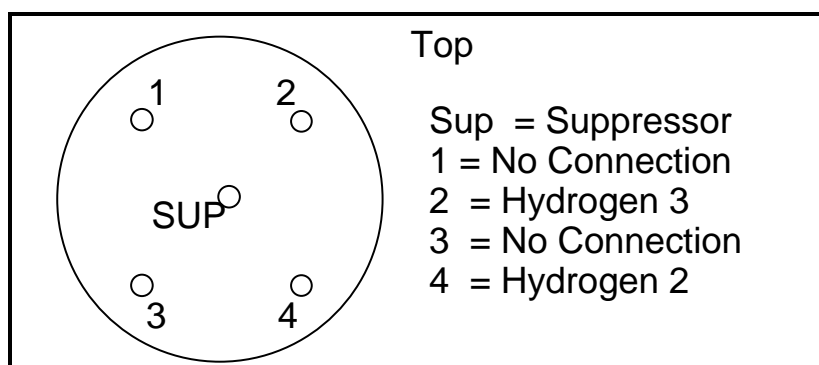


**HD Collector Five Pin Feedthrough Connections Outside View**

# Differential Pumping Option

There are two possibilities for the differential pumping option:

1. Differential Pumping without HD option

2. Differential Pumping with HD option

## Differential Pumping without HD option

An extra turbomolecular pump is mounted under the Differential Pumping Tee Chamber which replaces the source end of the analyser tube. The backing to this pump is provided from the analyser rotary pump. The pressure of the analyser is monitored using the additional ion gauge located in an elbow fitted at the collector end of the analyser tube. The vacuum at the source is isolated from the analyser by means of a bellows assembly fitted to the front of the source. This means that the only route for the sample gas into the analyser region is via the alpha slit, giving a differential pressure, as the gas flow is highly restricted.

## Differential Pumping with HD option

In this case the Differential Pumping Tee Chamber is not required as the extra turbomolecular pump is fitted directly under the HD collector housing. All other parts are as above.
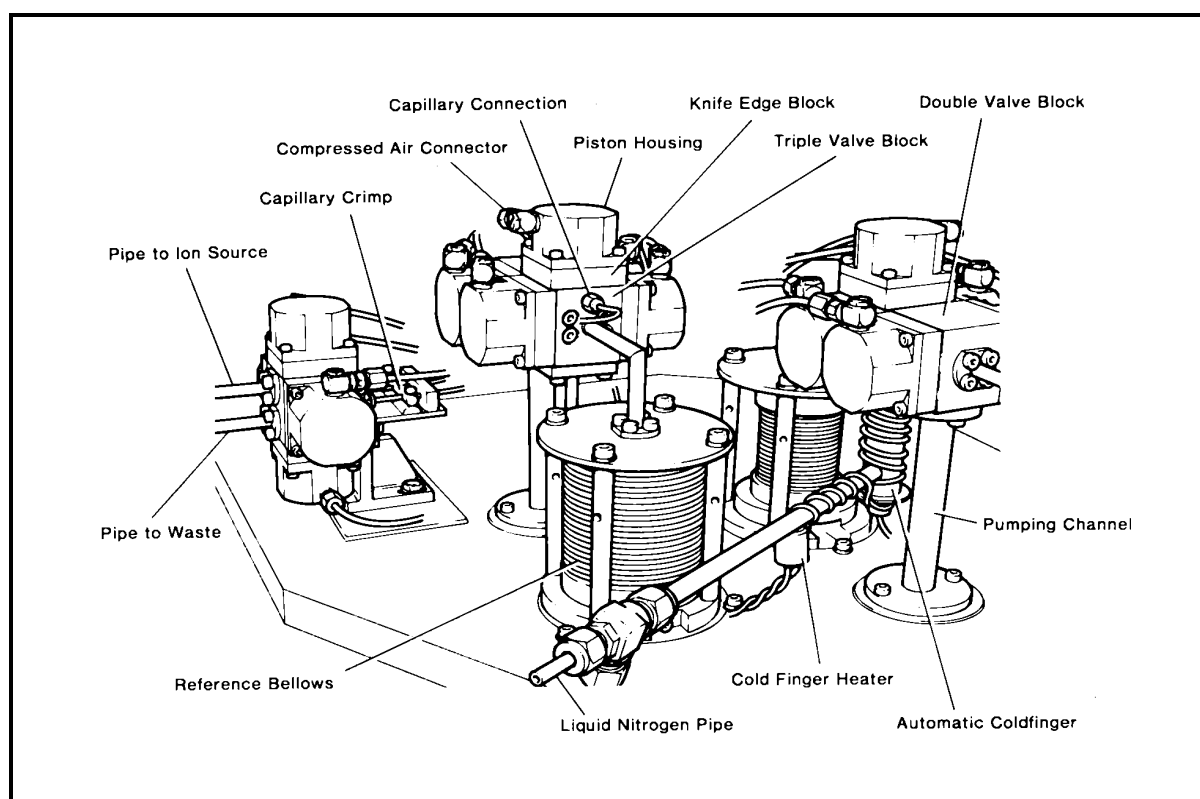
# The Dual Inlet System

## Overview

The dual inlet is built on its own cast aluminium base, (except for the changeover valve which is situated near to the source housing on the analyser bench), and can be removed as a complete module (along with its bench if necessary - to allow easy movement of the instrument). All the valve components are sited above the table for ease of maintenance. Below the table are sited the bellows stepper motors, the high and low vacuum pump valves, the pipework associated with the vacuum system and the pneumatic solenoids. The Charles Austen pump for the cold finger is mounted under the main bench.

## Principle of operation

The purpose of the dual inlet is to present identical flow rates of a reference gas of known isotopic composition and an unknown sample gas to the ion source. To achieve this it comprises two identical inlets. Each half of the dual inlet is connected to the changeover valve via capillaries which restrict the flow of gas, such that equal gas pressures in each half of the inlet produce identical ion beams at the ion source. Whilst one gas is flowing into the ion source, the other is flowing at exactly the same rate into a waste line, connected to a high vacuum pump. In this way all the relative isotopic fractionisation and mass discrimination effects associated with the gas handling and ion beam formation are held equal for both gases. Since these effects are cross cancelling, they need not be considered in the calculation of the isotopic enrichments for the samples being measured.



**Dual Inlet**

The inlet is assembled using ¼ inch gold O rings for all the joints.

**Note:** The inlet can be run manually or fully automatically from the computer when running samples from the preparation systems available with PRISM, (see later sections of this manual for details).

# Valves

There are 5 valves in each half of the inlet, 4 in the changeover valve block and 4 pump valves below the bench (although 2 of the under bench valves are used for the manifold they come as standard with the dual inlet).

Each valve is known by a two letter mnemonic. Valves in the reference half start with "R", in the sample half with "S". The other letters are:

| | |
|---|---|
| **I** | Inlet Valve, to either reference bottle or sample reparation systems. |
| **P** | Pump Valve, opening to the pump channel. |
| **V** | Variable volume or bellows isolation valve. |
| **F** | Cold Finger isolation valve. |
| **M** | Mass Spectrometer valve, opening inlet to changeover valve. |
| **C** | Changeover valve, opening to the ion source. |
| **W** | Waste valve, opening from changeover valve to waste pumping line. |

The four additional valves below bench are::

| | |
|---|---|
| **LV** | Low Vacuum pump valve, for rough pumping of the inlet. |
| **HV** | High Vacuum pump valve, for high vacuum pumping of the inlet. |
| **ML** | Low Vacuum pump valve, for rough pumping of the manifold and other accessories. |
| **MH** | High Vacuum pump valve, for high vacuum pumping of the manifold and other accessories. |

## Inlet Valves (Sample and Reference)
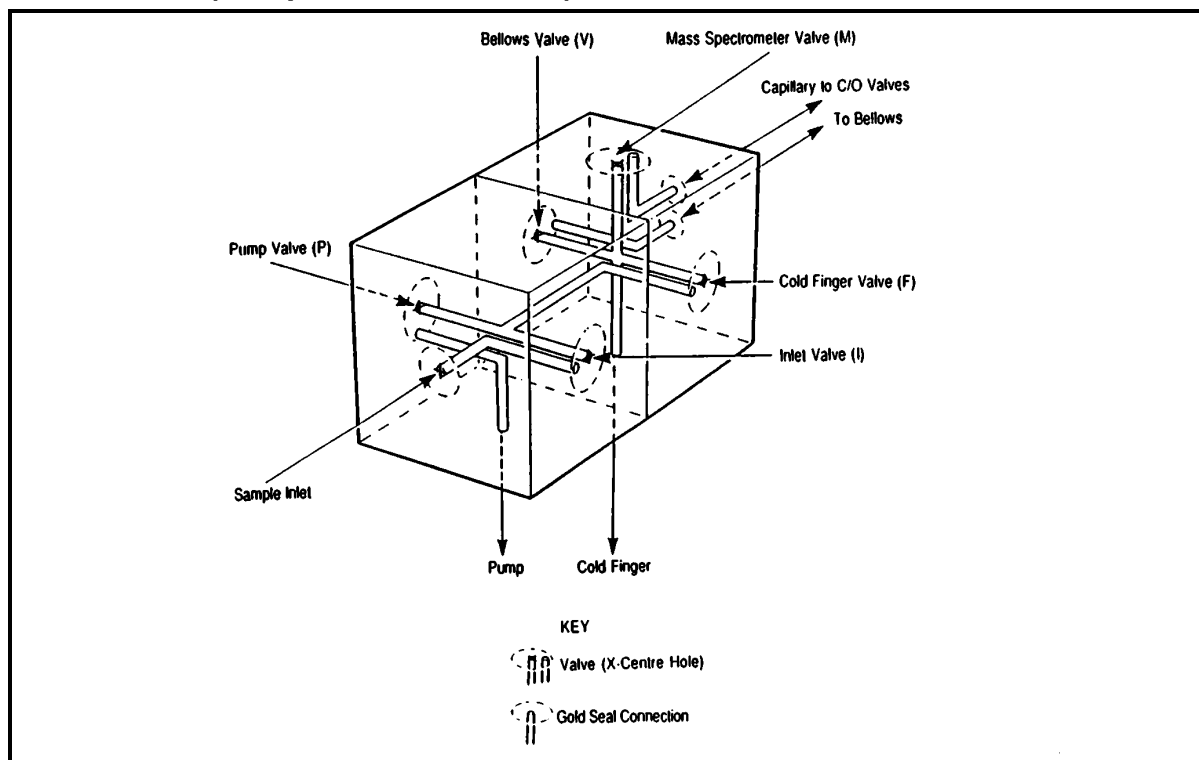


**Arrangement of Valves in Dual Inlet**

Both the sample and reference inlet valve blocks are identical and consist of a number of individual valve assemblies machined out of two blocks of vacuum grade stainless steel. The inlet valves are therefore configured in two sections: a two valve block containing the pump(P) and inlet(I) valves and a three valve block containing the cold finger isolation (F) valve, the bellows (V) valve and the mass spectrometer valve (M). The two blocks are joined with a 9.9mm gold ring. To aid identification of each valve, assignments are stamped on the blocks.

This construction minimises the distance between valves and results in a very low dead volume design with a minimum of welded joints. The volume of individual valves is less than 0.1ml and the volume of one complete valve assembly and interconnecting orifices (i.e. the analysis volume in a cold finger run)is approximately 0.2ml. The minimisation of these volumes whilst maximising gas conductance is an important design feature.

## Changeover Valve
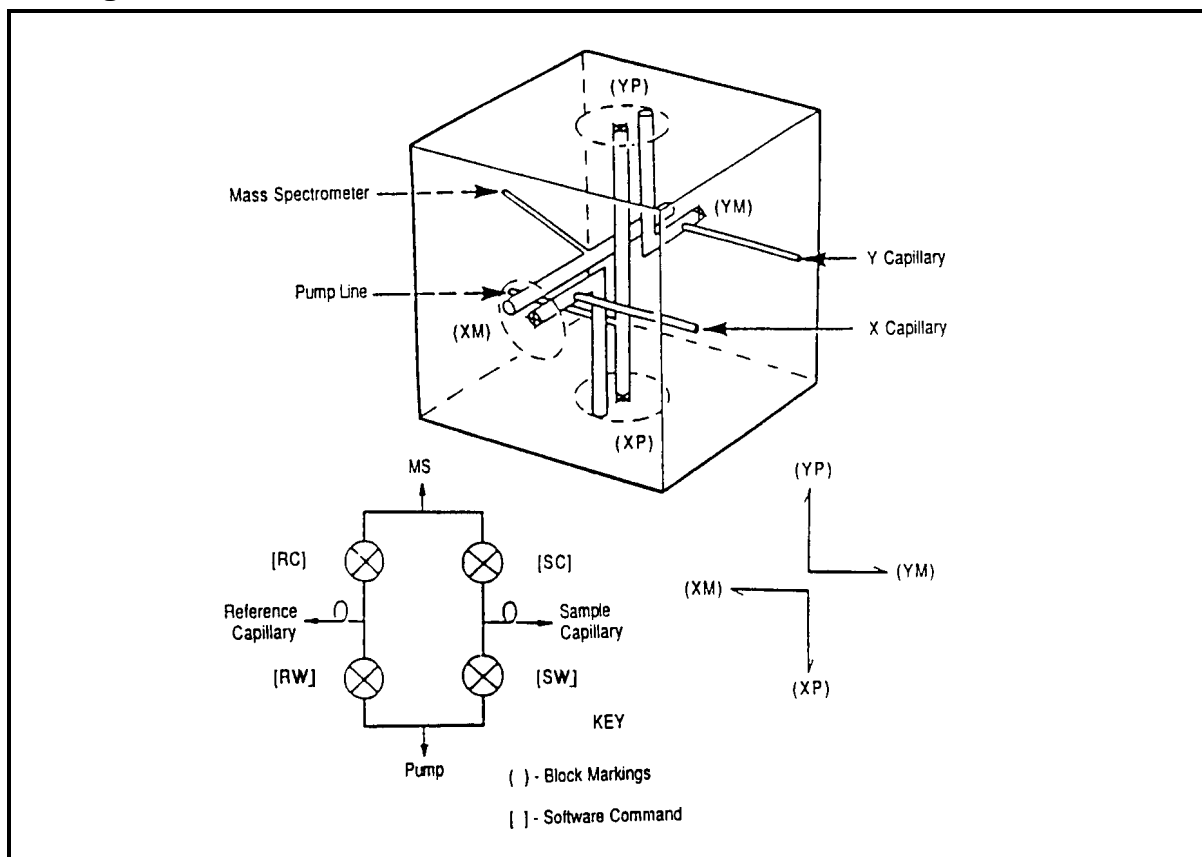


**Arrangement of Valves in Changeover Valve**

This valve assembly is machined from a single block of vacuum grade stainless steel. It makes up four valves in total which are split into two halves, two valves for the reference side, the other two for the sample side, (i.e. RC and RW for the reference, SC and SW for the sample). When in operation if RC is open (i.e. reference gas into the ion source) then RW and SC are closed and SW is open (i.e. sample gas to the waste). In this way whilst one gas is flowing into the ion source the other is flowing to the waste line.

## Under Bench Vacuum Valves (LV, HV, ML, MH) and Inlet Pumping Line
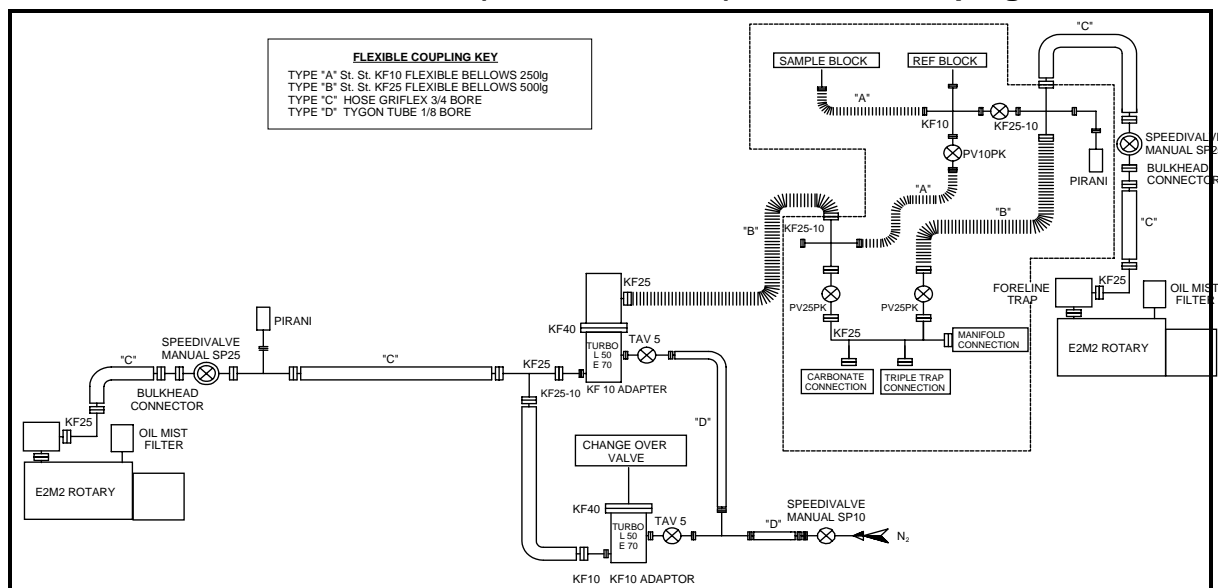


### Under Bench Inlet Vacuum Schematic

These are commercially purchased valves designed for high speed actuation from the inlet pneumatic lines. They are connected using stainless steel bellows, standard vacuum fittings. and specifically designed pipework to produce the inlet pumping line. Please note that all viton O-rings are degassed prior to build to ensure the highest possible vacuum. There is also a pirani gauge which measures the roughing pressure in the inlet prior to pumping the inlet with the turbo pump (high vacuum) and has the identification Pirani 3 in the software.

For more information regarding these valves please refer to the manufacturers manuals (Edwards PV10 and PV25).

## Pneumatic Valves

All the above valves are operated by the action of compressed air actuated pistons which <u>close</u> the valves. The compressed air supply is switched on and off by computer operated solenoid valves. The solenoids are mounted in banks on manifolds below the inlet table. These are hinged, and swing out for easy maintenance. The accompanying diagrams show which solenoid corresponds to which valve.
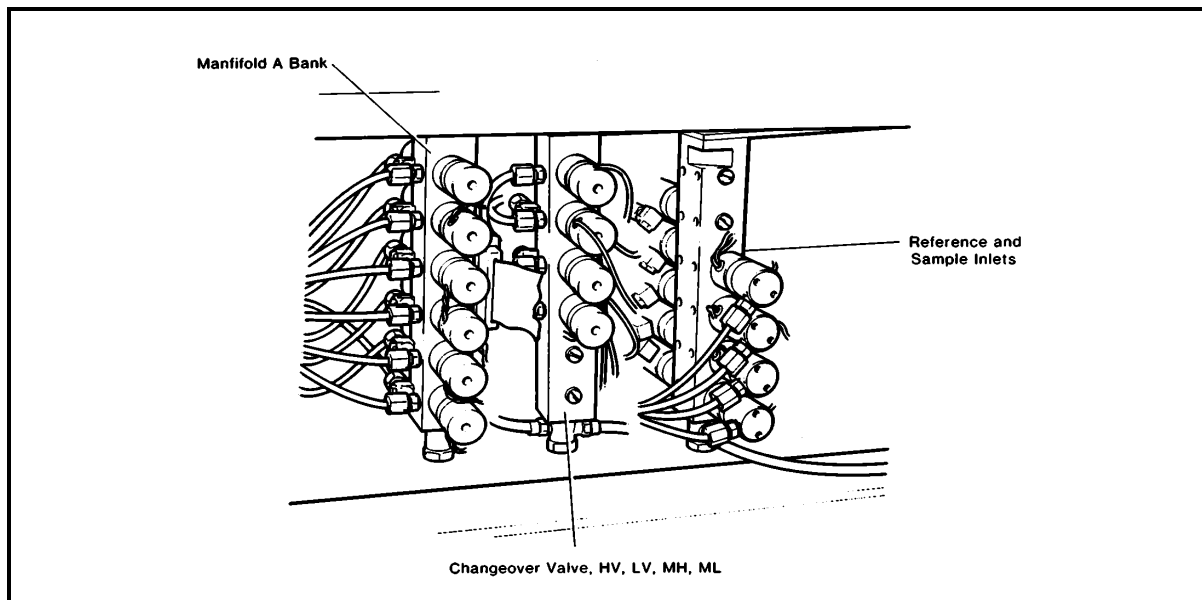


**Solenoid Valve Assignments**
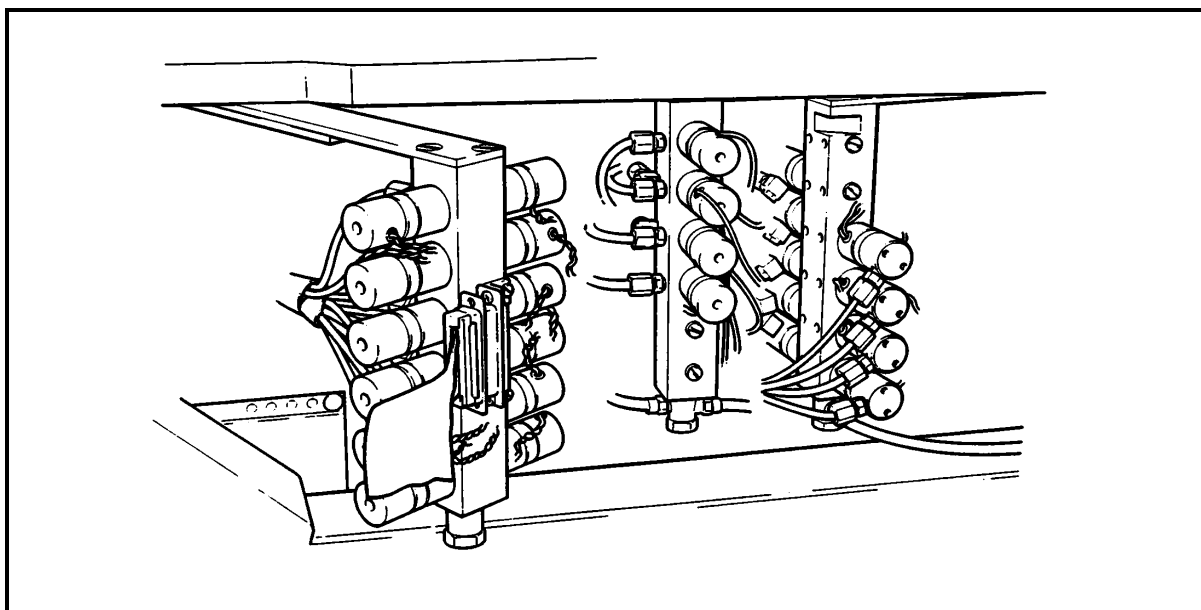


**A Bank Solenoid Out For Access**

The air supply from the solenoid to the valve is carried by a colour coded PTFE tube, to help in identification. PTFE is used because of its bakeability.

The colours used are:-

| | | | | | |
|-----|---|--------|-----|---|--------|
| RI | - | Blue | SI | - | Brown |
| RP | - | Purple | SP | - | Red |
| RF | - | Grey | SF | - | Orange |
| RV | - | White | SV | - | Yellow |
| RM | - | Black | SM | - | Green |
| RC | - | Yellow | SC | - | Red |
| RW | - | Orange | SW | - | Brown |
| LV | - | Clear | | | |
| HV | - | Pink | | | |
| ML | - | White | | | |
| MH | - | Black | | | |

The air supply is provided from a filter regulator assembly mounted at the rear of the main bench and should be set at approximately 7 Bar (120 p.s.i.).
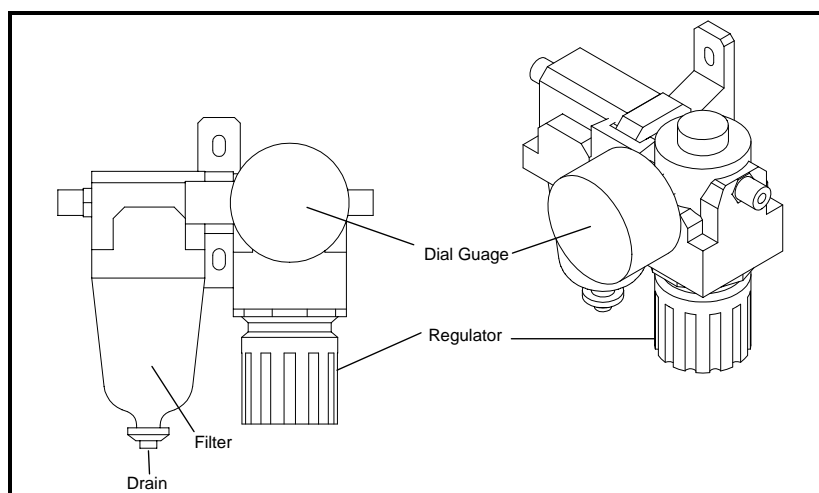


**Filter Regulator Assembly**
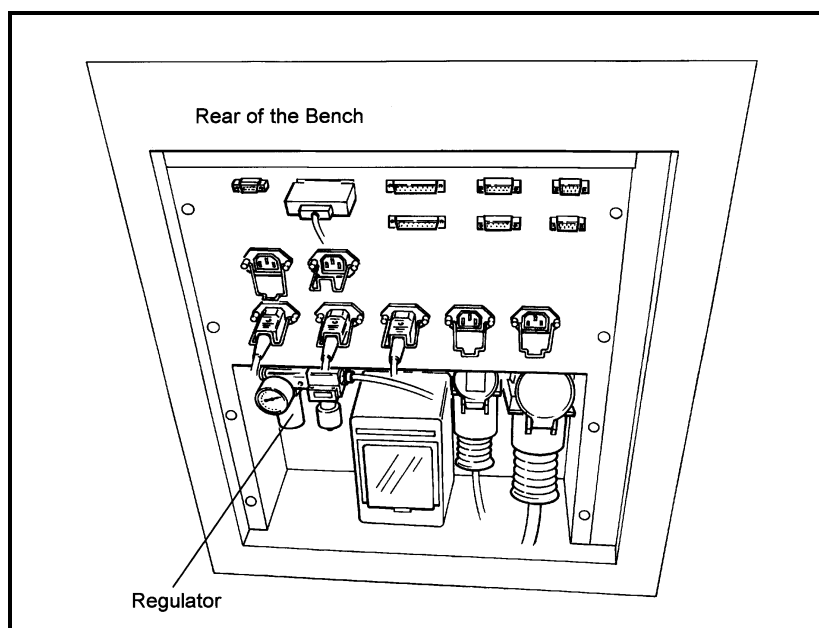


**Filter Regulator Assembly Location**

# Bellows Assemblies (Variable Volumes)

These are variable volumes which can be compressed or expanded to adjust the gas pressure in the two halves of the inlet. Each side of the inlet has its own bellows assembly. Both Bellows have approximately 20:1 maximum compression ratios. You will see that they are of different overall size, however. The reference bellows have a maximum volume of 100 ml, so that they form a large standard reservoir which does not require frequent refilling. The sample bellows have a maximum volume of 40 ml, chosen to give the best range of inlet pressures for the sample transfer distances in the system.

The bellows are moved by stepper motors mounted below the bench. The bellows are prevented from moving beyond their end stops by mechanical "flags" which break opto-electronic relays that act as signals to the stepper motor controller. Together with their robust construction, this is a major contributor to the high reliability of the bellows. The end stops are set in the factory and should not require further alteration.

The bellows assemblies are connected to the inlet system by a ¼ inch gold flange seal, which is connected to the inlet valve blocks via a low volume welded elbow which is welded directly into the valve block.
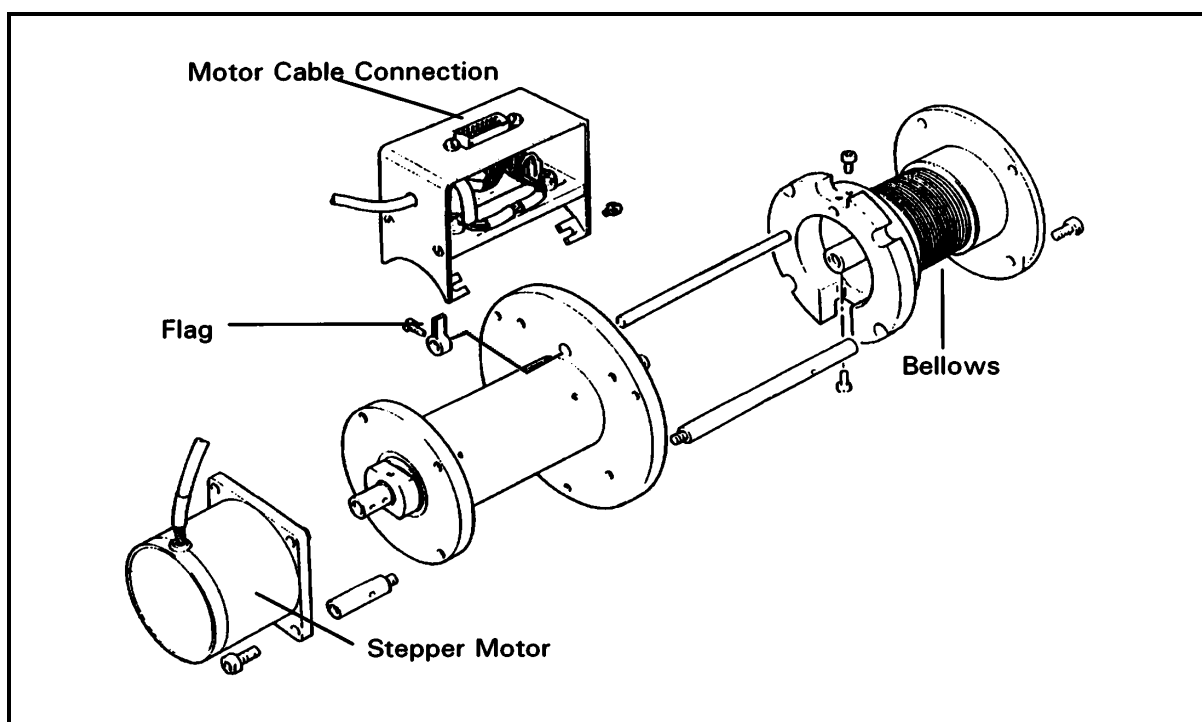


**Bellows Assembly**

# Capillaries

The two halves of the dual inlet are connected to the changeover valve via two stainless steel capillaries which are crimped to give the correct flow into the mass spectrometer. The crimped capillaries provide a viscous flow leak of gas into the mass spectrometer, which can be adjusted for identical flow rates by means of the crimps (for details of this procedure see later sections of this manual). The viscous leak ensures that there is no isotopic fractionation of the gas during introduction to the mass spectrometer.

The capillaries are of 0.004 inch internal diameter stainless steel with $\frac{1}{16}$ inch bore Swagelok connectors at each end. The capillaries, each 25 inches long are made from one continuous length of tube. The capillary crimping screws have differing crimping effects; the inner screw giving a coarse and the outer a fine adjustment.

# The Cold Finger

This is a micro volume (0.2 ml) into which samples of condensable gas can be frozen and isolated. The sample cold finger is a **computer controlled (see later sections of this manual for details)** auto cold finger, used to automatically analyse very small samples of $CO_2$ or $SO_2$ (for $N_2$ cold finger see later sections of this manual). The reference has an identical micro volume ("dummy" cold finger), to retain equal volumes in the two halves of the inlet. Concentrating the sample in the cold finger increases the inlet pressure to ensure that: (a) viscous flow conditions are maintained and the sample does not fractionate: and (b) ion beam intensities are increased to give good analytical precision.

The cold finger is attached to the sample side inlet valve block using a ¼ inch gold flange seal, with the "dummy" cold finger similarly attached to the reference side. The pumping for the liquid nitrogen required by the cold finger is provided by a Charles Austen pump located under the main bench of the PRISM (for details of the Charles Austen pump please see manufactures manual).

# Sample or Reference Connection

Each side of the dual inlet (sample and reference) has a connection point onto which a sample or reference gas bottle can be attached. This connection point is a 6.35mm (¼ inch) stainless steel pipe fitted to the system with a ¼ inch gold flange seal. The pipe is sealed by a Swagelok fitting using a PTFE seal. To ensure no dirt can enter the system filters are fitted to the connections with the inlet at the ¼ inch gold flange.

**CAUTION**

**When no sample bottle is connected it is essential that the inlet pipe is blanked off to prevent entry of dirt and to protect the instrument from the possible danger of accidentally allowing a large quantity of gas to enter the vacuum system.**

# Transducer

A total pressure transducer is fitted via a ¼ inch gold flange seal to the sample side of the dual inlet. This is used to measure the sample gas pressure prior to admittance to the dual inlet. The software can then decide how the sample will be handled (cold finger or bellows -please see later sections of this manual for details).

# Bakeout

The dual inlet has a heater assembly mounted on the bench top, so when the system is then covered with the fabric insulating bake-out cover provided an even bake-out temperature is obtained. **The bake-out is controlled via the software (see later sections of this manual for details)** with the temperature monitored by a thermistor mounted over the bellows.

# Vacuum System

## Overview

The pumping system is designed to produce the clean high vacuum required for reliable high precision stable isotope analyses. It also provides the high pumping speeds essential for the high gas flow rates encountered in continuous flow applications.
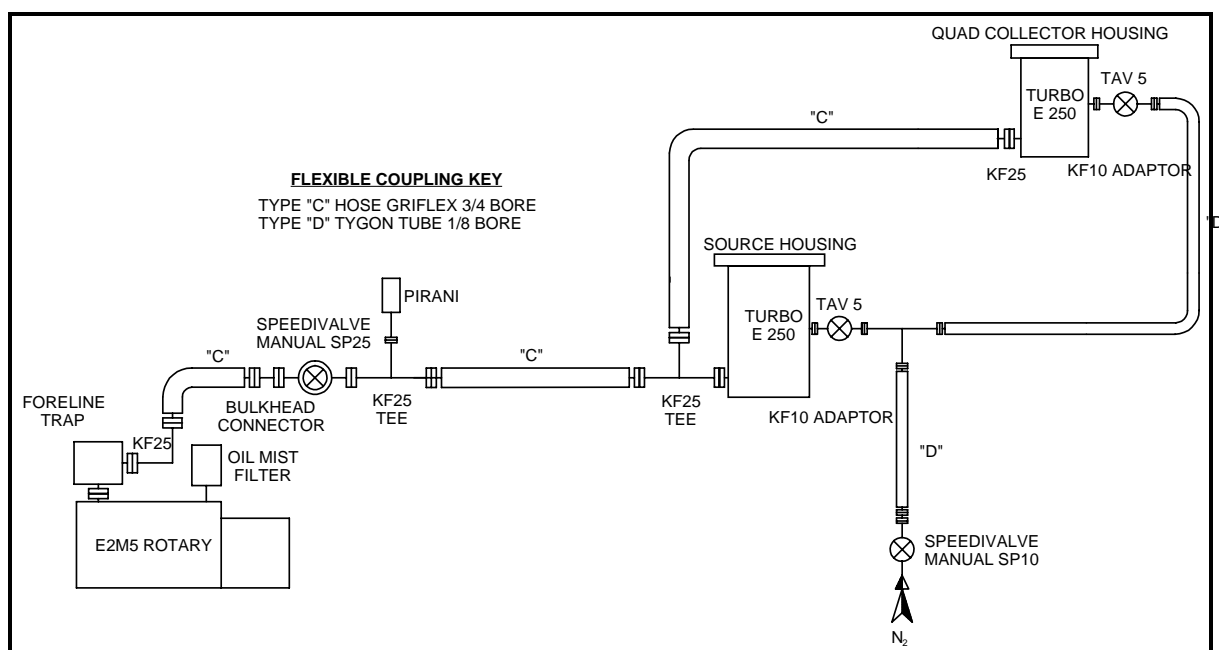


**Vacuum Schematic**

Please refer to the manufacturers manuals for more information on the vacuum pumps and accessories.

---

**CAUTION**

**The manufacturer's manuals emphasise the very important fact that regular routine maintenance is essential to reliable performance. A good clean vacuum is absolutely essential to correct operation of an isotope ratio mass spectrometer, and a day spent on service every six months or so will reap its reward in continued trouble free operation.**

---

# Analyser Pumping System

Two Edwards 250 l/s turbomolecular pumps are used for the PRISM analyser pumping. These are backed by the same Edwards RV3 rotary pump. The backing line is isolated from the rotary pump with Rotary Valve 1 at the rear of the bench.

**Note:** Each rotary pump is supplied with a foreline trap to prevent back streaming of oil vapours and an oil mist filter.

The pressure in the analyser backing line is measured with Pirani 1. To enable the system to be vented to a dry gas (e.g. dry nitrogen) the vent valves of each turbomolecular pump are joined together and exit the bench at Vent Valve 1. The turbomolecular pumps are cooled by fans fitted to the base of the pumps.
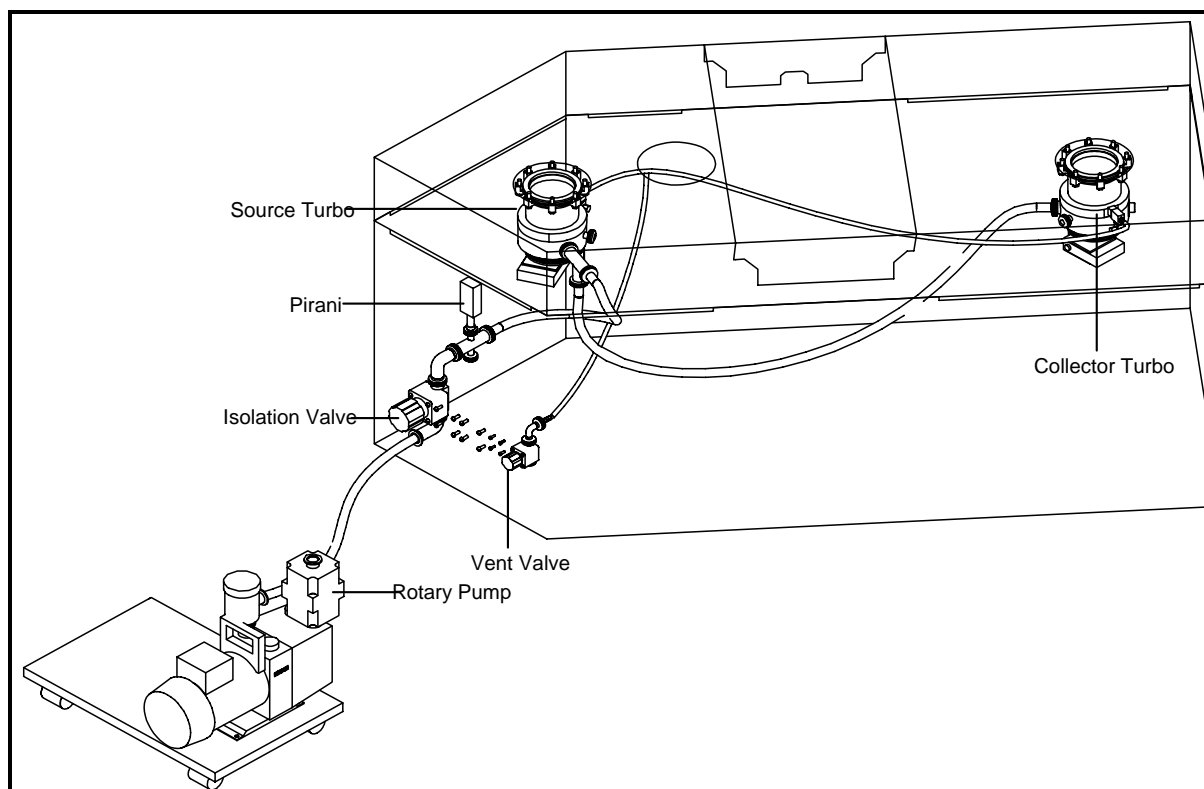


**Analyser Pump Layout**

Please refer to the manufacturers manuals for the turbomolecular and rotary pumps if you need detailed trouble shooting information.

# Dual Inlet Pumping System

On systems fitted with a dual inlet, two additional turbomolecular pumps are fitted. These are Edwards 70 l/s pumps, one used for the high vacuum pumping of the inlet, and the other for pumping the waste line from the changeover valve block. These are both backed by another Edwards RV3 rotary pump. The backing line is isolated from the rotary pump with Rotary Valve 2 at the rear of the bench. An additonal rotary pump is supplied to rough pump the inlet prior to pumping by high vacuum

**Note:** Each rotary pump is supplied with a foreline trap to prevent back streaming of oil vapours and an oil mist filter.

The pressure in the dual inlet backing line is measured with Pirani 4. To enable the system to be vented to a dry gas (e.g. dry nitrogen) the vent valves of each turbomolecular pump are joined together and exit the bench at Vent Valve 2. The turbomolecular pumps are cooled by fans fitted to the base of the pumps.
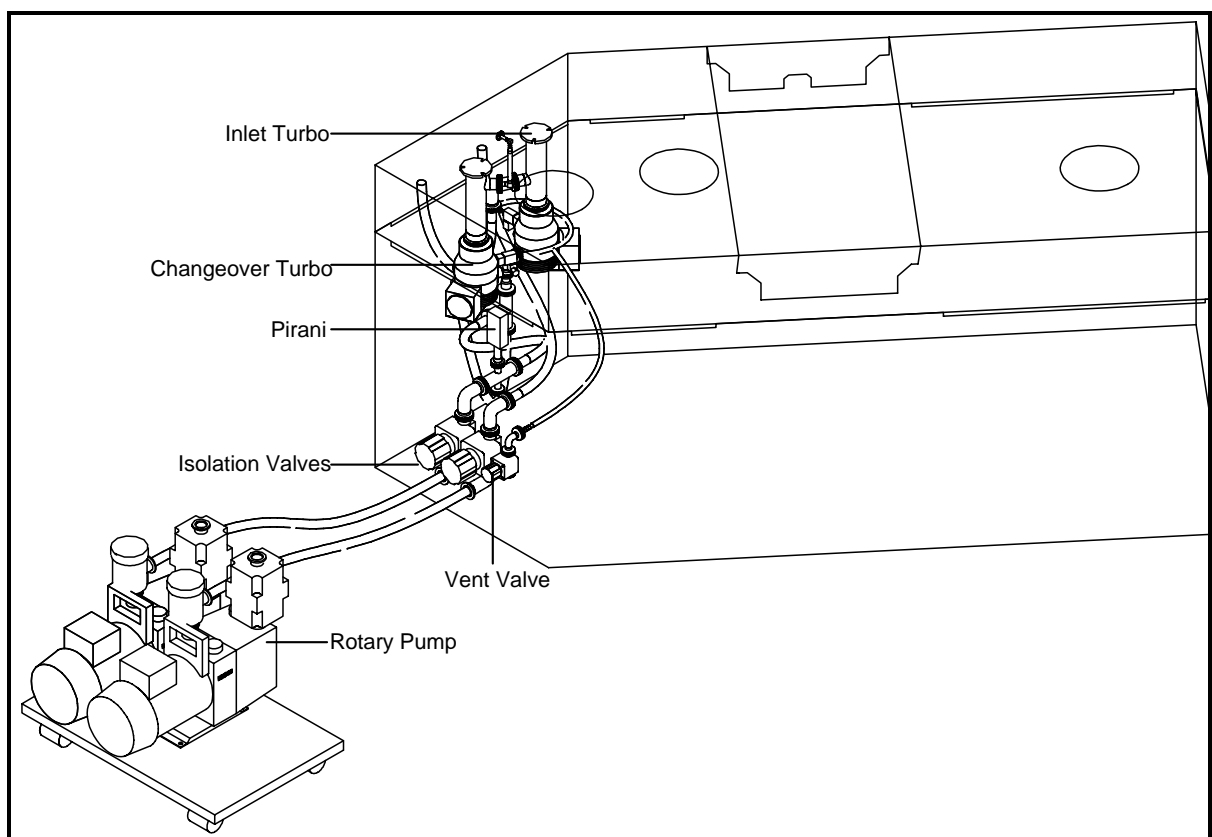


**Dual Inlet Pump Layout**

Please refer to the manufacturers manuals for the turbomolecular and rotary pumps if you need detailed trouble shooting information.

# Differential Pumping Option

An Edwards 250 l/s turbomolecular pump is used for the Differential Pumping Option. It is attached to the Differential Pumping Tee Chamber (or to the HD housing if the HD Option is chosen) under the analyser bench. The backing port of this turbomolecular pump is connected to the analyser pumping backing line. It therefore shares the Edwards RV3 rotary pump used by the analyser. It also shares vent valve 1 with the analyser turbomolecular pumps.
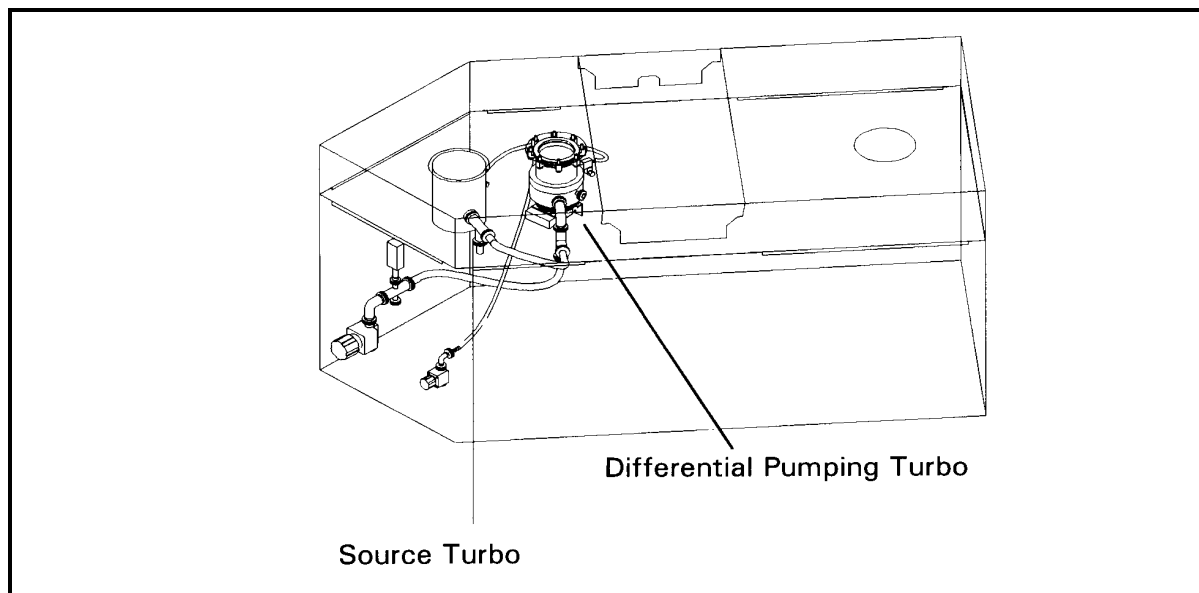


**Differential Pumping Layout**

Please refer to the manufacturers manuals for the turbomolecular and rotary pumps if you need detailed trouble shooting information.

# Rotary Pumps

The rotary pumps are located on a separate trolley and are connected to the main bench via flexible hose connections. The connections to the rest of the vacuum system is via a service panel located at the rear of the main bench. This allows easy access to the pumps for routine oil, filter and foreline trap maintenance, and ensures complete vibration isolation from the ion optics.
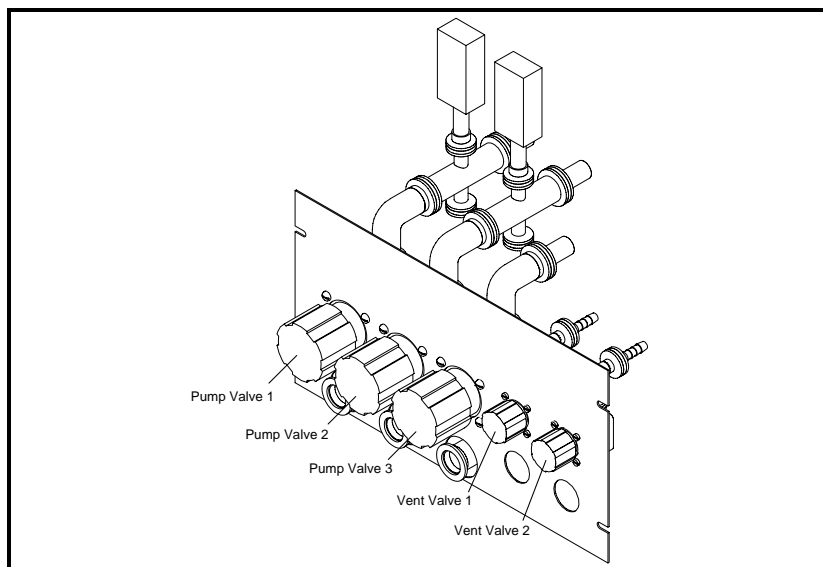


**Vacuum Service Panel**

The valve assignments are as follows:

| | | |
|---|---|---|
| 'ROTARY PUMP VALVE 1' | - | Analyser turbomolecular pumps backing |
| 'ROTARY PUMP VALVE 2' | - | Inlet turbomolecular pumps backing |
| 'ROTARY PUMP VALVE 3' | - | Inlet roughing pump |
| 'VENT VALVE' 1 | - | Inlet turbomolecular pumps vent |
| 'VENT VALVE 2' | - | Analyser turbomolecular pumps vent. |

# Pressure measurement

## Ion gauge

A VIG24 ion gauge is fitted to the source housing to monitor the pressure of the system. The ion gauge is mounted in a copper flanged (CU38) elbow so that it is not in line of sight of the source. If Differential Pumping is fitted to the system an extra ion gauge is fitted in the analyser to read the analyser pressure, while the ion gauge in the source reads the source pressure. The ion gauge is controlled via the software and the pressure displayed on the computer screen.

The gauge is fitted with two filaments so that in the event of one failing the other can be easily selected by software control (see later sections of this manual for details), thus removing the need to break vacuum. It is best however that the filament array be replaced (see later sections of this manual for details) the next time the vacuum is broken for other reasons.

The ion gauge circuitry in the STE gauge controller is fitted with a trip, which in the event of the ion gauge pressure rising above 5E-5 mBar, will automatically cause the source electronics to be switched off thus protecting the source filament.

**Note:** The source electronics will not come back on until the vacuum is better than 1E-5 mBar

If the ion gauge pressure rises further to a pressure in excess of 9E-3 mBar , safety trip circuitry in the ion gauge controller will switch the ion gauge itself off to protect the filament. In the event of this happening the status will be relayed to the operator via the software. If the pressures were later to improve to better than 9E-3 mBar, for safety reasons the gauge does not automatically switch back on but must be reset (see later sections of this manual).

In the event of the ion gauge becoming contaminated the controller has a built in facility for degassing the filament, which can be selected from the software, together with the on / off control and filament selection. (For information on the ion gauge control see later sections of this manual for details).

## Pirani Gauges

There can be a maximum of four Pirani gauges on a PRISM system. One of these is for the Carbonate Preparation System and will be discussed in the Carbonate section of this manual.

The Pirani gauges carry the following identifications as seen on the computer:

P1 Analyser       :Analyser Turbomolecular Pump Backing Line
P2 Inlet            :Inlet Rough Pumping Line
P3 Isocarb       :Carbonate Pumping
P4 Waste        :Changeover/Inlet Turbomolecular Pump Backing Line

Pirani 1 and 4 are located under the main bench at the rear where the rotary roughing lines enter, Pirani 2 is located under the inlet table.

## Vacuum Protection

The pressure measuring devices, in conjunction with the vacuum control electronics and the electrical control panel, provide vacuum protection for the ion gauges and ion source. Such that over pressure on the ion gauge will trip the ion source power supplies and high pressures on the ion gauges cause them to switch themselves off (see ion gauge section above for details).

# Electrical and Electronics

| WARNING |
|---|
| **All Electrical and Electronic Units can cause injury, so care must be taken.** |

## Overview

This section gives a brief description of the functions of each of the PRISM's electronic units.

## Unit Locations
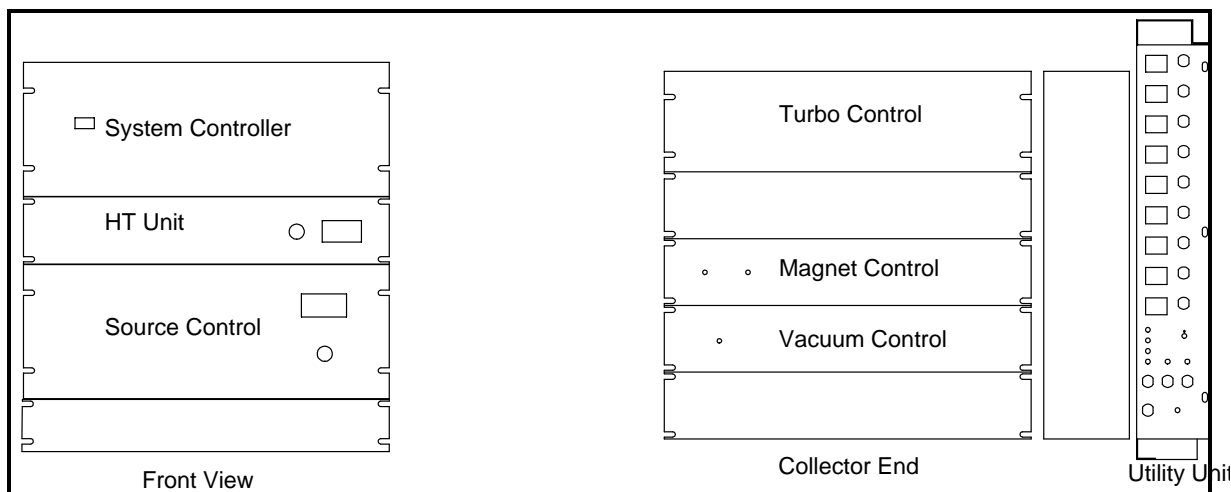


**Electronic Unit Layout**

All of the Electronic units are of the standard 19" rack type with the exception of the Utility Unit and the Head Amplifier.

The following 19" electronic units are situated centrally at the front of the instrument under the magnet:

- System Controller

- High Voltage Supply Unit

- Source Control Unit

- The other 19" electronic units are situated in the side of the bench under the main analyser collector:

- Turbomolecular Pump controller Unit

- Electromagnet Supply Unit

- Ion Gauge Controller Unit

The Utility Unit is situated to the right-hand side of the units beneath the collector.

The Head Amplifiers are bolted on to the side of the collector housings (two are possible if HD option is chosen).

At the rear of the bench are the Mains Inlet and Outlet Service Panels. These are sometimes grouped together as the "Electrical Service Panel".

# Mains Inlet Service Panel

The Mains Inlet Service Panel at the rear of the analyser bench has the Main Power Inputs to the PRISM; these can be unplugged from the instrument if required. Also mounted on this panel are the Main Circuit Breakers. These provide double-pole isolation, over-current protection and residual current (earth leakage) protection. The 32A breaker isolates the pumping circuit and the 16A breaker isolates the electronics circuit. For more details of power requirements please refer to the PRISM Site Planning Guide.



**Input Service Panel**
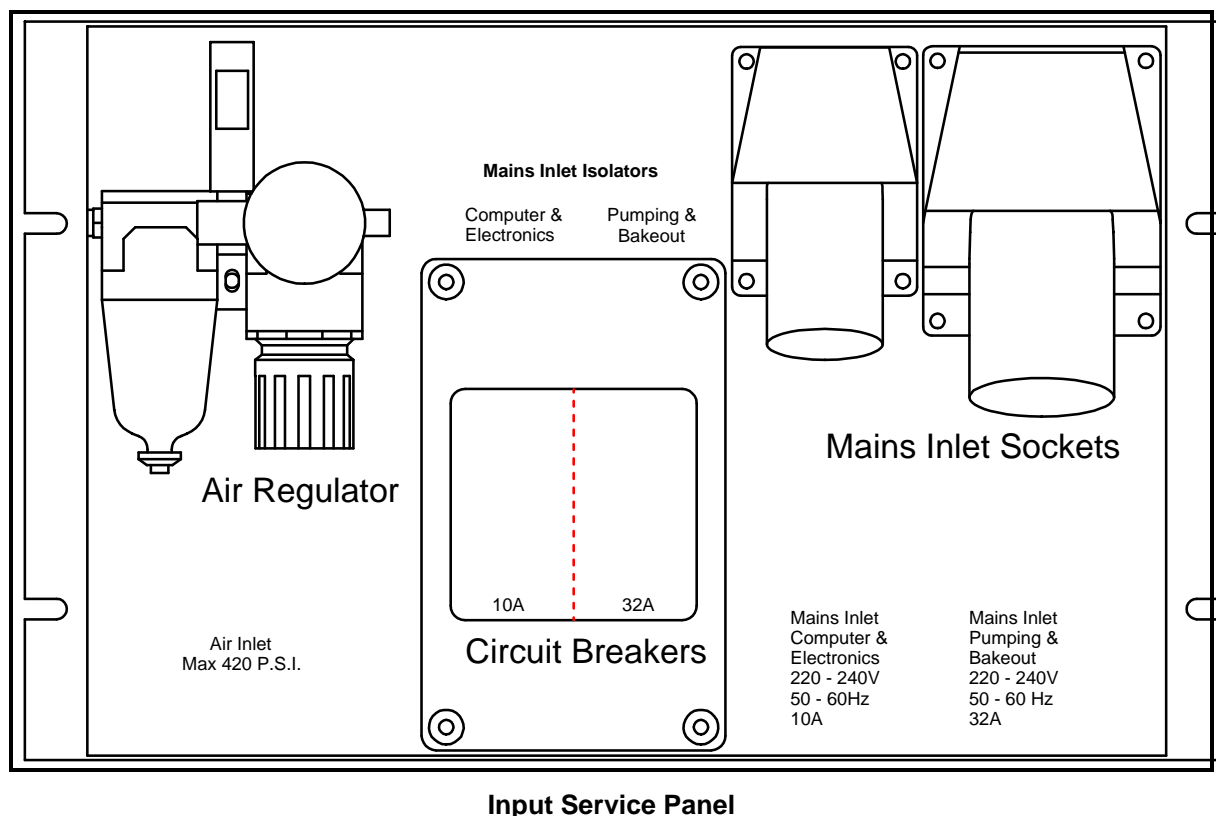
# Mains Outlet Service Panel

The Mains Outlet Service Panel is situated above the Mains Inlet Service Panel at the rear of the analyser bench. It allows peripherals to be connected to the system without the user having to gain access to the inside of the bench.
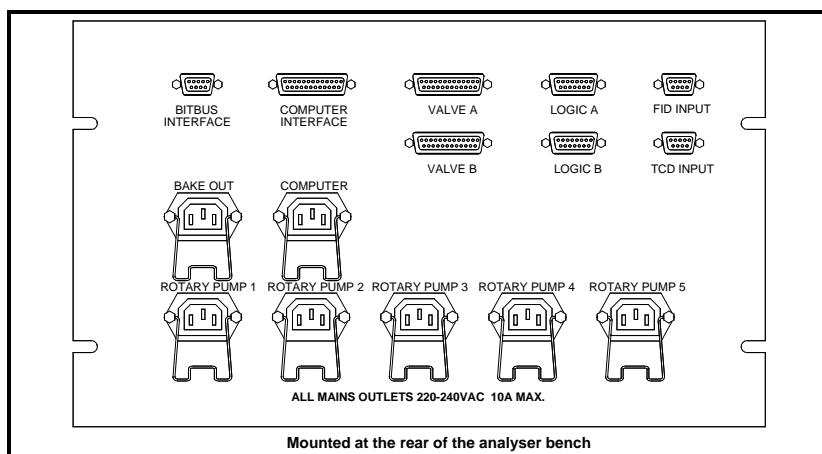


**Mains Outlet Service Panel**

# Head Amplifier

The 4-channel amplifier is housed in a rugged aluminium casting. It is secured to the side of the collector housing by two recessed bolts; location is aided by two dowel posts on the side of the housing. The connection to the four collectors and to the secondary electron suppresser is by a 5 pin gold feedthrough. The connectors in the amplifier are spring-loaded to ensure good contact.

Internally the amplifier is divided into 2 compartments, each housing a printed circuit board. The upper board (the "analogue board") is responsible for amplifying the collector currents by converting them into a voltage (0V to 10V). Prominent on this board are the 4 low temperature coefficient resistors.

The resistors used are:

| | | |
|---|---|---|
| Low Mass 2 | $5 \times 10^8$ | ohms |
| Low Mass 1 | $5 \times 10^8$ | ohms |
| Axial | $5 \times 10^{10}$ | ohms |
| High Mass | $1 \times 10^{11}$ | ohms |

When used for HD analysis, the Low Mass 2 channel amplifies mass 2 ($H_2$) and the High Mass channel amplifies mass 3 (HD).

As the maximum output voltage of each amplifier is 10V, the maximum source current can be calculated:

| | |
|---|---|
| Low Mass 2: | 2E-8A |
| Low Mass 1: | 2E-8A |
| Axial: | 2E-10A |
| High Mass: | 1E-10A |

A unique feature of the amplifier is that it digitises the amplified signal prior to transmission. This is performed by voltage to frequency converters on the analogue board. The conversion is scaled such that the full-scale 10V output from the amplifier is represented by a frequency of 1 MHz.

The second printed circuit board is called the 'digital board'. Its function is to turn the digitised signal from the amplifier board into pulses of red light which are then transmitted to the System Controller via optical fibre. The use of a fibre optic link makes the transmission of data immune to external electrical interference.

The system controller is responsible for counting the number of light pulses which arrive at its VFC card in each 100mS integration period. Some simple arithmetic shows that a full-scale signal is equivalent to 100,000 "counts" in each period.

The power supply for the amplifier and the secondary electron suppresser is connected to the digital board via a DIN plug.

---

**CAUTION**

**It is essential to remove the head amp prior to baking out the analyser.**

---

---

**CAUTION**

**The amplifier contains very static sensitive components. It should be handled carefully. Before disconnecting the amplifier, turn off the source to prevent static build-up on the feedthrough pins, then turn off the DC-supplies to remove power from the amplifier. Short the five feedthrough pins to the collector housing (using some tinned copper wire) before turning the source back on. Only remove the shorting wires AFTER the source has been turned-off again.**

---

---

**CAUTION**

**Never touch any of the components inside the amplifier, as contamination could serious affect performance.**

---

# System Controller

In the front of the bench is the System Controller. This unit controls all the electronic systems of the instrument. It consists of a rack-based microcomputer with all the additional electronics required to control the instrument.

The Data System controls the instrument by communicating with the System Controller via an RS232 serial link. There is a constant exchange of instructions and data between these two computer systems.

The System Controller uses the industry standard STE bus to provide an easily up-gradeable card based system. The Eurocard-based STE bus has gained wide acceptance for industrial control applications. Its use in this applications allows flexibility and the use of off-the-shelf components. The central processing unit is based upon a powerful Motorola 68000 microprocessor, running an in-house firmware package written almost entirely in the high-level language "C".

Depending on the configuration of your instrument, your system controller will contain some or all of the following cards:

- **Processor Card** : single card microcomputer which controls the bus.

- **VFC Service Card** : processes the optical signals from the amplifier.

- **Unit Interface Card** : handles communications to the other electronic units.

- **EMS Card** : digital controller for the electromagnet supply.

- **Stepper Motor Card** : provides control of up to 3 stepper motors.

- **Valve Card** : Each card provides control of up to 48 valves.

- **Analogue Card** : general purpose card for analogue signal processing (used for thermocouples, etc.).

- **'SPIBB' Digital I/O Card** : general purpose digital input and output card used to control the Utility Unit

- **Gauge Controller Card** : controls and monitors the vacuum gauges.

The use of this card based system makes it easy to alter your configuration. For example, if you have sample preparation systems that require extra valve outputs, a second valve card will enable you to run up to 96 valves.

Similarly the design makes repair straightforward: A defective card can be easily exchanged for a replacement.

**Note:** The maintenance of any electronic unit should only be carried out by qualified service personnel. See fault finding section for details.

# Source and High Voltage Supplies

> **WARNING**
>
> **These units generate the voltages and currents required to drive the gas source. There function is controlled by the system Controller via the in-house standard 'Unit Interface'.**

## High Voltage Supply Unit

This unit is situated beneath the System Controller. Its function is to generate the High Voltages used to drive the gas source. All voltages generated by this unit are "ground referenced". In other words one end of each supply is connected to ground/earth potential. These voltages are:

- Accelerating Voltage (often referred to as HT)

- Half plate focus voltage (Extraction Voltage)

- Z plate focus voltage

The front panel includes the following features:-

1. A red indicator to signify that mains power is applied to the unit.

2. A green indicator signifying that the high voltage circuitry is operating correctly.

   **Note:** This will not illuminate until about 1 minute after power up.

   To the right of the panel is a meter and a four position selector switch which allows the following source parameters to be monitored:-

- Accelerating Voltage.

- Half plate focus voltage.

- Z plate focus voltage (+ve or -ve).

## Source Control Unit

This unit is situated beneath the High Voltage Supply Unit. Its function is to generate the source voltages and currents which are not ground referenced. These are:

- Filament current

- Trap current

- Delta half plate voltage

- Electron volts

- Ion - repeller voltage

These voltages and currents are referenced to the accelerating voltage and half-plate voltage, which are generated by the High Voltage Supply Unit, and connected via two high voltage BNC type cables.

All the source voltages and currents are attached to the source via the source cable, which connects to an output from the Source Control Unit. A spur of this cable also attaches to the High Voltage Supply Unit to connect the Z-plate supply.

The front panel includes the following features:

A red indicator to signify that the mains power is applied to the unit.

To the right of the panel is a meter and six position selector switch which allows the following source parameters to be monitored:-

- Electron volts

- Ion repeller voltage (+ve or -ve)

- Trap current

- Source current

- Filament current

# Turbomolecular Pump Controller Unit

In the side of the bench under the main analyser collector the uppermost unit is the Turbomolecular Pump Controller Unit. This unit is capable of holding up to five Edwards turbomolecular pump controllers (EXC 100E). They are secured in the unit by screws going through the base of the unit into the controllers themselves.

---

**CAUTION**

**For details please see manufactures manual supplied.**

---

# Electromagnet Supply Unit

The Electromagnet Supply Unit is situated beneath the Turbomolecular Pump Controller Unit. This unit performs high efficiency power control, switching and sensing for both the main analyser magnet and the HD magnet (if fitted). Swapping between the two magnets is controlled by the computer (please see later sections of this manual for details). The basic operation is to turn one magnet off before switching the other on.

The control electronics for the electromagnet are situated on an STE board in the System Controller, and the raw DC supply is generated in the Utility Unit. This modular design ensures that the STE controller can maintain tight control of magnet functions (such as background scanning), whilst still delivering the high power required by the magnets.

# Utility Unit

This unit is situated so that its front-panel is exposed at the left hand corner of the instrument looking from the front. The panel houses the mains switches which isolate the supplies to major electrical components. Each rocker switch has a green light incorporated; when this is illuminated the power is on. Several of these mains circuits are interlocked via switches/relays for safety/vacuum protection.

Next to each switch is a 'pop-out' thermal fuse; which will 'trip' if the circuit draws excessive current. Pressing the fuse back in will restore the power. If the device does not reset, further investigation is required. The current rating (in Amps) of each circuit is shown on the face of the fuse.

Lower down on the panel are the neon indicators and pop-out fuses for the system controller power supplies, and trips for the input power supplies.
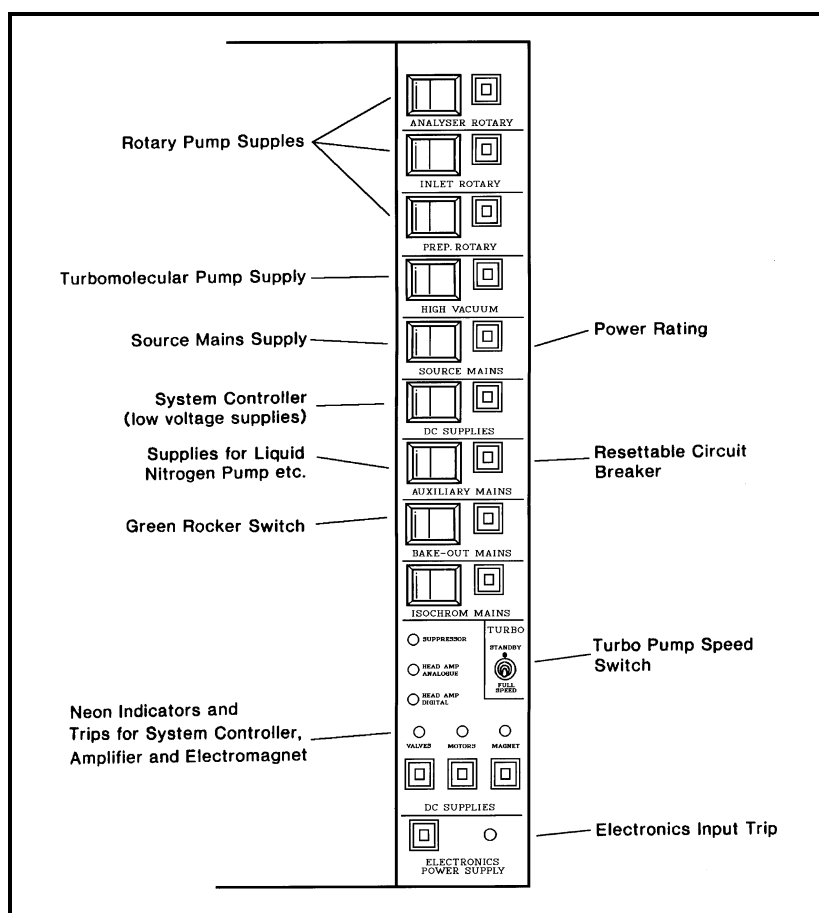


**Mains Distribution Panel**

Inside this unit is the Utility PCB, which generates DC supplies for Valves, Motors, Electromagnets and Head Amplifiers. It also has mains relays to control bake-out heaters, the Source/HT Unit, magnetic stirrer etc. The PCB is controlled by the System Controller via the STE SPIBB input/output card.

# Ion Gauge Controller Unit

At the bottom of this rack of units is the Ion Gauge Controller Unit. This unit can contain two Edwards Ion Gauge controllers (EXC EBEAM 2 HEAD PCPS). Standard PRISMs contain one controller to drive the analyser Ion Gauge, whereas systems with the differential pumping option require an extra controller to drive the Collector Gauge. The Edwards units are controlled by the System Controller via the STE Gauge Controller Card.

---

**CAUTION**

**For details please see manufactures manual supplied.**

---

# Hall Probe

The optional Hall Probe can be used to provide field mode control of the magnet (A standard system uses current mode control). The Hall Probe device is mounted between the poles of the main analyser magnet, whilst the control PCB plugs directly onto the EMS card in the System Controller. Selecting whether 'field mode' (Hall Probe active) or 'current mode' (Hall Probe inactive) can then be performed from the software (please see later sections of this manual for details).

# Fuses

The majority of circuits in the system operating at mains voltages are protected by the pop-out fuses next to the appropriate switches on the Utility Unit.

---

**CAUTION**

**See the fault-finding section for details of all fuses in the system.**

---

# Data System

The data system used on the PRISM mass spectrometer has the following characteristics:

- 486  33 MHz microcomputer with 8 Mb RAM.

- 120 Mb hard disc drive.

- 1.44 Mb  3.5" floppy disc drive.

- Super VGA high resolution colour monitor.

- Mouse.

- 2 Serial ports.

- 1 Parallel port.

- Epson LQ 570+ dot matrix printer with parallel printer cable.

- OS/2 Operating system.

---

**CAUTION**

**For details please see manufactures manuals supplied.**

---

The data system is mounted on a table next to the mass spectrometer and is connected to the instrument via the Mains Outlet Service Panel is situated at the rear of the analyser bench. This panel supplies power to both the computer and printer, as well as the communications link.

# Section 5



# User Interface

# Overview

The 'user interface' is the way you interact with the mass spectrometer from the computer system. This section of the manual will therefore detail the essential aspects that are needed to accomplish this, however it will be necessary to read the OS/2 manuals supplied with the instrument to complement this section.

Its purpose is therefore to guide you through the features necessary in running the PRISM software, and in conjunction with the context sensitive HELP facility, enable you to learn the basics of the software which will lead to instrument operation in the next section of this manual. The screen displays are used to familiarise you with the appearance of the software.

# OS/2 Environment

This section of the manual cannot serve as a full guide to all the powerful features provided with the OS/2 operating system. but is intended to give you the basic features required to start running the PRISM software package.

**Note:** PLEASE READ THE OS/2 MANUALS SUPPLIED WITH YOUR SYSTEM.

The PRISM is controlled largely through the use of a 'mouse' (a pointing device), pull down menus, and graphical representations of the system called 'icons'. There is no need to learn complicated keyboard sequences, and the system is easy to operate. Some people may prefer to use the Keyboard: there are simple 2 key alternatives to virtually all "mouse" operations. "Hot Keys" give simple access to frequently required options. The Keyboard is of course used in any case to enter data, such as sample names, reference gas compositions, to set integration times etc.

# Computer Start Up

OS/2 preserves the layout of your Desktop even when switched off. This means you no longer have to move icons to the top of a list to get them to start automatically - anything open at shutdown will be opened again next reboot. As well as leaving applications open at shutdown you can place applications in the start-up folder, these will then be started each time you reboot.

## Automatic Start-up

To start an application automatically simply drag the application icon (e.g. Dual Inlet) into the Start-up Folder (in the OS/2 System Folder on the Desktop). For example to start-up the system clock automatically :- Open the OS/2 system folder. Open the System Setup and the StartUp folders. Now pick the Clock icon from System Setup and Copy it into the StartUp folder. This application will now be run each time you reboot.

**StartUp Problems**

As OS/2 will always start applications that were running when it was ShutDown, which may cause possible problems. However, it is possible to disable the automatic program startup. To do this reboot the machine and when the white screen first appears press and hold the left Ctrl, left Shift, and F1 keys. Hold them down until the icons appear on the desktop.

# Computer Shut Down

## Why ?

To speed up access to commonly used files and applications OS/2 now uses a Caching system. A Cache is an area of memory set aside for the storage of data. This means that some files are accessed and left open, stored in the Cache. Any reference to these files accesses the copy in memory (in the Cache), thus saving the time to search and load from disk. When the computer is switched off, or re-booted, the contents of this memory disappears, possibly losing data or corrupting files. Wherever possible before turning the machine off or rebooting you should perform the ShutDown operation. This will clear the Cache saving any changes and closing files.

In addition to clearing the Cache, ShutDown will close down all the applications and folders which are open. OS/2 keeps track of the layout of the Desktop, where the folders are and what applications are running. Next time you boot into OS/2 these will be opened automatically and the Desktop will look exactly as it was at ShutDown. This means ALL folders/programs open at ShutDown will be opened again . The more applications open the slower the system runs so any folders you do not want to be opened next time should be closed BEFORE performing the ShutDown (See Window List section).

**Note:** Some applications will ask for confirmation to close down - this ensures that you do not lose data in applications you may have forgotten are open.

**How ?**

On the desktop, move to an area away from any icons. Bring up the Desktop menu (single click Right mouse button), and select shutdown. Wait for the message Shutdown Completed. You can now reboot or switch off the computer.



# OS/2 Desk Top

On starting the computer the first screen to be seen is the OS/2 Desk Top, an example of which can be seen below. From this, access to any of the applications can be achieved by using the mouse to click on the various icons displayed.
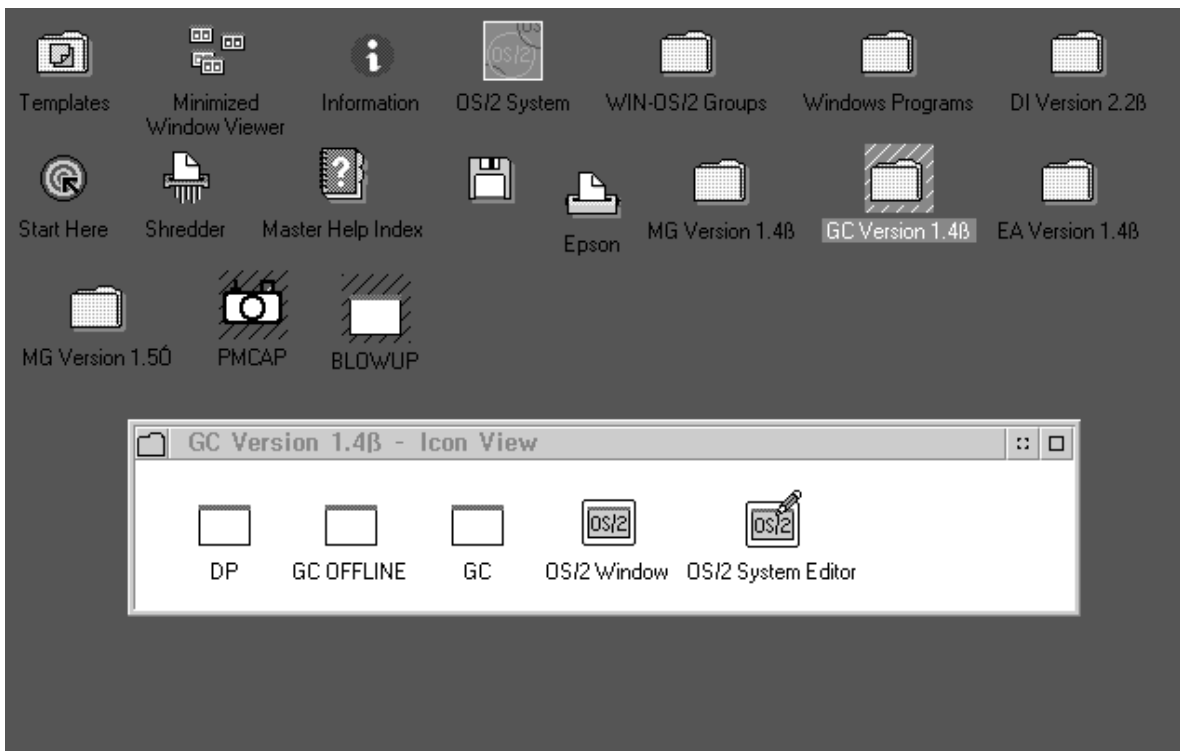
# Desk Top Environment

Each application started runs in its own window and a number of application windows can be open on the desk top at any one time. The screen can therefore be used by a number of Windows, which each display information related to a specific area of the software.

One helpful way of thinking about this is to regard the windows as sheets of paper on a "DESKTOP" represented by the whole screen display. Just like paper on a desktop, the windows can be picked up and moved around, put on top of each other etc. Unlike sheets of paper, some of the windows can be shrunk or expanded, either to fill the entire screen, or to occupy a minimum amount of screen space.

The entire PRISM program is itself a Window. If you wish to run another program, such as a spreadsheet, as well as the mass spectrometer, you can size the PRISM window to suit your display needs. It can even be shrunk to an Icon, and run in the background while the whole screen is available for other applications - the choice is yours.

# Using the mouse

The mouse is a device incorporating a tracker ball, which moves a pointer around the screen. The mouse pointer is the 'hand' which picks things up and moves them around on the desktop. The mouse also has two buttons at the top which can be depressed (clicked). The left mouse button is the most used button. It is used for selecting, resizing, dragging, copying objects and starting applications etc., within the PRISM software. The right button is used for moving objects and setting their attributes.

## Clicking

When the pointer is over the menu item or icon that you wish to select, a press or "click" on the left mouse button will highlight that item.

In the case of the main menu bar, clicking on one of the menu options will 'pull down' a further menu. Use the mouse to highlight the option you want. A further click will then action that option. It is possible to do this more quickly by doing two button presses in quick succession. This is called 'double clicking'.

More detail of Clicking is given below:

**Single Clicking (with the left mouse button)**
Single clicking on an object (icon, window, or menu)will select it, highlight it. This means that the object now has input focus (i.e. it will respond to anything typed at the keyboard and the other objects won't) or is just selected (depending on what type of object it is).

**Double Clicking (with the left mouse button)**
Double clicking on a program icon will start that program, (double clicking on a valve icon will open that valve or close it).
Also it is worth noting that double clicking on the small icon in the top left corner of a running application will generally close the application (this is the same as single clicking here and selecting close).

**Single clicking (with the right mouse button)**
When the user single clicks on a menu name this will bring up a menu list (can also use the left mouse button). The other place this is used is when closing down the system.

**Double Clicking (with the right mouse button)**
This has no effect.

**Pressing both buttons together**
This **only** has effect if the mouse pointer is currently pointing at the

desktop, it will bring up a list of all of the programs currently running on your system (see Window List).

**Note:** Use right mouse button to move icons, etc. and the left button to drag windows.

## Dragging

To drag an object (essentially move it), position the mouse pointer over the object and press and hold down the right mouse button, now when you move the mouse the object will move with the mouse pointer. You must however be careful where you drop the object.

The most important uses of dragging are :

**Window Positioning:** any window with a Title Bar can be repositioned by dragging on the title bar.

**Window Sizing:** a window can be expanded or contracted by dragging on its borders. Dragging on the corners will size in two dimensions simultaneously.

**Note:** the mouse pointer changes to a double headed arrow to show you that you are able to resize.

**Using Slider Bars**. Slider bars are used to scan through long amounts of text in text windows (e.g. Help Index) , and in two important parts of the PRISM software : Bellows adjustment and source tuning.

**Note:** It is also possible to move an object to another location by selecting the move option from the System Menu.

# Windows

All Windows have the same features and can therefore be discussed generally, however we will use the PRISM software as our example. Windows can have other windows running in them, with the highest level known as the 'Application Window' and the inner windows known as 'Child Windows'

The entire PRISM software runs as an 'Application Window'.
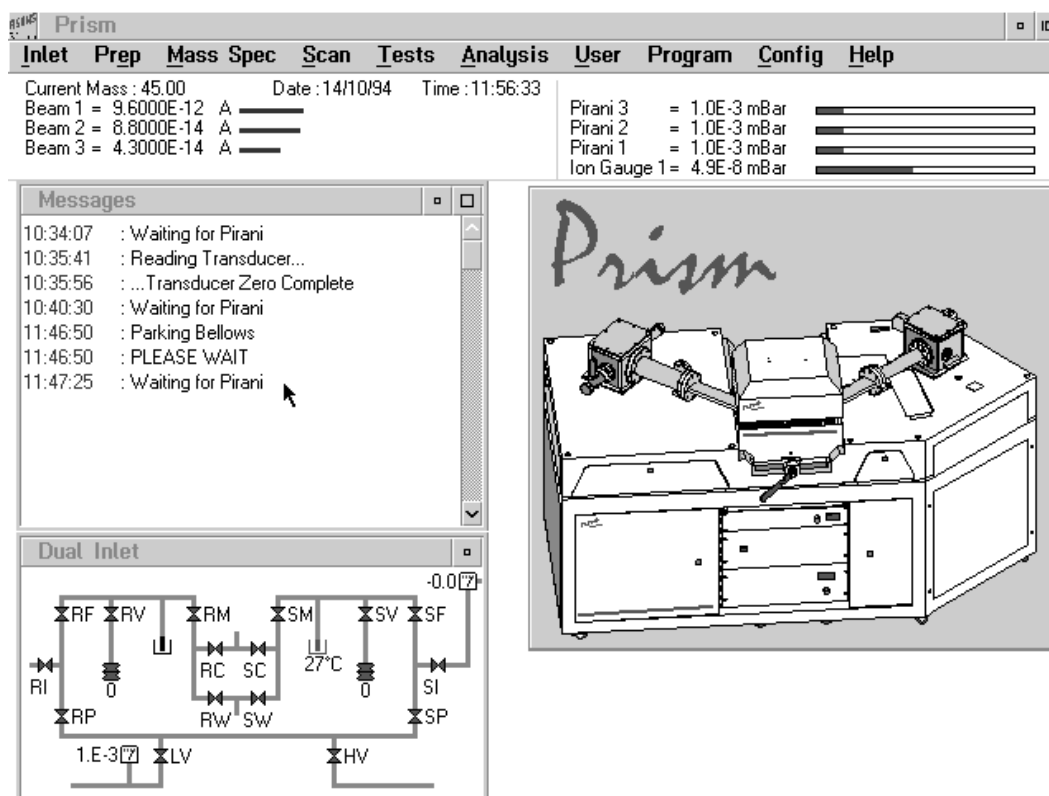


At the top is the 'Title Bar'. At the left end of this is the 'System Menu'. At the right end are the 'Minimize' and 'Maximize' boxes. The System Menu has the following features, which appear as a menu list by clicking once in the System Menu (please beware that a double click will close the PRISM software down, double clicking in most System Menus will close that particular application down):



**Restore**
If an application has been resized then the clicking in restore will restore the window to its default / original size.

**Move**

This enables the window to be moved around the screen, the window follows the mouse until the left or right mouse button is clicked (see also dragging a window).

**Size**

This is used to change the size of a window, by selecting size and moving the mouse pointer to one of the window borders that border will be picked up and move with the mouse until the left mouse button is clicked (see also sizing a window).

The next three commands can either be found in this menu or in the top right hand corner of a window as icons for maximizing, minimizing and restoring.

**Minimize**



This is used to minimize a window to an icon. When you minimize a window the particular 'Minimize' icon for that window is placed in the 'Minimize Icon Viewer' folder.

**Maximize**



This is used to maximize a window to its largest possible size.

**Restore**



The restore icon can be used to 'restore' the window to its original size, for example, if you were to maximize, or indeed re-size the PRISM window and you wished to return to the original window size then you could do this with the 'Restore' icon.

**Hide**

This is not used by the PRISM software and hence is 'Greyed Out' (see section on Greyed Out).

**Close**

This is used to close the application window down, ending any programs running from that window.

**Windows List**

This opens the 'Window List' dialog box which lists the active programs (also possible using <CTRL. .ESC>).
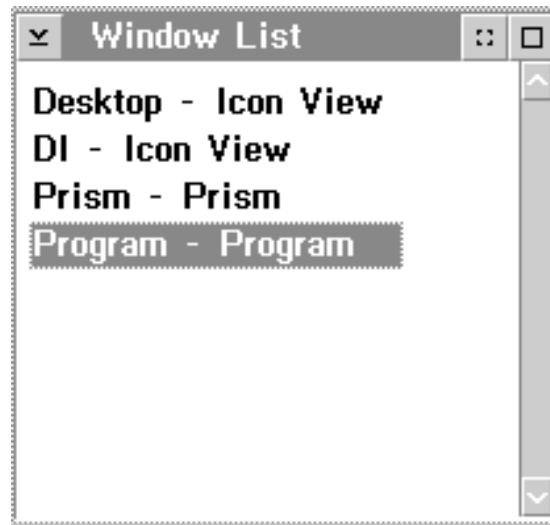


**Arrange Windows**

This is used to arrange the windows 'Child Windows' in the 'Application Window', hence tidying the screen layout.

## Moving Between Windows

You will often need to move between windows to view or change information.

To move between windows when the window you want is visible:

Click anywhere in the window in which you want to work.

The software activates the window and brings it to the front if there are other windows covering it. The active window has a coloured border and title bar, inactive windows are monochrome. This window now has 'input focus'.

## Dragging A Window

Windows can be moved to any location on the screen by dragging them to a different location. This is achieved by:

Move the mouse pointer to the title bar of the window to be moved.

Press and hold down the left or right mouse button.

Drag the window in the direction you want to move it.

When the window is in the new location, release the mouse button.

## Sizing A Window

The windows can be made smaller or larger, by moving the borders of the window in or out.

Move the mouse pointer to the border of the window (the pointer changes shape to a double arrow when it is position).

Press and hold down the left or right mouse button.

Drag the border in the direction desired.

When satisfied with the new size release the mouse button.

**Note:** Going to the corner of a window enables the window to be re-sized in two directions at once.

# Icons

Icons are small picture symbols used to represent either graphical objects (valves, bellows, etc.), or windows. An icon which represents a window or application has the name of the application beneath it. When a window is minimized it is represented by its Icon. Within some windows, notably the 'Dual Inlet Child Window', icons are used to represent real objects, such as valves, bellows, pressure gauges, etc. (clicking on these icons can produce an action or open a dialog box).
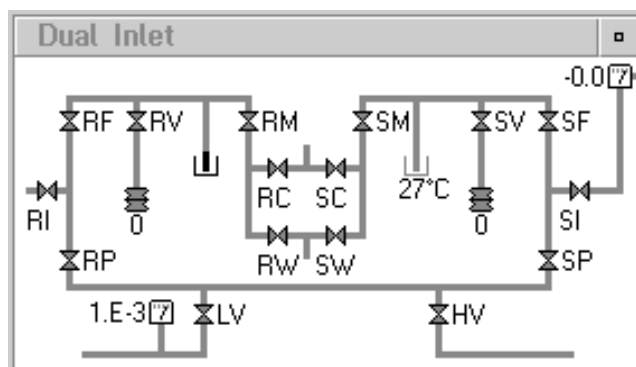


## Iconising A Window

When any of the child windows are minimized, they will appear in the ICON AREA at the bottom of the main PRISM window. Windows that can be iconized are the Message Window, the Scan Window, the Data Acquisition Window, the Dual Inlet Window, and the Preparation System Window. The Example above shows the Dual Inlet and Message windows iconized.
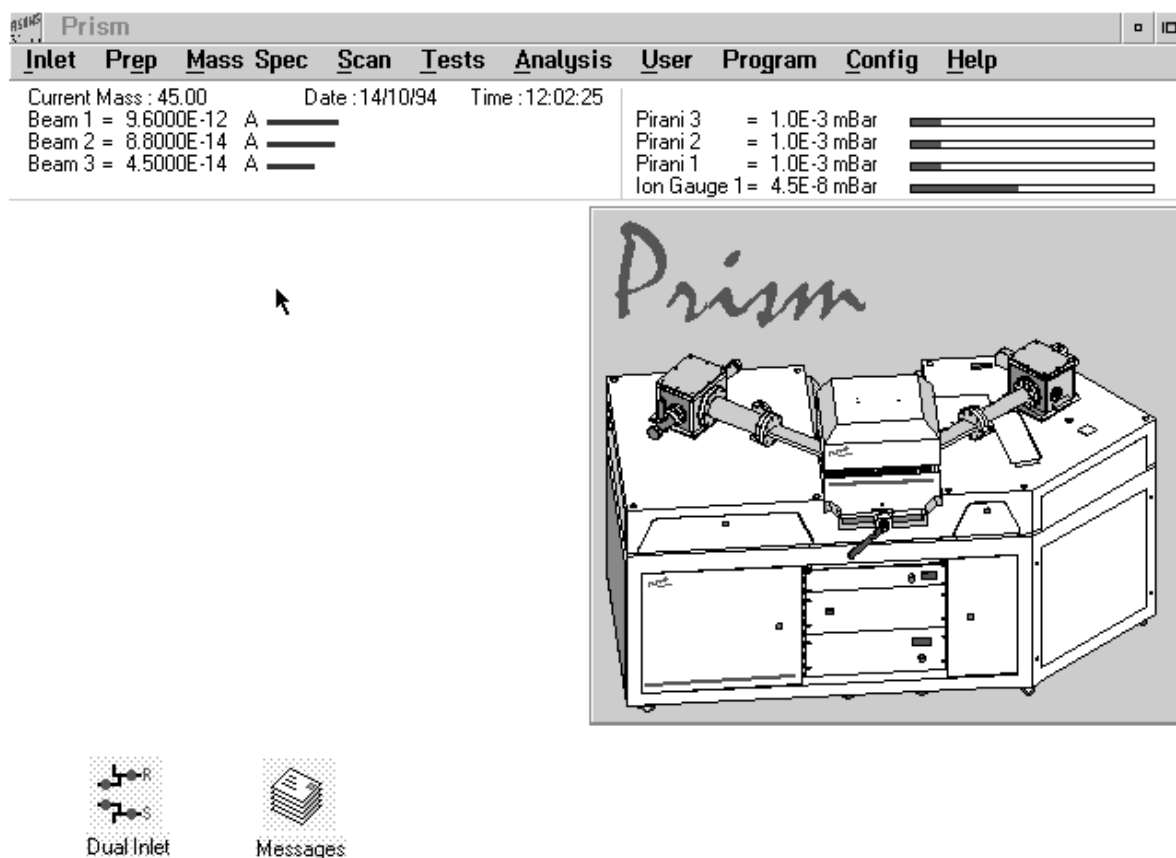
## Restoring an Iconised Window

If you double click on the icon , the window will be restored to its previous size. If the iconised window had a System Menu, a single click on the icon with the right mouse button will access a menu with options to restore, reposition or maximize the window.

# Using The Keyboard

## Selecting Menus

Each of the Main Menus has an underlined letter. Keying this letter whilst holding down the ALT key will select that menu. Items within the menus can also be selected by using the underscored key.

**Note:** The System Menu is selected by ALT SPACE.

## Moving the menu selection bar

The selection bar can be moved with the cursor keys. When the desired option is highlighted (shown in black), it can be actioned by pressing the return key.

## Special Keys

Certain keys have special applications , or have been allocated as "Hot Keys" which speed up software operation  without needing to use the mouse. These are listed and explained below.

- **F1**
  The F1 key will open the Help Window. If you are using a part of the software for which detailed Help is available, the appropriate Help Text will be displayed.

- **Esc (Escape)**
  This will leave the Help Window  and most dialog boxes without causing any action.

- **Enter (or carriage return)**
  Usually this will action the OK  or Run Button in a dialog box.

- **Tab**
  This key is used to move between different fields in Dialog Boxes.

- **Cursor Keys**
  These are used to move within dialog box fields,  and between Menus and Commands.
  They are also available to control the bellows motors after the Bellows Dialog Box has been selected. The arrow keys give a fine adjustment, the Page Up and Page Down Keys  a coarse step.

- **Alt**
  Holding the Alt key down while keying the Underlined letter in a Menu or Command will select that option.

- **Control**
  The control Key is used in the same way as the ALT key to access certain commands quickly:

Ctrl + A - Autorun Start

Ctrl + C - performs a Peak Centre

Ctrl + D - opens the DAPC Edit Dialog Box

Ctrl + G - opens the Ion Gauge Control Box

Ctrl + H - opens the H3+ calibration  dialog box

Ctrl + I  - opens the Identify peak dialog box

Ctrl + J  - starts a peak jump

Ctrl + Q - performs the peak quality routine

Ctrl + R - Runs an analysis

Ctrl + S - Stops a scan and  closes the Scan Window

Ctrl + X - Runs a scan from the parameter file

Ctrl + Y - Opens the Scan window and allows setting of Y - axis
parameters.

Ctrl + Z - Zeros the Amplifier.

For example**:** To toggle the changeover valve with the keyboard: Hold
down the Alt key and key I (Inlet) and T (Toggle changeover) in turn.

## Dialog Boxes

You can move between entry fields in Dialog boxes by using the TAB key
and cursor keys. The buttons in dialog boxes such as Yes, No, Help, etc.,
are controlled via pressing ALT and the underlined letter or using the
cursor keys to give focus to the key you need to select and then pressing
the return key.

## Menu

Menus are a list of functions which can be accessed by selecting the menu
item from the list. After selecting (clicking on the menu item or using one
of the special keys) some action will take place or the software will
require some further information from the user or further menus will be
revealed.

**Note:** If a menu or button has a **'...'** it means that there is a dialog box
activated by this menu or button, rather than starting a task.

The menu lists will be discussed in further detail in later sections of this
manual, however there are some features of menus that need to be
discussed at this stage.

## Menu Tick (On/Off)

When a tick appears at the right hand side of a menu item then that function is selected, when the menu item is again selected the tick will disappear hence de-selecting the menu item function. This therefore indicates the application of a toggle type function on the menu item. An example this can be found on the 'Zoom In' menu item in 'Scan Display Window' (see later for details of Scan Display Window).

```
✓ Zoom In
  Zoom Out
  Full Size
  Pan
  Drag
  RawData
  Link Zoom x
```
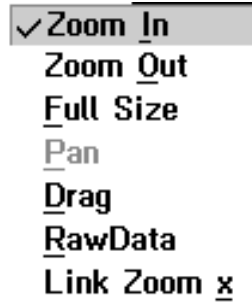
## Sub Menu Arrow

If an arrow appears on the right hand side of a menu item then that item will have a sub menu. Therefore another menu will be below the original menu. An example of this can be seen in PRISM software 'Help' menu option 'Language'.

```
✓ English Help            Language        →
  Aide en Français
```

## Grey Menu Items

If a menu item appears grey instead of black, then that menu item cannot be accessed at that time. An example of this can be seen in the System Menu when the window is at full screen the 'maximize' option is greyed out, as this has no function when the window is already maximized.

```
      Prism
  Restore         Alt+F5
  Move            Alt+F7
  Size            Alt+F8
  Minimize        Alt+F9
  Maximize        Alt+F10
  Hide            Alt+F11

  Close           Alt+F4

  Window list     Ctrl+Esc
  Arrange Windows
```

# The User Interface

This section of the manual describes the input controls available in OS/2, however the examples will be taken from the PRISM software. The main example will be the Data Acquisition Control Parameters Dialog Box as this shows most of the required controls.
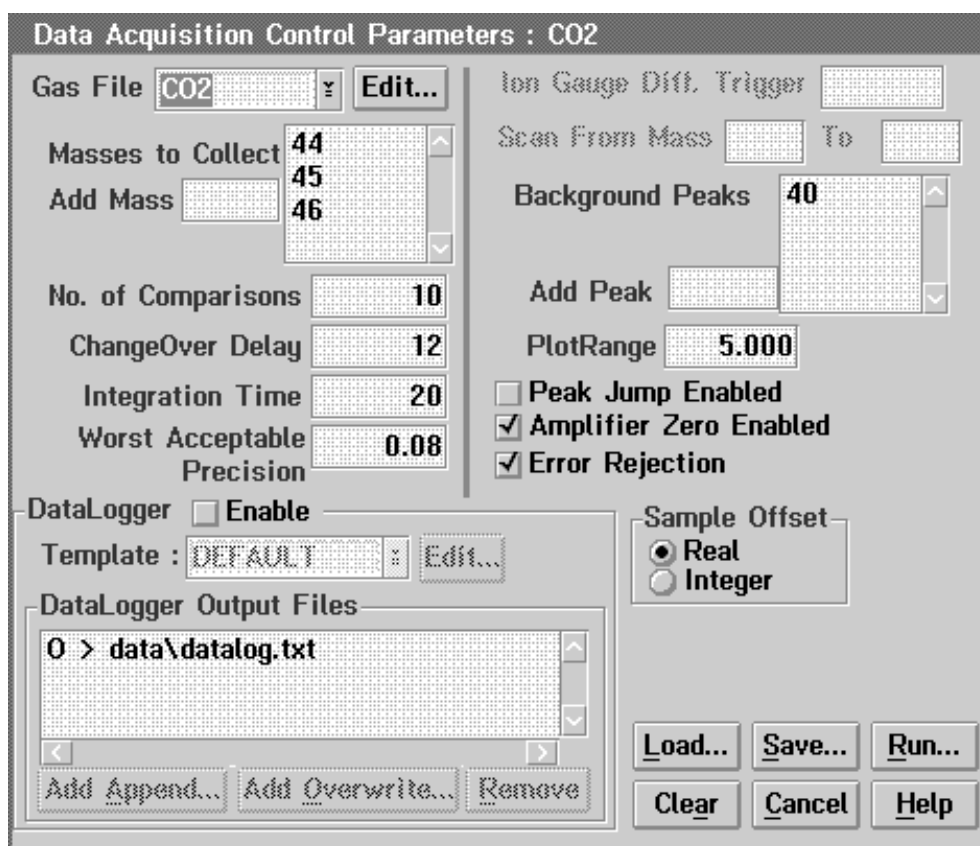


## Dialog Box

Dialog boxes are windows through which you can answer requests for data or provide information. They may contain any of the controls to be discussed later in this section, for example: entry fields for text or numerical input, push buttons for Help, To OK or Cancel an action, and 'Check Boxes' or 'Radio Buttons' to select or de-select options.

Dialog boxes generally do not have System Menu and can therefore not be minimized, etc. This generally means they take precedent over any other function and must be answered or cancelled prior to other user operations.

The operational sequence facility available in the software enables you to create your own dialog box questions for instrument control.

## Radio Button



These have an on/off function if they are on their own, however the more usual example is as part of a list within a box, where if one function from the list is chosen, the other functions are de-selected (there can be various boxes each containing a different set of radio buttons). In our example if 'Real' is chosen then 'Integer' is turned off, and visa versa. A radio button is selected by clicking on the circle next to the item description, on is indicated by a solid black dot in the centre of the circle.

**Check box**



These have a toggle function where the option is selected or not. Selection is shown with a tick or a cross in the box and is achieved by clicking on the box next to the item description. An example above is the Error Rejection Check Box.

**Push Button**



These perform a command when selected, for example saving data, calling the help file, opening another dialog box, etc. The default Push Button is emphasised by a heavy border and is selected by simply pressing the keyboard Return key. An example above is the Help Push Button.

**List Box**



This gives the user a list of options to be chosen from. These appear in alphabetical order and can be selected with a double click or by a single click followed by a command (Push Button). If the list is longer than the box the list can be scrolled up and down using the scroll bar at the side of the list. The items are selected with a single click and activated with a double click. An example above is the datalogger output files box.

**Entry Field**
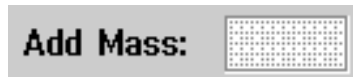


This is an area set aside to accept user inputted data into the dialog box, for example file names, data, etc. An example above is the Add Mass entry field.

**Combo Box**
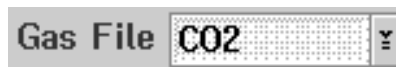


This is a combination of an entry field and a list box. When the arrow at the side of the box is clicked on a list box appears from which the various options can be selected as with a list box. Alternatively if the user knows the option name then this can be typed directly into the entry field of the combo box. An example above is the Gas File Combo Box.

## Scroll Bar

**Accelerating Voltage**  ‹ [_____] › | 4501.3

This is sometimes known as a slider bar and can be horizontal or vertical. The purpose of the Scroll Bar is to move a list (see list box)or control a mass spectrometer parameter, for example the source tuning controls.

Clicking on the arrows will move the slider a small amount, clicking to the side of the slider moves the slider a medium amount and if large moves are required then drag the slider by clicking and holding down the mouse button and moving in the direction required.

## Tab

You can move between entry fields in Dialog boxes by using the TAB key and cursor keys.

## Greyed Functions

**Template :** DEFAULT  ▼ | Edit...

If a function is in grey text rather than black text, then that function is not available at this moment. For example if the datalogger is disabled then the datalogger options are not required and are therefore greyed out.

# Printing

When a job is sent to the printer then a print job object will be created within the printer folder. This print job object represents your print job in the queue. It is possible to view any queued jobs by double clicking on the icon.

**Note:** If the spooler is disabled then jobs are sent directly to the printer and they will NOT appear in the printer folder.

There are a number of ways that you can print :

The application you are running generates a print job.

Use the Print facilities available in the software.

Use the 'Print' command from the DOS or OS/2 command line.

Drag an object and drop it onto a printer object.

Position the pointer over the dialog box or window that you wish to print and press the "Print Screen' key to dump the dialog box or window to the printer.

# Multitasking

The multitasking capabilities extend the power of the software enormously.

These are of two levels of multitasking.

First, each program that runs under OS/2, such as the PRISM software, may be "multithreading" i.e. many processes can be active simultaneously. For example you can be performing an automatic analysis whilst preparing samples on another part of the system, carry out data acquisition simultaneously with any other activity, tune the ion source whilst scanning etc.

Secondly, the system has true "multitasking" i.e. more than one program or "application" can be running at any given time. For example you can be running the instrument at the same time as analysing data with a spreadsheet and whilst writing a report with a word processor.
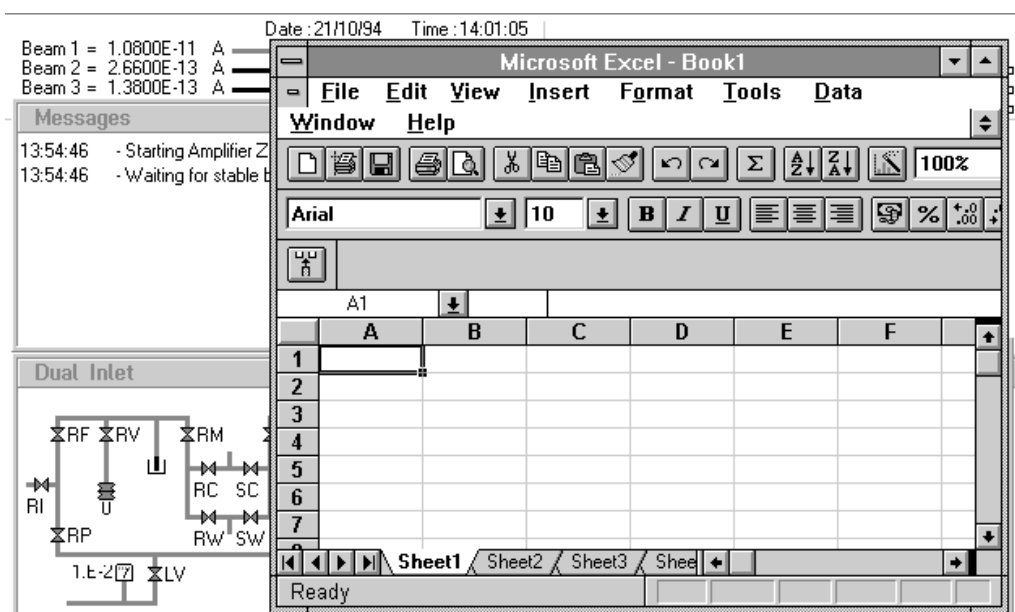
# PRISM Operating Environment

This section of the manual will specifically describe the PRISM Operating Environment. Some of this section will however have been described as examples in the previous section on the OS/2 Environment.

## Screen Layout

The entire PRISM software runs as an 'Application Window' with various 'Child Windows' running inside it. The application window and / or the child windows can be re-sized to fill the entire screen area, or they can be re-sized / repositioned to occupy a screen area of your choice. For example, you may wish to reduce the screen area used by the PRISM if you are using a spreadsheet application. The software can minimized and run as a icon if desired.
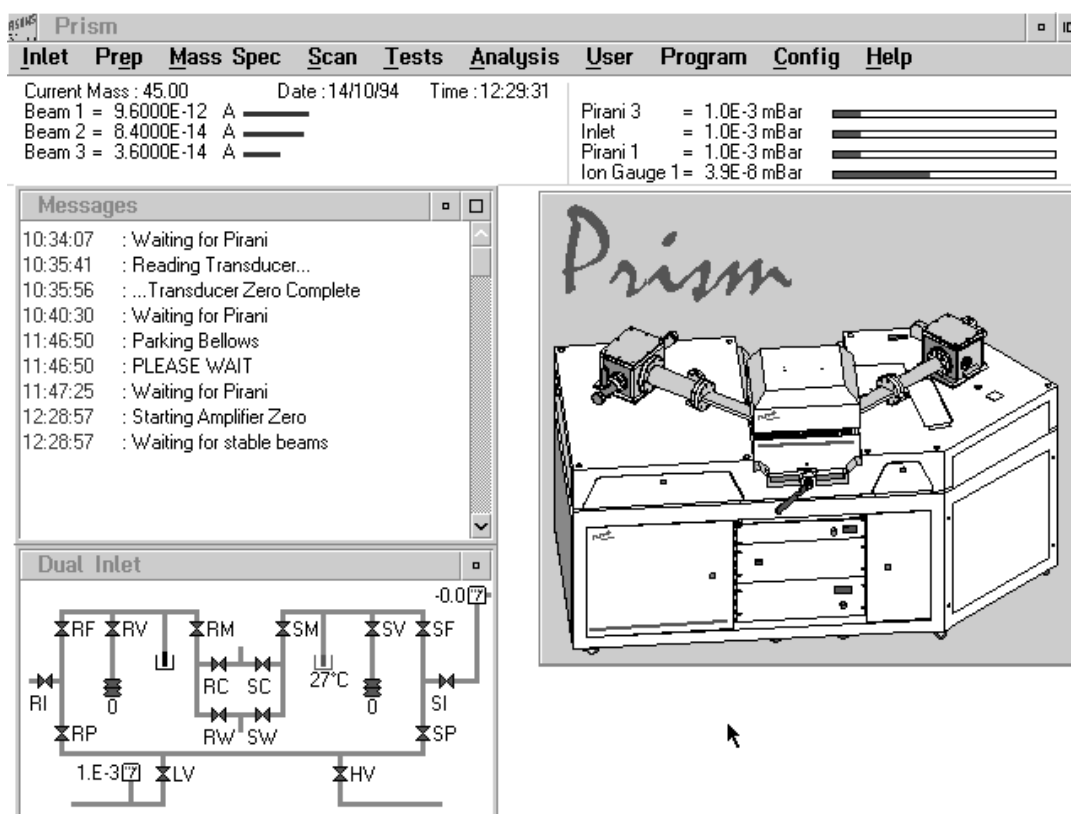


The Application Window and its child windows form four main areas.

Below the Menu Bar is the Monitor Window, which contains numerical and graphical readouts of the ion beams and vacuum gauges. This cannot be moved, re-sized, or iconized.

Below the Monitor Window is the main work area of the PRISM software. The Message Window appears in this area. Additional windows, and dialog boxes, etc. will appear here as you use the software.

At the bottom of the screen are windows which contain representations the appropriate preparation system valves, etc. fitted to your machine (Dual Inlet, etc.).These are user definable and will depend on which inlet and / or preparation system are fitted. They can be minimized to icons if required.

# Main Menu Bar

The diagram below shows the Main Menu Bar from which the operator gains access to the rest of the pull down menus available in the PRISM. The Main Menu Bar is therefore the primary menu.

## Choosing Commands Using The Mouse

For example, to choose the Mass Spec, Tune Source command:

Click the Mass Spec menu option in the Menu Bar.
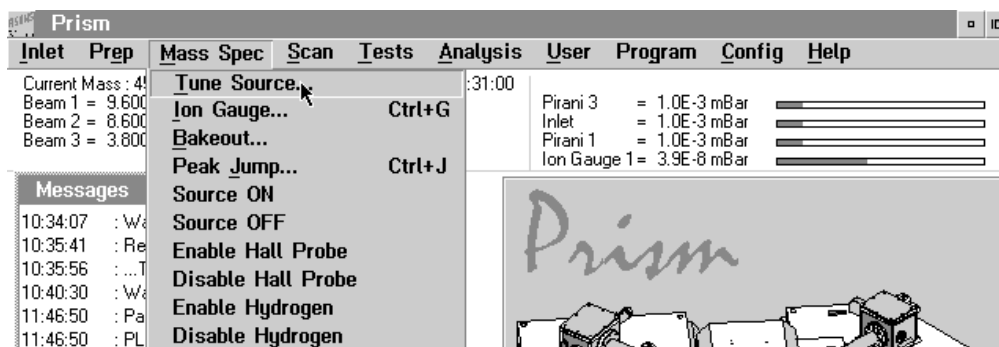


The Mass Spec menu appears with a list of commands.

Click the desired command, in this case, Tune Source

After you click the command, the menu disappears. Your command will either perform an action or display a dialog box to get more information from you.

Click the cancel button to cancel a dialog box.

## Choosing Commands Using The Keyboard

Using the same example as above to choose the Mass Spec, Tune Source command with the Keyboard:

Notice that there is an underlined letter in each of the menu names. For the Mass Spec menu, the underlined letter is a M.

Press ALT and hold down and type the letter M.

The Mass Spec menu appears.

Some of the commands in the menu have an underlined letter. For the Tune Source command, the letter T is underlined

Type the letter T to choose the Tune Source command.

Press ESCAPE to cancel a dialog box.

# The Monitor Window

The monitor window is situated just below the Main Menu Bar at the top of the screen. Unlike the other windows, it cannot be re-sized or iconised. It is used to display important information about the mass spectrometer: The right hand half shows the vacuum gauge status (Numeric and Graphical Displays), and the left hand half shows the Ion Currents (Numeric and Graphical Displays), as well as the information on the Current Mass (mass in the axial collector) and the date and time.



## Vacuum Gauges

The right hand half in our example shows the readings from three Pirani gauges and an ion gauge.

**Note:** This area can display up to 5 gauges which are user definable.

The pressure in milli-Bars is shown next to a horizontal bar which gives a graphical readout of the pressure. The bars are colour coded : green indicates pressure in the normal safe range, yellow a warning of high pressure, and red indicates an unsafe high pressure.

## Ion Beams

The signal from 3 Faraday Detectors (2 in the case of Hydrogen) is shown as Amps to the left of a horizontal bar which gives a visual impression of the beam intensity. On the PRISM which has 4 Faraday Detectors the 3 detectors to be displayed depends on the current mass selected. The bars are on a logarithmic scale.

# The Message Window

The message window appears in the left hand half of the central screen area. It can be re-sized or iconized if required, but has no System Menu and therefore cannot be closed down. This window is used to display text messages from the software. If you wish you can send messages to the text window from your own programs.

| Messages | | |
|---|---|---|
| 10:34:07 | : Waiting for Pirani | |
| 10:35:41 | : Reading Transducer... | |
| 10:35:56 | : ...Transducer Zero Complete | |
| 10:40:30 | : Waiting for Pirani | |
| 11:46:50 | : Parking Bellows | |
| 11:46:50 | : PLEASE WAIT | |
| 11:47:25 | : Waiting for Pirani | |
| 12:28:57 | : Starting Amplifier Zero | |
| 12:28:57 | : Waiting for stable beams | |
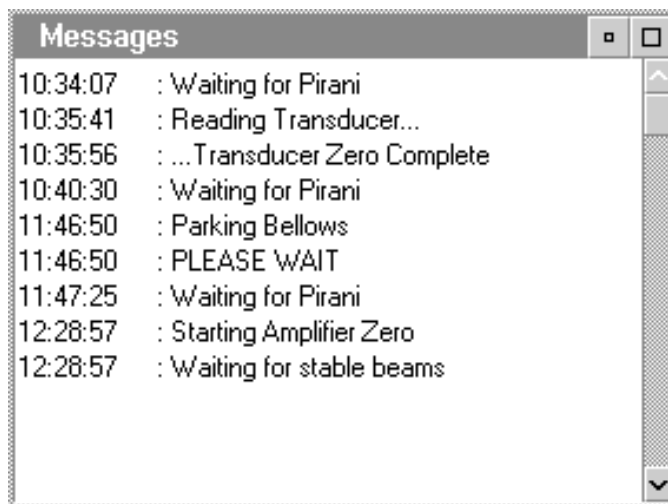
The message consists of a time for the message followed by the text. After the display portion of the window is full, it is possible to step backwards and forwards through the messages via the scroll bar at the right hand side of the window. This window forms a commentary on the operations performed which provides a record of events during an overnight automatic run, for example.

# The Mimic Diagram Windows

The Mimic Diagram windows normally appear at the bottom of the screen area. They can be re-sized or iconized if required, but as with the message window they have no System Menu and therefore cannot be closed down.

These windows are used to display graphical representations, made up of icons, of the Inlet system (see below for details) and the various preparation systems (see the appropriate preparation system sections of this manual for details) available for use on the PRISM. The preparation systems can be extended or modified by the user to allow for expansion of the system.

Most of the icons are interactive, allowing operation of them by pointing the mouse at them (the mouse pointer will change shape when correctly positioned over the icon) and clicking once with the left mouse button. This will bring up the dialog box for the icon allowing valve operation, bellows control, cold finger control, etc.

## Dual Inlet Window (Mimic Diagram)

The window contains a graphical representation of the inlet system. In this the valves are represented by coloured icons. A red icon is a closed valve, a green icon an open valve. The identity of each valve is shown by a two letter mnemonic next to its icon. Valves in the reference half start with "R", in the sample half with "S". The other letters are :

I    - Inlet Valve, to either reference bottle or sample preparation systems.
P   - Pump Valve, opening to the pump channel.
V   - Variable Volume or bellows isolation valve.
F   - Cold Finger isolation valve.
M  - Mass Spec valve, opening inlet to changeover valve.
C   - Changeover valve, opening to the ion source.
W  - Waste valve, opening from changeover valve to waste pumping line.
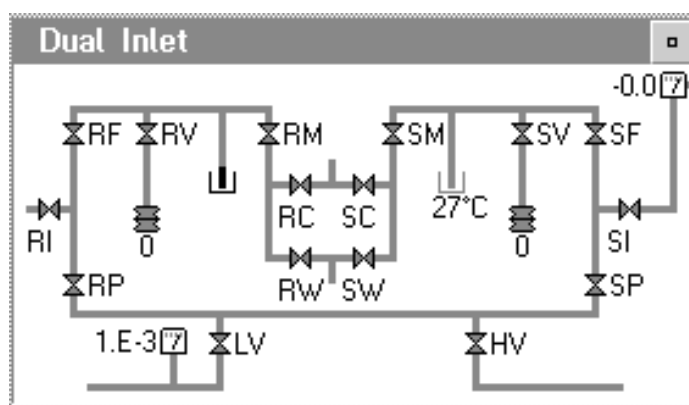


There are two additional valves :

LV - Low Vacuum pump valve, for rough pumping of the inlet.
HV      - High Vacuum pump valve, for high vacuum pumping of the inlet.

The valves are shown connected by the appropriate pipework (this is only a graphical representation - pipe lengths are not accurate).

The three other interactive icons in the mimic diagram are the 2 bellows icons (reference and sample) and the cold finger icon. The cold finger temperature and the bellows positions are shown next to the icons and are constantly updated.

The pressure gauges associated with the dual inlet are also shown in the dual inlet window, but these are not interactive. The pressure transducer is next to valve SI in the sample transfer line, and Pirani 2 (P2) in the backing line next to LV. The pressure is shown next to each gauge in mBar and which are again constantly updated.

# PRISM Menus

Across the top of the Main Window is the PRISM Main Menu Bar, from which further menus can be selected to control the mass spectrometer. For details of menu selection please see previous section.

| Prism | | | | | | | | | | □ |▭| |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Inlet | Prep | Mass Spec | Scan | Tests | Analysis | User | Program | Config | Help | | |

This section of the manual will therefore give a description of all the menus as they appear on the screen.

## PRISM Inlet Menus

The dual inlet is operated via the Dual Inlet Window and the PRISM Inlet Menu. This section can therefore be looked at in two parts, firstly the PRISM Inlet Menu and secondly from the Dual Inlet Window details to follow below:

## Inlet

| Inlet | Prep | Mass Sp |
|---|---|---|
| **Close All Valves** | | |
| Toggle C/O Valve | | |
| Reset Change Over | | |
| Enable/Disable CO | | |
| Pump Out Ref | | |
| Pump Out Sam | | |
| Pump Out All | | |
| Park Bellows | | |
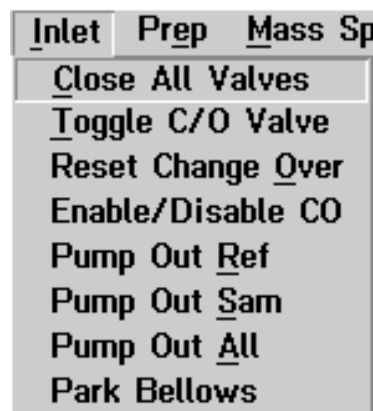
The menu options are :

### Close All Valves

This closes all valves in the dual inlet (all the valves shown in the dual inlet mimic diagram, except for RW and SW which remain open).

### Toggle C/O Valve

Switches the changeover valve between reference and sample sides of the Dual Inlet, therefore if gas is entering the analyser from the reference side of the inlet then the sample side is being pumped to the waste line and vice versa. If the sample side is open to the analyser then this changes to the reference side and vice versa in a toggle action.

## Reset Change Over

This closes the changeover valve to the analyser, closes valves RC and SC, and opens valves RW and SW.

## Enable/Disable CO

Enables or disables the changeover valve (toggles between them and gives a message in the message window as to the state). If the changeover valve is disabled then the **'Toggle C/O Valve'** command has no function until it is enabled again. This function is particularly useful when testing the inlet for contamination.

## Pump out Ref

Pumps out the reference side of the inlet including the reference port. Care must therefore be taken to ensure the reference port is closed before this command is chosen.

## Pump Out Sam

Pumps out the sample side of the inlet (not including the sample port).

## Pump Out All

Pumps out both sides of the inlet (not including the reference or sample ports).

## Park Bellows

Sets both bellows to the zero position (opens them to the fully open position, which is the lower flag reset).

# Dual Inlet Window (Mimic Diagram)
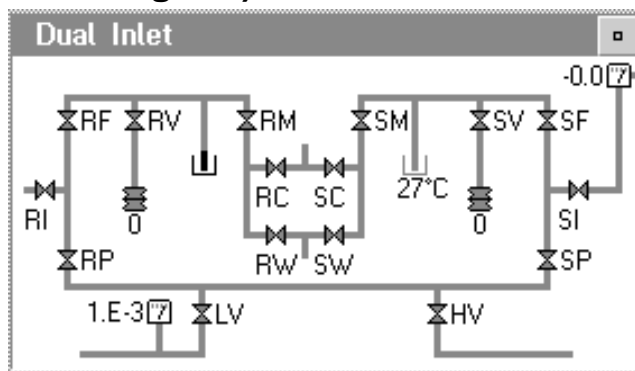


The options are:

## Valves

Use the mouse to point to the desired valve in its mimic diagram window.

**Note:** the pointer will change size when in range.

Click on the valve icon and the **'Valve Control'** dialog box for that valve will appear, the radio buttons close or open indicate the state the valve will go to on clicking OK.
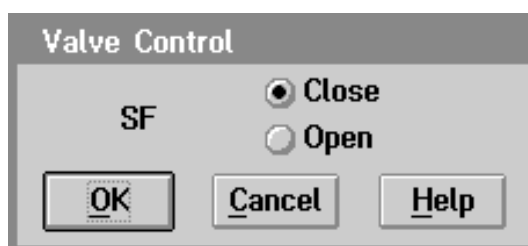


To toggle the valve either (i) click on the **'OK'** push button or (ii) press the return key on the keyboard.

**Note:** The radio buttons **'Close'** and **'Open'** can be disregarded as the valves toggle i.e. if the valve is open the next valve dialog box **'OK'** will close the valve and vice versa.

The **'OK'** push button appears at the current pointer position, so in practice, a double click toggles the valve.

The pump valve HV (and MH on the preparation systems) is the exception to the above operation (see below for details).

The Valve control dialog box also has the following push buttons:

**'Cancel'** will exit the dialog box without taking any action.

**'Help'** gives information on the operation.

**Note:** The pump line valves obey special rules. The pairs LV/HV ML/MH cannot be opened simultaneously as opening LV or ML will close HV or MH respectively.

Opening HV will invoke the following sequence ; LV will be opened and HV will only be switched to open when Pirani 2 reads less than $10^{-2}$ mBar. Opening MH operates in the same way with ML.

Closing HV or MH operates as usual.

## Bellows Adjustment

Click on either one of the two bellows icons in the dual inlet mimic diagram and the **'Bellows Control'** dialog box for the bellows chosen will appear.
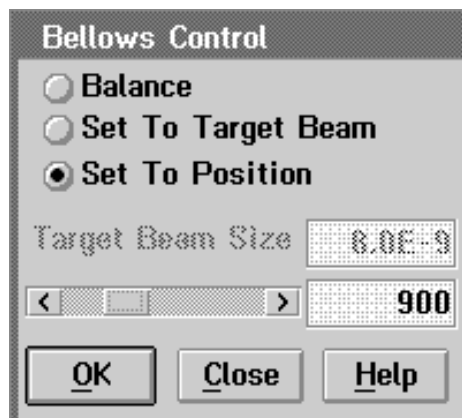


This contains the following radio buttons:

**'Balance'** when selected followed by the **'OK'** push button, reads the ion current on the opposite side of the inlet and then uses the bellows selected to balance the two ion currents of the major beams. If balance cannot be achieved the bellows are left in a position closest (usually top or bottom as the beam is too small or too large). The slider bar and the **'Target Beam Size'** entry field are greyed out and can therefore not be accessed if **'Balance'** is selected.

**'Set To Target Beam'** when selected followed by the **'OK'** push button sets the ion current (in Amps) of the major beam to the value selected in the **'Target Beam Size'** entry field (if the target cannot be reached the software will leave the bellows fully closed so the beam size is as near to the target as possible). The slider bar is greyed out and can therefore not be accessed if **'Set To Target Beam'** is selected.

**'Set To Position'** is the default radio button and when selected allows the bellows to be adjusted manually using the slider bar below. The slider bar works in the same fashion as a scroll bar. The entry field to its right both displays the bellows position in stepper motor steps and can be used to enter a new stepper value, by overwriting the value in the entry field and then clicking anywhere on the scroll bar.

The dialog box also contains the following push buttons:

**'OK'** starts the bellows action selected by the radio buttons above and closes the dialog box.

**'Close'** will exit the dialog box without taking any action.

**'Help'** gives information on the operation.

## Cold Finger Operation

To operate the cold finger click on the cold finger icon in the dual inlet mimic diagram and the **'Cold Finger Parameters'** dialog box will appear.

**Temperature Control**

Temperature  -100  °C
Duration  5  Mins

| Heat | Cool | Stop | Cancel | Help |

The dialog has the following entry fields:

**'Temperature'** allows the temperature at which the cold finger is controlled, to be entered. If no temperature is entered then the cold finger will go to its maximum or minimum settings (i.e. 150ºC if **'Heat'** is chosen or -200ºC (liquid N$_2$ temperature) if **'Cool'** is chosen)

**'Duration'** allows the time the cold finger is controlled at the set temperature, to be entered. If no duration is entered then the cold finger will go to its set temperature until the **'Stop'** command is given.

The dialog box has the following push buttons:

**'Heat'** turns on the heater wound round the cold finger and heats the cold finger to the temperature set in the **'Temperature'** entry field and holds at that temperature for the time set in the **'Duration'** entry field.

**Notes:**

a)  If no temperature has been set then the software will control the temperature at 150ºC.
b)  If no duration has been entered then the cold finger will go to its set temperature until the **'Stop'** command is given.

**'Cool'** uses a combination of the Charles Austen Pump (used to pump liquid Nitrogen into the cold finger) and the heater to cool the cold finger to the temperature set in the **'Temperature'** entry field and holds at that temperature for the time set in the **'Duration'** entry field.

**Notes:**

a)  If no temperature has been set then only the Charles Austen pump is used and the cold finger  temperature will achieve liquid Nitrogen temperature.
b)  If no duration has been entered then the cold finger will go to its set temperature until the **'Stop'** command is given.

**'Stop'** will turn off the heater or the Charles Austen pump depending which is operating.

**'Cancel'** exits the dialog box without taking any actions. The cold finger will continue heating or cooling for the time set even though the dialog box is closed.

**'Help'** gives information on the operation.

## Prep

This menu is different from the other menus in that its contents will depend upon which preparation systems are present on your system. The menu is therefore user definable and will not be discussed in this section. Please refer to the Preparation System sections later in this manual.

## Mass Spec

This menu contains all the necessary commands to effectively operate the mass spectrometer.
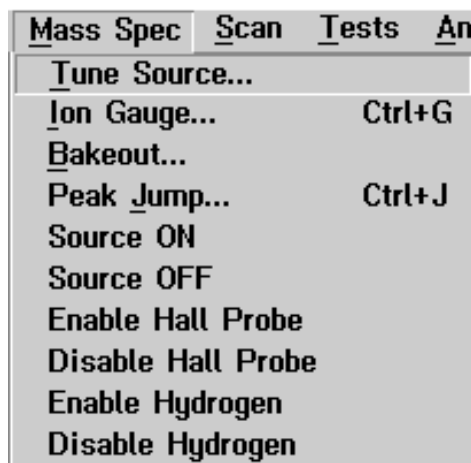


The options are :

### Tune Source

This menu item enables adjustment of the source potentials. It opens the **'Tune Source Electronics'** dialog box, which can be moved around the screen if required.
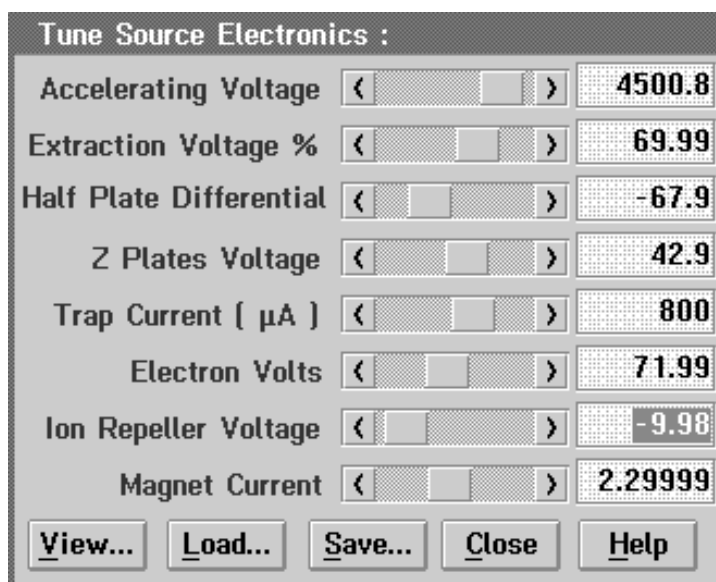


As can be seen the dialog box controls the following parameters:

**Accelerating Voltage** between 0 and 5kV

**Extraction Voltage** between 0 and 100% of the accelerating voltage

**Half Plate Differential** between -150V and +150V

**Z Plate Voltage** between -225V and +225V

**Trap Current** between 0 and 1000µA

**Electron Volts** between 50 and 100 V

**Ion Repeller Voltage** between -15V and +50V

**Magnet Current** between 0 and 5 Amps

To alter an individual tuning parameter either enter the new value in the entry field next to the source parameter (and click on another box) or use the scroll bar.

The dialog box has the following push buttons:

**'View'** allows stored tuning files to be viewed or deleted via the **'View Tuning'** dialog box. This facility therefore allows the parameters to be checked for accuracy and safety prior to loading.
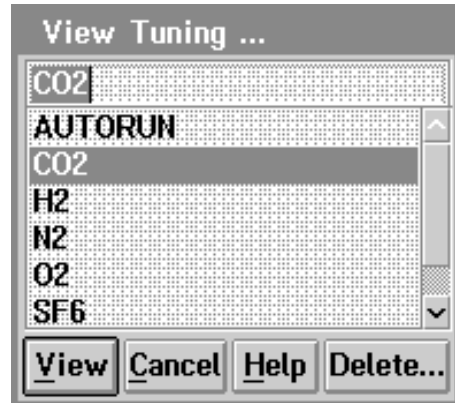


This dialog box has the following push buttons:

**'View'** loads the source tuning file selected from the list box. This opens the dialog box below which gives a list of the source parameters for the chosen file.
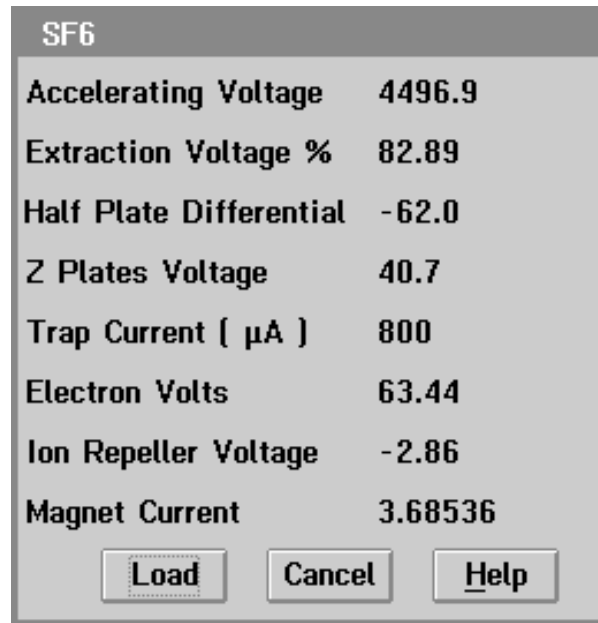


From here the tuning file can be loaded using the **'Load'** push button. The dialog box can be closed using the **'Cancel'** push button or user help can be obtained using the **'Help'** push button

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the source tuning file chosen from the list box. A dialog box is opened to confirm you wish to delete the file

**'Load'** opens the **'Load Tuning'** dialog box, which allows the source tunings files for the various gas species to be loaded (or deleted).
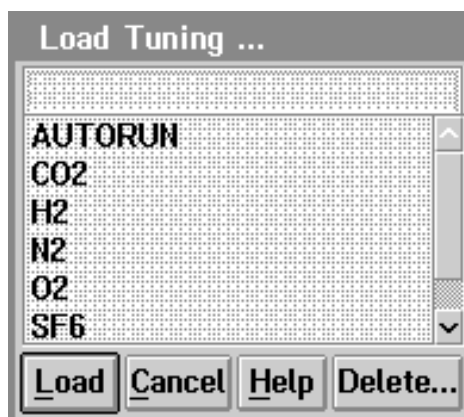


This dialog box has the following push buttons:

**'Load'** loads the source tuning file selected from the list box. The tuning parameters in that file are immediately loaded (if unsure then use View first).

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the source tuning file chosen from the list box. A dialog box is opened to confirm you wish to delete the file.

**'Save'** opens the **'Save Tuning file'** dialog box, which allows a tuning file that has been changed or a new tuning file to be saved.
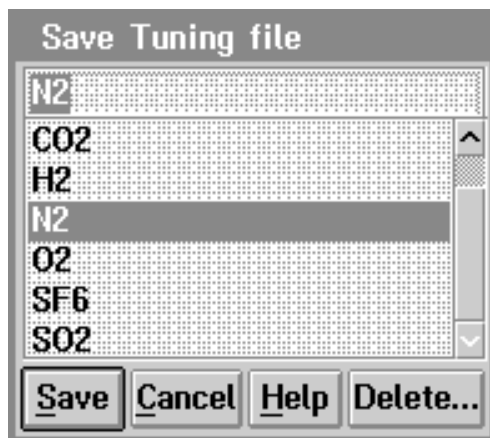


The dialog box has the following push buttons:

**'Save'** saves the tuning file under a new file name by typing in a new name in the entry field or can be over written on an existing file name by selecting a name from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the source tuning file chosen from the list box. A dialog box is opened to confirm you wish to delete the file

**'Close'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Ion Gauge

This menu option when selected enables control of the ion gauges via the 'Ion Gauge Control' dialog box.
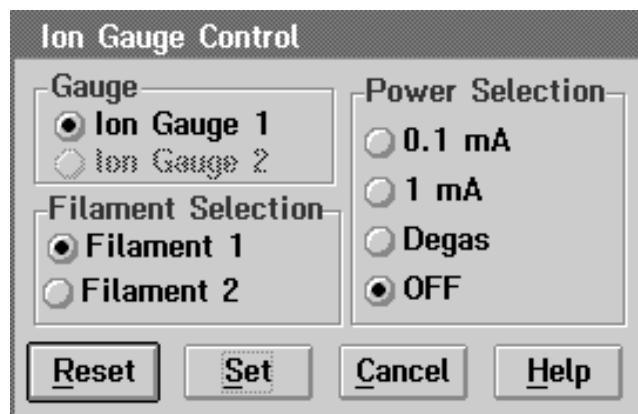


This dialog box has the following radio button sections:

Gauge - The PRISM can have two ion gauges fitted, if the Differential Pumping option is chosen. Thus the rest of the controls in the Ion Gauge Control dialog box act on either 'Ion Gauge 1' or 'Ion Gauge 2' depending on which of the radio buttons is selected.

**Notes:**

a) Selecting and controlling either ion gauge does not effect the other gauge.

b) If the system does not have a second gauge, the Ion Gauge 2 radio button is in grey not black script (for details of selecting ion gauges and setting up the monitor window see further sections of this manual).

**'Filament Selection'** - As discussed in previous sections of this manual the ion gauge has two filaments, to save the need for venting the system after filament failure. The filament in use on a particular ion gauge is selected by the **'Filament 1'** or **'Filament 2'** radio buttons on the left of the dialog box.

**'Power Selection'** - The Filament can be set in any of 4 states:
0.1mA  - sets the filament to 0.1 mA emission (recommended emission).
1.0mA  - sets the filament to 1.0mA emission.
Degas  - used to outgas the filament.
Off

The dialog box also has the following push buttons:

**'Set'** actions the selected options for the ion gauges and closes the dialog box.

**'Reset'** sets the ion gauge to off and must always be used between filament selection changes, or when switching from degas to pressure measurement.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Bakeout

This menu option when selected enables control of the bakeout temperatures and times via the 'Bakeout' dialog box.



Bakeout of the mass spectrometer (**Analyser**), dual inlet (**Inlet**), and the manifold (**Manifold**) are controlled from this dialog box, which enables a time / temperature profile with up to 3 set points to be programmed for each of the three areas of the instrument. The temperatures are entered in the **'Temperature'** entry fields and the time at that temperature is entered in the **'Duration'** entry fields.

**Notes:**

a) Each of the three areas can have different temperature profiles.

b) Do not have to bake all the sections (e.g. can bake only the Analyser)

c) Can bake using only one temperature and one duration.

The Bakeout dialog box has the following push buttons:

**Start'** starts the bakeout, using the profiles entered in the entry fields

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

| CAUTION |
|---|
| **It is essential to remove the head amp prior to baking out the analyser** |

## Peak Jump

This command allows you to position another mass in the central collector by moving the HT voltage or the magnet current. It opens the 'Peak Jump' dialog box, which can be moved around the screen if required.

```
┌─────────────────────────────────────┐
│ Peak Jump                            │
│        Current Mass : 45.0           │
│   Mass Calibration HT : 3547.0       │
│          Current HT : 3552.5         │
│                                      │
│   Mass to jump to    │40      │      │
│                                      │
│  ○ Magnet  Jump    □ Peak Centre     │
│  ● HT  Jump                          │
│  ┌──────┐   ┌────────┐   ┌──────┐    │
│  │  OK  │   │ Cancel │   │ Help │    │
│  └──────┘   └────────┘   └──────┘    │
└─────────────────────────────────────┘
```
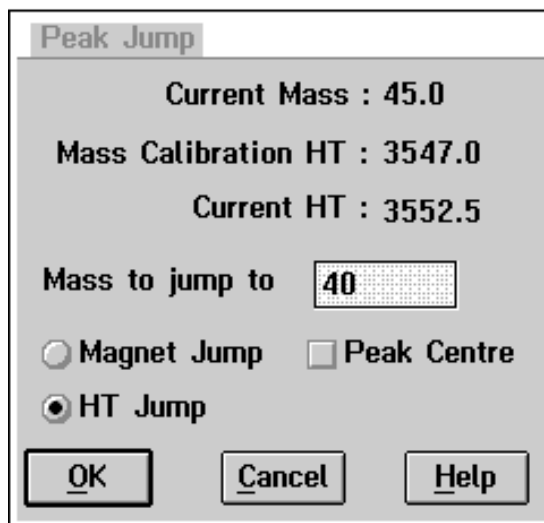
This dialog box displays the following:

**'Current Mass'** (the Identified mass which can be seen in the monitor window)

**'Mass Calibration HT'** (this is the source voltage at which the mass calibration with respect to the magnet current was taken)

**'Current HT'** (the source voltage, which for an accurate magnet peak jump must be the same as the mass calibration HT).

The new mass to be focused in the axial collector is entered in the **'Mass to jump to'** entry field. The option is then whether to **'Magnet Jump'** or **'HT Jump'** by selecting one of the radio buttons. After peak jumping the software can be asked to perform a peak centre, by selecting the **'Peak Centre'** check box.

The dialog box has the following push buttons:

**'OK'** starts the peak jump routine.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Source On

This menu option when selected turns **ON** the power to the source electronics. No source potentials are loaded, but the filament is turned on.

## Source Off

This menu option when selected turns **OFF** the power to the source electronics.

## Enable Hall Probe

This menu option enables the magnet to function with the Hall Probe (i.e. Field Mode), giving a message in the message window 'Hall Probe Enabled'.

**Note:** This command should be ignored if there is no Hall Probe fitted to the system.

## Disable Hall Probe

This menu option disables the Hall Probe (i.e. magnet operating in Current Mode), giving the message 'Hall Probe DISABLED'.

**Note:** This command should be ignored if there is no Hall Probe fitted to the system.

## Enable Hydrogen

This menu option moves the Hydrogen magnet into position and switches the magnet current from the main magnet to the Hydrogen magnet. The system is then ready to run Hydrogen, after the **'Current Mass'** is specified as mass 2 in the **'Identify Peak'** menu option in the **'Tests'** menu.

**Note:** This command should be ignored if the Hydrogen option is not fitted to the system.

## Disable Hydrogen

This menu option moves the Hydrogen magnet out and switches the magnet current from the Hydrogen magnet to the main magnet, allowing for the higher masses to be measured.

**Note:** This command should be ignored if the Hydrogen option is not fitted to the system.

# Scan

This menu enables the user to perform all the necessary scan functions required, e.g. HT, magnet etc..
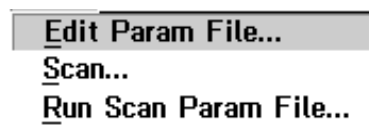


The menu options are :

## Edit Param File

This menu option accesses **'Load Scan Parameter File'** list box.
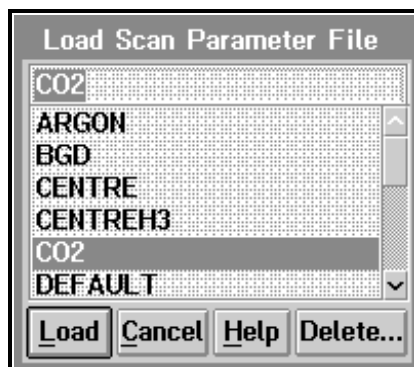


The list box enables you to select a scan parameter file to be edited (for details of editing scan parameter files see next section as the details are the same once the scan setup dialog box is opened) and has the following push buttons:

**'Load'** loads the **'Scan Setup'** dialog box (see next section for details) for the file chosen from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the scan parameter file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

## Scan

This menu option accesses **'Scan Setup'** dialog box which enables you to edit a set of scan parameters and save them on disc in a the **'Scan Parameter File'** (.SPF). This menu option loads the previously run Scan Setup file (or in the case of just starting the software the Default file).



The dialog box will be described in three areas as given below:

**'Select scan variable'**

This area contains the following radio buttons which enable you choose the X-axis variable to be scanned. (voltage, time or magnet)

**'Accelerating voltage'** allows the amplifier signals to be scanned with respect to the source accelerating voltage, keeping all other parameters at the same conditions.

**'Time'** allows the amplifier signals to be scanned with respect to time (useful when leak checking), keeping all other parameters at the same conditions.

**'Magnet'** allows the amplifier signals to be scanned with respect to the magnet current (or field if the Hall probe is enabled), keeping all other parameters at the same conditions.

**'Mnemonic'** is selected, a source parameter can be scanned by inputting the appropriate mnemonic into the entry field. The mnemonics must be entered as capitals and they are:

| | | |
|---|---|---|
| EX | - | Extraction Voltage |
| HP | - | Half Plate Differential |
| ZV | - | Z Plate Voltage |
| TR | - | Trap Current |
| EV | - | Electron Volts |
| IR | - | Ion Repeller |

**'Select Scan Range'**

This area controls the X axis variable of the scan. There are entry fields for scan width (**'Width'**), step size between points (**'Step'**), and integration time at each point (**'Integ Time'**).

The **'Default Scan'** check box is selected (ticked) then the scan range is input in the **'Width'** entry field, (this is the total width of the scan, e.g. if when scanning voltage 50V is entered in the entry field, then the scan will be ±25V around the current accelerating voltage). However if the **'Default Scan'** check box is not ticked, the **'Width'** entry field is no longer accessible and in this case extra entry fields become available **'Min X'** and **'Max X'**, which allow you to select the lower and upper limits of the x values (minimum and maximum to be used).

**Notes:** If a Time scan is selected, only the **'Max X'** entry field is used to enter the duration of the scan in seconds (there is no 'Min X' as time always starts at time zero), and the **'Step'** field is used to enter the time step.

If the **'AutoRepeat'** check box is selected then the scan will clear at the end of the scan, before starting the scan again (this is particularly useful when peak shaping).

**Lower Entry Field (Line Details)**

Each line in the bottom part of the dialog box sets up one trace on the scan (up to a maximum of four).

The fields are:

**'Collectors'**

Identifies the collector output to plot.

**Note:** This can be a ratio.

Entries should be made in the exact form shown below i.e. Minor1 , Major,  Minor2 ,Minor1/Major etc. with no spaces.

**'Label'**

The text in this field will appear on the screen to label the scan at the right hand edge of the scan window.

**'y min', 'y max' and ' % '**

These 3 fields control the Y-axis scale. The entry fields **'y min'** and **'y max'** are used to enter specific values (using scientific notation). By entering a value in the **' % '** column, the software will automatically scale a ratio or beam intensity within the requested range, according to the intensities measured before starting the scan.

**'Colour' and 'Graph'**

The **'Colour'** entry fields determine the colour in which a line is drawn in the Scan Display Window. The colour is indicated by a number. The numbers are the same as used in screen messages, the main colours being:

| | | |
|---|---|---|
| -2: White | 4: Green | 10: Dark red |
| -1: Black | 5: Cyan | 11: Dark Pink |
| 0: Background | 6: Yellow | 12: Dark Green |
| 1: Blue | 7: Neutral | 13: Dark Cyan |
| 2: Red | 8: Dark Grey | 14: Brown |
| 3: Pink | 9: Dark blue | 15: Pale Grey |

The **'Graph'** entry fields allows a number of separate graphs to appear in the Scan Display Window at the same time. You may have up to 4 graphs (entered 0 - 3 in the entry field). If you are plotting beam intensities it may be best to show these all on the same graph (in which case use the same number), to illustrate beam coincidence, but, for example, a ratio is best shown on its own graph, because of the different Y scale.

The Scan Setup dialog box also has the following push buttons:

**'Delete Line'** deletes a selected line from the lower entry details (line details). The line is selected by selecting any of the entry fields on the line. A confirmation dialog box is accessed to safeguard against accidental deletion.

**'Load'** accesses **'Load Scan Parameter File'** list box, which enables you to select a scan parameter file to be loaded and has the following push buttons:
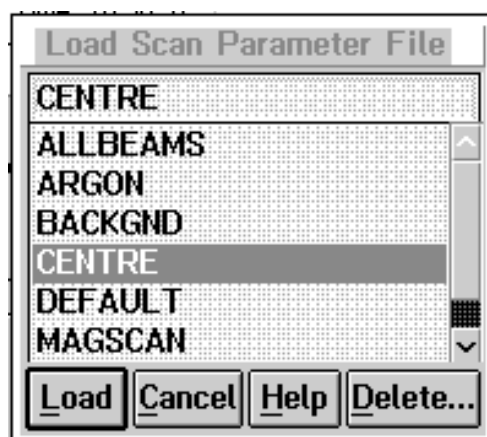


This dialog box has the following push buttons:

**'Load'** loads the **'Scan Setup'** dialog box for the file chosen from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the scan parameter file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**'Save'** saves the scan setup file in the existing Scan Parameter File (overwrites the file called up).

**'Save As'** selects the **'Save Scan Parameter File'** dialog box which enables new scan files to be saved and has the following push buttons:
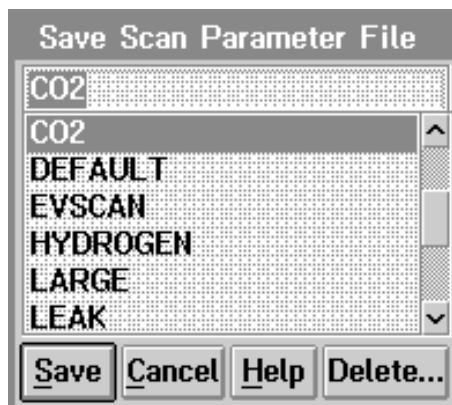


This dialog box has the following push buttons:

**'Save'** saves the scan setup file in a scan parameter file chosen from the list box or under a new name entered in the entry field.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the scan parameter file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'GO'** starts the scan using the parameters in the current scan setup file.

## Run Scan Param File

This menu option accesses **'Run Scan Parameter File'** list box.



The list box enables you to select a scan parameter file to be run and has the following push buttons:

**'Run'** runs the selected scan parameter file from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the scan parameter file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**Note:** For details of the Scan Window see below.

## Scan Display Window

When you start a scan, the 'Scan Display Window' appears in the middle of your screen. It can be re-sized and positioned to suit your own requirements. The window has its own Menu Bar and will show a graph for each plot you have set up in the 'Scan Setup' dialog box.
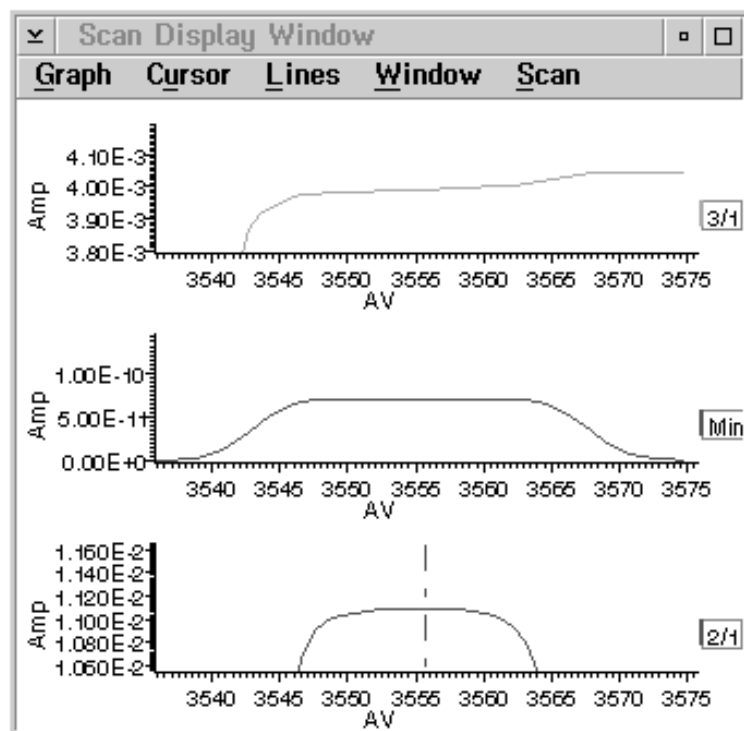
## Scan Display Window Menu Bar

The 'Scan Display Window' has the following options on its menu bar:

## Graph

| Graph | Cursor | Lines | Window | Scan |
|---|---|---|---|---|
| Print... | | | | |
| Print Setup... | | | | |
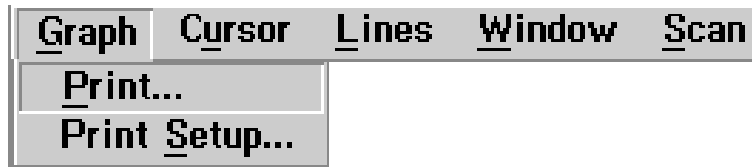
This menu option has the following commands:

**'Print'**

This menu option opens the **'Graph Print'** dialog box.

**Graph Print**

Printer : LQ-570 on LPT1

Title [                    ]

**Print Graphs**
- ○ **All**
- ◉ **Currently Visible**
- ○ **Selected**

[ **Ok** ]

[ **Cancel** ]

[ **Setup...** ]
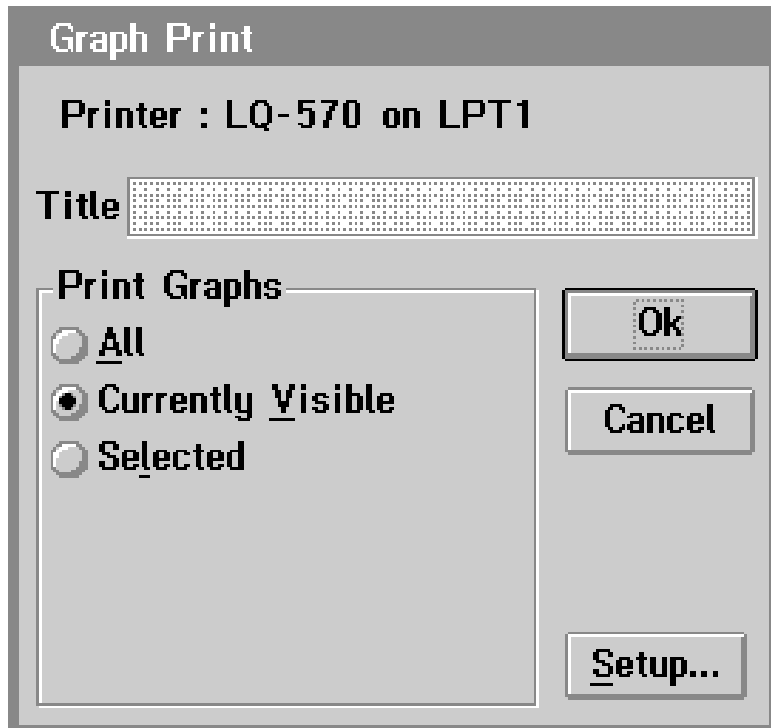
This dialog box allows you to give the print out a title in the **'Title'** entry field and to select the graphs to be printed using the radio buttons in the **'Print Graphs'** section. The radio buttons are:

**'All'** when selected prints all the graphs selected from scan.

**'Currently Visible'** when selected will print all the graphs in the scan window.

**'Selected'** when selected opens a list box from which graphs to be printed can be selected, by clicking on the required graph title.

The dailog box also has the following push buttons:

**'OK'** will then start the print out.

**'Cancel'** push button exits the dialog box without taking any actions.

**'Setup'** will open the **'Select Printer'** dialog box.

**'Print Setup'**

This menu option opens the **'Select Printer'** dialog box. This allows the type of printer to be printed to, to be chosen. A list of available printers appears in the list box
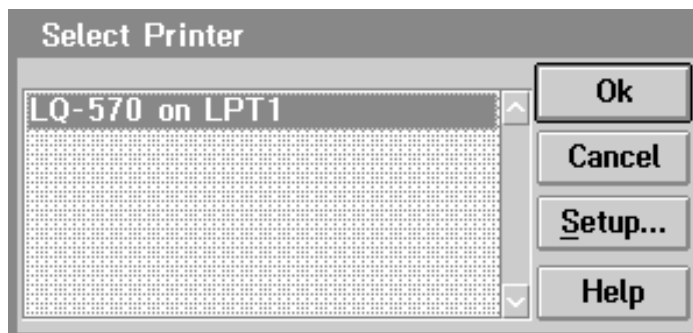


The dialog box has the following push buttons:

**'OK'** will specify the printer selected in the list box as the preferred printer until changed (by selecting a different printer).

**'Cancel'** push button exits the dialog box without taking any actions.

**'Setup'** will open the selected printers **'Job Properties'** dialog box, from which the print out details can be edited. For details of the 'Job Properties' dialog box see OS/2 Manual.
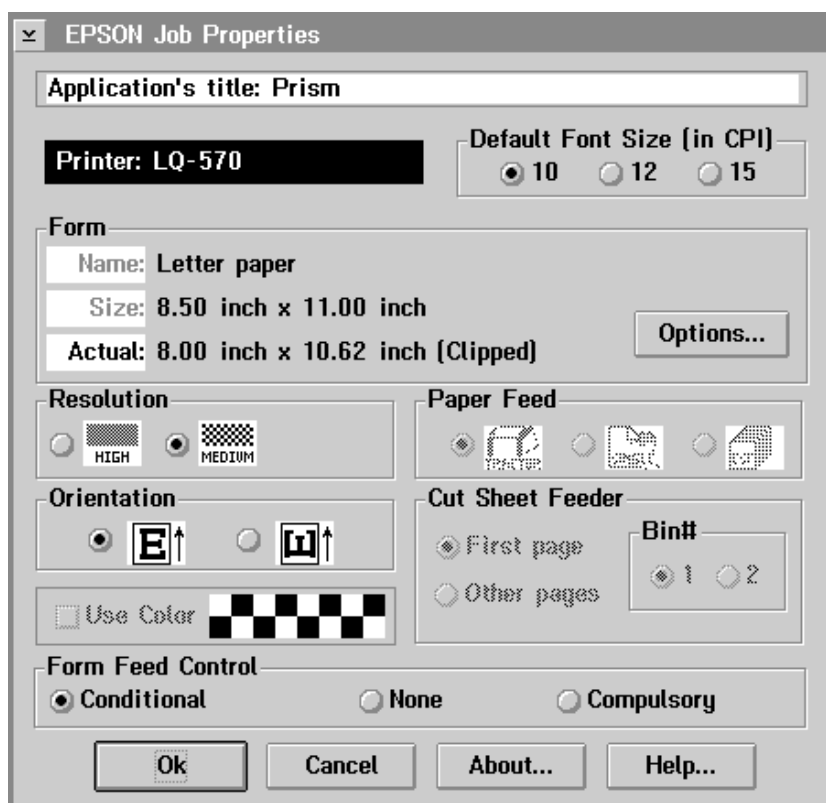


**'Help'** push button gives information on the operation.

## Cursor
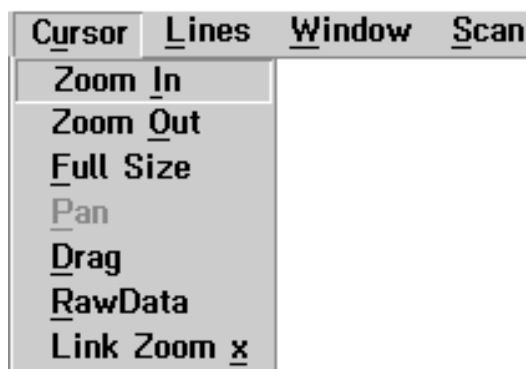
This menu controls the behaviour of the mouse pointer within the graphical display area. The commands all act with a 'Menu Tick' function and are:

**'Zoom In'**

When chosen the mouse cursor becomes a magnifying glass symbol. By dragging the magnifying glass over an area of the scan you can examine it in greater detail. If required you can 'Zoom In' in several stages.

**'Zoom Out'**

By clicking on a 'Zoom In' graph after selecting 'Zoom Out', the plot will go back to the size previously displayed prior to a 'Zoom In', this can be done several times until the graph is back to full scale use.

**'Full Size'**

This will return a graph to full scale, simply by clicking on the plot.

**'Pan'**

This is greyed out and has no function.

**'Drag'**

When Drag is selected, you can use the mouse to pick up and move the vertical cursor line used to show the current voltage. This is useful if a new analysis position is required on the flat top.

**'Raw Data'**

Raw Data mode provides an additional vertical cursor, which can be used in conjunction with the drag facility to examine the raw scan data in a small window at the bottom left of the screen. It should be noted that only those lines selected (see below for details) will show.

**'Link Zoom'**

When selected, the mouse symbol becomes 2 linked loops. Using this, you can link the x scale of two graphs by clicking on them in turn.

## Lines
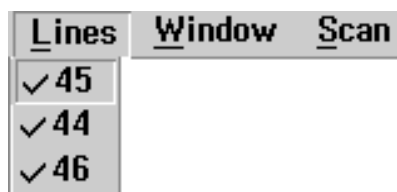
This menu contains a list of the lines available for display. The text in the menu is the label you have entered in the Scan Setup dialog box. By ticking lines on or off from this menu you can simplify the display window.

## Window
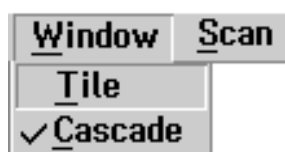
The commands all act with a 'Menu Tick' function and are:

**'Cascade'**

This default setting will arrange multiple graphs one above the other in the display window (i.e. one column).

**'Tile'**

This option will arrange graphs side by side , as well as above each other. (i.e. two or more columns).

## Scan

The commands are:

**'Centre'**

This command automatically centres the voltage cursor (peak centre).

**'Quality'**

This command performs a peak quality check and sends the results to the printer, (see section on peak quality).

**'Repeat'**

This repeats the scan once, if auto repeat has not been set.

**'Halt'**

This command halts a scan while in progress.

# Tests

This menu contains a set of commands used to check the status of the instrument, as well as being required for day to day running.

| | |
|---|---|
| **_P_eak Quality** | **Ctrl+Q** |
| **_I_dentify Peak...** | **Ctrl+I** |
| **Mass Calibration...** | |
| **_P_eak Centre** | **Ctrl+C** |

The menu options are :

## Identify Peak

This opens **'Identify Axial Peak'** dialog box which allows you to identify the mass in the axial collector (i.e. the mass presently in the axial collector or the mass your going to move to the axial collector).

This is particularly important as this sets the 'beam mask', which selects the collector array into which the beams are measured (see section on the PRISM collector array).

Examples of the 'Beam Mask' are given below:

If mass 45 is selected (measuring $CO_2$) then the following mask is used;

| | | |
|---|---|---|
| Beam 1 | (mass 44) | Low 1 Collector |
| Beam 2 | (mass 45) | Axial Collector |
| Beam 3 | (mass 46) | High Collector |

If mass 29 is selected (measuring $N_2$) then the following mask is used;

| | | |
|---|---|---|
| Beam 1 | (mass 28) | Low 2 Collector |
| Beam 2 | (mass 29) | Axial Collector |
| Beam 3 | (mass 30) | High Collector |

If mass 2 is selected (measuring Hydrogen) then the following mask is used;

| | | |
|---|---|---|
| Beam 1 | (mass 2) | Hydrogen 2 Collector |
| Beam 2 | (mass 3) | Hydrogen 3 Collector |

Please see collectors section in Equipment Description chapter of this manual for details of beam assignments for other gases.

**Identify Axial Peak**

Current Axial Mass  45.0

New Mass Identity  45.0

| OK | Cancel | _H_elp |
|---|---|---|
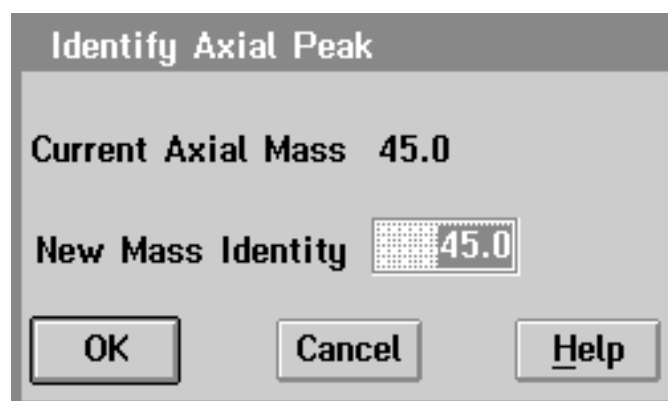
The **'Identify Axial Peak'** dialog box displays the **'Current Axial Mass'** (also shown in the monitor window) which is the mass, the mass spectrometer accepts as the axial mass. However, if the magnet current or accelerating voltage has been moved by the user then a new axial mass must be identified in the **'New Mass Identity'** entry field.

**Note:** Always identify the current axial mass when changing gas species.

The dailog box has the following push buttons:

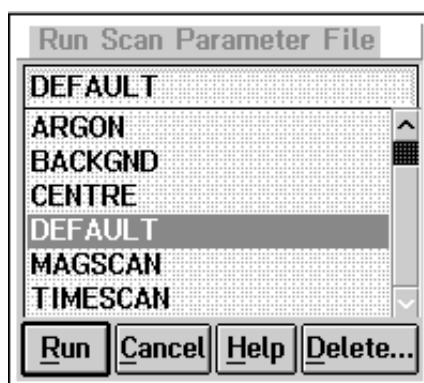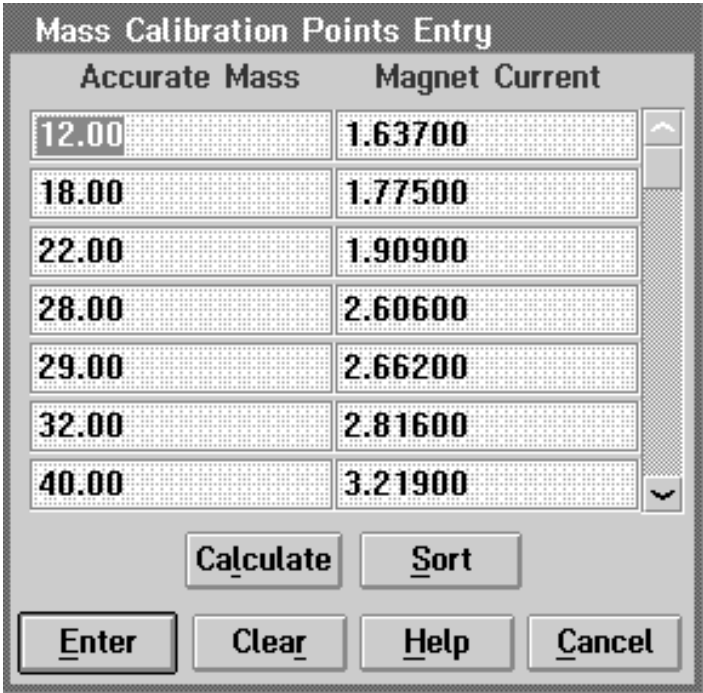**'OK'** enters the **'New Mass Identity'** given in the entry field and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Mass Calibration

This menu option opens the **' Mass Calibration Points Entry'** dialog box, which enables the mass spectrometer to be mass calibrated (i.e. the values obtained for mass value against magnet current are inputted into the computer in this dailog box).

| Mass Calibration Points Entry | |
|---|---|
| **Accurate Mass** | **Magnet Current** |
| 12.00 | 1.63700 |
| 18.00 | 1.77500 |
| 22.00 | 1.90900 |
| 28.00 | 2.60600 |
| 29.00 | 2.66200 |
| 32.00 | 2.81600 |
| 40.00 | 3.21900 |

Calculate   Sort

Enter   Clear   Help   Cancel

The dialog box contains a list box with 2 entry fields on each line. The entry fields expect the **'Accurate Mass'** and the **'Magnet Current'** at which the mass is found.

**Note:** The list box has no limit to the number of peaks and the greater the number of peaks inputted the more accurate the mass calibration will be.

The dialog box also has the following push buttons:

**'Clear'** removes all the entries from the list box (i.e. all the mass and magnet current data).

**'Calculate'** performs a line fit on the data points entered in the list box, which will be used to fill in the gaps in the data.

**'Sort'** puts the entries in the list box above in mass order, with the lowest mass number at the top.

**'Enter'** saves the data to disc and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Peak Centre

This menu option will automatically set the accelerating voltage to focus (centre) the mass in the axial collector. This is accomplished using the scan procedure file 'Centre' to scan the axial collector. If a peak is found within the scan range then the accelerating voltage is set to the peaks central value (marked with a broken vertical line). The peak shape is displayed, and may be printed out if desired, the window will stay open until closed by the operator.
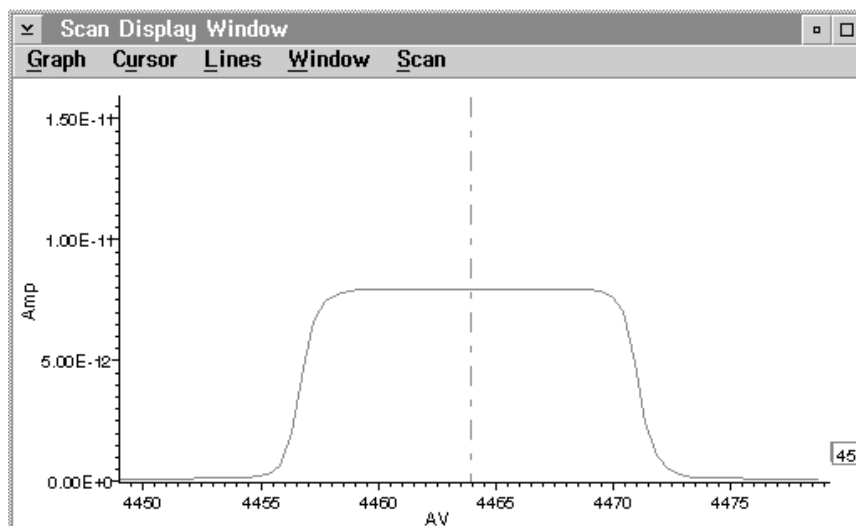


Example of a Peak Centre Window for $CO_2$ (mass 45)
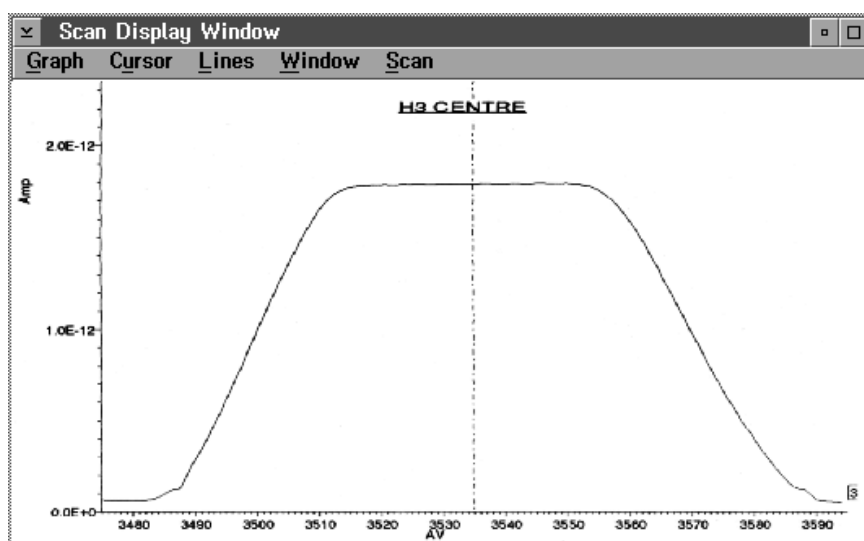


Example of a Peak Centre Window for Hydrogen (mass 3)

If the Hydrogen peak centre is required, then the scan procedure 'CentreH3' is used instead of 'Centre'. This is handled by the software once Hydrogen has been selected, by identifying the current mass as 2.

## Peak Quality
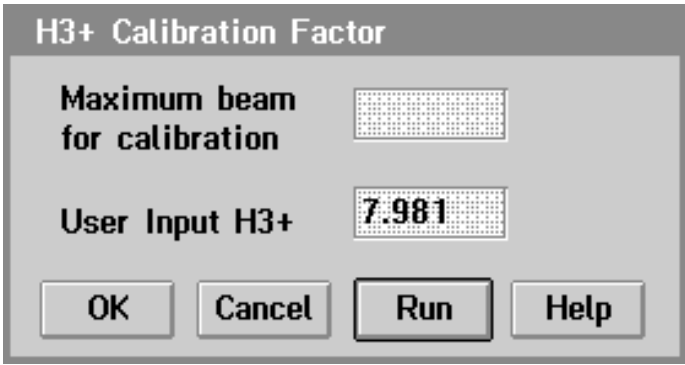
This menu option provides a quantitative measurement of the quality of your peak shapes, in terms of flatness, symmetry and resolution. When the **'Peak Quality'** command is chosen a peak centre is carried out (as above) and the resolution, symmetry, and peak top flatness are measured of any peak found within the range of the centre scan, these are then outputted to the printer.

**Note:** If the peak quality of all the collectors are required then the other beams need to be added to the centre scan procedure. However they should be returned to the original settings, as the changed files may effect the accuracy of the instrument to 'Peak Centre'.

## H3+ Calibration

This menu option is only applicable to instruments fitted with the Hydrogen option and is used to calibrate the contribution of the H3+ ion to the mass 3 ion current before performing H/D analyses. For further details please see the section on the Hydrogen option.

When selected this option opens the **'H3+ Calibration Factor'** dialog box.



The current value of the H3+ correction factor (previously measured value) is stored on disk in the parameters file 'Constant Parameters', and is displayed in the **'User Input H3+'** entry field. This value may be updated either by inputting a new value directly, or by performing a new calibration.

The pressure range that the calibration is performed over is user selectable. This is done by inputting the maximum ion current (in Amps) required in the entry field **'Maximum beam for calibration'**.

**Note:** A 5 point calibration is performed at currents equal to 100%, 80%, 60%, 40%, and 20% of this value. It is therefore important that this maximum value should be at least 5 times bigger than the beam height in the reference half of the inlet, when the bellows are fully open (ideally load Hydrogen into the reference bellows to give a signal of 2E-9 Amps with the bellows fully open). During the calibration the message window will inform on the progress of the calibration.

The dialog box also has the following push buttons:

**'OK'** saves the **'User Input H3+'** value if a new value is entered and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Run'** starts the H3+ calibration using the value entered in the **'Maximum beam for calibration'** entry field as the target ion beam.

**'Help'** gives information on the operation.

## Amplifier Zero

This menu option measures the background signal at the detectors with no gas flowing into the mass spectrometer from the dual inlet. After isolating the source from the inlet (resets the changeover valve), the routine waits for 20 seconds, and then measures the residual signal of all the amplifiers. The values are output to the printer, and are stored on disk in the parameters file 'Constant Parameters' for subtraction from subsequent analyses.

**Notes:**

a) It is essential to perform an amplifier zero before performing an H3+ Calibration or hydrogen analysis.

b) The beams displayed in the monitor window are not zero corrected.

## Transducer Zero

This menu option when selected sets the Transducer reading to zero. It should therefore only be selected when the sample side is at vacuum level (i.e. no sample in the sample port). The transducer reading is used to set the sample gas handling procedure, for the sample into the inlet. It should therefore be selected regularly as Transducers tend to drift, especially when the temperature changes.

## Analysis

This menu contains the necessary commands to run samples on the PRISM mass spectrometer with a Dual Inlet and associated preparation systems (for details of the preparation systems see their manuals).

| Analysis | User | Program | Config |
|----------|------|---------|--------|
| Run... | | | Ctrl+R |
| DAPC Edit... | | | Ctrl+D |
| Autorun Start/Stop... | | | Ctrl+A |
| Autorun Batch Edit... | | | |
| Parameter File Edit... | | | |

The menu options are :

## Run

This menu option opens the **'Select DAPC File to Run'** dialog box, which is used to initiate a single manual analysis (single run).

The dailog box has a list box from which the DAPC file to be run can be selected (it gives a list of all available DAPC files) and an entry field into which the sample name is entered (this is used on the analysis printout to identify the sample). The dialog box also has the following push buttons:

**'Run'** starts the analysis using the DAPC file selected from the **'File'** list box with the identifier entered in the **'Sample Name'** entry field

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**Delete'** allows the user to delete the DAPC File chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

## DAPC Edit

This menu option accesses the **'Data Acquisition Control Parameters'** (DAPC) dialog box, which enables existing data acquisition parameters files to be edited or new files to be created. These data acquisition control parameters (DAPC) decide how a particular sample is run (for example $CO_2$ and $N_2$ samples are run using different parameters), therefore this dialog box is used to set the parameters that control the way an isotopic composition is measured.

**Note:** This menu option loads the DAPC file accessed previously, however if this is the first time this menu option has been accessed, then load an existing file using the **'Load'** push button or create a new file.

The example below of the Data Acquisition Control Parameters dialog box is for $CO_2$ and will use this as an example in this section of the manual (meaning any specifics relate to $CO_2$).



The fields are:

**'Gas File'** combo box specifies the name of the gas species associated with the DAPC file. Therefore to create a new file, type in the name and select the **'Edit'** push button.

**Note:** For details of the **'Edit'** push button see Reference Gas Details section below.

**'Masses to Collect'** list box details the peaks for which data is to be collected. For example, in the case of $CO_2$ this should be 44, 45 and 46. The masses are added into this list in the **'Add Mass'** entry field. The masses are deleted by selecting the mass and pressing the <DEL> key.

**'Add Mass'** entry field enables mass numbers to be added to the **'Masses to Collect'** list. After entering the mass number in the entry field, then enter this into the list using the TAB key.

**'No. of Comparisons'** entry field allows the number of data blocks to be collected during the analysis to be entered.

**'Changeover Delay'** entry field allows the time in seconds between the toggling of changeover valve and the start of an integration to be entered (in seconds).

**'Integration time'** entry field allows the length of the data block (i.e. counting time) to be entered (in seconds).

**'Worst Acceptable Precision'** entry field allows the worst analytical error (internal precision) to be entered (in per mil). If the error is greater than this value, the run is repeated once.

**'Ion Gauge Diff Trigger'** entry field allows the pressure differential (in %) between the sample gas and the reference gas measured on the ion gauge to be entered. If the pressure differential is greater than this value a background scan is triggered on the sample. The background scan is specified in the **'Scan from Mass'** **'to'** entry fields.

******* **Note:** This menu option is not yet implemented in the software. *******

**'Scan from Mass'** **'to'** entry fields allow the start and end masses of the background scan to be specified.

******* **Note:** This menu option is not yet implemented in the software. *******

**'Background Peaks'** list box details a list of additional peaks to measure, after each sample. This is carried out on both the sample and reference gas and is used for checking gas purity by peak jumping to other gas peaks e.g. mass 18 (water) and 40 argon) used to check for leaks. The masses are added into this list in the **'Add Peak'** entry field and this function is enabled in the **'Peak Jump Enable'** check box.

**Note:** Remember a valid mass calibration must be carried out prior to using this feature.

**'Add Peak'** entry field enables mass numbers to be added to the **'Background Peaks'** list. After entering the mass number in the entry field, then enter this into the list using the TAB key.

**'Plot range'** entry field sets the value (in per mil) of the y axis scale range in the ratio traces shown during data acquisition. For example an entry of 5.0 gives a y scale of ± 5‰ .

**'Peak Jump Enabled'** check box enables the measurement of background peaks specified above in the **'Backgrounds Peaks'** list box.

**'Amp Zero Enable'** check box allows the user to decide whether the amplifier zeros are removed from the data. This has a default of **ON** should be left as this when running samples.

**'Error Rejection'** check box allows error rejection of raw data to be enabled or disabled. This is normally enabled and rejects deltas that are greater than 2 standard deviations outside the mean.

**'Sample Offset'** radio button section allows for the sample data to be plotted in the same graph window as the reference data, for this to happen an offset is calculated. This can set the data plots to be either a full real delta value or an integer only, by use of the radio buttons **'Real'** and **'Integer'**.

**'DataLogger'** section please see section on the DataLogger below.

The Data Acquisition Control Parameters dialog box has the following push buttons:

**'Load...'** opens the **'Load DAPC Parameters'** dialog box which contains a list box, from which previously saved parameter files can be loaded, which is useful for checking or editing prior to running.
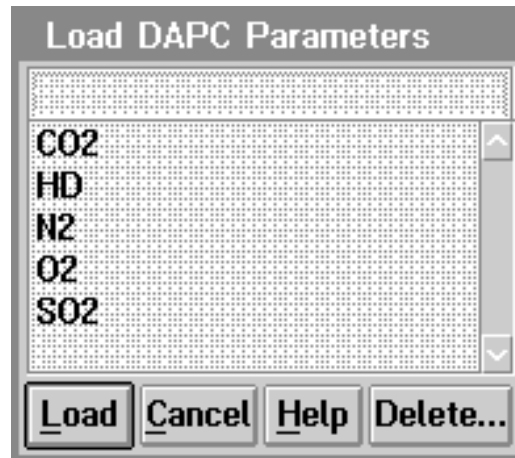


The Load DAPC Parameters dialog box has the following push buttons:

**'Load'** loads a DAPC Parameters for the file chosen from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the DAPC Parameters file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**'Save...'** opens the **'Save DAPC Parameters'** dialog box, so when a new parameter file is created it can be saved to a file.
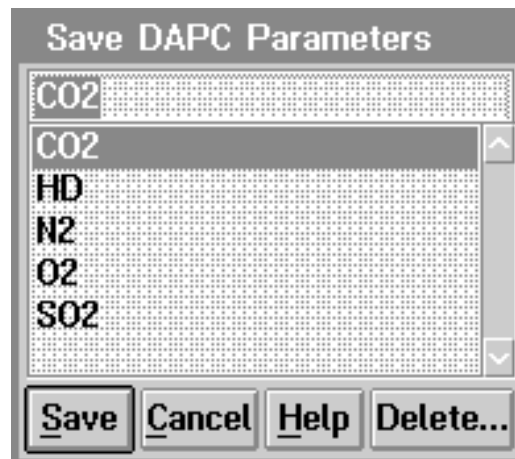


The Save DAPC Parameters dialog box has the following push buttons:

**'Save'** saves the DAPC Parameters in an existing DAPC Parameters file chosen from the list box or under a new name entered in the entry field.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Delete'** allows the user to delete the DAPC Parameters file chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

'Run...' opens the **'Select DAPC File to Run'** dialog box, which is used to initiate a single manual analysis (single run).
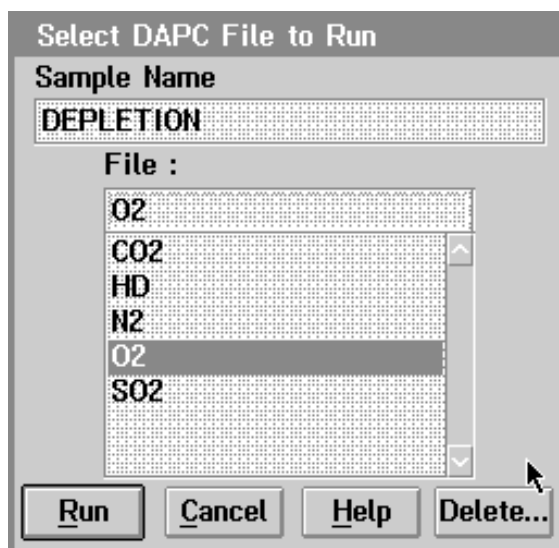


The dialog box has a list box from which the DAPC file to be run can be selected (it gives a list of all available DAPC files) and an entry field into which the sample name is entered (this is used on the analysis printout to identify the sample).

The dialog box also has the following push buttons:

**'Run'** starts the analysis using the DAPC file selected from the **'File'** list box with the identifier entered in the **'Sample Name'** entry field

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**Delete'** allows the user to delete the DAPC File chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**'Clear'** removes all the data from the dialog box on the screen.

**Note:** The data is not removed from the DAPC file only from the screen.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**There are 2 other sections of the Data Acquisition Control Parameters dialog box details of which are given below:**

## Reference Gas Details ('Edit' push button)

Each DAPC (Data Acquisition Parameters File) has associated with it a Reference Gas Details file. This file is edited from the DAPC dialog box by clicking on the **'Edit'** push button at the side of the **'Gas Species'** combo box. This opens the **'Reference Gas Details'** dialog box, which is used to enter the delta values for your reference gas.



This 'Comment' entry field allows the user to input a comment in the gas details dialog box (for example the reference gas name). The 'Gas Species' combo box allows the type of gas used as a reference (e.g. $CO_2$) to be selected (if the gas required is not in the list then select 'OTHER' and create your own).

The 'Reference Values' section of the dialog box has entry fields which allow the delta value(s) of the specified gas to be entered. The type and name of the delta value(s) is taken from the 'Reference Gas Constants' dialog box (see below) and will depend on the gas species. Therefore if the reference gas delta(s) has changed, input the new delta value(s).

The Reference Gas Composition Edit dialog box has the following push buttons:

**'Constants...'** opens the **'Reference Gas Constants'** dialog box details of which are given below.

**'Corrections...'** opens the **'Reference Gas Corrections'** dialog box details of which are given below.

**'Save'** saves the Reference Gas Details in the specified DAPC file and closes the 'Reference Gas Details' dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Reference Gas Constants

Each Reference Gas Details file has associated with it a Reference Gas Constants file. This file is edited from the Reference Gas Details dialog box by clicking on the **'Constants'** push button. This opens the **'Reference Gas Constants'** dialog box.

This box varies depending upon the gas species selected from the 'Reference Gas Details' dialog box, the example below is for $CO_2$ :



**Note:** If other gases are selected then the dialog box is similar, however only the features relating to that gas are valid (the other functions are greyed out).

The dialog box has the following features:

**'No Of Deltas'** entry field for the gas species to be entered, (for example for $CO_2$ this would be 2, but for non enriched $N_2$ this would be 1).

**'Delta Details'** section enables the name(s) for the delta value(s) to be entered in the **'Text'** entry field(s) and the number of decimal places to be entered in the **'No Decimals'** entry field(s). These entries are used in the 'Reference Gas Details' 'Reference Values' section to specify the delta(s) name(s) and the number of decimal places allowed, they also used on the report printed after the data run.

**'Craig Constants'** section allows the Craig constants for the specific gas to be specified in the four entry fields (please refer to the Isotope Ratio Mass Spectrometers chapter of this manual for details (Chapter 3)). The table for the various gases is given below, however any values can be entered. The constants allowed are **'C1'** , **'C2'** , **'C3'** and **'C4'**.

|      | $CO_2$ | CO | $SO_2$ | $N_2$ | HD |
|------|--------|--------|--------|-----|-----|
| C1   | 1.0676 | 1.0378 | 1.09   | 1   | 1   |
| C2   | 0.0338 | 0.0169 | 0      | 0   | 0   |
| C3   | 1.0010 | 1.0010 | 0      | 1   | 0   |
| C4   | 0.0021 | 0.0021 | 0      | 0   | 0   |

**'Atom Percent'** section allows atom percent reference ratios to be input into the entry fields (please refer to the Isotope Ratio Mass Spectrometers chapter of this manual for details (Chapter 3)). The number of entry fields will vary with the gas species, the example above being for $CO_2$ which requires three entry fields. The reference ratios allowed are **'R1'** , **'R2'** and **'R3'**.

**'With Respect To'** entry field allows the correction factor label to be input. This is used in the print out from the data analysis.

**'CO2 Delta To Use'** section allows the user to select whether the delta values are to be either of the type $\delta 45$ or $\delta 13$ by selection of the appropriate radio button. This section is greyed out for any gas species other than for $CO_2$ .

**'Santrock & Hayes'** section contains the Santrock & Hayes constants which are user definable if required (please refer to the Isotope Ratio Mass Spectrometers chapter of this manual for details (Chapter 3)). The constants allowed are **'Alpha'** , **'k'** , **'Ratio 13'** and **'Ratio 18'**.

**'Delta Temperature Coefficients'** section allows the user to input the various information required when correcting the data for temperature (for example Carbonate Sample preparation system or Isoprep 18 preparation system). The section also allows the user to select which delta ($\delta$) value the correction should **'Apply To'** by selection of the appropriate radio button. The constants and coefficients allowed are **'Equilibrium Constant'** , **'Temp Coefficient'** , **'Reaction Temp'** and **'Standard Temp'**. (Please refer to the preparation system manuals for details).

**'SMOW Equilibration Constant'** section allows the constant **'E3'** to be altered if required (please refer to the Isotope Ratio Mass Spectrometers chapter of this manual for details (Chapter 3)). This is used if the oxygen isotopes are to be corrected relative to SMOW.

**'Ok'** push button saves the new data and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Reference Gas Corrections

Each Reference Gas Details file has associated with it a Reference Gas Corrections file. This file is edited from the Reference Gas Details dialog box by clicking on the **'Constants'** push button. This opens the **'Reference Gas Corrections'** dialog box.

This box varies depending upon the gas species selected from the 'Reference Gas Details' dialog box, the example below is for $CO_2$ :
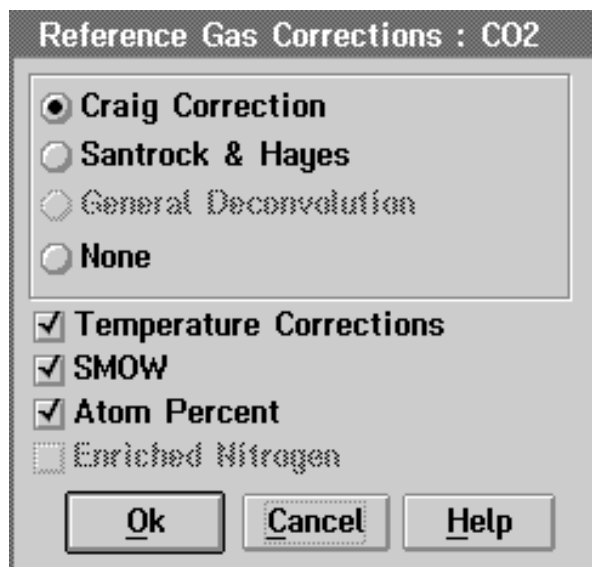


**Note:** If other gases are selected then the dialog box is similar, however only the features relating to that gas are valid (the other functions are greyed out).

The dialog box has the following radio buttons:

**'Craig Correction'** when selected applies the Craig Correction method to the data.

**'Santrock & Hayes'** when selected applies the Santrock & Hayes Correction method to the data.

**'General Deconvolution'** when selected applies the Craig Correction method to the data for any gas other than for $CO_2$ .

**'None'** when selected applies no corrections to the data.

The dialog box has the following check boxes:

**'Temperature Corrections'** when selected applies the temperature corrections to the data.

**'SMOW'** when selected corrects the data with respect to SMOW, as well as using the corrections from the constants file.

**'Atom Percent'** when selected prints out the atom percent and atom percent excess data using the constants from the constants file.

**'Enriched Nitrogen'** can only be selected if the 'Atom Percent' is selected and the gas is Nitrogen. When selected will measure the mass 30 and give the δ30 ratio.

The dialog box has the following push buttons:

**'Ok'** push button saves the corrections required and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Datalogger

The Datalogger is a utility which enables you to archive data to disc whilst running samples, in a format which is easily transferable to spreadsheets, etc. The type of data to be saved during sample running is selected from a 'Template' which can be edited and saved. The data is saved in either of two file formats 'Overwrite' or 'Append':

a)  The 'Overwrite' file format as its name suggests saves the data of the sample currently running over the sample data from the previous run. This keeps the file small, but is of little use when running autoruns where the samples need to be compared with each other.

b)  The 'Append' file format saves the sample data in chronological order, so that all the data from a autorun can be saved. This format is therefore more useful when samples are to be compared.

The data can also be saved to more than one file (up to 10 files) which is useful if data is required for a individual autorun, as well as to keep an on going check on the instrument performance.

The data logging software is enabled by the Datalogger 'Enable' check box in the Data Acquisition Control Parameters dialog box.

**Note:** If this is disabled then the other Datalogger functions are grey and cannot be accessed.

The 'Template' combo box is used to select template (the type of data requiring to be saved) to be used. Selecting the 'Edit' push button opens the 'Edit Template' dialog box, which enables the selected template to be checked and / or edited.
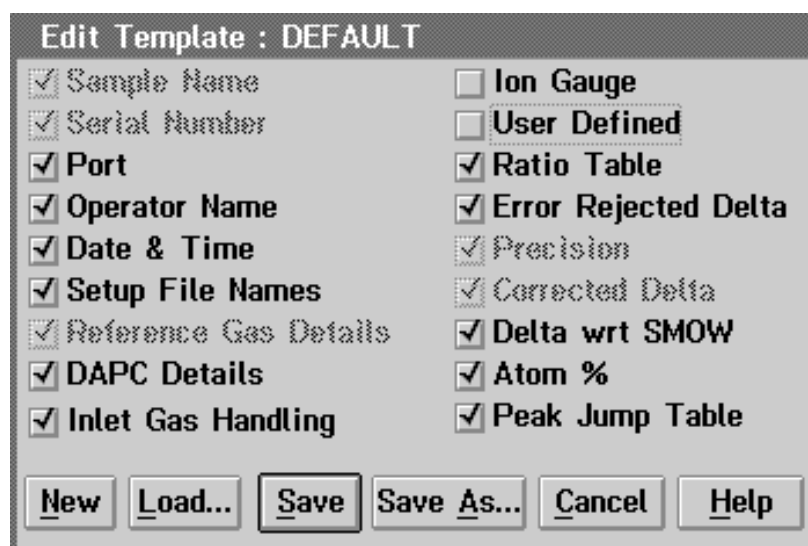


This dialog box shows check boxes next to the variables which can be saved on file. The variables shown in grey are always saved. i.e. Sample Name, Serial Number, Reference Gas Details, Precision and Corrected Delta. By ticking the appropriate check boxes, you can tailor the saved data to your own needs.

The dialog box also has the following push buttons:

**'New'** clears the check boxes so that a new template can be created.

**'Load'** opens the **'Load Template'** dialog box, which allows a template previously saved to be loaded.
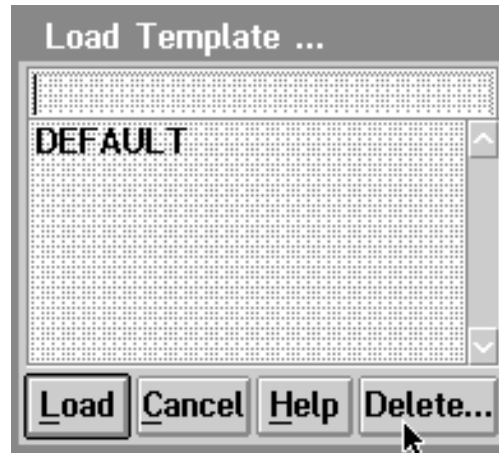


This dialog box contains a list box from which the file to be loaded can be chosen. When the file name has been selected, the details are loaded with the **'Load'** push button or alternatively the file can be deleted with the **'Delete'** push button (as usual with 'Delete' a confirmation dialog box is opened). The **'Cancel'** push button exits the dialog box without taking any actions and the **'Help'** push button gives information on the dialog box.

**'Save'** saves the template in the file already created.

**'Save As'** opens the **'Save Template As'** dialog box, which saves the template to a new file.
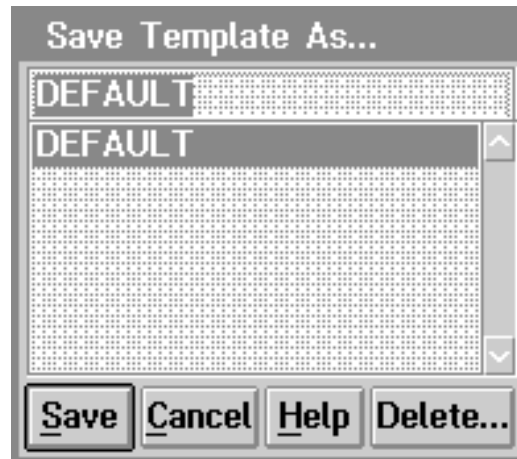


This dialog box contains a list box from which a file name can be selected to save to and a entry field to allow for a new file name to be created. When the file name has been selected, the template is saved with the **'Save'** push button or alternatively the file can be deleted with the **'Delete'** push button (as usual with 'Delete' a confirmation dialog box is opened). The **'Cancel'** push button exits the dialog box without taking any actions and the **'Help'** push button gives information on the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

The output file(s) to be saved to are selected in the **'DataLogger Output Files'** section of the Data Acquisition Control Parameters dialog box. These files may be of **'Overwrite'** or **'Append'** type as indicated by the prefix **'A >'** or **'O >'** in the output files list box and any file in this list box has the data stored to, if the datalogger is enabled.

To add a file to the list, click on either the **'Add Append'** or the **'Add Overwrite'** push buttons depending which style of file is required. In either case this opens the **'DataLogger Append File Selection'** dialog box.
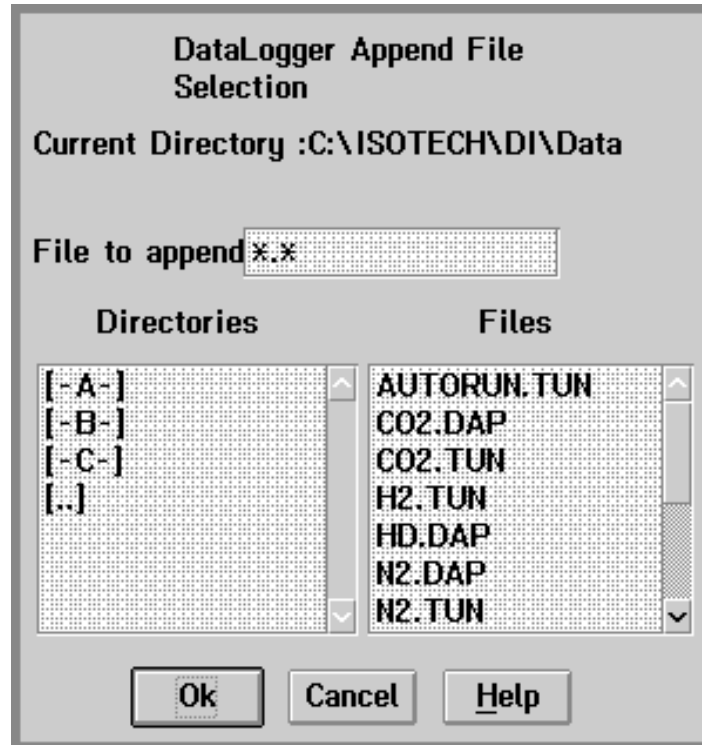


A new file name can now be entered in the **'File to append'** entry field or a file name can be selected from the **'Files'** list box. The file is then added to the **'DataLogger Output Files'** with the **'OK'** push button. The **'Cancel'** push button exits the dialog box without taking any actions and the **'Help'** push button gives information on the dialog box.

**Note:** It is essential the file name has an file name extension, and it is advised to use **'.TXT'** .

To remove a file from the **'DataLogger Output Files'** list, highlight it, and click on the **'Remove'** button.

**Note:** The file is not deleted from the hard disc by this action.

## Autorun Start/Stop

If an autorun is not presently running then this menu option opens the **'Select Autorun File'** dialog box, which is used to initiate an autorun analysis (a sample set from one of the preparation systems - for details see the section on preparation systems). If an autorun is running then this menu option will stop the autorun, after finishing the sample it is running.
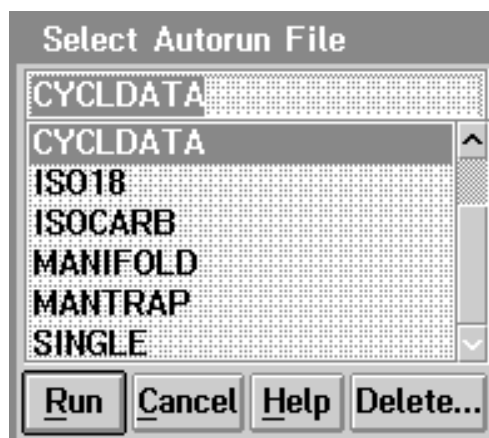


The dailog box has a list box from which the Autorun file can be selected (it gives a list of all available Autorun files).

The dialog box also has the following push buttons:

**'Run'** starts the analysis using the Autorun file selected from the list box.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**Delete'** allows the user to delete the Autorun File chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

## Autorun Batch Edit

This menu option accesses the **'Load Autorun Setup File'** dialog box, which allows the **'Autorun Batch'** files to be edited.

## Batch Files

In order to run a set of samples from a preparation system automatically, you need to provide the system with some basic information about them. This information is put in a **BATCH FILE**.

For each sample, the batch file contains:

**A sample name** This is an alphanumeric identifier for your sample, used for output and stored data.

**Position on preparation system** Either e.g. manifold port "A1" or a sample carousel position.

**A data acquisition file** This determines the integration procedures etc. used while measuring the isotopic composition of your sample.

When you set up a batch file, you associate it with a particular preparation system on which you have attached the samples. There is a file for each possible sample preparation device.

| MANIFOLD | - | Manifold (with or without crackers) |
| MANTRAP | - | Manifold and cryogenic traps |
| BAS50 | - | Breath carousel |
| CETRAP | - | Elemental analyser/cryogenic traps |
| ISO18 | - | Isoprep 18 water equilibration device |
| ISOCARB | - | Carbonate preparation device |
| SINGLE | - | Single sample run from inlet port |
| CYCLDATA | - | Repeats the same sample |
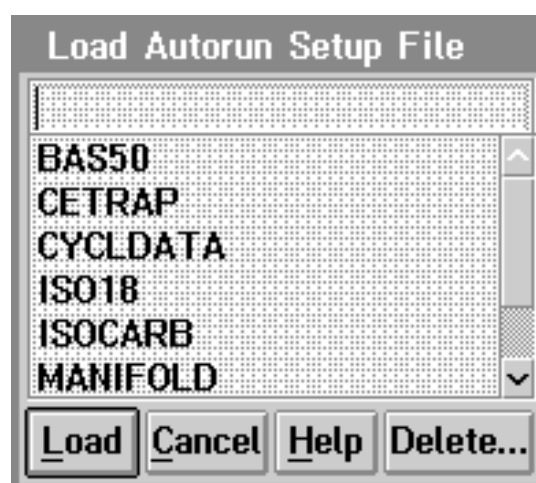


This dialog box contains a list box from which a sample preparation batch file can be selected.

It also has the following push buttons:

**'Load'** opens the **'Autorun Parameters Edit'** dialog box.

**AutoRun Parameters Edit**

Autorun File Name : MANIFOLD

Select A Prep System

| ISO18 |
| ISOCARB |
| **MANIFOLD** |
| MANTRAP |
| SINGLE |

| <u>O</u>k | <u>S</u>etup... | <u>C</u>ancel | <u>H</u>elp |
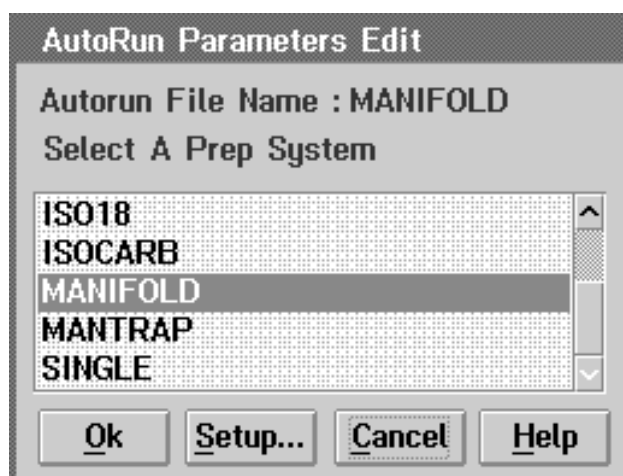
This dialog box contains a list box which indicates the file to be edited and the following push buttons:

**'OK'** saves the autorun parameter file, after it has been edited and exits the Autorun Batch Edit menu option.

**'Setup'** opens the **'Autorun : Sample Details'** dialog box.

**AutoRun : Sample Details**

| | Sample Name | Position | DAPC File | |
|----|-------------|----------|-----------|---|
| 1 | Sample_1 | A1 | CO2 | ⅀ |
| 2 | Sample_2 | A2 | CO2 | ⅀ |
| 3 | Sample_3 | A3 | CO2 | ⅀ |
| 4 | Sample_4 | A4 | CO2 | ⅀ |
| 5 | Sample_5 | A5 | CO2 | ⅀ |
| 6 | Sample_6 | A6 | CO2 | ⅀ |
| 7 | Sample_7 | A7 | CO2 | ⅀ |
| 8 | Sample_8 | A8 | CO2 | ⅀ |
| 9 | Sample_9 | A9 | CO2 | ⅀ |
| 10 | Sample_10 | A0 | CO2 | ⅀ |

| <u>D</u>elete | Delete <u>A</u>ll |
| <u>O</u>k | <u>R</u>elease | Release <u>A</u>ll | <u>I</u>nsert | <u>H</u>elp |
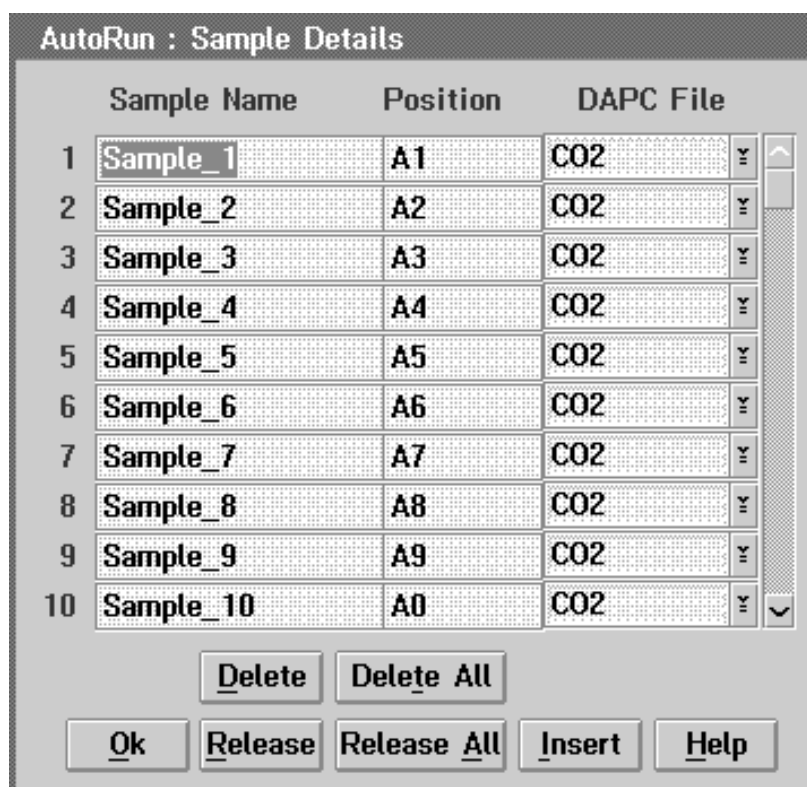
This contains 1 line per sample, with three fields in each line. The first column is used to enter a **'Sample Name'**. The second column is used to enter the sample's **'Position'** on the preparation device. (This will be either a valve mnemonic e.g. A2 for a port on the manifold, <u>or</u> a number to indicate the position on a carbonate or elemental analyser carousel). The third column is the gas species to be run which corresponds to the **'DAP File'**. There are 2 short cuts to entering the **'DAP File'** name in this combo box

(i) typing the initial letter (i.e. C for $CO_2$ , N for $N_2$ , etc.).

(ii) Click on the arrow to the right of the box - this will display a list box of the DAP Files available from which can be selected the required one using the mouse.

A normal text cursor shows which field you are in and information can be entered by typing in the normal way. To complete the entry in each box and TO MOVE BETWEEN FIELDS USE THE **TAB** KEY, not return.

When a line of sample details has been successfully entered, you will hear a **'Beep'**. There is display room for up to 10 samples. Further space will automatically appear for subsequent samples if the batch file is longer. There is a scroll bar at the right for moving through a long file.

The Autorun : Sample Details dialog box also has the following push buttons:

'OK' saves the edited Sample Details and exits the dialog box.

**'Release'** makes available the sample at the present cursor position.

**Note:** If a sample has already been analysed, its entry is changed from black to grey. Before it can be deleted or edited, it should be made available by using the **'Release'** command.

**'Release All'** makes available all the samples that have already been run.

**'Insert'** allows for an additional sample to be added at the current cursor position.

**'Delete'** removes the sample details at the current cursor position.

**'Delete All'** removes all the sample details in the file.

**'Help'** gives information on the operation.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

Delete' allows the user to delete the Autorun Setup File chosen from the list box. A dialog box is opened to confirm you wish to delete the file (**'Yes'** or **'No'** push buttons).

**Note:** It is very unwise to delete any of the files in the Autorun Setup File as they may be required if the system is up-graded at a later date.

## Parameter File Edit

Parameter files are used to control the way your samples are processed during an autorun and to store the various instrument constants. There is a file for the Dual Inlet, and one for each sample preparation mode, as well as files for the instrument constants.

**Note:** For details of the parameters see later sections of this manual.

This menu option opens the **'Edit Parameter Files'** dialog box, which allows these parameters to be edited.
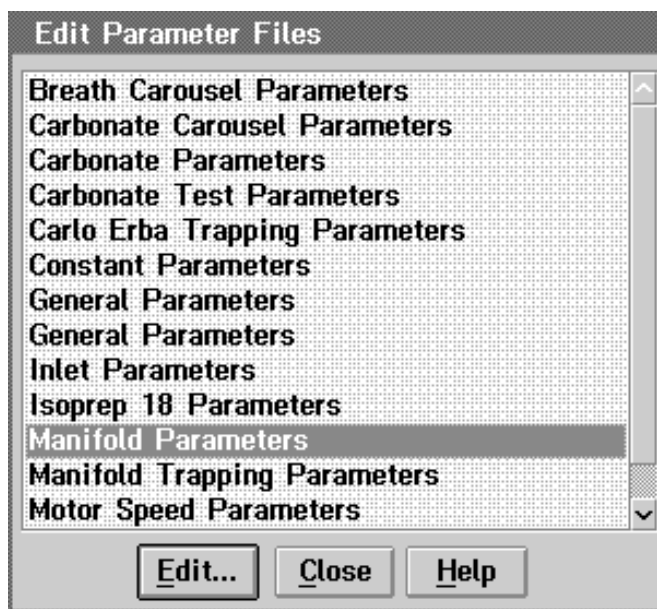


This dialog box contains a list box containing all the parameter files available to the user, from which a file to be edited can be chosen. The dialog box also has the following push buttons:

**'Edit'** opens the **'Edit Parameters'** dialog box, which allows you to edit any of the parameters within that file.
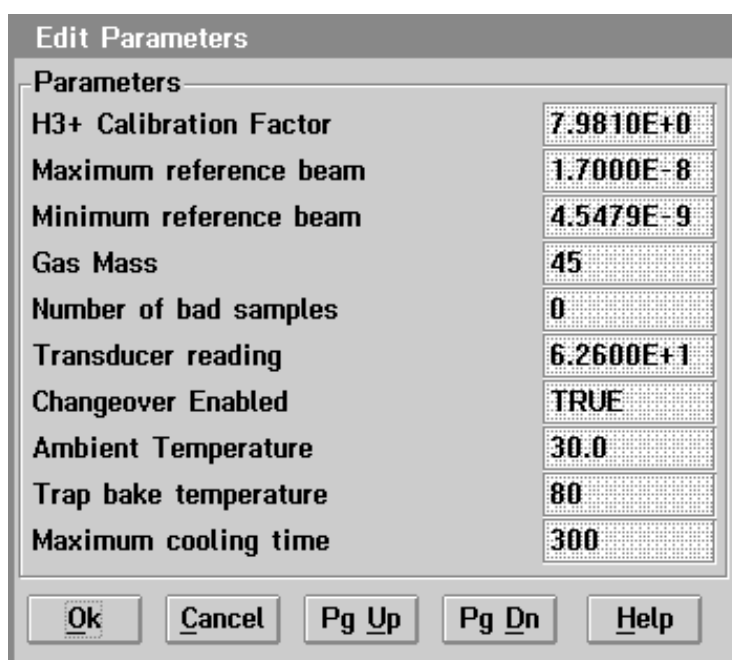


Each file contains one or more pages of parameters contained in entry fields, which can be changed as with any entry field. Please see the next section of this manual for details of the parameters.

The dialog box contains the following push buttons:

**'OK'** saves the parameters and exits the dialog box.

**'Cancel'** exits the dialog box without taking any actions.

**'Pg Up'** goes up to the next page of parameters.

**'Pg Dn'** goes down to the next page of parameters.

**'Help'** gives information on the operation.

**'Close'** exits the dialog box.

**'Help'** gives information on the operation.

# User

The PRISM software incorporates User levels, passwords, and a Login facility. This enables you to protect your machine against unauthorised use, and perhaps more importantly, restrict the access of inexperienced users to portions of the software, so that the consequences of inadvertent mistakes can be avoided, (use of the software is simplified for low level users).

****** The User Levels function of the software is not functioning at time of print. ******

The portions of the software appropriate to the user level are:

## Level 1

This allows access to sample batch entry, autorun start, and a restricted range of the manual control features. This level is appropriate for beginners or visitors who simply need to initiate automated analysis of their samples.

## Level 2

This intermediate level allows access to most of the manual control features of the instrument, and allows editing of selected run parameters etc.

## Supervisor

Users at this level have access to all of the software.

```
Log In...
Log Out...

List Users
Create New User...
Modify User...
Delete User...

View Own Details...
Change Password...
```

**Note:** When there is no user logged in, all menus except USER and HELP are disabled.

The menu options are :

## Log In

This menu option opens the **'User Login'** dialog box which allows the user to log into the system.



The dialog box has 2 entry fields which allow the user to input their **'User Name'** and their **'Password'**.

The dialog box has the following push buttons:

**'OK'** checks the **'User Name'** and **'Password'** for validity, and either exits the dialog box if they are valid giving access to the functions of the appropriate user level or opening a dialog box giving the reason for denied access.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Log Out

This menu option when selected logs the user off the system.

**Notes:**

a) Only the 'User' and 'Help' menu options are now available until the user logs in again.

b) Even with no user logged in, the software remains running. This means that you can start an Autorun, and then logout to prevent unauthorised interruptions or access.

## List User

This menu option opens the **'User List'** dialog box which gives a list of all authorised users.
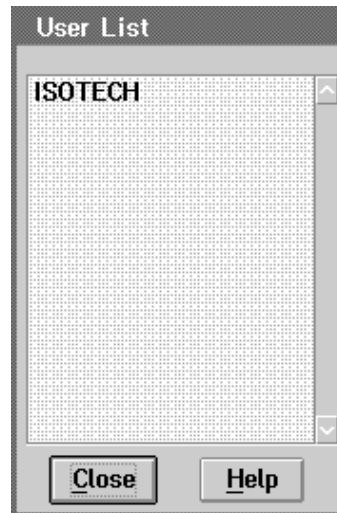


The dialog box has the following push buttons:

**'Close'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Only Supervisor level users have access to the next group of commands in the 'User' Menu:

## Create New User

This menu option opens the **'Create New User Details'** dialog box, which allows new users to be given access to the system.



This dialog box expects the following information in the entry fields:

**'User Name'** requires the new name the user will use to gain access to the software (up to a limit of 10 characters). It is advisable to use a abbreviated version of the user full name to make entering the name easier.

**'Password'** requires a character set with a minimum of 5 characters known only to the new user (maximum 10 characters).

**'Full Name'** requires the full name of the user.

The user level is then selected using the radio buttons in the **'User Access Level'** section

**Note:** The **'Config File'** combo box is not yet implemented and should be ignored.

The dialog box then has the following push buttons:
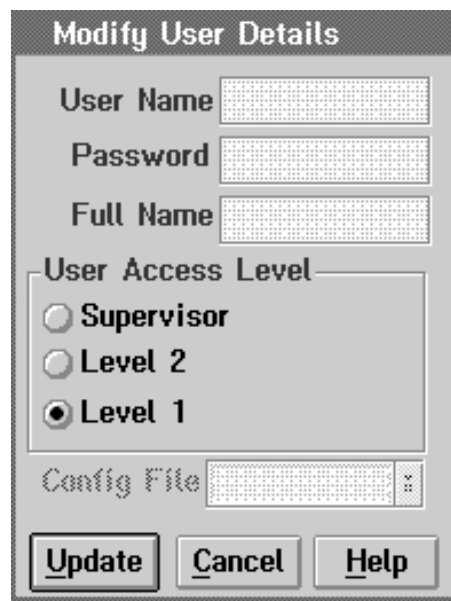
**'Add'** updates the user list with the new user and clears the dialog box, allowing another user to be added.

**'Cancel'** exits the dialog box without taking any actions and should be used to exit the dialog box when no further users are to be added.

**'Help'** gives information on the operation.

## Modify User

This menu option opens the **'Modify User Details'** dialog box, which allows the user access level or password to be altered.



This dialog box expects the following information in the entry fields:

**'User Name'** requires the name of the user whose details are to modified, this once entered will load the 'Full Name' of the user, when the TAB key is used to move to the next entry field.

**'Password'** requires the new password if the password is to be modified.

**'Full Name'** gives the full name of the user once the 'User Name' has been inputted.

The new user level can then selected if required, using the radio buttons in the **'User Access Level'** section

**Note:** The **'Config File'** combo box is not yet implemented and should be ignored.

The dialog box then has the following push buttons:
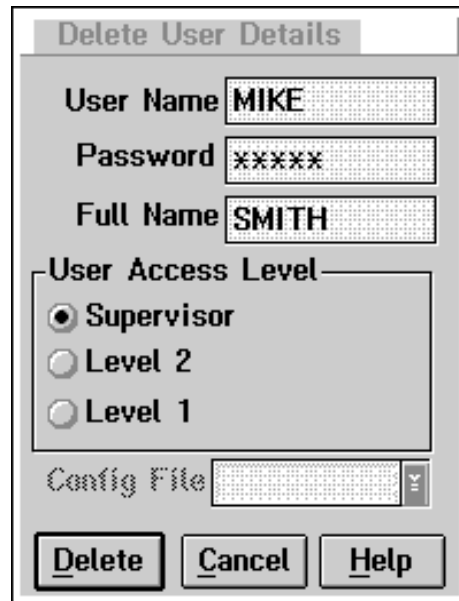
**'Update'** changes the user access level or the password.

**'Cancel'** exits the dialog box without taking any actions and should be used to exit the dialog box when no further users are to be modified.

**'Help'** gives information on the operation.

## Delete User

This menu option opens the **'Delete User Details'** dialog box, which allows user access to be deleted.



This dialog box expects the following information in the entry fields:

**'User Name'** requires the 'User Name' whose details are to deleted, this once entered will load the 'Full Name' of the user, when the TAB key is used to move to the next entry field..

**'Full Name'** gives the full name of the user once the 'User Name' has been inputted.

The new user level is then selected using the radio buttons in the **'User Access Level'** section

**Note:** The **'Config File'** combo box is not yet implemented and should be ignored.

The dialog box then has the following push buttons:

**'Delete'** opens a delete confirmation box, expecting **'Yes'** (to delete the user) or **'No'** (to not delete the user).

**'Cancel'** exits the dialog box without taking any actions and should be used to exit the dialog box when no further users are to be deleted.

**'Help'** gives information on the operation.

## All users have access to the final two User menu commands.

## View Own Details

This menu option opens the **'View Current User Details'** dialog box, allows user to view their own details.



The dialog box then has the following push buttons:

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Change Password

This menu option opens the **'Change Password'** dialog box, allows user to change their password.



The dialog box has the following entry fields:

**'User Name'** gives the present logged in user name.

**'New Password'** requires the new password.

**'Confirm Password'** requires the new password entered to be confirmed by re-typing the new password in this box. Failure to do this and the password will not be changed.

The dialog box has the following push buttons:

**'OK'** enters the new password for the user logged in.

**Note:** For this reason it is advisable to log out of the software when you are not present to avoid changes to your password.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

# Program

One of the unique features of the PRISM software suite is the incorporation of a powerful programming facility. This enables the user to completely program the operation of sample preparation systems, the dual inlet, control valves, stepper motors, read all analogue outputs from the instrument (including ion beams), and to set all digital registers available via the mnemonics (e.g. re-tuning the ion source from a program). In short the objective is to provide all the flexibility of an interpreted language like BASIC whilst retaining all the power and facilities of the operating system and the compiled source code. For details of programming see later sections of this manual.

The PRISM software runs using these sequence files which allows the user to modify the way their system runs if desired.

**Note:** Care must be taken when altering sequences to avoid damage to the system.

This menu gives the user the ability to use these programs individually from within the software.



The menu options are :

## Run
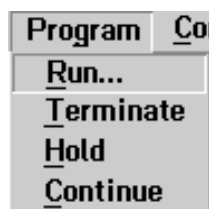
This menu option will open the **'Select Sequence to Run'** dialog box, which executes a selected program.
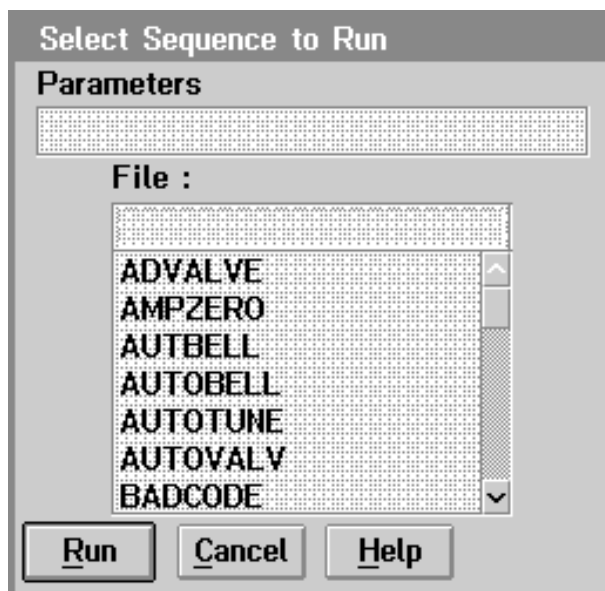


This dialog box contains a **'File'** list box from which the program to be run is selected and a **'Parameters'** entry field into which are put any parameters the program expects can be inputted. The dialog box also has the following push buttons:

**'Run'** starts the selected program.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Terminate

This menu option ends the execution of the currently running program.

## Hold

This menu option pauses the execution of the currently selected program.

## Continue

This menu option resumes execution of the held program.

# Config

This menu gives the user the ability to decide what is monitored in the monitor window and to tell the software the number and type of vacuum gauges that are fitted to the system, thus allowing for further expansion.



The menu options are:

## Monitor

This menu option will open the **'Monitor Configuration'** dialog box. This gives a list of which vacuum gauges, thermocouples, and Thermistors are used on the system and to specify where on the screen they are displayed, either in the monitor window or in the various mimic diagrams on the main screen.
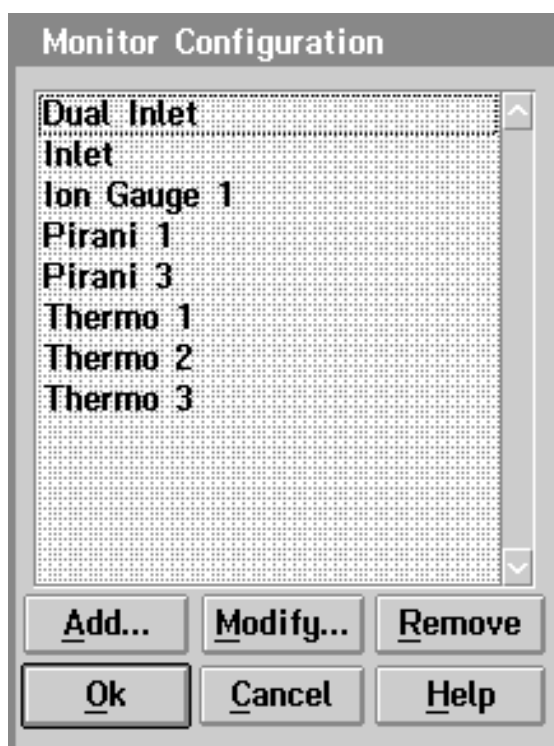


The dialog box comprises of a list box of the various vacuum gauges, thermocouples and Thermistors specified and the following push buttons:

**'Add'** when selected opens the **'Add Monitor Item'** dialog box.

**Add Monitor Item**

Description

Mnemonic

Line Number

Ok    Cancel    Help

This allows for further gauges or thermocouples can be added to the system, if the system expands. The entry fields in this dialog box expect the following information when adding a new gauges, Thermistors or thermocouples:

**'Description'** is the name of the device, and will appear in the 'Monitor Configuration' dialog box.

**'Mnemonic'** is the mnemonic code that identifies the particular gauge or thermocouple in the software (see Table below for details).

**'Line Number'** is a number identifier which specifies where the gauge is displayed in the monitor window (up to 5 lines). If line '0' is chosen then the gauge or thermocouple is displayed only in the appropriate mimic diagram.

| Description | Mnemonic | Normal Line Number |
|---|---|---|
| Source Ion Gauge | I1 | 1 |
| Analyser Ion Gauge | I2 | 2 |
| Analyser Backing Pirani | P1 | 3 |
| Inlet Roughing Pirani | P2 | 4 |
| Carbonate Roughing Pirani | P3 | 0 |
| Inlet Backing Pirani (or Turbo Speed) | P4 | 5 (or 0) |
| Inlet Cold Finger Thermocouple | T1 | 0 |
| Triple Trap Thermocouple | T2 | 0 |
| Carbonate Cold Finger Thermocouple | T3 | 0 |
| Unassigned | T4 | 0 |
| Dual Inlet Transducer | DI | 0 |

**Note:** For ISOCHROM MS mnemonic P4 is used for monitoring the turbomolecular pump speed, as there is no dual inlet.

This dialog box has the following push buttons:
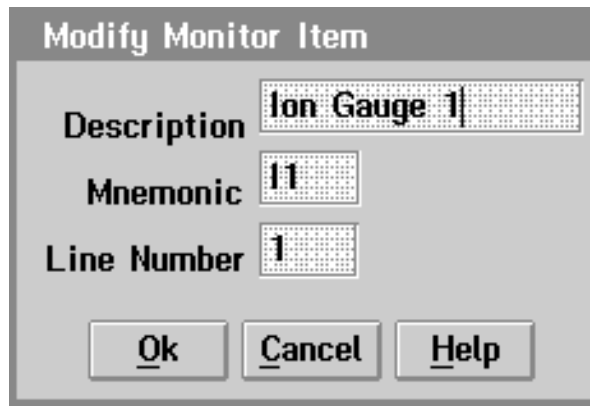
**'OK'** adds the new gauge, Thermistor or thermocouple to the list and updates the screen to include the new gauge, Thermistor or thermocouple.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Modify'** opens the **'Monitor Configuration'** dialog box for the gauge, Thermistor or thermocouple selected from the list box in the 'Monitor Configuration' dialog box.

**Modify Monitor Item**

Description [Ion Gauge 1]

Mnemonic [I1]

Line Number [1]

[ <u>O</u>k ] [ <u>C</u>ancel ] [ <u>H</u>elp ]

This dialog box contains the details for the selected gauge, Thermistor or thermocouple, and can be modified by changing any of the entry fields which have the same function as described above. The dialog box has the following push buttons:

**'OK'** will modify the gauge, thermistor or thermocouple details and update the screen.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Remove'** will remove the gauge, thermistor or thermocouple selected in the list box from the screen.

**'OK'** will action any alterations and exit the dialog box.

**'Cancel'** will exit the dialog box without taking any actions.

**'Help'** gives information on the operation.

## Gauge

This menu option will open the **'Gauge Configuration'** dialog box, This gives a list of which gauges are present on the system and their type, to enable the software to use the appropriate calibration routine, for pressure measurement.
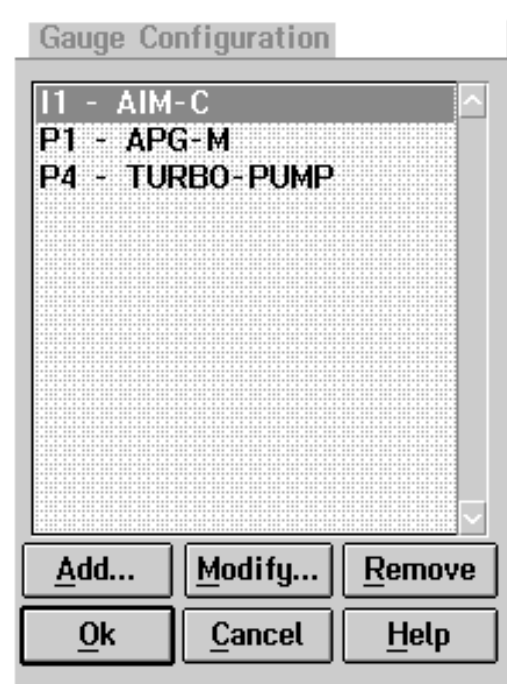


The dialog box comprises of a list box of the various vacuum gauges, specified and the following push buttons:

**'Add'** opens the **'Add Gauge Item'** dialog box, which allows for further gauges to be added to the system in case of expansion.
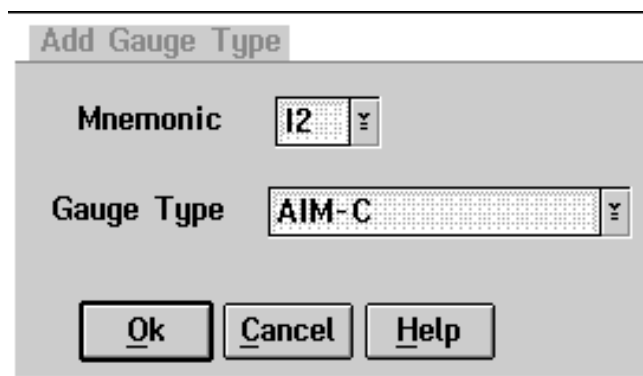


The combo boxes in this dialog box expect the following information when adding a new gauge:

**'Mnemonic'** this is the mnemonic code that identifies the particular gauge in the software (see Table above for details).

**'Gauge Type'** this is where the gauge type is specified, the options for a particular mass spectrometer are listed in this combo box, details of which are listed in the followingTable.

| Gauge Type | Description |
|---|---|
| AIM-C | Penning Gauge (type AIM-C) |
| APG-L | Pirani Gauge (type APG-L) |
| APG-M | Pirani Gauge (type APG-M) |
| Ion Gauge | Non Active Ion Gauge |
| Pirani | Pirani Gauge (type PVG-5) |
| Turbo Pump | Turbomolecular Pump Speed |

**Note:** The system cannot necessarily use all of the above gauge types (e.g. only ISOCHROM MS can use Turbo Pump and AIM-C).

The dialog box has the following push buttons:

**'OK'** will add the new gauge details to the list and update the software to include the new gauge.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Modify'** opens **'Modify Gauge Type'** dialog box for the gauge selected from the list box in the 'Gauge Configuration' dialog box.
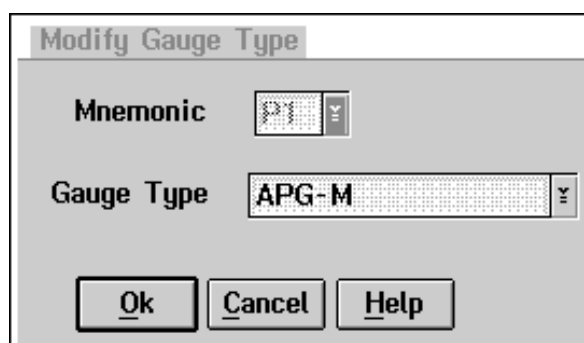


This box will contain the details for the selected gauge which can then be modified by changing the 'Gauge Type' combo box (for details of the combo box see above). It will be seen that the gauge mnemonic is grey as this cannot be modified. The dialog box has the following push buttons:

**'OK'** will then modify the gauge details and update the software to include the modified gauge.

**'Cancel'** exits the dialog box without taking any actions.

**'Help'** gives information on the operation.

**'Remove'** will remove the gauge selected in the list box from the screen.

**'OK'** will action any alterations and exit the dialog box.

**'Cancel'** will exit the dialog box without taking any actions.

**'Help'** gives information on the operation.

# Help

This menu allows the 'Help' facility of the PRISM software to be accessed by the user. The 'Help' files provide a guide to the software as you are running and should be used in conjunction with this manual.

**Note:** As an alternative to using the 'Help' menu most windows have a 'Help' push button, which can be used to open the **'Help Window'** for that section.

## Help Window

Whenever a Help function is selected the information is display in the **'Help Window'**. Within this window the PRISM menu option functions are displayed as separate windows.
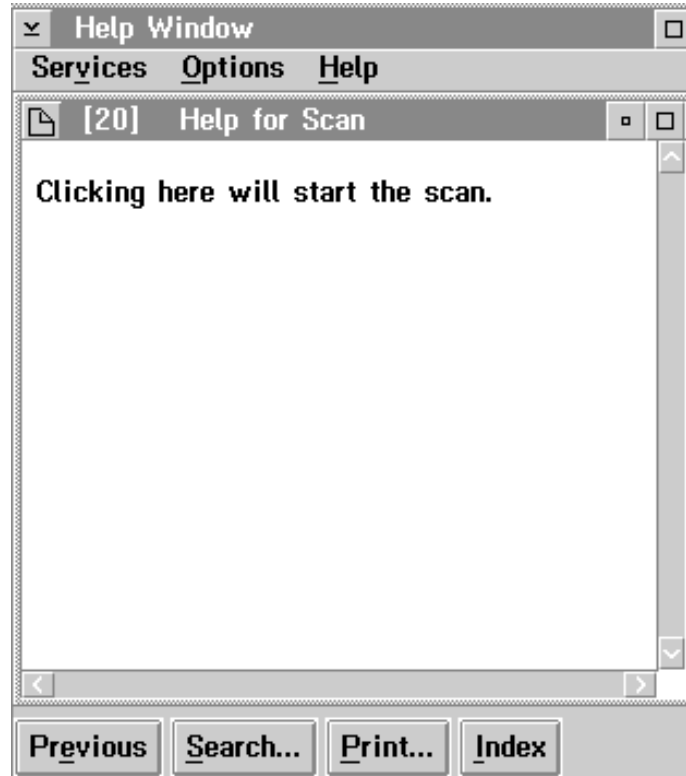


For further details on this window please consult your OS/2 Manual supplied with this system.
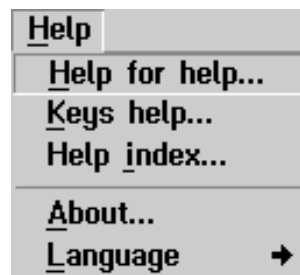
The menu options are :

## Help for Help

This menu option when selected opens the **'Help for Help'** window. This details the way the help files can be accessed.
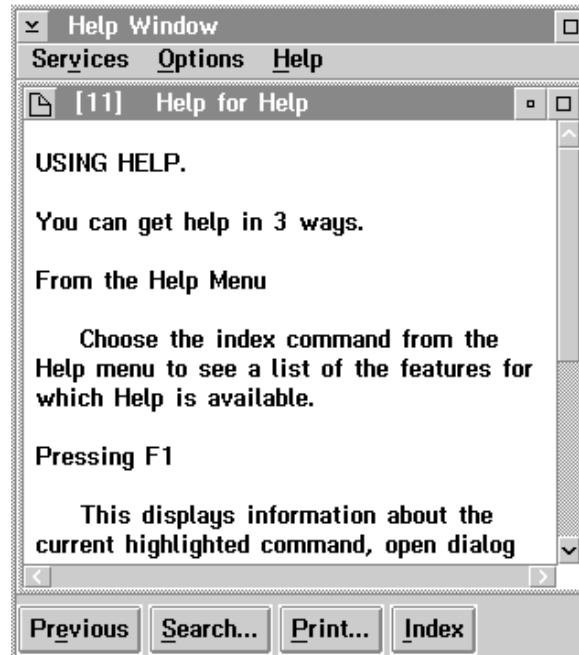


## Keys help

This menu option when selected opens the **'Keys help'** window. This details the functions of the various keys e.g. Tab, Alt, etc..

## Help index

This menu option opens the **'Help Index'** list box within the 'Help Window'.
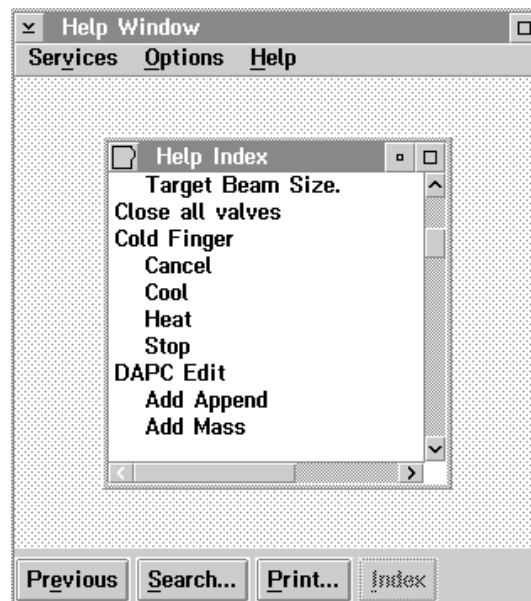


From this list box the titles of the Help files are stored, therefore if for example, you may wish to find the file relating to Bellows, step down the list box (alphabetic order) to the section on Bellows. Then by double clicking on the section required the Help file will then open and you can read the text available on the Bellows.

## About

This menu option when selected displays the **'application about box'** which details the application presently running.

```
┌─────────────────────────────────────────┐
│ Prism                                     │
│                                           │
│            VG Isotech                     │
│                                           │
│    (c) FISONS Instruments Plc  1995       │
│                                           │
│           All rights reserved             │
│                                           │
│                                           │
│    Serial Number                          │
│                                           │
│    Firmware Version : Offline             │
│                                           │
│              ┌──────────┐                 │
│              │  Cancel  │                 │
│              └──────────┘                 │
└─────────────────────────────────────────┘
```

**Note:** The **'Cancel'** push button will exit this window.

## Language

This menu option when selected opens a sub-menu, which enables the user to select the language for the help text.

```
┌──────────────────────┐  ┌────────────────────────┐
│ ✓English Help         │  │ Language            →  │
│  Aide en Français     │  │                        │
└──────────────────────┘  └────────────────────────┘
```

**Note:** The language being used is indicated by the tick to the side of the menu items (this is not a menu on / off tick).

# Parameter Files

This following pages of this section will give a brief description of the parameter files, for reference. For details of how to edit them see earlier in this section and for more in depth details of their function see later sections on the individual preparation systems.

The parameters for your system are set during the test and installation period and are tailored to your system, however you can, if desired, edit them to more suit your particular needs.

## Breath Carousel Parameters

If samples from the Breath Carousel preparation system are run, then these parameters are used.

### Time to hold in $H_2O$ trap.

This is the time to expand from the vacutainer into the water trap before starting $CO_2$ transfer to the trap.

### Time to trap $CO_2$.

The time $CO_2$ is frozen from the breath sample. 120 seconds should be adequate.

### Time to pump over samples.

This is the time that the frozen sample will be pumped over in the trap by the high vacuum.

### Trap pumpout time

This is the time the trap is pumped between samples. At this time the trap is held at the temperature set in the parameter 'Trap bake temperature'.

### Maximum Cooling Time

If the trap takes longer than this time to cool down, a problem with the liquid $N_2$ supply is diagnosed and the run is paused.

### Maximum Warm-up Time

If the trap takes longer than this to warm-up, a problem with the heater is diagnosed and the run is paused.

### Temperature to release sample

This is the temperature to which the trap is raised before the sample is released up to the transducer of the dual inlet.

### Trap bake temperature

This is the temperature at which the trap is baked between samples.

### Inner Ring Position

This is the number of steps of the stepper motor (which drives the carousel) at which point the outer ring of samples have been measured and the sampling needle should be moved to the inner ring.

### First Sample Position

This is the offset in steps on the stepper motor required to position the sampling needle over the first vacutainer.

# Carbonate Carousel Parameters

If samples from the Isocarb preparation system are run, then these parameters are used, as well as the Carbonate parameters.

## Full Turn

Whether the stepper motor does a half step or full step each time. Possible entries are TRUE for a full step, FALSE (Default) for a half step. The half step setting gives a smoother carousel motion.

**Note**: The appropriate setting must be selected on the stepper motor card in the system controller.

## Belt Tolerance

The number of steps required to take up the drive belt slack. Generally around 5 steps.

## Position of Sample 1

The number of steps from Park position to the sample number 1 hole in the carousel disk. Generally around 40 steps.

## Flick Buckets

This allows the carousel to be moved past the bucket drop zone, back again and finally to the bucket drop zone. This gives added confidence that the sample bucket has dropped. Possible entries are TRUE (Default) for carry out the flick, and FALSE for don't carry out the flick.

# Carbonate Parameters

If samples from the Isocarb preparation system are run, then these parameters are used, as well as the Carbonate Carousel parameters.

## Carbonate Reaction Time

This is the length of time (in seconds) for which the sample is reacted with the phosphoric acid and transferred to the cold finger.

## Carbonate Pumpout Time

This is the time for which the Isocarb is pumped out between samples. The cold finger is also baked at this time to a temperature set in the parameter 'Temperature in pumpout'.

## Carousel Flag Offset

This is the number of steps that the first '0' hole is away from the reset flag (Park position) on the carousel. (This value will not change and is set during installation).

## Temperature to stop heater

This is the temperature at which the cold finger heating is stopped. The cold finger is then controlled at the temperature set in parameter 'Temperature for sample release'.

## Maximum temp during freezedown

This is the temperature that the cold finger must reach before the reaction time begins. If this temperature is not exceeded during cooling / freezing, a problem with the liquid $N_2$ is diagnosed, and the run is paused.

## Maximum cool down time

If the Isocarb cold finger takes longer than this to reach base temperature as given in the parameter 'Maximum temp during freezedown', a problem with the liquid $N_2$ supply is diagnosed and the run paused.

## Maximum Warm-up time

If the ISOCARB cold finger takes longer than this to warm-up to the temperature in parameter 'Temperature to stop heater', a problem with the heater is diagnosed, and the run paused.

## Wait for warm up

The time in seconds that the cold finger is allowed to warm up before the transducer is read and the sample is transferred into the inlet system.

## Temperature in pumpout

The ISOCARB cold finger is kept at this temperature during pumpout to help remove moisture etc.

## Temperature for sample release

The temperature at which the cold finger is allowed to warm up to and held at (e.g. -70°C) before the sample is released to the dual inlet.

# Carlo Erba Trapping Parameters

If samples from the Elemental Analyser (Carlo Erba) are to be run via the trap (Double or Triple), then these parameters are used.

## Trap Nitrogen

This parameter is used to decide whether $N_2$ or $CO_2$ is trapped from the Elemental Analyser. Set to TRUE to trap $N_2$, FALSE for $CO_2$.

## Time to pump over sample

Once the sample has been trapped, it will be pumped over on high vacuum for this number of seconds.

## Time to fill with Helium

This time is used to build up helium pressure in the traps before starting the sample combustion. This prevents accidental trapping of air.

## Trap cooling time

This time is used to ensure the trap is cold before starting sample transfer.

## Time to flow through traps

This time (seconds) should be sufficiently long to ensure that all the sample $N_2$ or $CO_2$ will have flowed through the trap after starting the Elemental Analyser.

## Temperature to release sample

This is the temperature the trap must reach before the sample is released to the dual inlet. Room temperature is OK for $CO_2$, but a higher value should be set for $N_2$.

## Trap Pumpout Time

This is the time the trap is evacuated by the high vacuum between samples.

## Trap bake temperature

This is an elevated temperature at which the trap is held during pump out etc.

## Maximum Warm-up Time

If it takes longer than this time to warm-up the trap, a problem with the heater is diagnosed.

## Maximum cooling time

If the trap takes longer than this to reach base temperature, a problem with the liquid $N_2$ supply is diagnosed and the run paused.

# Constant Parameters

Generally the software uses these parameters for storage of general system constants.

## H3+ Calibration Factor

The current H3+ calibration factor is stored here.

## Maximum reference beam

The maximum reference beam height, measured at the beginning of a sample autorun, is stored here (when the bellows are fully closed the major beam height is measured).

## Minimum reference beam

The minimum reference beam height, measured at the beginning of a sample autorun, is stored here (when the bellows are fully open the major beam height is measured).

## Gas Mass

The current axial mass is stored here (also displayed in the monitor window).

## Number of bad samples

Number of bad samples that have occurred during the autorun at present running. If this number exceeds the number of bad samples set in Inlet parameters file then the autorun will be paused.

## Transducer reading

The current transducer reading is stored here, from which the software decides which inlet method to use (cold finger or bellows).

## Changeover Enabled

This location stores whether the changeover valve is enabled or disabled from the inlet menu.

## Trap bake temperature

This is a temporary storage location taking the data from the trapping parameter files of the temperature the trap is baked to during pumpout.

## Maximum cooling time

This is a temporary storage location taking the data from the trapping parameter files of the maximum time allowed for the trap to reach base temperature. If the trap takes longer than this to reach base temperature, a problem with the liquid $N_2$ supply is diagnosed and the run paused.

## Magnet Constant

General Mass Spectrometer equation used when HT peak jumping and is updated when the peak is identified.

## HT Settle Time

This is the time allowed after a HT peak jump for the system to settle prior to data being measured.

## Hall Probe Enabled

This location stores whether the Hall Probe has been enabled.

## Use Mass Cal With Hall Probe

This location stores whether the mass calibration used the Hall Probe.

## Reference bellows maximum

This is the maximum stepper motor steps of the reference bellows.

## Sample bellows maximum

This is the maximum stepper motor steps of the sample bellows.

## Amplifier Zero Channel 0

The location the main analyser Low 1 collector amplifier zero is stored.

## Amplifier Zero Channel 1

The location the main analyser Axial collector amplifier zero is stored.

## Amplifier Zero Channel 2

The location the main analyser High collector amplifier zero is stored.

## Amplifier Zero Channel 3

The location the main analyser Low 2 collector amplifier zero is stored.

## Amplifier Zero Channel 4

Not used at this time.

## Amplifier Zero Channel 5

Not used at this time.

## Amplifier Zero Channel 6

Not used at this time.

## Amplifier Zero Channel 7

Not used at this time.

## Amplifier Zero Channel 8

The location the main analyser Hydrogen 3 collector amplifier zero is stored.

## Amplifier Zero Channel 9

The location the main analyser Hydrogen 2 collector amplifier zero is stored.

## Amplifier Zero Channel 10

Not used at this time.

## Amplifier Zero Channel 11

Not used at this time.

## Resistor Gains Channel 0

The location the main analyser Low 1 collector amplifier resistor value is stored.        (5E+8)

## Resistor Gains Channel 1

The location the main analyser Axial collector amplifier resistor value is stored.  (5E+10)

### Resistor Gains Channel 2

The location the main analyser High collector amplifier resistor value is stored.  (1E+11)

### Resistor Gains Channel 3

The location the main analyser Low 2 collector amplifier resistor value is stored.        (5E+8)

### Resistor Gains Channel 4

Not used at this time.

### Resistor Gains Channel 5

Not used at this time.

### Resistor Gains Channel 6

Not used at this time.

### Resistor Gains Channel 7

Not used at this time.

### Resistor Gains Channel 8

The location the main analyser Hydrogen 3 collector amplifier resistor value is stored.        (1E+11)

### Resistor Gains Channel 9

The location the main analyser Hydrogen 2 collector amplifier resistor value is stored.        (5E+8)

### Resistor Gains Channel 10

Not used at this time.

### Resistor Gains Channel 11

Not used at this time.

# Inlet Parameters

If samples are run using the Dual Inlet, then these parameters are used.

## Inlet Pumpout Time

This is the time in seconds for which the sample side of the dual inlet is pumped (on high vacuum) after each analysis.

## Sample Inlet Time

This is the time in seconds for which the valve SI remains open when transferring a sample to the bellows volume.

## Auto Peak Centre

If this is set to TRUE, the mass spectrometer will peak centre between each analysis.

## Reload Tuning

If this is set to TRUE, your source tuning will be reloaded before each sample.

**Note:** You must have saved the source tuning file as AUTORUN.TUN.

## Maximum Consecutive Bad Samples

If a sample is not analysed successfully (e.g. too small etc.) it is counted as a 'bad' sample. If the number of bad samples exceeds that set by this parameter, the Autorun will be paused. You may restart it if you wish.

## Target Ion Beam

This is the aiming ion current at which the data acquisition is carried out, if the sample is sufficiently large.

## High Enrichment Samples

If this is set to TRUE, the software will watch for saturation on the minor beams during beam balancing, (this slows down the analysis time and should not be used if the samples are not enriched). It is not necessary to use this facility for samples of less than 5 At% enrichment.

## Beam Stabilisation Delay

This is the time in seconds that is waited for when pressure balancing is completed, immediately prior to data acquisition.

## Max Transducer Bellows

This is the transducer pressure in milli-Bars above which the sample is considered too large for safe introduction to the ion source from the bellows. This is a precaution to protect the mass spectrometer from over pressure (e.g. leaking manifold port). A sample size below this is considered safe. If the sample is greater than this value it will be chopped a number of times until the pressure is equal to or less than this value.

## Min. Transducer Bellows

This is the pressure in milli-Bars measured on the transducer, which represents the smallest sample size which can be analysed without use of the cold finger.

## Min Transducer Coldfinger

This is the transducer pressure in milli-Bars which represents the maximum sample size for the cold finger (i.e. without excessive chopping) if the entire sample is transferred.

## Bellows Closed during analysis

This is normally set to TRUE, to maintain identical inlet volumes for reference and sample. However, for hydrogen analysis, it should be set to FALSE, to maintain relatively constant signal/noise and minimise sample depletion.

## Nitrogen Coldfinger Fitted

This should only be set to TRUE if you are using the optional Nitrogen cold finger with trapping agent for analysis of small $N_2$ samples.

## Cold Finger Enabled

This should be set to FALSE if either you have no liquid $N_2$ available, or if you do not wish to use the cold finger (e.g. when running hydrogen).

## Freezedown Time

This is the time in seconds for which a sample is frozen into the cold finger.

## Temperature to stop heater

When the cold finger is being warmed up, the heater is switched off at this temperature, to avoid the temperature overshooting room temperature.

## Temperature in pumpout

The cold finger is kept at this temperature during pumpout to help remove moisture etc.

## Maximum temp during freezedown

If, during freezing, this temperature is exceeded, a problem with the liquid $N_2$ supply is suspected and the autorun is paused.

## Ambient temperature

This is the 'aiming' value of room temperature for warm-up (i.e. the cold finger is considered above room temperature when this is reached).

## Maximum cool down time

If it takes longer than this number of seconds to reach base temperature on the cold finger, a problem with the liquid $N_2$ supply is diagnosed and the autorun is paused.

## Maximum warm-up time

If it takes longer than this time to warm-up the cold finger, a problem with the heater is diagnosed and the autorun is paused.

## Retry on LN2 Fail

If this is set TRUE, the system will retry cooling the cold finger (by restarting the Charles Austen pump) after a liquid $N_2$ problem has been diagnosed.

## Large vol Depletion factor

This is the multiplying factor for the measured sample sample beam, the product is used to set the reference beam value. This factor is used when the large depletion volume is used (i.e. double coldfinger volume). This allows for the sample depleting whilst the reference bellows is being adjusted.

## Small vol depletion factor

This is the multiplying factor for the measured sample sample beam, the product is used to set the reference beam value. This factor is used when the small depletion volume is used (i.e. coldfinger volume). This allows for the sample depleting whilst the reference bellows is being adjusted.

## Length of Bakeout (Hrs)

This is not used, however if you wish to bake the system without temperature feedback then the bakeout time in hours is set in this parameter prior to running the sequence 'Bakeout.Seq'.

## Time For Thermal Equilibration

This is the time delay for the cold finger sample to reach equilibrium after the ambient temperature has been reached.

## Cycle Data Delay Time

Time between sample analysis runs when using the 'Cycldata' autorun procedure

# Isoprep Parameters

If samples from the Isoprep 18 preparation system are to be run, then these parameters are used.

## Sample -2nd Bank Fitted

Set to TRUE if you have a 48 sample version of the ISOPREP 18.

## CO$_2$ Loading Time

This is the time that CO$_2$ from the reservoir is equilibrated with the sample flasks if you choose the automatic filling option.

## Loading Pumpout Time

This is the time the Isoprep 18 is pumped out before loading the CO$_2$ over the samples.

## Dry Run

Set to TRUE if you are testing the system by using empty sample flasks - this saves time.

## Sample Equilibration Time (Hrs)

Enter here the time (in hours) to shake the samples before analysis.

## Pumpout Time

This is the time (in seconds) for which the Isoprep 18 is pumped between samples.

## Sample Transfer Time

This is the time in seconds for which the sample gas is expanded from the flask before the valve SI is opened.

# Manifold Parameters

If 'clean' samples are to be run from the manifold, then these parameters are used.

## 20 Sample - B Bank Fitted

Set to TRUE if you have a 20 sample manifold.

## Use Crackers

Set to TRUE if you have Crackers fitted and wish to use them in the Autorun.

## Pumpout Time

This is the high vacuum pumpout time of the manifold between samples.

## Sample Release Time

This is the time delay from opening the port valve to the next action (taking the sample into the dual inlet).

# Manifold Trapping Parameters

If samples are to be run from the manifold via the trap (double or triple), then these parameters are used.

## Trap Nitrogen

This is set to TRUE to trap $N_2$, FALSE for $CO_2$.

## Time to trap $H_2O$

The sample gas will first be held in the water trap for this many seconds.

## Time to trap $CO_2$

The sample gas will then be held in the $CO_2$ trap for this time.

## Time to trap $N_2$

If $N_2$ is being trapped, it will be frozen down for this length of time.

## Time to pump over Sample

Un-trapped gases will be pumped away for this period of time (on high vacuum).

## Temperature to release sample

The trap will be warmed to this temperature before the sample is released to the dual inlet.

## Trap pumpout time

The trap will be pumped for this time between samples at the temperature set in the 'Trap bake temperature' parameter.

## Trap bake temperature

This is an elevated temperature at which the trap is held during pump out etc.

## Maximum Warm-up Time

This is the maximum time allowed before a problem with the trap heater is diagnosed and the run paused.

## Maximum cooling time

If the trap takes longer than this to reach base temperature, a problem with the liquid $N_2$ supply is diagnosed and the run paused.

# Motor Speed Parameters

If the bellows motor speed is to be changed then these parameters are used.

## Ref. bellows rate (10-200 s/s)

This is the speed in bellows stepper motor steps per second the reference bellows moves at. This is useful if the motor slips due to low system mains voltages.

## Sam. bellows rate (10-200 s/s)

This is the speed in bellows stepper motor steps per second the sample bellows moves at. This is useful if the motor slips due to low system mains voltages.

**Note:** After changing either of these parameters it is necessary to run the program 'Motorate' from the 'Program' menu.

# Single Sample Parameters

If single samples are to be run semi-manually then these parameters are used.

## Inlet Pumpout Time

This is the time the inlet is pumped in the high vacuum between samples.

## Connector Pumpout Time

The time in seconds the transducer region is pumped in high vacuum, after a sample has been confirmed to be attached to the sample inlet port.

## Sample Inlet Time

The time in seconds the sample expanded from its sample bottle.

# Section 6



# Operating Instructions

# Introduction

This section of the manual will describe operating the PRISM. It will therefore guide the operator from electrical switch-on the instrument to running the first zero enrichment sample run and simple autoruns. This section can be followed through by new users wishing to set up the instrument or each sub section can be used for reference, giving detailed information on specific subjects. Please also refer to the User Interface section of this manual.

This section assumes that the system is fitted with a dual inlet. If a continuous flow system is fitted, refer to the relevant section of this manual.

# Switching the Electrical Circuits

This section of the manual describes switching on/off the electrical circuit of the PRISM. The electrical circuit is separated into two halves:

1. Pumping Circuit.

2. Electrical Circuit.
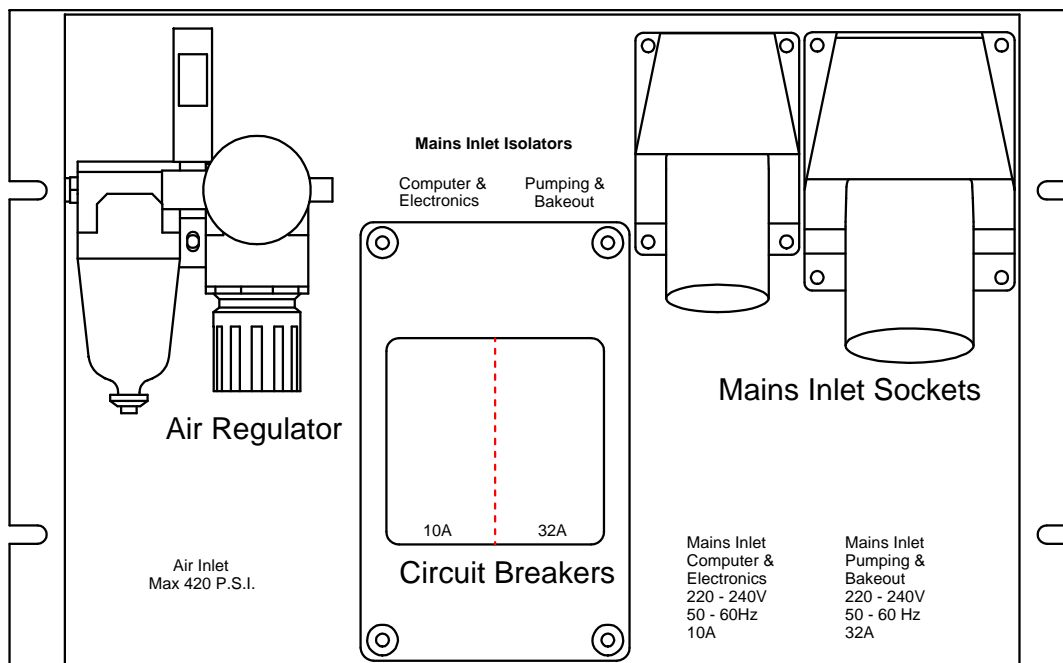


each with its own mains cable, which are connected from the user's supply panel to the PRISM's Mains Inlet Service Panel located at the rear of the analyser bench.

The mains for these two circuits are supplied by the user in accordance with the site specification guide supplied.

# Switching-on the Pumping Circuit

This circuit supplies mains for the Pumps, bake-out heaters and auxiliary devices such as the cold-finger heater and magnetic stirrer.

Before following this procedure ensure that all switches on the Utility Unit front panel are in the **OFF** position (the right hand side of the rocker switch pressed in, the left hand side sticking out).

Ensure that all peripheral devices taking power from the Pumping Circuit (e.g. the rotary pumps) are connected.

Ensure that the pumping circuit mains cable is connected to the inlet on the Mains Inlet Service Panel.

Ensure that the mains supply to the cable is switched-on.

Switch on the pumping circuit via the 32A mains isolator on the right-hand side of the mains isolator box (labelled "Pumping and Bakeout").

# Switching-on the Electronics Circuit

This circuit supplies mains to the electronics units (described in "Electrical and Electronics" section of this manual) with the exception of the Turbomolecular Pump Controller Unit, which is part of the pumping circuit.

Ensure that all peripheral devices taking power from the Electronics Circuit (e.g. the Data System) are connected.

Ensure that the electronics circuit mains cable (from the site service panel or transformer) is connected to the inlet on the Mains Inlet Service Panel.

Ensure that the mains supply to the cable is switched-on.

Switch on the electronics circuit via the **10A** mains isolator on the left-hand side of the isolator box. (labelled "Electronics").

Wait at least 10 seconds for signals to settle.

Switch on the "DC Supplies" via the switch on the Utility Unit front panel.

Switch on the source via the Utility Panel Switch.

Switch on the printer, monitor and computer.

CAUTION

As a general rule never turn the system controller from off to on (via the switch on its rear panel, or the 10A breaker) unless the D.C. Supplies are off, and have been off for at least 1 minute.

# Switching off the Electrical Supplies

Switch off is generally in reverse order of switch on.

**Note:** Before switching off the system ensure that the system is in a safe state e.g. Ion Source off, Ion Gauge off, Pumping system closed down, etc..

# Running The PRISM Software

The computer system is shipped with the instrument and has the software pre-loaded, there should therefore be no need to load the software onto the system, however the software comes with full instructions on installation (any updates of the software will also come with full instructions).

This section of the manual will therefore assume that the software is already loaded onto the computer, the computer is unpacked and on the table provided and the various parts of the computer system (monitor, base unit and printer) are cabled together (for further information refer to the manufacturers guide).

## Starting Up The Software

The following procedure should be followed:

Connect the computer interface cable provided between the Mains Outlet Service Panel connector marked 'Computer Data' and the Computer 'Serial 1' input.

Connect the mains cable provided from the Mains Outlet Service Panel connector marked 'Computer' to the Computer mains sockets.

Turn on the computer, monitor and printer mains switches, this will bring up the OS/2 Desk Top.
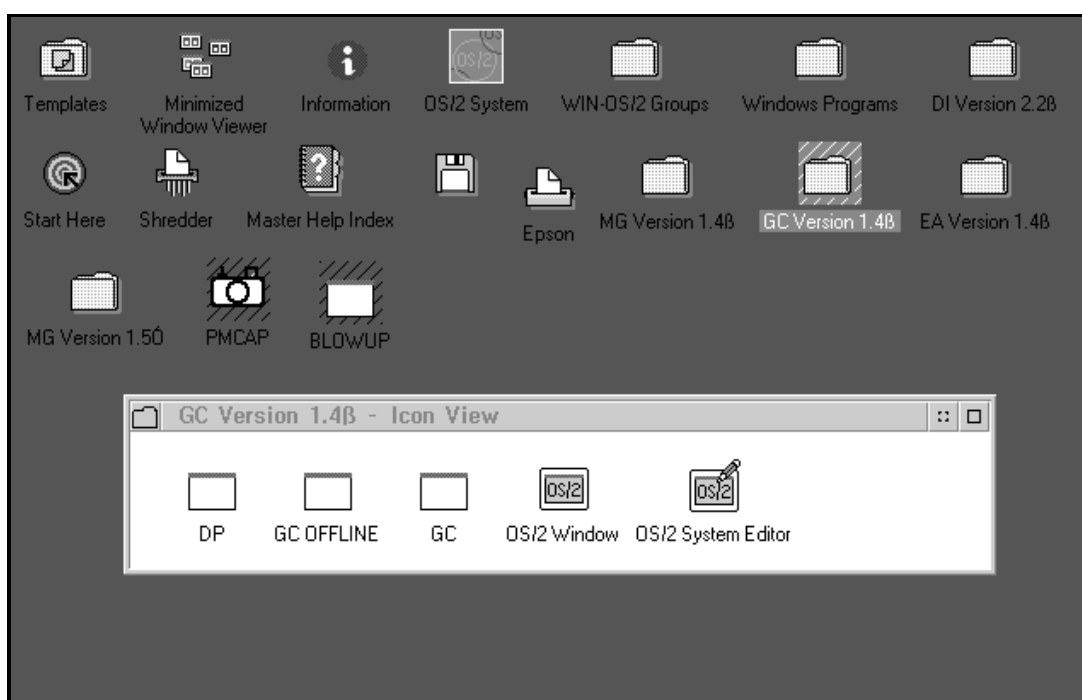


Select Dual Inlet (DI) icon and double click, this will open the Dual Inlet folder.
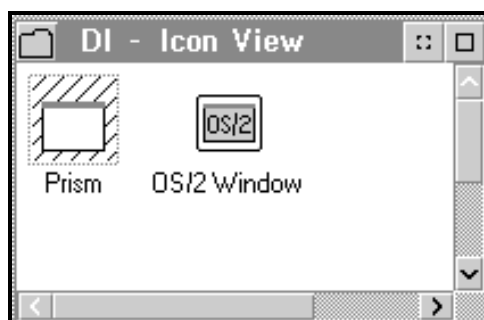
Select the PRISM icon from the folder, a double click on the icon will start the software and the 'User Login' dialog box will appear.

**User Login**
Please Log Into The System
User Name [ ]
Password [ ]
[Ok] [Cancel] [Help]
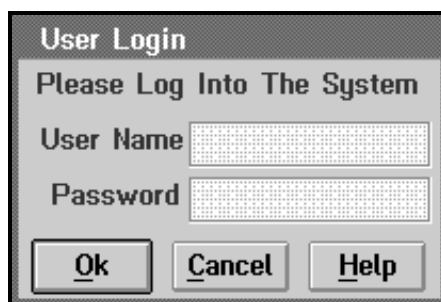
Input your user name and password in the appropriate entry fields of the 'User Login' dialog box, and click on the 'OK' push button.

The software appropriate to your user level can now be accessed and you have control of the instrument.

**Notes:**

a) The default 'User Name' is **ISOTECH** and the default 'Password' is **ISOTECH**, which give the user supervisor level operation.

CAUTION

Please remove these as soon as you have set up your own 'User Name' and 'Password'.

b) Refer to the fault finding section if the software fails to start.

c) If the software has already been run and the software has been shut down with the Dual Inlet folder open, then the software will re-open with this folder active and you can start at step 5 above.

d) If the Dual Inlet icon has been placed in the 'Startup' folder then the software will start-up at step 6 above.

## Logging In

The procedure for logging into the PRISM software, if the software has been just started or the software has been logged out of, is:

Select 'Log In' from the 'User' menu.

**Note:** If the software has just been started then ignore step 1, because the software goes directly to the Login dialog box.

Enter your user name and password in the Login dialog box. Your password will not be displayed on screen.

Click on OK.

You are now logged into the software and have access to the functions appropriate to your user level.

## Logging Out

To log out of the software, select 'Log Out' from the 'User' menu.

You are now logged out of the software. However, the software remains running, which means you can start an 'Autorun', and then logout to prevent unauthorised interruptions or access (to regain access you will need to log in to the system again).

## Configuring the system

The system can be configured for different types of gauges and where they appear on the main window. This is done normally only once at the time of installation, however if details are required, for example, if you are adding extra preparation system, please refer to the previous user interface section on 'Config' menu option.

## Closing Down The Software

To close the PRISM software down, select the system menu from the main menu bar. Selecting the 'Close' Option opens the dialog box shown below .
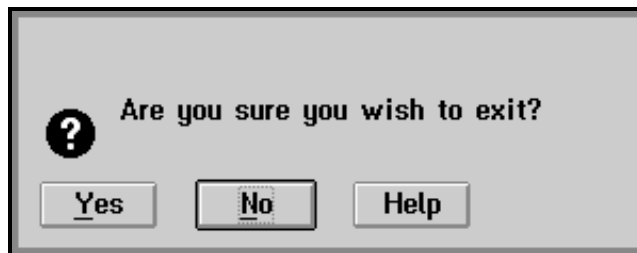


Choose the 'Yes' push button to shut the program down. This will take you back to the OS/2 Desk Top screen with the Dual Inlet folder open, from which the software can be re-started.

**Note:** The system controller maintains the mass spectrometer in a safe condition even though the software is inactive (the mass spectrometer will remain in the same state e.g. source on, valves open, etc.).

It should not normally be necessary to exit the program, as the multitasking features of the operating system remove the need to close the software down to perform other activities. However it is necessary to close down the software if you wish to shut down the system controller or re-read the set-up files for the mimic diagrams and menus (e.g. adding a new preparation system).

Selecting the 'No' push button will ignore the close command and the system will run as normal.

# Pumping Operations

This section of the manual will look at pumping the PRISM system down, setting the vacuum gauges and venting the system. It will assume that the system is "powered up" (which will be carried out during installation) and all the electronic units are working.

# Pumping Down The System

This section of the manual will deal with pumping down the mass spectrometer and also starting the pumping for the Dual Inlet. You should however also refer to the section on evacuating the Dual Inlet. Please also refer to the manufacturers manuals for the rotary and turbomolecular pumps.

## Starting the Rotary Pumps

The procedure for starting the rotary pumps is as follows:

The rotary pumps should be isolated from the system using the manual isolation valves (close the valves) at the rear of the bench prior to starting to avoid oil vapours, which can be produced on start up of the rotary pumps entering the high vacuum system.

Plug the rotary pump leads into the appropriate power socket on the Mains Outlet Service Panel at the rear of the bench. The socket identification is as follows:
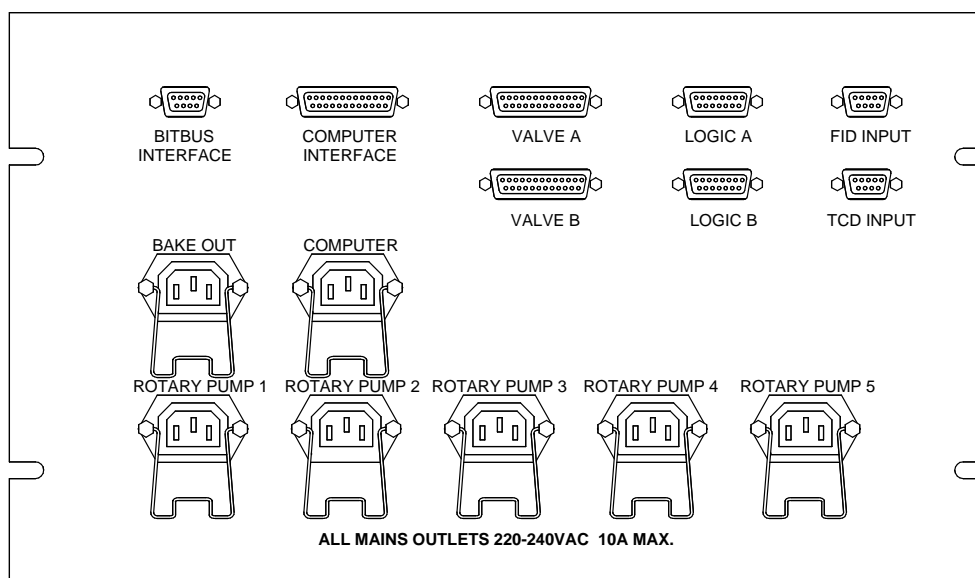


**Mounted at the rear of the analyser bench**

| | | |
|---|---|---|
| Rotary Pump 1 | - | Analyser backing pump |
| Rotary Pump 2 | - | Inlet backing pump |
| Rotary Pump 3 | - | Inlet roughing pump |
| Rotary Pump 4 | - | Prep system pump (Isocarb) |
| Rotary Pump 5 | - | Prep system pump (Isochrom) |

Switch ON the rotary pump switches on the utility unit as necessary.

| | | |
|---|---|---|
| 'Analyser Rotary' | - | controls Rotary pump 1 |
| 'Inlet Rotary' | - | controls Rotary pumps 2 and 3 |
| 'Prep Rotary' | - | controls Rotary pumps 4 and 5 |

The switches required will depend on the number and use of the rotary pumps.

**Note:** these switched power outputs have individual fuses.

Follow the start up procedure in the rotary pump manual. It is advisable to follow the gas ballast procedure to de-contaminate the pump oil and the foreline trap sieve (if the pump has not recently been used or if the oil or sieve is new).

When the rotary pumps have been gas ballasted, then put the rotary settings to 'High Vacuum' mode and gas ballast position '0' (see manufacturers guide for details), and then leave for one hour.

## Starting the Turbomolecular Pumps

When the rotary pumps have been running for one hour up to the isolation valves the turbomolecular pumps can be started using the following procedure:

Switch ON the 'High Vacuum' switch on the Utility Unit. This controls all the turbomolecular pumps (turns ON/OFF). The action of powering up the turbomolecular pumps closes the vent valves and the pumps start to speed up.

Immediately after step 1, open **ALL** the backing line isolation valves, located on the Vacuum Service Panel at the rear of the analyser bench. The turbomolecular pumps will then accelerate to full operating speed. When full speed is reached the LED on the turbomolecular pump controller will light.

The Pirani pressure readings should now start to decrease (monitor window) and when Pirani 1 reaches 2E-2 mBar then the ion gauge may be switched on. If there are no vacuum problems the pressure will continue to fall and the source can be turned on and /or the system baked.

**Note:** For vacuum problems refer to fault finding section of this manual or to the relevant pump manufacturers manual.

# Setting the Pirani Gauges

The Pirani gauges are used to measure the pressure in the mass spectrometer backing lines, with the pressures being displayed in the monitor window.

The Pirani gauges are calibrated on the system for vacuum (1E-3 mBar) and atmosphere (1E+3 mBar). These are factory set, but if new Pirani gauges are fitted the following procedure may be used to calibrate them:

CAUTION

This procedure <u>cannot</u> be followed with the High vacuum pumps running.

Open Pirani to atmosphere and check the pressure reading in the monitor window for a reading of 1E+3 mBar. If the pressure does not read 1E+3 mBar then adjust the potentiometer in the top of the Pirani marked 'ATM'. (atmosphere) until it reaches 1E+3 mBar.

Connect the Pirani gauge to the rotary pump direct using a KF25 to KF10 adapter.

Start the pump and wait four minutes before adjusting the 'VAC' potentiometer (also in the top of the Pirani) for 1E-3 mBar

Repeat steps 1 to 3 until no further adjustment is required and the correct display is achieved.

# Operating the Ion Gauge

The ion gauge(s) are used to measure the pressure in the mass spectrometer, with the pressures being displayed in the monitor window.

The PRISM can have two ion gauges fitted, if the Differential pumping option is chosen. the following description of turning the ion gauge on / off will discuss the source ion gauge (Ion Gauge 1), however it is equally valid for Ion Gauge 2.

## Turning ON the Ion Gauge

The procedure to turn on the source ion gauge is as follows:

Check that Pirani 1 is reading less than 2E-2 mBar (if this reads higher the ion gauge will not switch on and a message will be given in the message window).

Select Ion Gauge from the Mass Spec menu.

Select the appropriate radio buttons for the Ion Gauge, Filament and Current you require (e.g. Ion Gauge 1, Filament 1 and Current 1 for the source ion gauge).

Select Set push button.

The ion gauge should now start and the reading will be displayed in the monitor window. For further details of the Ion Gauge menu, see the User Interface section of this manual.

**Turning OFF the Ion Gauge**

The procedure to turn off the source ion gauge is as follows:

Select Ion Gauge from the Mass Spec menu.

Select the Filament radio button for the ion gauge you wish to turn off.

Select the OFF radio button.

Select the Reset push button.

The ion gauge selected should now be off, as indicated by the display going to 1.3E-3 mBar.

# Zeroing the Transducer

Pump out the Transducer region of the Dual Inlet (see later for details) and select the 'Transducer Zero' command from the 'Tests' Menu.

# Venting the PRISM

Venting the PRISM mass spectrometer is basically the reverse of pumping the system down and the following procedure should be carried out:

Turn OFF the source.

Close all the inlet valves, as this will keep the inlet components dry.

Turn OFF the ion gauges.

CAUTION

It is advisable to wait for at least 30 minutes at this stage to let the source and ion gauges cool down. This helps to avoid contamination or breakage of these components caused by the reaction when hot with air.

Switch OFF the 'High Vacuum' switch on the Utility Unit. This turns off all the power to the turbomolecular pumps. The turbomolecular pump automatic vent valves will not open until the speed of the pumps drop below 50% of full speed.

Immediately after switching the turbomolecular pumps off, close the backing line isolation valves and open the manual vent valves (next to the isolation valves).
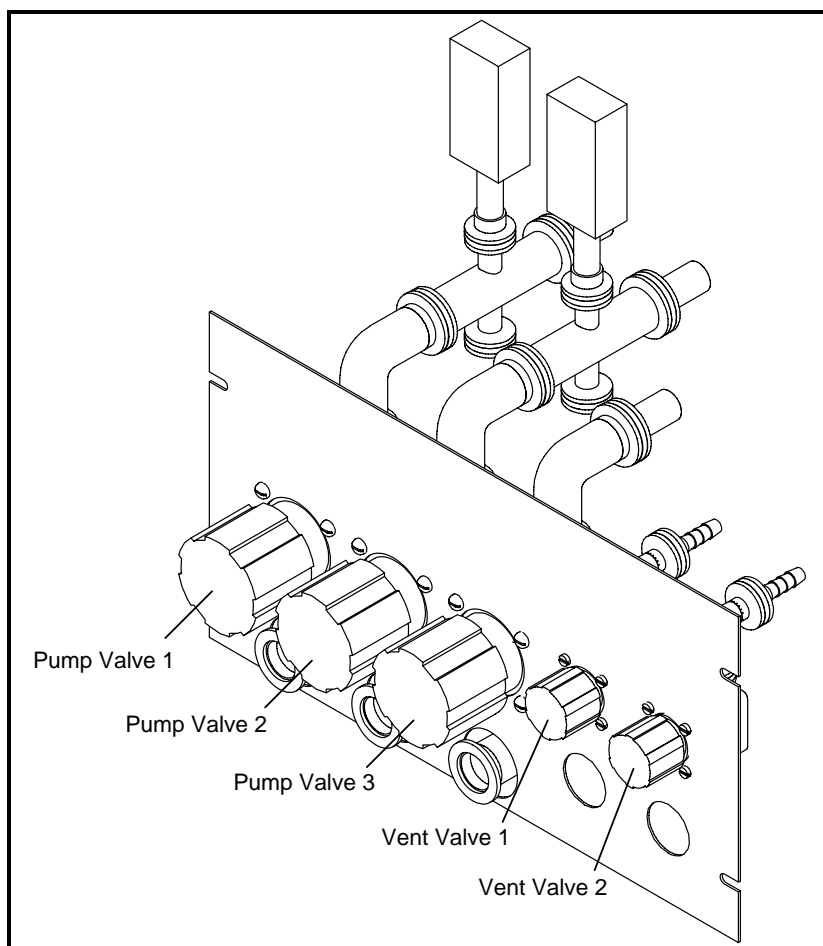
Pump Valve 1

Pump Valve 2

Pump Valve 3

Vent Valve 1

Vent Valve 2

**Note:** Dry Nitrogen or dry Air can be connected to the other side of the vent valves if required. Venting to dry gas will aid the return to high vacuum conditions.

The turbomolecular pumps will now slow down and after approximately 5 minutes the vent valves will open and the system will come up to atmosphere, as shown by the Pirani gauges in the monitor window. The various parts of the system can then be opened as required.

# Dual Inlet Operation

The dual inlet can be operated via the Dual Inlet Window and / or the PRISM Inlet Menu. Please also refer to the User Interface section of this manual.

**Note:** At this stage it will be necessary to check that the 'DC Supply' and 'Auxiliary Mains' switches on the Utility Unit are in the **ON** position.

## Valve Operation

Opening and closing the valves can be done manually (individually) using the Valve Control dialog boxes accessed from the mimic diagram or automatically using the various programs available from the Inlet menu.

### Multi Valve Operation

Valves may be operated rapidly by holding the return key down, and then selecting each desired valve icon in turn. This avoids the need to use the valve dialog box, and makes valve operation very convenient.

## Bellows Operation

Adjustment of the bellows can be done manually or automatically from the bellows dialog box or the bellows can be parked (position at the lower flag position i.e. fully open) if the **'Park Bellows'** command is selected from the **'Inlet'** menu.

### Setting Beam Target

Manually this is achieved using the slider in the Bellows dialog box, selected by clicking on the bellows icon, and visually watching the beam current in the monitor window until the desired beam height is achieved or for automatic operation follow the method below from the Bellows dialog box:

Enter the value (beam target) in the entry field ('Target beam size' in Amps)

Click on 'Set To Target Beam' radio button

Click on the 'OK' push button

The selected bellows will then adjust, until the target beam size is achieved. This can be done on either side of the inlet.

### Balancing Beams

Balancing the pressure in the 2 inlet halves can be done manually using the slider in the Bellows dialog box, selected by clicking on the bellows icon, and visually watching the beam current or for automatic operation follow the method below from the Bellows dialog box:

Click on the 'Balance' radio button

Click on the 'OK' push button

The system will measure the pressure in the other half of the inlet, from the bellows selected and then adjust the bellows in the selected half to achieve balanced ion beam currents on each side of the inlet. This can be done on either side of the inlet.

## Cold Finger Operation

Cold finger operation can be done manually from the Cold Finger Parameters dialog box, selected by clicking on the cold finger icon or automatically using the various sequences during sample running.

# Evacuating The Inlet

If you wish, you can use the mouse to control the valves, and pump the inlet out (or part of the inlet) manually.

CAUTION

Care must be taken to avoid vacuum accidents, e.g. opening the pump lines to an open reference port, etc.

However, the simplest method is to choose one of the 3 pumpout options from the DUAL INLET menu

# Evacuating Both Sides of the Inlet Manually

Before the inlet can be evacuated the reference and sample inlet pipes must be sealed. It is however good practice to keep these pipes sealed at all times, except when a sample or reference container is fitted to the inlet.

To evacuate both sides of the inlet at the same time the following procedure should be followed:

Select 'Close All Valves' from the Inlet menu, to ensure that all the valves are shut and that both halves of the changeover valves are pumped into the waste line.

Open LV to rough pump the inlet pump line, at this stage Pirani 2 (P2) may be seen to rise, wait until a pressure of 1E-3 mBar has again been reached.

Open RP and SP to open each side of the inlet to the pumping line and again wait until Pirani 2 has reached 1E-3 mBar.

Open RI and SI to open the sample and reference ports (please make sure that both sides are sealed off before opening the valves - no risk of opening to atmosphere or pumping away a sample) and again wait until P2 has reached 1E-3 mBar.

Open SF and SV to evacuate the sample bellows and again wait until P2 has reached 1E-3 mBar.

Open RF and RV to evacuate the reference bellows and again wait until P2 has reached 1E-3 mBar.

Open RM and SM to evacuate the capillaries and again wait until P2 has reached 1E-3 mBar.

Open HV, this will cause the software to automatically check that LV is still open (if not it will open it) and then check that the pressure of P2 indicates less than 1E-2 mBar. If all is well then the software will close LV and open HV and hence the inlet to the high vacuum.

The inlet is now being pumped into the high vacuum pump and will stay in this state until HV is closed. It should be left to pump for several minutes to evacuate all the residual gas.

CAUTION

At this stage it is important that the inlet pipe is not removed or any preparation line valves are opened, as this could possibly dump a large quantity of gas into the turbomolecular pump (high vacuum pump).

When the inlet is successfully evacuated the inlet valves can be all closed using 'Close All Valves' from the Inlet menu.

### Evacuating the reference side of the inlet

This is just a copy of the procedure above but without opening any valves on the sample side of the inlet (prefixed S).

### Evacuating the sample side of the inlet

This is just a copy of the procedure above but without opening any valves on the reference side of the inlet (prefixed R).

**Note:** when pumping the sample port the Transducer reading should reach its zero setting (Please refer to the User Interface section of this manual).

# Loading Reference Gas

In order to ensure that enough gas is present to carry out complete analyses it is it is recommended that the reference side be filled such that, with the bellows fully open, the reference major beam is approximately 2E-9 Amps. If unsure about the sample size a lower value can be used but this may result in chopping of large samples. This signal size sets the minimum sample size that can be measured using balanced beams.

To load the reference gas the following procedure should be followed:

After ensuring that all the inlet valves are closed, attach the reference gas cylinder or cracker with reference gas tube (filled to a pressure of between 1 and 1.5 ATM) to the reference inlet pipe.

Set the bellows to their fully open position by selecting 'Park Bellows' in the Inlet menu. This will fully open the sample bellows as well, but this will not matter.

Follow the procedure for evacuating the reference side of the inlet described above and wait 5 minutes by which time the major beam should have stabilised to less than 1E-12 Amps.

**Note:** when waiting for the reference port to reach 1E-3 mBar on P2 if this takes a long time suspect a leak on the reference gas cylinder or connection.

Close all the inlet valves using the 'Close All Valves' from the Inlet menu.

Open RV and RF.

Release the reference gas from its container up to RI, allowing 30 seconds for equilibration, and in the case of a factory reference gas cylinder close the manual valve.

CAUTION

Ideally the gas pressure in the cylinder should be between 0.5 to 1.5 Bar, never use high pressure gas cylinders as these may damage the instrument.

Open RI to allow the reference gas into the reference bellows and wait 60 seconds to equilibrate, before closing RI.

**Note:** At this stage it is unwise to open RM and let the reference into the mass spectrometer unless it is certain that the pressure in the inlet is less than 100 mBar. If unsure the safest way to check this is to let the gas into the sample side of the inlet where the pressure transducer is located. Experience with the instrument will soon be gained and this stage will not be required, as reference gases cylinders can be filled to deliver the correct amount of gas into the inlet.

Open RM and 'Toggle C/O Valve' in the Inlet menu, which will open RC and SW whilst closing RW and SC.

The major beam should now read approximately 2E-9 mBar (if there is no beam or it is unusually low, check that the peak is centred, the source is on and tuned correctly or there is no gas in the reference cylinder as indicated by no rise in the ion gauge reading). This major beam represents the minimum reference beam and thereby determines the minimum sample size that can be measured during the first run in an autorun. If the major beam is set to high a coldfinger sample with a major beam less than this minimum reference limit cannot be measured and that particular sample may be aborted. Therefore if the reference major beam is too high (greater than 3.5E-9 Amps), then the pressure in the reference side must be reduced using the following procedure known as 'Chopping'.

## Chopping

Ensure RP and SP are closed then open HV, this will open LV first and check that P2 reads less than 1E-2 mBar. When HV is open wait 2 minutes.

Close HV and open RP, this will let the gas in bellows expand into the pumping line, thus reducing the pressure and hence the major beam signal size.

After 2 minutes to allow for equilibration close RP. If the beam is still not as required then this procedure can be repeated.

**Note:** Chopping can be done on either side of the inlet or on both sides at the same time by opening and closing RP and SP together.

# Loading Gas for a Zero Enrichment Analysis

This procedure will enable approximately 15 to 20 mBar of reference gas to be loaded into the inlet, which can be used for setting up the mass spectrometer or running a zero enrichment analysis. The exact amount of gas will vary slightly depending on the gas species and the beam height required, however experience with the instrument will aid this.

The procedure is as follows:

Ensuring that all the inlet valves are closed attach the reference gas cylinder or cracker with reference gas tube (filled to a pressure of between 1 and 1.5 ATM) to the reference inlet pipe.

Set the bellows to their fully open position by selecting 'Park Bellows' in the Inlet menu.

Follow the procedure for evacuating the inlet above and wait 5 minutes by which time the major beam should have stabilised to better than 1E-12 Amps for $CO_2$ .

**Note:** when waiting for the reference port to reach 1E-3 mBar on P2 if this takes a long time suspect a leak on the reference gas cylinder or connection.

Close RI, RM, SM and ensure that LV and HV are also closed.

Release the reference gas from its container up to RI, allowing 30 seconds for equilibration and in the case of a reference gas cylinder close the manual valve.

CAUTION

Ideally the gas pressure in the cylinder should be between 0.5 to 1.5 mBar, never use high pressure gas cylinders as these may damage the instrument.

Open RI to allow the reference gas into the inlet and wait 60 seconds to equilibrate, before closing RI.

Read the transducer to see if the pressure in the inlet is between 15 to 20 mBar, this can be adjusted either up or down using the bellows or by chopping.

**Note:** Experience with the instrument will soon be gained and this stage will not be required, as reference gases cylinders can be filled to deliver the correct amount of gas into the inlet.

Open RM and 'Toggle C/O Valve' in the Inlet menu, which will open RC and SW whilst closing RW and SC.

There is now gas flowing into the mass spectrometer and the beam height required can be set using the bellows commands. A zero enrichment analysis can now be run (please refer to later sections of this manual).

# Setting Up The Mass Spectrometer

This section of the manual will describe setting up the PRISM mass spectrometer. This section can be followed through by new users wishing to set up the instrument or each sub section can be used for reference, giving detailed information on specific subjects. Please also refer to the User Interface section of this manual.

**Note:** for Hydrogen see later sections of this manual.

This section will assume the system has a dual inlet attached to introduce samples and that the previous section on the Operation of the dual inlet has been read.

**Note:** At this stage it will be necessary to check that the 'DC Supply' and 'Auxiliary Mains' switches on the Utility Unit are in the **ON** position.

## Source On/Off

Before turning the source on via the computer the 'Source Mains' switch on the Utility Unit must be switched into the **ON** position. Turning the source on or off is then easily achieved by the 'Source ON' and 'Source OFF' options in the Mass Spec menu.

Some points to be aware of when turning the source on:

Ensure the vacuum is better than 1E-6 mBar, especially if turning the source ON for the first time.

The source will not turn on unless the ion gauge is on and the vacuum is better than 1E-5 mBar (see ion gauge trip).

The 'Source ON' option applies the filament current to the source, it does not load any source tuning parameters, etc.

The source parameters are set to their safe zero settings when the source is turned on.

WARNING

If any operation is required to be performed on the source it is essential to turn **OFF** the 'Source Mains' switch, so as to isolate the source electronics from the mains supply.

## Tuning the Source

This section of the manual will look at tuning the PRISM source. It will assume that the mass spectrometer is to be tuned for $CO_2$ , however this procedure can be used for any of the other gas species. It will also assume that the source is turned ON, the source pressure is less than 5E-8 mBar and about 15 to 20 mBar of $CO_2$ is in the inlet with the changeover valve open on the reference or sample side.

**Note:** For Hydrogen specific information please see later sections of this manual.

## Recommended Values

Normally when an instrument is installed a set of source tuning parameters for each gas species of interest is provided for the user. However if for any reason these are not available the following table provides a starting point, prior to optimising, for each gas species. The final optimum parameters for any individual machine may be some way from these values, and should not be worried about if there is still suitable tuning adjustment available.

|  | HD | $N_2$ | $CO_2$ | $SO_2$ | $SF_6$ |
|---|---|---|---|---|---|
| Accelerating Voltage | 4.5kV | 4.5kV | 4.5kV | 4.5kV | 4.5kV |
| Extraction Voltage | 75% | 75% | 75% | 75% | 80% |
| Half Plate Differential | 0 | 0 | 0 | 0 | 0 |
| Z Plate Voltage | 0 | 0 | 0 | 0 | 0 |
| Trap Current | 800μA | 800μA | 800μA | 800μA | 800μA |
| Electron Volts | 100V | 70V | 70V | 70V | 70V |
| Ion Repeller Voltage | +45V | -5V | -5V | -5V | -5V |
| Magnet Current | 2.85A | 2.25A | 2.85A | 3.4A | 4.7A |

## Recommended Source Tuning Procedure

This procedure should be followed for all gases except Hydrogen (see later sections).

Select 'Identify Peak' in the Tests menu and identify the peak as 45 (or any other of the centre masses). This will ensure the correct collector combination has been selected for $CO_2$ .

To identify the mass:

a) Choose Test, Identify Peak.
b) Key the mass into the entry field.
c) Click 'OK'.

Load the source tuning file for the gas species to be measured, in this example $CO_2$ .These files will have been created during the installation and have been saved under an appropriate file name, if not then load the values from the table of recommended values above.

To load a source tuning file:

a) Choose Mass Spec, Tune Source.
b) Click 'Load'.
c) Select tuning file required from the list box ($CO_2$ in this example).
d) Click 'Load'.

Adjust the magnet field and/or the accelerating voltage to focus the appropriate masses at the collector.

**Note:** It is better to use the magnet current and only use the HT for fine adjustment.

Use the Peak Centre command to ensure correct focusing in the axial collector.

Adjust the extraction voltage to give maximum sensitivity.

At this stage it may be necessary to adjust the inlet pressure to provide a suitable working ion beam (8E-9 Amps).

The half plate differential voltage should now be adjusted to give maximum sensitivity.

The z focus should next also be adjusted to give maximum signal.

It may be found periodically necessary to repeat the peak centre procedure, as slight beam steering effects influence the focusing.

The ion repeller should now be adjusted to give maximum sensitivity within a range of -2 to -10 volts. It may be found that lowering the voltage by about 2 volts from the most sensitive position will give an improvement in peak flatness and shape.

The electron volts should also be adjusted for maximum sensitivity. There may be more than one maximum in the electron volt - beam intensity function (the maximum with the best peak shape should be chosen).

Steps 3 to 11 should be iterated until satisfactory peak shapes and sensitivity have been obtained.

Save the source tuning parameters if required.

To save a source tuning file:

a) Choose Mass Spec, Tune Source.
b) Click 'Save'.
c) Select tuning file name to save the file in from the list box ($CO_2$ in this example) or enter a new name in the entry field.
d) Click 'Save'.

The collectors can now be adjusted for coincidence (see below for details), prior to running samples, etc..

# Adjusting the Collectors for Coincidence

When changing gas species or setting up the mass spectrometer it will be necessary to adjust the collector positions, using the collector micrometer adjusters. When there is coincidence of the peaks, each of the isotopes can be measured simultaneously as the flat top of each peak will coincide.

The positions for the collectors will have been established during installation and these should be used as the starting position when moving the collectors for coincidence. The procedure is as follows:

Peak Centre the axial collector

Scan all 3 collectors with auto-repeat over a scan width large enough to get all three peaks on screen. (As this procedure continues it will probably be necessary to keep re-starting the scan to get a new Y max scale).

Move the high mass collector adjuster either way by a small amount (if this is the wrong direction for coincidence then move the other way), continue to move the adjuster until the high mass peak is coincident with the axial peak.

Repeat 3 above with the low mass collector (remember to have identified the new gas species as this will set which collector is used at the low mass - see section on Identify peaks in the User Interface chapter of this manual).

The peaks should now all be coincident and should look similar to the plot below
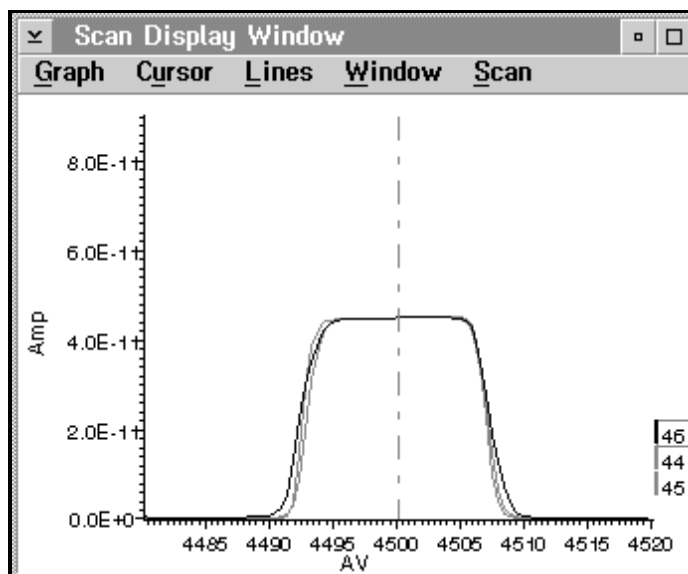
# Peak Shaping

Peak shaping is a very important part of instrument operation. The multi tasking features of the PRISM software are particularly useful for this, as it is possible to simultaneously scan the peak shapes, tune the ion source, and observe the ion current intensities in the monitor window.
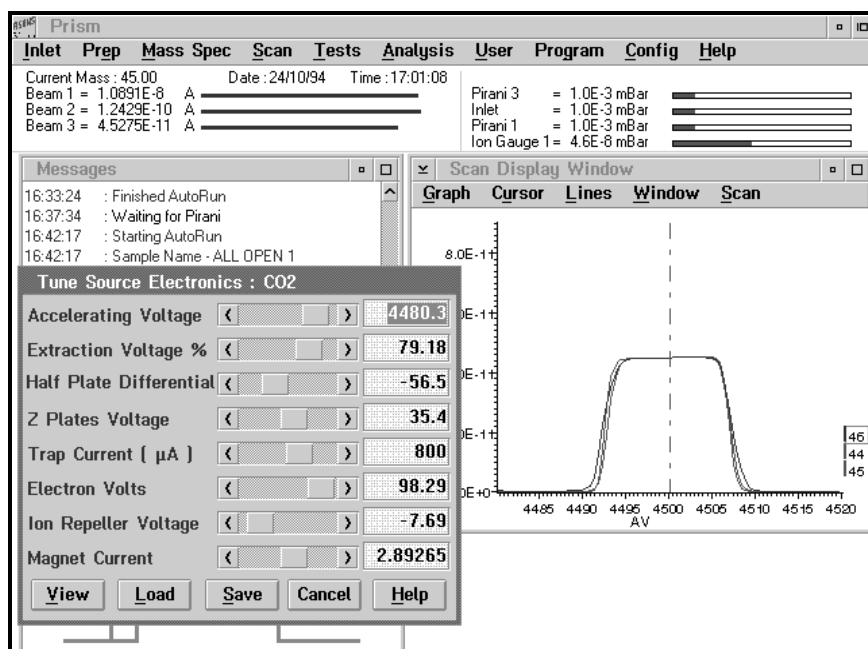


Start an auto-repeat scan, scanning the three collectors (do not worry if the beams are not coincident).

Choose Mass Spec, Tune Source

Adjust the source tuning parameters using the recommended procedures, whilst observing the effect on sensitivity and peak shape.

When happy with the peak shapes, select Scan, Stop

Save your tuning if required.

Choose the Peak Centre command.

A typical $CO_2$ peak shape is shown below:
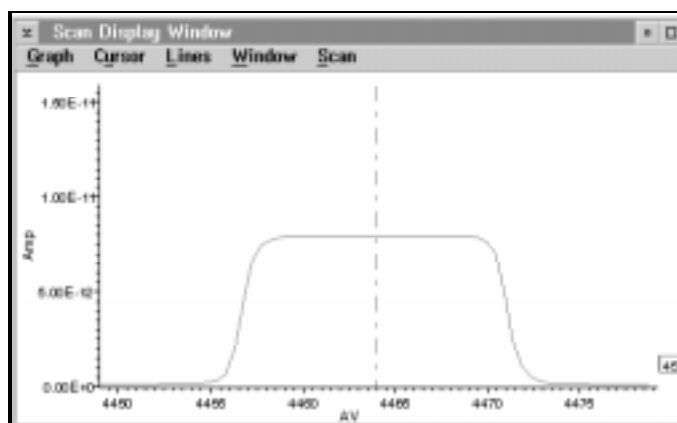
## Adjusting the magnet

Another factor influencing the peak shape is the position and pole angle of the magnet. Movement of the magnet about the flight tube in any direction is a skilled operation and will have been carried out during installation.

Experience of the PRISM has shown that after installation it is rarely the magnet position that causes poor peak shapes. Hence it is **strongly recommended** that the magnet is **NOT** adjusted without careful consideration preferably consulting the factory.

Loss of peak shape is more likely to be caused by:

Poor source tuning

Old or Moved filament

Dirty flight tube

Poor Vacuum

Fault in the electronics.

# Peak Jumping

This operation allows you to focus another mass in the central collector by moving the HT voltage or the magnet current.

**Notes:**

a) If HT peak jump is selected, when jumping to lower masses the HT supply may not be able to go to a high enough HT (5kV maximum).

b) If Magnet peak jump is selected then a mass calibration must have been carried out (see later sections of this manual).

c) If **no** Hall probe is fitted and Magnet jump is selected the magnet will cycle (go to 5 Amps then to 0 Amps) before jumping to the selected mass.

To jump to a new mass:

Ensure the current mass is identified using the Identify Peak command

Choose Mass Spec, Peak Jump

```
┌─ Peak Jump ──────────────────────┐
│         Current Mass : 29.0        │
│   Mass Calibration HT : 4500.3     │
│           Current HT : 4492.3      │
│                                    │
│   Mass to jump to    │ 45 │        │
│                                    │
│  ⦿ Magnet Jump   ☐ Peak Centre     │
│  ○ HT Jump                         │
│  ┌────┐  ┌────────┐  ┌──────┐      │
│  │ OK │  │ Cancel │  │ Help │      │
│  └────┘  └────────┘  └──────┘      │
└────────────────────────────────────┘
```
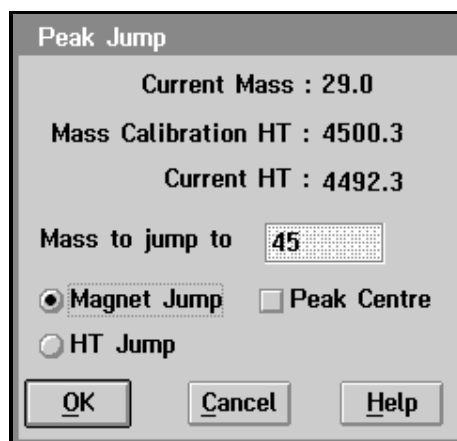
Key the mass into the entry field

Select HT or Magnet jump

Select whether Peak Centre is required after the jump.

Click OK

## Balancing the Capillaries (Adjusting the Crimps)

The capillaries are set during installation and should not need adjusting prior to running samples. However there may be a need to periodically re-adjust the capillary crimps; for example, thermal expansion/contraction effects during a bakeout may move the crimps slightly out of adjustment.

The procedure for adjusting the crimps is as follows:

Pump out the entire inlet

Admit a suitable pressure of gas (say 15 mBar) around the entire inlet (i.e. RP, SP, open). The capillaries should be adjusted (balanced) at the target ion beam of 1E-8 Amps for $CO_2$ and $N_2$ , therefore use the bellows to adjust the ion beam height.

Allow the gas to equilibrate for 10 minutes.

Finally check the beam heights on reference and sample sides. They should be identical down to the 4th significant figure i.e. they should agree to less than 5 in this place:

| **For example** | Ref: | 10.1062 } | not balanced |
| | Sam: | 10.1180 } | |
| | Ref: | 10.1062 } | balanced |
| | Sam: | 10.1088 } | |

If final adjustment is necessary, it is always best to increase the flow rate on the lower beam side than to crimp down further. This avoids the risk of over-crimping.

# Scanning

This section of the manual will look at scanning, which is a useful technique when looking at peak shapes or leak checking. Please also refer to the User Interface section of this manual.

**Note:** If all you want to do is perform a peak centre, choose the 'Peak Centre' command from the 'Tests' menu.

## Starting a Scan

The scanning process can be started in two ways:

Select 'Scan' from the 'Scan' menu, and click on **'GO'** from within the 'Scan Setup' dialog box when the scan details are correct (see User Interface section of this manual for details).

Select 'Run Scan Param File' from the 'Scan' menu, and then selecting the 'Scan Parameter File' required from the list box which appears, click on the **'Run'** push button.

The software will then proceed to scan as per the specified set-up details, the scan will be displayed in the 'Scan Display Window' (see below for details).

## Scan Display Window

When you start a scan, the 'Scan Display Window' appears in the middle of your screen. It can be re-sized and positioned to suit your own requirements. The window has its own Menu Bar and will show a graph for each plot you have set up in the 'Scan Setup' dialog box.
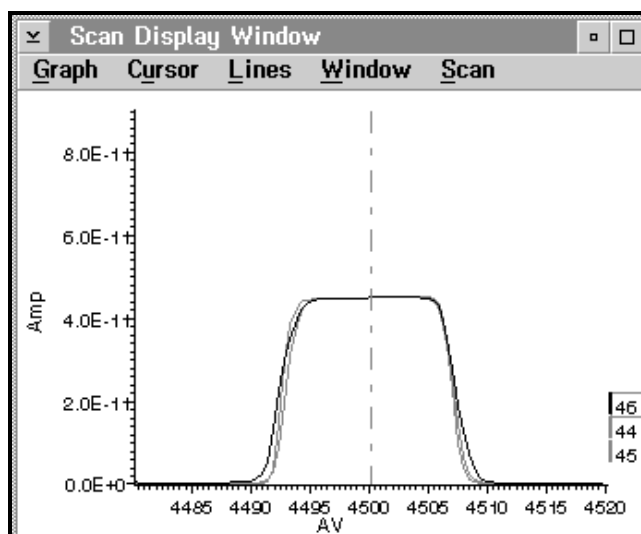


For details please see the User Interface section of this manual.

## Closing the Scan Display Window

Choose 'Close' from the system menu at the top left of the Title Bar of the Scan window.

**Note:** This is a standard system menu bar and can be used to do all the normal system bar functions.

## Printing a Scan

Please see sections above on Graph Menu for details of printing graphs.

**Note:** If you are using OS/2 version 2.0 or higher, you can print the Scan Display Window using "Print Screen" on your PC keyboard (this will print the active window).

# Background Scans

Background scans can be used to investigate suspected contamination and also to check for possible vacuum system leaks to atmosphere. Diagrams depicting three typical background scans are shown below:
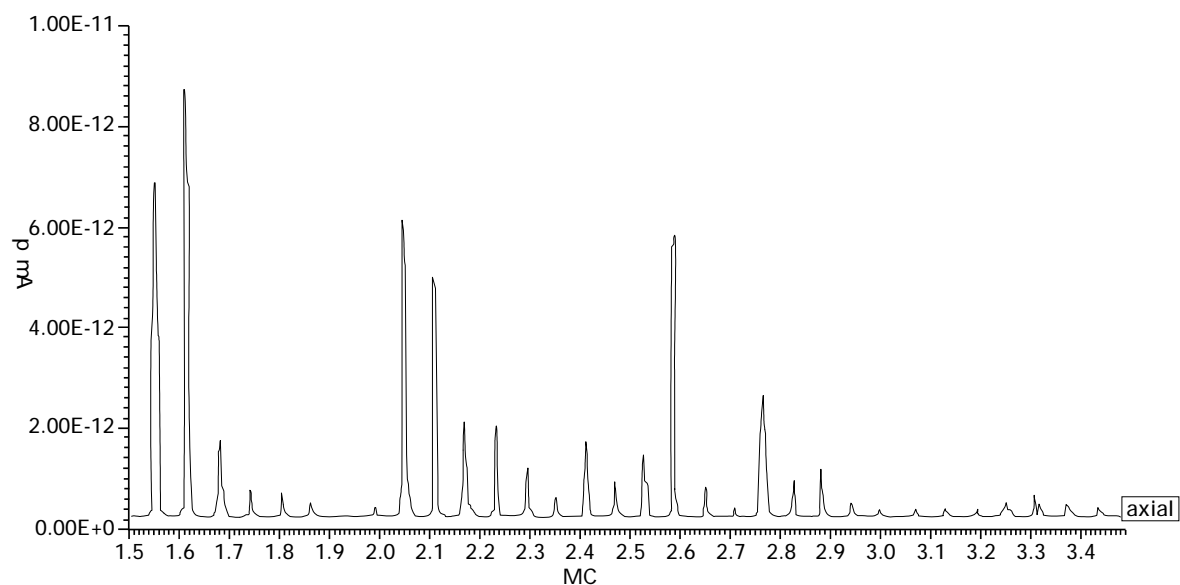


Typical Background Scan Showing Dirty Background Prior To Bakeout
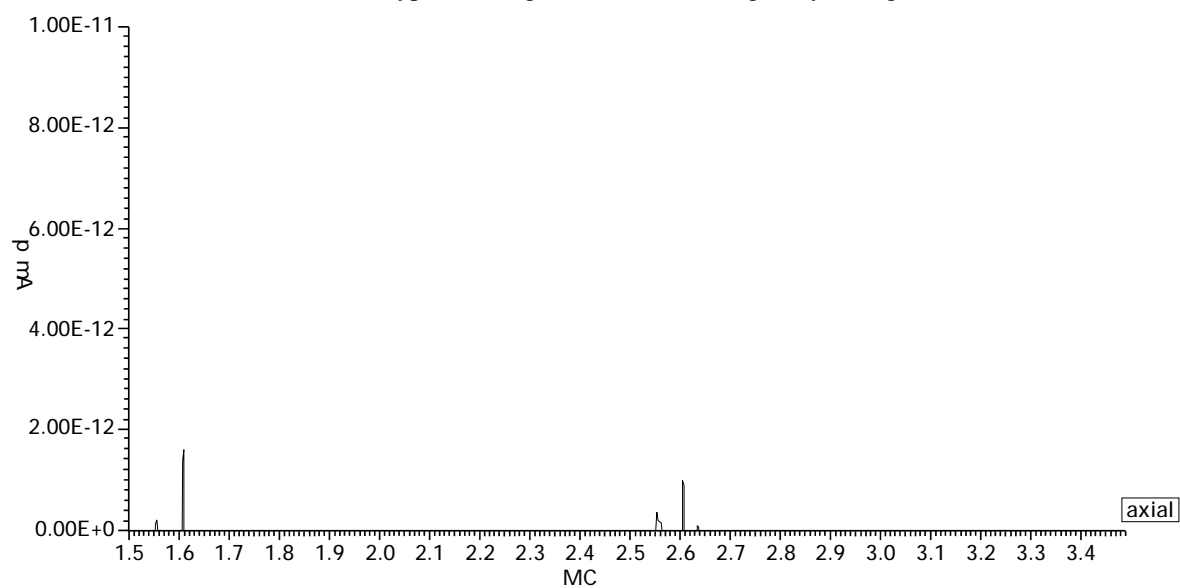


Typical Background Scan Showing Clean Background After Bakeout
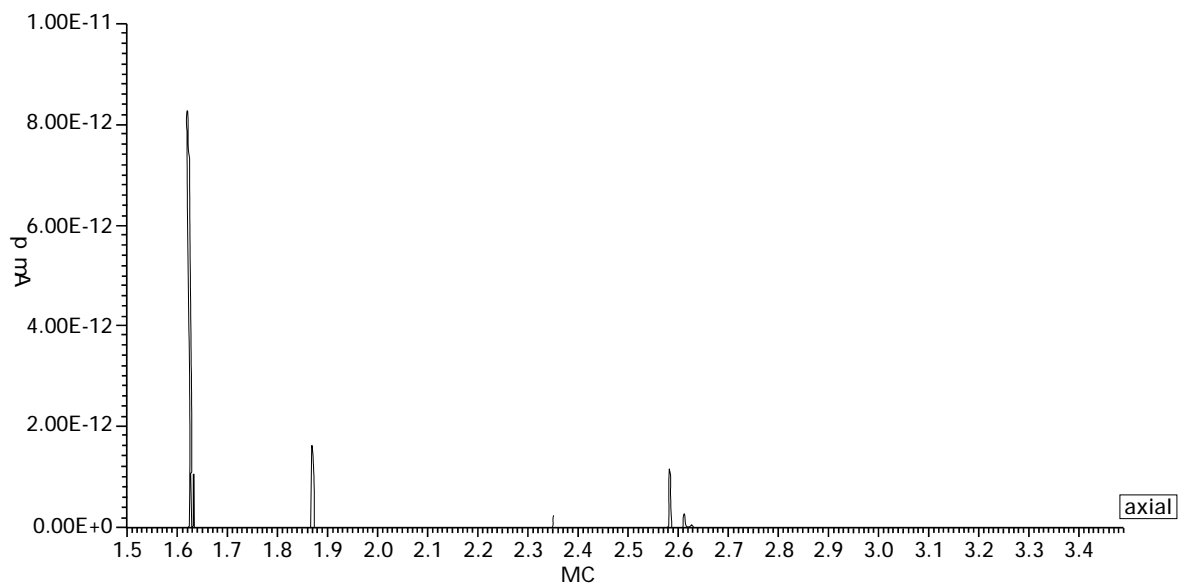
Typical Background Scan Showing Air Leak

These scans can be obtained using the scan programs (Please refer to the User Interface section of this manual.). It is best to scan only the axial channel and to set the maximum (Y) peak height to give sensible peak heights (e.g. 1E-11 Amps).

**Note:** The magnet current is scanned between the masses of interest - see section Tuning the Source for approximate magnet currents or take the magnet currents from the mass calibration.

# Mass Calibration

This section of the manual looks at mass calibrating the mass spectrometer, which is essential when peak jumping with the magnet, as it tells the computer what mass is at which magnet current. The mass calibrations with or without Hall Probe are different and you should **not** therefore assume that if you have carried out a mass calibration with a Hall Probe, it will be valid when you take away the Hall Probe and vice versa. Please also refer to the User Interface section of this manual.

## Mass Calibration without Hall Probe

When carrying out a mass calibration on a system without a Hall Probe fitted the procedure below should be followed:

Tune the PRISM mass spectrometer in the normal way (At say 4.5 kV accelerating voltage), centred in axial collector

Ensure the parameter **'Use mass cal with Hall Probe'** in the **'Constants'** parameter file is set to **'FALSE'**.

Use the Identify Peak command on the Tests and Calibrations menu to set up the mass to be calibrated in the axial collector.

Introduce the calibration gas to the dual inlet. (This can be a gas mixture or several different gases can be introduced one after another).

Go to the Tune Source dialog box. Use the slider control on the magnet current field to focus the mass you wish to set up in the axial collector. The magnet will then need to be cycled (i.e. taken to 5 Amps then 0 Amps then to the mass to be calibrated in one jump) to get rid of any hysteresis effects in the magnet cycle. (The magnet will be cycled in this fashion when peak jumping) This may take some time before the centre of the peak is obtained after jumping from 0 Amps.

When you have noted down the magnet setting for at least 5 masses, leave the source tuning box.

Choose Mass Calibration from the Test and Calibration menu. Enter the values for mass and magnet current from step 5 in the table below.

Click on Calculate and Enter in turn to store the calibration.

Extra points may be added to the first 5 one at a time, or in groups. Remember to click Calculate and Enter in turn to update the calibration.

| Mass Calibration Points Entry | |
| --- | --- |
| **Accurate Mass** | **Magnet Current** |
| 12.00 | 1.63700 |
| 18.00 | 1.77500 |
| 22.00 | 1.90900 |
| 28.00 | 2.60600 |
| 29.00 | 2.66200 |
| 32.00 | 2.81600 |
| 40.00 | 3.21900 |

Calculate  Sort

Enter  Clear  Help  Cancel

**Note:** Enter the magnet setting to full 5 decimal places to ensure accurate calibration.

# Mass Calibration with Hall Probe

When carrying out a mass calibration on a system with a Hall Probe fitted the procedure below should be followed:

Ensure that the Hall Probe is enabled from the Mass Spec menu

Tune the PRISM mass spectrometer in the normal way (At say 4.5 kV).

Ensure the parameter **'Use mass cal with Hall Probe'** in the **'Constants'** parameter file is set to **'TRUE'**.

Use the Identify Peak command on the Tests and Calibrations menu to set up the mass in the axial collector.

Introduce the calibration gas to the dual inlet. (This can be a gas mixture or several different gases can be introduced one after another).

Go to the Tune Source dialog box. Use the slider control on the magnet current field to focus the masses you wish to set up in the axial collector.

**Note:** There is no need to cycle the magnet as the value in the magnet current box is accurate. It is easy to find the exact centre of the peak by using the arrows on the end of the slider box to manually scan the field, and noting the central value.

When you have noted  down the magnet setting for at least 5 masses, leave the source tuning box.

Choose Mass Calibration from the Test and Calibration menu. Enter the values from step 7 in the table.

**Note:** Enter the magnet setting to full 5 decimal places to ensure accurate calibration.

Click on Calculate and Enter in turn to store the calibration.

Extra points may be added to the first 5 one at a time, or in groups. Remember to click Calculate and Enter in turn to update the calibration

# Drift Correction

Once the calibration points have been entered (with or without Hall Probe), there should be no need to update the table. The relationship between magnet setting and mass will remain constant. If you are running manually, perform a peak centre, the go to the mass calibration box, and click on Calculate and Enter to update the calibration.

If you wish to correct for drift in an autorun, make sure the parameter 'Auto Peak Centre' in the Inlet parameter file is set to TRUE.

# Hydrogen Option

This section of the manual will deal with the operation of the Hydrogen option when this is fitted to the PRISM mass spectrometer. Please also refer to the User Interface section of this manual.

## Selecting Hydrogen Mode

The method for selecting Hydrogen mode is as follows:

Pump out the existing sample and reference gases, this may take some time, depending on the gas type, however experience with the instrument will give an insight as to how long to leave it pumping.

Load Hydrogen into the reference side of the inlet (see Dual Inlet section).

Toggle the changeover valve so the mass spectrometer is open to the reference side of the inlet (a rise in source pressure should be observed as the gas goes into the source).

Go to 'Identify Peak' in the Tests menu and identify the mass as 2, this will set up the correct beam mask for Hydrogen, i.e. display the Hydrogen signals in the monitor window (only 2 signals).

Select 'Enable Hydrogen' in the Mass Spec menu. This will remove the magnet current from the main magnet, bring up the current on the Hydrogen magnet and move the Hydrogen magnet into position.

**Note:** the Hydrogen magnet position runs up to a stop when going forwards and is clamped securely to prevent movement up and down or side to side, so after installation magnet will always return to the same position when Hydrogen enabled.

Load 'Tune Source' from the Mass Spec menu, and load the Hydrogen source tuning file (if a Hydrogen tuning file does not exist then a new file will need to be created - see section on Hydrogen source tuning).

Adjust the magnet current so that the Hydrogen 3 peak is approximately in the centre of the H3 collector (the magnet may need cycling) and perform a 'Peak Centre' from the Tests menu.

H3+ calibration should be carried out at this time (see section on H3+ calibration).

**Note:** Remember to do an Amplifier Zero prior to running a H3+ calibration.

The instrument is now ready for the analysis of Hydrogen.

## De-Selecting Hydrogen Mode

Select 'Disable Hydrogen' in the Mass Spec menu. This will remove the magnet current from the Hydrogen magnet, bring up the current on the main magnet and move in the Hydrogen magnet into its rest position away from the flight tube. The mass spectrometer can then be changed to the new gas species.

## Tuning the Source

For Hydrogen follow the procedure in the previous Tune Source section of this manual, with the exception that the Ion Repeller should be set to about +45 volts and the Electron volts to 100V (the higher these values are, normally the better the H3+ calibration).

**Note:** Leave the values for the Ion Repeller and Electron Volts high, **do not** be tempted to lower these values by a great deal as this will effect the H3+.

## Peak Shaping

Peak shaping is a very important part of instrument operation. The multi tasking features of the PRISM software are particularly useful for this, as it is possible to simultaneously scan the peak shapes, tune the ion source, and observe the ion current intensities in the monitor window. The procedure for Hydrogen peak shaping is as follows:

Start an auto-repeat scan, scanning the Hydrogen collectors.

Choose Mass Spec, Tune Source

Adjust the source tuning parameters using the recommended procedures, whilst observing the effect on sensitivity and peak shape.

When happy with the peak shapes, select Scan, Stop

Choose the Peak Centre command.

Typical Hydrogen peak shapes are shown below:
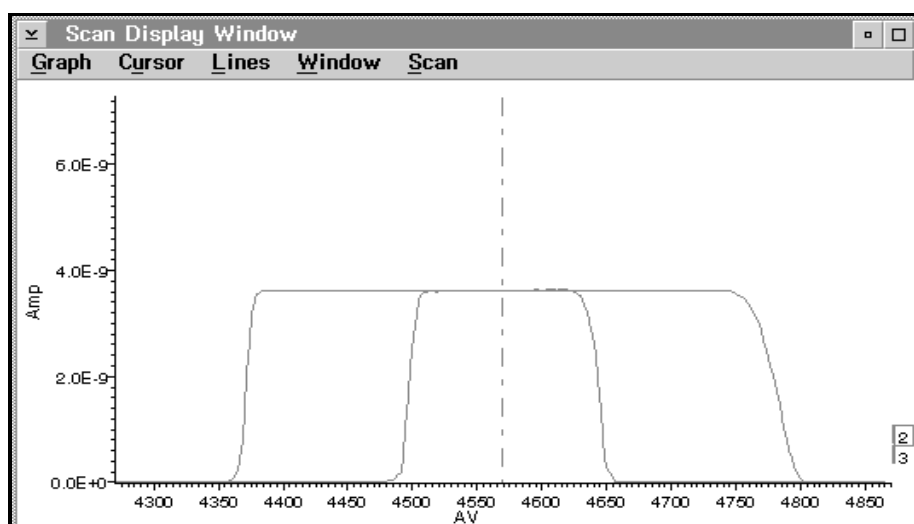
## Adjusting the magnet

Another factor influencing the peak shape is the position of the magnet. Movement of the magnet about the flight tube in any direction is a skilled operation and will have been carried out during installation.

Experience of the PRISM has shown that after installation it is rarely the magnet position that causes poor peak shapes. Hence it is **strongly recommended** that the magnet is **NOT** adjusted without careful consideration preferably consulting the factory.

Loss of peak shape is more likely to be caused by:

Poor Source Tuning

Old or Moved filament

Dirty Flight Tube

Poor Vacuum

Fault in the Electronics.

# Adjusting the Collectors for Coincidence

The Hydrogen collectors are only semi-adjustable in that the vacuum system needs to be vented to move the collectors. The collector buckets are set in the factory and should not need adjusting to get coincidence of the Hydrogen peaks.

# H3+ Calibration

This procedure is used to calibrate the contribution of the H3+ ion to the mass 3 ion current before performing H/D analyses. The procedure is as follows:

Perform an amplifier zero.

Ideally load Hydrogen into the reference bellows to give a signal of 2E-9 Amps with the bellows fully open. A 5 point calibration is performed at currents equal to 100%, 80%, 60%, 40%, and 20% of the **'Maximum beam for calibration'** value.

Select 'H3+ Calibration' from the 'Tests' menu.

Enter 'Maximum beam for calibration' value in the entry field.

**Note:** This must be at least 5 times greater than the beam height in the reference half of the inlet, when the bellows are fully open.

Click on 'Run'

The H3+ calibration will now commence and the message window will inform on the progress of the calibration. At this stage a graph window is opened ready to display the results.

At the end of the calibration, the data collected at each point and the calculated correction factor are printed out. A graph is also displayed on which 2 lines are drawn : 1 joining the raw data points, and a 2nd representing the best fit straight line through the data.

| H3+ Calibration | | | | | |
|---|---|---|---|---|---|
| | **0.2** | **0.4** | **0.6** | **0.8** | **1.0** |
| Mass 2 (nAmps) | 1.81090 | 3.64092 | 5.44286 | 7.28528 | 9.08330 |
| Ratio 3/2 (ppm) | 334.143 | 346.039 | 359.585 | 373.685 | 387.051 |
| | H3 Factor | ....**7.33731** | | | |

**Note:** The H3+ correction factor is the slope of the best fit line.
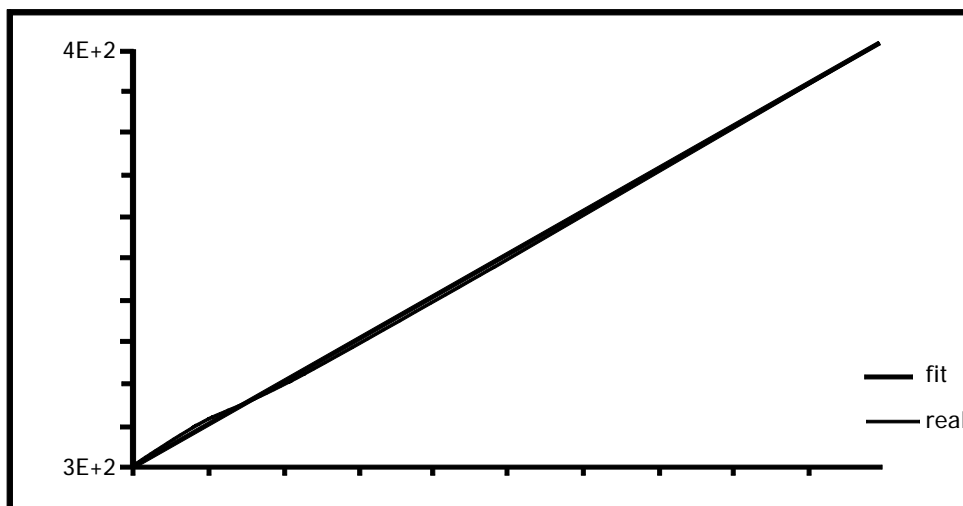


The value of the H3+ correction factor is stored on disk in the parameters file 'Constant Parameters' (see Parameter File section of this manual). The graph is printed out, and the **'H3+ Calibration'** dialog box is opened to display the details of the updated correction value.

Click on the **'OK'** push button which exits the dialog box and the H3+ calibration.

The mass spectrometer now has a H3+ calibration which will be used to correct sample data.

# Hall Probe Option

One of the options available with the PRISM mass spectrometer is to fit a Hall Probe to the magnet. This gives feedback to the magnet supply allowing peak jumps to be achieved at greater speed. Please also refer to the User Interface section of this manual.

# Installing the Hall Probe.

The probe is fitted into the magnet gap at the rear of the flight tube. It is a very tight fit so apply some oil (e.g. Rotary pump oil or any non-corrosive oil) to the surfaces of the probe before pushing it into the gap. The two locking lugs on the cable connector should face down.

## Connecting the Hall Probe.

Fit the Hall probe cable so that the HE14 connector is by the magnet and the 9 way D type is in the STE rack. Connect the HE14 connector to the Hall probe.

## Connecting the EMS Card

CAUTION

Follow procedure in maintenance and fault finding to switch off the power to the system controller (STE rack).

Switch off power to the STE rack and the magnet. Remove the 15 way connector from the EMS card and fit the Hall probe interface PCB in its place. Connect the free 15 way and the 8 way connectors to the Hall probe interface PCB

# Enable/Disable Hall Probe

If a Hall Probe is fitted to the PRISM then it can be enabled and disabled by selecting the 'Enable Hall Probe' or 'Disable Hall Probe' commands in the Mass Spec menu.

**Note:** If there is no Hall Probe fitted do not use these commands.

# Operation

After power on the Hall probe needs approximately 45 minutes to stabilise its operating temperature. This time period should be allowed whenever power is removed from the device.

CAUTION

For optimum performance the probe should be protected from draughts, direct sunlight or any other heating/cooling sources.

# Sample Running

The PRISM can be used to run samples manually, where you manipulate the sample gas yourself or in automatic mode via one of the preparation systems available. This section of the manual will therefore deal with the running samples from setting up sample run files (DAP) to autoruns, as well as detailing information specific to the various gases.

If information is required for an individual preparation systems then the section on that system should be referred to for more detail. Please also refer to the User Interface section of this manual.

# Single Sample Run

The PRISM can be used to run single samples manually, where you manipulate the sample gas yourself, or in semi-automatic mode (see Single Sample Autorun section of this manual). Before running a sample make sure that you an appropriate DAPC file is set up.

### DAPC File Set up

The PRISM software can have numerous DAPC files for the same gas species, however experience with the instrument will mean that often only one DAPC file is required for each gas species (however each user and/or application is slightly different, hence the flexibility in the software). This means that once you have a Parameter file for each gas species, then you will seldom require to edit the Data Acquisition Parameters, unless for some special reason (e.g. new reference gas).

To edit a set of Data Acquisition Parameters:

Select 'DAPC Edit' from the 'Analysis' menu

Click on 'Load' and select the desired DAPC file by entering the filename in the entry box, or selecting one from the list box in the 'Load DAPC Parameters' dialog box.

Click on 'Load'.

Edit the file as required.

**Note:** Please also refer to the User Interface section of this manual.

Click on 'SAVE' and enter the filename in the entry box, or select a name to overwrite from the list box in the 'Save DAPC Parameters' dialog box.

Click on 'Save'.

Click on 'Cancel'.

## Manual Analysis Run

Before running a sample manually, make sure that you have balanced the ion currents for sample and reference using the bellows controls, the valves in the reference and sample halves of the inlet are in identical states. The procedure is as follows:

Select 'Run' from the Analysis menu which will allow a manual (single) data run to be started.

Select the required DAPC file from the list box in the 'Select DAPC File To Run' dialog box .
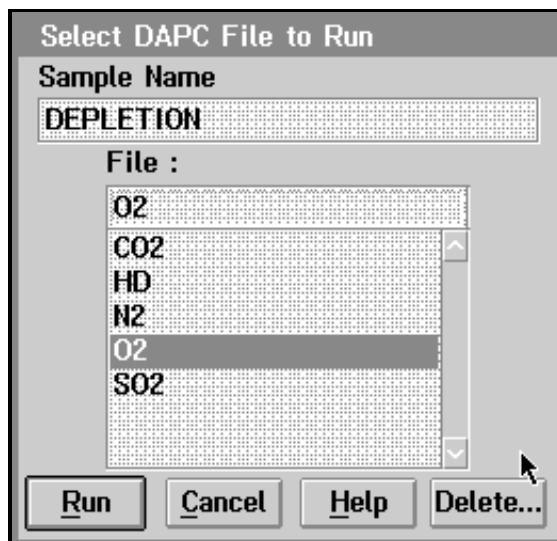


Enter a sample name in the entry field provided.

**Note:** If a sample name is not input at this stage, you will be prompted to do so.

Select 'Run'.

The analysis will now commence and **'Data Acquisition'** Window will open.

## Data Acquisition Window

During data acquisition, the ratios measured are shown on the screen graphically in a form analogous to a chart recorder display. Each ratio measured is shown as an x-y plot, with time as the x axis, isotopic ratio as the y axis. The x axis is divided into blocks by vertical lines which represent each integration interval between toggles of the changeover valve. A red line is drawn between each integration point collected by the data acquisition routine.

**Note:** Please also refer to the User Interface section of this manual for details of the x and y axis's.
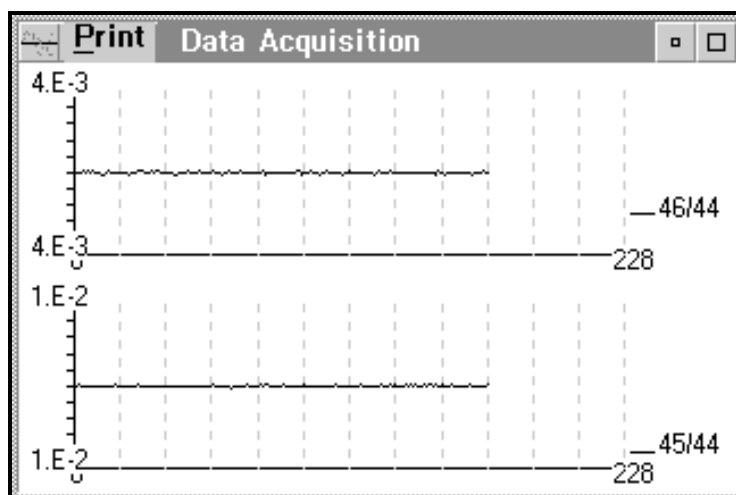


The graph window defaults to about 20 % of the screen area, but it can be repositioned, re-sized, minimised or maximised if you wish to examine the ratio traces in greater detail. The traces are printed out after each analysis, along with the analytical results.

## Aborting a Run

If you wish to stop an analysis in progress, choose the 'Close' option from the system menu bar of the Data Acquisition Window.
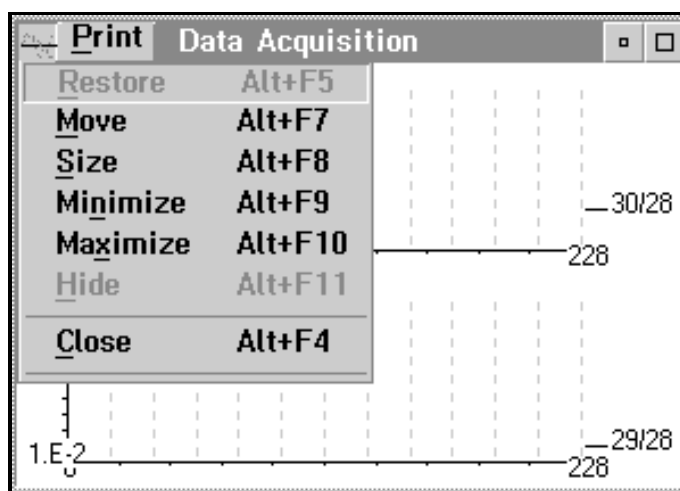
## Results Format

The analytical results are printed out in the format shown below. They are designed to fit on one sheet of paper.

If the Printer spooler is enabled, there will be a short delay while the output is spooled to the PRINT MANAGER application, which means that if the printer malfunctions or runs out of paper, no output is lost as all the results, including graphs, are saved on disk until the printer is available. However, if the spooler is disabled then the data will be lost if the printer malfunctions. To avoid this save the data in the Datalogger.

```
                              Sample Name           : test_2
                              Run Serial Number     : 256
                              Port                  : 2
                              Operator Name         : ISOTECH
Date            :16-12-94     Time                  :11:35
DAPC File       :CO2          Tuning File           :CO2.dap
Sequence        :CYCLDATA.sqfDataLogger Template    :DEFAULT
Ref Gas Name    :CO2              Ref Gas Delta(s)   :delta 45 = -31.114delta 46 = -19.404

Cold Finger Used      : No                  Sample Not Chopped
Transducer Reading    : 6.26E+1
Ion Gauge             : Reference =  1.2E-7 Sample = 1.2E-7

 R/S    Major        45/44          Delta 1        46/44          Delta 2

  R    9.99327E-9   0.0115280                      0.0039869
  S    9.98644E-9   0.0115284                      0.0039868
  R    9.99018E-9   0.0115280      0.031           0.0039868       0.004
  S    9.98341E-9   0.0115280      0.020           0.0039866      -0.003
  R    9.98954E-9   0.0115280      0.003           0.0039867      -0.021
  S    9.98329E-9   0.0115279     -0.004           0.0039866      -0.024
  R    9.98813E-9   0.0115275      0.014           0.0039865      -0.015
  S    9.98210E-9   0.0115274      0.014           0.0039866       0.008
  R    9.98409E-9   0.0115274     -0.003           0.0039864       0.030
  S    9.98223E-9   0.0115276      0.009           0.0039864       0.028
  R    9.98399E-9   0.0115269      0.038           0.0039863       0.016
  S    9.97909E-9   0.0115269      0.030           0.0039862      -0.004

Error Rejected Delta                 0.015                          0.002
Precision                            0.005 (0)                      0.006 (0)
Corrected Delta wrt PDB             -32.547                       -19.353
Delta 18 wrt SMOW                                                  10.910
Atom %                               1.0755                         0.2035
Atom % Excess                       -3.578E-2                      -4.006E-3




The Following Corrections Have Been Applied :
        Amplifier Zero Subtraction
        Craig
        SMOW
        Atom Percent
```



**Example of the Results Format for Zero Enrichment $CO_2$**

# Zero Enrichments Sample Running

A useful way of checking the integrity of the inlet system and mass spectrometer is to run zero enrichment analyses. This is simply to run a sample which is the same as the reference. The easy way to do this is to load the inlet completely with the same gas (i.e. let the gas flow everywhere in the inlet). If a single sample run is now performed and the system is OK then the delta values should be zero. If repeated measurements are required to check for reproducibility then the autorun file CYCLDATA can be used.

The inlet can be investigated further by running cold finger volumes with the same gas.

**Note:** This method is used as part of the specification tests upon installation.

## CYCLDATA Autorun File

This autorun procedure runs the same sample (without manipulating the inlet valves) several times. The number of repeat runs (cycles) is decided by the user by how many times lines are input in the CYCLDATA Autorun file. In our example below, the sample will be run 5 times.



For further details of Autorun refer to the User Interface section of this manual and later in this chapter.

# Changing Gas Species

If the purpose of a mass change is to re-tune the instrument for the analysis of a different gas species, (e.g. when changing from $CO_2$ to $N_2$ analysis). The procedure should be followed as below:

Pump out the existing sample and reference gases. This may take some time, depending on the gas type, however experience with the instrument will give an insight as to how long to leave it pumping.

Load the appropriate new reference gas into the reference side of the inlet.

Toggle the changeover valve, so the mass spectrometer is open to the reference side of the inlet (a rise in source pressure should be observed as the gas goes into the source).

Load 'Tune Source' from the Mass Spec menu, and load the source tuning file for the new gas species (if mass spectrometer has not been tuned before for the new gas species then a new file will need to be created - see previous sections on source tuning).

Go to 'Identify Peak' in the Tests menu and set the axial mass for the new gas (e.g. 29 for $N_2$), this will set up the correct beam mask for the gas species.

Adjust the magnet current so that the centre beam is approximately in the centre of the axial collector (the magnet may need cycling if no Hall probe is fitted, or if a Mass Calibration has been carried out a 'Peak Jump' will do this automatically) and perform a 'Peak Centre' from the Tests menu.

It is now necessary to adjust the collector position, using the micrometer adjusters, following the procedure given in previous sections of this manual.

**Note:** it may be necessary to increase the beam size using the reference bellows to 8E-9 Amps, to make this process easier.

If Hydrogen is the new gas species then H3+ calibration should be carried out at this time.

The instrument is now ready for the analysis of the newly selected gas species.

# Autorun

The PRISM is intended for prolonged unattended operation. Samples are loaded onto one or more of the sample preparation systems, and are then analysed in the sequence and with the procedure that you want. If information is required for an individual preparation systems then the section on that system should be referred to for more detail. Please also refer to the User Interface section of this manual.

## Creating a new Batch File

The procedure for editing a Batch File is as follows:

Choose the 'Analysis' Menu

Select the 'Autorun Batch Edit' command

Select the preparation system file (e.g. MANIFOLD for samples to be run from the Manifold - see preparation system sections of this manual for details) from the list and 'Load'.

Choose Setup to enter your sample details.

Select 'Delete All'.

Select 'Yes', which clears all the previous sample details.

Enter the sample details as required, giving Sample Name, Position and DAP File for each sample to be analysed.

Select 'OK'.

Select 'OK'.

The Batch file is now complete and is ready to run.

## Editing an existing batch

The 'Insert' command allows the adding of a line anywhere in the batch (chosen by positioning the cursor with mouse or cursor keys - the new line is prior to the selected line). The 'Delete' command removes the sample at the current cursor position. The 'Delete All' command clears the file.

## Starting An Autorun

To start an autorun follow the procedure below:

Select 'Analysis' from the main menu bar

Choose 'Autorun Start/Stop' (this is the same menu item for start and stop, it therefore toggles the start/stop).

Select the batch file to run from the 'Select Autorun File' dialog box.

The system will now prompt you to ensure there are adequate supplies of paper etc.
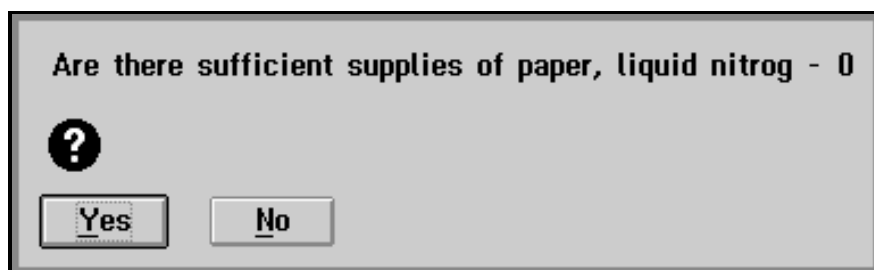


If supplies are short (answer is No) there will be a further question.
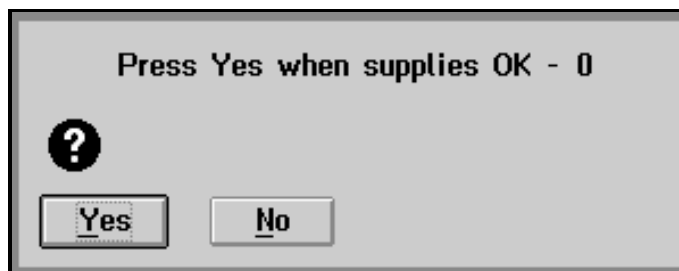


With certain preparation systems, you will be asked further questions about your samples. Once these are complete, the instrument can be left to run your samples unattended.

## Autorun Log

During the progress of an autorun, the Message Window on the screen provides a timed event log of the major steps. Any errors which might have occurred are also logged here. The log is stored, and can be printed out for later examination (when running with OS/2 Version 2.0 or higher).

## Editing the Batch during Autorun

With the editing facilities, you can append or insert samples into the batch file when an autorun is already in progress. This allows you to start the run and continue to add samples to an elemental analyser carousel for example, or simply to correct errors in sample names without stopping the run. The exception is that it is not possible to append further samples if the final sample in the batch is already being prepared.

## Completed Samples

If a sample has already been analysed, its entry is changed from black to grey. Before it can be deleted or edited, it should be made available by using the 'Release' command (chosen by positioning the cursor with mouse or cursor keys). 'Release All' makes available all samples that have been run already.

**Note:** This is also a method of checking which samples have been run by opening the Sample Details dialog box and seeing which samples have been greyed out.

## Multitasking

Because of the multitasking facilities of the software, you can run any part of your instrument not being used by the autorun, such as an idle preparation system. More importantly, you can be processing data in a spreadsheet or a database, or run any other application you wish.

## Editing Parameters during an Autorun

The Parameter Files can be edited during an Autorun, which allows you to make modifications to the way your samples are running. This facility enables you to change timings etc. if you find that the early samples in a run have, for example, had inadequate reaction times etc.

The procedure for this is given below:

Choose the 'Parameter File Edit' from the Analysis menu.

Select the Parameter file you wish to edit with either the mouse or the cursor keys, and then click on the 'Edit' push button.

Edit the required Parameter in the entry fields available.

**Notes:**

a) Parameters are of 3 types - Real numbers, Integer numbers, or Booleans. Real numbers should be entered in scientific notation. Booleans have the value TRUE or FALSE. Edit a parameter value by typing in the normal way. Use the cursor keys to move between fields.

b) Some files contain more than 10 parameters - use the Pg Up and Pg Dn push buttons to move through the extra values (pages).

When the Parameter(s) has been edited select 'OK'.

Select 'Cancel'.

The next time the sequence file containing the edited parameter is loaded then the new value will be used, (this means the subsequent samples will be processed with the new parameters).

## Output from an Autorun

At start of the run, the date, time, and batch being analysed are output to the printer. For each sample analysed, a full report identical to that for a manual analysis is output. When the batch is complete, the batch report is printed.

If the Printer spooler is enabled, there will be a short delay between samples while the output is spooled to the PRINT MANAGER application, which means that if the printer malfunctions or runs out of paper, no output is lost as all the results, including graphs, are saved on disk until the printer is available. However, if the spooler is disabled then the data will be lost if the printer malfunctions.

**CAUTION :To avoid lost data, save the data in the Datalogger.**

## Batch Report

As well as the detailed results report printed out for each sample, at the end of the run a batch report is printed out which gives a one page 'at a glance' summary of the data for the entire autorun.

## Stopping an Autorun (Interrupting a Batch)

If an autorun is running it can be stopped by selecting Autorun Start/Stop in the Analysis menu. The software will then continue until the sample presently being run has completed its analysis.

**Note:** To ensure a fast stop, select the 'Program' menu option 'Terminate' immediately after 'Autorun Start/Stop'

If an Autorun is stopped for any reason, restarting will resume at the first unprocessed sample.

## Single Sample Autoruns

If you choose SINGLE when starting an autorun, you can attach samples to the sample inlet pipe and run them semi-automatically. The software will prompt you when to change the sample, open the container etc.

**Note:** It is necessary in this mode to set up a Batch File with at least 2 samples in it. You can append extra samples as you decide which order to run them in.

# Sample Gas Considerations

Various gases can be analysed on the PRISM. These can derived from a number of different preparation systems (to be discussed in their own manuals). This section of the manual is intended to give a brief insight into the running of the major sample gases.

## Nitrogen (N$_2$ )Analysis

Nitrogen isotope analysis is important in many fields of biological and medical science. The accurate analysis of N$_2$ isotope ratios is critically dependent on avoidance of : (i) impurities, particularly CO$_2$ and; (ii) leaks which introduce atmospheric N$_2$.

If you are working with low enrichment samples, there is no need to adjust collectors when going from CO$_2$ to N$_2$ measurement on the PRISM. The extra collector is set up to remove this requirement. If you are working at high enrichment, and need to measure mass 30, move the high mass collector to the appropriate position after loading the N$_2$ source tuning file and, importantly, using the Identify Peak command, mass 29, to ensure use of the correct low mass Faraday cup.

Leak monitoring should be carried out using mass 32 (O$_2$), and perhaps also mass 40 (Argon). Remember that small quantities of the latter will probably be present in your sample. Excessive O$_2$ levels not only indicate a leak that will have caused dilution of your sample with atmospheric N$_2$, but also will lead to increased mass 30 due to NO production.

Mass 44 should be monitored to check for CO$_2$ impurities. The presence of CO$_2$ causes raised 29/28 ratios because of CO production in the ion source, giving an increased background signal for the sample.

Typical Nitrogen DAPC File

## Carbon Dioxide (CO₂) Analysis

This is the most commonly analysed gas, used for both $^{13}$C and $^{18}$O determination. There are no mass spectrometric difficulties associated with its analysis, as modern instruments have evolved to perform isotopic measurements on ever smaller $CO_2$ samples.



Typical Carbon Dioxide DAPC File

The main factor affecting result quality on an instrument performing to specification is the purity of the $CO_2$ being analysed. Key impurities are: (1) $H_2O$ which can cause isotopic exchange of $^{18}O$, and also protonation of $CO_2$ if present in significant quantity. (2) Organic materials such as cleaning solvents or pump oils, which can create unresolved mass interference's. It is particularly important to avoid such impurities for small samples which use the cold finger, which can concentrate the impurities as well as the $CO_2$. The likely sources of such problems are either inadequate off-line sample preparation (for example, faulty rotary pump on glass line), lack of care (e.g. residual solvents left on glassware), or small leaks on sample vessels (input of atmospheric $H_2O$).

$H_2O$ in the sample can be directly monitored by measurement of mass 18. When checking for leaks, do not use mass 28. $CO_2$ breaks down to give an intense 28 peak from CO. Leaks should be monitored using mass 40 (Argon).

Examination of the ratio trace output of the results report can help greatly in diagnosis of impurities. For example, significant $H_2O$ in the sample produces a "herring bone" zigzag on the 45/44 trace characterised by a rise in sample ratio, followed by a fall in reference ratio. Similarly, organic impurities frequently manifest themselves as steady upward drift in sample ratios, not shown on the reference side.

Remember, you can do a full background mass scan of your sample gas to help in the identification of impurities.

# Hydrogen ($H_2$) Analysis

Hydrogen has certain characteristics which differentiate it from the other gases measured.

The minor beam at mass 3 (HD) is very weak in intensity compared to the other minor isotopes measured, and so results are intrinsically less precise.

An ion-molecule reaction produces unresolved $H_3^+$ ions which cause a pressure dependent variation in the mass 3 beam intensity. This must be corrected for (the "$H_3^+$ correction").

The mass difference between the 2 isotopes is very large, and the fractionation effects are magnified.

Hydrogen is difficult to store for long periods of time and is prone to leakage.

Because of low signal for mass 3, it is essential to have performed the amplifier zero routine before measuring the $H_3^+$ correction factor or carrying out analyses. Remember, the amplifier offset is a much higher proportion of the total signal at mass 3. Ensure the amplifier is fully warmed up.

An $H_3^+$ correction factor should be measured prior to analysis. This factor is an intrinsic function of the ion source, and is very stable long term, but will change slowly as the filament ages. Once measured and stored, the $H_3^+$ correction is automatically applied to all your analyses. It is important that the correction is linear as well as numerically within specification.

**Typical Hydrogen DAPC File**

The physical characteristics of hydrogen mean that care should be taken not to fractionate it during gas handling. The transfer times set as default in your parameter files are suitable for hydrogen analysis. Always allow adequate time for isotopic equilibration. Note that because of their intrinsic mode of operation the vacuum gauges in your system will show larger pressures when handling $H_2$ than for say $CO_2$. This is a characteristic of the gauges, so do not be alarmed when Pirani gauges show high readings during initial pumpout of hydrogen. Remember also that turbomolecular pumps are less efficient at pumping hydrogen than they are other gases, because of its low compressibility. Therefore allow slightly longer high vacuum pumpouts during hydrogen measurement.

On the PRISM, hydrogen analysis is accomplished with the separate spur, magnet, detectors and amplifier. This will have been calibrated during installation. Operation is initiated by the "Enable Hydrogen Mode" command on the mass spectrometer menu. If you have had the HD amplifier disconnected, allow sufficient time for warm up before commencing analysis.

The final point to remember is the purity of the gas itself. Most hydrogen samples will have been prepared by reduction of water either using Zinc or Uranium. Incomplete reaction can lead to the presence of variable quantities of water in the sample. In the worst cases the gas can be saturated with water vapour. This manifests itself by high ion gauge readings, and very slow equilibration of hydrogen beam intensities on admission to the mass spectrometer. Needless to say, such poor samples will give inaccurate results.

## Sulphur Dioxide (SO₂) Analysis

$SO_2$ is the normal gas used for measurement of sulphur isotopes. This is because it is relatively easy to prepare by combustion in oxygen.



Typical Sulphur Dioxide DAPC File

Unfortunately, $SO_2$ is a toxic gas which is quite soluble in water to give sulphurous acid, $H_2SO_3$. The presence of any water in a sample can cause significant analytical problems in terms of inter-sample memory, inaccurate ratios, and long pumpout and transport times. Historically, many analysts thought it necessary to run with inlet systems at elevated temperatures to minimise these difficulties. With a modern micro-inlet system such as that of the PRISM, the analysis of $SO_2$ is easily performed at room temperature, provided the $SO_2$ is clean. All sample measurements should include a measurement of the mass 18 background.

A single analysis of 12 changeover periods (6 sample and 6 reference measurements) can be achieved in 7 minutes using a changeover valve delay of 20 - 25 seconds. A pump out time of several minutes should be allowed between samples.

It is possible to run with the inlet system hot if you wish by using the bakeout heaters and covering oven during analysis. Remember to let the system cool down before returning to normal operation.

When returning to other gases, it is good practice to pump the inlet for at least an hour, or longer if you wish.

# Sulphur Hexafluoride (SF$_6$) Analysis

Sulphur Hexafluoride has become of increasing interest as a gas for isotopic analysis of sulphur. This is because of 2 main reasons:

Unlike SO$_2$, SF$_6$ is essentially inert at room temperature and pressure, and so is not subject to the pitfalls associated with impure SO$_2$ in preparation lines.

Fluorine is mono-isotopic ($^{19}$F), and so measurement of SF$_6$ gives <u>all</u> the sulphur isotopic ratios directly, unlike the under-determined SO$_2$ system.



**Typical Sulphur Hexafluoride DAPC File**

The high mass range and resolution of the PRISM is designed to easily accomplish SF$_6$ analysis. In the ion source, SF$_6$ ionises by loss of F$^-$ to produce SF$_5^+$, and so the peaks collected normally are 127 ($^{32}$SF$_5$), 128($^{33}$SF$_5$), and 129($^{34}$SF$_5$). If you specified the analysis of SF$_6$ for your system, collector positions and source tuning values will have been established for your instrument. If you are attempting SF$_6$ analysis for the first time, the magnet current should be set at about 4.0 to 4.2A in order to focus mass 128 in the axial collector. Masses 127 and 129 will be found by moving the adjustable collectors close together. As when setting any gas up, the easiest procedure is to first put the easily identifiable major beam (127) in the axial collector, then jump one AMU to the small 128 peak. Finding masses 127 and 129 in the adjustable collectors is then easy, because of their high intensities.

$^{34}$S is 4.2% of sulphur, and so the typical operating range for the major beam (127) is up to about 3.5nA.

SF$_6$ is easily condensable in the normal automatic cold finger (the sublimation temperature for SF$_6$ being -63.8ºC), and so the standard inlet system is ideal for small sample analysis. Experience has shown that because of its high mass, SF$_6$ moves slower around inlet and pumping system pipework than other gases, <u>at low pressures</u>. It is advisable therefore to use increased pumpout times and cold finger freeze down times for SF$_6$ analysis. (For example 50% longer than for CO$_2$). Other than this, there are no particular problems associated with the analysis of SF$_6$.

## Oxygen (O$_2$) Analysis

Oxygen can be measured directly as the molecular gas in modern systems such as the PRISM. This has the advantages that there are no ion correction to be applied to raw data, as is the case when $CO_2$ is used to measure oxygen isotope ratios, and the direct measurement of $\delta^{17}O$ is possible.

Because of the high sensitivity of the mass spectrometer, only low $O_2$ pressures are required, and so the introduction of pure $O_2$ to the ion source has no significant effect on filament lifetime.



**Typical Oxygen DAPC File**

The collectors should be adjusted so that mass 33 is in the axial collector and masses 32 and 34 in the low and high mass collectors respectively. If you specified the analysis of $O_2$ on your system, a suitable source tuning file will have been set up. If you are starting from scratch, there are no particular features of source tuning peculiar to $O_2$ : tune the instrument as you would for $CO_2$ or $N_2$. Experience has shown that it may be useful to set changeover valve delay times in the range 15-20 seconds for good results.

Analytical precision for $\delta^{18}O$ measurement using $^{34}O_2/^{32}O_2$ ratios is similar for conventional analysis using $CO_2$. For major ion beams around 10nA mass 32 internal precision for $\delta^{17}O$ measurement using the $^{33}O_2/^{32}O_2$ ratio should be around 0.015‰.

As with $N_2$, the main pitfall with $O_2$ analysis is inaccuracy due to atmospheric leaks affecting sample quality. It is therefore good practice to measure the intensity of $N_2$ at mass 28 as a check of sample purity. Any moisture in the sample can also encourage isotopic exchange.

# Bakeout

The PRISM analyser, dual inlet and manifold incorporate individual bakeout heaters enabling one or more of the sections to be baked at pre-selected temperatures. Baking the system generally improves the vacuum and cleanliness of the system, hence tending to give better results. Bakeout of the mass spectrometer is therefore advisable after, for example, filament changes, and inlet bakeouts are important if you suspect a bad sample has introduced contamination.

## Bakeout Procedure

Bakeout of the mass spectrometer, dual inlet, and the manifold is firmware controlled. A time - temperature profile with up to 3 set points can be programmed for each of the three areas, and independently followed using thermistor feedback. Each of the three sections of the instrument have a slightly different procedure for putting on bake, but if two or more sections require baking at once then follow the sections together. Please also refer to the User Interface section of this manual.

### Analyser Bakeout

This entails removing any components which will be adversely affected by baking (e.g. Head Amplifiers, magnets, etc.), covering the system with a bakeout cover (supplied with the instrument) and starting the bakeout. The procedure for baking the Analyser is as follows:

Disable Hydrogen (if Enabled).

Remove Head amplifier following the procedure below.

Load Source tuning file Bake.tun

Move the main magnet to the rear of the bench having set the return stop.

Place the flight tube heater under the flight tube and plug into the Bakeout socket in the Mains Outlet panel at the rear of the bench.
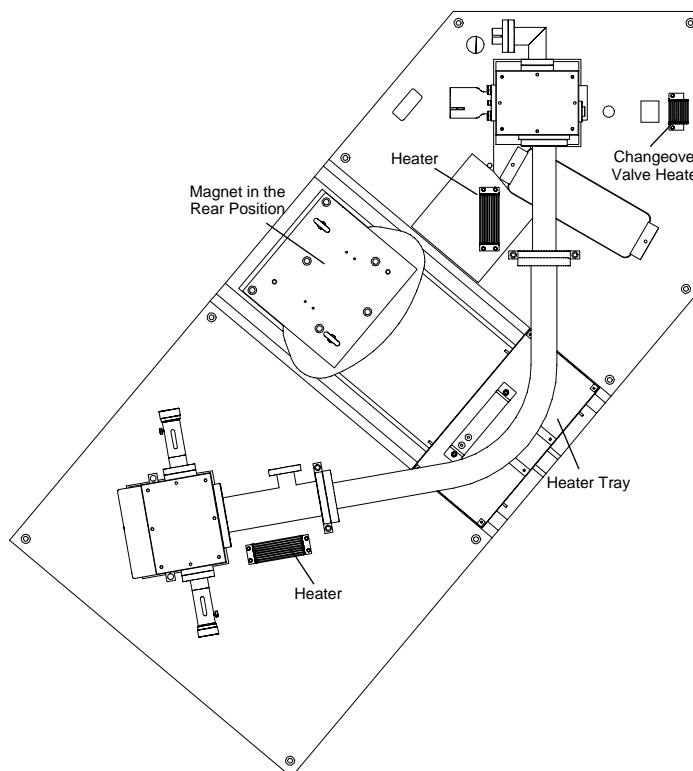
Clean the exterior metalwork to remove any dirt/fingerprints (using alcohol or acetone - avoiding contact with any viton o-rings).

Fit the bakeout cover (supplied with the instrument).

Turn on the Bakeout switch (rocker switch on Utility Unit).

Start the bakeout following the procedure below.

## Inlet Bakeout

The procedure for baking the Inlet is as follows:

Close all inlet valves

Remove all sample and reference containers, and ensure all pipework is sealed.

Manually evacuate the inlet and leaving all the valves open.

Open all the changeover valves using the mouse.

Set the bellows stepper position to '800'.

Clean the exterior metalwork to remove any dirt/fingerprints.

Fit the bakeout cover (supplied with the instrument), ensuring that the Transducer Head is outside the cover, as it is not bakeable.

Turn on the Bakeout switch (rocker switch on Utility Unit).

Start the bakeout following the procedure below.

## Manifold Bakeout

**Note:** When baking the manifold it is essential that the pneumatic air supply is maintained as the valves are normally closed and therefore require air to hold them open, to avoid damaging the valve seats.

The procedure for baking the Manifold is as follows:

Close all manifold valves

Remove all sample and reference containers, and ensure all pipework is sealed.

Evacuate the manifold and leave all the valves open.

Turn on the Bakeout switch (rocker switch on Utility Unit).

Start the bakeout following the procedure below.

**Note:** There is no need to fit a cover on the manifold as the manifold housing forms the bakeout cover.

# To Start a Bakeout

CAUTION

When baking the dual inlet or manifold, ensure that all valves are open.

Always remove the amplifier prior to bakeout of the analyser, observing the anti-static precautions.

Having prepared the appropriate sections of the system for bakeout (as above) then the bakeout can be started using the following procedure.

Select the Bake.Tun source tuning file (i.e. HT = zero and Trap current = 200µA).

Select Bakeout from the Mass Spec Menu

The Bakeout Dialog box appears:



Enter at least 1 time/temperature set point for each component to be baked. Times should be input in hours. One, two or three set points can be chosen to give ramp heating and cooling to the various parts (input in degrees C).

Click on the Start button

The Bakeout Monitor Window appears which shows the current status of the bakeout for each region. There is an **'Abort'** push button associated with each region, which if selected aborts the bakeout for that region without affecting the bakeout for the other regions. The **'Close'** push button is greyed out until all the bakeouts have been aborted (all regions).

| Bakeout Monitor Window | | | | | |
|---|---|---|---|---|---|
| | Temperature°C | | Time | | |
| | Target | Actual | Elapsed | Remaining | |
| Inlet | 90 | 13 | 00:00:22 | 09:59:38 | **Abort** |
| Analyser | 120 | 19 | 00:00:22 | 07:59:38 | **Abort** |
| Manifold | 90 | 19 | 00:00:23 | 09:59:37 | **Abort** |
| | | | | Close | |
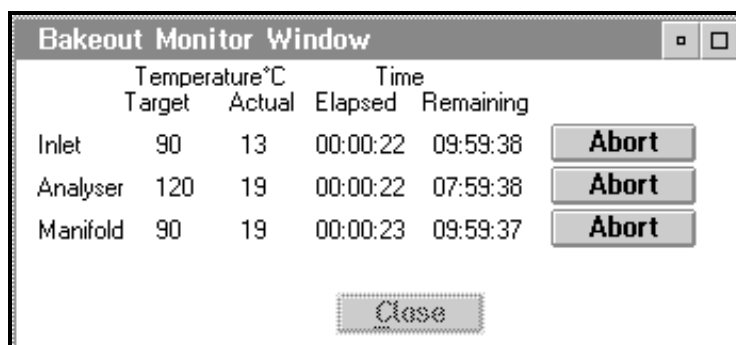
**Notes:**

a) If another region is required to be baked, whilst running a bakeout for one of the other regions, then re-enter the Bakeout menu and input the new details (it will be seen that the running bakeouts are greyed out).

b) If the warning **'Tripped'** appears and the actual temperature indicates **'N.C.'** there is a problem with your thermistors.

## To Stop a Bakeout:

The procedure for stopping a bakeout is as follows:

Click on the required **'Abort'** push button(s) in the Bakeout Monitor window, for the region(s) you wish to stop baking. If a bakeout is aborted , it will be indicated here as shown below.

| Bakeout Monitor Window | | | | | |
|---|---|---|---|---|---|
| | Temperature°C | | Time | | |
| | Target | Actual | Elapsed | Remaining | |
| Inlet | 90 | 13 | 00:01:17 | 09:58:43 | **Abort** |
| Analyser | 120 | 19 | 00:01:17 | 07:58:43 | **Abort** |
| Manifold | 90 | 19 | 00:00:27 | 09:59:33 | Aborted |
| | | | | Close | |
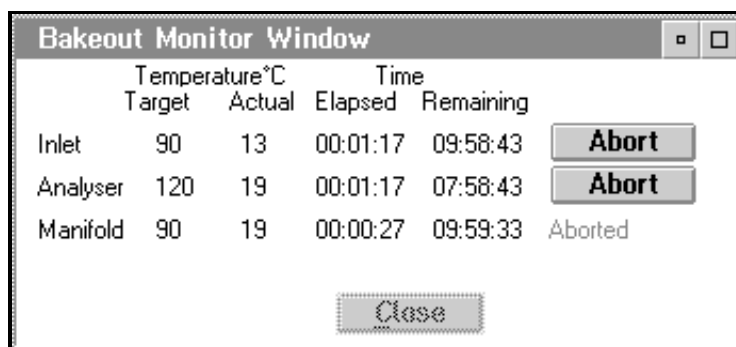
When all bakeout programs have finished or have been aborted, click on the **'Close'** push button in the Bakeout Monitor window and this will close the window.

## Bakeout Completion

Upon completion of the bakeout the system should be put back using the reversal of the bakeout procedures above.

# Removing the Amplifier

The amplifier must be removed occasionally, and **always** before an analyser bakeout.

Evacuate the inlet.

Close inlet valves (these will close anyway when the DC supplies are turned off).

Turn off source.

Turn off the DC supplies (rocker switch on Utility Unit).

Unscrew the two bolts holding the amplifier to the optics housing.

Carefully withdraw the amplifier from the two locating dowels.

Carefully disconnect the fibre optic leads and the DIN power plug from the amplifier (remember which connector is which - connectors are numbered to aid this).

Store the amplifier safely in an anti-static container/bag.

Turn on the DC supplies

Turn on source.

# ReplacingtheAmplifier

Replacing the amplifier is the reverse of removal, noting especially:

Ensure DC supplies are off until the unit is fully reconnected.

Use a screwdriver to discharge any static charge build up from the five feedthrough pins on the optics housing before reconnecting the amplifier (i.e. ground the pins to the mass spectrometer housing/frame). **FAILURE TO DO THIS MAY CAUSE DAMAGE TO THE UNIT.**

# Section 7



# Advanced Software

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# Introduction

The purpose of this chapter is to describe the remaining features of the software package. As such it is essentially an Advanced User's guide, which describes in detail aspects of the package which enable users to 'customise' the appearance and operation of their software.

Descriptions are also included of the interface to 'add-in' features such as the 'ISOLINK' remote modem support package.

# System Files

Various files are used by the software to set-up the behaviour and layout of your system. These include files to define the appearance of pull-down menus, mimic diagrams, and parameter files. Further files are used to define the programs used to run samples in an Autorun, and perhaps most interestingly for the advanced user, files can be written which allow full programming control of instrument and sample preparation system operation.

# Editing and Creating Files

The simplest way to edit system files is with the OS/2 System Editor. If this is not on your OS/2 Desktop, it can be found by opening the 'Productivity' Folder in the 'OS/2 System' folder. Alternatively, open an OS/2 Window from the Command Prompts, and type 'E' at the C: prompt.

**Note:** The facilities available in the OS/2 System Editor are fully described in the OS/2 manuals, which should be referred to in this section.

When saving a file, you will be prompted to define a file type. All files should be identified as 'Plain Text', which are simple ASCII Text Files. Use the Save-As command to save the file in the appropriate directory and **always** use the correct filename extension:

| | | |
|---|---|---|
| *.SEQ | - | sequence files |
| *.PRM | - | parameter files |
| *.SQF | - | autorun sequence files |
| *.TXT | - | menu and Mimic diagram files |

It is also possible to create system files using a more sophisticated package, such as Microsoft Word. However it is essential to save such files as **Text only**, as the software will not expect to encounter any hidden formatting characters.

# Parameter Files

The values of any of the system parameters can be changed from the Analysis menu, using the Parameter File Edit commands. The actual files however contain much more information. Parameter Files are found in the directory **C:\ISOTECH\DI\PARAMS**. Their filenames have the form *.PRM.

## Format

An example of the format is shown below:

```
1"Manifold Parameters"
"20 Sample - B Bank Fitted"    =    FALSE  BooleanT    1
"Use Crackers"                 =    FALSE  BooleanT    1
"Pumpout Time"                 =    60     IntegerT    1
"Sample Transfer Time"         =    30     IntegerT    1
"Sample Release Time"          =    15     IntegerT    1
```

The first line is the text which will appear in the Parameter Files list box on selection of the Parameter File Edit command in the user interface. It is preceded by a number which indicates the minimum User Level needed to access the file from software (see user levels in the User Interface Section of this manual).

**Note:** The text must be included in quotation marks (").

Each subsequent line details one parameter, and has the general form:

**"Text" = Value, Type, User Level**

**Text**

This must be included in quotation marks, and is the text which will appear in the Parameter File Edit dialog box. You may write this in a foreign language if you wish. This text is also used by the IMPORT and EXPORT commands (see later).

**Value**

This is the value initially assigned to the parameter. It must be in the appropriate format and range for the type of variable used.

**Type**

This declares the parameter type. Valid selections are:

BooleanT -     logical, TRUE or FALSE
IntegerT  -     integer numbers
RealT     -     real numbers
CardinalT -     Cardinal numbers
StringT   -     string of characters

**User Level**

A number from 1 to 3 declaring which levels of user have access to changing the parameters.

# Autorun Sequence Files

Autorun Sequence Files are found in the directory **C:\ISOTECH\DI\AUTORUN**. Their filenames have the form *.SQF. They are a list of the 6 main sequences which are used during an autorun, of any particular preparation system (see Preparation System manuals for more details).

## Format

Each Sequence File has the following format consisting of 6 lines, each of which must include the name of a valid *.SEQ file in the **C:\ISOTECH\DI\SEQUENCE** directory. Each line starts with SEQUENCE (upper case) followed by a space, a colon (:), a space, and then the sequence filename. The line is terminated by a semi-colon (;).

The example below shows MANIFOLD.SQF, used during a manifold autorun.

```
SEQUENCE : startup.seq;
SEQUENCE : manstart.seq;
SEQUENCE : manreles.seq;
SEQUENCE : maninlet.seq;
SEQUENCE : pumpsam2.seq;
SEQUENCE : nopump.seq;
```

The first sequence named is executed once, at the start of the autorun. It is used to initialise the mass spectrometer and dual inlet. Actions which occur at this stage include measurement of the reference gas pressure limits and, evacuation of the sample side of the inlet, and prompts to the user if any actions / decisions are required.

The second sequence named is executed once, and is used to initialise the sample preparation system in use. Actions which may occur can include prompts to the user about samples, and initial pumpout.

The remaining 4 sequences are executed in turn, for each sample named in the autorun batch file. If there are 5 samples in the autorun batch file, each sequence will run 5 times.

The third sequence performs sample preparation using the preparation system chosen. This third sequence also gets passed two parameters to allow events to be controlled relative to the sample and port being run. The first parameter (%0) is the port ID and the second is a flag which is set true if the current sample is the final one in the autorun. Using this flag users have control over actions performed at the end of the autorun (e.g. modifying the pump sequence to leave the preparation system pumping after the sample run).

The fourth sequence takes the sample into the dual inlet and balances ion beams prior to data acquisition.

Following data acquisition, the fifth sequence pumps away the old sample, and the sixth sequence will pump out the preparation system.

# Text Files

The appearance of Mimic Diagrams on your screen is controlled by a set of files found in the

**C:\ISOTECH\DI\SETUP\ directory**

All these files have names of the form *.TXT.

## Adprep.Txt

This file contains a list of the mimic diagram files to be used on your system. In the example below, three mimic diagrams are defined.

dualinlt.txt 260 0 0 290 165
10man.txt  259 290 0 170 165
isoprep.txt 259 290 165 330 165

Each line has the form:

**<File name> <Icon Number> <xstart> <ystart> <xwidth> <yheight>**

**Filename** is a file of the form *.txt which contains the graphical details of the mimic diagram (e.g. 10man.txt is the 10 sample manifold mimic diagram).

**Icon Number** is a number (260 or 259) which defines the icon which will appear on screen when the mimic diagram is minimised.

**Co-ordinates** define the position and size of each mimic diagram window on screen. The bottom left-hand corner of the screen is defined as position X=0, Y=0. The co-ordinates $x_1$, $y_1$ thus give the <u>position</u> of the bottom left hand corner of the mimic diagram with repeat 0, 0. Co-ordinates $X_2$, $Y_1$ then give the <u>size</u> of the window, by defining the width and height respectively.

So the line for example:

**dualinlt.txt 260 0 0 290 160**

defines a mimic diagram which starts in the bottom left hand corner, is 290 pixels wide, and 160 pixels high.

By defining these 4 co-ordinates carefully, mimic diagram windows can be made to join together. Thus ADPPEP.TXT has a line which describes the position and size of each mimic which appears on screen, and the name of the file which contains the detailed information needed to draw within the window.

## Mimic Diagrams (*.TXT)

These contain details of the icons for valves, bellows, pipework, thermocouples, pressure gauges, etc., which appear on screen.

The Dual Inlet example below demonstrates the principles involved.

Dual Inlet

Mnemonic L0 Valve 20 1000 Vert

Mnemonic L1 Valve 20 1020 Vert

Mnemonic L2 Valve 20 1040 Vert

Mnemonic L3 Valve 20 1060 Vert

Mnemonic RP Valve 20 44 Vert

Mnemonic RF Valve 20 106 Vert

Mnemonic LV Valve 80 20 Vert

Mnemonic RI Valve 10 75 Horz

Mnemonic RM Valve 100 106 Vert

Mnemonic RV Valve 50 106 Vert

Mnemonic RC Valve 113 85 Horz

Mnemonic RW Valve 113 55 Horz

Mnemonic SP Valve 234 44 Vert

Mnemonic SF Valve 234 106 Vert

Mnemonic HV Valve 185 20 Vert

Mnemonic SI Valve 248 75 Horz

Mnemonic SM Valve 151 106 Vert

Mnemonic SV Valve 204 106 Vert

Mnemonic SC Valve 139 85 Horz

Mnemonic SW Valve 139 55 Horz

Mnemonic SB Bellows 203 70 0 2400

Mnemonic SR Bellows 203 300 10 200

Mnemonic RB Bellows 49 70 0 3400

Mnemonic RR Bellows 49 300 10 200

Mnemonic T1 ColdFinger 174 86 Vert

Mnemonic XX ColdFinger 80 86 Vert

Mnemonic P2 Gauge 60 20 Vert

Mnemonic DI Gauge 265 125 Vert

Mnemonic HM Valve 300 300 Vert

Mnemonic J6 Valve 300 300 Vert

Pipe 00 30 5 83 8

Pipe 00 64 5 67 20

Pipe 00 83 5 86 20

Pipe 01 23 35 237 38

Pipe 01 23 35 26 44

Pipe 01 83 29 86 38

Pipe 01 237 35 240 44

Pipe 01 188 29 191 35

Pipe 02 23 53 26 106

Pipe 02 19 78 23 81

Pipe 03 23 115 26 122

Pipe 03 23 119 106 122

Pipe 03 53 115 56 122

Pipe 03 84 95 87 122

Pipe 03 103 115 106 122

Pipe 04 53 81 56 106

Pipe 05 103 58 106 106

Pipe 05 103 58 113 61

Pipe 05 103 88 113 91

Pipe 06 122 88 139 91

Pipe 06 129 91 132 100

Pipe 07 122 58 139 61

Pipe 07 129 49 132 58

Pipe 08 237 53 240 106

Pipe 08 237 78 249 81

Pipe 09 237 115 240 122

Pipe 09 157 119 240 122

Pipe 09 207 115 210 122

Pipe 09 178 95 181 122

Pipe 09 154 115 157 122

Pipe 10 207 81 210 106

Pipe 11 154 58 157 106

Pipe 11 147 58 157 61

Pipe 11 147 88 157 91

Pipe 12 256 78 269 81

Pipe 12 269 78 272 125

Pipe 12 275 129 285 132

Pipe 13 188 5 191 20

Pipe 13 188 5 250 8

Pipe 14 5 78 10 81•

The first line is text, which appears on the Title Bar of the mimic diagram window. This is followed by a line which describes each icon to be drawn in the window, with each icon corresponding to a mnemonic (which must be a valid mnemonic for your system). Three types are available, illustrated below in example lines.

**Mnemonic RP Valve 20 44 Vert**

Tells the system to draw a valve icon at co-ordinates x=20, y=44 with respect to the 0, 0 corner of the mimic diagram window. This will correspond to Mnemonic RP, and will be labelled as such on the window.

**Note:** Valves can be shown in either vertical 'Vert', or horizontal 'Horz' orientation, to better illustrate the connectivity of the system.

**Mnemonic SB Bellows 203 70 0 2400**

Tells the system to draw a bellows icon at x=203, y=70, representing SB. The limits of the motor are defined as 0 to 2400.

**Mnemonic T1 Coldfinger 174 86 Vert**

Tells the system to draw a coldfinger icon at x=174, y=86. Its temperature, which will be shown in the window, is monitored by thermocouple T1.

**PIPE n $x_1$ $y_1$ $x_2$ $y_2$**

Icons drawn in the mimic diagram window can be shown connected by lines of varying thickness. The command in the *.TXT file takes the form:

The number n is not currently used. The co-ordinate pairs $x_1$ $y_1$ and $x_2$ $y_2$ define a rectangular area of pixels with respect to the 0, 0 position of the window. This will be filled in grey to form a pipe or connection on the finished screen display.

# User Menus

It is possible to add your own commands and procedures (written as *.SEQ files) to certain of the pull down menus.

The files **C:\ISOTECH\DI\SETUP\USERMENU.TXT** contains a list of editable commands on the Inlet, Mass Spec, and Prep menus. An example is shown below. The file contains one line for each added menu command.

Each line has the following format:

**Text : number : *.SEQ : optional parameters :**

**Text** is the text which will appear on the pull down menu. If a character is preceded by a tilde (~), then that character will appear underlined, and can be used for keyboard operation via the "alt" key. Note that you may write the text in a foreign language if you wish.

**Number** is a key to the menu on which the command will appear. Valid selections are

 2 -      Inlet menu
35 -      Prep menu
 7 -      Mass Spec menu

**\*.SEQ** is the name of a valid sequence file in the C:\ISOTECH\DI\SEQUENCE directory. This sequence will be executed when the menu command is selected.

**Optional Parameters** are added if the named *.SEQ file expects to receive parameters, they should be placed here, separated by either spaces, or commas.

**Note:** If no parameters are passed, the line has the form.

**Text ; number ; *.SEQ;;**

# Using CSV Format Files

The **Datalogger** facility in the software suite stores raw results information in files where each data item is separated by a comma. This gives rise to the term CSV (Comma Separated Variables). Such files can be imported into third party suites for further processing.

The details of how to read in such files will vary from one spreadsheet package to another. The example below describes how to import a file into Microsoft Excel 5.0 running under Windows.

From the File menu, choose the open command.
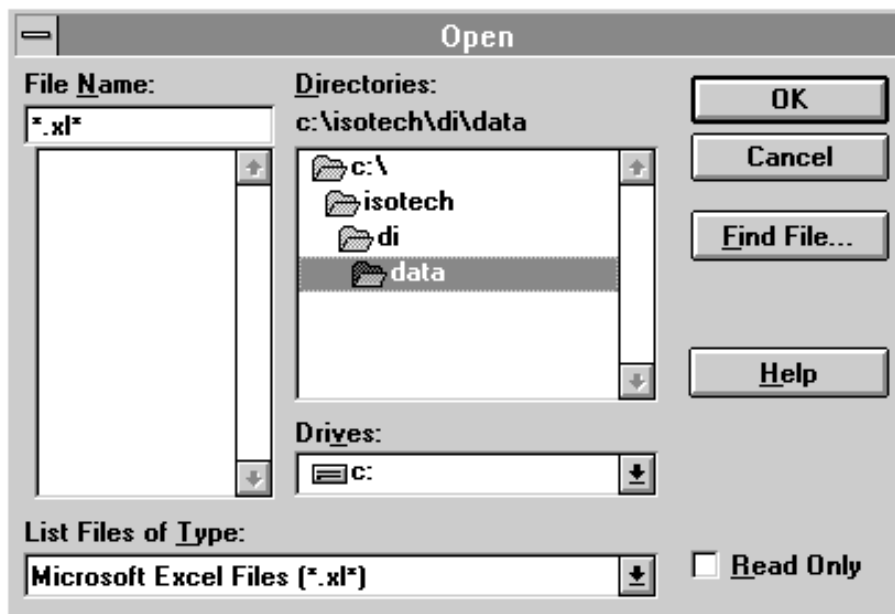


Set the directory to **C:\ISOTECH\DI\DATA**

From the 'List Files of Type' box, choose the Text Files options.

Click on the file name, then OK.

The file will now be imported into Excel.

**Note:** Because the software is multitasking, you can use the spread sheet programs while the VG PRISM is carrying out analytical work, maximising your productivity.

# Compressing and Archiving Data

If you are using the Datalogger system to store your raw data, it is good practice to copy your files onto media which can be stored in a safe place. In the Dual Inlet mode of operation on the PRISM the amounts of data generated are not large, so that compression of the data is not necessary.

Files may be copied from the C:\ISOTECH\DI\DATA directory onto the a:\ drive using either the Drives program from the OS/2 system folder, or perhaps more easily using the copy command from an OS/2 Window e.g.:

**COPY C:\ISOTECH\DI\DATA\\*.TXT A:**

would copy all *.TXT files into the current directory on the disk in drive A.

However should you wish to compress your data we would recommend you use a more sophisticated compression package such as 'PKZIP' or 'LHA'.

# Programming The PRISM

One of the unique features of the PRISM suite is the incorporation of a powerful programming facility. This enables you to completely program the operation of sample preparation systems, the dual inlet, control valves, stepper motors, read all analogue outputs from the instrument (including ion beams) , and to set all digital registers available via the mnemonics ( e.g. retuning the ion source from a program). The language includes all arithmetic operations, real ,integer and logical variable types, conditional statements, IF's GOTO commands, labels, subroutine calls etc.

In short the objective is to provide all the flexibility of an interpreted language like BASIC whilst retaining all the power and facilities of the operating system and the compiled source code.

## Program Language Syntax

### Commands

Commands available are :

**OPEN** mnemonic

Opens the valve identified by mnemonic

**CLOSE** mnemonic

Closes the valve identified by mnemonic

**START** mnemonic

Turns on the switch identified by mnemonic

**STOP** mnemonic

Turns off the switch identified by mnemonic

**ANOUT** mnemonic,value

Sets the specified analog output to the desired value

**STEPOUT** mnemonic,value

Tells the stepper motor identified by mnemonic to move to position identified by value

### Read Statements

Read Statements available are :

**READBEAM** mnemonic

- Reads the specified ion beam, the mnemonics are:

- J0  Major
  J1  Minor 1
  J2  Minor 2
  J3  Minor 3

  **READMOTOR** mnemonic, variable

- Reads the position of the stepper motor identified by mnemonic and stores the result in variable.

  **READVALVE** mnemonic, variable

- Reads the state of the valve and stores the result in the variable (Boolean)

  **DIGIN** mnemonic,variable

- Reads the digital signal identified by mnemonic and stores the result in variable

  **ANIN** mnemonic, variable

- Reads the analog signal identified by mnemonic and stores the result in variable (which must be real).

- For example:

- ANIN T1,FINGERTEMP

- Reads thermocouple 1 (the cold finger) and stores the result in FINGERTEMP

## Declarations

Declarations available are:

**INTEGER** list of variables

- Declares a list of variables as of integer type: an integer can be in the range -2,147,483,648  to +2,147,483,647

  **REAL** list of variables

- Declares a list of variables as of real type. A real can be in the range +/- 2.3E-308 to +/-1.7E308 with 15 digits of precision.

  **BOOLEAN** list of variables

- Declares a list of variables as of Boolean type (i.e. Logical) type. A BOOLEAN variable may adopt the value TRUE or FALSE

  **FLAG** list of variables

- Declares the variables as GLOBAL booleans. This means that they will be valid in all programs which are running concurrently.

  **STRING** list of variables

- Declares a list of variables as of string (i.e. text/character) type. STRING variables may be up to 80 characters long.

## String Handling Commands

**APPEND** StringVariable StringVariable/String

- Appends the second parameter to the first string.

**PREPEND** StringVariable StringVariable/String

- Prepends the second parameter to the first string.

**COMPARE** BooleanResultVariable StringVariable StringVariable/String

- Compares the first string to the second and if they are the same then returns true.

**SLICE** StringVariable StartPosition Length StringVariable/String

- Copies the portion of the last string from the 'start position' for the next 'length' characters into the first string.

**VALSTR** String ASCIIValue

- Converts the ASCII value into a character and returns it as a string.

- **Note:** ASCII codes can be used in String commands APPEND, PREPEND and ASSIGN by preceding the number with the @ symbol (e.g. ASSIGN Cr @10 would put the ASCII text for the character 10 into the string Cr).

**STRVAL** ASCIIValue StringVariable/String

- Returns the ASCII value of the first character in the string.

## Assignment Statements

Assignment Statements available are:

**ASSIGN** variable,value

- Sets variable to value

**ADD** variable, value1, value2

- Adds value2 to value1 and stores the result in variable

**SUBTRACT** variable,value1, value2

- Subtracts value2 from value1 and stores the result in variable

**MULTIPLY** variable,value1, value2

- Multiplies value1 by value2 and stores the result in variable

**DIVIDE** variable, value1, value2

- Divides value1 by value2 and stores the result in variable

**OR** Variable Boolean1 Boolean2

- Performs a logical OR on the 2 booleans and stores the result in the variable

**NOT** Variable Boolean1 Boolean2

- Performs a logical NOT on the booleans and stores the result in the variable

**AND** Variable Boolean1 Boolean2

- Performs a logical AND on the booleans and stores the result in the variable

## Program Flow Control

Program Flow Controls available are:

**IF** value1, conditional, value2, label

- Compares value2 to value1 ; if the conditional operator (=, >, < ) is satisfied, the statement is TRUE, and execution will proceed at the line following "label". Otherwise execution will proceed at the next line.

**:label-name**

- A valid label name is an alphanumeric string preceded by a colon. Label-names are referred to by GOTO and IF statements.

**GOTO** label

- Execution proceeds from the line after the label referred to.

**EXIT** Return Value

- Ends the program, returning the real variable Return Value to the calling program. The last line of the program must be an EXIT .

**CALL** return variable SEQUENCE FILENAME <parameters>

- Starts execution of the program specified by SEQUENCE FILENAME. Any required parameters must be passed. The execution of the calling program is suspended until the called program executes an EXIT statement. The Return value of the EXIT statement is stored by the calling program in return variable.

**SPAWN** SEQUENCE FILENAME <parameters>

- Starts execution of the program specified by SEQUENCE FILENAME . Any required parameters must be passed. The execution of the Spawning program continues, so that the two programs run in parallel. No return variables are possible.

**WAIT** value

- Waits for value seconds before proceeding with the program. Value may take the form of a suitable variable name.

## Input/Output

Input/Output available are:

**MESSAGE** colournumber String

- Sends a text message to the message window. The message is shown in the specified colour. String contains the text to be output. The contents of a variable can be output by specifying the variable name preceded by a $ (e.g. MESSAGE -1 Value is $Val would output the contents of a variable Val).

- **Note**: Upto five variables can be used in one message.

- The main colour numbers are:

  |        |           |              |
  |--------|-----------|--------------|
  | -1: Black | 4: Green | 9: Dark blue |
  | 1: Blue | 5: Cyan | 10: Dark red |
  | 2: Red | 6: Yellow | |
  | 3: Pink | 8: Grey | |

- By convention, red should be reserved for error or warning messages.

**QUESTION** Answer_Variable String

- Opens a question dialog box on the screen. String contains a prompt to the user which requires a yes/no response. If the response is Yes, the Boolean answer_variable is set to TRUE, if no it is set to FALSE. This enables the user to control the flow of execution of the program.

## Communication with Communication (Com) Ports

**SETUP** Comport BaudRate Parity DataBits Stopbits SoftwareHandshaking HardwareHandshaking

- Opens the communication port with the specified settings

**PUTPORT** ErrorVariable PortMessage

- Writes one character to the communication port:

**READPORT** ErrorVarible Port Message

- Reads a string from the communication port. This will read until the terminator string is returned.

**GETPORT** ErrorVarible Port Message

- Writes one character to the communication port then read one character immediately.

**WRITEPORT** ErrorVarible Port Message

- Writes a string from the communication port.

**READPORTFILEIN** ErrorVariable Port FileName

- Reads text from the communication port and writes it out to the file C:\ISOTECH\DI\FileName.PRT. It will keep reading until the terminator is received.

**WRITEPORTFILEOUT** ErrorVariable Port FileName

- Reads the text file C:\ISOTECH\DI\SETUP\FileName.PRT and writes it to the communication port.

**Errors**

- The values returned as errors from the communication port are as follows:

- 0 - OK
  1 - Unknown Error
  2 - Cannot read from or write to the port

## Communication Routine Parameters

**Maxresp**

- Is an INTEGER with a default of 50 characters to read (Note: 0 will give as many characters as needed).

**Timeout**

- Is an INTEGER with a default of 10000 milli-seconds to wait for a response.

**WaitTime**

- Is an INTEGER with a default of 100 milli-seconds to wait before re-trying the communication port.

**Retry**

- Is an INTEGER with a default of 1 times to re-try writing to the communication port.

**RdTerm**

- Is an STRING with a default value of CrLF. The communication port will be read until the read terminator is received.

**WrTerm**

- Is an STRING with a default value of CrLF. The communication port will be written to until the write terminator is sent.

If any other settings are required declare the variable of the correct type and assign the required value.

**Note:** This must be done in each sequence using the communication port commands. If any variables are undeclared the default value is used.

## Miscellaneous

Miscellaneous commands available are:

**JUMP** value HTJump PeakCentre

- Jumps to the mass specified by value, using HT if HTJump is set TRUE or by Magnet if HTJump set FALSE. If PeakCentre is set TRUE it will perform a peak centre after the jump.

**LOAD** Tfname 255

- Retunes the ion source by loading the specified tuning file. The 255 is a bit set value which specifies which of the source parameters to load (e.g. 1 = HT only, 4 = Half Plates only, etc.).

**Mnemonics**

- Mnemonic 2 letter identifiers for valves switches etc.

**string**

- Any set of alpha-numeric characters.

**Variable Names**

- Variable Names consist of a string , started by a letter, of between 3 and 12 characters. The names are case sensitive.

- FINGtemp and FingTEMP are different variables.

- Before use, a variable name must be declared in a REAL, INTEGER, or BOOLEAN declaration statement.

**Parameters**

- A parameter is identified by a % symbols

- e.g.   WAIT %0

- Will use the value passed in to the sequence as the first parameter.

**Comments**

- A line is treated as a comment if its first character is %

**IMPORT** variable name, "*.PRM","Text"

- Will read the value of the parameter identified by "Text" from the parameter file *.PRM, and store the result in variable name. This command thus enables sequences to import values from parameter files.

- **Note:** If, and only if, a variable is imported from a parameter file, it <u>does not</u> need to be declared in an BOOLEAN, REAL, INTEGER etc. statement. This is because the parameter file will define the variable type.

**EXPORT** variable name, "*.PRM", "Text"

- Will write the value of variable name to the file *.PRM, stored on the new value of the parameter identified by "Text".

- **Note:** variable <u>must</u> be of the <u>same type</u> as that defined in the parameter file.

    **PARK** mnemonic

- Where mnemonic is a valid stepper motor, this command steps the motor device back to its zero reset position, as controlled by the appropriate expanded position.

    **CONTROL TEMP** Mnemonic Setpoint Duration.

- This will allow the device identified by Mnemonic to be controlled at Setpoint (°C) temperature for the specified duration (minutes).

    **CONTROL TEMP STATUS** Mnemonic Status Variable name

- This will query the status of the temperature device identified by Mnemonic. Status values retuned are

- -998          - no control in progress
  -999          - device tripped
  Other value    - temperature reading (°C)

- and will be stored in the <u>Integer</u> variable given as status variable name.

    **ABORT CONTROL TEMP** Mnemonic

- This will abort a temperature control operation.

# An Example Program

```
% An example program
% This program illustrates some of the features available
% It monitors the pumpout of the reference gas by measuring the major beam
intensity. If it does not fall sufficiently in a given time, the user is asked if they
want to try again
% Declare variables
INTEGER Pumptime,Counter
REAL  Majorbeam,Allgone
BOOLEAN Answer

% Initialise variables
ASSIGN Pumptime, 60
ASSIGN Allgone 1.0E-12
ASSIGN Counter,0

% Set valves to pump out reference
OPEN LV
OPEN RP
OPEN RF
OPEN RV
OPEN RM

% Set changeovervalve to reference
CALL Dummy SETCHOV.SEQ TRUE

:Pumpout
ASSIGN Counter,0

:Loop
WAIT 5

% Read Majorbeam
ANIN J0, Majorbeam

% Increment the loop counter
ADD Counter,Counter,5
IF Counter < Pumptime Loop

% has the gas been pumped out?
IF Majorbeam < Allgone Finish

% The gas has not all been evacuated
QUESTION Answer Pumpdown too slow -Do you want to continue?
IF Answer = FALSE Quit

% The user wants to retry the pumpout
GOTO Pumpout

:Quit
MESSAGE -1 Program Aborted
GOTO END

:OK
MESSAGE -1 Reference evacuated OK

:END

% Close all valves
SPAWN CLOSEDIV.SEQ
EXIT 0
```

## Good Practice When Programming

Make the first line of a program a comment which identifies the author, date of revision and purpose of the program

Declare all variables at the start of the program

Use self- explanatory variable names

Use frequent comments.

Break programs down into subroutines using labels and comments, and use CALL, rather than writing complex programs that are hard to follow and may be too lengthy.

Use integer variables and arithmetic where the precision of real variables is not absolutely needed.

Take back ups of your program on floppy disc.

## Running a Program

Choose the PROGRAM, RUN option from the main menu bars

Select the program to run from the dialog box which appears: remember to enter any necessary parameters using the parameter entry field.

Press OK or Return to start the program.

## Editing a Program

You can enter a program on your VG PRISM data system by using the OS/2 System editor, which is a powerful text processor. It has extensive search and replace features and on screen editing facilities, such as cut and paste, which are similar to those provided by any Text processing software. Fonts and text colours are also selectable. A detailed manual for this editor is provided with your system.
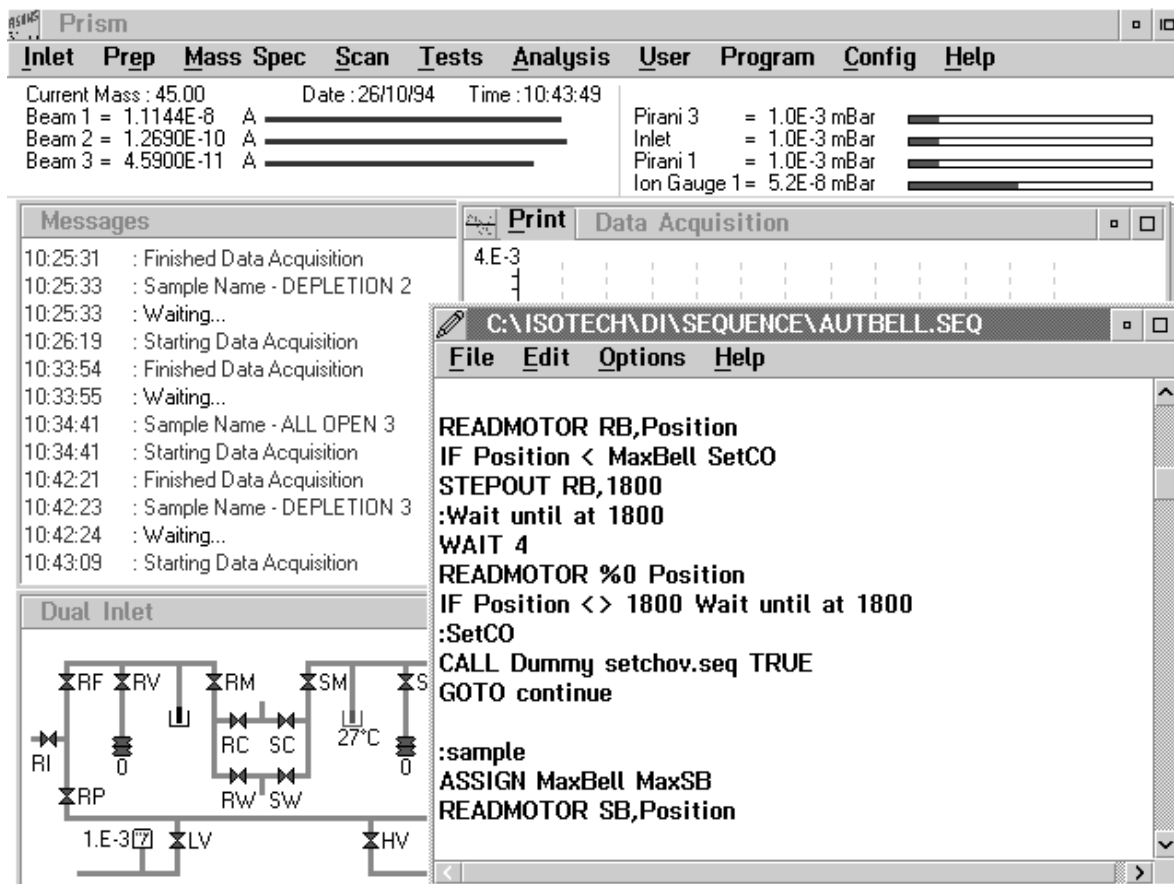


**Note:** Because the software is multitasking, you can write your programs while the VG PRISM is carrying out analytical work, maximising your productivity. However be careful not to modify sequences which are currently running.

## Testing a Program

The best way to test a program is to run it first using the off-line software which is provided with your system . Again, your productivity is maximised, and you can test your programs without risking damage to your instrument, however sequence commands setting the bellows, motor positions, etc. will obviously not drive the specified devices while running off-line.

# Section 8



# Maintenance and Fault Finding

# Introduction

This section of the manual will give details of the routine maintenance and fault finding of the PRISM and Dual Inlet. It is important that routine maintenance is carried out on a regular basis, this will improve the reliability of your instrument and in turn the affect the accuracy of your sample running. Two things to bear in mind before attempting any maintenance or fault finding:

Read the manufacturers guides supplied for the details of routine maintenance for the components of the system e.g. Rotary pumps, Turbomolecular pumps, Air Compressors, etc.

Fault finding and system repairs should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

# Useful Tools

A basic set of tools is described below which should be kept on hand to perform the routine maintenance tasks described in this section of the manual.

Set of metric Allen keys (hexagon keys)
Pair of tweezers
Lint-free gloves (dental gloves are ideal i.e. non-powdered)
A digital multimeter ('AVO')
Medium size screwdriver (straight head)
Medium size screwdriver (cross head / Philips head)
Adjustable spanner (Monkey/Crescent Wrench)
Set of Jeweller's screwdrivers
Mini-spanner / wrench set
Scalpel / sharp knife

# Emergency fault conditions

WARNING

Before returning the power to the system ensure the reason for the shutdown is corrected.

# Emergency Instrument Shutdown

In exceptional circumstances (such as fire or exposure to hazardous voltages) it may be necessary to shut down the instrument quickly. If such a situation arises, the instrument should be isolated from the electrical supply, as close to the supply as possible:

Isolate the supply at the distribution box which connects the instrument to the site supply, if this can be done quickly. This will usually mean operating the isolation lever on the outside of the box.

Switch off the two (pumping and electronics) circuit breakers on the electrical service panel, then unplug the mains inlet cables situated below them.

Disconnect the power cables of any peripherals connected to the instrument (such as sample preparation systems).

When there is no further risk to personnel:

Close the rotary pump isolation valves.

Turn off the power switches to the PC, Monitor and Printer.

Turn off all the power switches on the Utility Unit rear panel.

# Electrical power failure

WARNING

Repair the Electrical fault before returning the power to the system.

If the power supply to the instrument fails for any reason (e.g. during an electrical storm) the following procedure should be followed:

Close the rotary pump isolation valves.

Switch off the two (pumping and electronics) circuit breakers on the electrical service panel.

Turn off the power switches to the PC, Monitor and Printer.

Turn off all the power switches on the Utility Unit rear panel.

When the failure has been rectified:

Follow the standard power-up procedure for the Pumps/Electronics

# Other supplies failure

## Compressed Air

WARNING

Care must be taken when dealing with compressed air to avoid injury from loose fitting.

If the compressed air fails the valves on the Dual Inlet (and the magnet movement for the Hydrogen magnet if fitted) will fail to operate. This will cause some of the samples to be lost, however the number will only be small, because of the 'Maximum Consecutive Bad Samples' parameter is set relatively low.

The reason for the compressed air failure should then be located and rectified. This could include:

Check the air compressor (see manufacturers guide).

Check air lines.

Check for blockages.

Check the filter on the mass spectrometer has been drained.

## Liquid Nitrogen

WARNING

Care must be taken when handling Liquid Nitrogen.

If the liquid Nitrogen fails then this is normally indicated by the software. This halts the sample run until the fault has been rectified. This could include:

No liquid Nitrogen.

Blocked or missing Nitrogen lines.

Fittings loose or missing from the cold finger or trap.

Level set too low in the trap dewar.

## Special Gases (Continuous Flow only)

Please see sections on the continuous flow products.

# Routine Maintenance

CAUTION

Read the manufacturers guides supplied for the details of routine maintenance for the components of the system e.g. Rotary pumps, Turbomolecular pumps, Air Compressors, etc.

## Overview

Many of the problems associated with poor instrument performance can be stopped with regular and effective maintenance. It cannot be stressed too strongly that two days service every six months will save time and increase sample throughput in the long term.

The main aim of routine maintenance is to therefore rectify potential problems before they affect the accuracy of the instrument, so in this section we concentrate on vacuum cleanliness, inlet performance and overall instrument noise levels.

## Planned maintenance

To break this down further the following procedures should be carried out every **six months,** unless specified differently in their own manufacturers manual:-

### Change the Rotary Pump Oil

The rotary pumps are a mechanical pump and as such the force of their operation slowly breaks down the oil which in turn reduces their pumping efficiency. New oil is a light brown colour, while old oil varies from dark brown to black. Oil which is cream coloured has been pumping a lot of water and the source of this water must be identified before resuming sample analysis. Running with old oil for extended periods of time will contaminate the analyser and inlet. (Please remember to ballast new oil as per the manufacturers manuals.)

CAUTION

Follow the Maintenance plan in the manufacturers Rotary Pump manual supplied. Also supplied in the Rotary Pump manual are details of the procedure.

### Change the Molecular Sieve in the Foreline Traps

The foreline traps fitted to the rotary pumps contain molecular sieve, which traps oil vapour from the rotary pumps and prevents it from contaminating the system, so to ignore this procedure will lead to a build up of hydrocarbons in the analyser and inlet.

CAUTION

Follow the Maintenance plan in the manufacturers Foreline Trap manual supplied. Also supplied in the Foreline Trap manual are details of the procedure.

## Change Oil Mist Filter Elements

While this does not necessarily effect the performance of the instrument, changing the Oil Mist filters ensures that the environment that the system operates in, is kept as safe as possible for the user.

CAUTION

Follow the Maintenance plan in the manufacturers Oil Mist Filter manual supplied. Also supplied in the Oil Mist Filter manual are details of the procedure.

## Bakeout the Analyser, Inlet and Preparation Lines

No matter how clean and careful the preparation of samples, there will be a gradual build up of water, hydrocarbons and other contaminants in the instrument. In order to remove these we must bake the system, failure to do so will result in poor performance and increased memory between samples. Baking simply vaporises the volatile contaminants and so allows them to be pumped away.

CAUTION

Follow the Bakeout procedures given earlier in this manual for the Analyser and Inlet Bakeout, and for the Preparation Lines follow the procedures given in the relevant sections relating to the Preparation Line fitted to your system.

## Check the Vacuum Integrity

By running a background scan to cover the masses 26 to 50 we can check on the cleanliness of the vacuum and the presence of any leaks to atmosphere. If there are large numbers of hydrocarbons, the system will probably require baking. A leak is indicated by a nitrogen to oxygen peak ratio of 4:1 and an oxygen peak very much greater than the argon peak, in which case it should be identified and cured. Failure to obtain a clean background vacuum will give rise to problems obtaining a good peak shape, good precision and low memory.

CAUTION

Follow the Leak Checking procedures given in this manual.

## Check the Cross-Seat Leakage of all Inlet and Preparation Line Valves

The technique of gas stable isotope analysis relies on constant and consistent reference and sample gas flows. To this end it is imperative that each valve that the gas comes in contact with opens and closes correctly every time it is operated. A valve which is fractionally open will cause fractionation of the gas and subsequent errors in the results, while some valves may cause complete loss of the sample, e.g. a leaking pump valve. It is important that the changeover valve be included in any investigation of inlet cross-seat leakage, failure in this area will lead to a reduction in the measured delta between two isotopically different gases. Other inlet valve problems can show up in the zero enrichment run, while prep line problems must be investigated by the transportation of gas around the line.

CAUTION

Follow the Leak Checking procedures given in this manual.

## Optimise the Peak Shapes

By comparing sample and reference ratios we can reduce many of the errors involved with peak height measurement. However, in order that variations in the accelerating potential and magnet field do not affect the precision of the results it is necessary to have a flat area over which to measure the ratio. It is important to realise that there are many other causes of poor peak shape as well as the obvious one of bad magnet position and they should be investigated prior to moving the magnet.

CAUTION

Follow the Peak Shaping procedures given in this manual.

## Balance the Capillaries

In the comparison of sample with reference there should be equal flows of both gases through the capillaries into the source, such that the pressure of both gases are equal. This is achieved by the use of variable crimps on each capillary to give identical major beam heights. Poor capillary balance gives poor zero enrichment. A high value for the zero enrichment delta leads to poor accuracy in the measurement of real samples.

CAUTION

Follow the capillary adjusting procedures given in this manual.

## Check Noise Levels with Zero Enrichment Data Runs

During normal operation the instrument measures ratios of gas stable isotopes, for those measurements to be realistic, the output from the data collection system must be as noise free as possible. A zero enrichment is used so that confusion due to gas effects are reduced to a minimum. The source of the noise must then be determined and cured before attempting to run gas samples.

See the Operation section of this manual for details of running zero enrichment samples.

## Compressor (if supplied) Maintenance

The performance of your instrument relies on the smooth running of the valves on the inlet. These are mostly pneumatically operated, so the continued performance of the Air Compressor and air lines is of great importance. The Air Compressor (if supplied with your instrument) should therefore be maintained in accordance with its manufacturers guide supplied with it. This involves checking the system **two or three times weekly**.

## Compressed Air Water Trap Drainage

The Compressed Air Water Trap is fitted as standard on the system and should be checked and drained **two or three times weekly**. Failure to do this may cause damage to occur to the Predyne valves used to operate the Dual Inlet valves. The procedure is simply to press the small drain button under the filter ('glass' section). This will produce a hissing sound as the air escapes and any liquid in the filter will be expelled (a cloth or similar is advised).

# Fault Finding

CAUTION

Fault finding should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

## Overview

This section of the manual covers fault finding on the PRISM Mass Spectrometer with Dual Inlet, for information on the preparation systems please refer to the sections of this manual appropriate to that system.

If you experience any difficulties that this section of the manual does not cover or you have any problems with your instrument please contact the Customer Service Department at Micromass or your local Micromass representative.

## Computer System Faults

### Computer

Please refer to the manufacturers manual for details.

### Monitor

Please refer to the manufacturers manual for details.

### Printer

Please refer to the manufacturers manual for details.

## Vacuum problems

If a vacuum system fails to reach the required pressures, there are a number of potential causes:

### Cleanliness :

Surfaces outgassing.  Fingerprints, hairs etc.

### Solution :

Clean and bake.

### Leak:

Hole in the vacuum envelope. If large it may be detected using acetone. If small use a helium leak checker or a mass spectrometer tuned to a suitable mass.

To tackle any vacuum problem it is first necessary to identify the best level of vacuum we can obtain, using the gauges at our disposal. Below gives various levels of vacuum seen on the system and some of the probable causes. It then goes on to describe some of the methods that can be employed in detection:

## Pirani gauge at atmosphere

The parts of the system to be checked are:

Check operation of the rotary pumps.

Check operation of the rotary pump isolation valves.

Check operation of the Pirani gauges.

Check for very large leaks.

Check for Vent Valve leakage.

On the PRISM with Dual Inlet there are three rotary pumps and three Pirani gauges, it is possible to swap them around to find if one of them is not working. Also it is possible to check the operation of a pump and integrity of any backing line pipework by putting one's hand over the open end of a piece of pipe and switching on briefly, (this is perfectly safe and painless). If the problem is a leak and the pressure is this high, it will be possible to hear the gross leak by the sound of the air being sucked into it.

CAUTION

NEVER use water for any leak checking test.

NEVER use acetone on viton seals.

**Note:** Pirani gauges are contaminated by large quantities of $CO_2$ so always allow plenty of time, up to 10 minutes, to pump away this gas.

## Pirani gauge between atmosphere and $10^{-3}$mBar

The parts of the system to be checked are:

Check for leaks with acetone (gold and copper seals only).

Check for Vent Valve leakage.

Check the Foreline traps are not saturated and require new molecular sieve.

Check both Piranis in the same pump system.

When leak checking with acetone at any given vacuum level, be prepared for either a rise or fall in pressure, as the liquid may block the hole depending on the size of leak. Always allow plenty of time for the pumps to pull down the vacuum before assuming that there is a leak; it can take up to fifteen minutes for the Pirani gauge to reach $10^{-3}$mBar. When a leak has been discovered try to tighten it out by evenly tightening the bolts holding the joint together, if this is not possible the gasket will have to be changed.

CAUTION

NEVER use acetone on viton seals (e.g. Housing lids, etc.).

## Pirani gauge at 10$^{-3}$ mBar but ion gauge will not read

The parts of the system to be checked are:

Check for leaks with acetone (gold and copper seals only).

Check operation of Turbomolecular pumps.

Check for Vent Valve leakage / failure.

Check ion gauge filament continuity.

Check ion gauge filament shorts to grid and earth.

If the ion gauge is lit then the ion gauge housing will be warm to the touch so suspect the ion gauge read circuit. Beware when handling the ion gauge connections as the grid circuit is normally 150v. If the ion gauge filament supply is incorrect then the filament will try to light but fail; iridium coated filaments run on 7 volts. Check the filament and cables for both short and open circuits, if one filament has blown check that it is not touching the grid and impending the operation of the other one. The gauge head must always be inserted such that the filaments are below the grid and so will sag away from the grid when they get hot.

CAUTION

NEVER use acetone on viton seals (e.g. Housing lids, etc.).

## Ion gauge between 10$^{-3}$ and 10$^{-7}$ mBar

The parts of the system to be checked are:

Check for leaks with acetone (gold and copper seals only).

Check for Vent Valve leakage / failure.

Check ALL the Turbomolecular pumps are running.

Expect that a clean system will take roughly 30 minutes to reach 10$^{-7}$ mBar from the time the Turbomolecular pumps were switched on. If the system reaches 10$^{-7}$ mBar and stays there, then the system probably requires baking.

CAUTION

NEVER use acetone on viton seals (e.g. Housing lids, etc.).

## Ion gauge reads better than 10$^{-7}$ mBar

The parts of the system to be checked are:

Check the Backgrounds by running a background scan.

Check for leaks with acetone (gold and copper seals only).

Leak check with helium or argon (see section on Leak Checking below).

If a vacuum problem is still suspected at this level after baking the system, then to identify the reason a background scan including peaks 26 to 50 should be run. The ratio of nitrogen to oxygen in air is approximately 4:1, so a ratio of 28 to 32 measured by peak jumping near to 4:1 will indicate a leak, and the system should then be leak checked using the procedure below. However if the ratio of 28 to 32 is very much greater than this, say 20:1 then mass 28 is most likely carbon monoxide. High non-air peaks indicate a need to bake the system again.

For details of leak checking please see Leak Checking section of this manual below.

CAUTION

NEVER use acetone on viton seals (e.g. Housing lids, etc.).

# Vacuum Pump Fault Finding

## Turbomolecular Pump

Please refer to the manufacturers manual for details.

## Rotary Pump

Please refer to the manufacturers manual for details.

## Vent Valve

Please refer to the manufacturers manual for details.

# Leak checking

There are various sections of the system that can be leak checked using the mass spectrometer, this section of the manual covers these and details the methods.

## Leak Checking the Mass Spectrometer to Atmosphere

To leak check the Analyser and Pumpline (bottled argon available) to atmosphere follow the procedure given below:

Close all inlet valves.

Expose valve RI to atmosphere (open the reference port).

Seal off reference inlet port.

Open valve RI.

Close valve RI.

Open valves RF RV RP SP SF SV and allow the inlet port atmosphere fill the inlet (to reduce the pressure).

**CHECK STATUS OF RI          IT MUST BE CLOSED!!**

Open valves RM SM.

Toggle changeover valve.

Peak jump to mass 40.

Peak centre on mass 40.

The Mass Spectrometer is now set up to measure the mass 40 beam ready for leak checking with Argon.

Pump out the inlet. The magnitude of the mass 40 beam in the centre collector should fall.

Close all inlet valves including RW and SW.

Set up a repeat scan on the centre collector using a y-axis scale based on the mass 40 background peak. A scale of zero to 10 times the background should be suitable. Set the x-axis scale to 5 minutes.

Use the Argon probe provided to check for leaks in the analyser, by spraying the gas on each joint, weld, or feedthrough in turn. When a possible site of a leak has been identified blow away the gas and repeat the exercise.

Check for leaks in the pumping system below the bench.

If a leak is present, then the rate of rise of the of the Argon peak will increase.

After a leak has been found, it should be repaired and the system re-leak checked as above.

**Notes:**

If a leak has been repaired and the system vented, it may be necessary to bake the system before re-leak checking, so as to obtain the same vacuum level.

Helium may be used instead of Argon for leak checking, however it will be necessary to inlet a small aliquot of Helium into the mass spectrometer, to find the helium peak.

To find a leak on the changeover valve waste line, pump out the analyser, spray argon on each joint in turn while operating the valve and look for a rise in the beam height.

## Leak Checking the Dual Inlet to Atmosphere

This uses a similar to the method used above to leak check the mass spectrometer, however mass 28 (Nitrogen) can be used so that no Argon is required. the set up procedure is as above only this time tuning the instrument to mass 28. The leak checking procedure is then given below:

Ensure reference and sample inlet ports are closed.

Pump out the inlet on high vacuum for 5 minutes. All valves including the 4 changeover valves should be open.

Set up repeat scan on the centre collector.

Close HV.

Monitor mass 28.

The rate of rise of the mass 28 peak can then have the following:

**No Increase in mass 28**

No further action required as there is no leak detected.

Increase in mass 28

The procedure to isolate problem area is as follows:

Open HV. Wait till scan shows mass 28 returned to background.

Close RM and SM.

RESULT 1  INCREASE IN MASS 28

Changeover valves and / or capillaries are leaking to atmosphere.

Close RC. If mass 28 falls to background or better then RC is leaking to atmosphere across the diaphragm gold ring seal or diaphragm itself may be split.  If mass 28 falls to some intermediate value then either the reference capillary is leaking or the diaphragm seal across RW is leaking. If mass 28 continues to rise at the same rate then the problem is on the sample side. Close RW. If mass 28 falls to background then the sample side is OK. Open RC. If mass 28 remains at background then RW is faulty. If mass 28 rises then the reference side capillary is leaking.

Repeat this procedure beginning with the closing of SC.

The above procedure assumes for simplicities sake that the leak is confined to one location. If this is not the case then it is worthwhile using the Argon probe as with the mass spectrometer leak checking procedure to locate the leak further.

RESULT 2  NO INCREASE IN MASS 28

Changeover valves and capillaries are leak tight.

Open RM and SM.

RC SC open RW SW closed.

Close RV SV RF SF RP SP LV HV

Open the valves in turn and monitor the response. If there is a rise in mass 28 at any point, then the leak is in that area of the inlet and should be repaired.

## Leak Checking the Inlet Valves for Cross Seat Leakage

CAUTION

Do not carry out any of the following procedures without first reading and understanding them, as air can easily be admitted to the mass spectrometer.

The procedure to test the inlet valves for cross seat leakage is given below:

Pump out the inlet for 5 minutes on high vacuum.

Close all valves.

Expose valve RI to atmosphere.

Seal off reference inlet port.

Open LV.

Toggle changeover valve such that RC is open and RW is closed.

Set up repeat scan on mass 28 as before.

Refer to Figure below
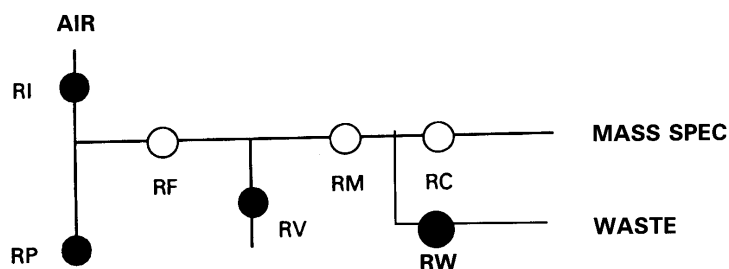


Open RM RF.

Any leak across RI now has the shortest possible route to travel into the mass spectrometer.

Observe the scan. A rise in mass 28 of greater than 5E-13A in 5 minutes signifies a leak.

If a leak is observed, open RP, wait till the background falls and then close both RP and RF.

RF is the next valve to be tested.

Refer to Figure below:

Open RI. Atmosphere is now up against RF.

Close RI.

Observe the scan.
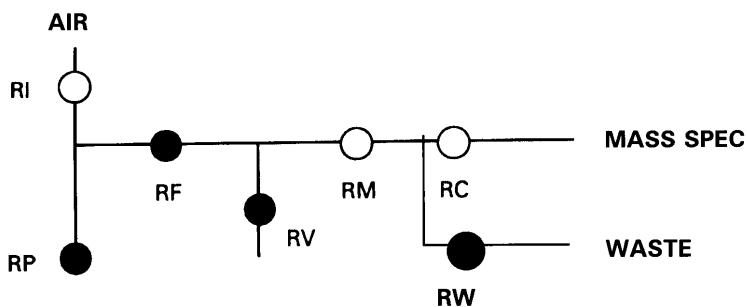
In sequence the remaining valves should be tested in the following order.

RM                  RV     SP     SF     SM     SV     SI
   RP

**Notes:**

In the case of the valves RV or SV, fill the bellows with air by admitting atmosphere into them and then closing RV or SV and pumping away the air left in the inlet for 5 minutes, prior to leak checking using the diagram above for RF as the method.

In the case of RP or SP, air can be admitted up to each side from the inlet valve (SI for RP and RI for SP) before leak checking using the method in the diagram above for RI.

If you suspect a virtual leak (degassing or trapped volume) then pump out the suspected region for 5 minutes and recheck the leak rate.

A leaky valve is considered to be significant when the observed leak rate is greater than 5E-13A in 5 minutes.

## Leak Checking the Changeover Values for Cross Seat Leakage

The procedure to test the changeover valves for cross seat leakage is given below (please refer to the Figures):
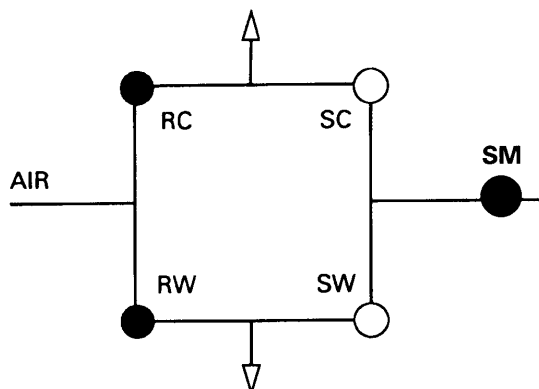


Evacuate the inlet and close all valves.

When checking RC and RW for cross seat leakage, since both valves lead directly to high vacuum BOTH must be closed at the same time when under test.
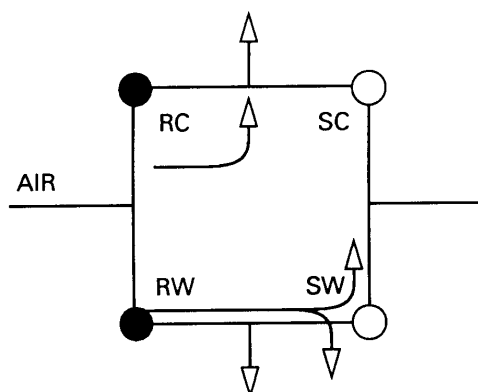


Air will be allowed up to the CLOSED valves RC and RW (see the procedure below):

Set up the changeover valves with RC, RW, SM closed and SC, SW open.

All other inlet valves closed.

From the reference inlet port aliquot some air to RF.

Close RI.

Open RF RM and allow this aliquot of air to reach RC and RW. At this stage the pressure of air is less than atmosphere so damage will be limited even in the worst case scenario of the leak being one of the valves stuck fully open.

Observe mass 28. If it rises sharply then immediately open RP and pump on the capillary. If the rise is slow then close SW to establish which of the valves is leaking. If there is no rise then open RI and allow atmosphere up to RC and RW. Recheck the rise in mass 28.

If RC leaks, a rise in the mass 28 signal will be observed on the mass spectrometer. Rather less obviously a rise will also be observed if RW leaks. Half of the leak will go down the waste Turbomolecular pump line, while the other half will pass through the open valves SW and SC and on into the mass spectrometer leading to a rise in the mass 28 peak.

To distinguish which of the valves RC or RW is leaking simply close SW.

If the rise in mass 28 persists then RC is leaking or if the rise stops then it is RW which is leaking.

CAUTION

Before checking SC and SW in a similar manner great care must be taken in pumping out the air contained in the capillary and changeover valve.

The procedure for this is given below:

Set acceleration voltage to zero.

Close SW.

Pump out reference capillary through the inlet valves LV and HV.

Leave the reference capillary pumping for 5 MINUTES.

Open RW first, wait 1 minute then open RC.

Tune acceleration voltage to mass 28 and WAIT till background is back to normal before attempting to leak check the sample side valves SC and SW.

# No Peaks Found

If no peaks can be found then the following list gives some of the areas to check:

Check the source lead is plugged into the source feedthrough flange.

Check the 'Head Amplifier(s)' is fitted and its power lead and fibre optic leads are correctly plugged in.

Check that the 'DC Supply', 'Auxiliary Mains' and 'Source Mains' switches on the 'Utility Unit' are in the ON position and non of the fuses are tripped.

Check the source is turned on and is tuned for the appropriate gas (check the source settings on the front panel).

Check the magnet is on (use a small screwdriver to check for magnetism) and is over the flight tube in the correct position.

Check that the correct mass for the tuning file has been identified to set the beam mask for the gas (e.g. mass 45 for $CO_2$).

Check if Hydrogen is to be measured that the Hydrogen mode has been enabled and the mass identified as mass 2.

Check the correct gas for the tuning file selected, is in the inlet and the changeover valve is set correctly (ensure the correct amount of gas is available).

Check that the collectors are approximately in the correct position for the gas to be measured, and ensure that peaks cannot be seen on any of the collectors.

Check for short circuits in the source, pin to pin and pin to ground.

Check for short circuits in the collector(s), pin to pin and pin to ground.

If after checking these points you are still having difficulty please contact the Customer Service Department at Micromass or your local Micromass representative.

# Ratio Trace Problems

Problems in the ratio traces can be split into two forms, noisy and systematic.

| **Noisy** | **Systematic** |
|---|---|
| Filament stability | Changeover valve sticking |
| High background | Inlet contamination |
| Source supplies | Inlet valve seat leakage |
| Amplifier stability | Capillary balance |
| Amplifier supply stability | Inlet pressure balance |
| High tension stability | |
| Peak Shape | |
| Suppressor stability | |
| Pumping stability | |
| Connection continuity | |
| Environmental stability | |

| Symptoms Zero Enrichment | Symptoms Enrichment | Cause |
|---|---|---|
| | | Correct |
| | | B, C, D, S, T, U |
| | | A, B, C, D, S, T, U |
| | | E, F, G, J, M |
| | | E, G, H, J, M |
| | | E, F, L |
| | | K, D, Q, R, T, U |
| Or | | N, O |
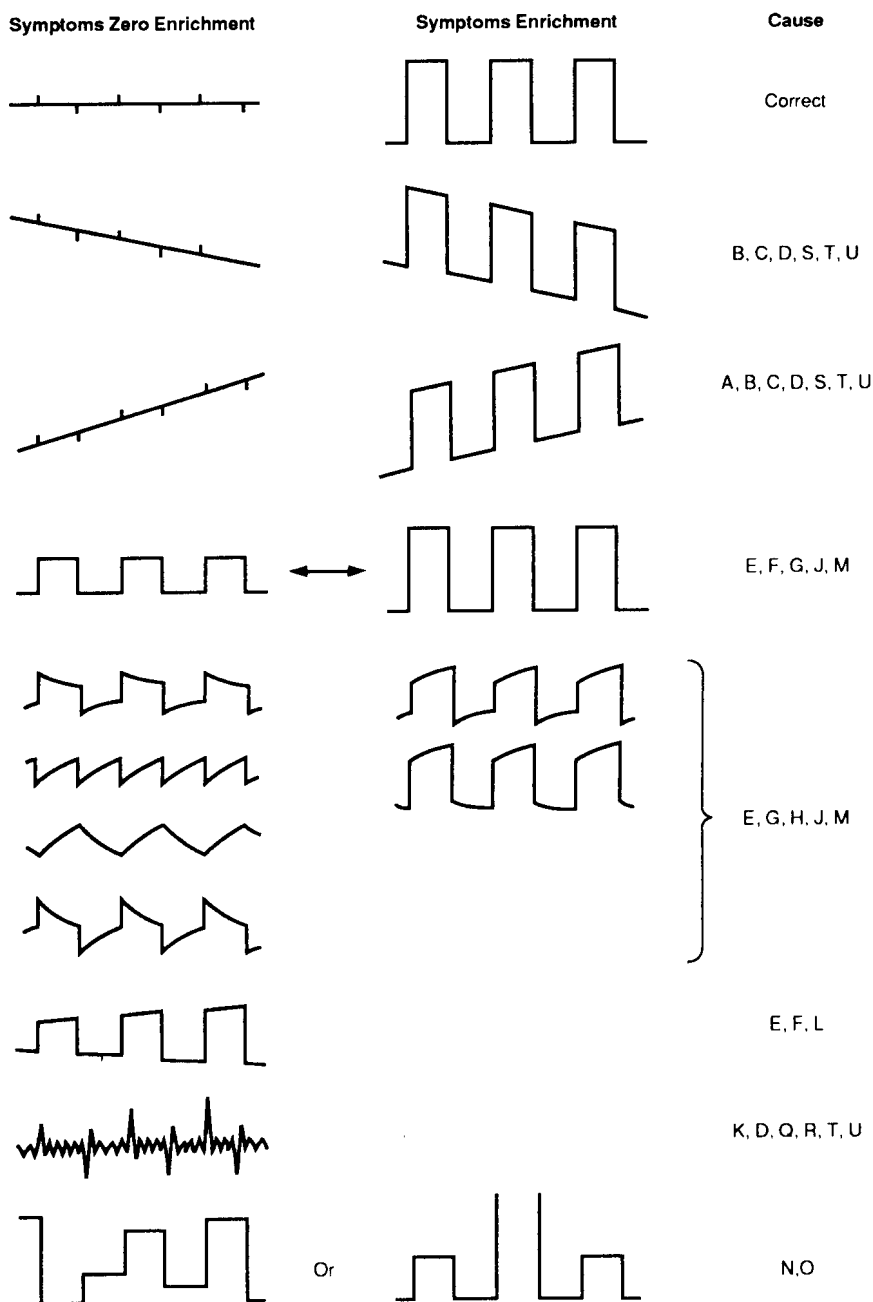
## Faults determined from Ratio Traces

These faults are determined from the ratio trace output at the end of each data run. They may apply just to the sample being run, to all samples or to the instrument itself. Simplified ratio traces are shown in the Figure above.

| Symptom | Cause | Cure |
|---------|-------|------|
| A | Sample depletion | Check capillaries are correctly set |
| B | Amp zero incorrect | Run zero amp routine |
| C | Major background changing | Bake system |
| D | Minor background changing | Bake system |
| E | Sample contamination | Check sample purity |
| F | Leak on inlet | Locate and cure |
| G | External leak on changeover valve | Locate and cure |
| H | Leak on bleed pipe | Locate and cure |
| I | Bleed pump failure | Identify and cure |
| J | Capillaries not balanced | Reset the capillary crimps |
| K | Poor resolution | Tune source for good peak shape |
| L | Unequal inlet volume | Set sample and reference inlet valves to same |
| M | Unequal inlet pressures | Adjust variable reservoirs to equal pressures |
| N | Inconsistent changeover valve | Check air supply, solenoids and changeover valve plungers |
| O | Changeover valve sticking | Strip and clean the changeover valve |
| P | Glitching filament | Change Filament |
| Q | Amplifier noise | Change faulty amplifier |
| R | Dirty source | Strip and clean source |
| S | HT or magnet supplies noisy | Check unit and load stability |
| T | Suppressor voltage faulty | Check voltage and shorts in collector |
| U | Electrical leakage on collector | Clean ceramics and input with acetone or similar solvent |

As can be seen from the above table of possible faults there are many symptoms which can be attributed to more than one cause. The next stage in any fault finding routine must be to separate the correct cause from all other possibilities.

The simplest approach is to try a process of elimination until the correct one is found. Indeed this might be the only approach in some situations, e.g., the inconsistent changeover valve. In this case we would first check the air supply, then use either a meter or test PCB to check the electrical signal to the Predyne valve. Finally we would dismantle, clean and re-grease the plunger. Other cases might be approached differently depending on our experience of fault conditions: noisy ratio traces are usually associated with a glitchy filament. So the first line of approach here would be to monitor the source current and look for changes in that current, in time with excursions in the ratio traces. A high source current would obviously be another pointer to the filament being the problem.

In all cases the logical approach must reign supreme. As an aid to solving the problem it can often be of help to draw a sketch of the relevant components of the system, especially when swapping components.

This brings us on to one of the most powerful fault finding techniques, that of exchanging components where more than one of those components are available. For example, if the nitrogen results are very noisy, but the carbon dioxide traces are as quiet as one would expect, we would suspect the only non-common item in the system, that is the magnet supply.

Another example of swapping components concerns the amplifier. Normally we measure the ratios 2/1 and 3/1 however, if both traces are noisy and we suspect the major channel we can swap leads 1 and 2 over. Now we are measuring the rations 1/2 and 3/2, if both results are still noisy then the problem is in the analyser. However if only the ration 1/2 is noisy the problem is in the major channel of the amplifier (or a fault on the VFC card).

# Technique for Finding Contamination

Anyone who has tried to prepare and measure a small (<1 uMol) sample of $CO_2$ will realise that even the smallest amount of contamination will have a detrimental effect on the accuracy and precision of the isotopic measurements, in particular, the oxygen isotope $^{18}O$.

If contamination is suspected, either in the inlet or the preparation line then the following gas transfer technique can be used to pinpoint the specific location:

Introduce $CO_2$ all round the inlet with the reference bellows compressed to 1,000 and a major beam of 1E-8 Amps. Close RI and SI. Carry out a zero, all open, measurement. This is the BASELINE ZERO.

Isolate both halves of the inlet by closing RP and SP.

Close SV and pump out the sample side on high vacuum for 2 minutes.

Close SM SP SI.

Open SV, wait 20 seconds.

Close SV. Enclosed in the volume between SM and SP is approximately 10μl of $CO_2$ which is isotopically identical to the gas in the reference bellows.

From the 'Program' menu, select CYCLE C/F.SEQ. Input freezedown time in the parameter box of 300 seconds. Select OK. (The time may need to be longer depending on the section requiring checking).

The 10μl of $CO_2$ will be frozen into the cold finger for 300 seconds, the valve SF closed, and the temperature of the cold finger raised to ambient automatically. When the sequence is finished, the message CYCLE COMPLETE will appear.

Open SM. Wait till the beam has stabilised.

Switch over to the reference side and set the major beam to about 0.2E-9 less than the sample beam. Close RM and RV and note the major beam. Open RV.

Switch over to the sample side. The beam should be depleting, but still greater than the reference just set. When the sample beam reaches a value just greater than the noted reference beam close RV.

An equal amount of reference and sample gas are now depleting from equal c/f volumes. Carry out a data run measuring the isotopic difference between the sample gas and the reference. Since the gases are the same the result should be zero.

If the inlet cold finger is contaminated then the $^{18}O$ result will show a shift, usually positive, away from zero.

This result is the **INLET COLD FINGER BASELINE ZERO**.

The next section to be checked is the transducer region. Pump out the transducer region and the sample cold finger volume.

Close SM SI SP open SV. Wait 20 seconds. Close SV.

Open SI and allow the gas to enter the transducer region.

Select **CYCLE C/F.SEQ.** as before this time selecting a freezedown time of 300 seconds.

Measure sample versus reference as before. The result will show whether the transducer section is clean.

Repeat the procedure for sections further away from the inlet where applicable e.g. Trapping system or Isocarb. The further the section under test is away from the inlet the longer the freezedown time will have to be.

If any of the sections appear to be 'Dirty' then it is advisable to repeat the section again (to avoid mistakes in procedure) before cleaning the appropriate section. For details on cleaning procedures see other sections of this manual.

# Bellows Faults

CAUTION

Fault finding should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

## Both Bellows Not Moving

If both of the bellows (reference and sample) are not moving then the likely causes are:

'DC Supplies' switch on the Utility Unit is in the OFF position or the fuse has blown.

Cables from the Stepper Motor Card to the bellows are missing or damaged.

Both of the motors are damaged. It is very unlikely for both motors to be damaged.

Stepper Motor Card in the System Controller is damaged. Check to see if there are any LEDs lit on the card.

Flags for the end stops of the bellows have moved together so the motor position is trapped. This is very unlikely for both motors.

24V supply to the motors is missing.

## Single Bellows Not Moving

If one of the bellows (reference and sample) is not moving then the likely causes are:

Cables from the Stepper Motor Card to the bellows are missing or damaged. This can be identified by swapping cables between the motors.

Motor is damaged

Flags for the end stops of the bellows have moved together so the motor position is trapped.

A third of the Stepper Motor Card in the System Controller is damaged. This can be checked out by swapping the cables from one bellows to the other.

CAUTION

Do not swap cables or remove the steeper card with the System Controller ON and 'DC Supplies' switch ON.

## Bellows Motors Stall

On systems where the sample and / or reference bellows have a tendency to stall (slip), the MOTORATE sequence can be used to change the motor speed, thus reducing the resonance which causes the problem.

**Note:** The problem if it appears is made worse if the Mains input voltage is low.

The speeds are defined in the 'Bellows Speed' parameter file, which can be edited to give sample and reference bellows speeds 10 steps per second and 200 steps per second. The default, set when the System Controller is powered-up, is 150. Users with resonance problems should try various values, each at a range of vacuum levels until a reliable drive speed is found.

Once the speed parameters have been changed, they can be transmitted to the System Controller by running the sequence 'MOTORATE'. This sequence should be run whenever the speeds in the parameter file are changed, and whenever the system controller is powered-up.

# Cold Finger Faults

CAUTION

Fault finding should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

## Fails to Heat

If the cold finger fails to heat up (e.g. between samples), then some of the likely causes are:

The heater element is open circuit. This can be checked with a multimeter.

The incorrect cable is connected to the heater or is damaged.

Thermocouple is connected incorrectly or is damaged (this is indicated in the Dual Inlet window). Check that the heater is on and that the Thermocouple is not reading.

'DC Supplies' switch on the Utility Unit is OFF and the fuse has not blown.

The LED 11 on the PCB in the Utility Unit lights when Heat is selected and the associated relay is operational.

SPIBB cable from the SPIBB card in the System Controller to the Utility Unit is disconnected or damaged.

SPIBB card is not functioning.

WARNING

The cold finger can get either very hot or very cold and should not be touched.

## Fails to Cool

If the cold finger fails to cool down (e.g. cold finger size sample runs), then the likely causes and some of the checks that can be carried out are:

No liquid Nitrogen available. (This is normally indicated by the software during sample running as the cause for the cold finger not reaching the base temperature.)

Charles Austen pump not functioning.

Pipe to the cold finger blocked or not connected correctly.

Check the thermocouple is connected correctly and is undamaged (this is indicated in the Dual Inlet window). Check the cool is not on, but the Thermocouple is not reading.

'DC Supplies' switch on the Utility Unit is ON and the fuse has not blown.

The LED 12 on the PCB in the Utility Unit lights when Cool is selected and the associated relay is operational.

SPIBB cable from the SPIBB card in the System Controller to the Utility Unit is connected and undamaged.

SPIBB card is not functioning.

WARNING

The cold finger can be either very cold or very hot and should not be touched.

## Charles Austen Pump Not Functioning

Refer to the manufacturers manual.

# Inlet Valve Faults

CAUTION

Fault finding should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

If the Inlet Valves fail to function correctly, either not opening or not closing fully, then some of the probable causes and checks to be made are:

'DC Supplies' switch on the Utility Unit is ON and the fuse has not blown.

Check that the compressed air is arriving at the valve. Carefully loosen the air line to the valve and listen to the air escaping.

Check for blockages in the air line.

Check the compressor operation and that the correct air pressure is set.

Check the correct valve is being selected from the software.

Check the Inlet Valve piston mechanism is operating. This can be checked by removing the piston housing and operating the valve.

Check the valve is correctly built, e.g. spring is not missing.

Check the operation of the solenoid (Predyne) valve is operating correctly. This can be done by swapping the air line with a working valves air line or splitting the Predyne valve and ensuring the solenoid is energising when the valve is operated (often the valve only needs cleaning).

Cables from the Valve Card to the Predyne is missing or damaged. This can be identified by swapping cables between the Predyne assemblies.

CAUTION

Do not swap cables or remove the valve card with the System Controller ON and 'DC Supplies' switch ON.

Valve card is damaged. This can be checked by swapping cards if more than one is fitted.

If the valve is not closing correctly then the valve pad may be leaking. This can only be confirmed by replacing the valve pad, so this should be left till last as it means venting the valve.

## HD Magnet Fails to Move

CAUTION

Fault finding should be carried out by a suitably qualified technician. Please contact the Customer Service Department atMicromass or your local Micromass representative with any queries.

If the HD magnet fails to move when the HD option is chosen from the software, then some of the probable causes and checks to be made are:

Any of the valve problems from above (Inlet Valve Faults), as the movement is accuated by Predyne Valves.

Check the air regulators fitted to either end are not fully closed.

Check the Piston is working correctly.

Check there are no obstructions.

## System Controller Error Messages

The errors tabulated below are reported by the system controller as numeric error code, and translated into the text description by the data system software. In the event of an error, the description, along with the command which caused the error, is displayed in the Message Window. If the error code is not recognised by the software, it will be displayed without a text description.

Possible causes are shown below for each type of error. 'Software Errors' are those generated by a bug in the software suite, and should be reported to the Customer Care department.

When reporting an error message to the Customer Care Department, always note down the whole line which appears in the message window, not just the error message itself. It is helpful, if you also note down:

The operation you were trying to perform (if any).

Any suspect behaviour of the software (e.g. Erroneous monitor window data)

Any message preceding or following the error.

| Code | Description | Possible cause |
|------|-------------|----------------|
| !0300 | invalid command | Software errors<br><br>Incompatibility between software and firmware versions |
| !0301 | invalid mnemonic | Incorrect mnemonic used in a sequence<br><br>Old version of firmware, which does not support new prep. system<br><br>Mimic diagram does not match actual hardware configuration<br><br>Missing/failed STE card<br><br>Incorrect STE card link settings |
| !0302 | invalid argument | Incorrect mnemonic used in a sequence<br><br>Parameter in sequence or parameter file out of range |
| !0333 | invalid number of parameters | Software error |
| !0400 | invalid beam mask | Attempt to acquire data from missing collectors (e.g. HD on a PRISM with no HD head amplifier).<br><br>Attempt to acquire data with no collectors selected. |
| !0401 | invalid integration time | Integration time in scan window out of range |
| !0405 | bad acquisition command sequence | Software errors |
| !0411 | invalid scan step | Step parameter in scan window out of range |
| !0600 | hardware not present | Missing STE hardware<br>Failed STE hardware |
| !0601 | invalid hardware type | Errors in configurable mnemonics table |
| !0602 | invalid hardware channel | Errors in configurable mnemonics table |
| !0603 | invalid operation | Software errors |
| !0604 | channel already in use | Software errors |
| !0610 | internal limit reached | Firmware has run out of data space to perform a specific operation |

| !0611 | no more mnemonic space | Configurable mnemonics table too big |
|-------|------------------------|--------------------------------------|
| !0620 | no acquisition hardware | Missing/failed STE VFC card |
| !0621 | no acquisition interrupt | Missing/failed STE VFC card |
| | | Incorrect link settings on VFC card |
| | | Incorrect link settings on processor card |
| | | Failed processor card |
| !0934 | gauge 1 tripped | Ion Gauge 1 tripped |
| | | Ion Gauge 1 filament blown |
| | | Ion Gauge 1 controller failure |
| | | STE gauge card failure |
| !0944 | gauge 2 tripped | as above for Ion Gauge 2 |

# General Notes on Fault Finding

Here are a few guidelines to help you when fault finding.

**ALWAYS** check every possibility outside the vacuum before opening up the system.

**ALWAYS** check the continuity of a filament and isolation from it's surrounding before closing up the vacuum system.

**ALWAYS** tighten up any vacuum seal evenly in a cris-cross fashion.

**ALWAYS** check that capillaries are open before fitting.

**ALWAYS** allow plenty of time for the vacuum to come down before assuming that there is a leak.

**ALLOW** the head amplifier at least an hour to warm up before taking measurements.

**ALLOW** the magnet at least an hour to warm up before taking measurements.

**ALWAYS** double check especially when swapping components.

**NEVER** jump to conclusions!

When the source of the problem has been located the repair must be carried out meticulously to ensure the cure is successful.

# Electronics

The following problems may be caused by faults in the electronics systems:

## System Crashes

As with any high voltage system, occasional flash-overs (caused by dirty source, high analyser pressure, etc.) can occur. In some circumstances this may cause the microprocessor in the system controller to "crash".

Signs that the system controller may have crashed are:

The software error message "System Controller Not Responding" appears on the PC.

The Source Unit turns off and cannot be tuned back on from the software.

The LEDs on the processor card show a fault condition (see firmware manual for details)

The system controller can be reset by following the electronics power down procedure, followed by the power up procedure.

Under no circumstances should the system controller be switched off then on alone via the switch on its rear panel. This could open inlet valves at random thus venting the analyser.

If resetting the system controller does not cure the problem, then an STE card may have been damaged. The system controller can run with only two cards (the Processor card and the VFC card) and so removing all other cards in turn may isolate the faulty card. If the VFC card is damaged the power-on LED sequence may show this (see Firmware Manual for details).

## Problems Controlling the Source/HT Units

If the source cannot be switched on from the software, the most likely cause is a system controller crash as described before. Other possible causes are:

The "DC Supplies" switch is off.

The Ion Gauge is switched off.

The analyser pressure is too high.

The System Controller SPIBB card is damaged.

The System Controller Gauge Controller card is damaged.

If the software's "Tune Source" option does not function, the most likely cause is a damaged System Controller Unit Interface card.

If "Invalid Mnemonic" error messages are displayed in the software message window for each of the source mnemonics "AV" etc. then the Unit Interface Card is almost certainly the culprit. If all looks well in the software, but the voltages still do not change, check the unit interface ribbon cable from the System Controller to the Source and HT units.

## Magnet Supply Problems

If attempting to change the magnet current results in an "Invalid Mnemonic" error message, the System Controller EMS card is almost certainly to blame.

If the magnet is off despite the programmed current, the possible causes are:

DC Supplies Switch off.

"EMS" pop-out fuse tripped.

Raw Supply Failure (check the "Magnet Power" LED on the magnet controller front panel).

Magnet Supply failure.

EMS card failure.

If the magnet current appears to be full on despite the programmed current, turn off the DC supplies switch to avoid damage to the electromagnet. Check that the cable between the system controller EMS card and the magnet supply is intact.

If a hall probe is fitted, check the position of the probe, and the integrity of the hall probe cable connections.

## Beam Problems

If is appears impossible to get any beam readings in the monitor window, check that the DC Supplies Switch is ON and that Head amp. power supply and fibre optic connections are intact.

The head-amp power supply status is reported by three LEDs on the utility unit rear panel (Analogue, Digital and Suppressor). Fuses for these are located on the Utility PCB and should only be replaced by a qualified technician.

## Fault Diagnosis Using a Terminal Emulator

If a fault has occurred which could be caused by either software or system electronics, communicating with the electronics directly can help. OS/2 has a terminal emulator which can be used for this. This will usually have been configured during factory testing, however complete instructions are included in the appendix.

Shut down the data-system software.

Open the "OS/2 System" window.

Open the "Productivity" window.

Start-up the application "PMTerminal"

Select the session configured for system controller communications.

Any characters typed are sent directly to the system controller. A full list of commands is included in the Firmware Manual (included in the appendix), but here are some tips:

Press the space bar before typing each command to get an echoing prompt.

use the read command "r" and write command "w" to access hardware which is suspect (valves, motors etc.). A full list of mnemonics is included in the firmware manual.

To read the beams of a standard PRISM, use the "b*..." command to read beam counts - see the firmware manual for more details.

The internal commands "i..." can be used to get detailed system information. If you contact the Isotopic Analysis factory with a problem, you may be asked to use these commands.

Always leave the cursor at the left-hand margin - not at the prompt - before leaving PMTerminal.

# System Repairs

CAUTION

System repairs should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

## Overview

This section of the manual will deal with repairs to the system. Care must always be taken when removing items from the system to avoid damage and contamination. It is also advisable for you to take notes as you disassemble the components to compliment this manual when it is time to reassemble.

**Remember:** If you have any queries please contact the Customer Service Department at Micromass or your local Micromass representative.

# Mass Spectrometer Maintenance

This section of the manual will deal with repairs to the Mass Spectrometer. The first section repeats the procedure for venting the system from the Operation section to avoid you having to turn back to that section, as most of the repairs require venting the system.

## Venting the PRISM

Venting the PRISM mass spectrometer is basically the reverse of pumping the system down and the following procedure should be carried out:

Turn OFF the source.

Close all the inlet valves, as this will keep the inlet components dry.

Turn off the ion gauges.

CAUTION

It is advisable to wait for at least 30 minutes at this stage to let the source and ion gauges cool down. This helps to avoid contamination or breakage of these components caused by the reaction when hot with air.

Switch OFF the 'High Vacuum' switch on the Utility Unit. This turns off all the power to the Turbomolecular pumps. The Turbomolecular pump vent valves will not open until the speed of the pumps drop below 50% of full speed.

Immediately after switching off the Turbomolecular pumps, close the backing line isolation valves and open the vent valves (next to the isolation valves).

**Note:** Dry Nitrogen or dry Air can be connected to the other side of the vent valves if required. Venting to dry gas will aid the return to high vacuum conditions.

The Turbomolecular pumps will now slow down and after approximately 5 minutes the vent valves will open and the system will come up to atmosphere, as shown by the Pirani gauges in the monitor window. The various parts of the system can then be opened as required.

## Removing the Ion source

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The procedure for removing the Ion source is as follows:

Ensure all high voltage supplies are turned off before carrying out operations on the source. Remove the source connector plug.

WARNING

Failure to complete Step 1 may result in Electrical Injury.

Ensure the system is safely vented.

Undo the bolts on the source housing lid.

If necessary, use jacking screws to loosen the lid.

Remove the lid. Place it on a clean lint free tissue on a flat work surface. Protect the sealing faces from abrasion.

Wear gloves when handling the source. Use a pair of tweezers to pull the gold barrel connectors off the feed through pins.

While holding the spring loaded inlet probe ceramic in the retracted position, undo the 2 captive screws on the source mounting bracket (See figure below).
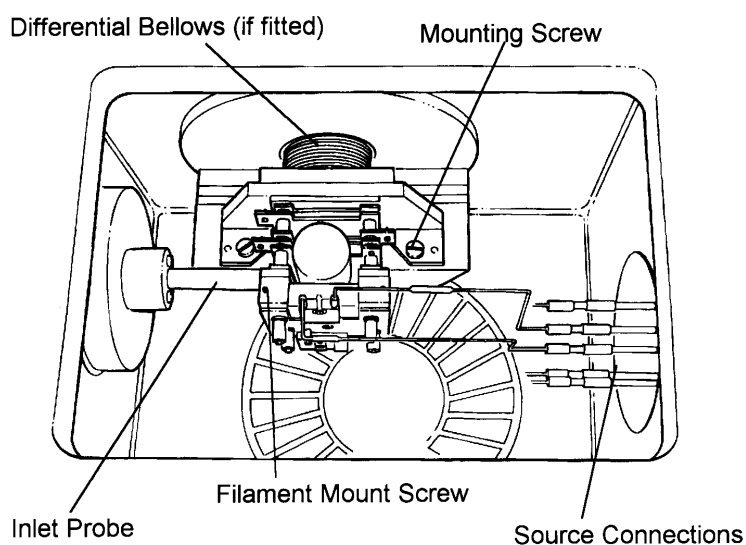


**Note:** The Differential Pumping Bellows (if fitted) make no difference when removing the ion source, however the operation for re-fitting is a little more difficult with the bellows fitted to the front of the source.

## Replacing the Ion Source

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The procedure for replacing the Ion source is as follows:

Before inserting the source:

a) Ensure all connecting screws and wires are tight.

b) Use an AVO meter to check for filament continuity and short circuits.

c) If a suitable device is available, perform a high voltage isolation test (pin to pin and pin to earth).

While holding the inlet probe ceramic in the retracted position and the source in position, locate the 2 mounting screws in their locating holes on the housing mounting lugs. Tighten these fully, doing this in stages so not to angle the threads.

Restore the inlet probe to the inserted position. Check it is properly located against the source block (flat face towards flight tube).

Using tweezers, push the gold barrel crimps on the connecting wires onto the source feedthrough pins. The Figure below shows the connections on the inside of the feedthrough, looking out.
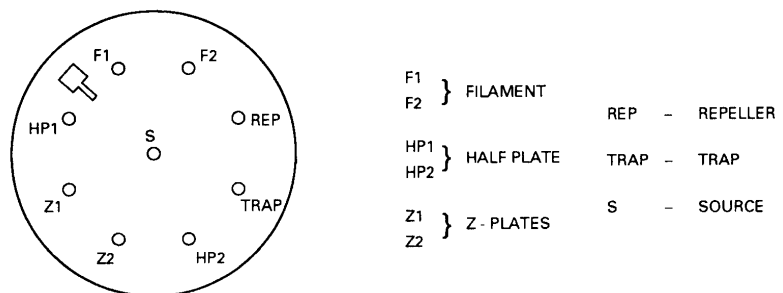


Check that the connecting wires are attached correctly and that there are no short circuits. Use a meter from the feedthrough pins to the appropriate source component to check continuity.

Check that the sealing surfaces on the housing and the source lid are free of dust etc.

Replace the lid, lining the bolt holes up carefully. Insert the bolts and tighten the bolts from the centre outwards, working in diagonally opposite pairs. This ensures an even seal and if no further internal work is required, pump out the system.

## Changing the Ion Source Filament

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The procedure for changing the Ion source filament is as follows:

Follow the venting procedure.

The filament may be changed without removing the source. If you wish to examine the source in detail, or to clean it, follow the source removal procedure, then go to step (4).

With the source in situ, use tweezers to pull the gold barrel connectors off the two filament leads from the feedthrough pins.

Use a small screwdriver (jeweller's type is ideal) to undo the two screws which attach the filament and source magnet assembly to the source block (Fig 2.11, item 4).

Carefully withdraw the assembly from the rest of the source.

Remove the filament lead barrel connectors from the two filament base posts.

Unscrew the filament retaining screw from the magnet yoke assembly, and remove the old filament.

Before attaching the new filament, make sure that the filament wire is securely attached to the posts, and has no kinks or other defects.

Secure the new filament to the magnet yoke with retaining screw.

Place the magnet / filament assembly on a flat metal surface, and adjust the filament position following the procedure given below.

Attach the filament leads to the base posts.

Attach the source magnet/filament assembly to the source. Tighten the two retaining screws evenly so that the assembly is parallel to the source block.

Use an electrical meter to check for filament continuity and for any short circuit between the filament and the source block.

Either (i) reconnect the two filament leads at the feedthrough pins, or (ii) follow the full source replacement procedure.

Filament Position

The procedure for positioning the filament is as follows:

Make sure the filament wire is parallel to the filament body, that it has a clean even coating of Thoria and the shield does not touch both of the filament legs.

Secure the filament to the magnet assembly (south pole - marked with an 'S') making sure the filament shield is against the magnet pole face.

Place the magnet assembly on to a flat, clean surface (As shown below). Adjust the height of the filament wire to approximately 1 mm from the flat surface. Once the filament wire is parallel to the flat surface and at the correct distance away then lock the filament in place by tightening the screw.
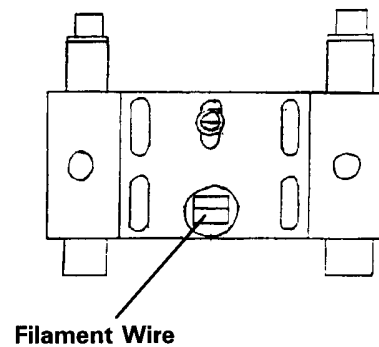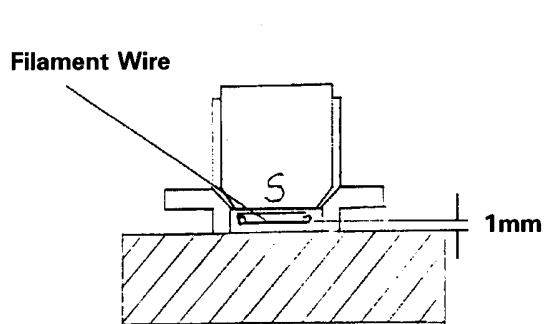
## Disassembly of the Ion Source

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

This should only be undertaken under clean conditions on a clean surface.

**It will be advantageous to make notes and record the orientation of the plates and keep the spacers from each layer separate as you disassemble the source to ease the re-assembly.**

The procedure for disassembling the Ion Source is as follows:

Remove the ion source from the following the procedure given above.

Place the on a flat clean surface, with the source mounting bracket (item 13) down. Note: If the Differential Pumping Bellows are fitted, then remove before placing down (See figure below).

Remove the magnet block assemblies from either side of the source (items 36), being especially careful not to damage the filament. Remember when reassembling the filament is mounted on the magnet marked with a 'S' for south.

Remove the circlips (item 27) from the ends of the ceramic rods (item 26) from the source block (item 22) end of the source.

The ion source can now be disassembled carefully removing the spacers and plates one at a time. The diagram below shows the exploded view of the source once it has been disassembled.
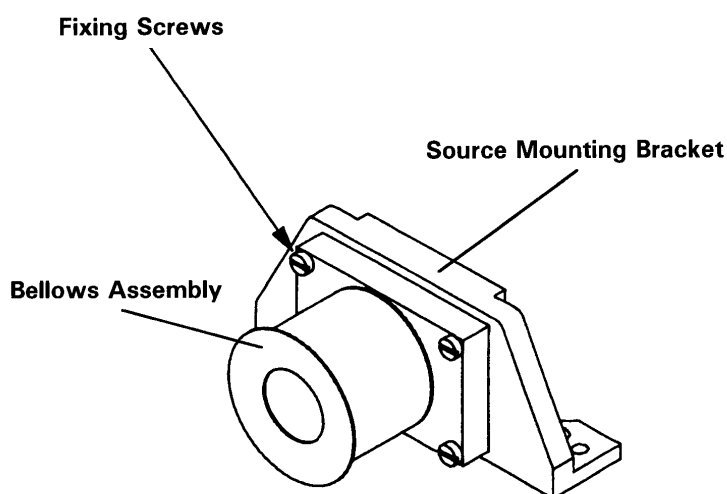


Figure : Source Mounting Bracket with Differential Pumping Bellows Assembly attached.

**Final Assembly**

**Note - Wiring Connections**

F1, F2  Connected to the Filament
Z1, Z2  Connectd to the Z Plates
HP  Connected to the Half Plates
TR  Connected to the Trap
R  Connected to the Ion Repeller
S Connected tothe Source Block

**Top of Source & HD Housing**

R⊙

TR○

HP○

F2

Z2

F1

Z1

HP

View of the Feedthrough from
inside the Housing
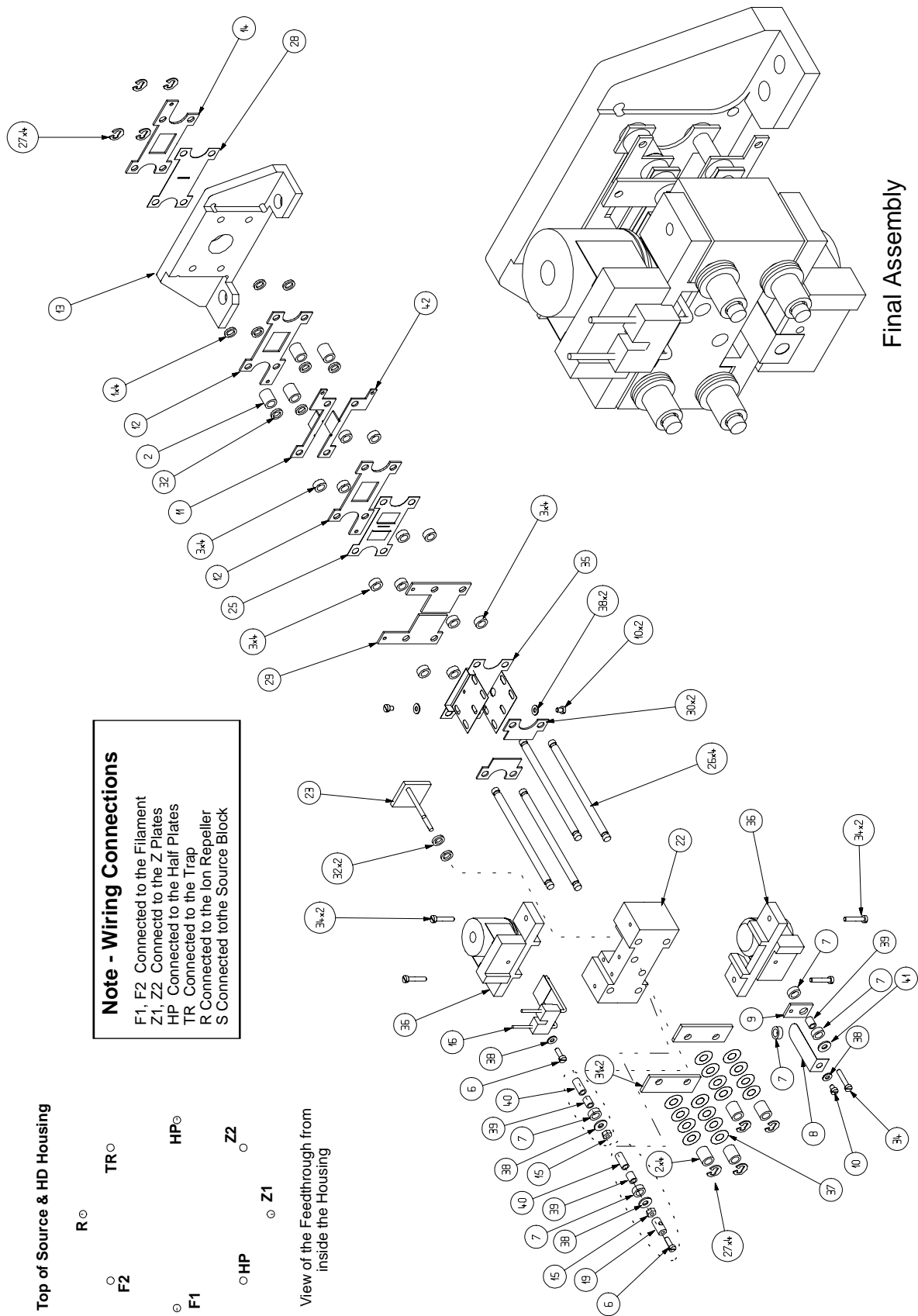
**Source Assembly Parts List**

| Reference | Part No. | Description | Quantity |
|---|---|---|---|
| 1 | 3009884 | Distance Piece  (075mm) | 4 |
| 2 | 1037037 | Spacer Metal  (0.219") | 4 |
| 3 | 1036052 | Spacer Ceramic  (1.98x2.08mm) | 12 |
| 6 | 1038018 | Screw SS CH HD  (10BAx3/16") | 3 |
| 7 | 1640052 | Spacer Ceramic  (0.071") | 5 |
| 8 | 3602899 | Trap | 1 |
| 9 | 1037018 | Bracket Trap | 1 |
| 10 | 1038019 | Screw SS CH HD  (10BAx1/8") | 3 |
| 11 | 3009878 | Z-Plate | 1 |
| 12 | 3009883 | Electrode Standard Plate 2 | 3 |
| 13 | 3010833 | Short Source Mounting Bracket | 1 |
| 14 | 3010533 | Electrode Plate | 1 |
| 15 | 1038026 | Nut Hex Full SS  (10BA) | 2 |
| 16 | 4003260 | Filament Assembly | 1 |
| 19 | 1637041 | Connector  (10BA) | 1 |
| 22 | 3602003 | Ion Box | 1 |
| 23 | 1637012 | Ion Repeller | 1 |
| 25 | 4002904 | Source Slit Shield Assembly | 1 |
| 26 | 1036059 | Ceramic Rod | 4 |
| 27 | 1026022 | Circlip (Truac 5133/12H) | 8 |
| 28 | 3009880 | Alpha Plate | 1 |
| 29 | 3009879 | Source Half Plates | 2 |
| 30 | 3009885 | Ion Exit Support Plate | 2 |
| 31 | 3602103 | Locating Plate | 2 |
| 32 | 1036054 | Ceramic Spacer  (0.755x0.8mm) | 6 |
| 33 | 1015031 | Screw SS CH HD   (M3x10) | 2 |
| 34 | 1038016 | Screw SS CH HD   (10bax3/8") | 4 |
| 35 | 3009886 | Ion Exit Plate | 1 |
| 36 | 4003109 | Filament Magnet Block Assembly | 2 |
| 37 | 1011048 | Washer SS Wavy  (M3) | To Suit |
| 38 | 1038025 | Washer SS Flat   (10BA) | 6 |
| 39 | 1640016 | Ceramic Sleeve  (3.5mm) | 2 |
| 40 | 1640018 | Ceramic Sleeve  (6.4mm) | 2 |
| 42 | 3010534 | Z-Plate B | 1 |
| 43 | 1037025 | Spacer Metal  (0.244") | 4 |

## Assembly of the Ion Source

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The assembly of the Ion source is the reverse of disassembly above ensuring that the filament is positioned correctly and that none of the plates are shorting together.

## Replacing the Inlet Probe

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The procedure for replacing the Inlet probe is as follows:

Follow the source removal procedure.

Use a small screwdriver to undo the two screws which hold the ceramic retaining plate in position.

The retaining plate, ceramic, and the spring which holds the ceramic against the source can all now be removed for cleaning or renewal.

Replacement is the reverse of removal.

**Note:** There is a machined flat on the probe, so that the tip is semicircular. Ensure that this flat locates properly in the source block against the ion exit slit plate. Failure to locate the probe correctly will cause a loss of sensitivity.

## Replacing the Ion Gauge filament

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

If both Ion Gauge filaments have been blown or a broken filament is causing a short circuit, it is necessary to change the Ion Gauge filament. The procedure is as follows:

Follow the venting procedure and ensure the instrument is left for at least 30 minutes to allow the Ion Gauge to cool.

Remove the Ion Gauge from the Ion Gauge housing, by removing the six bolts. Care must be taken to avoid damage to the Ion Gauge.

Stand the Ion Gauge on its end and undo the three set screws in the top of the barrel connectors holding the old filament array in place, using the Allen key supplied with the new filament. The old filament array can then be removed.

Insert the new collector array into the barrel connectors and tighten the set screws, ensuring the filament array is spaced equally around the curve of the grid.

Cut the small bars at the base (barrel connector end), using a small pair of wire cutters, which connect the filaments to the filament common. These are only there to provide support for the filaments during transportation and if not removed will prevent the Ion Gauge from working.

Check for continuity with an multimeter and then refit the Ion Gauge into its housing, remembering to use a new copper gasket (do not use the old gasket). It is important that the filament are mounted at the bottom of the housing, so if one filament fails it will not drop onto the grid.

Check again for continuity and shorts before pumping the system.

## Cleaning Procedures

This is a generalised cleaning procedure for both Ceramic and Stainless Steel components used in the source or dual inlet. If more details are required then please contact Micromass Customer Service Department or your local representative.

CAUTION

Care must be taken when handling the components to avoid damage.

Do not use these procedures to clean Thoria coated items, e.g. source filament, ion gauge filament.

WARNING

This procedure involves Boiling Water and chemicals therefore the necessary precautions must be taken.

## Rough Cleaning

Polaris Powder ($Al_2O_3$ powder) can be used to remove obstinate marks (burn marks, etc.).

**Note:** When cleaning the Source Repeller use metal polish to avoid scratching the surface.

## Pre-cleaning

For Stainless Steel components this should involve hot solvent cleaner in ultra-sonic for 30 minutes or Hot ultra-sonic wash in Decon 90 ( or similar aqueous cleaner) for 15 minutes followed by a rinse in de-ionised water. Either of these processes should remove any fingerprints and mild contamination.

For Ceramic components this should involve boiling in Decon 90 (or similar aqueous cleaner) for 30 minutes followed by a rinse in de-ionised water.

## Final Cleaning

To prepare the components for use in the Mass Spectrometer. This is a generic cleaning procedure for any component that can stand the conditions. Exceptions include Nylon, O-rings, Kel-F, etc.

Boil in de-ionised water for 30 minutes.

Change the water and boil for a further 30 minutes

Dry with dry Nitrogen in holes etc. (optional).

Dry in oven at 100 to 120 deg C for 12 hours.

After cleaning all components should only be handled using gloves.

## Bakeout Heater

CAUTION

Electrical repairs should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

The Bakeout heaters are fitted in various places around the system (see Equipment Description section of this manual). If for any reason they fail the procedure for replacing them is as follows:

Ensure the 'Bakeout' switch on the Utility Unit is in the OFF position and that Bakeout is not running.

WARNING

Failure to observe the above may result in injury.

Unscrew the connections to the appropriate heater, from the connectors mounted under the bench.

Remove the circlips from the ends of the heater mounting block, which hold the heater in place.

Remove the heater.

Fit the new heater using the reverse method of the above.

## Rotary Pump Repair

Please refer to manufacturers guide for details.

## Turbomolecular Pump and Vent Valve Repair

Please refer to manufacturers guide for details.

## Air Compressor (if supplied) Repair

Please refer to manufacturers guide for details.

## Electronics Repair

CAUTION

Electronics repairs should be carried out by a suitably qualified technician. Please contact the Customer Service Department at Micromass or your local Micromass representative with any queries.

## Controller Board

The system controller is an "STEbus" system made up of "eurocard" size PCBs plugged into a standard backplane. The position of any card in the backplane is not important: any card can be inserted into any position.

The system controller firmware automatically looks for STE cards when it is powered-up, and then creates mnemonics for any cards which are present. The system controller detects the type of card (valve card, motor card etc., and the "number" of the card: valve card 0, valve card 1 etc.). Links on the PCB define the card type and number.

On occasions it may be necessary to replace a faulty card, or to add a card for a new preparation system. This should only be carried out by a qualified technician:

CAUTION

Anti-static precautions should be taken when handling system controller cards.

Switch off the electronics circuit in the normal way.

Open the system controller front panel.

If a card is to be removed, note down the positions of any cables connected to the card: labelling any connectors as necessary. Remove the card.

Check the link settings of the new card by comparing them with the failed card. If the card is part of a new preparation system supplied by Micromass, the link settings should be ready configured.

Insert the new card into the backplane. The position is not important, but it is advised that a replacement card be placed in the same slot that the failed card occupied.

Connect any cables as required. If the card is part of a new prep. system, the system's cables will need to be threaded through the port in the rear of the system controller, and beneath the cards to the front of the unit.

Switch on the electronics circuit: check that the system controller processor card (usually placed in the left-most slot) has a single green LED lit: this means that the boot-up sequence has been successful.

Close the system controller front panel.

Start-up the software and check for error messages.

If the new card fails to operate check:

that the card has mated with the backplane socket correctly.

that the link settings are correct.

that the link settings are different from those of all other cards of the same type.

If in any doubt, contact a Micromass Service Engineer.

## Electrical Supply Fuse Replacement

All fuses accessible to the user are of the pop-out type, and are situated on the utility unit rear panel. In addition the two mains inlet circuit breakers on the electrical inlet service panel will switch off if the rated inlet current is exceeded.

All replaceable fuses are located inside the instrument, and can therefore only be changed by a qualified service technician. **All fuses are of the 20mm type**. These are tabulated below:

| Fuse | Location | Legend | Rating |
|------|----------|--------|--------|
| Head amp analogue | Utility PCB | FS2 | 300mA T |
| Head amp analogue | Utility PCB | FS3 | 300mA T |
| Head amp digital | Utility PCB | FS4 | 1A T |
| Head amp suppressor | Utility PCB | FS5 | 300mA T |
| Head amp suppressor | Utility PCB | FS1 | 300mA T |
| Utility Digital (+5V) | Utility PCB | FS6 | 1A T |
| System Controller | S.C. Rear Panel | FS1 | 1A T |
| System Controller U.I. | Unit Interface Card | FS1 | 1A T |
| H.T. Unit Output Socket | H.T. Rear Panel | FS1 | 2A T |
| H.T. Unit H.T. Supply | H.T. Rear Panel | FS2 | 300mA T |
| Source Unit | S.C. Rear Panel | FS1 | 1A T |
| Turbo-pump Controllers | T.M.P. Control Unit | See Manufacturer's Instructions | |
| Ion Gauge Controller(s) | Ion Gauge Control Unit | See Manufacturer's Instructions | |

# Computer, Monitor and Printer Repair

Please refer to manufacturers guide for details.

# Dual Inlet Maintenance

This section of the manual will deal with repairs to the Dual Inlet.

## Changing the Dual Inlet Valve Seat

CAUTION

Wear hand protection (gloves) for this operation to avoid contamination.

The Kel-F seats in the micro-inlet valves are designed for prolonged trouble-free operation, but periodically it may be necessary to replace a leaking, contaminated, or otherwise defective seat.

You will need:

1 pair tweezers
1 pair lint-free gloves (surgical type ideal)
1 new valve carrier assembly
1 new diaphragm
Paper tissues etc.
M3 Allen Key
M5 Allen Key

The procedure for changing a Dual Inlet Valve Seat is as follows (also see diagram below):

Close ALL valves in the dual inlet, except the one to be replaced, which should be left open.

**Note:** If the valve to be worked on is one of the four in the changeover block, it will be necessary to shut down and vent the mass spectrometer. It is a good precaution to turn off the ion source and ion gauge when opening the dual inlet up.

Unscrew the 4 M3 x 12 bolts which hold the Piston assembly in position.

Withdraw the piston assembly. Take care not to lose the coil spring, which will be loose.

Mark the orientation of the spacer flange so that when re-assembling the flange can be returned to the same orientation. Unscrew the 4 M5 bolts which hold the knife edge spacer flange in position. Take care not to lose the brass actuator plug.
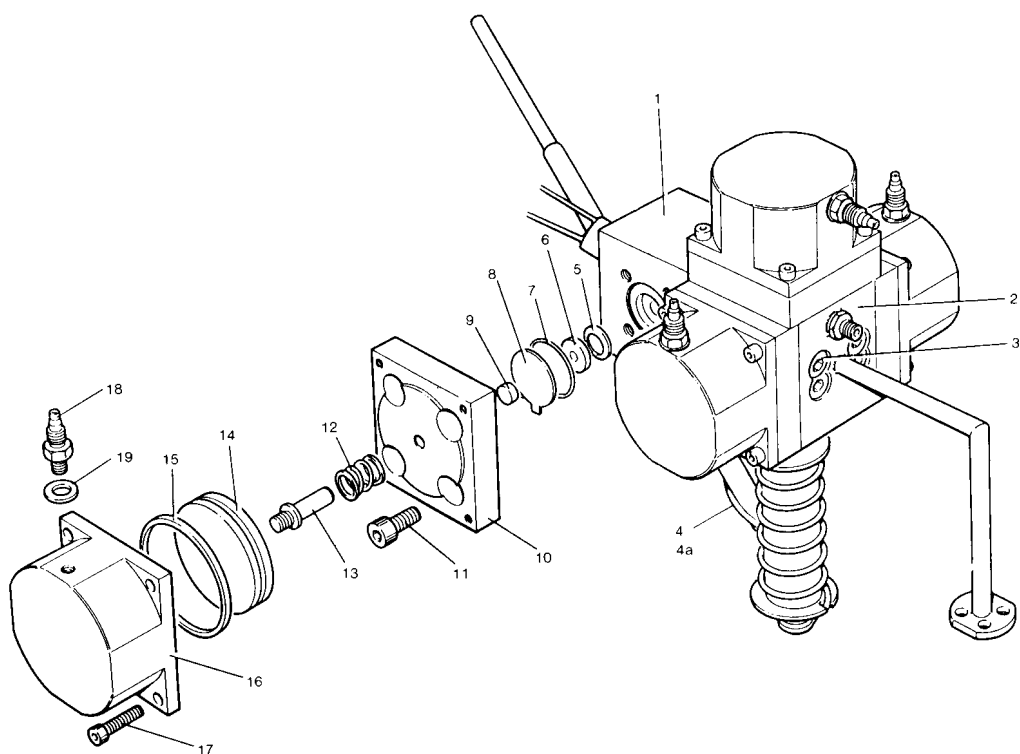
Put the spacer flange in a safe place.

**Note:** It is important not to damage the knife edge.

Use the tweezers to remove the old diaphragm by pulling on the projecting tab.

The old carrier assembly can now be removed.

**Note:** It may be necessary to ease it out by gently tapping on the **non-internal** parts of the valve block with your tweezers. **Do not scratch any internal surfaces.**

| Reference | Part Number | Description | Quantity |
|---|---|---|---|
| 1 | 3601830 | Double Valve Block | 2 |
| 2 | 3601831 | Triple Valve Block | 2 |
| 3 | – | M5 x 45 Bolts | – |
| 4 | 4002885 | Auto Cold Finger | 1 |
| 5 | 1640418 | Disc Spring | 14 |
| 6 | 3601836 | Valve Seat Carrier Assembly | 14 |
| 7 | 3601918 | MP3 Gold Ring | 14 |
| 8 | 3601847 | Diaphragm | 14 |
| 9 | 3601837 | Actuator Plug | 14 |
| 10 | 3601844 | Spacer Flange (with knife edge) | – |
| 11 | – | M5 x 10 Bolt | – |
| 12 | 1640420 | Compression Spring | 14 |
| 13 | 3601835 | Piston Rod | 14 |
| 14 | 3601833 | Piston | 14 |
| 15 | – | Piston 'O' Ring | – |
| 16 | 3601834 | Piston Housing | 14 |
| 17 | – | M3 x 12 Bolts | – |
| 18 | – | Compressed Air Connector | – |

The disc spring should also be removed and inspected.

Clean the disc spring if necessary.

Inspect the polished valve sealing face in the valve block for scratches etc. Remove any dust particles by **gentle** strokes with a lint-free tissue.

Re-insert the disc spring using tweezers. The spring should be oriented with its raised (inner) ring pointing out.

Insert the new valve carrier assembly. Handle it by its edges only using tweezers. The two 'dimples' should face inwards towards the disc spring.

Locate the new diaphragm over the new valve seat, positioned over the MP3 gold-ring. Ensure the handling tab is correctly located in the cutaway on the valve block.

**Note:** The gold ring is <u>very</u> seldom a problem and does not require removing or replacing.

Use firm hand pressure with the knife-edge spacer to ensure the diaphragm is located.

Having ensured that the brass actuator plug is in position, now bolt the knife-edge spacer flange back into its original position. Tighten the bolts in stages in a diagonal pattern.

Making sure the coil spring is located , bolt the piston assembly back into position.

Pump out the Dual Inlet to the low vacuum and then high vacuum for 5 minutes at least.

Operate the valve several times to ensure the new valve face seats properly.

The new valve seat should now be checked for correct operation (leak checks, etc.) and the inlet baked.

## Bellows removal and replacement

The two bellows are easily removed for maintenance, and the procedure is as follows:

Switch off the System Controller and unplug the ribbon cable to the bellows.

Undo the 4 bolts on the 1/4 inch gold flange seal at the connection to the inlet block.

Undo the three bolts which attach the bellows sub assembly to the inlet table.

Withdraw the assembly downwards and out of the bench.

The procedure for replacement is the reverse of above, making sure to use a new gold ring.

## Fitting new Capillaries

New capillaries are supplied as a matched pair. They should never be changed singly.

The procedure for fitting New Capillaries is as follows:

Pump out the dual inlet.

Turn off the ion source and ion gauge (precautionary measure).

Close ALL valves in the dual inlet

CAUTION

Close RW and SW also, so that the changeover valve is **fully** shut.

Remove the two upper crimping bars and the guide bar.

Undo the 4 nuts attaching the capillaries to the dual inlets and changeover valves.

Remove the old capillaries.

Use a file or cutters to retrieve the old nuts from the capillaries.

Fit a nut to one end of a new capillary, then fit the two part stainless steel ferrule, the smaller back ferrule, followed by the larger conical front ferrule. The "cones" point towards the valve block.

Fit the end to the valve block. The capillary should protrude a few mm beyond the ferrules, pushed into the stub on the valve block. (This is to avoid any dead volume).

Tighten up the nut carefully until it begins to bite and compress the ferrule.

Loosen the nut again and check that the ferrules and capillary protrusion are OK.

Tighten the nut up properly.

Repeat steps 8 to 12 for the other 3 attachments.

Refit the guide bar and crimping bars on the changeover valve assembly.

Pump out the capillaries on rotary vacuum via LV.

**Note:** Leave RW and SW shut. Pump for at least 15 minutes.

Switch to HV pumping. RW and SW may now be opened.

Crimp the capillaries down to the correct flow rate. **Take at least 2 days over this**. During the crimping process, both elastic **and** plastic deformation occurs. Over rapid crimping may result in the crimps closing after a bakeout.

## Adjusting the Capillary crimps

As with the Venting section above, this section is repeated from previous information and is only given here to ease use of the manual and need not be read if this is section is already understood.

Pump out the entire inlet.

Admit a suitable pressure of gas (say 15 mBar) around the entire inlet (i.e. RP, SP, open). The capillaries should be adjusted (balanced) at the target ion beam of 1E-8 Amps for $CO_2$ and $N_2$ , therefore use the bellows to adjust the ion beam height.

Allow the gas to equilibrate for 10 minutes.

Crimp the capillaries to approximately double the required flow rate (the flow rate required on the PRISM is measured as the depletion of the Major beam over an average analysis run and should be approximately 16% ) and bake the inlet.

After the system has cooled down adjust the capillaries to the correct flow rate, it is normally advisable to bake again to ensure the integrity of the capillaries.

Finally check the beam heights on reference and sample sides. They should be identical down to the 4th significant figure i.e. they should agree to less than 5 in this place:

| **For example** | Ref: | 10.1062 } | not balanced |
| | Sam: | 10.1180 } | |
| | Ref: | 10.1062 } | balanced |
| | Sam: | 10.1088 } | |

If final adjustrnent is necessary, it is always best to increase the flow rate on the lower beam side than to crimp down further. This avoids the risk of over-crimping.

## Changing Predyne (Solenoid) Valves

The procedure for replacing a Predyne valve approximately the same whether the valve is normally closed or normally open, and is as follows:

Switch off the 'DC Supplies' switch on the Utility Unit.

Switch off the System Controller following the procedure given previously.

Remove the compressed air from the system. This may involve switching off the compressor so check that nothing else is connected to the air line which may be damaged.

Disconnect the ribbon cable from the appropriate Predyne assembly.

Use a D-type connector pin removal tool to remove the pins from the D-type connector corresponding to the valve to be replaced (make a note of which wire goes to which pin number).

If the valve is a normally open type then unscrew the air fitting from the side of the valve.

Unscrew the old valve from the valve manifold.

De-solder the D-type pins from the old valve and solder them onto the leads of the new valve (Ensure that the new valve is of the same type as the old valve).

Fit the new valve onto the manifold and fit the pins in the correct orientation into the D-type connector.

If the valve is a normally open type then screw the air fitting into the side of the valve.

Connect the ribbon cable to the Predyne assembly.

Reconnect the compressed air to the system.

Switch on the System Controller following the procedure given previously.

Switch on the 'DC Supplies' switch on the Utility Unit.

The Inlet valve should now be checked for correct operation.

## Cleaning Procedures

The recommended procedures for cleaning components of various types in the Dual Inlet are given above.

## Changing Bakeout Heater

Please refer to the section 'Changing Bakeout Heater' in the 'Mass Spectrometer Maintenance' section of this manual.

## Changing CRT7 (if fitted) Valve Pad

Please refer to the Micromass  'All Metal Valve' operating instructions.

## Charles Austen Pump Repair

Please refer to the manufacturers 'Operating and Maintenance Instructions' supplied with this manual.

## PVA 25K / PVA 10K Repair

Please refer to the manufacturers guide for details.