

 *nu instruments*

Perspective
Stable Isotope Ratio Mass
Spectrometer

System Manual
Issue 1.0

Preface

Nu Instruments Ltd.

Unit 74, Clywedog Rd South
Wrexham Industrial Estate
Wrexham, LL13 9XS
North Wales (UK)

Tel.: +44 (0)1978 661304

Fax: +44 (0)1978 664301

Service Tel: +44 (0) 1978 665921

Service Email: irmsservice@nu-ins.com

www.nu-ins.com

This manual describes the Perspective Stable Isotope Ratio Mass Spectrometer. The contents include a description of the instrument, basic instrument operation and routine maintenance advice. All information contained in this manual is believed to be correct at the time of publication. All product specifications, as well as the information contained in this manual, are subject to change without notice.

I. Nu Instruments safety notices

Nu Instruments products are designed and manufactured in compliance with accepted worldwide practices and standards, to provide protection against electrical and mechanical hazards for the operator and the area surrounding the product.

All Nu Instruments products are designed and intended for professional use only, by skilled operators, for their intended purpose and according to all of the instructions, safety notices and warnings provided by Nu Instruments Ltd.

Those instructions, notices and warnings assume that an operator will not use any tool when using the Nu Instruments product, other than those detailed in the relevant instructions. The instructions, notices and warnings further assume that all operators clearly understand that the use of Nu Instruments products in any manner not specified by Nu Instruments may impair the protection provided by the products and expose operators and other personnel to hazards.

The hazard symbols and terms used in this document are defined below. To reduce or eliminate hazards, instrument operators must fully understand these symbols.

II. Warranty

Nu Instruments Ltd. makes no warranty of any kind regarding this material, including, but not limited to, the implied warranties of merchantability and fitness for a particular purpose. Nu Instruments shall not be liable for errors contained herein or for incidental damages in connection with the furnishing, performance, or use of this material.

III. Environmental conditions

According to BS EN 61010 - 1:2010, the normal environmental conditions to be maintained for all Nu Instruments instrumentation will be described below.

This standard applies to equipment designed to be safe under at least the following conditions:

- a) Indoor use
- b) Altitude up to 2000 m
- c) Temperature 5°C to 40 °C
- d) Maximum relative humidity 80% for temperatures up to 31 °C, decreasing linearly to 50% relative humidity at 40 °C
- e) Mains supply voltage fluctuations up to +/- 10% of the nominal voltage
- f) Transient overvoltages up to the levels of Overvoltage Category II

NOTE 1: These levels of transient overvoltage are typical for equipment supplied from the building wiring.

- g) Temporary overvoltages occurring on the mains supply
- h) Applicable Pollution Degree of the intended environment (Pollution Degree 2 in most cases)

NOTE 2: Manufacturers may specify more restricted environmental conditions for operation; nevertheless the equipment must be safe within these normal environmental conditions.

IV Software prerequisites

Nu Instruments software runs under a Windows multitasking operating system, with automatic updates and virus scanning options. The user should be aware of several possibly serious problems that could occur, arising from the automated updates and from the multitasking character of the operating system. Highly recommended precautions are described below, which should be taken to avoid any adverse consequences.

CAUTION

Microsoft Windows automatic updates and automatic virus scans should be disabled in order to avoid unexpected interruptions to the running of the instrument software and possible loss or corruption of acquired data.

Automatic updates and virus scans

It is vitally important to disable any automatic updates or automatic virus scans, since they have the potential to corrupt automated acquisition runs. Updates and scans should only be performed when the instrument is not acquiring data or when the computer is performing any other critical tasks. Updates and scans can assume complete control of the system and, in the case of automatic updates, may be followed by rebooting the PC, effectively totally stopping the control of the mass spectrometer, irrespective of any task being performed. This is a direct consequence of the multitasking nature of the operating system. By enabling automatic updates, the user is implicitly permitting other programs to be run at any time, without notice. The outcome may not be what the user would expect or desire and may occur when the user least expects it.

These options will have been disabled on any PC supplied by Nu Instruments, but this should be verified by the user before the mass spectrometer is used in earnest.

Select **Control Panel > Windows Update > Change Settings** to deselect automatic updates. Several updating options will be presented. Over the life of the instrument, the user is likely to replace the PC and the replacement will undoubtedly be delivered with these options enabled, together with numerous unnecessary free programs and options, which the user may well wish to remove. Automatic updates and scans must also be disabled before the PC is used in any critical manner.

In some rare cases, when an update has been allowed, the update may re-enable the automatic update option, without alerting the user. This change may not be apparent so the user should be aware that such possibilities can occur.

⚠ CAUTION

The Microsoft Windows multitasking environment allows other programs to be run on the instrument PC, as well as the instrument software. The user should be aware that other programs may have unwanted and undetectable effects on time-critical aspects of the instrument software.

Running other programs on the system PC

Since the main purpose of the PC is to control the mass spectrometer, it is highly recommended that it should not be used for other tasks that could be easily accomplished off-line. Again, since the operating system is multitasking, the user may expect that other tasks can be easily undertaken, such as analyzing data using programs like Microsoft Excel. However, since the user has no control over the allocation of the computer resource, it will be appreciated that if a computationally intensive calculation is performed by such a program, it may result in the mass spectrometer control program being suspended for a significant period.

Although buffering is used as much as possible to minimize such effects, if there are critical timing events being carried out, the possibility exists that unexpected problems may occur. Consider, for example, an event, denoted here as **Event 1**, being initiated by the software, and immediately afterwards in the code, a timer is started, denoted **Start timer**, which is there to monitor the timing of a subsequent process, starting from **Event 1**. Since the execution of each line of code in the mass spectrometer program is totally under the control of the Windows operating system, if a second program is running, it becomes theoretically possible for a large, and undefined, time delay to occur between **Event 1** and **Start timer**. The intention was that **Event 1** and **Start timer** should be virtually simultaneous, but now they are not, so the timing of the subsequent process will be inaccurate, which would be completely undetected by the software and by the user.

Microsoft Themes

As new versions of Windows emerge, new color schemes are provided by Microsoft, described as different Themes. Unfortunately, it has been found that some very unexpected effects can occur if certain of these themes are selected. It is therefore highly recommended to run the software using a basic or standard theme, without strong coloring. If programs or utilities are run off-line on another computer or laptop, some of the forms may unexpectedly become unreadable. If this occurs, check the computer or laptop Theme setting as a first resort.

⚠ CAUTION

Within this document, the software screenshots used are simply examples for explanatory purposes. The readings in these screenshots will differ from instrument to instrument. If, for example, the vacuum readings or tuning parameters do not match those seen on your instrument, this will not necessarily be an indication of a problem with your instrument. All screenshots are exemplars only. There is no requirement to try to match them.

V. Hazards, warnings and symbols

The terms **DANGER**, **WARNING** and **CAUTION** are used throughout this document. It is important that the user is familiar with the meaning of those terms in the context of this document and the meaning of the hazard symbols associated with potential hazards.

Hazard terms:

DANGER

Indicates a hazardous situation, which, if the necessary safety precautions are not adhered to, will result in death or serious injury. This signal word is limited to the most extreme situations.

WARNING

Indicates a hazardous situation, which, if the necessary safety precautions are not adhered to, could result in death or serious injury.

CAUTION

Indicates a hazardous situation, which, if the necessary safety precautions are not adhered to, could result in minor or moderate injury.

This term is also used with no hazard symbol, when highlighting preferred practices, or to warn of possible damage to the instrument, if instructions are not adhered to.

Hazard symbols:

If the nature of the hazard is not marked on the instrument, the user should consult this document.

Electric shock:



High voltage: Indicates any hazard relating to voltages that present a risk of electric shock.

Heat:

Hot surface: Indicates any hazard relating to hot surfaces that present a risk of burn injuries.

Corrosive chemicals:

Corrosive substance: Indicates any hazard whereby contact exposure can cause permanent damage to human tissues, such as eyes and skin.

Irritants:

Harmful or irritant substance: Indicates any hazard whereby the operator may be exposed to substances that may be harmful or cause irritation or inflammation to the eyes, skin or respiratory system.

Toxic substances:

Toxic substance: Indicates any hazard whereby the operator may be exposed to materials causing immediate and serious, potentially fatal, toxic effects.

General hazards:

General hazard: Indicates general hazards.

Magnetic field hazard:

Magnetic field: Indicates any magnetic field hazard.

Crush hazard:

CAUTION

Crush hazard: Indicates a crush hazard with moving parts or the handling of heavy objects.

Fire hazard:

CAUTION

Flammable gas: Indicates any hazard involving the use of flammable gas.

VI. Specific safety information

i. Magnetic field hazards

The instrument operates with a magnetic field intensity approaching 1 tesla between the electromagnet pole pieces. Do not insert objects constructed from magnetically permeable material into the gap between the top and bottom magnet coil assemblies whilst the magnet circuit is switched on. A high mechanical force will be exerted on any such materials between the pole pieces, when the magnet is energized.



WARNING

Magnetic field: Current static magnetic field guidelines restrict exposures for wearers of cardiac pacemakers to 0.5 mT.

Persons with pacemakers or other implanted electronic devices should keep at least 20 cm away from the edge of the magnet coil assemblies.

ii. Heat hazards

The instrument rotary pumps generate heat.

During the instrument bakeout procedure, heaters operating at up to 120 degrees Celsius are used.



⚠ CAUTION

Hot surface: Approved maintenance engineers or system specialists must ensure that the rotary pumps have cooled before conducting any pump maintenance procedures.



⚠ CAUTION

Hot surface: Approved maintenance engineers or system specialists must ensure that the bakeout heaters and any corresponding instrument sections have cooled before removing the bakeout covers.

iii. Crush hazard

During the bakeout procedure it is required that the electromagnet is moved to the back of the bench.



⚠ CAUTION

Crush hazard: Approved maintenance engineers or system specialists must take care when moving the electromagnet, to avoid trapping fingers behind the magnet during movement.

iv. Corrosive substance hazards



⚠ CAUTION

Corrosive substances: Corrosive substances may be used during service of the instrument. Approved maintenance engineers must take appropriate precautions when handling and disposing of them.

v. Gas hazards



⚠ CAUTION

Irritant gas: Operators and approved maintenance engineers must take appropriate precautions if irritant gases are used in conjunction with the instrument. It must be ensured at all times and especially during site preparation that extraction is sufficient and operational.



⚠ CAUTION

Crush hazard: Operators and approved maintenance engineers must take appropriate precautions when moving compressed gas cylinders.



⚠ CAUTION

Flammable gas: Ensure that the exhaust line is safely vented out of the laboratory, in accordance with local health and safety regulations.



⚠ DANGER

Toxic gas: Ensure that the exhaust line is safely vented out of the laboratory, in accordance with local health and safety regulations.

vi. High voltage hazards

High d.c. and a.c. voltages are present throughout the system.



DANGER

High voltage: Various electronic units within the Perspective are powered by or generate high voltage d.c. and a.c. voltages. Always isolate or disconnect the mains supply before removing the covers of the mains distribution unit (MDU) or any electronic units.



DANGER

High voltage: Dangerous high voltages are supplied to the source. Only trained personnel should access the source.



CAUTION

Trip hazards: Potential trip hazards from electrical cabling, water hoses, air line, vent line and gas lines. Ensure that only trained operatives have access to the instrument service area. Ensure that all lines are routed safely away from the instrument.



CAUTION

Slip hazards: Potential slip hazards from water spillage or broken water connection. Ensure that only trained operatives service the water chiller. Ensure that water lines are routed away from walkways and any water spills/leaks are cleaned up immediately to minimize any potential slip hazard.

VII. General safety information

i. Instrument safety notices

In addition to the warnings contained in this document, users must also observe all warnings and instructions on all notices fixed to the instrument, accessories and any devices associated with the operation of the instrument.

ii. Protective clothing

Appropriate safety clothing must be worn at all times whilst operating the mass spectrometer, particularly when handling hazardous materials such as corrosive solutions or gas bottles.

iii. Environment

The installation requirements, and the environmental conditions required for the safe operation of the Perspective, are detailed in the Site Requirements document.

iv. General maintenance

Only suitably qualified personnel should perform maintenance procedures. Substituting parts or performing any unauthorized modification to the instrument may result in a safety hazard. Compliance with all local and national requirements for electrical and mechanical safety is required.

Tools provided with the instrument are for use by engineers and other suitably qualified personnel. Always consult Nu Instruments before using the tools for the first time, to ensure that they are intended for your use. Only use tools for tasks described in the Maintenance chapters of the System Manual and only use the tools in the way that the manual describes. Nu Instruments can accept no responsibility for damage or injury caused by incorrect use of the tools.

v. Routine maintenance

The routine maintenance procedures described in this manual must be conducted in accordance with the safety information provided herein. This instruction applies to all maintenance tasks.

vi. Gas supplies

The condition of all gas tubing connected to the instrument should be regularly examined, for example to detect stress cracks. The user must carry out appropriate leak tests on any gas and liquid connections assembled during normal use or maintenance.

Any special national or local regulations for the safe use of gases and gas cylinders must be observed. The user must ensure that the type of connector used on the outlet side of pressure regulators conforms to applicable national or local requirements.

vii. Extraction lines

The condition of all vent lines should be regularly examined, for example for blockages or cracks.

Any special national or local regulations for the venting of exhaust gases out of the laboratory must be followed.

vii. Use of chemical reagents

Nu Instruments Ltd. is not aware of any substance that is more hazardous, when used in conjunction or in contact with the Perspective, compared with its use when not in conjunction or contact with the Perspective but this possibility is not precluded. Please contact Nu Instruments, if in doubt. It is the responsibility of the user to manage risks arising from the nature of materials being tested, for example radioactive samples.

viii. Hazardous waste material

The user is responsible for the collection and safe disposal of waste materials. The operator should note that potentially hazardous waste materials may be present and must be properly disposed of in accordance with national and local guidelines.

ix. Cleaning and decontamination

It is the responsibility of the user to carry out appropriate decontamination, if hazardous material has been analyzed, or spilled onto or inside the instrument. If the user intends to use any cleaning or decontamination methods that are not specified in this manual, please check with Nu Instruments Ltd. that the proposed method will not damage the instrument.

Nu Instruments Ltd. must be notified if any equipment being returned has been in contact with any hazardous substances. Nu Instruments Ltd. will not accept delivery of any equipment that has been contaminated, unless documentary proof of decontamination is provided. Fill in and sign the **Health and Safety Declaration** form and include the form with all pre-approved returns.

x. Disposal

Safely dispose of all equipment and any components in accordance with all local and national safety and environmental requirements.

VIII. Electrical power, gases and cooling water requirements

All power, gas and cooling water input / output requirements are listed in the **Site Requirements** document. Your Site Requirements document is specific to your region, particularly with respect to the electrical requirements and so this information is not specifically included in this manual.

The installation of the instrument can only proceed when the requirements of the Site Requirements document have been met. After installation and acceptance of the instrument, no changes should be made to electrical, gas or cooling water supplies to the instrument, unless they comply with the Site Requirements document.

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Chapter 1: System overview

1.1 Overview

The principal components of the Nu Instruments Perspective IRMS mass spectrometer will be described in outline in this chapter.

The Perspective is a sensitive stable isotope ratio mass spectrometer, under full software control. The instrument design and the materials used in its construction allow the precise measurement of isotope ratios of gases up to mass 150 in both dual inlet (DI) or continuous flow (CF) modes.

The Perspective has a Nier type electron impact ionization source, which can operate up to a maximum beam voltage of 8 kV. The differentially pumped source region incorporates two turbo-molecular pumps, with a third located in the collector region.

The mass separation is accomplished by an electromagnet. Two ion focussing (quad) lenses give a 60 cm effective radius. Simultaneous multiple ion beam collection is provided, with two Faraday collector configurations available, depending on the required applications. One configuration has 6 Faraday collectors and the other has 10 Faraday collectors.

Each Faraday collector has its own dedicated pre-amplifier. The pre-amplifiers are mounted in an evacuated, temperature-controlled bin for improved stability. The instrument is under full computer control, with a fully bakeable vacuum system.

The Perspective stable isotope ratio mass spectrometer can operate in two modes, continuous flow (CF), in which the sample is introduced into the instrument in a stream of helium carrier gas and dual inlet (DI), in which pure sample gas is used. The Perspective measures the ratio of isotopes in a variety of gas samples, which can be introduced to the instrument from a number of preparatory techniques. Continuous flow techniques include Elemental Analysis (EA), Gas Chromatography (GC) and a multi-sample gas preparation system (Gas Prep). In Dual Inlet mode, the Nu Carb system for the analysis of carbonates and a manifold system are commonly used.

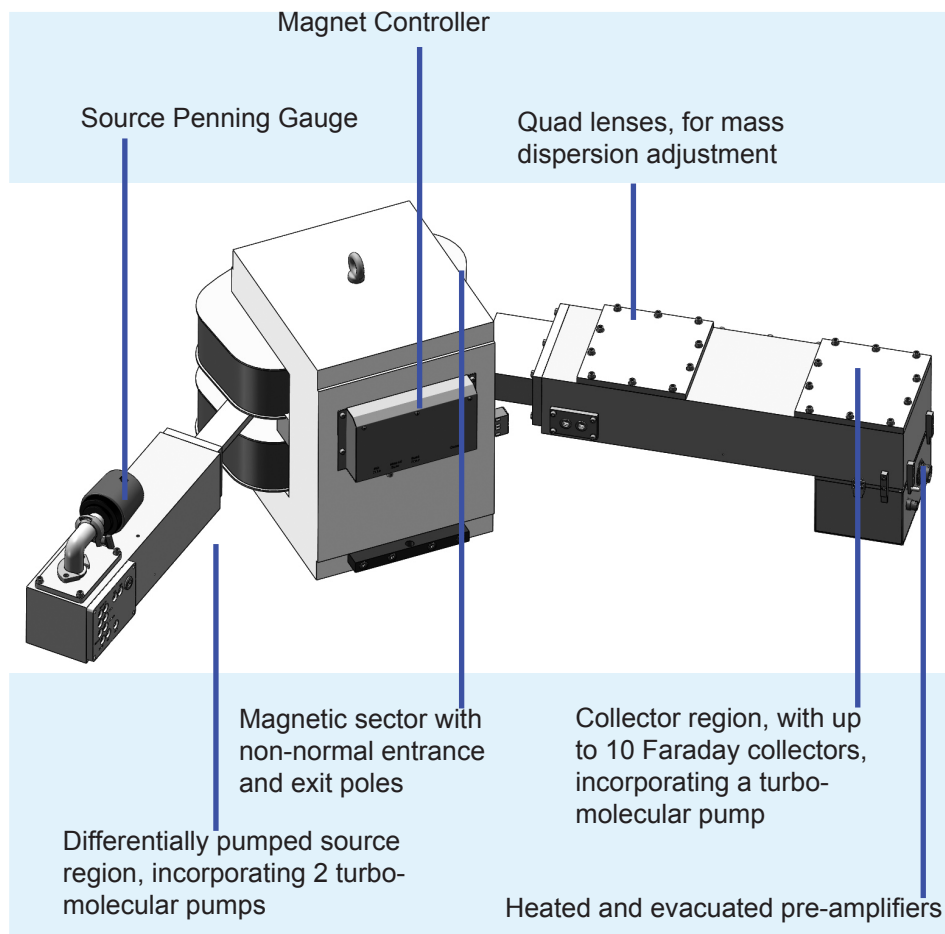


Figure 1.1-1: Schematic diagram of the Perspective above bench components.

The technique of stable isotope mass spectrometry does not measure absolute ratios of isotopes, but compares the isotope ratios measured in samples to the ratios in an international reference gas standard. The results reported are the difference between the two, usually in parts per thousand, also referred to in units of “per mil”, denoted ‰.

To measure the isotope ratio, a gas is introduced into the source, where a proportion of it is ionized, (approximately one ion per 800 molecules), by electron impact. The electrons are thermionically emitted by a hot filament and are accelerated through the source where they collide with some gas molecules, causing ionization by loss of an electron. Once formed, the resulting positive ions are accelerated out of the source through a potential of several thousand volts. The ions are then focused into a tight beam by electrostatic focusing plates and steered out of the source into a differential pumping region.

The differential pumping region allows a relatively high pressure to be maintained in the source region, while maintaining a much lower pressure in the rest of the instrument. This is important because (up to a limit) the more sample gas that can be introduced into the source, the larger the ion beams, which makes them easier to measure. On the other hand, once the ions have left the source they can be scattered out of the beam by collisions with residual gas molecules, and hence lost to measurement. For this reason, the fewer residual gas molecules present the better, so a total of three turbo-molecular pumps are provided, one directly beneath the ionization source, the second at the differential pumping region and the third in the collector region. All three turbo-molecular pumps are backed by a single rotary pump.

When the ions leave the differential pumping region they enter the flight tube, between the poles of the electromagnet. When the ions travel through a magnetic field they follow a curved trajectory, the radius of which depends upon the mass of the ion. Ions of all masses enter the flight tube following the same path, but each isotope leaves it following a different one, with lighter ions being deflected more by the magnetic field.

For ions of a particular mass, the path out of the flight tube is divergent, so the ions need to be refocused onto the entrance slit of the collector being used to measure ions of that mass. This second focussing is performed by a pair of electrostatic lenses (called Quad lenses), located in the analyzer housing close to the flight tube. The ions of each isotope are collected in separate Faraday collectors. Resulting signals are amplified and sent to the system computer for processing and reporting.

1.2 Ionization source

The Nu Instruments Perspective Nier-type electron impact ionization source is designed for both dual inlet and continuous flow application. The source is illustrated in the diagram below.

A filament, made from tungsten or thorium coated iridium, emits electrons, which are accelerated towards the ionization region. Small permanent magnets on either side of the source provide a vertical magnetic field in the ionization region, collimating the electron beam and causing the beam to follow a spiral path, thus increasing the path length and the chances of collision with gas molecules. After leaving the ionization region, the electron current is sampled by an electron trap. The measured current, known as the trap current, is usually about 500 microampere. The trap current is used to regulate the filament heating current, so as to ensure a stable ion beam.

Positive sample ions generated in the source are repelled by a positively charged repeller plate, directing them out of the source block in a trajectory perpendicular to the direction of the electron beam.

Extraction of positive ions is further aided by the half plates, which are situated directly after the source block. These are set to a lower voltage than the source block and can have a differential voltage applied, for horizontal steering.

Immediately after the defining slit are the Z plates. Equal positive voltages applied to both pairs of plates provides vertical focussing; a voltage difference between top and bottom pairs is used to provide vertical steering.

The final plate in the source assembly is a Horizontal deflect plate. It is used in conjunction with the differential voltage on the half plates, to provide horizontal steering of the ion beam.

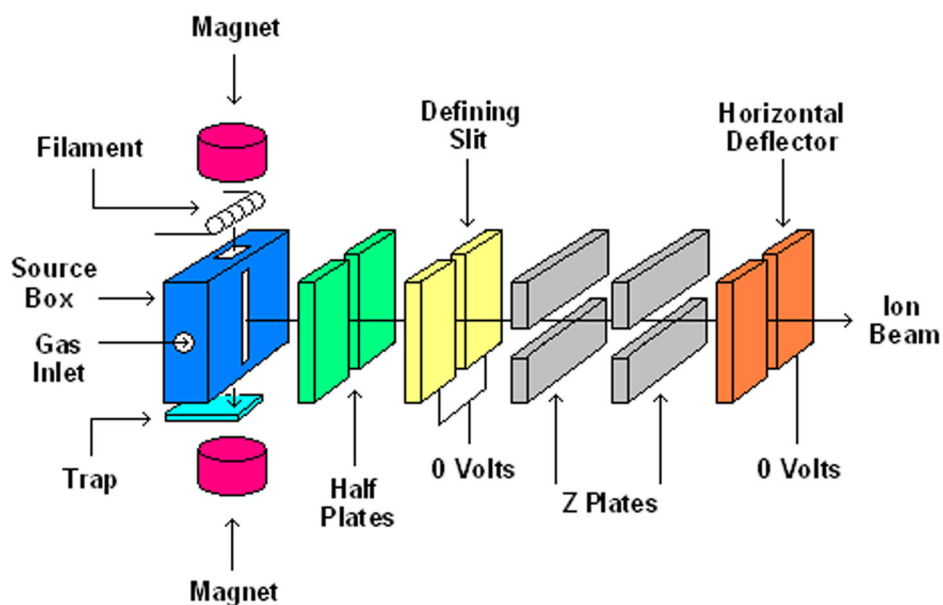


Figure 1.2-1: Schematic of the Nier-type source.

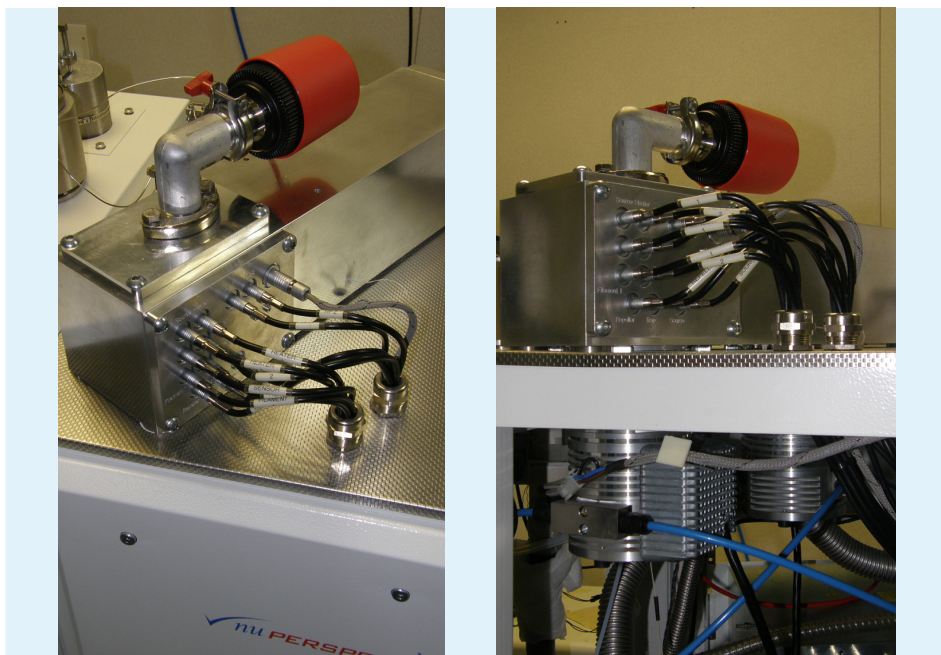


Figure 1.2-2: Perspective source region and Penning gauge, with source and differential turbo-molecular pumps below the bench.

1.3 Inlet valve

A pneumatically operated isolation valve enables the user to switch between Dual Inlet and Continuous Flow (DI and CF) modes, without the need to make any manual changes to gas connections in the source region. If no dual inlet is fitted, the valve port is blanked off. When the isolation valve is closed, gas is admitted from the dual inlet to the source. When the valve is open, gases from the reference gas system and from the sample preparation systems are allowed to enter the source.

1.4 Magnetic sector

Most of the focusing of the ion beam shape, in the X, Y and Z directions, is achieved after the source region. This is performed by the shapes of pole-pieces at the entrance and exit of the magnetic sector and by number electrostatic lens elements. The single focussing magnetic sector design has non-normal entrance and exit poles to provide a dispersion of the ion beams at the collector plane.

⚠ CAUTION

The magnet is supported in three positions, two towards the front of the instrument and one to the rear. These positions are height adjusted on installation and they are critical to the trajectory of the ions through the flight tube. Although the adjustments are clearly visible and accessible, they are not normally adjusted by the user.

The magnet separates ions according to their mass but also acts as a focusing device, forming ion images of the source slit at the collector slits. The shapes of the magnet pole pieces at the magnet entrance and exit are critical to this focusing and allow the focus positions of the different dispersed masses to lie in the same plane, approximately perpendicular to the beam axis.

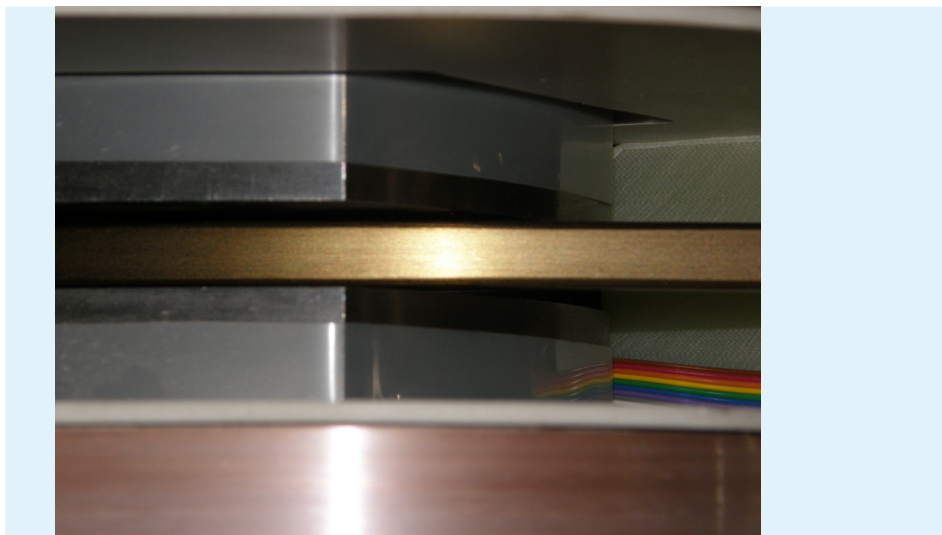


Figure 1.4-1: Flight tube entering the magnetic sector.

1.5 Zoom optics

Between the magnet exit pole and the collector are two lens arrays, known as the Quad lenses. These arrays act as a zoom lens, permitting the user to adjust the mass dispersion. This patented feature allows a fixed collector array to be incorporated, eliminating the requirement to move any of the individual detectors from one isotopic application to the next.

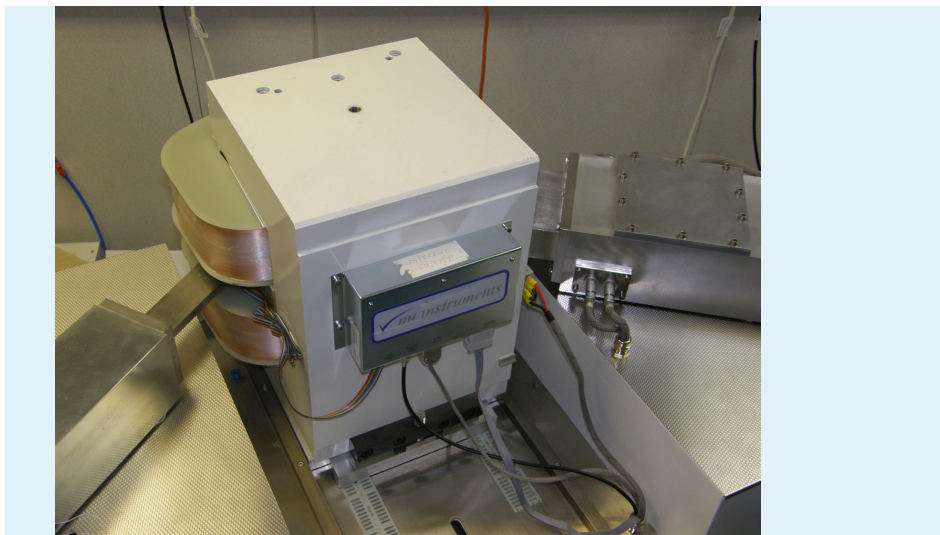


Figure 1.5-1: Magnet and quad lens arrays.

With the zoom lenses switched off, the image plane coincides approximately with the collector slit plate. A small amount of focussing by the quad lenses is usually required to obtain good peak shapes. Then, by adjusting the two lenses together, it is possible to change the dispersion.

The quad lenses are very versatile and as well as controlling the dispersion, they also allow fine adjustment of the ion beam focussing.

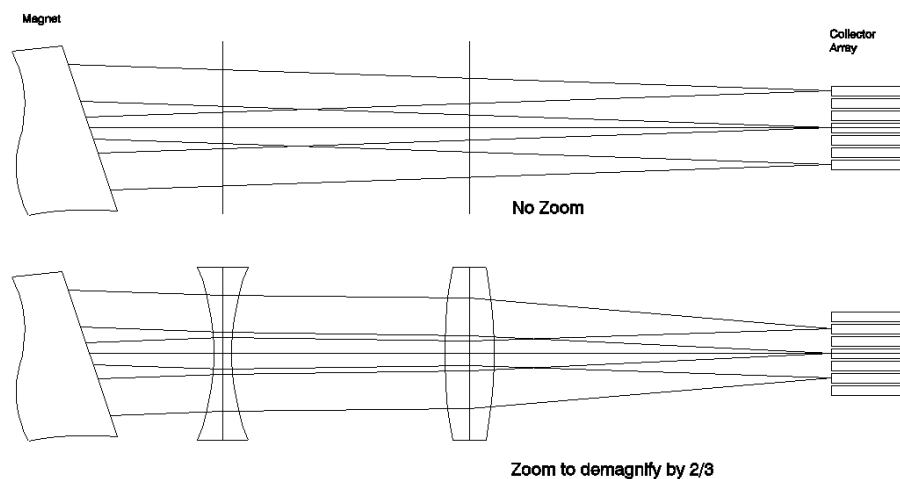


Figure 1.5-2: Dispersion adjustment by the quad lens arrays.

1.6 Collector configurations

Data acquisition is carried out using simultaneous multicollection. Several configurations of collectors are available, depending on the applications required of the instrument. The most common configuration, shown in the diagram below, consist of 6 collectors, four of which are used in applications involving CO₂, N₂, SO₂ and CO. The outer two collectors are dedicated to the measurement of H₂ and HD at masses 2 and 3. The HD collector is fitted with a curved lens, acting as an energy filter to eliminate low energy ions of a higher mass.

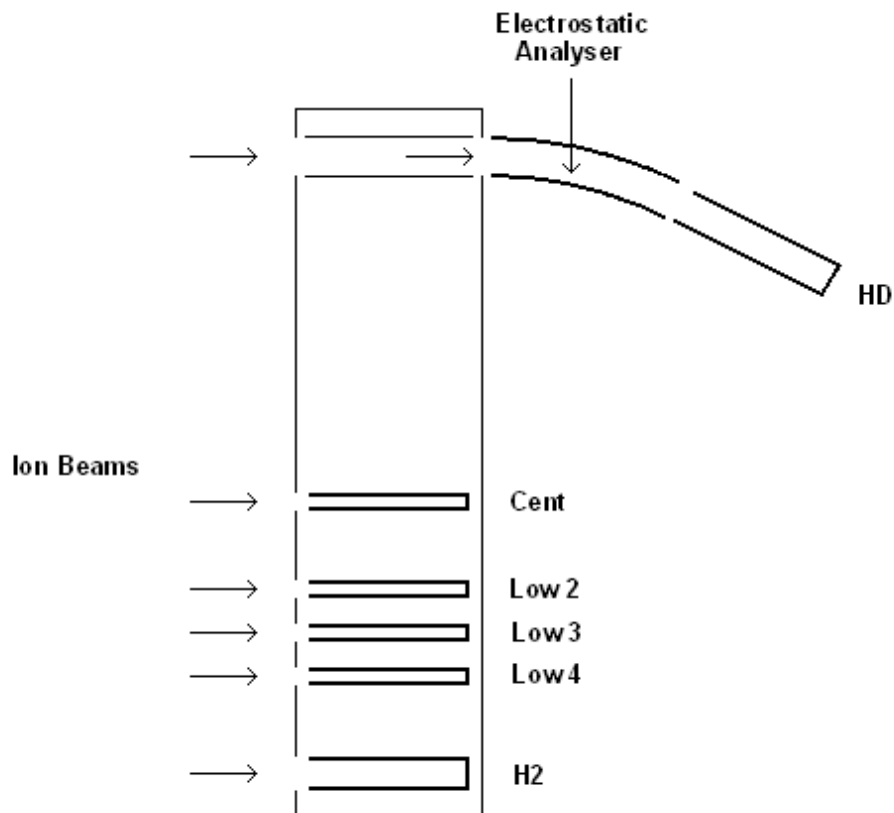


Figure 1.6-1: The 6 Faraday collector configuration.

A 10-collector configuration, illustrated below, includes four such high mass collectors, with curved lenses for the elimination of low energy ions. The outer two collectors are again used for the measurement of H₂ and HD at masses 2 and 3. The three collectors below the HD collector are primarily used to measure masses 47, 48 and 49, all low abundance CO₂ molecular ions. These require detection using the curved lenses for elimination of low energy ions of higher mass.

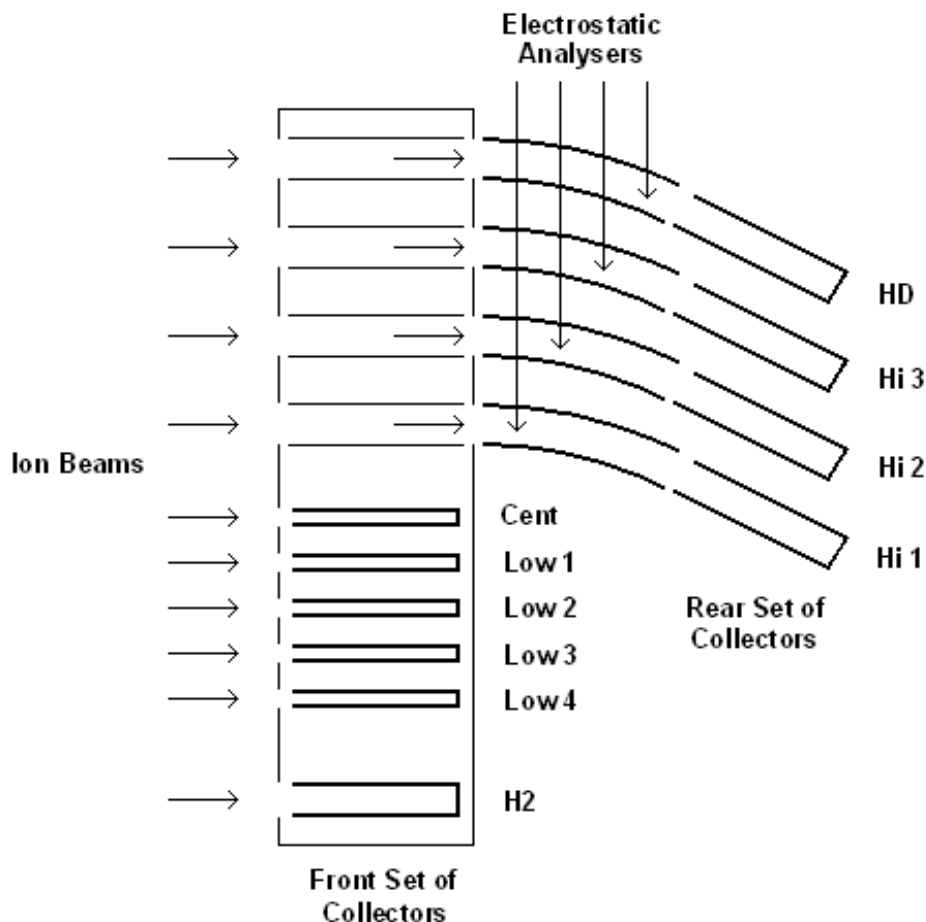


Figure 1.6-2: The 10 Faraday collector configuration.

The choice of multicollection strategies depends on the application and the size of samples available. The table below illustrates the way that the collectors are used in a number of applications. The table includes SO₂ and SO measurements, which could both be measured during SO₂ applications, since a proportion of the SO₂ gas in the electron impact ionization source will fragment to form SO.

Gas	H2	Low 4	Low 3	Low 2	Low 1	Cent	Hi 1	Hi 2	Hi 3	HD
CO		28		29		30				
CO ₂		44		45		46	47	48	49	
H ₂	2									3
N ₂		28		29		30				
N ₂ O		44		45		46	47	48		
SO		48		49		50				
SO ₂		64	65	66						

Figure 1.6-3: Collector Designation, the entries in the table are the cardinal masses of the isotopes.

1.6.1 Collector preamplifiers

Some of the collectors are only used for specific applications, whilst others may be used for a wider range of applications and typical beam sizes. Each collector has a dedicated pre-amplifier, with the optimum gain resistor to suite the application or range of applications expected. The standard values of the gain resistors and associated maximum beam currents are listed in the table below.

	H2	Low 4	Low 3	Low 2	Low 1
Gain (GΩ)	1	1	100	100	100
Max Current (A)	5×10^{-8}	5×10^{-8}	5×10^{-10}	5×10^{-10}	5×10^{-10}

	Cent	Hi 1	Hi 2	Hi 3	HD
Gain (GΩ)	100	1000	1000	1000	100
Max Current (A)	5×10^{-10}	5×10^{-11}	5×10^{-11}	5×10^{-11}	5×10^{-10}

Figure 1.6-4: The standard gain resistors and maximum beam currents.

1.6.2 Suppressors and mop plates

Suppressors and mop plates are devices which prevent the entry of stray ions and electrons into the Faraday collectors, which would affect the accuracy and precision of measurements.

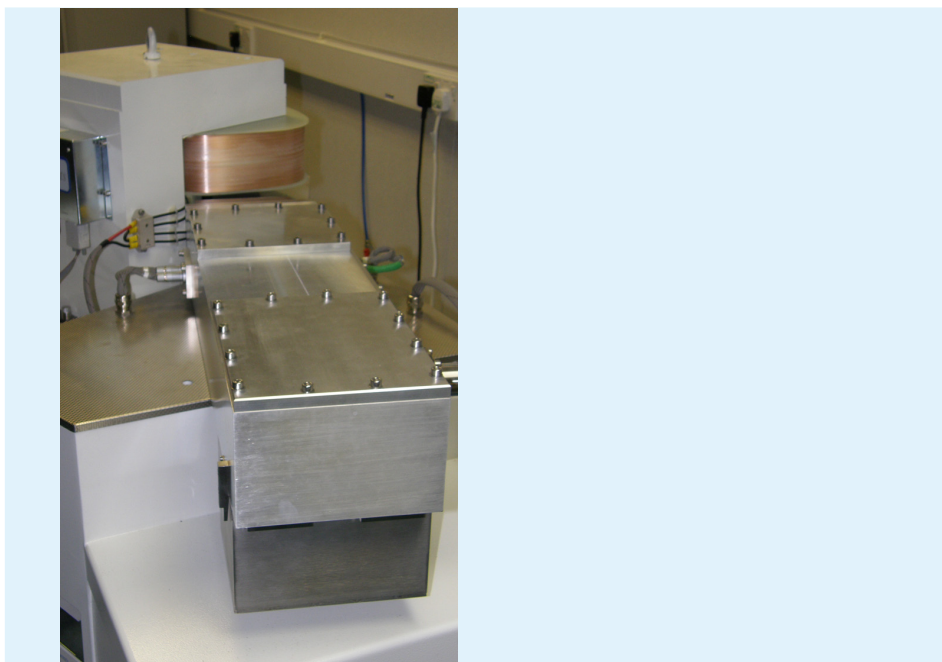


Figure 1.6-5: The collector region.

Unwanted stray ions may be measured directly if they enter a collector, or they may also collide with any internal surface of the mass spectrometer. During such collisions, low energy electrons may be produced, which may also enter a collector. This would be of particular concern when measuring very low abundance ions, in which case the stray ions or electrons would have a greater proportional effect on the apparent beam intensity.

In front of each Faraday collector is a suppressor, which ensures that low energy electrons do not enter the collector. A negative potential, usually around -50 Volts, is applied to the suppressor, repelling the low energy electrons away from the collector. There are also two ground plates in front of and behind the suppressor.

The suppressor also serves an additional function. When a high energy analyte ion enters the collector, the ion impact at the base of the Faraday cup produces secondary electrons. If a secondary electron is allowed to exit the collector, the contribution of the analyte ion to the preamplifier response would be counted twice; once when the positive ion enters the collector and then again if a negatively charged electron were to exit the collector. The suppressor plate repels secondary electrons generated inside the Faraday back into the collector.

The mop plate is directly in front of the collector array, before the suppressor plate. It has a positive voltage, typically around +50 V. Its main purpose is to repel stray ions that may approach the collector from a trajectory that is outside the normal path of the incoming ion. A secondary purpose is to attract low energy electrons and prevent them from entering the collector array.

1.6.3 Collector magnets

Beneath the collectors are six small permanent magnets, aligned so that their field is in the same sense as the main magnet. Three are under the front set of collectors and three under the rear set.

As described in the previous section, when an ion enters a collector, it is involved in a high energy collision with an internal surface of the collector, producing a shower of electrons. These electrons must remain within the collector because the response of the pre-amplifier to a negative electron leaving the collector is exactly the same as a positive ion arriving, leading to errors in measurement. The magnets cause the electrons to move in curved paths, increasing the probability that they hit the side wall of the collector and hence cannot escape.

These magnets have little effect on the analyte ions entering the Faraday collector, since these ions have high energies and have much more mass than the secondary electrons.

1.7 Vacuum control

The source region is differentially pumped, with a source turbo-molecular pump and a differential turbo-molecular pump. The analyzer region also incorporates a third turbo-molecular pump. All three of these pumps are identical and all three are backed by a single rotary vane pump. All of the pumps are within the instrument, but the backing pump is mounted directly to the floor, within the instrument enclosure, to eliminate any vibration. The overall arrangement of the vacuum system is shown in the figure below.

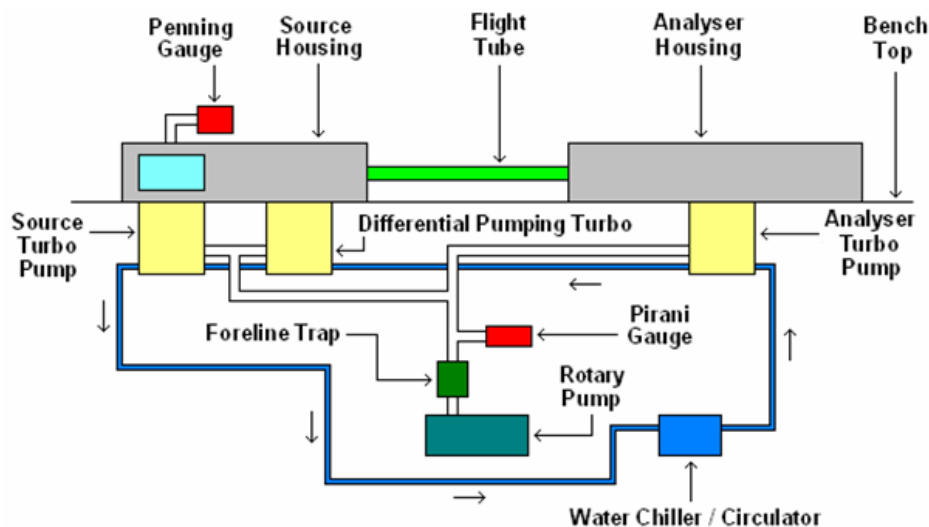


Figure 1.7-1: Schematic diagram of the pumping system.

A Penning gauge monitors the source housing vacuum and operates in the high vacuum region from 10^{-4} to 10^{-9} mBar. A Pirani gauge measuring the low vacuum region down to 10^{-3} mBar is positioned in the backing line between the rotary pump and the three turbo-molecular pumps.

The Pirani gauge may be constantly monitored at all pressure conditions and does not need to be switched off, even when the source region has been vented to atmospheric pressure. The Penning gauge, however, is only switched on when the Pirani gauge pressure reaches 5×10^{-2} mBar or below. This would normally be achieved after 5 minutes of pumping from atmosphere on the rotary backing pump.

The foreline trap is a small chamber close to the rotary pump, containing activated alumina spheres a few millimeters in diameter. These alumina spheres absorb oil vapor from the rotary pump and prevent backstreaming into the turbo-molecular pumps.

A water chiller/circulator cools the turbo-molecular pumps. The cooling for the pumps is connected in series, with the coolant supplied firstly to the pump in the Analyzer region and then to the Differential and Source pumps. If a Dual Inlet is also installed, the turbo-molecular pump on the dual inlet is also included in the circuit, usually after the Source turbo-molecular pump.

It is recommended that the operating temperature of the chiller be set to 15°C. This is to avoid condensation on the pipes within the body of the instrument. In humid conditions, the set temperature may need to be increased if condensation forms on the cooling circuit within the instrument enclosure.

It is recommended to inspect the visual flow meter regularly, to ensure that water is flowing to the instrument. This flowmeter is attached to the outlet of the instrument, between the instrument **Water Out** and the chiller **Water In**.

If the cooling water supply should fail for any reason, the turbo pumps will continue to function normally and there is no need to take any immediate action. The fault should be rectified as soon as is convenient, in order to ensure that the lifetime of the pumps is not reduced.

⚠ CAUTION

Inspect the visual flow meter regularly to ensure that there is water flowing to the instrument.

The exhaust from the rotary backing pump is connected via a polymer tube to a tail pipe on the rear panel of the instrument. This allows oil vapor and other exhaust gases to be vented safely outside the laboratory.

⚠ CAUTION

It is recommended that all exhaust gases are safely vented out of the laboratory, in accordance with local regulations.

1.8 Instrument control panels

The following section describes the instrument control panels. A number of specially designed electronic units are used on this instrument.

The units present in the instrument are:

Mains Distribution Unit (MDU): The main power input enclosure also contains the bakeout output and control area.

Source power supply unit: Contains all of the filament supplies and related source outputs. Also contains two high voltage units to provide the HT for the mass spectrometer source.

HV1000/16 V power supplies: Either two or three HV1000 units are supplied with each instrument, depending on the configuration. They each have 16 channels of programmable +1000 V to -1000 V output and are used for the horizontal deflector, the voltages on the quad lenses and the suppressor voltages for the Faraday array.

Magnet slave: With the Hall probe and magnet control (mounted on the rear of the magnet), the magnet slave is used to control the field in the magnet.

Magnet control: Contains a precision digital to analog converter and Hall probe amplifier, to control the magnetic field.

Preamplifier Bin: Houses the Faraday preamplifiers and the connection circuitry for the Faraday collectors.

System Controller: Communication between the controlling PC and any unit on the instrument is directed via the system controller.

Micro Rack: Houses the System Controller card, Dual Inlet and Continuous Flow cards and supplies power to the preamplifiers.

1.8.1 Mains Distribution Unit (MDU)

The MDU is located at the rear of the instrument, below the magnet. The mains power input to the instrument is via the MDU, which then distributes this mains power around the instrument. Most of the electronic units in the Perspective require input of 24 Volts and this is also supplied by the MDU.



Figure 1.8-1: Location of the MDU and source power supply.

The **Source** keyswitch is a secondary safety measure. When the keyswitch is off (service), all high voltage supplies will be isolated. A number of the high voltage supplies can be switched off individually using manual switches. These primary interlock switches turn off the 24 V output to the electronics that provide the specific high voltage supply.

On the panel is a series of switches. From left to right these are;

Power On/Off:	Switches the mains power on and off.
Circulator Power:	This turns on the chiller/circulator. This provides water cooling to the turbo-molecular pumps. There is a dedicated socket on the MDU panel for the circulator.
Rotary Pump:	Turns on the rotary backing pump.
Turbo:	Turns on the turbo-molecular pumps. This switch is operative only if the rotary pump is on.
Micro rack:	Switches on the power to the instrument control systems, the data handling and the communications.
Magnet:	This turns on the magnet controller and magnet slave unit. The slave unit is a low voltage, high current supply that provides the current for the electromagnet.
HV Supplies:	Turns on the power supplies for the HV1000 supplies.
Source Filament:	This switches on the source power supply unit, but does not switch on the HT supplies within the unit.
Source HT:	Switches on the HT power supplies in the Source Supply unit. This switch will not operate unless the keyswitch is in the correct position. The keyswitch is a safety feature normally used during maintenance. If the keyswitch is turned off and the key removed, it is not possible to turn the source HT power supplies on.
Preamp Pump:	If the instrument is configured only for use in continuous flow mode, this switch operates a separate pump used to evacuate the preamplifier bin.
Bakeout:	This enables the bakeout facility. The bakeout is software controlled, but as a safety feature, this switch must be manually operated. Below the bakeout switch is a small socket for the flight tube temperature sensor and a power output for the flight tube heater.

The above switches are separately fused.

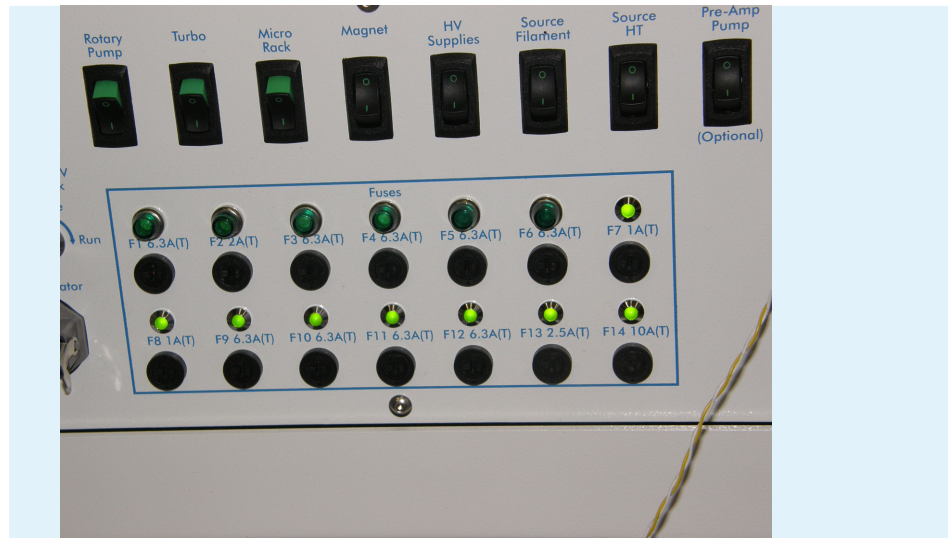


Figure 1.8-2: MDU panel - Fuses.

Fuse	Description
F1	24V PSU
F2	Electronics Rack
F3	Rotary Pump
F4	Heaters
F5	Chiller output
F6	Pre-Amp Bin Pump
F7	Pneumatics & Prep Bench
F8	Prep Bench
F9	Turbo 1
F10	Turbo 2
F11	Turbo 3
F12	Turbo 4
F13	Interlocks
F14	Source Supply & HV1000 Units

Figure 1.8-3: Fuse table.



Figure 1.8-4: MDU panel - Bakeout Control.

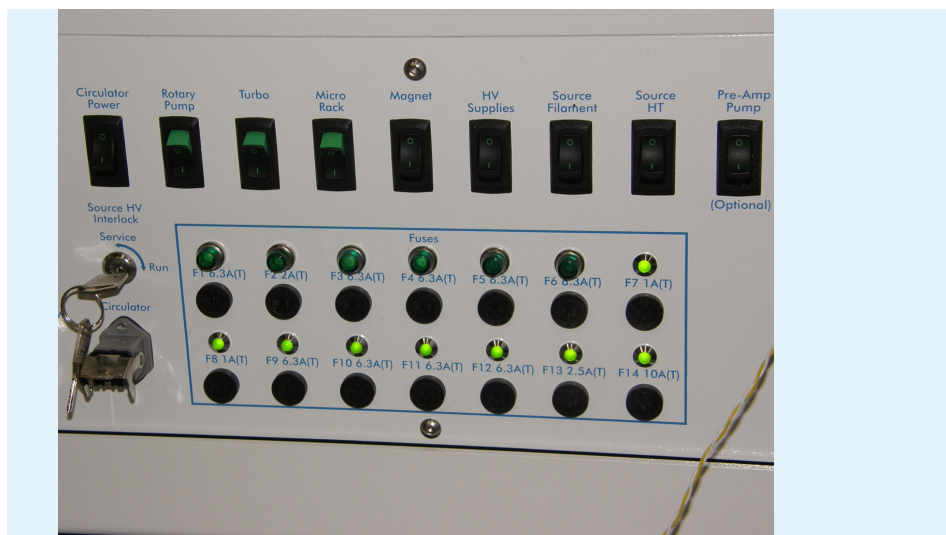


Figure 1.8-5: MDU panel - Individual switches.

1.8.2 Source Supply Unit



⚠ DANGER

The voltages inside this unit are potentially lethal. Even if the source HT and half plate extraction voltages are set to zero, the lid should not be removed.

**DANGER**

The input and output of the unit consists of 10 kV rated SHV connectors. Under no circumstances should the unit be operated with the connectors disconnected.

The Source Supply unit controls all of the high voltages to the source. The unit will operate continuously up to 10 kV, but normally supplies a voltage in the region of 8 kV. The Source Supply is a high precision current and voltage controller and incorporates the circuitry to resistively heat the filament, with a temperature sensor at the source potential. Output to the filament is controlled automatically by the software, using feedback from the trap current.

There are two switches on the MDU that control this unit. They are labelled **Source Filament** and **Source HT**. The Source Filament switch applies power to the unit. When this is switched on, a blue **Power** LED is illuminated. Under this condition, the power will be applied to the source, corresponding to the voltages specified in the **Tuning** window. Current will be slowly applied to the source filament and the orange status LED will flash approximately once per second, until the trap current is stable. Once the trap current is stable, the orange LED will be permanently illuminated.

The high voltage will not be applied to the source until the Source HT is switched on. A green LED indicates that the high voltage supply is operative. The high voltage is ramped up slowly to the value set in the Tuning window.

The orange **status** LED flashes at approximately 3 Hz when the source is initially turned on. This should only last for up to 30 seconds. If the flashing persists for a longer period, or if the LED starts flashing during normal operation of the instrument, this indicates that the filament is open circuit and needs replacing.

The source power supply is located at the rear of the instrument, above the MDU.

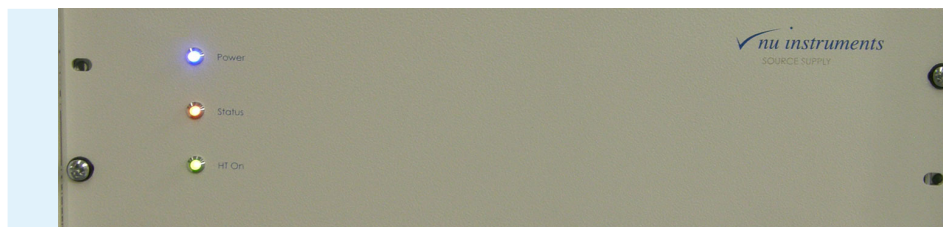


Figure 1.8-6: Source Supply Unit.

1.8.3 Magnet control unit

The magnet control consists of three separate parts, the Hall probe, the Magnet Controller and the Magnet Slave.

The Hall Probe measures the magnetic field. Its output is temperature-sensitive, so it is housed in a thermostatically controlled enclosure. It is located between the magnet pole pieces at the rear of the flight tube. The Hall Probe connects to the magnet controller via a 9 way 'D' plug.

The Magnet Controller is mounted on the rear of the magnet yoke, so as to minimize the possibility of interference pickup on the small controlling signals, and contains the Hall Probe reading and temperature control circuit, along with a precision 20 bit DAC and associated circuitry, to enable the magnet field to be set to the required value. There are four connectors on the Magnet Controller unit labelled **Hall Probe**, **Magnet Slave**, **Board Power** and **Comms**. The connectors are all different, so it is impossible to connect the cables to the wrong sockets.

The Magnet Slave unit is a high precision current controller, operating in the range from 0 to 25 Amps. The Magnet Slave supplies power to the electromagnet and is located in a 19-inch rack behind the front panel, beneath the HV1000/16 units.



Figure 1.8-7: Magnet Slave and HV1000/16 units.

The magnet slave is also connected to the magnet controller via two 13 way D-connectors. These cables carry power, as well as digital control signals to define the output requirements for the slave. Communication between the system control and the magnet control circuitry is via the RS422 connector, mounted on the rear of the magnet slave unit.

When in use, the system control defines the required magnetic field (using a 32 bit value sent out by the PC), and this is read by the Magnet Control unit. This measures the actual field, using the Hall Probe, and ensures that these two are same. This is achieved by the unit producing a small (0-5 V) demand voltage, which is sent to the Magnet Slave using the coaxial cable. This voltage is then used by the slave to control its output current.

When in use, the unit performs checks to ensure that its output is within a safe operating range. If the safe range is exceeded, the unit will limit the output or shut down, so as to protect itself.

Other connectors at the rear of the Magnet Slave are the mains input (240 V AC) and the magnet power connectors. These are polarized and the red positive output should go to the upper of the two magnet coil connectors, with the negative output going to the lower coil connector.

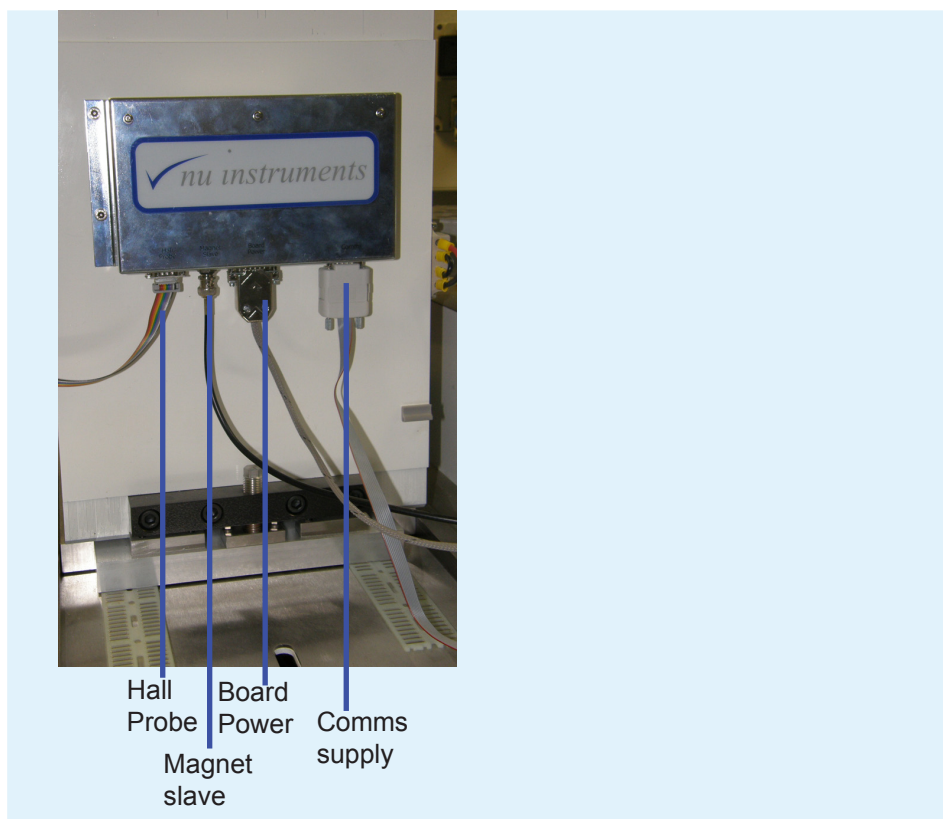


Figure 1.8-8: Magnet controller.

The **Magnet slave** supplies power to the electromagnet and is driven by the magnet controller.

1.8.4 High voltage supplies (HV1000 units)

**⚠ DANGER**

The voltage within the HV1000 units could be lethal and so it should not be opened for service work.

There are either two or three HV1000/16 power supplies provided with the Perspective. They are located in a 19-inch rack, behind the front panel beneath the analyzer housing. They each provide sixteen independently programmable dual polarity 1000 Volt outputs.

The HV1000/16 units require a 24 volt input, which comes from the MDU.

The sixteen outputs are assigned by labels on the rear of the unit, output 0 being closest to the 24 volt power input, output 15 furthest away. Also present on the rear panel is a 9 way 'D' plug which provides the RS422 connection to the system micro, and an earth stud.

The HV1000 units provide the voltages for the Quad lenses and the ES filters. The outputs from the units are controlled from the **Quad** panel in the **Tuning** window. The units can provide linear, quadratic and cubic potential distributions - or combinations of these.

Although the HV1000/16 supplies are identical, each has an individual address set, so if the units are interchanged, the addresses must be reset, using the DIP switches on the right hand side of each unit. Note down the DIP switch settings before interchanging any of the HV1000/16 units.

The 24 V connector has the following connections:

Pin 1	Ground
Pin 2	+24 V
Pin 3	0 V

HINT: Pin 3 lies between pins 1 and 2 for this connector.



Figure 1.8-9: HV1000/16 units and Magnet Slave units.

1.8.5 The Micro Rack

The Micro Rack is located behind the small side panel at the right hand end of the instrument, below the pre-amplifier bin. It contains several electronic cards, depending on the system configuration.

System Controller: The left hand slot (slot 0) contains the System Controller card. This card must only be located in slot 0. The System Controller carries an onboard microprocessor which is in direct communication with the system computer and controls all of the other cards. Communication between the controlling PC and any unit on the instrument is directed via the System Controller card.

DVM Card: In slot 8 is the DVM card. This card provides the power for the pre-amplifiers and outputs a voltage proportional to the beam current collected in each amplifier.

Dual Inlet and Continuous Flow cards: To the left of the DVM card are the Dual Inlet and Continuous Flow cards. Depending on how many, and the type of preparation systems for which the instrument is configured, there may be multiple dual inlet and continuous flow cards. The dual inlet card controls all of the functions of the dual inlet preparation system and associated peripherals. The continuous flow card performs a variety of functions. It controls the reference gas system, monitors the pressure in the instrument and it monitors the speed and the temperature of the turbo molecular pumps. The card also controls the bakeout and pneumatic panels.

1.8.6 The Preamplifier Bin

This is the housing beneath the far right hand end of the analyzer housing and contains the high gain pre-amplifiers, one for each collector.

To achieve low background noise in the amplifiers, this housing is both temperature controlled, to a few degrees Celsius above room temperature, and evacuated. The temperature control is automatic. Whenever the instrument is switched on, the heaters and the control circuitry are activated.

There are two electrical connectors. The larger one brings the power for the amplifiers from the DVM card in the Micro Rack and takes the amplified signals from the amplifiers back to the DVM card. The smaller connector carries the power for the heater and the connection to the temperature sensor used for thermostatic control.

1.8.7 The Service Panel

The Service Panel is located on the rear of the instrument, to the left of the MDU. This panel is where various supplies enter and exit the instrument. This includes the water cooling input and output, compressed air input, reference gas input to the reference gas unit, helium carrier gas and the exhaust output from the rotary backing pump and reference gas unit. There is also an Edwards Speedivalve, used in the evacuation of the pre-amplifier bin (see section 2.4). A photograph of the service panel is shown below.

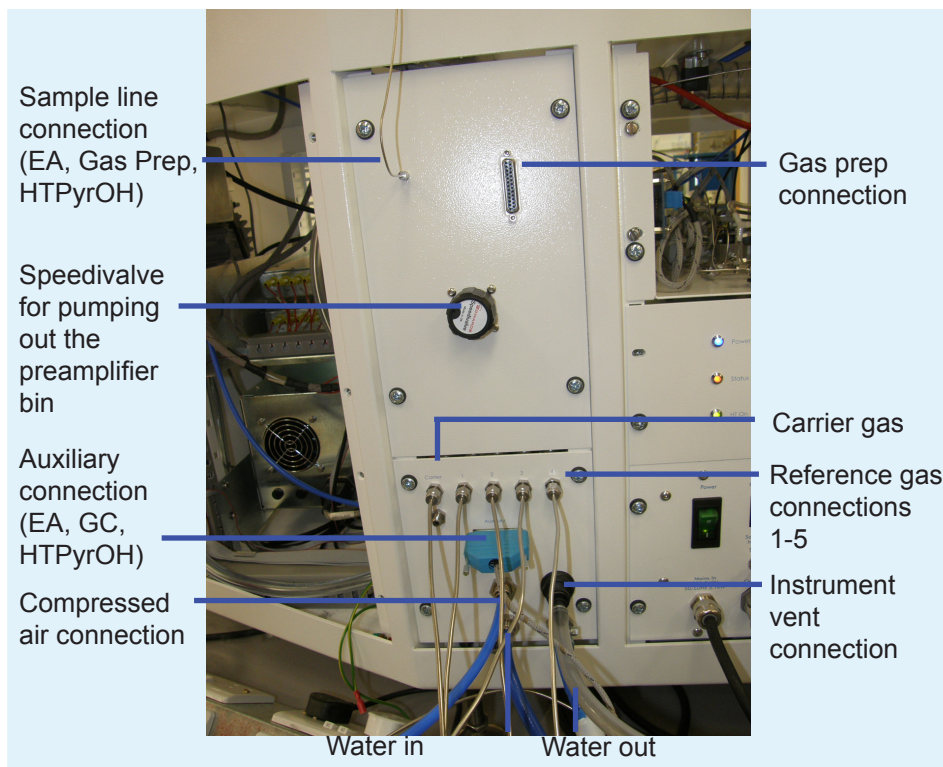


Figure 1.8-10: Service panel.

The following fittings are used on the Service Panel:

1/8 inch stainless steel Swagelok union for the reference gases and the helium carrier gas.

1/16 inch SGE union for the CF sample preparation systems.

8 mm ID barbed tail pipes for the water connections.

8 mm OD metric push-fit nylon connector for the compressed air.

10 mm ID barbed tail pipe for the exhaust from the rotary pump and reference gas unit.

25 way AUX connection for the EA/HT-PyrOH and GC

25 way GP connection for the Gas Prep

To the left of the service panel, the only other connection is that for the system computer. This is the socket on the rear of the instrument to the left of the service panel, below the pre-amplifier bin.

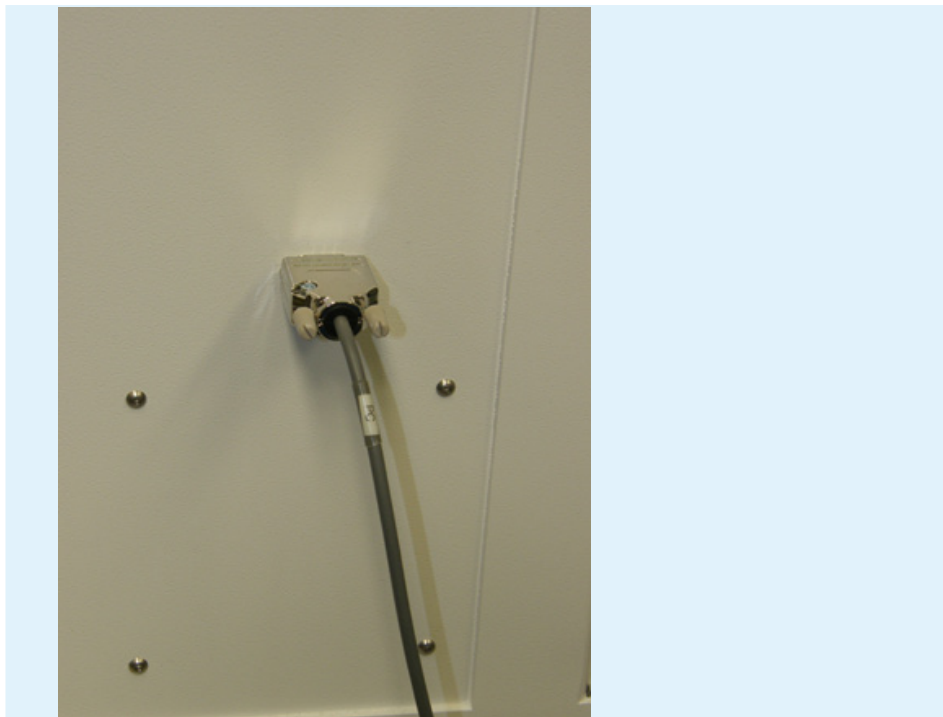


Figure 1.8-11: Connection to computer.

Chapter 2: Vacuum control procedures

Many vacuum system control functions, such as opening and closing of valves, venting of the system and bakeout are performed through the software.

The mass spectrometer chamber needs to be vented in order to clean the source or to exchange the filament. Whenever the mass spectrometer chamber has been vented for a long period, the bakeout procedure should be carried out afterwards, as described in detail in section **2.5 Bakeout procedure**.

There are five different status modes:

1. **Operate** mode: The source is on and the Mass Spec valve is open.
2. **Standby** mode: The source is on and the Mass Spec valve is closed. This Standby mode is recommended when the instrument is not in use, for example overnight.
3. **Safe** mode: The source is off and the Mass Spec valve is closed. This status must be adopted before the instrument can be vented.
4. **Vent** mode: The source is off and the instrument is vented. Maintenance procedures, such as cleaning the source or exchanging the filament, can now be performed.
5. **Shut down** mode: The instrument is completely powered down, which is required, for example, prior to scheduled power cuts.

HINT: Whenever the filament has been exchanged, a bakeout procedure can be carried out, as described in section **2.5 Bakeout procedure**, describing the venting and baking of the instrument. The user may also refer to these steps whenever there is a need to vent the instrument or perform a bakeout, even if these actions are required in a different context. The steps described will apply equally well. If the bakeout procedure has to be performed simply to improve the vacuum, the user may go straight to section **2.5 Bakeout procedure**, as the instrument does not need to be vented first.

HINT: The exchange of a filament is performed in the following steps:

1. Check if the filament is working correctly. If it is not, it needs to be replaced.
2. Vent the instrument. In order to replace the filament, the instrument must be vented to atmospheric pressure.
3. Exchange the filament. This will be explained step by step.
4. Pump down the instrument, which will be explained in detail.
5. Briefly check that the system is working.
6. Carry out the bakeout procedure.
7. Check that the new filament is working correctly.

The exchange of a filament is explained in detail in **Chapter 3 Maintenance**.

2.1 Turning off the ion source (from Standby to Safe mode)

This section contains step by step instructions for safely turning off the ion source. It is assumed that the instrument is in the **Standby** mode, with the source running and the mass spectrometer valve closed.

1. Go to **Mass Spec > Tuning > Load** to open the **Instrument Tuning Parameters** folder. All of the tuning parameters are stored in this folder.

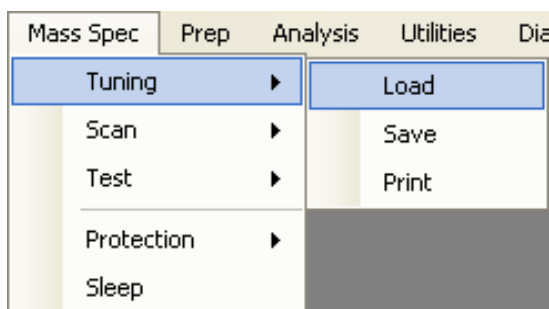


Figure 2.1-1: Mass Spec > Tuning > Load.

2. The **Instrument Tuning Parameters** folder opens. All of the tuning parameters are stored in this folder. Select the file **<SetToZero.Tun>** and click **Open**. This sets all instrument voltages and currents to zero.

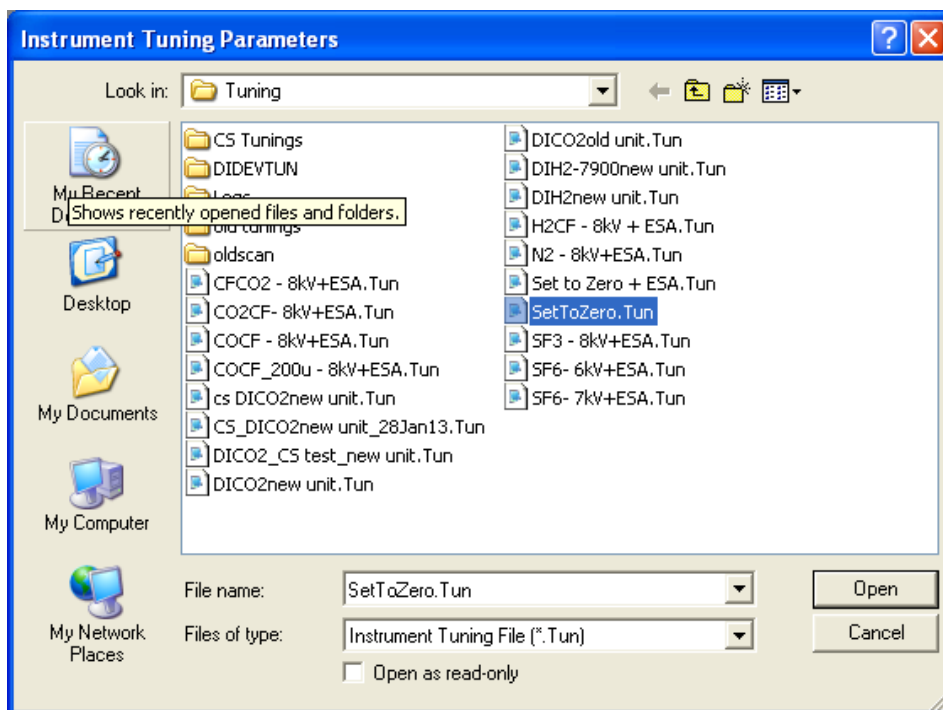


Figure 2.1-2: Instrument Tuning Parameters window with <SetToZero.Tun> selected.

3. Go to the **Digital > Power** tab. The status should show that all supplies are on (checkmark placed) except for **Bakeout ON** and **Fil In Control**. Click in the box of each parameter to switch them off, in the following order:

- HT
- Source
- Filament Supply
- Deflector Supplies
- DVM Supplies

All boxes are now unchecked. A message flashing at the top of the screen will state that the source is out of regulation. No action is required. The **Digital** window should appear as shown in the right hand image below.

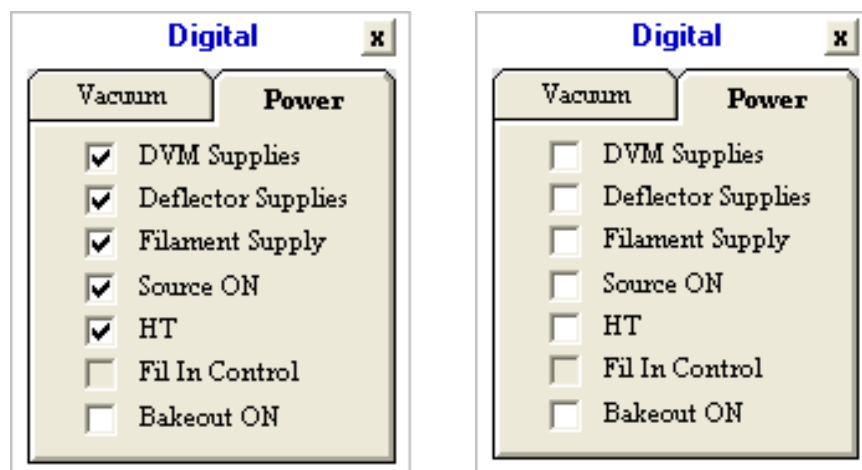


Figure 2.1-3: Switching off supplies in the Digital window

4. The actions in this step depend on whether the instrument is configured for continuous flow, for dual inlet only, or both.

a) If the instrument is configured as **continuous flow only**, the only requirement is to close the mass spec valve. The state of the valve can be toggled by clicking on the icon in the **Gas Control** window (red indicates that the valve is closed, green indicates that it is open).

b) If the instrument is connected to a dual inlet, the valves on the changeover valve assembly block, labelled **SC**, **RC**, **SW** and **RW** should be closed. The status of each valve changes by clicking on the icons in the **Dual Inlet** window (red indicates that the valve is closed, green indicates that it is open).

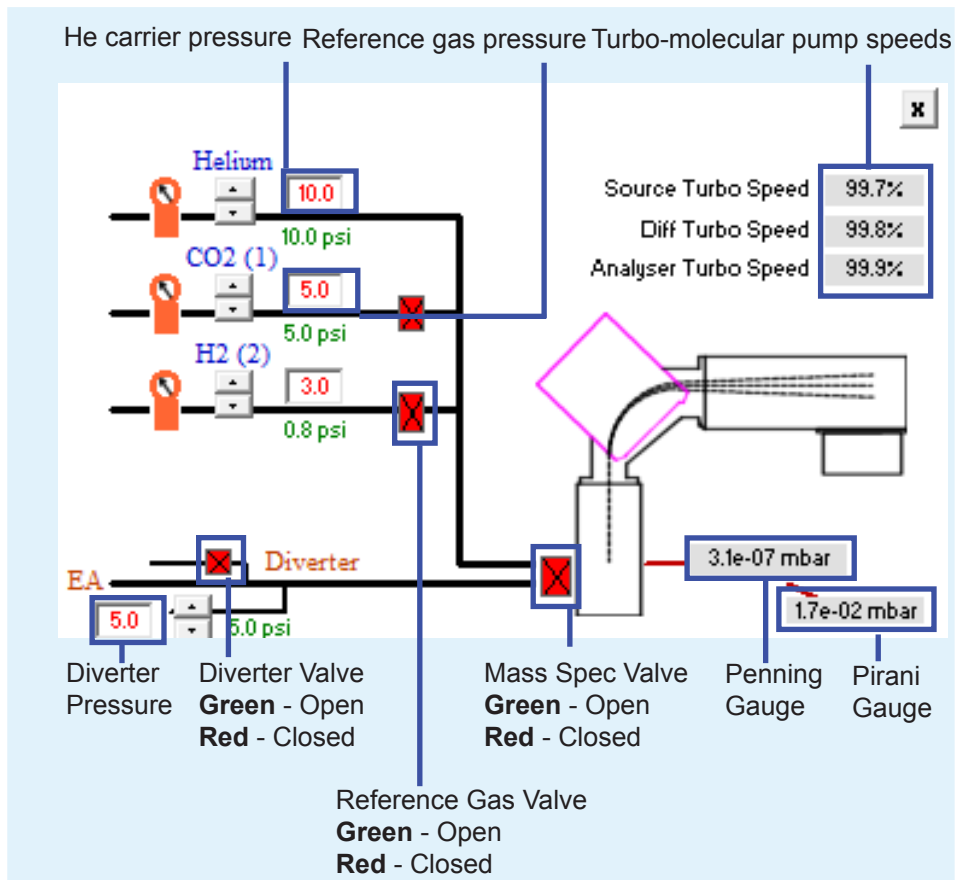


Figure 2.1-4: Closing the Mass Spec valve in the Gas Control window (continuous flow).

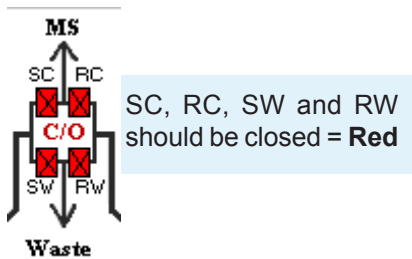


Figure 2.1-5: The valve status of the changeover assembly in the Dual Inlet window.

c) If the instrument is configured as both continuous flow and dual inlet, the mass spec valve and all four valves on the changeover valve assembly block should be closed.

- At the rear of the instrument on the Mains Distribution Unit (MDU), turn off the switches in the following order:
 - Source HT
 - Source Filament
 - HV Supplies

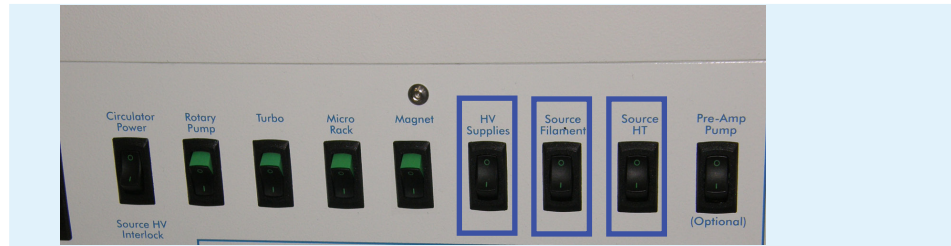


Figure 2.1-6: Switching off the Source HT, Source Filament and HV Supplies.

6. The source is now off and the instrument is now in **Safe** mode. The instrument can now be vented, following the procedure in the next section **2.2 Venting the instrument**.

2.2 Venting the instrument (vent mode and shutdown mode)

In this section, the steps in the venting procedure will be described, changing the instrument mode from **Safe** mode, either to vent the instrument or to place it into **Shutdown**. It is assumed that the instrument is in Safe mode, otherwise follow the steps in **2.1 Turning the source off (from Standby to Safe mode)**.

1. Go to the **Digital > Vacuum** tab and turn off the Penning gauge, by unchecking the **Penning ON** checkbox.

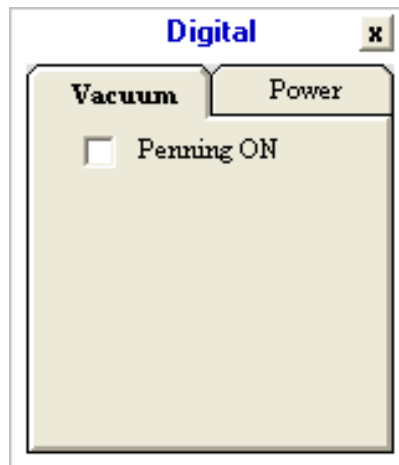


Figure 2.2-1: Turning off the Penning gauge.

2. Check that there are no checkmarks in any of the boxes in the **Digital > Power** tab. All of these parameters were unchecked in the previous section.
3. Ensure that the main inlet valves to the mass spectrometer are closed and that all of the valves in the Dual Inlet changeover valve assembly, **SC**, **RC**, **SW** and **RW**, are closed. These valves were all closed in the previous section.
4. Go to the Mains Distribution Unit (MDU) and turn off the switch labelled **Turbo**. Ensure that the **Source HT**, **Source Filament** and **HV Supplies** are already switched off, as described in the previous section.



Figure 2.2-2: Switching off the turbo-molecular pumps.

5. Go to the **Gas Control** window and monitor the **Turbo Speed** boxes. Wait until the speed of all turbo-molecular pumps has decreased to less than 40%. This may take up to 30 minutes.

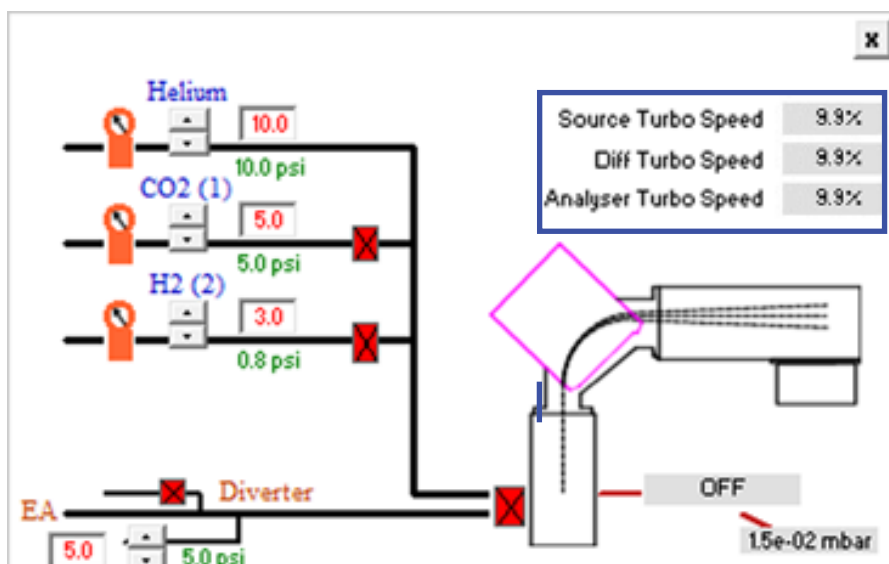


Figure 2.2-3: Monitoring the speed of the turbo-molecular pumps in the Gas Control window.

6. When the turbo speeds have all fallen below 40%, turn off the switch labelled **Rotary Pump** on the MDU at the rear of the instrument.

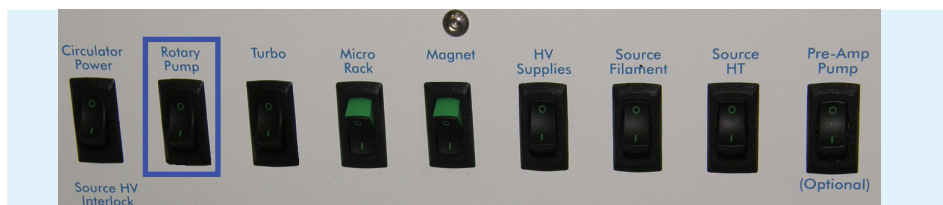


Figure 2.2-4: Switching off the Rotary pump.

7. Immediately after turning off the rotary pump, open the vent valve on the side of the turbo-molecular pump. To access this, remove the panel. Slowly open the black knurled vent valve screw on the side of the turbo-molecular pump under the source housing. This must be done slowly, to avoid damage to the turbo-molecular pump. As soon as a faint hissing is audible, stop turning the knurled screw and let the instrument come to atmospheric pressure. This is indicated by the Pirani Gauge in the **Gas Control** window reaching 10^3 mBar. This will take a few minutes.



Figure 2.2-5: Removing the panel to open the vent valve on the turbo-molecular pump.

HINT: Instead of steps 6 and 7, a completely safe way of venting is to wait until all of the pump speeds have reached 0%, after turning off the rotary pump. Then slowly open the vent valve on the side of the source housing turbo-molecular pump. This takes longer than steps 5 and 6, but minimizes any risk of damage to the turbo-molecular pump.

The instrument is now ready for maintenance procedures, such as changing the filament or cleaning the source.

HINT: If a system vent, rather than a complete shut down is required, the procedure can be terminated here. If a full shutdown is required, follow steps 8 to 10. A complete shutdown is recommended, for example, for a scheduled power cut.

8. At the rear of the instrument on the Mains Distribution Unit (MDU), turn off the switches labelled **Circulator Power**, **Magnet** and **Micro Rack**. In fact, provided that the Micro Rack is switched off last, the order of operation of the other two does not matter.



Figure 2.2-6: Switching off the Circulator Power, Magnet and Micro Rack.

9. On the MDU, turn off the **Power** switch above the entry point of the mains lead.

10. Disconnect the power lead from the mains supply.

This completes the instrument shut down procedure.


2.3 Pumping down (Safe mode)

This section describes step-by-step instructions on how to pump down the Perspective from atmospheric pressure. During this process, various electrical components will need to be switched on. These switches are all located on the Mains Distribution Unit (MDU) to the rear of the instrument. It is assumed that the power lead is connected to the supply and that the power switch is turned on.

1. Go to the **MDU** panel and turn on the **Micro Rack** switch. This is only required if the system was completely shut down, for example for a scheduled power cut. If the instrument was in **Vent** mode, the Micro Rack will be still switched on.



Figure 2.3-1: Switching on the Micro Rack switch.

2. Open the **Stables Control** software by double clicking the  software icon on the desktop.

HINT: If the software is already running, an error message will indicate that there is **no communication with Mass Spectrometer**. This message will revert to **OFFLINE running of software**. If this message appears, close the software and restart it. There is no instrument fault. The instrument electronics must be running before the computer can establish communications. To close the software, right click on the **Nu Instruments** symbol in the top left of the screen and click **Close**.

3. Close the vent valves on all of the turbo-molecular pumps to finger tightness. The valves are located on the side of the pumps. To access the pumps, some side panels will have to be removed. The image below shows the location of a vent valve on the side of a turbo-molecular pump.

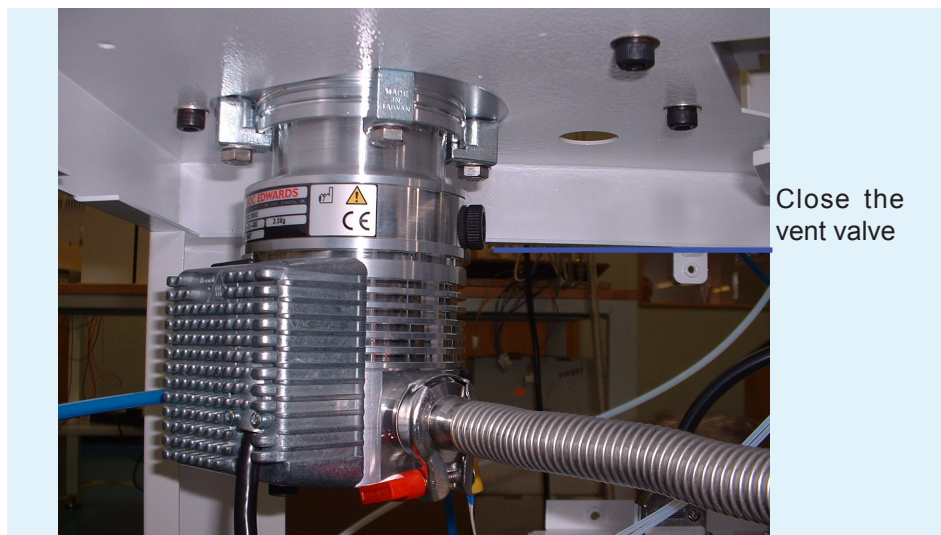


Figure 2.3-2: Location of the vent valve on the turbo-molecular pump.

4. The main inlet valve to the mass spectrometer should also be closed. The actions in this step depend on whether the instrument is configured as continuous flow only, dual inlet only, or both.

a) If the instrument is configured as continuous flow only, the only requirement is to make sure that the mass spec valve is closed. The state of the valve can be toggled by double clicking on the icon in the **Gas Control Window**, shown below. Red indicates that the valve is closed, green indicates that it is open.

b) If the instrument is configured as dual inlet only, the valves on the changeover valve assembly, labelled **SC**, **RC**, **SW** and **RW** should be closed. The states are changed by double clicking on the icons in the **Dual Inlet window**. Red indicates that the valve is closed, green indicates that it is open.

c) If the instrument is configured as both continuous flow and dual inlet, the mass spec valve and the four changeover assembly block valves must be closed.

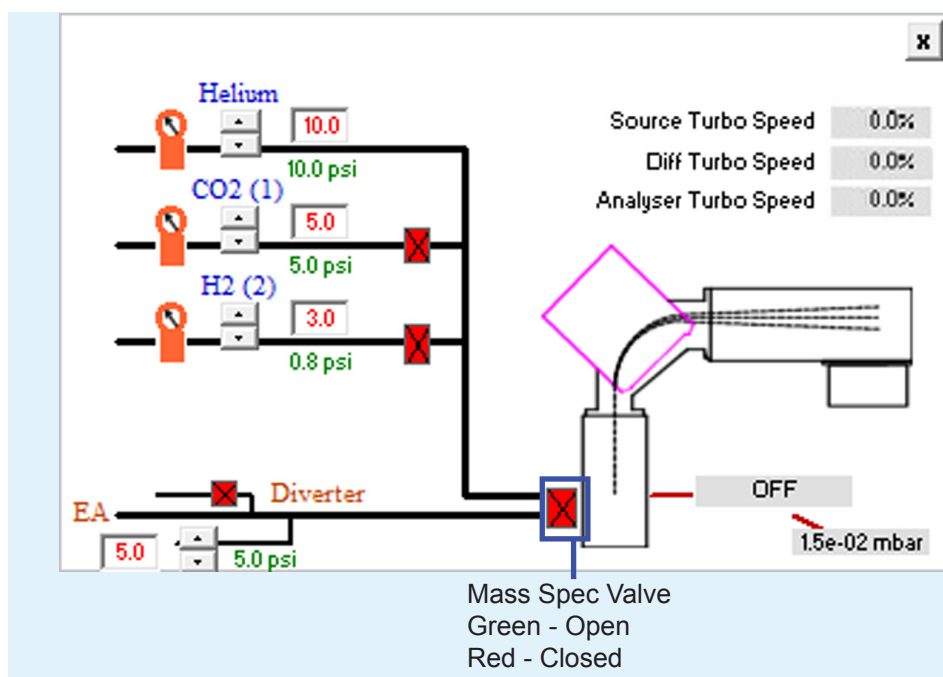


Figure 2.3-3: Closing the Mass Spec valve in the Gas Control window (continuous flow).

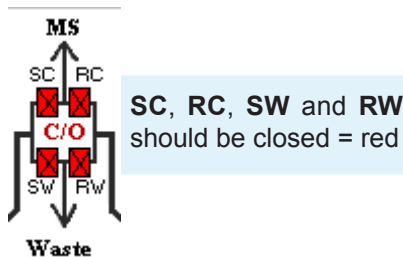


Figure 2.3-4: The valve state of the changeover assembly in the Dual Inlet window.

5. On the **MDU** panel, turn on the switch labeled **Rotary Pump**. This starts the rotary pump. Check that the Pirani reading in the **Gas Control** window starts to decrease from its initial value of about 10^3 mBar.



Figure 2.3-5: Switching on the Rotary pump switch.

6. When the Pirani gauge reading has fallen to less than 1 mBar, turn on the switch labelled **Turbo** on the MDU panel. This switches on all three turbo-molecular pumps. Watch the pump speeds in the **Gas Control** window, to ensure that they all increase at approximately the same rate.

HINT: If after about 1 minute, the Pirani gauge reading has not begun to decrease, the vent valve may not be closed or there may a significant leak somewhere.

- a) Ensure that the vent valve on the turbo-molecular pump has been closed.
- b) Switch off the rotary pump and fix the leak before continuing.



Figure 2.3-6: Switching on the Turbos switch.

7. When the pumps have reached full speed, wait for about 30 minutes before switching on the Penning gauge.

8. Go to **Digital > Vacuum**. The **Penning ON** checkbox should be empty. Place a checkmark next to it to initiate the Penning gauge. The reading can be checked in the **Gas Control** window and should be in the 10^{-6} mBar range, followed by 10^{-7} mBar after about 1 hour.

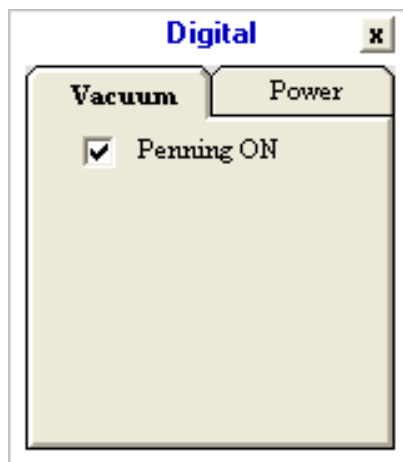


Figure 2.3-7: The Vacuum tab in the Digital Window with the Penning gauge on.

This completes the start up procedure. The instrument is now in **Safe** mode.

2.4 Turning the ion source on (Operate mode)

HINT: When the instrument has been vented, the bakeout procedure can be carried out, which is described in detail in section **2.5 Bakeout procedure**. After cleaning the source or exchanging the filament, it is recommended to perform a quick check that the source is working, before proceeding to the bakeout procedure. Once it is confirmed the ion source is working, it should be switched off again for the bakeout procedure. Do not use the instrument for analyses until the bakeout procedure has been completed.

It is assumed that the instrument is in **Safe** mode, as described in section **2.3 Pumping down (Safe mode)**. The following is a step by step description how to safely turn on the source. At this point an error message indicating that the source is out of regulation will be flashing at the top of the screen. This is normal, as the source is not yet on.

1. The Instrument must be under vacuum with a Penning pressure below 10^{-5} mBar, otherwise the software will not allow the source to be switched on. Ideally the Penning pressure should be in the low 10^{-6} mBar range before attempting to turn on the source. This prolongs the life of the filament.

2. This step depends on the instrument configuration.

HINT: Before opening the main isolation valve for continuous flow, helium must be flowing and helium must continue to flow whenever the main isolation valve is open. If the flow of helium needs to be interrupted, for example for helium cylinder exchange, the isolation valve must firstly be closed. See **Chapter 3 Maintenance** for full details.

a) If the Instrument is configured for continuous flow mode only, go the **Gas Control** window and set the helium carrier gas and the helium sample gas to about 10 psi. Once the He carrier set pressure (red numbers) matches the actual pressure (green numbers), wait for about 2 minutes and then open the **Mass Spec** valve. When the Mass Spec Valve is opened, there will be a small pressure surge. After this initial pressure surge, the pressure should return to the range 5×10^{-6} to 1×10^{-5} mBar, depending on the configuration.

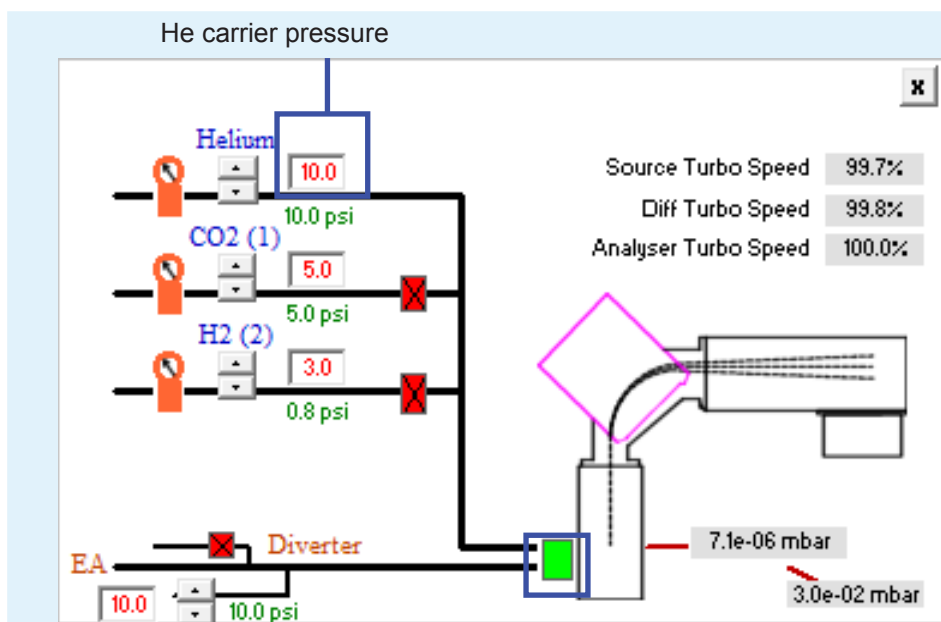


Figure 2.4-1: Setting the He carrier pressure in the Gas Control window.

HINT: If the source is on and an attempt is made to open the Mass Spec valve, the software will prompt the user to turn the source off. Open the Mass Spec valve and turn the source on again. This is to protect the source during the initial pressure surge.

b) If configured for Dual Inlet mode only, the valves on the changeover valve assembly block labelled **SC** and **RC** should be closed and those labelled **SW** and **RW** should be open. The states of the valves are changed by clicking in the icons in the **Dual Inlet** window. Red indicates that the valve is closed, green indicates that it is open. Open **SW** and **RW**.

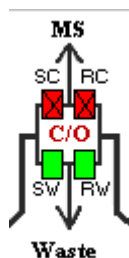


Figure 2.4-2: Switching SW and RW on in the Dual Inlet changeover valve assembly.

c) Some instruments have both Continuous Flow and Dual Inlet capability. In this case, when working in Continuous Flow mode, set up as for Continuous Flow only and the valves on the changeover valve assembly block as shown in the figure above, with **SC** and **RC** closed and **SW** and **RW** open.

Working in Dual Inlet mode, set up as for Dual Inlet mode only, with the Mass Spec valve closed.

3. The next step is to turn on the magnet. It is assumed that the magnet and Hall probe are secure and fixed in place.

4. Turn on the switch labelled **Magnet** on the MDU panel.

5. Go to **Diagnostics > Monitor > Magnet** in the Stables software. After 30-60 minutes the **Proportional Error** should settle down to ± 10 , indicating that the magnet has warmed up and has settled down.

6. Before switching on the source power supply, go to **Mass Spec > Tuning > Load** to open the **Instrument Tuning Parameters** window.

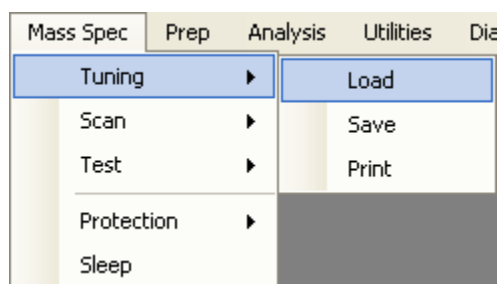


Figure 2.4-3: The Mass Spec/Tuning/Load tabs.

The **Tuning** folder, which contains all of the stored tunings, will open. An example is shown below. Select **SetToZero** and click **Open**. This sets all instrument voltages and currents to zero, so no sudden voltage changes will occur when any power supplies are switched on.

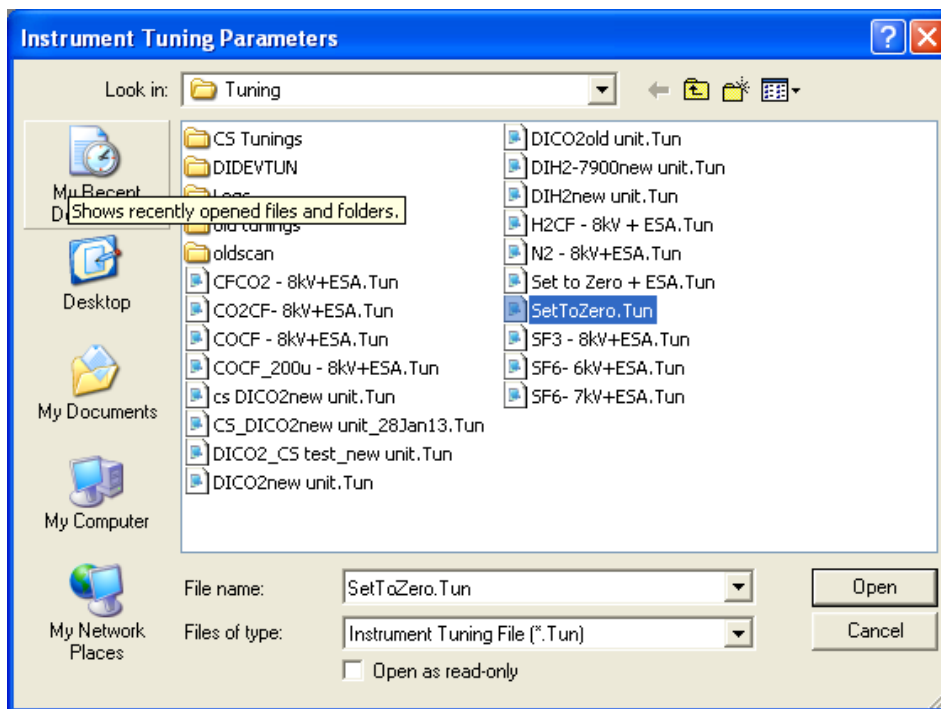


Figure 2.4-4: The Tuning Panel with tuning SetToZero selected.

7. On the MDU panel at the rear of the instrument, switch on the **Source Filament**, **Source HT** and **HV Supplies**, in that order.



Figure 2.4-5: Switching on the Source Filament, Source HT and HV Supplies.

8. Go to the **Digital > Power** tab and ensure that all checkboxes are unchecked. Now place a checkmark in the boxes labelled **DVM Supplies**, **Deflector Supplies**, **Filament Supply** and **Source ON**, in that order.

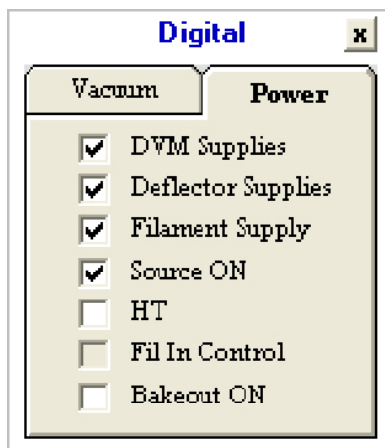


Figure 2.4-6: Activating DVM Supplies, Deflector Supplies, Filament Supply and Source ON in the Digital window.

9. Go to **Mass Spec > Tuning > Load** to open the **Instrument Tuning Parameters** window. Select one of the pre-installed tunings, other than **SetToZero**, and click **Open**. The source parameters specified by the tuning will now appear in the **Source**, **Quads** and **Collectors** panes in the Tuning window. Typical examples are shown below.

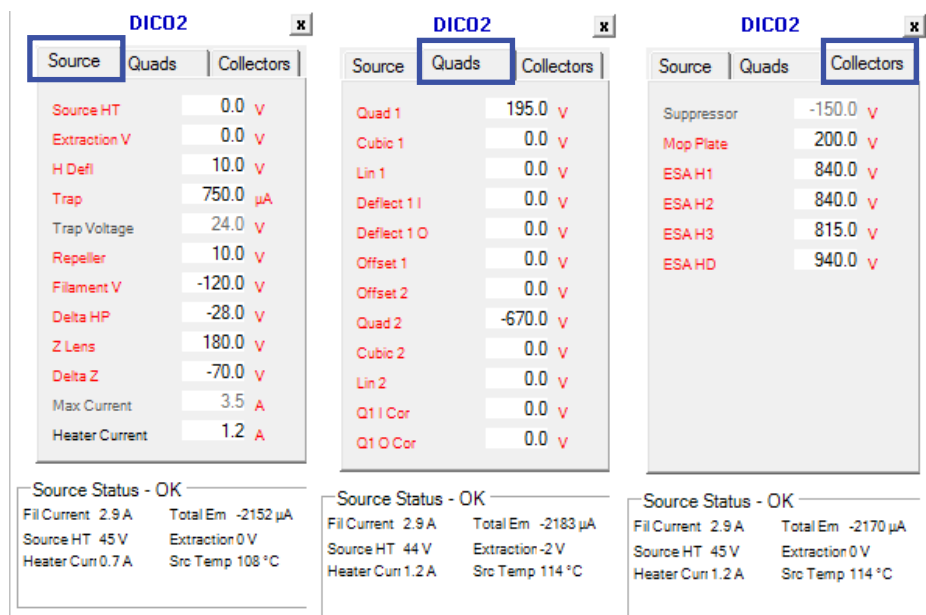


Figure 2.4-7: Typical values for the Source, Quads and Collectors panes.

The filament and heater currents in the **Source Status** window will slowly rise and when the filament current approaches its operating value, typically somewhere between 2.5 and 3 Amps, the total emission will also begin to rise. When the electronics stabilizes the trap current at the specified value, a checkmark will appear in the **Fil in Control** box in **Digital > Power** tab.

Click on the **Mass Spec > Protection > Reset Trips** tabs. The error messages at the top of the screen indicating that the source is out of regulation should now disappear.

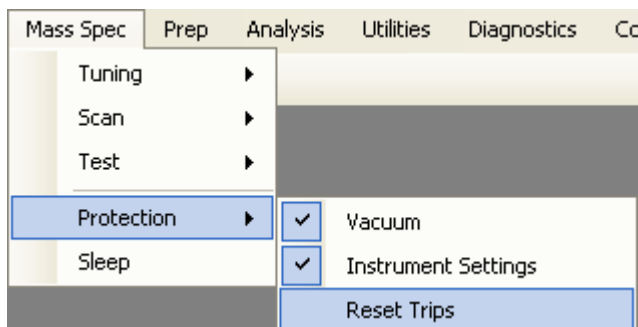


Figure 2.4-8: The Mass Spec >Protection > Reset Trips.

10. Go to **Digital > Power** tab and check the **HT** checkbox. A checkmark will be displayed and the **Source HT** and **Half Plate** voltages specified in the tuning will appear in the corresponding boxes on the **Source** pane and in the **Source Status** box in the **Tuning** window.

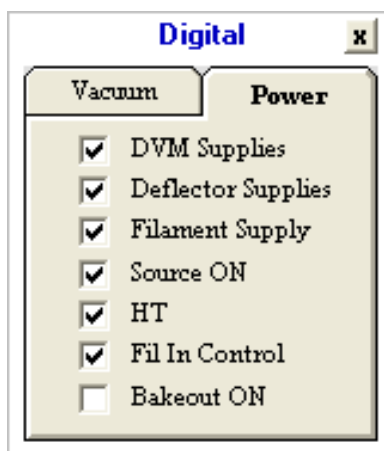


Figure 2.4-9: Switching parameters on in Power tab of the Digital window.

The source is now operational.

11. Switch the source off again, as described in section 2.1 **Turning off the ion source**, as the next step is to carry out the bakeout procedure.

HINT: If the source is being switched on after the bakeout procedure has been completed, leave the source switched on. Over the course of a few hours, the **Source Temperature** in the **Source Status** window will slowly rise. If the heater current is set to zero, the temperature will stabilize at approximately 100°C. If the source heater is on at 1.5 Amps, the source temperature will stabilize at about 180°C. The source can run continuously in either condition, although it is recommended that the heater current does not exceed 1.5 Amps.

2.5 Bakeout procedure

The aim of the bakeout procedure is to improve the vacuum. Bakeout can be completed after any venting of the vacuum region, or at other times when the system vacuum has degraded.

The bakeout raises the temperature of the whole housing. This elevated temperature speeds up the desorption of species from the inner surfaces of the vacuum chamber and allows them to be pumped away before they readsorb onto any other surface.

CAUTION

Bakeout procedures should only be carried out by trained personnel.



CAUTION

Hot surface : Hot surfaces will be present throughout the bakeout procedure. Make sure there are no flammable materials near the instrument. Also ensure that the system has cooled down for several hours before touching any surfaces.

The bakeout procedure removes water vapor and other hard to pump gases from the vacuum region. To perform the bakeout, the instrument must be under vacuum with the source switched off, which is described as the Safe mode in section 2.1 **Turning off the source**.

HINT: If a dual inlet is fitted, it is recommended that it is baked at the same time. This is to ensure that any contaminants do not accumulate in any cold spots. If, however, the dual inlet is not baked at the same time, it is recommended that the dual inlet is isolated from the Perspective by closing all of the valves, **SC**, **RC**, **RW** and **SW** in the changeover assembly.

HINT: If the system is set up for continuous flow it is recommended that the main isolation valve is open for the bakeout procedure. Helium must be flowing at all times, so ensure that it will not run out during the bakeout procedure.

1. The main CF isolation valve must be open to make sure that the seat is not affected during the bakeout, which could mean that it can not be opened again. The valve contains a valve seat which is made from Kel-F. If this material experiences temperatures above 70°C it may deform, with the consequent risk of baking the valve into the closed position.

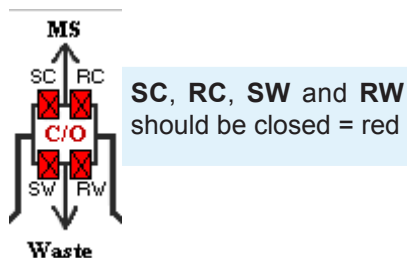


Figure 2.5-1: Changeover assembly valves closed, to isolate the dual inlet, if not being baked at the same time.

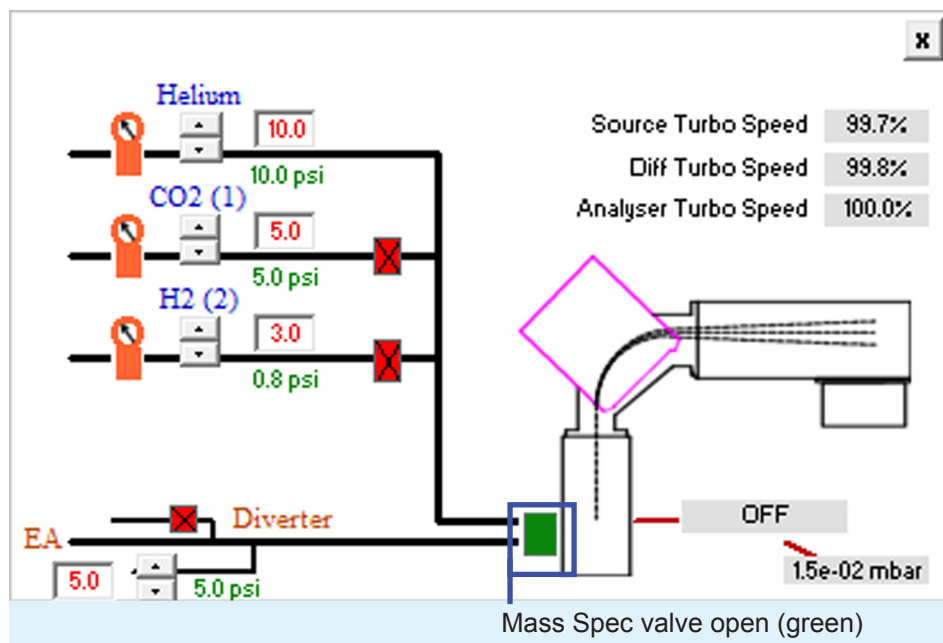


Figure 2.5-2: Mass Spec valve open.

2. Ensure that there are no checkmarks against any supplies in **Digital > Power**. Everything must be unchecked.

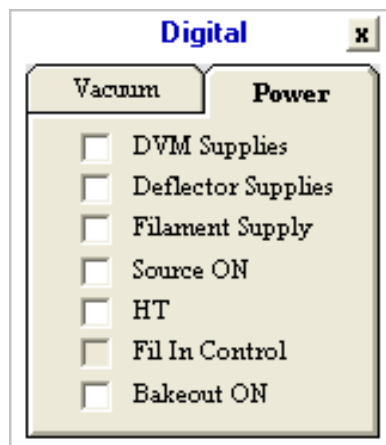


Figure 2.5-3: Switching off supplies in the Digital window.

3. On the MDU, the following switches should be off: **Magnet, HV Supplies, Source Filament, Source HT, Pre-Amp Pump** and **Bakeout**. The Bakeout switch will later be switched on to initiate the bakeout.



Figure 2.5-4: Switches in the off position.

2.5.1 Removing electrical feedthroughs

Before removing electrical feedthroughs, ensure that the voltages are off and the **HV Supplies, Source Filament** and **Source HT** switches are off on the **MDU**, as described above.

1. Remove the cover over the electrical connections to the source and unplug all of the cables from the feedthroughs on the source plate.
2. At the flight tube end of the analyzer housing are four feedthroughs, two on each side. These are the feedthroughs that provide the voltages to the plates of the Quad lenses. Remove their protective covers first and then remove the feedthroughs.
3. At the opposite end of the analyzer housing is another feedthrough, the ESA feedthrough. This supplies various voltages used by the collectors and should also be removed.

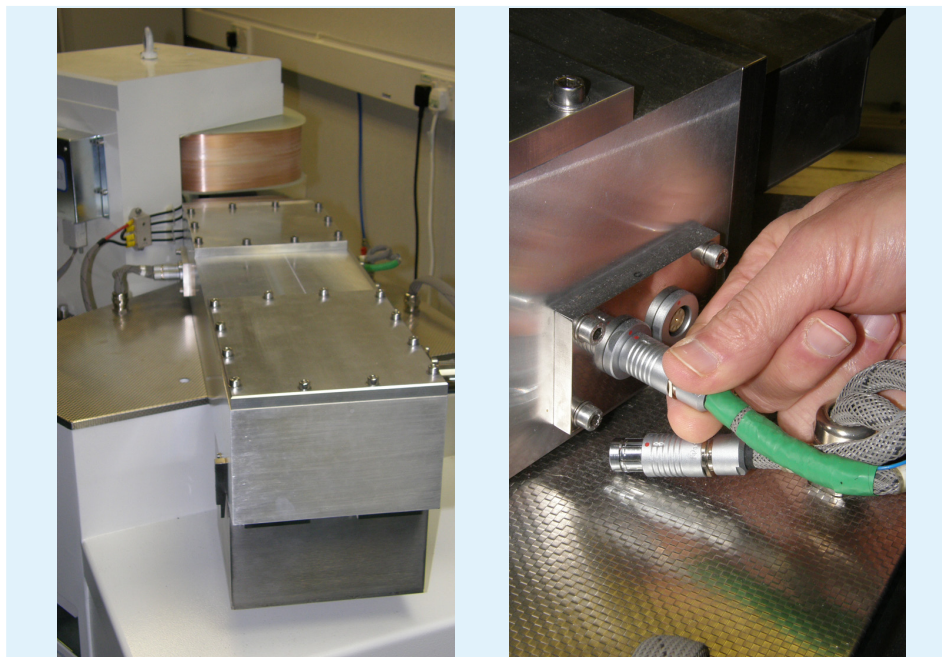


Figure 2.5-5: Quad connectors.

2.5.2 Removing the pre-amplifier bin

The preamplifier needs to be removed from the system prior to baking to protect the delicate electronics inside. In order to remove the pre-amplifier bin, it must be vented. Since the bin holds a low vacuum, no detailed procedure to vent the bin is necessary.

HINT: The way that the pre-amplifier bin is evacuated depends on the configuration of the system. For a continuous flow-only instrument, a small vacuum pump is provided. For a system with a dual inlet, the low vacuum rotary pump is used to evacuate the bin. In both cases, the bin is isolated from the vacuum system via a speedivalve located on the back of the instrument. The bin does not need to be actively pumped but instead only needs to be evacuated about once a month to ensure that a good vacuum is maintained. The housing, once evacuated, can be left under static vacuum for many weeks.

1. The preamplifier bin must be vented and **DVM Supplies** switched off. Go to the **Digital > Power** tab and ensure that **DVM Supplies** is unchecked.

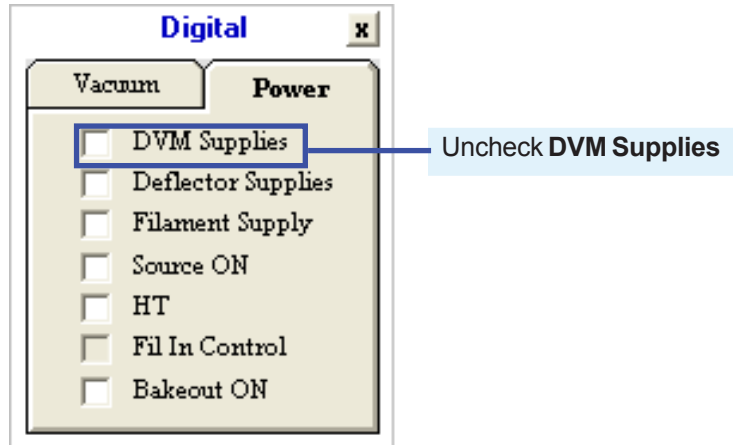


Figure 2.5-6: Unchecking DVM Supplies in the Power tab of the Digital window.

2. Check that the clips on both sides of the preamplifier bin are in place and secure. This is to ensure that the bin does not fall onto the bench once the vacuum is lost.



The pre-amplifier bin is very heavy. Before venting the pre-amplifier bin, ensure that the clips on both sides are secured, so that the pre-amplifier bin can not fall onto the bench once vented.

Figure 2.5-7: Checking that the clips of the pre-amplifier housing are secured.

3. Check that the Edwards Speedivalve on the service panel is closed.

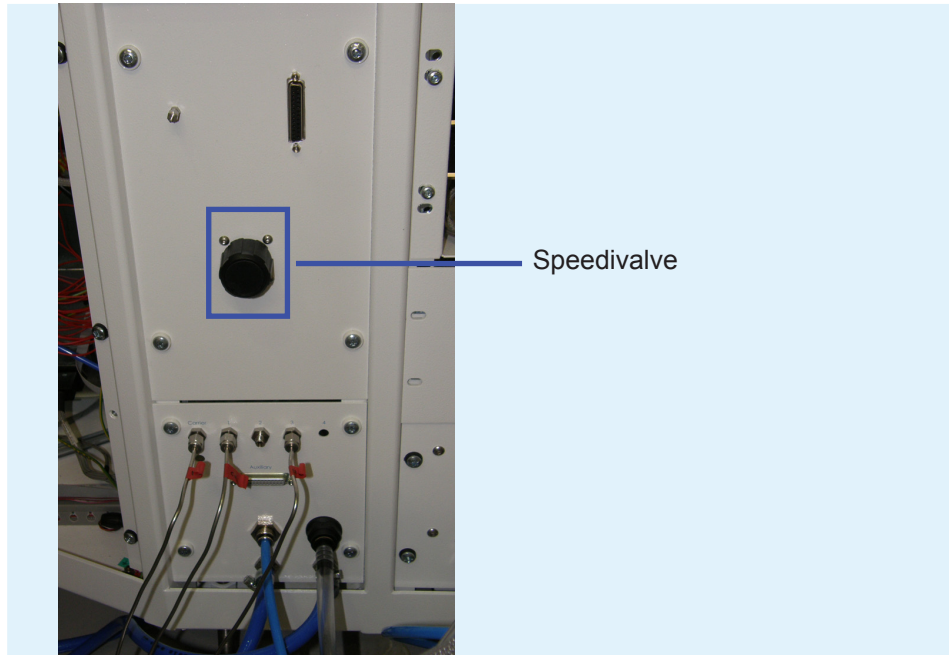


Figure 2.5-8: The Edwards Speedivalve on the Service panel.

4. Unscrew the small silver knurled nut shown in the next image.
5. Loosen the clear plastic vacuum pipe by moving it from side to side, until air is heard entering the bin, then remove the clear plastic pipe from the tailpipe.

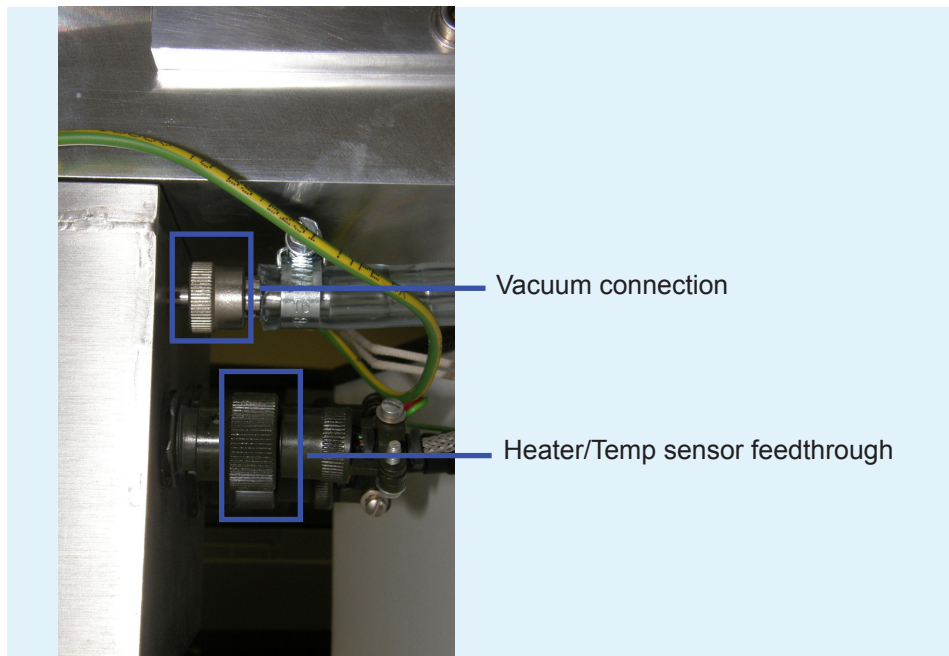


Figure 2.5-9: Connections to the pre-amplifier bin.

The venting procedure is complete. The bin should now be supported on the clips located on either side of the analyzer housing.

6. The pre-amplifier bin can now be removed. Remove the two dark green electrical connectors.

7. The pre-amplifier bin should now be supported only by the clips on either side of the analyzer housing. Support the pre-amplifier bin, remove the clips and gently lower the bin onto the shelf below. The bin can be supported by hand.

The bin should be completely removed from the instrument by sliding it sideways. Take great care not to damage the amplifier connector pins on the guides at the side of the analyzer housing.

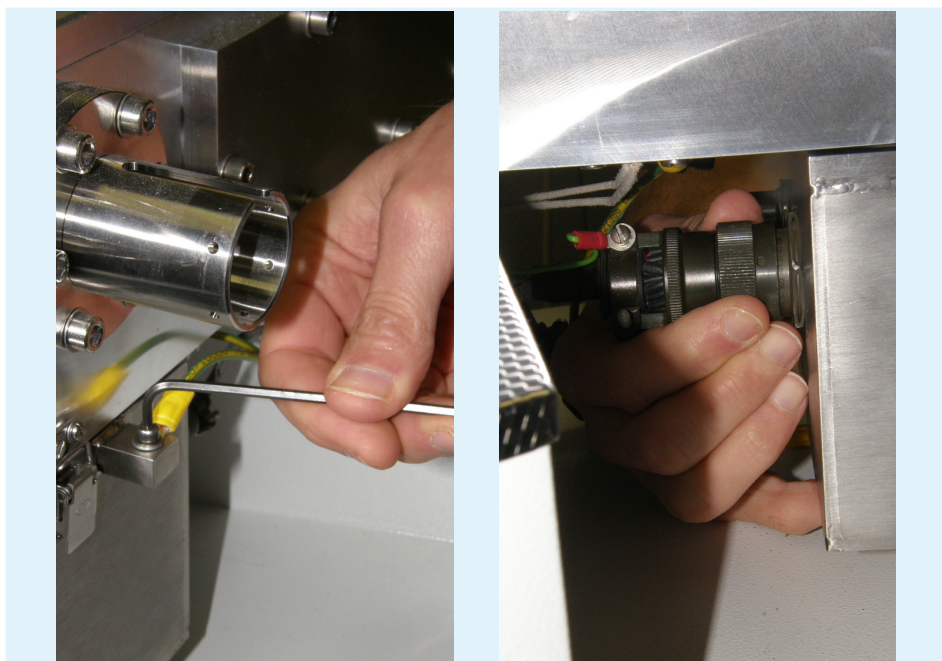


Figure 2.5-10: Loosening the bolt and disconnecting the cable.

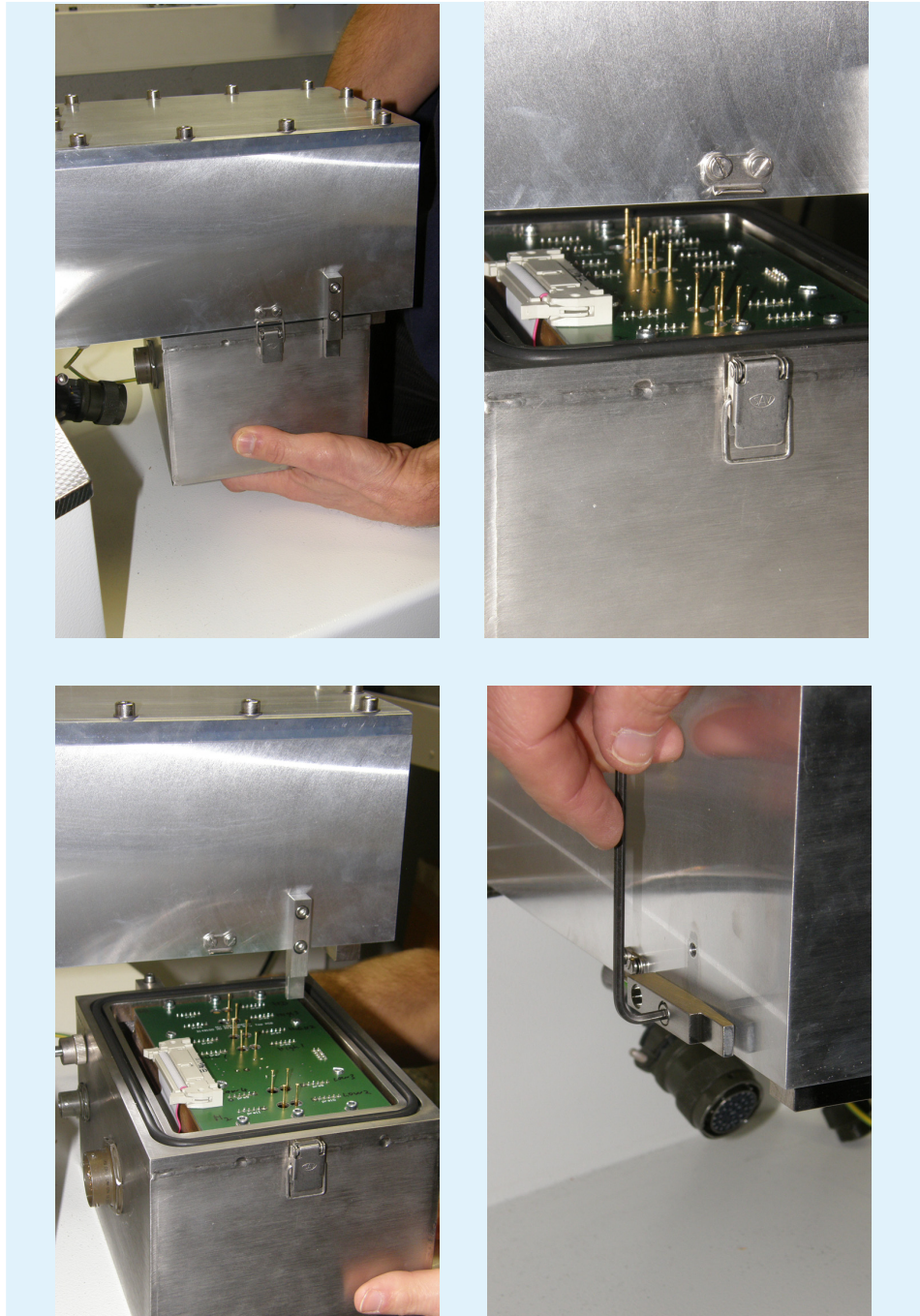


Figure 2.5-11: Carefully removing the pre-amplifier bin.



Figure 2.5-12: Re-tightening the bolt.

2.5.3 Removing the Penning gauge body

Prior to the bakeout, the red body of the Penning gauge must be removed.

1. Go to **Digital > Vacuum** and uncheck the Penning gauge.

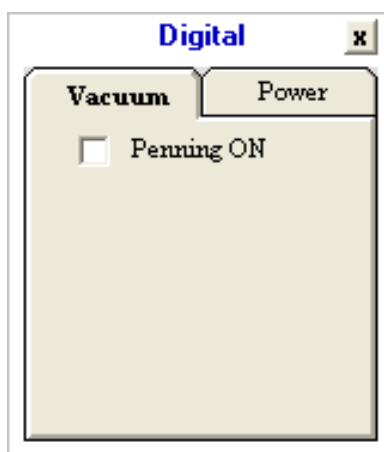


Figure 2.5-13: Turning off the Penning gauge.

2. Unplug the cable from the red body of the Penning gauge. Release the red body of the gauge by twisting it and then remove it by drawing it backwards over the vacuum fitting.

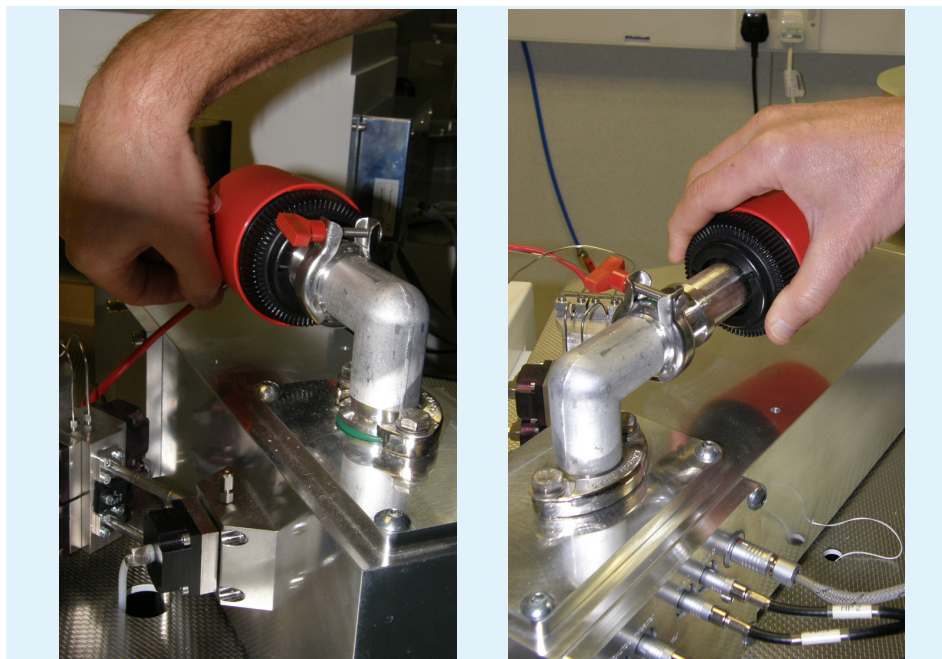


Figure 2.5-14: Removing the red body of the Penning gauge.

2.5.4 Moving the magnet back

⚠ CAUTION

The magnet is very heavy and it is secured to the frame of the instrument by a large retaining bolt. The bolt ensures that in the event of seismic activity, the magnet and the rest of the instrument move together, reducing the risk that the magnet becomes displaced, potentially causing catastrophic damage to the flight tube and danger to personnel. It is highly recommended that when the magnet is moved back for the bakeout, it is held in place by the securing bolt supplied.

⚠ CAUTION

Before moving the magnet, move the cables that are behind the magnet. There is no need to disconnect them.

-
1. Before moving the magnet, ensure that the magnet is switched off at the MDU.

⚠ CAUTION

While pushing the magnet back, move the magnet slowly and observe that there is clearance between the magnet and the flight tube during its travel. Be careful not to damage the cables behind the magnet.

2. The magnet needs be moved clear of the flight tube. Above the MDU at the back of the instrument, three knurled screws attached to the base of the magnet can be seen, as shown in the image below.

3. Before the magnet can be pushed back, the bolt underneath the bench, which is holding the magnet in place, must be removed and stored safely.

HINT: This holding bolt for the magnet gives seismic protection to the instrument, preventing displacement of the magnet in the event of an earthquake. Once the magnet has been pushed back, the same bolt can be inserted again in a different position to fix the magnet into place during the bakeout procedure. This is recommended procedure for any countries which may suffer from earthquakes.

The two outer screws define the position of the magnet relative to the flight tube, while the center one is a locking nut. The center locking nut must be removed, but do not adjust the position of the outer locating screws. Once the locking nut has been removed, the magnet can be moved away from the flight tube. Caution is required and the magnet must be moved very slowly. It is very heavy, but it moves easily on its roller bearings, so little force is required to move it.

It is not necessary to remove the cables from the magnet controller.

HINT: Only unscrew the middle locking nut and not the two outer alignment screws. Keeping the alignment screws fixed will ensure that the magnet returns to the same position after it is pushed back after bakeout.



Figure 2.5-15: The locking nut and locating screws on the magnet base.



CAUTION

Crush hazard: Take care when moving the electromagnet, to avoid trapping fingers behind the magnet during movement.

4. Slowly push the magnet back. It is mounted on runners, so it will glide easily. Ensure that it does not impact against the end stops.

HINT: It is recommended that two persons move the magnet back, one either side of the magnet, one pushing gently, the other guiding it.

2.5.5 Install the flight tube thermocouple

1. Install the flight tube thermocouple, if not already fitted, using high temperature tape, which is supplied with the instrument. Three other thermocouples, permanently fixed to the instrument, are also used to control the bakeout process.

HINT: The thermocouple could remain fitted after bakeout, if the thermocouple is secured with tape to the side of the flight tube, so that the magnet can be moved over it. If the thermocouple is fitted to the central upper surface of the flight tube, it has to be removed in order to be able to move the magnet back in position after the bakeout.

2. Ensure that the flight tube thermocouple is connected to the bakeout control panel.

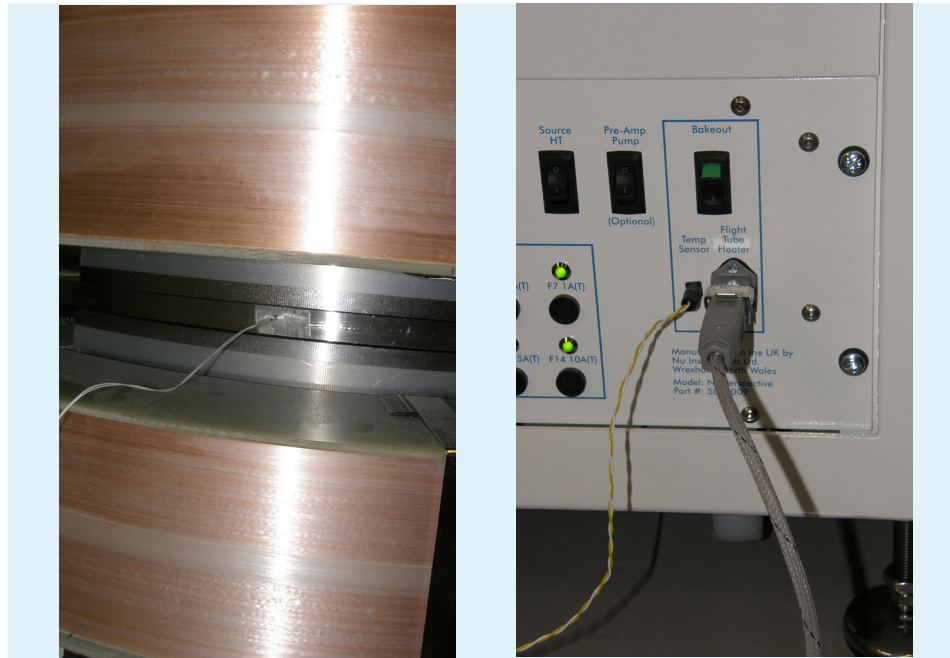


Figure 2.5-16: Thermocouple fitted to the flight tube and connected to the MDU.

2.5.6 Placing the bakeout heater and covers

1. Position the flight tube heater tray beneath the flight tube, as shown in the image below.
2. Plug the flight tube heater into the socket on the MDU. At this point it, the heater will not become hot.

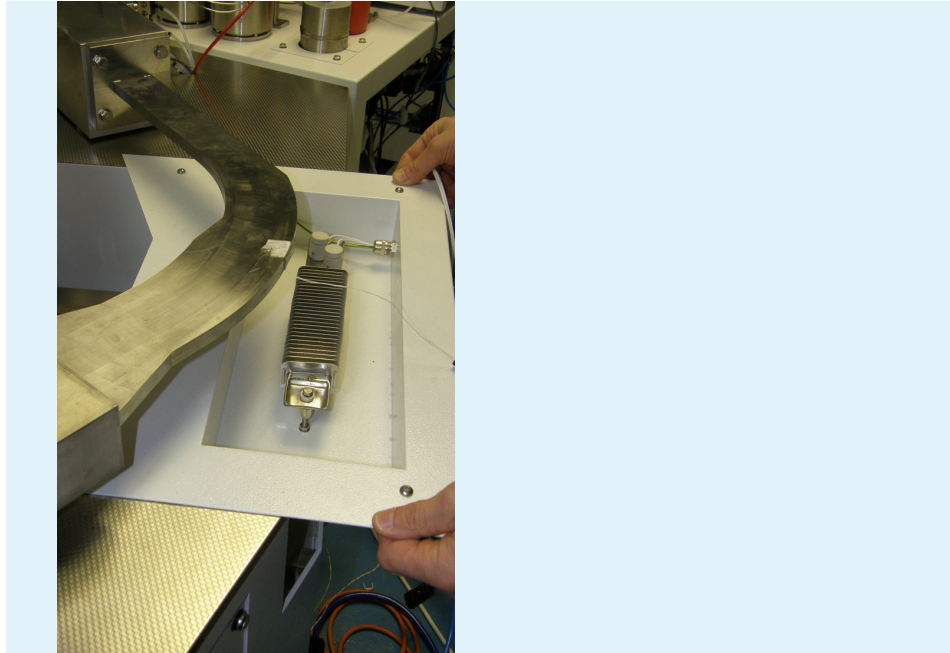


Figure 2.5-17: Placing the heater tray under the flight tube.

3. Put the bakeout covers in place. There are two, joined by velcro strips close to the point where the flight tube meets the source housing.



Figure 2.5-18: The bakeout cover fitted over the analyzer housing and the flight tube.

HINT: Before the covers are fitted, some electrical leads were removed. Make sure that they have not been left inside the covers.

2.5.7 Starting the bakeout

1. On the MDU at the back of the instrument, turn on the switch labelled **Bakeout**. The bakeout will not start until the timings and the temperature have been set in the software.

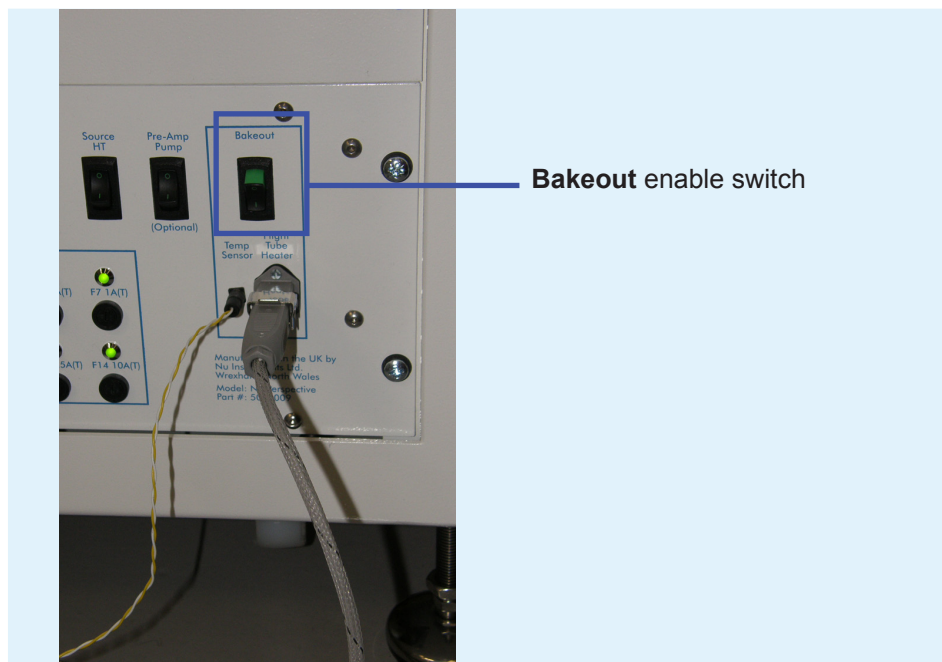


Figure 2.5-19: Switching on the Bakeout Enable switch.

2. Go to **Utilities > Bakeout**, the **Bakeout Control** window will now open.

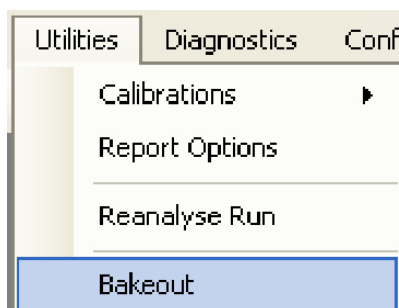


Figure 2.5-20: The Utilities dropdown menu.

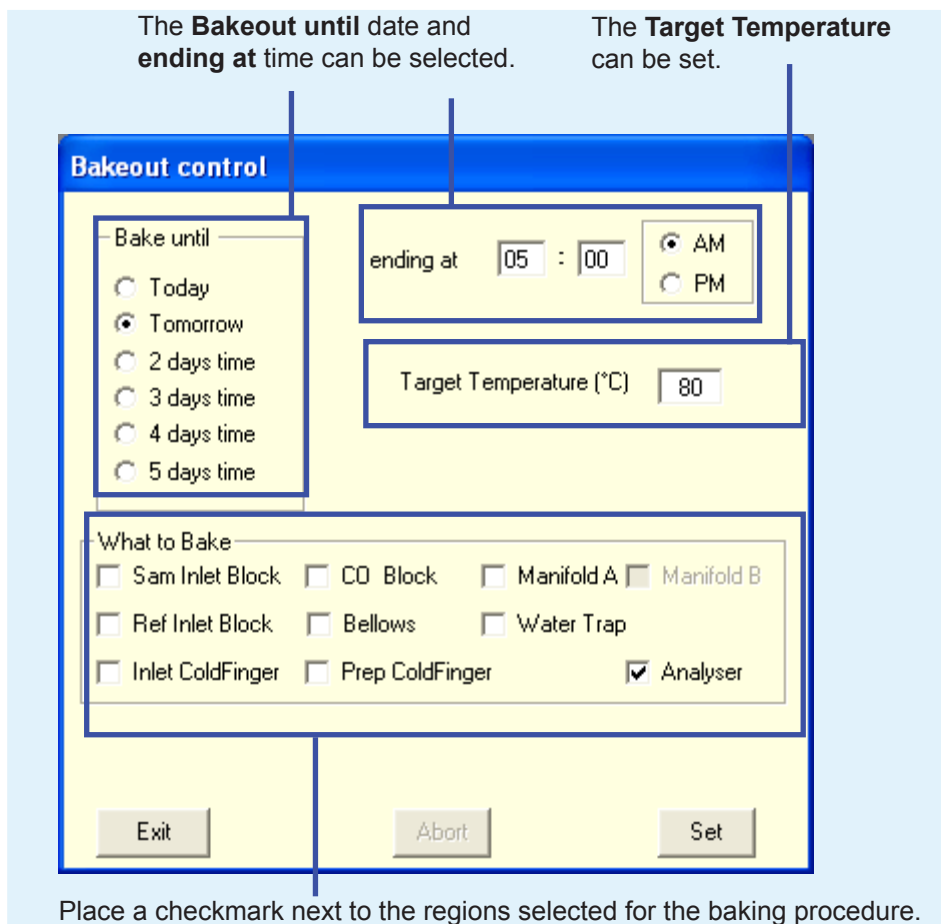


Figure 2.5-21: The bakeout control window.

3. The bakeout control window sets the time for the end of the bakeout, in the **Bake until** and **ending at** boxes. In the example above, the bakeout will end at 5:00 am, the day after the bakeout has started.

The target temperature is the bakeout temperature - all parts of the instrument will bake at this temperature. 80°C is the recommended bakeout temperature, which should not be exceeded, to avoid damaging the Kel-F valve seats.

HINT: If a dual inlet is fitted, it is recommended to bake this at the same time, to avoid any cold spots where contamination could accumulate.

In the **What to Bake** box, select **Analyser** and leave the other boxes empty. The other options in this box concern the Dual Inlet and this is covered in a separate manual. Selecting Analyser will bake the source housing and the flight tube, as well as the analyzer housing.

Once the desired parameters have been input, click **Set** and the bakeout will start.

HINT: After the bakeout has started, if any of the parameters need to be changed, clicking **Abort** will stop the bakeout. The parameters can then be edited and the bakeout restarted by pressing **Set**.

4. Go to **Diagnostics > Monitor > Engineer** to monitor the temperatures of the source housing, flight tube and analyzer housing.

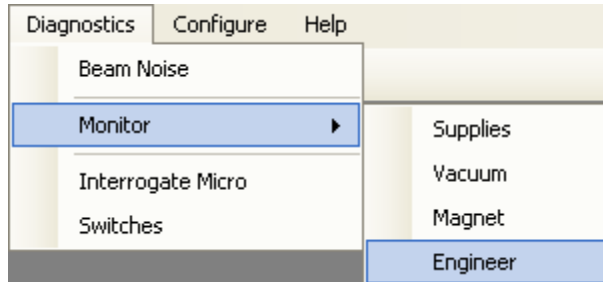


Figure 2.5-22: Select Diagnostics > Monitor > Engineer to open the Engineer Monitor.

5. The **Engineer Monitor** window will open. Select **Analysers-Bakeout** from the dropdown list.

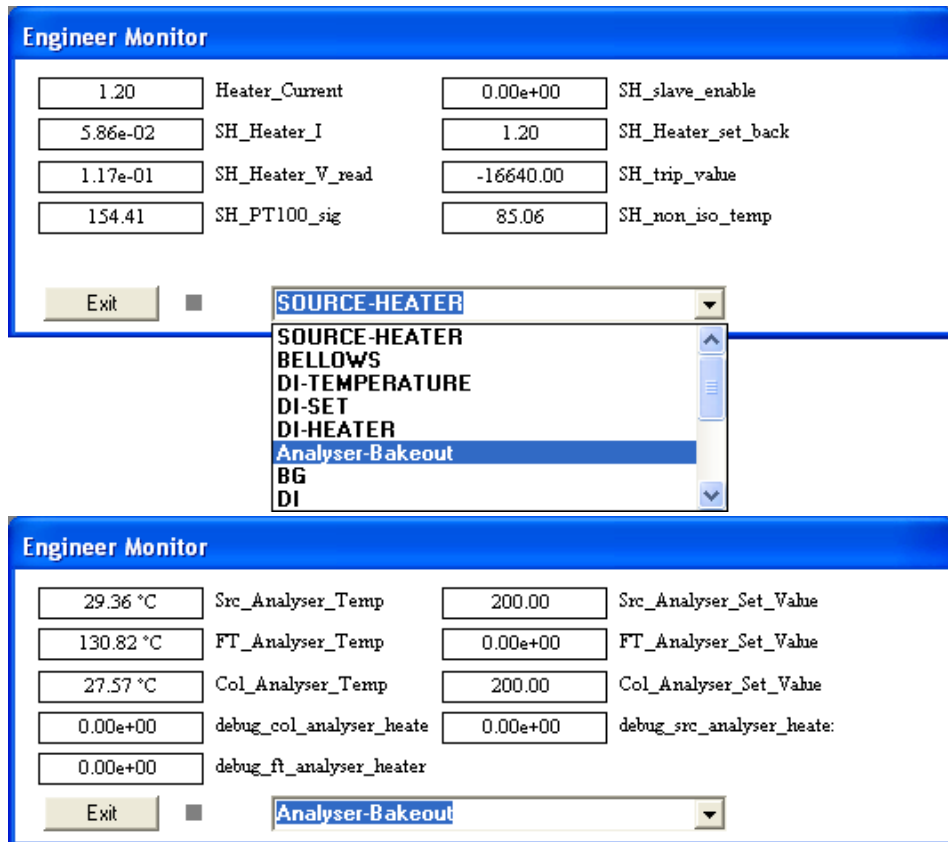


Figure 2.5-23: Monitoring the Analysers-Bakeout in the Engineer Monitor window.

The top three left hand boxes are the source, flight tube and collector temperatures. The rest of the boxes monitor how the instrument control systems are working and are be used for software debugging and troubleshooting.

The temperature readings will automatically update. The temperature of the flight tube will rise faster than the housings. These can take several hours to reach bakeout temperature.

Clicking **Exit** will clear the panel from the screen.

6. This next step can be performed after the bakeout has finished. When the temperatures of the housings have fallen below 50°C, remove the bakeout covers.

7. Replace the red body of the Penning gauge by sliding it over the vacuum fitting and twisting it to lock it in place. Reconnect the cable and turn the Penning gauge on using the **Vacuum** tab in the **Digital** window, turn the Penning gauge on by checking the box.

At this point, the pressure will appear to be quite high, probably somewhere in the 10^{-6} mBar range. This will initially fall rapidly and then the rate of decrease will become slower, as the instrument reaches room temperature.

8. Replace all of the cables on the source plate (the cables and sockets are labelled) and replace the source plate cover. Do not turn the source on yet.

9. Unplug and remove the flight tube heater tray. Slowly move the magnet over the flight tube, until it contacts the locating screws. Replace the locking nut. Finger tight is sufficient. Assuming that the locating screws have not been disturbed, the magnet should be in exactly the same position that it was before the bakeout. Check that the Hall probe is in position by visually inspecting it.

10. At the flight tube end of the collector housing, reconnect the four cables for the Quad lenses. The cables labelled **Quad 1** connect to the sockets nearest to the magnet.

11. At the opposite end of the collector housing, reconnect the cable supplying the ESA voltages.

12. Refit the pre-amplifier bin in its position under the end of the analyzer housing.

Firstly, slide the bin along the shelf so that it is in position below the housing. As with removal, be very careful not to damage the amplifier connector pins on the guides at the side of analyzer housing.

Slowly lift the bin so that it slides up the guides on the side and the rear of the analyzer housing. It is a good idea to crouch down and watch the connector pins as they approach the feedthroughs. They are not visible on contact, but they are very close to the feedthroughs when lost to sight, at which point it is possible to ensure that the bin is in the right place.

Fasten the clips on each side of the analyzer housing.

13. Evacuate the pre-amplifier bin, following the steps below:

- a) Make sure that the clips on either side of the analyzer housing are properly located. These will prevent the bin falling onto the shelf below if for any reason the vacuum is lost.
- b) If not already off, turn off the DVM supplies by going to **Digital > Power** and unchecking **DVM Supplies**.
- c) Locate the connection to the vacuum pump. It is the small metal knurled nut on the end of a clear plastic pipe. Make sure this nut is attached firmly to the tail pipe located on the side of the bin. Finger tight is sufficient.
- d) If the system is set up for continuous flow only, switch on the **pre-amp pump**, which is located at the right hand end of the MDU. When the pump starts, it will initially seem quite noisy. This is normal. For systems fitted with a Dual Inlet, this pump is not required, as the roughing pump is used to pump out the pre-amplifier bin.
- e) On the **Service Panel** at the back of the instrument is a small Edwards Speedivalve. Open this valve to start the evacuation of the pre-amplifier bin.

HINT: It may be necessary to lift the pre-amplifier bin against the bottom of the analyzer housing for a few seconds for the evacuation to start.

- f) Leave the pre-amplifier bin pumping for 2 to 3 minutes and then close the speedivalve.
- g) If required, turn off the **pre-amp pump** at the switch on the MDU.
- h) Go to **Digital > Power** and place a checkmark next to **DVM Supplies** to turn the supplies on.

The evacuation of the pre-amplifier bin is now complete.

14. Turn on the source as described in section 2.4.

Chapter 3: Maintenance

A number of maintenance procedures need to be carried out at specified intervals, to maintain performance of the system. All procedures will be explained in detail in separate sections. Maintenance of the turbo-molecular pump water chiller and rotary pumps are explained. Maintenance of the source and exchanging the filament will be described in detail. Also explained is how to safely exchange empty helium and reference gas bottles.

CAUTION

Suitable nitrile or latex (powder free) gloves should be worn when working inside the vacuum chamber and when handling any source components.



CAUTION

Irritant liquid : Appropriate personal protective equipment should also be worn when handling any cleaning solvents and hydrocarbon pump oil.

3.1 Maintenance table overview

The following table gives an overview of the required maintenance procedures and some guidelines for the approximate intervals between procedures.

HINT: Note that the time intervals are very dependent on the usage of the system and the sample matrices being analyzed.

Maintenance procedure	Approximate time interval
Recirculating chiller water – Turbo-molecular pumps	3 months
Changing E2M1.5 rotary pump oil and foreline trap alumina beads	12 months
Changing the filament	12 to 24 months
Cleaning the source	18 to 36 months

Figure 3.1-1: Maintenance interval guidelines.

3.2 Maintenance of the water chiller

The Perspective utilizes one water chiller for the turbo-molecular pumps. The water level in the chiller should be checked regularly and requires changing periodically.



⚠ CAUTION

The chiller is not manufactured by Nu Instruments Ltd. Follow the operating and safety instructions detailed in the manufacturer's operating manuals that are supplied with the chillers.

A visual flow indicator is provided at the rear of the instrument, connected inline between the instrument water outlet and the chiller water inlet. It is recommended to check this every week, to ensure that reasonable water flow is present.

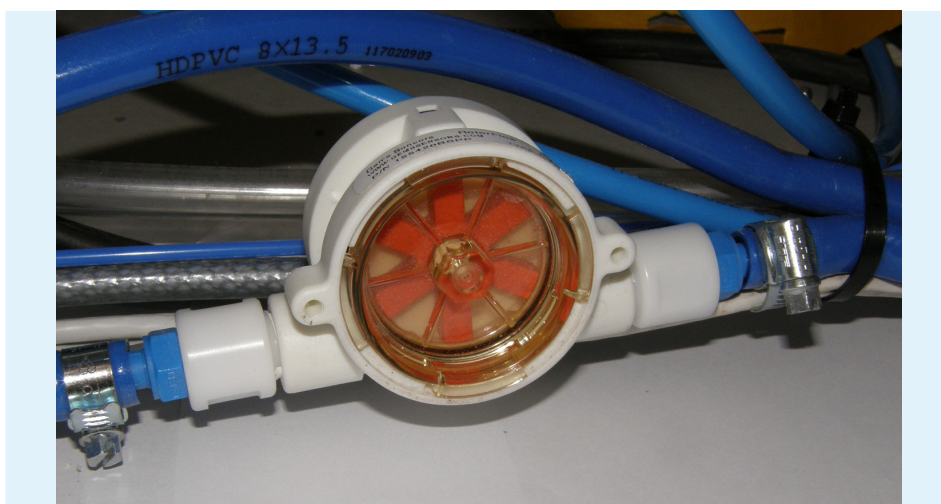


Figure 3.2-1: Checking the flow meter.

This chiller is used to regulate the temperature of all of the turbo-molecular pumps.

HINT: This chiller is set to cool the recirculating water to between 4 and 6 °C below that of the ambient air temperature. It is recommended to periodically monitor the temperature of the water in the chiller reservoir, using a suitable temperature probe.

Contact Nu Instruments Ltd for details of adjustments to the cooling conditions of this chiller, if necessary.



Figure 3.2-2: Checking the water level of the chiller.

The water level in the chiller should be checked monthly and topped up, if required, with tap water, or with deionized water, depending on the manufacturer's recommendation.

The images show a typical water chiller. Whichever chiller is installed, ensure that adequate access is available for checking the water level and topping up the water.

HINT: If a large amount of algae growth is visible in the water then it is recommended to change the water. Algae growth will be more extensive when the coolant is exposed to artificial or natural light, such as when transparent tubing is used.

HINT: If the water needs to be topped up more than once a week, check all tubing and connections for water leaks.

To drain the water from the chiller, follow the steps outlined.

1. Ensure that the water chiller is turned off and that the mains plug is disconnected.

**⚠ WARNING**

High voltage : Ensure that the chiller mains plug is temporarily held up away from the floor, perhaps using a cable tie. Do not leave it trailing on the floor.

2. Remove the lid of the reservoir.
3. It is recommended to raise the chiller and place a tray underneath it.

**⚠ CAUTION**

Lifting hazard : The water chiller is heavy and should not be lifted by only one person.

4. Unplug the **In** connector at the chiller bulkhead on the left hand side. Remove the connector from the chiller. In the example shown (ATC KT chiller with quick release fittings), this is done by pressing the gray button, as shown in the image below.
5. Press the button inside the connector to allow the water to drain from it. Hold the connector over the tray. The water will keep draining as long the button is pressed down. As soon as the button is released, the water will stop flowing.

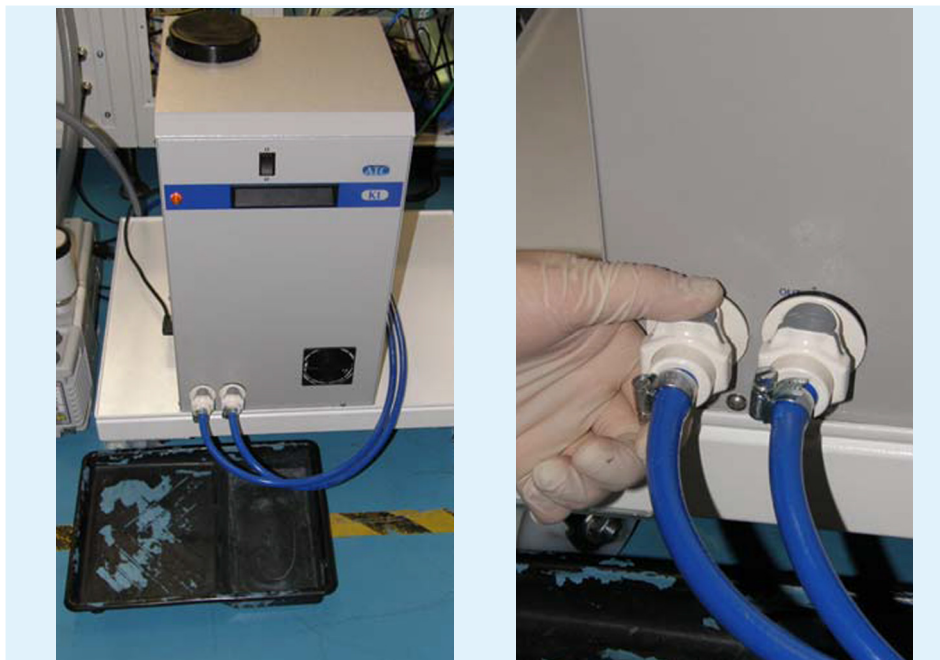


Figure 3.2-3: Raising the chiller prior to drainage and disconnecting the IN connector.

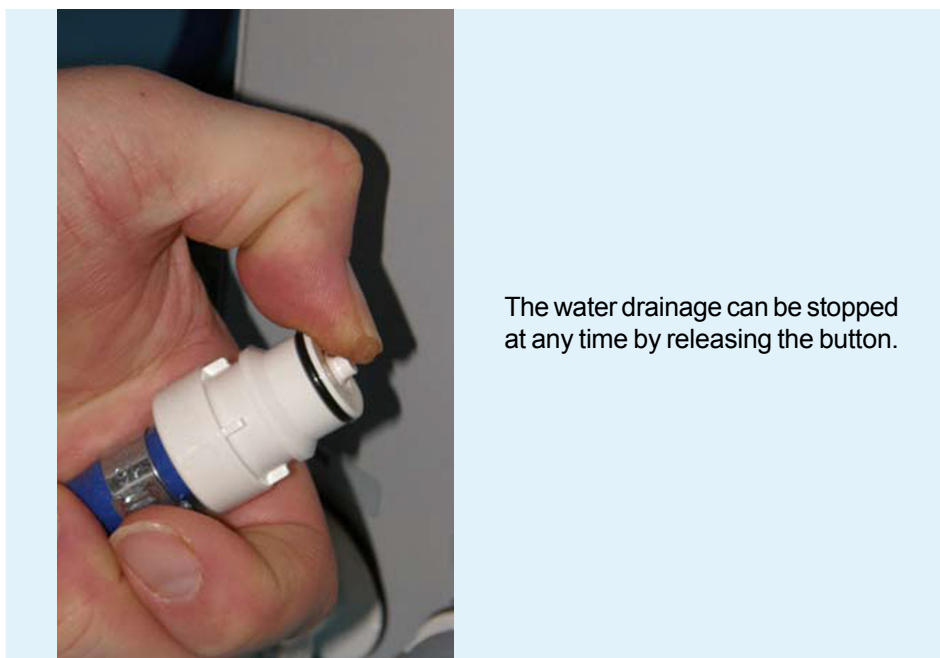


Figure 3.2-4: Draining the chiller.

6. The next step is to fill the water reservoir again. Fill the tank to a level recommended by the chiller manufacturer.
7. Turn on the chiller and check that water is being circulated, which can be observed by rippling of the water surface. Bubbles of air will appear in the tank for a short time, after which the water level may need topping up.
8. Observe the chiller water circulating into the reservoir for at least a minute, before replacing the lid.

⚠ CAUTION

Air locks can occur at the exit and entry hoses of the reservoir of the turbomolecular pump chiller, if the water has been completely drained and replaced. If an air lock is present, water circulation can be reduced. To release the air lock, tip the chiller at an angle of 30 degrees or so, with the reservoir lid in place.

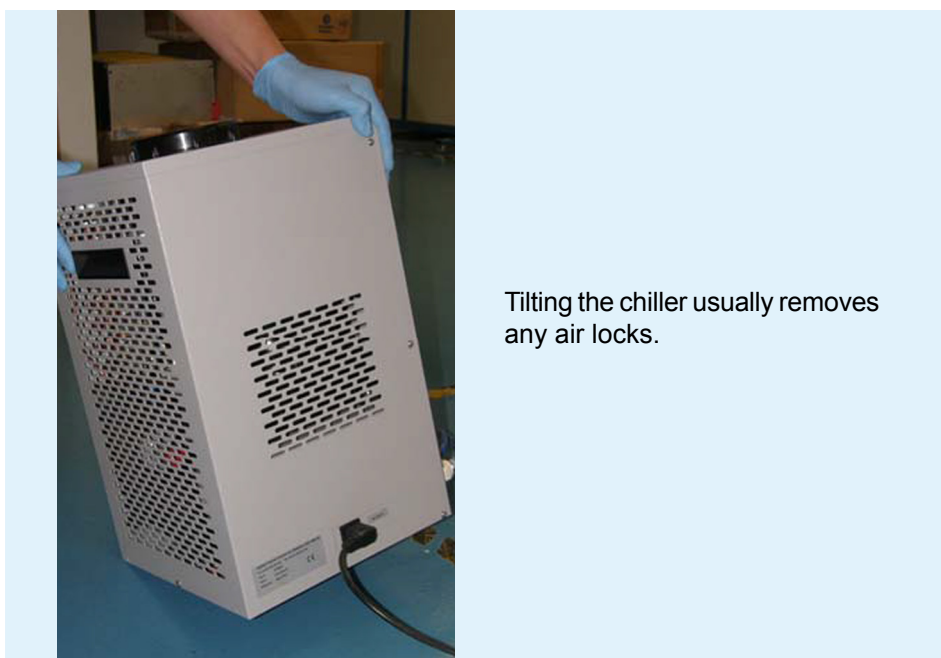


Figure 3.2-5: Releasing an air lock by tipping the chiller at an angle.

3.3 Maintenance of the rotary pump

The backing rotary pump of the Perspective needs regular oil changes. One E2M1.5 rotary pump is fitted to the main instrument for backing of the 3 instrument turbo-molecular pumps.

Edwards Ultra grade 15 oil should be used for the E2M1.5 rotary pump.



WARNING

Hot surface : Allow the pump body and the oil to cool sufficiently before carrying out the oil change.

3.3.1 Changing the oil in an E2M1.5 backing rotary pump

The oil in the E2M1.5 rotary pump can only be changed with the system vented. See Section 2.2 **Venting the instrument** for a description of venting the system.

Items required to change the oil include:

- a low trolley or platform to raise the pump before draining the oil.
- a tray of suitable capacity, into which the oil will be drained.
- the correct grade of oil to refill the pump.
- a funnel that fits into the oil fill hole.

To change the oil, complete the following steps:

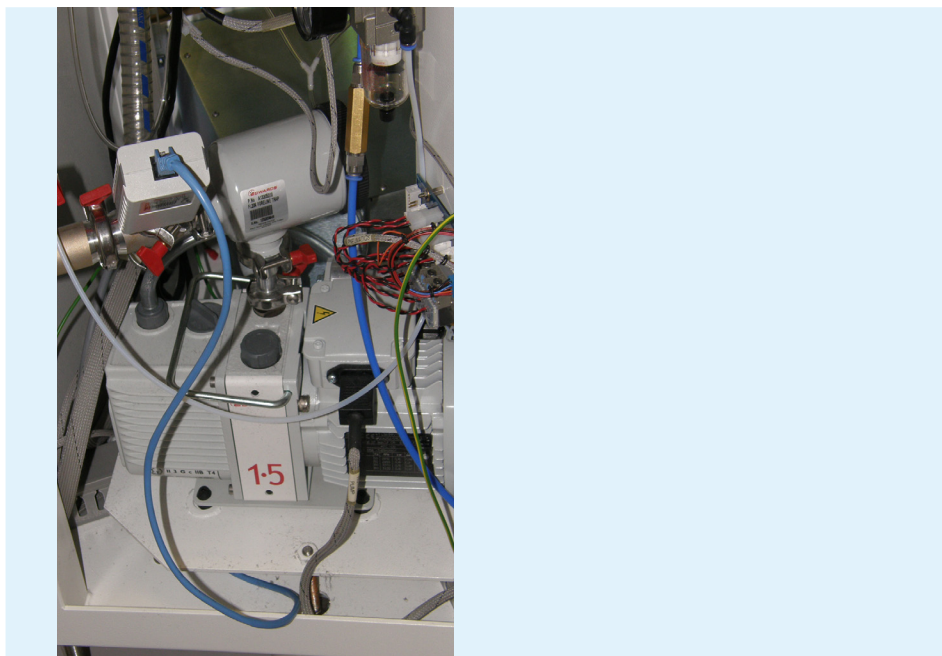


Figure 3.3-1: E2M1.5 rotary pump.

1. Part of the vent procedure is to turn off the rotary pump. Also now disconnect the mains lead to the rotary pump.
2. Disconnect the exhaust tubing from the rotary pump.
3. Allow the pump to cool for 5 minutes and then break the connection between the rotary pump and the instrument pump line, by carefully unscrewing the C-clamp connection directly on the rotary pump.
4. Lift the rotary pump off the rotary base onto the platform and place the tray underneath the drain plug, in order to drain the oil.
5. Allow the pump to cool for a further 5 minutes (ten minutes cooling in all).



Figure 3.3-2: Placing the pump on a platform or trolley to drain the oil.

6. Open the oil fill cap. This is important, since removing the fill cap allows the oil to drain quickly.

7. Unscrew and remove the oil drain plug and allow the oil to drain into the tray.



Figure 3.3-3: Removing the oil fill cap and drain plug.

Refilling the oil

HINT: Ensure that the correct oil is used for filling.

1. Replace the oil drain plug and place the funnel in the oil fill hole.
2. Fill the pump with new oil through the oil fill hole. The oil level in the viewing glass will rise.

CAUTION

Do not fill too quickly, in case the oil level exceeds the maximum oil fill indication, on the oil level viewing glass.

3. Replace the oil fill cap once the pump is at least 2/3 full.
4. Remove the tray containing the old oil and dispose of the oil according to local environmental guidelines.
5. Lift the pump back to its operating position and remove the trolley or platform from the working area.
6. Clean up any oil spill from around the oil fill cap and the drain plug.

Switching the pump on again

1. Reconnect the pump to the main instrument, ensuring that the C-clamp connection is fully tightened.
2. Reconnect the exhaust tube and the power connector.
3. Switch on the pump, observing that the oil level remains sufficiently high.
4. Return the instrument to vacuum, following the procedure in **Section 2.3 Pumping down**.

3.3.2 Changing the beads in the foreline trap cartridge

HINT: Ensure that all of the tools to be used in this procedure have been cleaned with propanone (acetone) before starting.

HINT: It is recommended to change the alumina beads in the foreline trap cartridge whenever the oil in the rotary pump is exchanged.

HINT: If the alumina beads that will be placed into the foreline trap have been exposed to atmosphere for any length of time, they should be dried in an oven at 100°C for 1 hour before putting them into the cartridge.

The purpose of the foreline trap is to prevent migration of oil vapor from the rotary pump to the vacuum system. The foreline trap contains beads of an activated alumina molecular sieve, housed in a cartridge.



CAUTION

Irritant gases: Take appropriate precautions if irritant gases are used in conjunction with the instrument. Irritant gases may adsorb onto the alumina beads and could be released when the beads are removed from the foreline trap. Do not bake the alumina beads to reactivate them if irritant gases have been used.

Removing the used alumina beads from the foreline trap

1. To change the beads in the foreline trap cartridge, the foreline trap must be removed. Unfasten the upper retaining clip that secures the trap to the pump and loosen the retaining clip that secures the trap to the Pirani gauge.
2. Lift off the trap and place the trap on a clean work surface.



Figure 3.3-4: Removing the foreline trap.

3. Unscrew the large retaining wing nut and lid from the body of the foreline trap. A spring beneath the lid keeps the cartridge in place at the bottom of the trap.
4. Remove the cartridge and spring from the trap body.



Figure 3.3-5: Opening the foreline trap and removing the spring and cartridge.

**⚠ CAUTION**

Irritant gases: Take appropriate precautions if irritant gases are used in conjunction with the instrument. Irritant gases may adsorb onto the alumina beads and could be released when the beads are removed from the foreline trap. Ensure safe disposal of used alumina beads immediately after they have been removed from the cartridge.

5. Place the cartridge on the work surface with the wing nut facing upwards.
6. Remove the wing nut and washer from the spindle of the cartridge.



Figure 3.3-6: Opening the cartridge.

**⚠ CAUTION**

Irritant powder : Alumina powder is an irritant to the respiratory system and to the eyes. Avoid inhaling alumina powder during the removal and replacement of the alumina beads and wear safety glasses during this stage of the procedure.

7. Lift off the end cap from the cartridge, along with the washer below the end cap.
8. Remove the compression spring and perforated disk.

9. The used beads can now be removed from the cartridge for safe disposal.



Figure 3.3-7: Removing the end cap, revealing the perforated disc.

10. If the replacement beads have been stored in an airtight container, they may be placed directly into the cartridge. If, however, the beads have been exposed to the atmosphere for any length of time, they should be dried in an oven at 100°C for one hour to dry them.
11. Add alumina beads, so that the level of beads added is approximately level with the marker on the spindle.
12. Tap the cartridge to settle the beads into place, then add more beads if necessary.
13. Position the perforated disk on top of the beads with its smooth surface facing down onto the beads.
14. Position the spring, the washer and the end cap.
15. Place the second washer and wing nut onto the spindle and tighten the wing nut, pushing downwards gently on the lid.
16. Place the cartridge into the trap, ensuring that it is correctly located on the bottom O-ring seal.
17. Replace the spring and screw the large wing nut and lid onto the foreline trap.
18. Refit the trap onto the rotary pump.

3.4 Filament, lens and source block procedures

Before any work is performed on the source or filament the first step is to vent the instrument, as described in **Section 2.2 Venting the instrument**.

Next, turn the **Source HT** key on the back of the instrument to the service setting. For added safety, turn off **Source HT**, **Source Filament** and **HV Supplies** on the MDU panel.

In order to access the lens stack, source block and filament, the cover and connectors must firstly be removed, followed by removal of the complete source assembly from the vacuum chamber, which will be explained in **Section 3.4.1**.

If only an exchange of filament is required, the source assembly does not need to be disassembled for this task. The exchange of a filament is described in **Section 3.4.2**.

In order to clean the lens stack or the source block, the source assembly will be dismantled. Before this is possible, all of the connectors must be removed. This is described in **Section 3.4.3**.

The separation of the source block and the lens stack is described in **Section 3.4.4**.

Once separated from the source block, the lens stack can be dismantled, which is described in **Section 3.4.5**.

The user may then wish to dismantle the source block, which also houses the trap, for cleaning purposes. The disassembly and re-assembly of the source block is described in **Section 3.4.6**.

The assembly of the source block is described in **Section 3.4.7**.

The cleaning procedure, which is applicable to both the lens stack and the source block, is described in **Section 3.5**.

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all tools to be used have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component, as it is a contaminant for CO₂ analysis.

HINT: To ensure that components are not lost during disassembly, place them in clean glass beakers or storage boxes. Keep ceramic and metal components in separate containers, to ensure that they are not damaged.

3.4.1 Schematic of the source assembly

The schematics below show the filament, lens and source block inside the vacuum chamber.

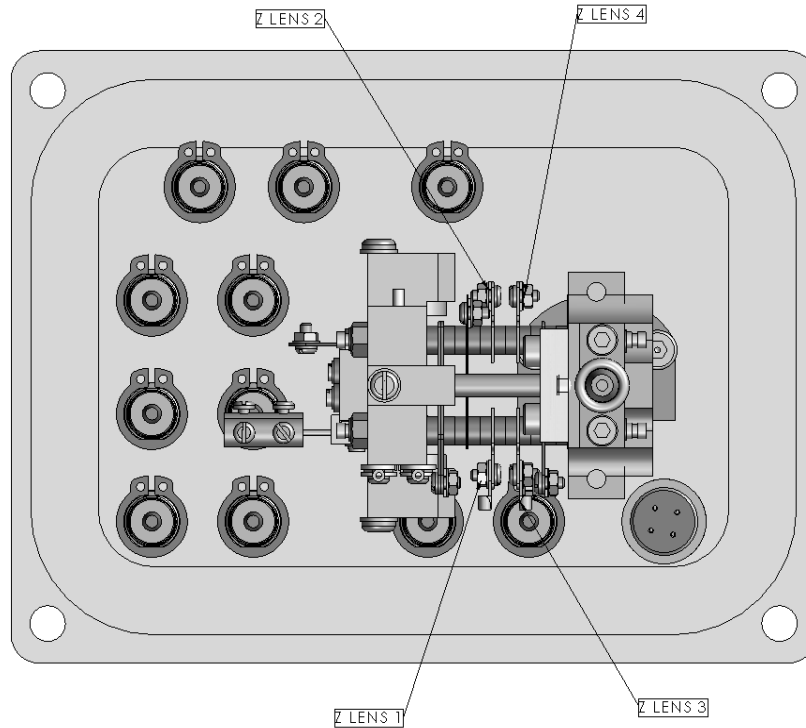


Figure 3.4-1: Filament, lens and source block inside the vacuum chamber. Note that the number of ceramic spacers between the source plates may be different to that displayed in this diagram.

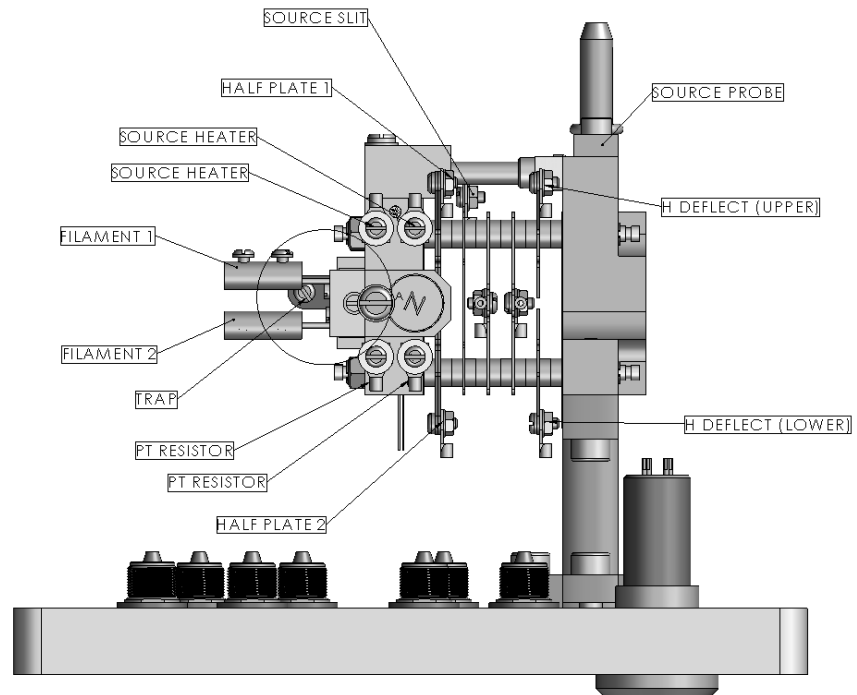


Figure 3.4-2: Schematic of the lens stack and source block. Note that the number of ceramic spacers between the source plates may be different to that displayed in this diagram.

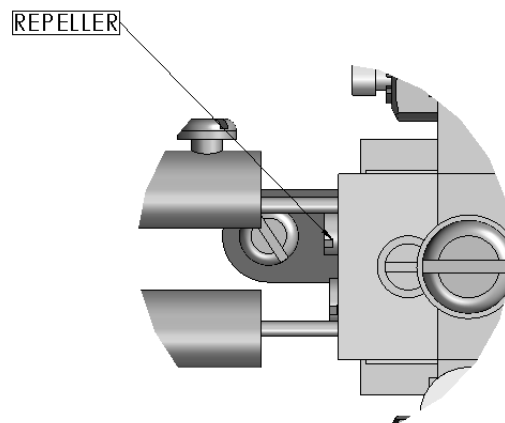


Figure 3.4-3: Detailed view of the repeller.

The table below lists and describes all of the wiring connectors to the source block, filament and lens stack.

WIRING INFORMATION		
CONNECTOR/CONNECTION	CONNECTS TO	WIRE TYPE
FILAMENT 1 CONNECTOR	FILAMENT 1	0.9MM GOLD PLATED COPPER
FILAMENT 2 CONNECTOR	FILAMENT 2	0.9MM GOLD PLATED COPPER
TRAP CONNECTOR	TRAP	0.6MM GOLD PLATED COPPER
SOURCE CONNECTOR	SOURCE	0.6MM GOLD PLATED COPPER
REPELLER CONNECTOR	REPELLER	0.6MM GOLD PLATED COPPER
HP 1 CONNECTOR	HALF PLATE 1	0.6MM GOLD PLATED COPPER
HP 2 CONNECTOR	HALF PLATE 2	0.6MM GOLD PLATED COPPER
HEATER 1 CONNECTOR	SOURCE HEATER	0.9MM GOLD PLATED COPPER
HEATER 2 CONNECTOR	SOURCE HEATER	0.9MM GOLD PLATED COPPER
SENSOR 1 CONNECTOR	PT RESISTOR	0.6MM GOLD PLATED COPPER
SENSOR 2 CONNECTOR	PT RESISTOR	0.6MM GOLD PLATED COPPER
SOURCE SLIT	SOURCE PROBE	0.6MM GOLD PLATED COPPER
H DEFLECTOR (UPPER)	SOURCE PROBE	0.6MM GOLD PLATED COPPER
4 WAY CONNECTOR PIN 3	H DEFLECTOR (LOWER)	22AWG PTFE RED SOLID CORE
4 WAY CONNECTOR PIN 1	Z LENS 1	22AWG PTFE BLUE SOLID CORE
4 WAY CONNECTOR PIN 2	Z LENS 2	22AWG PTFE BLACK SOLID CORE
Z LENS 1	Z LENS 3	0.6MM GOLD PLATED COPPER
Z LENS 2	Z LENS 4	0.6MM GOLD PLATED COPPER

Figure 3.4-4: Listing of all wiring connectors.

3.4.2 Removing the source assembly from the vacuum chamber

Tools required: Hexagon keys

1. Remove the source region cover by taking out the two screws and then lifting off the cover.

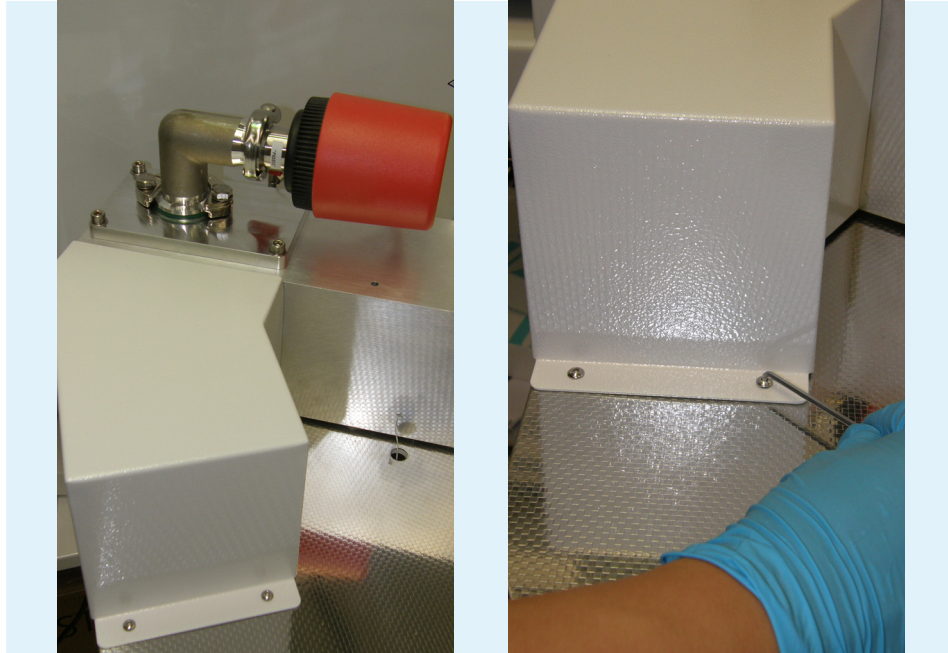


Figure 3.4-5: Removing the cover.

2. Use both hands to take off the cover. Remove the earth connector.

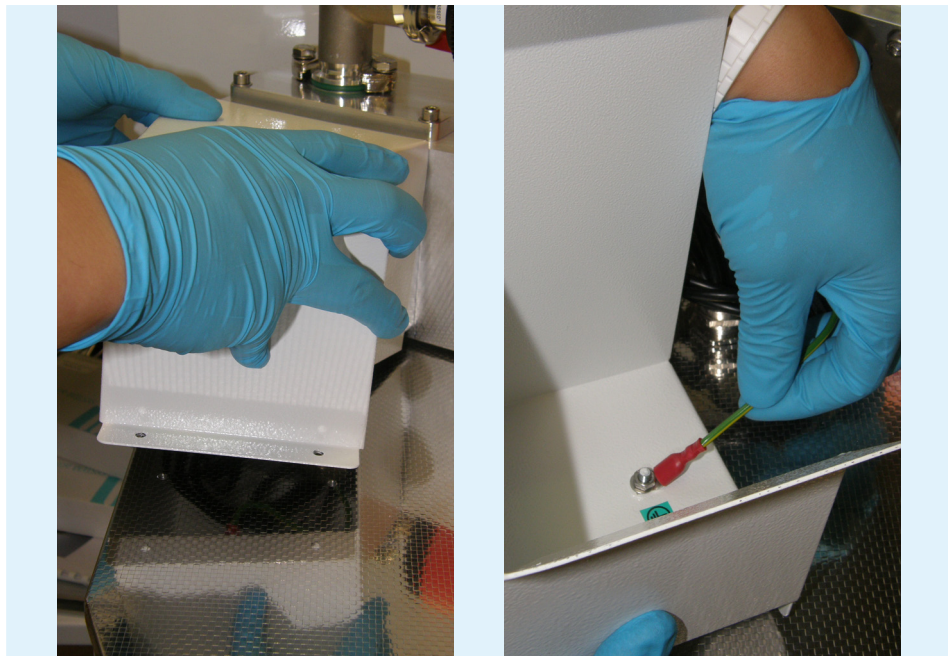


Figure 3.4-6: Taking off the cover and removing the earth connector.

3. Unplug all of the cable connectors. Do not pull on the cables, only pull on the connectors to unplug them.

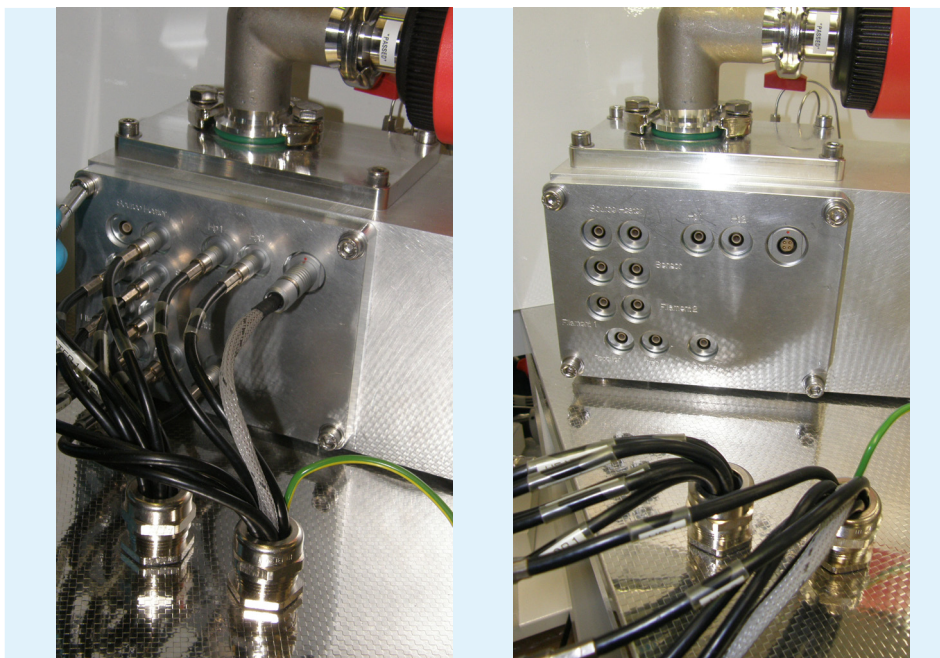


Figure 3.4-7: Removing the cable connectors.

4. The next step is to remove the flange. Take out the hexagon screws that secure the flange to the vacuum chamber. Carefully remove the flange.

5. It is recommended to place aluminium foil over the source housing, when the source flange has been removed, to reduce the risk of contaminating the mass spec.

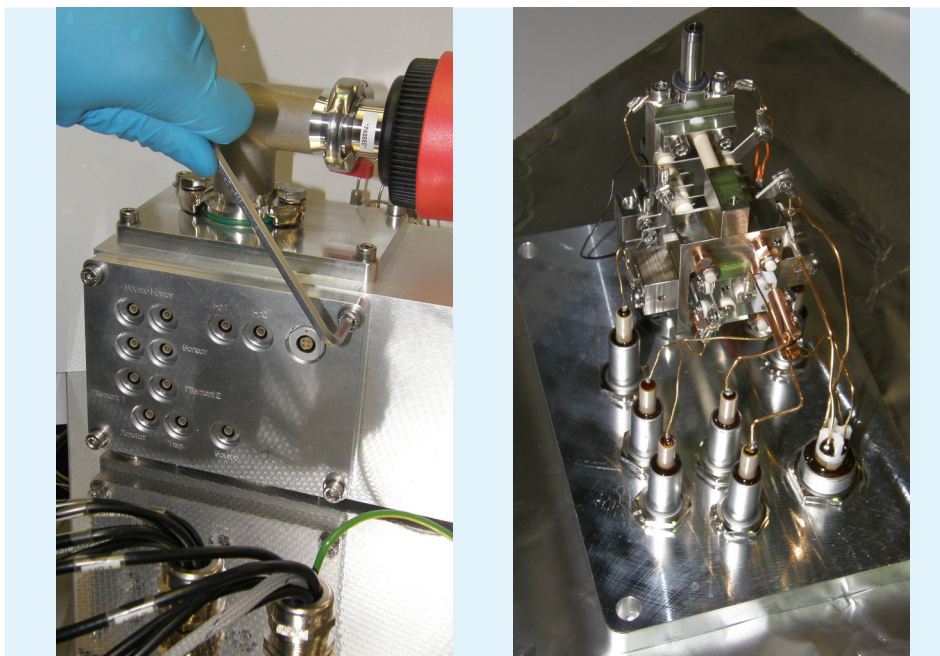


Figure 3.4-8: Removing the flange.

3.4.3 Replacing the filament

1. When the flange has been removed, as described in **Section 3.4.1**, turn it on its side and place it on some bunched up aluminium foil.

Removing the old filament will be described first, followed by the insertion of the new filament.

Removing the old filament

Tools required: Small flat bladed screwdriver; tweezers

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and ensure gloves are worn at all times.

HINT: Ensure that all tools to be used have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component, as it is a contaminant for CO₂ analysis.

2. Firstly remove the source inlet gasket to a safe location.
3. Loosen the screws connecting the filament legs to the brass connector block. Only loosen both screws on the side attached to the filament, not the side going down to the feedthrough.
4. Carefully pull off the connector to the filament.

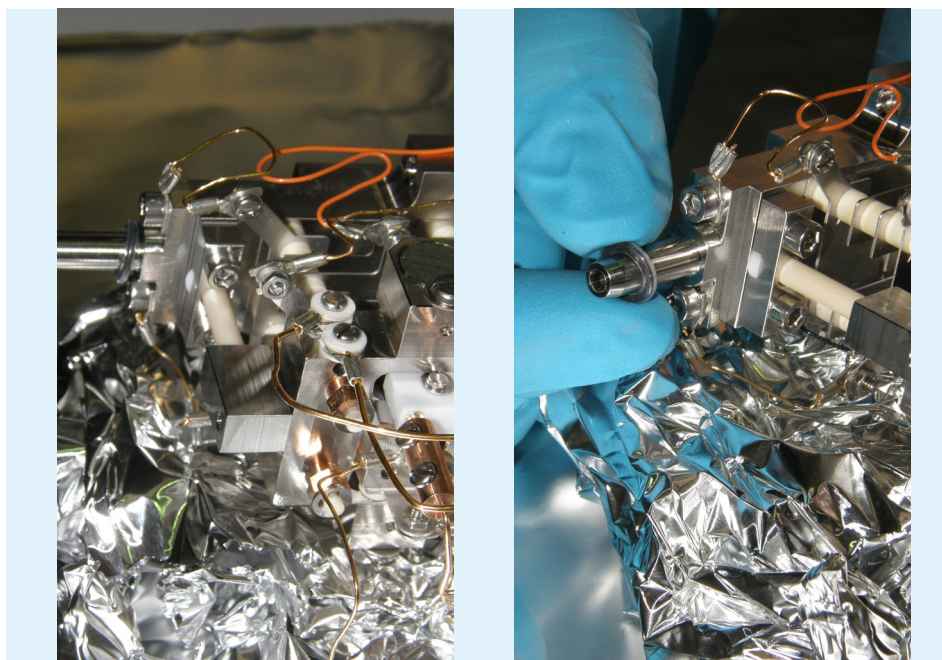


Figure 3.4-9: Taking off the source inlet gasket.

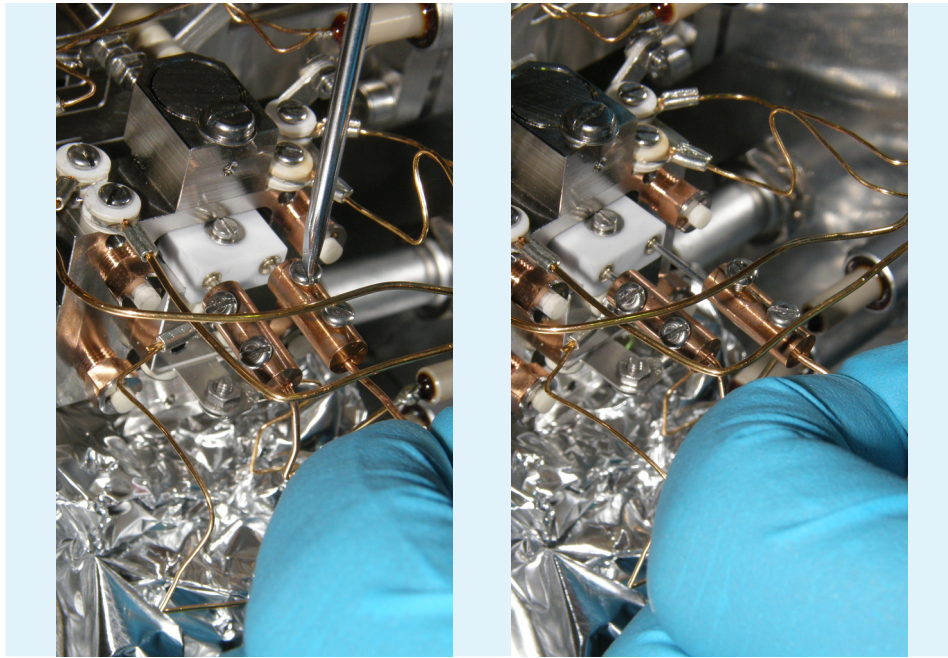


Figure 3.4-10: Removing the connector.

5. Loosen the screw on the top of the filament insulator.
6. Once the screw is loosened, take it out using tweezers.

HINT: Be aware that there is a magnet adjacent to these screws, so they will be attracted towards it.

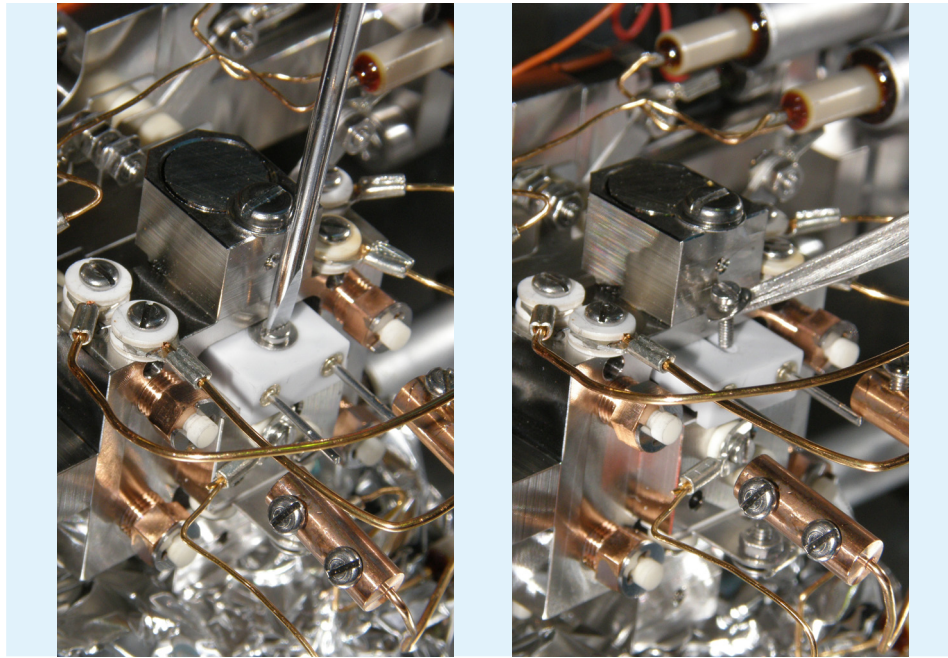


Figure 3.4-11: Removing the top screw and lifting it out with tweezers.

7. Pull the filament out using tweezers.

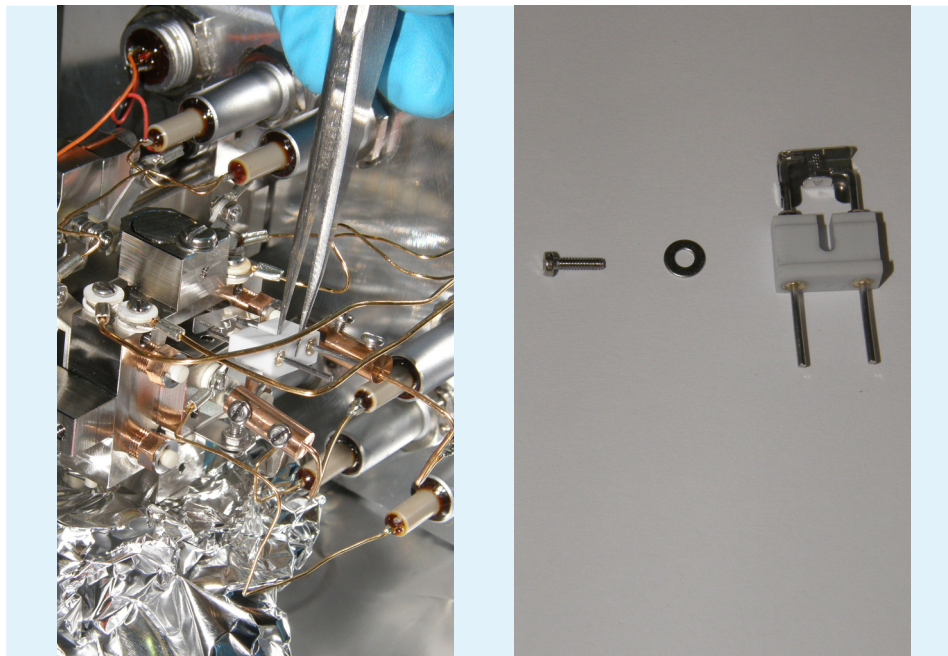


Figure 3.4-12: Pulling the filament out and showing the filament parts.

8. Exchange the filament. The filament components, shown left to right in the above figures are M1.6 mm x 6 mm screw, M1.6 washer and the filament.

Fitting the new filament

1. Firstly, place the filament into the source block on top of the wraparound. The lip of the filament should be facing the wraparound, so that the white part of the filament facing upwards is flat. The lip acts as a guide to ensure that the filament is positioned correctly.
2. Put in the screw and the washer.

HINT: The fitting of the new filament is shown in this section away from the main assembly for clarity.

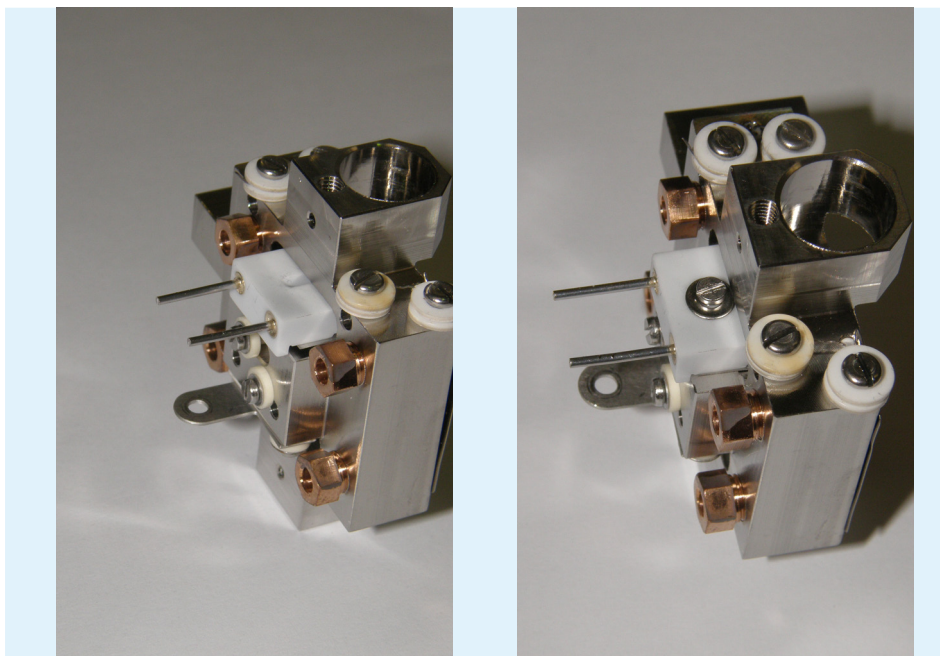


Figure 3.4-13: Fitting the new filament.

3. Screw the filament in loosely to begin with, then push the filament in as far as it will go.
4. The filament lip should fit up against the source block. Finally tighten the screw to just beyond finger tight.
5. The side view of a filament correctly fitted in the block is shown in the figure below.

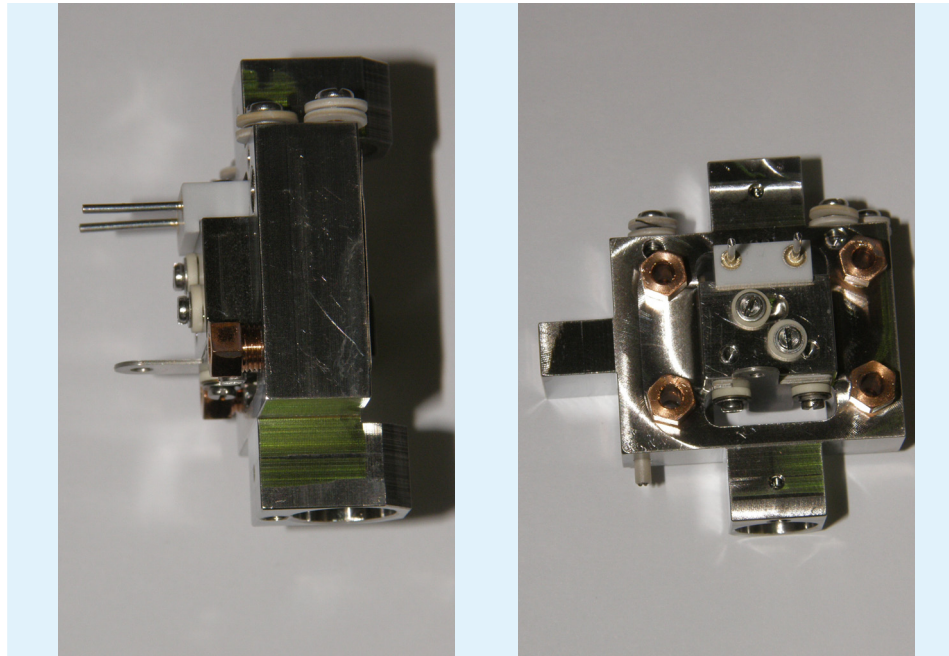


Figure 3.4-14: Correct alignment of the new filament.

6. If all that was required was a filament change then follow the reverse of the above procedure.

HINT: If the source assembly has been removed from the housing it is worth checking the continuity of the connections on the outside to ensure that none of the uninsulated wires in the source are touching the housing.

3.4.4 Taking off the connectors

Tools required: Small flat bladed screwdriver; tweezers; 4 mm spanner

There are a number of connectors, all of which must be removed before removing the source assembly from the source flange. The image below shows the source assembly removed from the flange, with all of the connectors removed, which is the aim of this section.

All of the connectors have to be removed, before the source block and the lens stack can be separated, as will be described in **Section 3.4.4**.

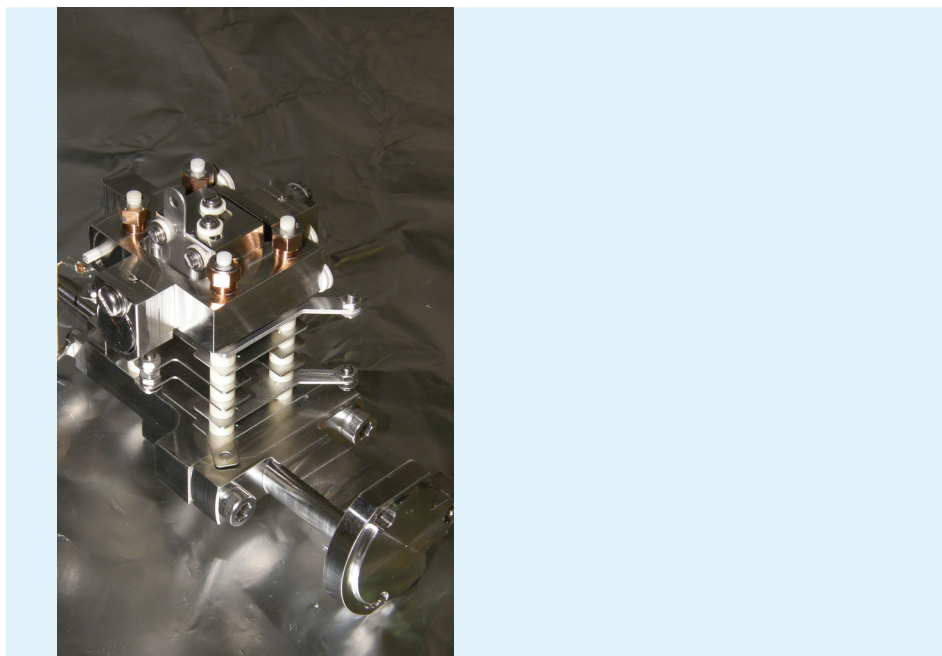


Figure 3.4-15: Source assembly with all connectors removed (aim of this section).

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all of the tools have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component as it is a contaminant for CO₂ analysis.

HINT: After removing the connections, it is recommended to tighten the screws back in a little, so that they do not fall out.

1. Firstly, using the screwdriver and spanner, loosen the connectors to the repeller and the trap and remove the connectors.

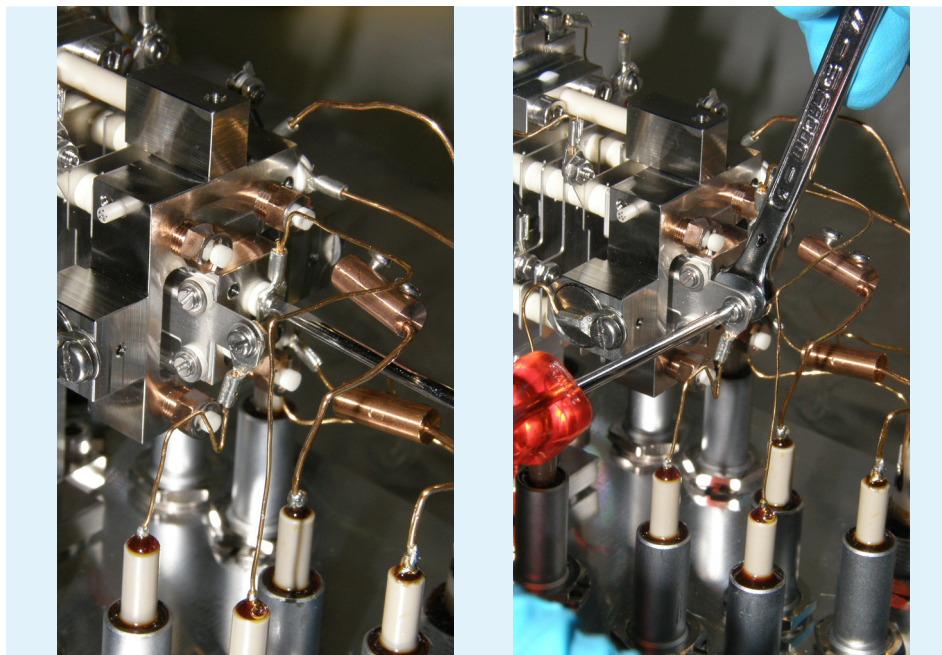


Figure 3.4-16: Loosening the connectors to the repeller and the trap.

2. Remove the connector to the source heaters, then continue to remove all other connectors.

HINT: Take extra care when disconnecting the heater end sensor crimp terminals, as they are sandwiched between two fragile ceramic insulating washers.

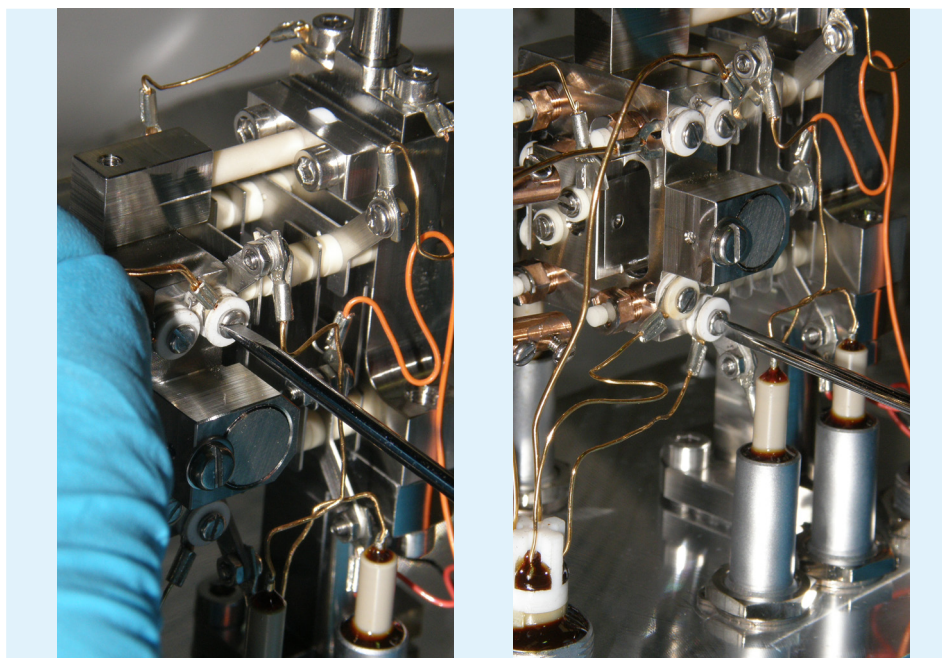


Figure 3.4-17: Removing the connector to the source heater, then the other connectors.

3. Continue to remove all other connectors, using the 4 mm spanner where required. Remember that there are connectors all round the source assembly.

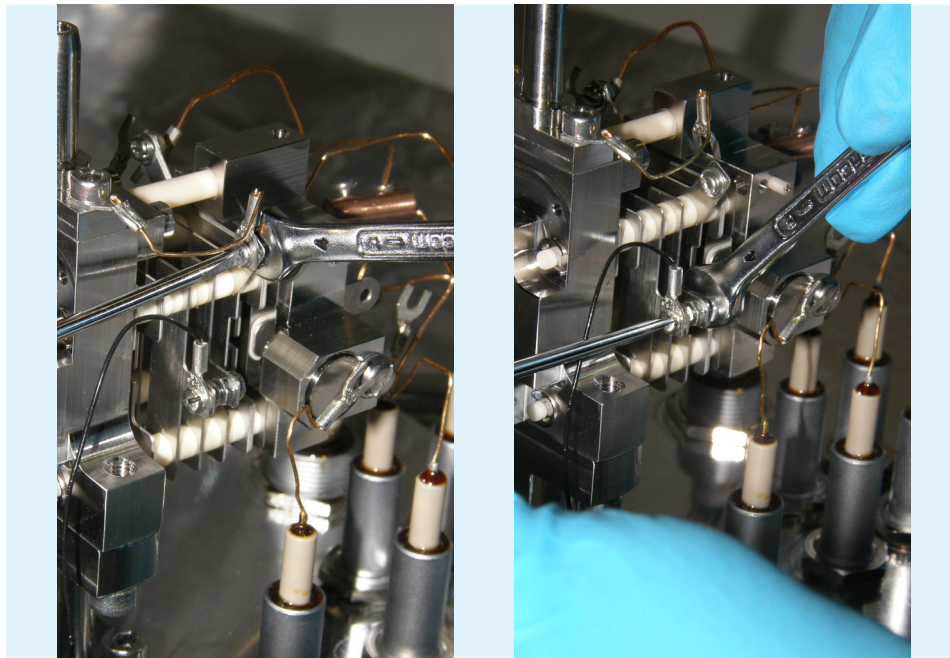


Figure 3.4-18: Removing the connector to the source heater, then the other connectors.

4. Once all of the connectors are removed, the source assembly should appear as it is shown in the figure at the start of this section.

3.4.5 Separating the source block and lens stack

Tools required: Hexagon keys, tweezers, 5.5mm spanner, flatbladed screwdriver.

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all of the tools have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component as it is a contaminant for CO₂ analysis.

HINT: To ensure that components are not lost during disassembly, place them in clean glass beakers or storage boxes. Keep ceramic and metal components in separate containers, to ensure they are not damaged.

Once all of the connectors are removed, the source block can be separated from the lens stack.

1. Using a hexagon key, remove the three screws that secure the source to the flange.
2. The source assembly will now lift off the source flange.

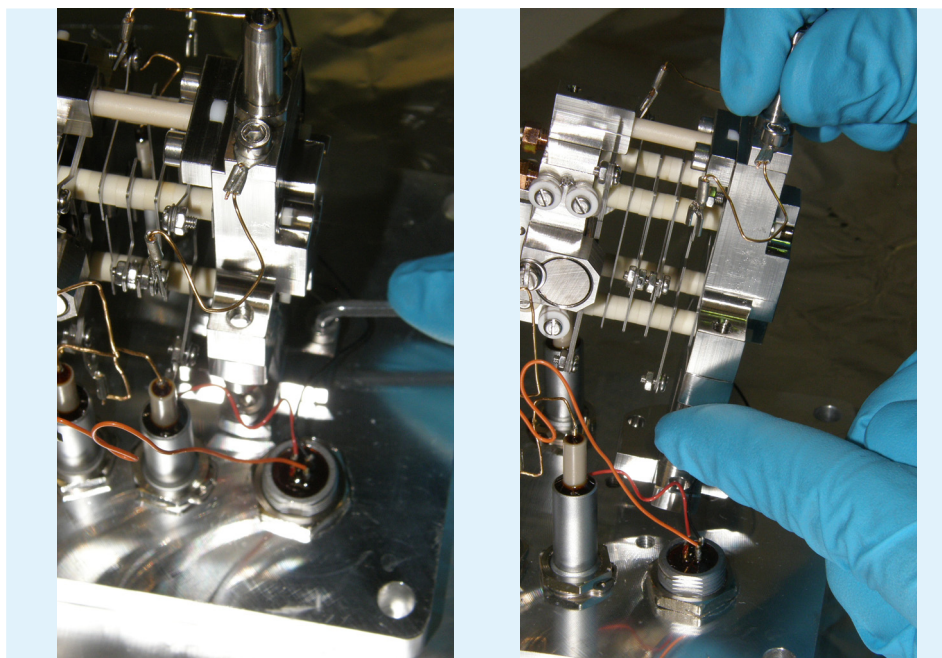


Figure 3.4-19: Removing the screws to lift off the source assembly.

3. Cover a table surface with aluminium foil and place the source assembly on it.
4. The next step is to remove the source block. Before removing it, the tension on the ceramic block should be released. Turn the ceramic rod tensioning nut clockwise using the 5.5 mm spanner. Screwing in will release the tension on the ceramic block.

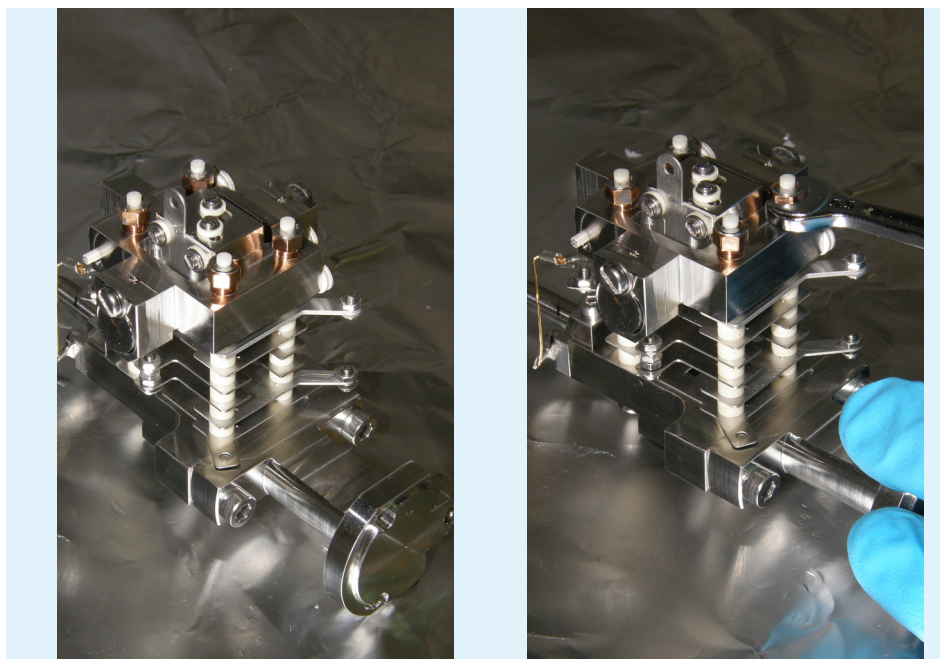


Figure 3.4-20: Releasing the tension on the ceramic block.

5. Use a small flat bladed screwdriver to remove the C-clips. Remove them on all four rods.

HINT: With the C-clips removed, do not lift up the entire source assembly. All components are free to move and the rods can easily fall out.

6. The next step is to lift off the complete source block.

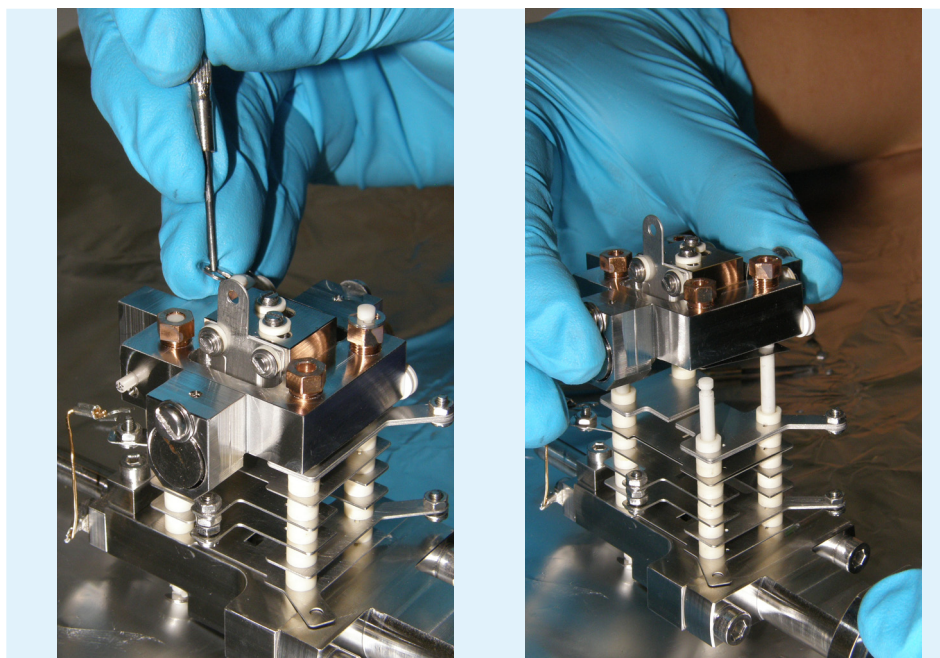


Figure 3.4-21: Removing the C-clips and lifting off the source block.

3.4.6 Dismantling the lens stack

HINT: Note down the order of placement, as well as the orientation of the source lens stack plates before disassembly.

Tools required: Tweezers

HINT: Since the source assembly is an internal component it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all of the tools have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component as it is a contaminate for CO₂ analysis.

HINT: To ensure that components are not lost during disassembly place them in clean glass beakers or storage boxes. Keep ceramic and metal components in separate containers to ensure that they are not damaged.

The source lens stack requires periodic cleaning, with a timescale that depends on the number and types of samples analyzed. This section describes the disassembly of the lens stack. The cleaning procedure is described in **Section 3.5**.

1. Take off the ceramic spacers.
2. Take off the two half plates. Each half plate consists of two plates.
3. Take care to remove the metal plates from the ceramic rods as gently as possible. Scraping the metal against the rods can deposit metal onto the ceramic. This may affect the insulating capability of the rods.

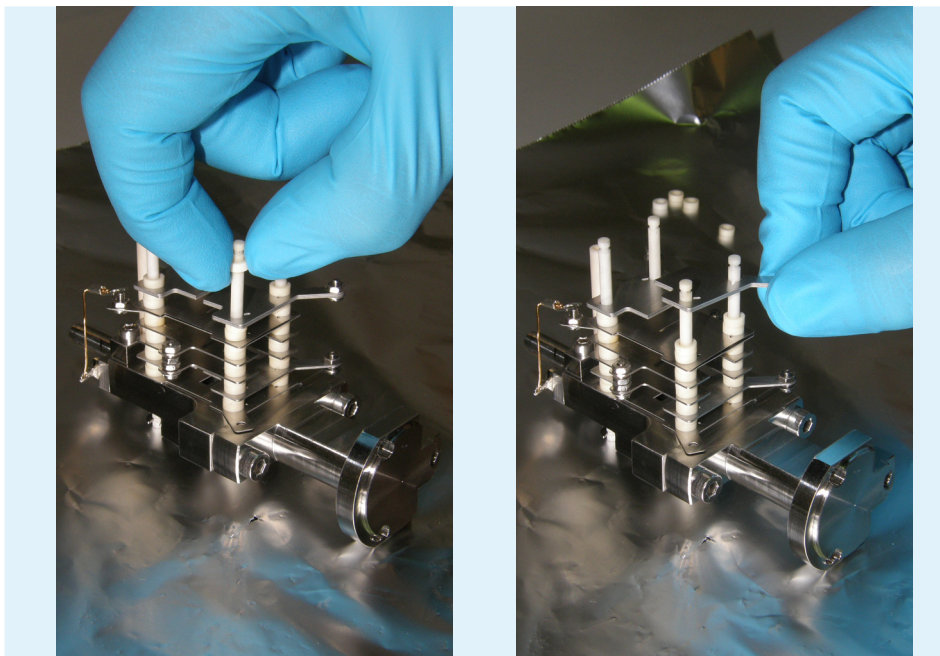


Figure 3.4-22: Removing the ceramic spacers and half plates.

4. Next, remove the ceramic spacers.
5. Remove the two source defining plates. The two plates are mounted together, each with a different slit size.

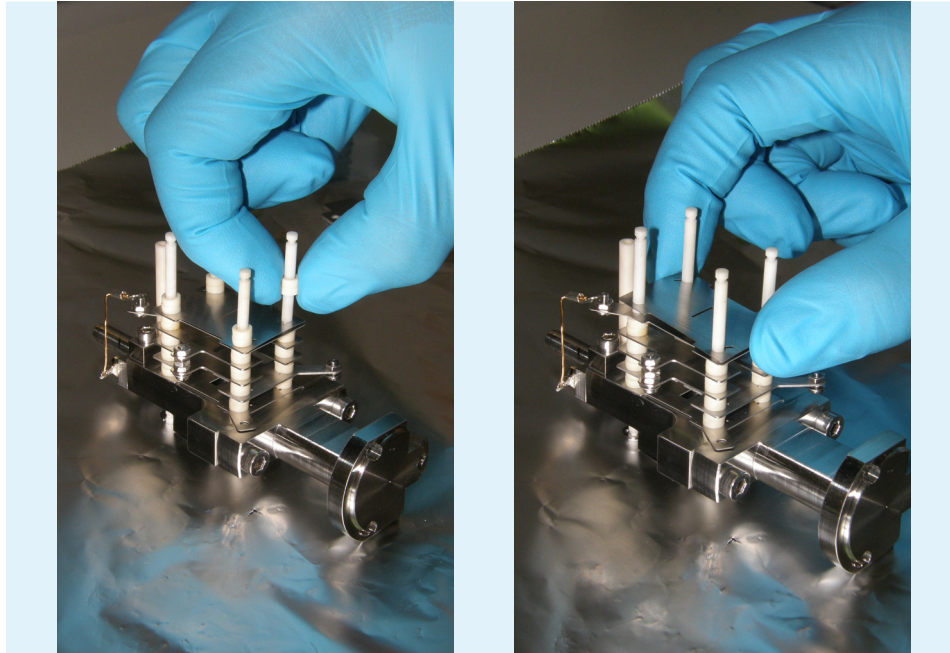


Figure 3.4-23: Removing the ceramic spacers and two source defining plates.

6. The figure below shows the two source defining plates next to each other, for comparison.
7. Remove the ceramic spacers.

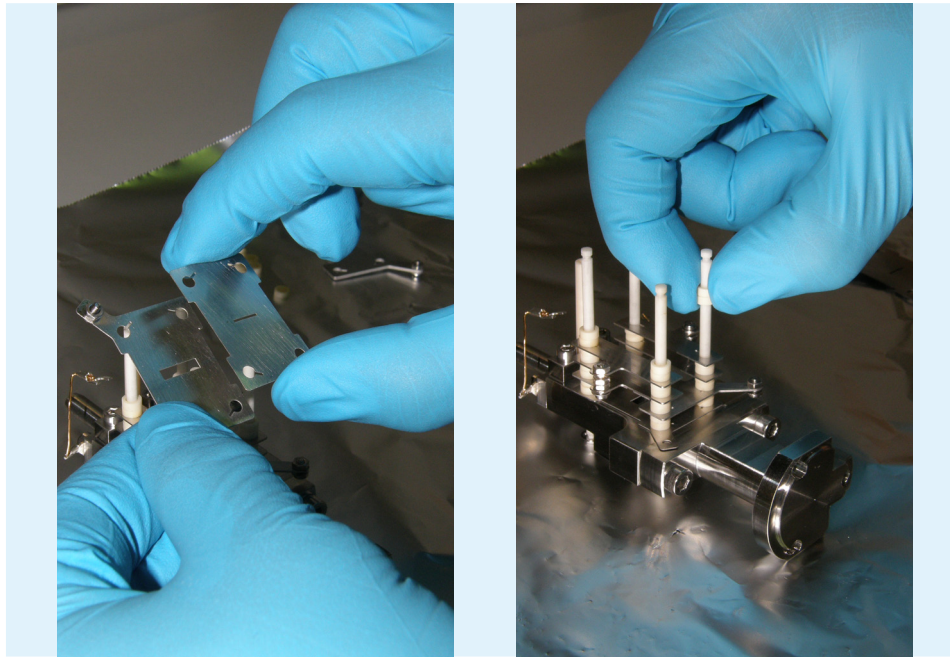


Figure 3.4-24: Comparing the two source defining plates and removing ceramic spacers.

8. Remove the two Z-plates, which are joined together. They must be removed in two steps.

9. Remove two ceramic spacers from one Z-plate pair, followed by the plate pair.

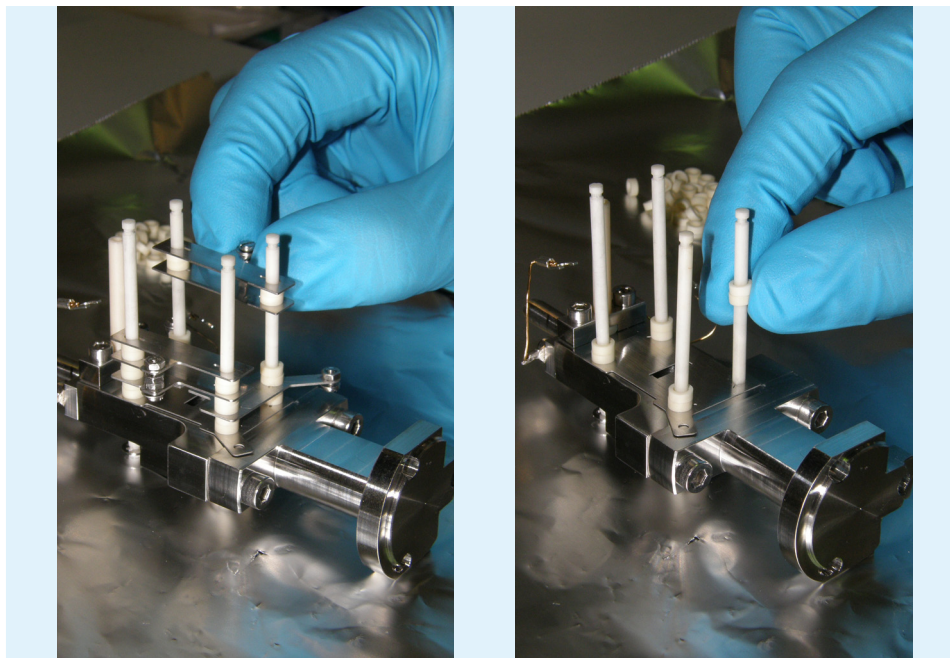


Figure 3.4-25: Removing the two Z-plates and 8 ceramic spacers.

10. Repeat for the other Z-plate pair.

11. Note that there are 8 ceramic spacers in total.

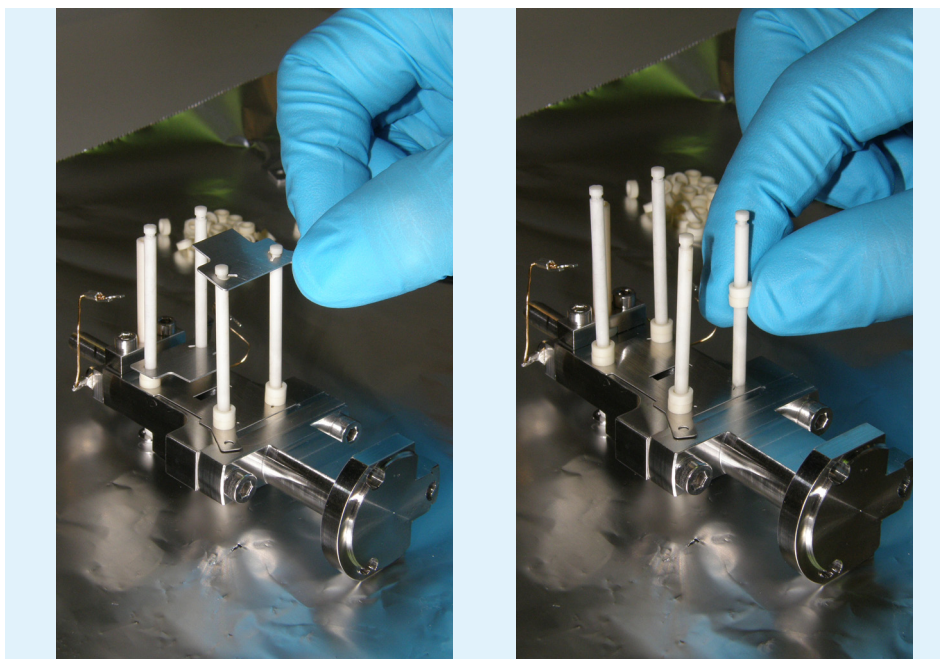


Figure 3.4-26: Removing the two horizontal deflector plates and 8 ceramic spacers.

12. Remove the H-defl plates.

13. The lens stack is now disassembled. If the ceramic rods require cleaning, the remaining block can be removed, then take off the C-clips.

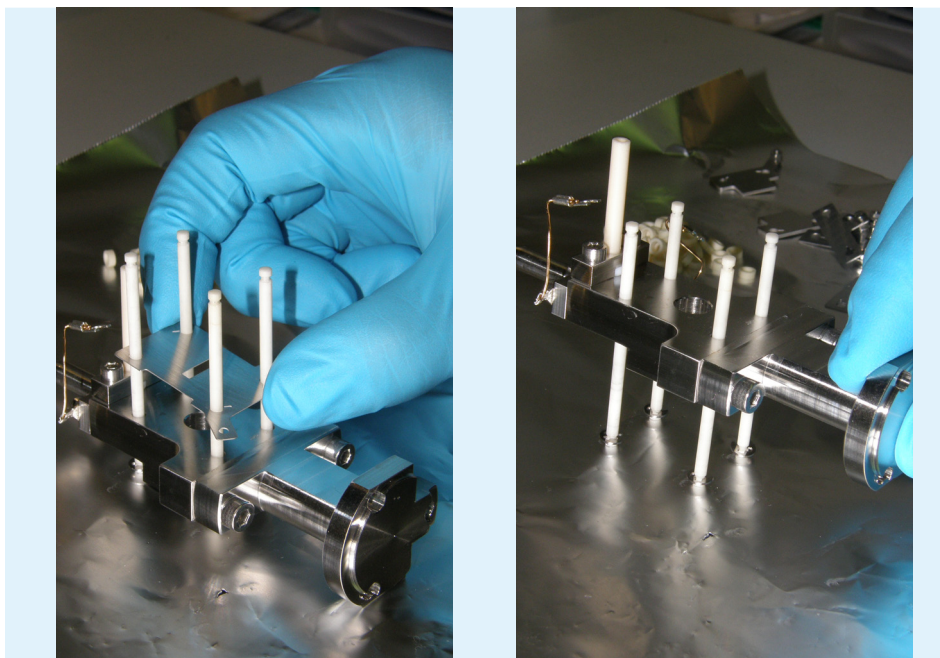


Figure 3.4-27: Removing the final source plate.

14. Follow the cleaning procedure described in **Section 3.5**.

15. Reassemble the lens stack in the reverse order to the sequence above.

3.4.7 Disassembling the source block

This section will describe the disassembly of the source block and the next section will describe the reassembly of the source block, as special care has to be taken with certain steps.

Tools required: Small flat bladed screwdriver; tweezers; 5.5 mm spanner

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all of the tools have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component as it is a contaminant for CO₂ analysis.

HINT: To ensure that components are not lost during disassembly, place them in clean glass beakers or storage boxes. Keep ceramic and metal components in separate containers, to ensure that they are not damaged.

To disassemble the source block, follow the procedure described below.

1. Take off the trap. Remove the first two screws and insulating ceramics to gain access to the trap.

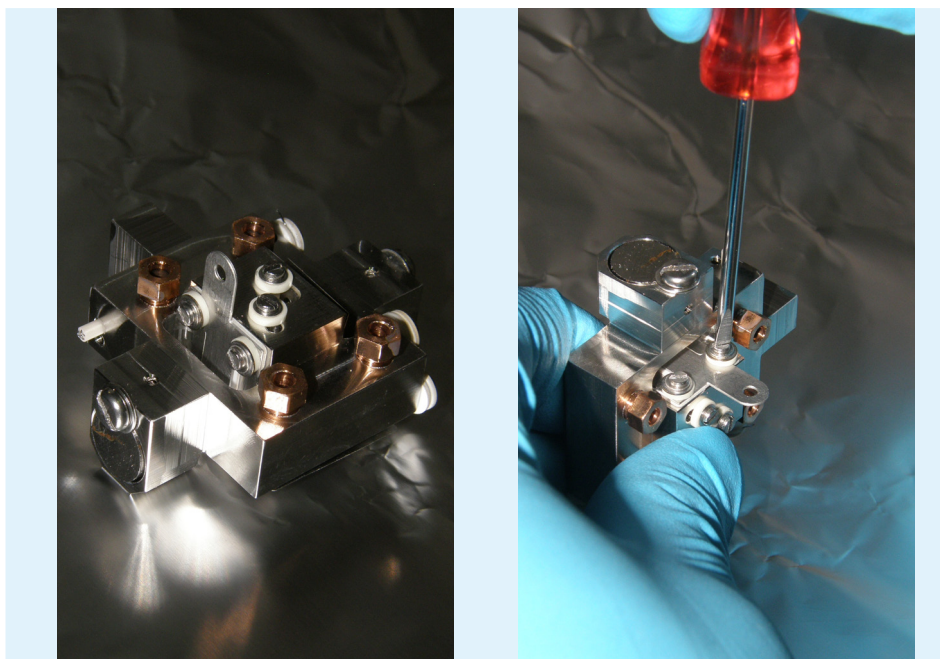


Figure 3.4-28: Removing the first two screws to disassemble the source block.

2. After removing the first two screws, take out the metal trap using tweezers.

3. Take out the ceramic plate beneath the trap, which is the same shape as the trap itself.

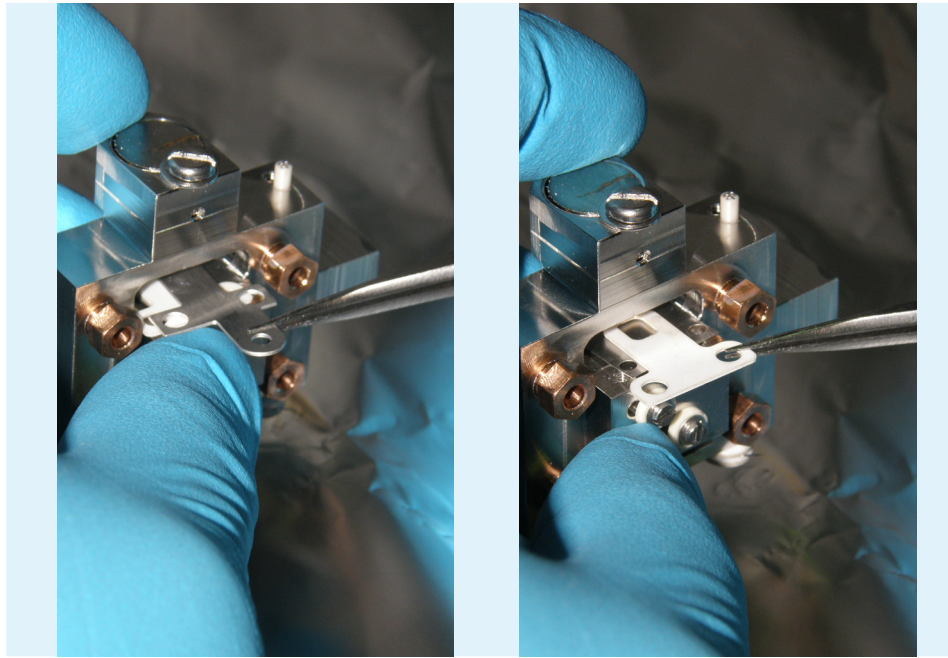


Figure 3.4-29: Removing the trap and the ceramic spacer beneath.

4. If not already removed, remove the filament as described in **Section 3.4.2**.
5. Turn the remaining source block over and remove the source wraparound.

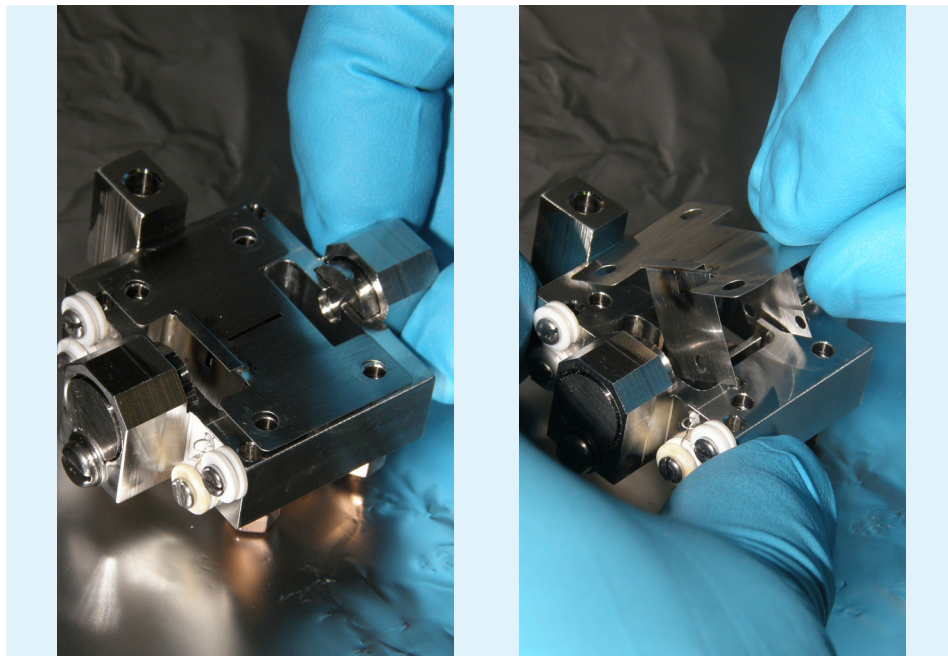


Figure 3.4-30: Turning over the block and removing the source wraparound.

6. Once the source wraparound has been removed, the repeller inside the source block can be seen.

7. Turn over the block. Remove the two screws to take out the repeller for cleaning.

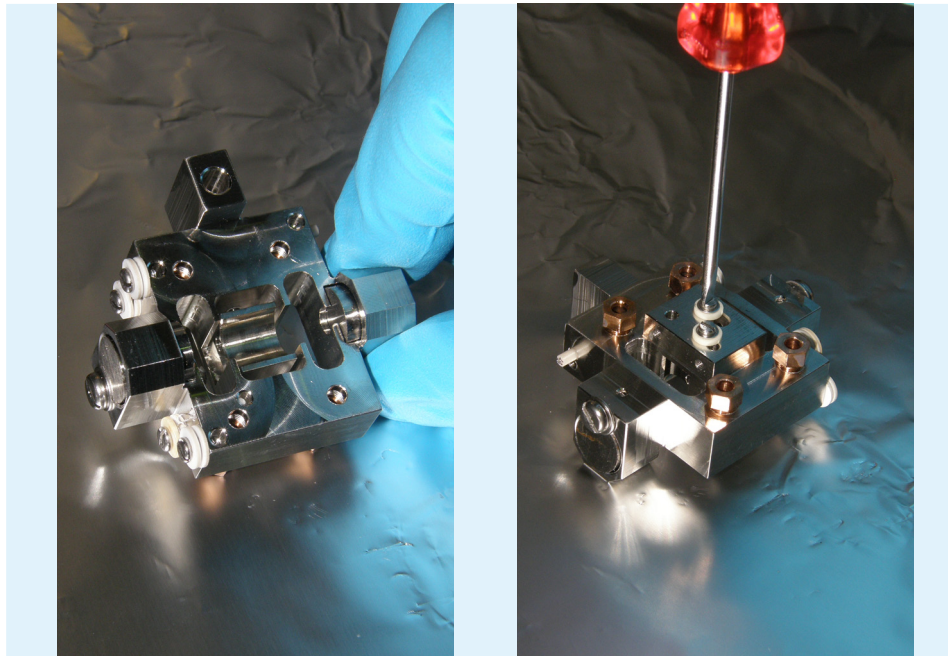


Figure 3.4-31: Viewing the repeller and turning the block over for further disassembly.

8. The screws have very long ceramic sleeves. Take them out using tweezers.

9. Lift off the remaining source block. Only the repeller is now left, with two washers on top.

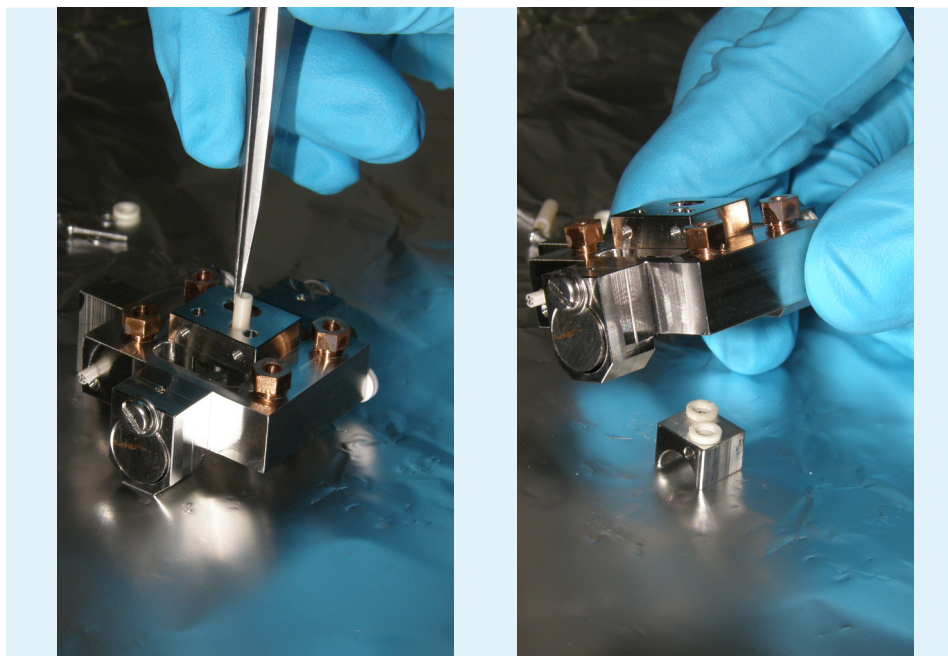


Figure 3.4-32: Removing the long ceramic sleeves and lifting off the block.

10. To fully disassemble the source, remove the magnets, source heater and temperature sensor.

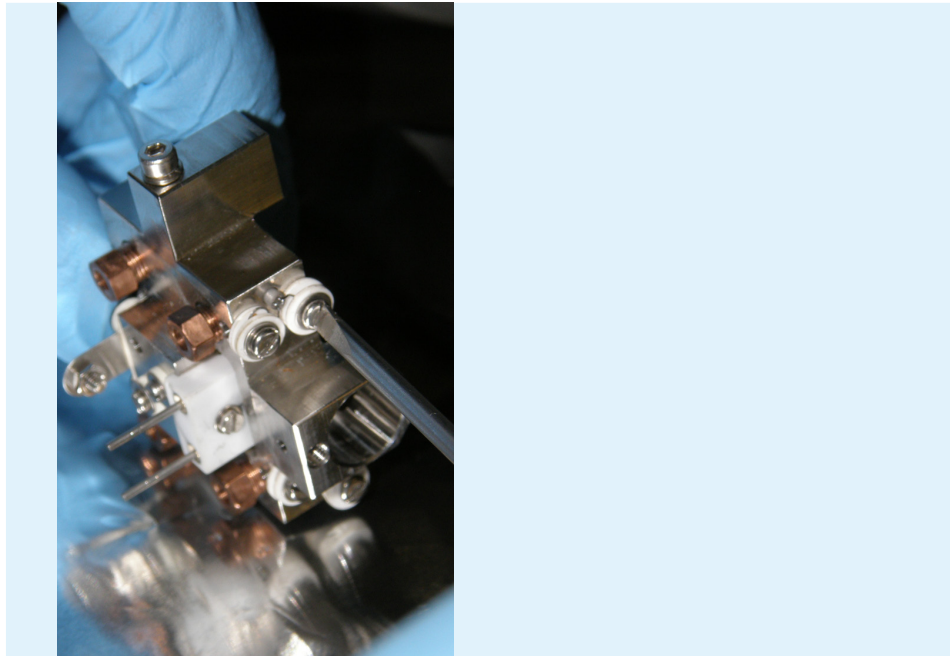


Figure 3.4-33: Removing the screws to take out the source heater and magnets.

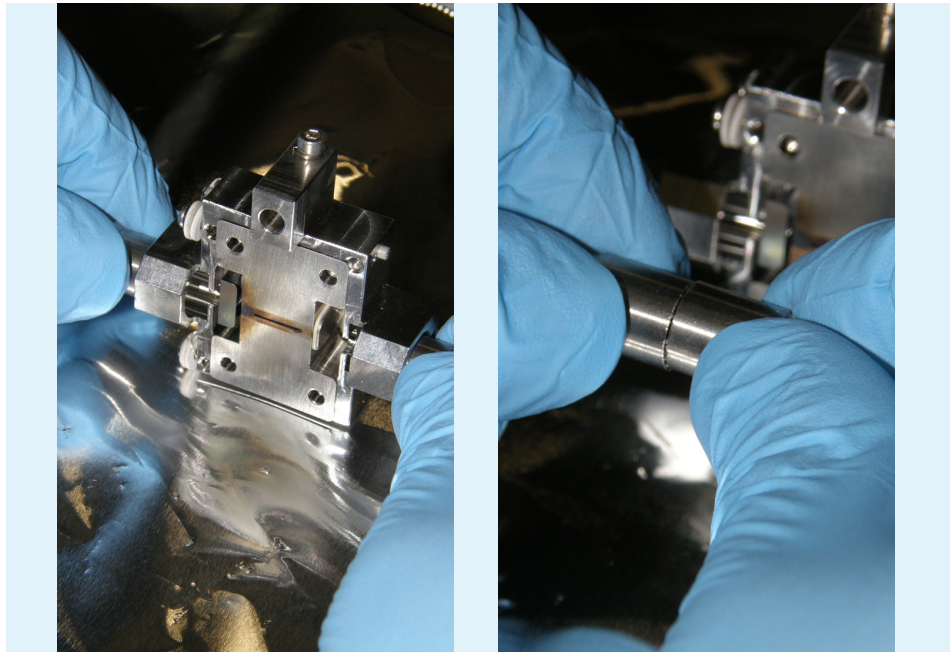


Figure 3.4-34: Removing the magnets.

3.4.8 Assembling the source block

This section will describe the assembly of the source block, as special care must be taken with certain steps.

To assemble the lens stack, reverse the steps performed in **Section 3.4.6**. To reattach the source block and lens stack, reverse the steps performed in **Section 3.4.4**. Refer to **Section 3.4.1** for the schematic view of the source assembly and the wiring table for the source.

Tools required: Small flat bladed screwdriver; tweezers; 5.5 mm spanner.

HINT: Since the source assembly is an internal component, it must remain as clean as possible. Perform any work in a clean area and wear gloves at all times.

HINT: Ensure that all of the tools have been cleaned with acetone (propanone) beforehand. Do not use isopropyl alcohol (IPA or propan-2-ol) on any internal component as it is a contaminant for CO₂ analysis.

HINT: To ensure that components are not lost during disassembly, place components in clean glass beakers or storage boxes. Keep ceramic and metal components in separate containers, to ensure that they are not damaged.

The image below shows the repeller disassembled. The parts, from left to right are:

M1.6 x 10 mm screw, M1.6 washer, 1 mm ceramic spacer, 7.5 mm ceramic sleeve, source block, 1 mm ceramic spacer, repeller.

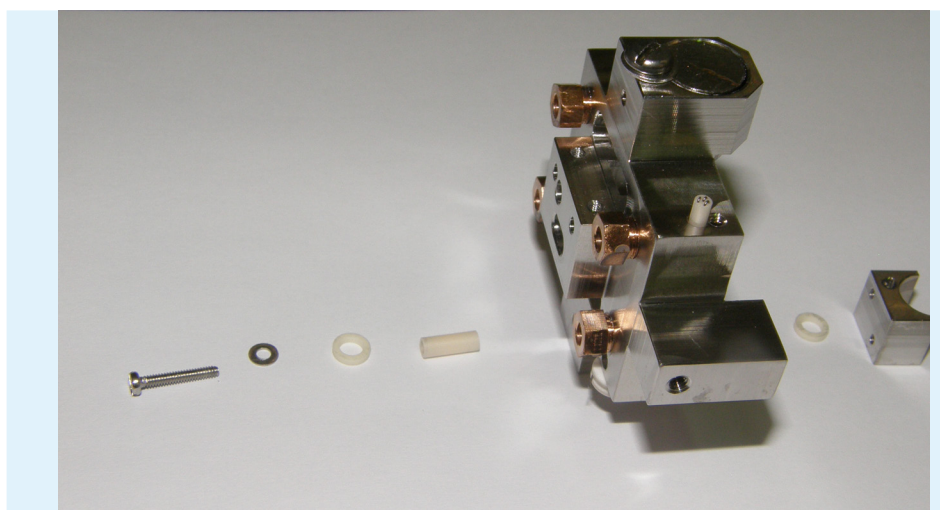


Figure 3.4-35: Parts for the source rebuild

1. The first step is to build up the long screw, as shown in the image above. This screw holds the repeller in place. Build it up in the sequence shown below:

- a) place the M1.6 x 10 mm screw on a flat surface.
- b) place the M1.6 washer over it.
- c) place the 7.5 mm ceramic sleeve over it.
- d) finally place the 1 mm ceramic spacer over it



Figure 3.4-36: Building up the repeller screw.

2. The images above show the build up of the repeller screw and the images below show its position on the repeller. For illustration purposes, the source block is not in place.

3. Place the source block in position, on a flat surface, ready to insert the repeller.

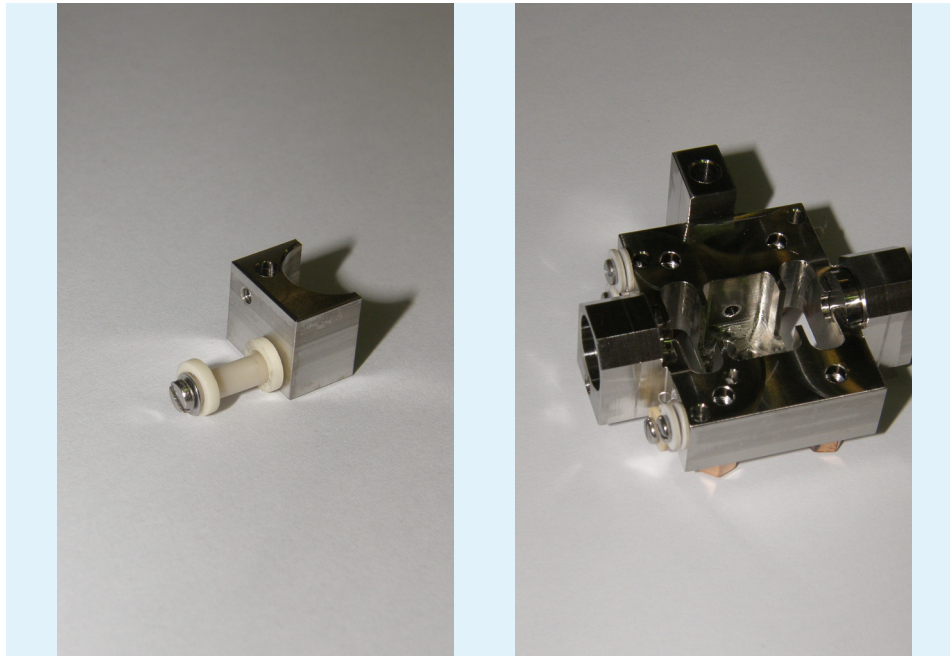


Figure 3.4-37: The repeller screw built up, shown without the source block in place (left) and with the source block in position.

4. Place 2 x 1 mm ceramics in the source block.
5. Place the repeller into position.

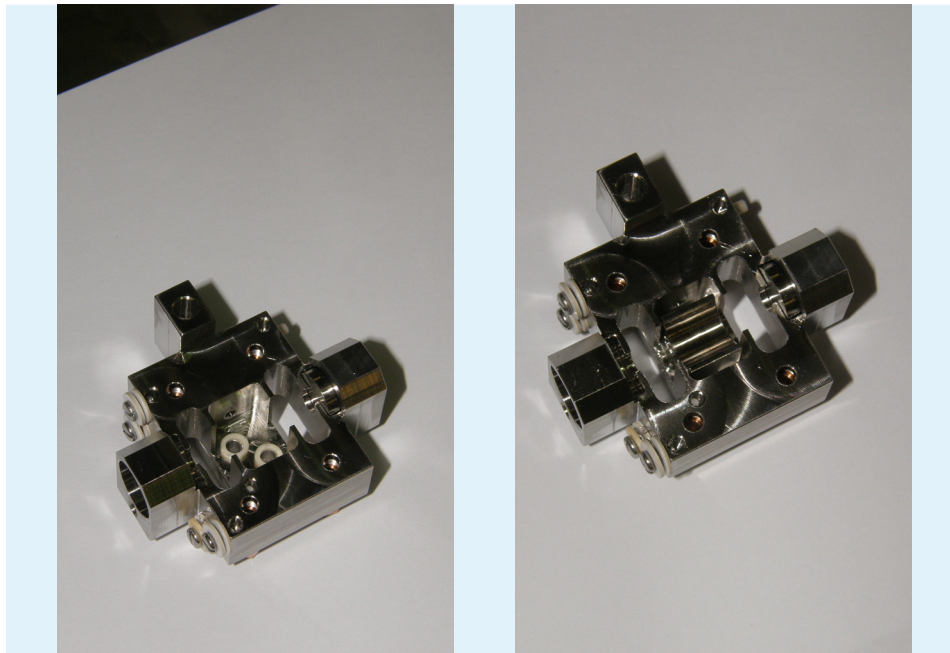


Figure 3.4-38: Placing the ceramics and the repeller into the source block.

6. Using finger pressure to hold the repeller in position, turn the whole source block over. Keep the repeller in the same position for the next few steps.
7. Place the two ceramic sleeves into the source block so that they slot into the 1 mm ceramic spacers between the repeller and source block.

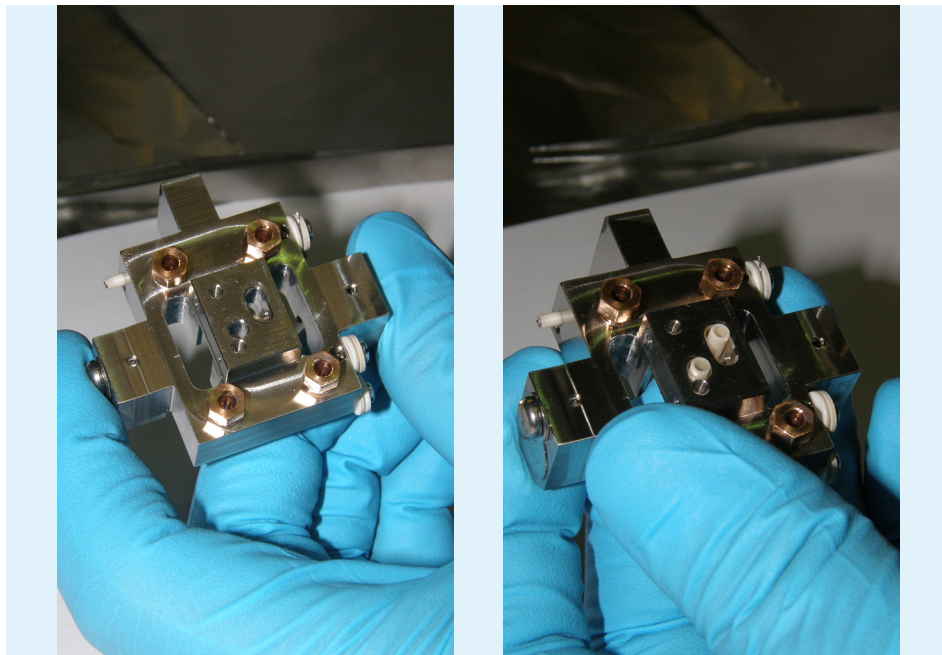


Figure 3.4-39: Turning the source block over and inserting the ceramic sleeves.

8. Place a 1 mm ceramic spacer over each ceramic sleeve, if this has not already been done.
9. Place 1 x M1.6 washer and 1 x M1.6 x 10 mm screw into one of the holes and loosely tighten (finger tight).

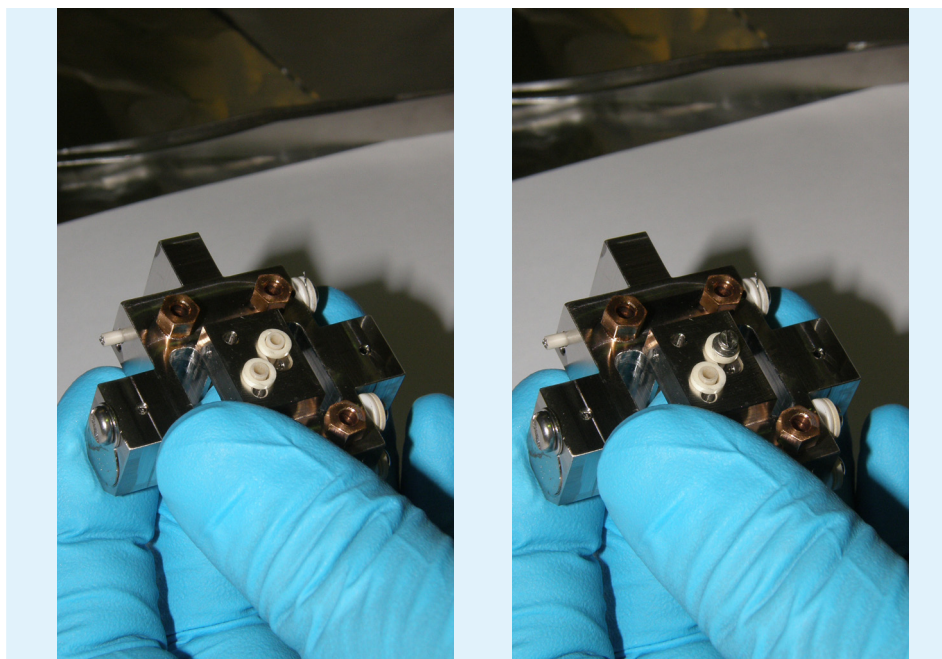


Figure 3.4-40: Placing the ceramic spacer, the washer and screw into the holes.

10. Place the other washer and screw in position (again only finger tight). The screws should be tight enough so that the source block can be flipped over without the repeller moving out of place but not so tight that the repeller cannot be moved into the correct position.

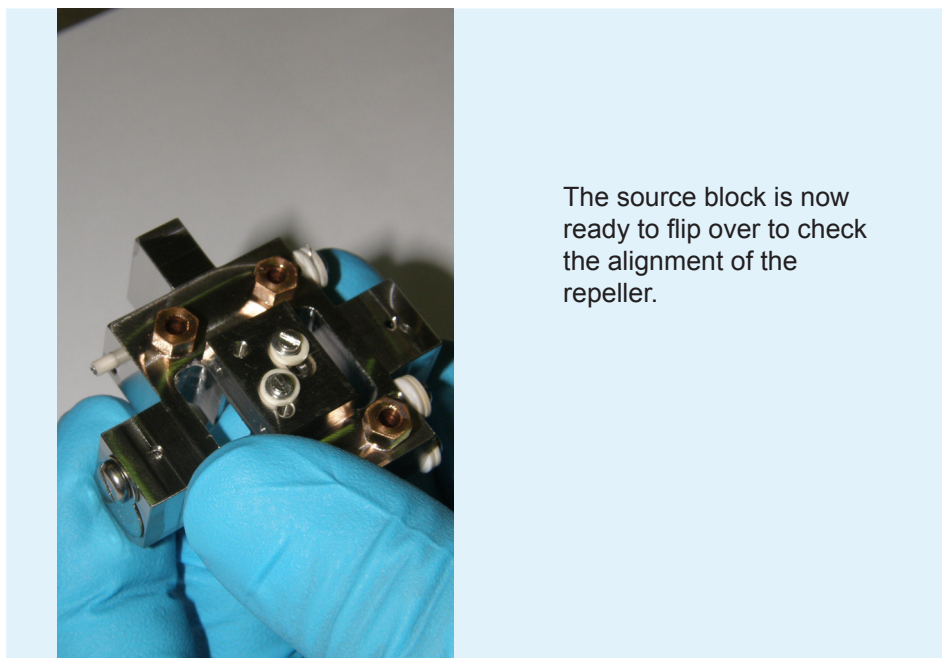


Figure 3.4-41: Source block ready to be turned over.

11. Flip over the source block. The next step is to align the repeller. The ideal position is in line between the two magnet holders. The image below shows the incorrect (left) and the correct (right) alignment.

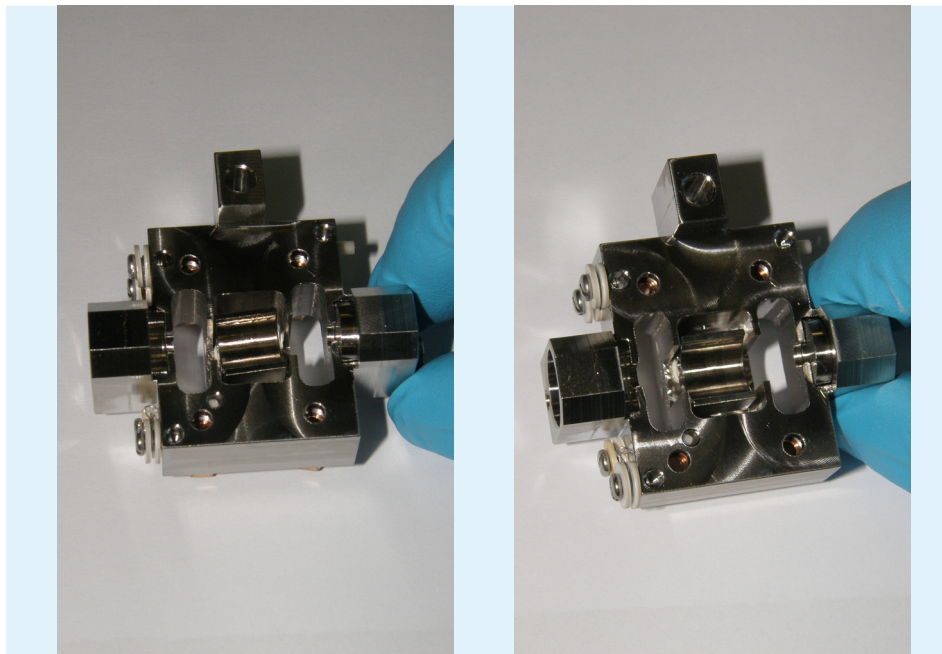


Figure 3.4-42: Comparison between incorrect (left) and correct (right) repeller alignment.

12. Finally, tighten the repeller screws just beyond finger tight so that the repeller does not move. Do not over-tighten the screws as this may crack a ceramic.

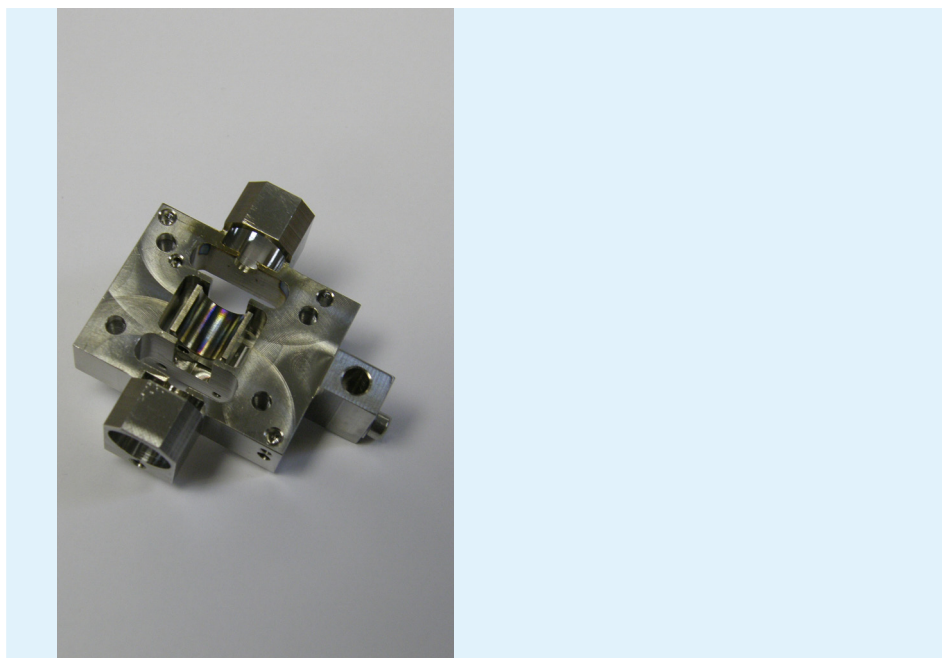


Figure 3.4-43: Repeller tightened.

13. The next step is to build the trap. The image below shows the components of the trap, from left to right:

M1.6 x4 mm (or 6 mm) screw, M1.6 washer, shim ring (looks like a washer but is a precision thickness with two flat faces), 1 mm ceramic spacer, 2.1 mm ceramic sleeve, trap, trap insulator, source wraparound.



Figure 3.4-44: Trap components.

14. Firstly, the building of one screw will be described. The other screw will be built in exactly the same way later.

- a) place the M1.6 x 4 mm (or 6 mm) screw on a flat surface.
- b) place the M1.6 washer onto the screw.
- c) put the shim ring onto the screw.
- d) place a 1 mm ceramic onto the screw.
- e) place the short ceramic sleeve onto the screw, so that it falls down between the ceramic spacer and the screw.



Figure 3.4-45: Building up a trap screw.

15. Next, place the wraparound onto the source block. This will hide the repeller. The wraparound has a small piece of metal welded onto it (not shown in these images).

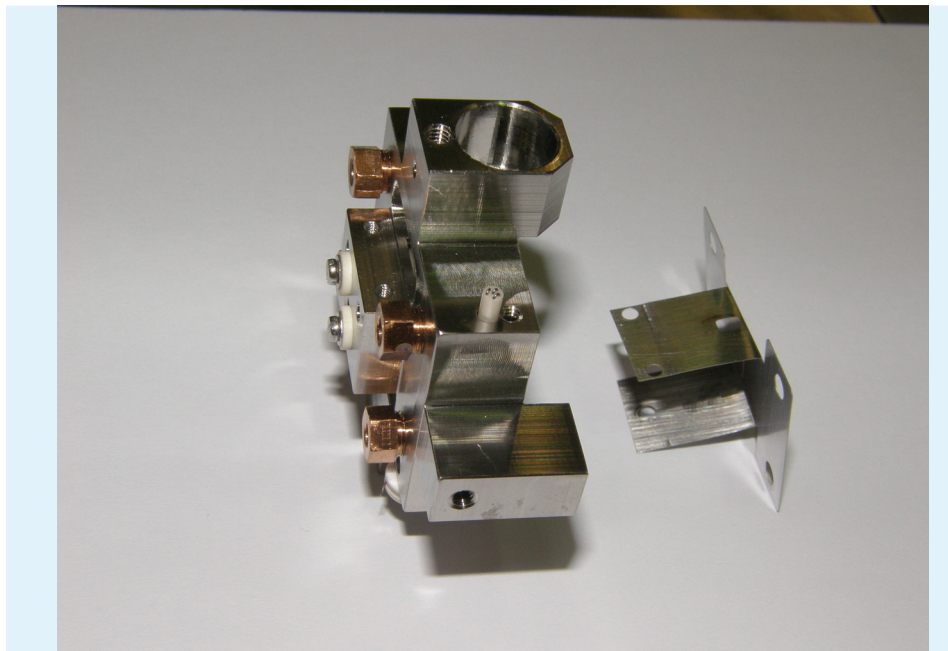


Figure 3.4-46: Placing the wraparound onto the source block.

16. One side of the wraparound has two holes and the other has one hole, so make sure it is the correct way round, as shown in the figure below.

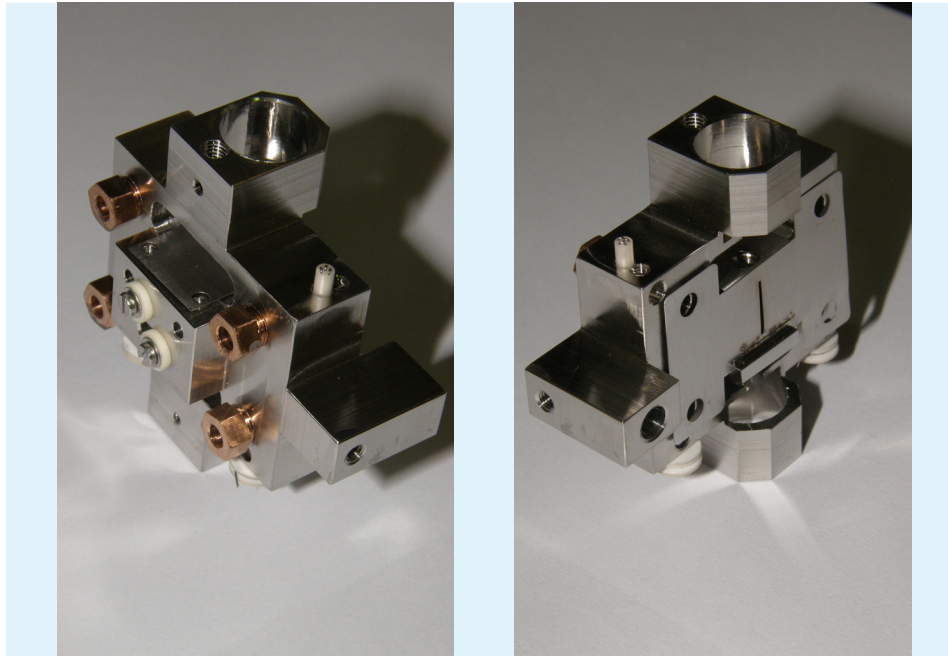


Figure 3.4-47: Placing the trap on the source block.

17. Place the trap insulator onto the wraparound.

18. Place the trap on top of the insulator.

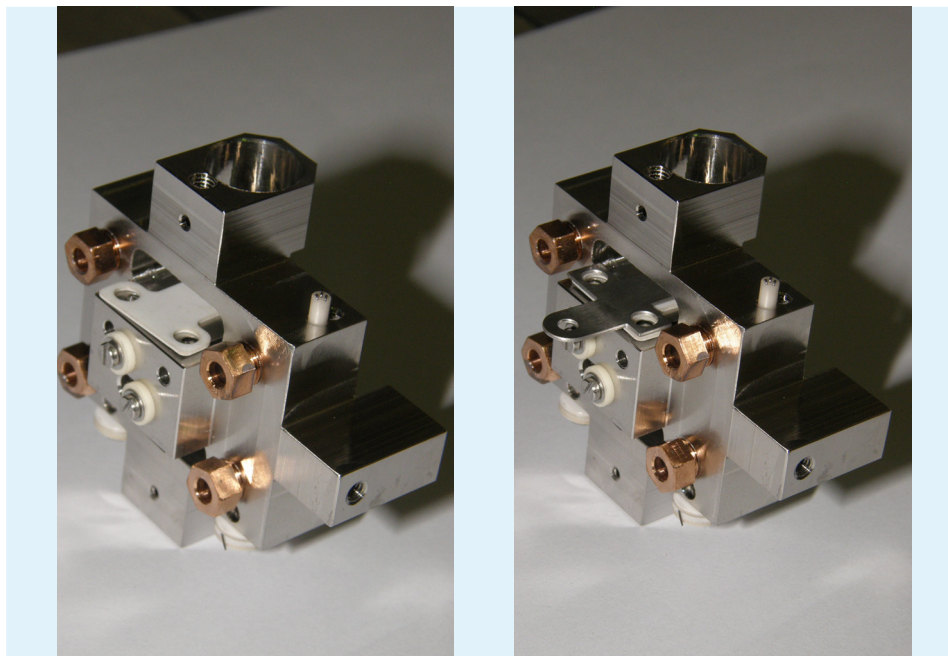


Figure 3.4-48: Placing the trap on top of the insulator.

19. Place the screw that is not built up into one of the holes. This is to keep the parts aligned during the next build process.

20. Place the built up screw into position, just loosely to begin with.

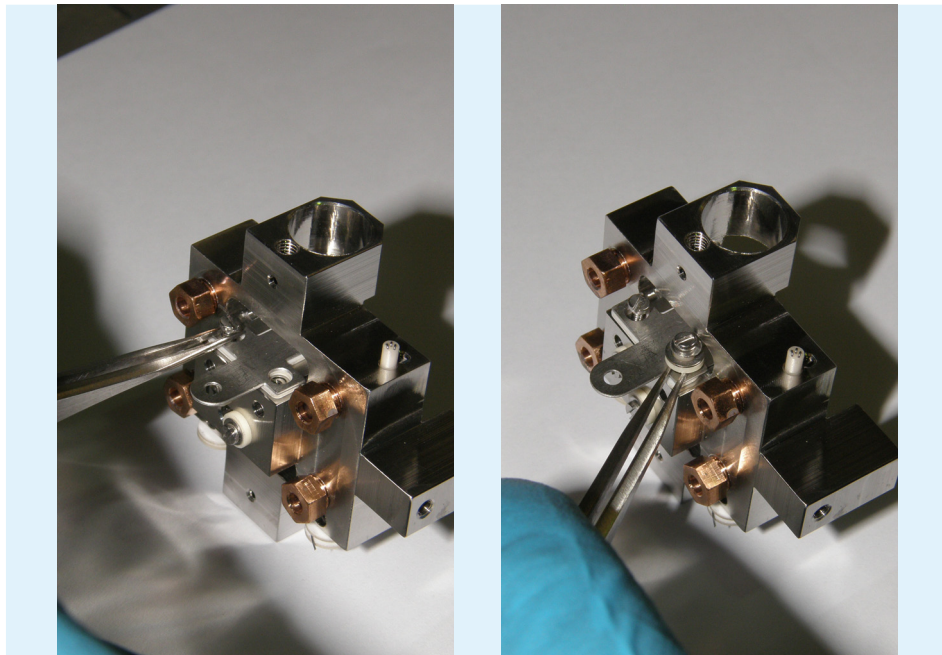


Figure 3.4-49: Placing the screws.

21. Remove the single screw and build the screw in the same way as the other one (M1.6 washer, shim ring, 1 mm ceramic spacer, short ceramic sleeve).

22. Screw in the second screw, again loosely to begin with. Make sure that the inner ceramic sleeve falls down completely into the hole in the trap insulator. Then the 1 mm ceramic spacer and shim ring should align around the sleeve.

23. Tighten the screws down to just beyond finger tight. If everything is correct, the trap should not move. If it does move, then the ceramic sleeve is not fully in the holes of the trap and trap insulator.

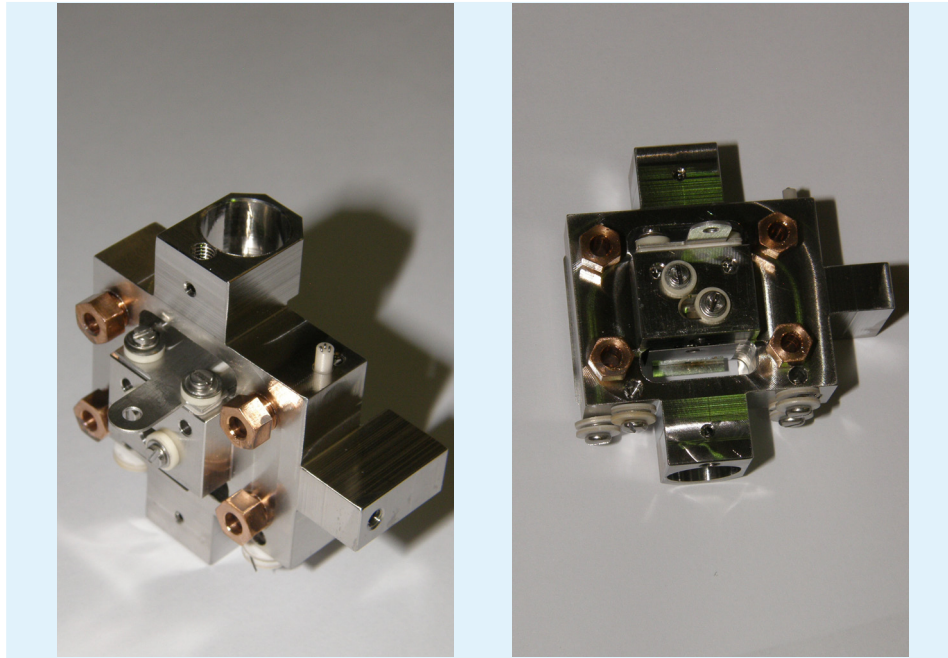


Figure 3.4-50: Repeller, trap and wraparound in position.

24. The next step is to fit the filament, as previously described in **Section 3.4**.

25. If not already fitted, reattach the source heater and temperature sensor to the block. Have the two screws with insulating sleeves and the ceramic washers ready, then insert the temperature sensor.

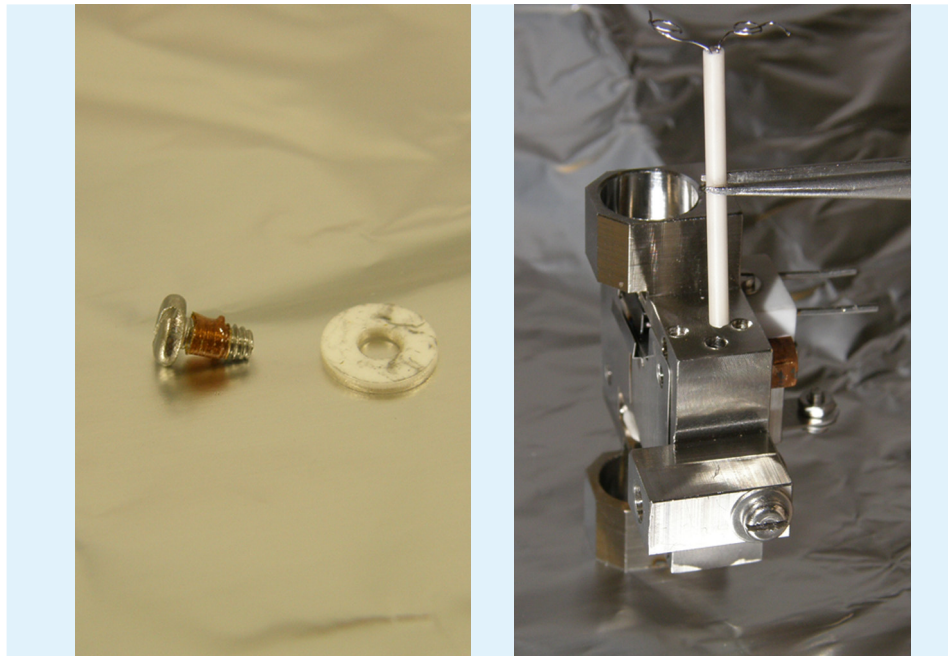


Figure 3.4-51: Have the screw and insulator ready. Insert the temperature sensor.

26. Now place both insulators on top of the source block and then insert the first screw holding it with tweezers and tighten the screw in lightly. Tightening too hard will break the ceramic washer.

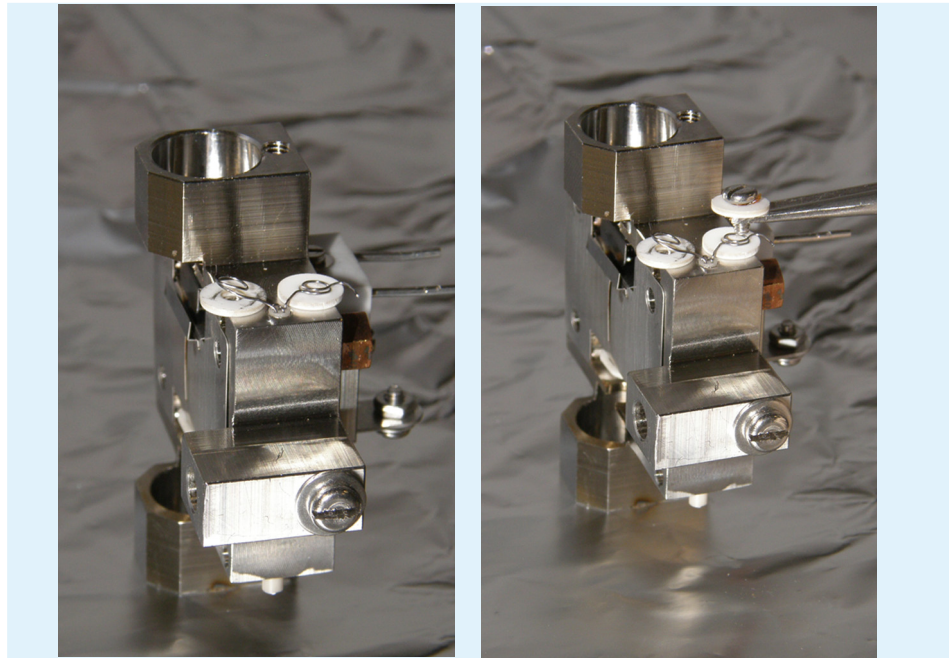


Figure 3.4-52: Placing both insulators (left), before inserting the first screw (right).

27. Now place the second screw in and tighten it lightly.

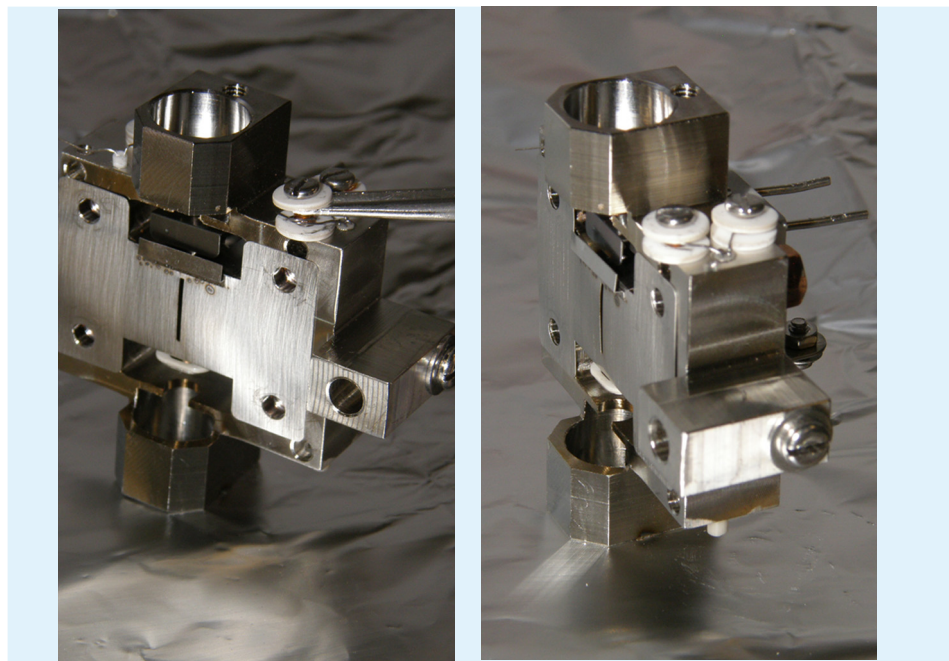


Figure 3.4-53: Inserting the second screw (left) and both screws tightened (right).

28. Finally, fit the magnets to the source block. The orientation of the magnets is very important. The magnetic field orientation should be the same for both magnets, i.e. the visible parts of the magnet on either side of the block should be of opposite polarities. The easiest way to ensure this is to orient the magnets together so that they attract each other and then place them into position with the magnets in that same orientation.



Figure 3.4-54: Placing the magnets back in with the correct orientation.

29. The source rebuild is now complete.

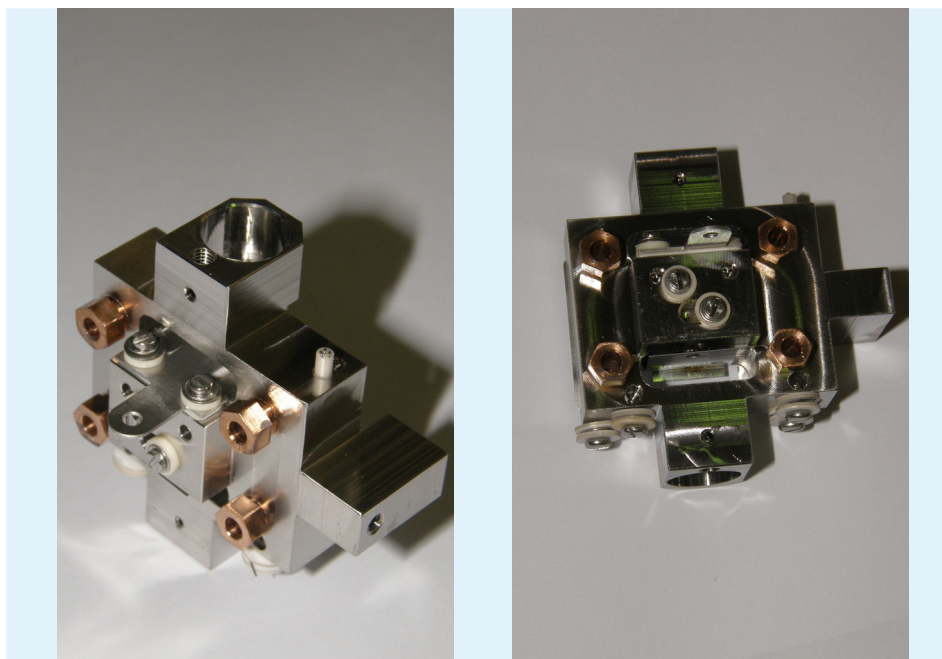


Figure 3.4-55: Repeller, trap and wraparound in position.

To fully build up the source assembly, firstly build up the source lens stack, following the reverse of the procedure described in **Section 3.4.6**.

Next, connect the source block and source lens stack, following the reverse of the procedure described in **Section 3.4.4**.

Next, reconnect the connectors to the various locations, following the reverse of the procedure from **Section 3.4.3**.

Finally, reconnect the assembled source flange to the source housing, following the reverse of the procedure described in **Section 3.4.1**.

HINT: Remember to reattach the source inlet gasket before reinserting the assembled source into the housing.

3.5 Cleaning procedures

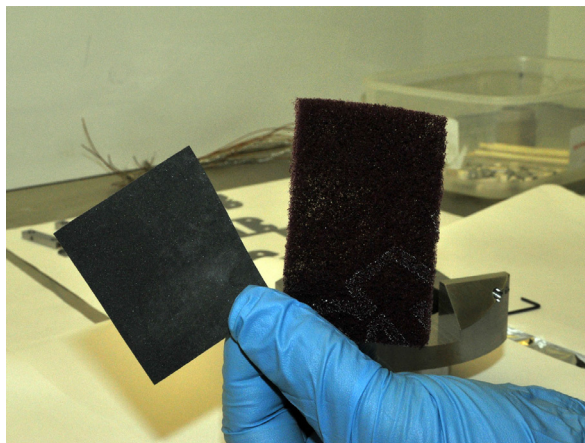
The following sections describe maintenance of stainless steel and ceramic components.

3.5.1 Cleaning of stainless steel components

HINT: Several of the components require a specialist cleaning procedure. It is recommended to contact Nu Instruments if it is intended to clean anything other than source assembly components.

HINT: If paper towel or tissue is to be used during the cleaning or drying process, it is strongly recommended that this is of a low loss or lint-free type.

1. It is recommended to use a fine grade of Scotch-Brite, or similar abrasive cloth, and fine abrasive paper for the cleaning process. Clean off any discoloration, firstly using the Scotch-Brite, followed by the abrasive paper. The abrasive will need to be rolled up tightly to clean into some of the holes.
2. Place the stainless steel components in a 10% solution of a surface active cleaning agent, such as Decon 90, in a glass beaker, in an ultrasonic bath for 20 minutes.
3. Rinse all of the components with deionized water several times to remove most of the cleaning agent residue.
4. Place the components in a beaker of deionized water in an ultrasonic bath, again for 20 minutes, to remove any possible trace of the cleaning agent.
5. The preferred drying method for the parts is to oven dry at 80 °C for two hours. Alternatively, air dry on lint-free paper tissues for an extended period, ensuring that no liquid remains in any holes.



It is recommended to use Scotch-Brite and fine abrasive paper first, to remove the discoloration.

Figure 3.5-1: Cleaning materials.



Figure 3.5-2: Placing the stainless steel components in a beaker for cleaning.

3.5.2 General cleaning of ceramic components

1. Any ceramic components may only require cleaning with abrasive paper to remove discoloration. Use the abrasive very gently. If immersed in a cleaning solution, ceramic components may require extended drying times, due to their porosity.
2. Propanone (acetone) can be used to wipe and clean the surface of the ceramic components.

If ultrasonic cleaning is required, follow the method for non-ferrous materials and ceramics described in **Section 3.5.3**.

HINT: If paper towel or tissue is to be used during the cleaning or drying process, it is strongly recommended that this is of a low loss lint-free type.



WARNING

Flammable solvents : Observe normal safety precautions when using volatile solvents, such as propanone and propan-2-ol.

HINT: Avoid cleaning ceramic insulators with corrosive chemicals, such as acids or acidified cleaning solvents. Acidified solvents may attack and destroy the structure of the ceramic.

3.5.3 Cleaning of non-ferrous materials and ceramics

HINT: Do not use an alkaline cleaning agent like Decon-90 on non-ferrous materials (aluminium or copper alloys) or ceramics. These agents may corrode or attack the structure of the material.

1. Make up a 3% solution of a pH neutral cleaning agent, such as Neutricon, in a glass beaker and immerse the components in the solution. Place the beaker in an ultrasonic bath for 20 minutes (5 minutes for aluminium components).

HINT: For aluminium components, reduce the time in the ultrasonic bath to 5 minutes to reduce the risk of sonic burn.

2. Rinse all of the components with deionized water several times to remove most of the cleaning agent residue.

3. Next place in a beaker full of deionized water and place in an ultrasonic bath, again for 20 minutes (5 minutes for aluminium components). This is to remove any possible trace of the cleaning agent.

4. The preferred drying method for the parts is to place them in an oven at 80 °C for two hours. Alternatively, allow them to air dry on lint-free paper tissues for an extended period, ensuring that no liquid remains in any holes.

3.6 Changing the helium cylinder

In this section, the procedure for changing the helium cylinder and reference gas cylinders will be discussed.



WARNING

Compressed gases : Only trained personnel should work with compressed gases. Follow local regulations when working with them.



WARNING

Crush Hazard : Compressed gas cylinders are heavy. Follow local cylinder handling regulations.

**WARNING**

Hazardous gases : Ensure that the exhaust line is vented safely out of the laboratory, in accordance with local health and safety regulations.

3.6.1 Helium change

HINT: Any cylinder should be changed before it is completely empty. This reduces the risk of any contaminants from the internal walls of the cylinder leaching out and contaminating the gas lines or instrument.

As the helium may be connected to various prep systems, refer to their individual manuals regarding interruption in helium flow.

The following steps will ensure that the internal components are protected and will minimize system downtime.

1. Turn off the HT and Source by unchecking the relevant box in the **Digital** window

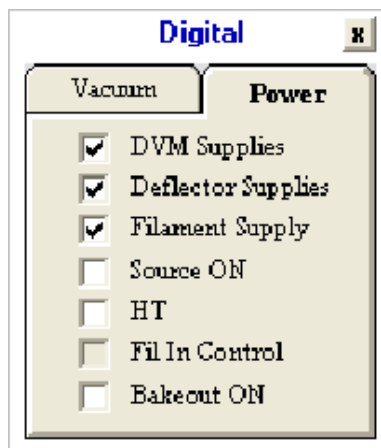


Figure 3.6-1: Turning off HT and Source in the Digital window.

2. Set all connected gas pressures to a low value of around 5 psi.
3. Close all of the continuous flow valves, including the reference valves and the main isolation valve.

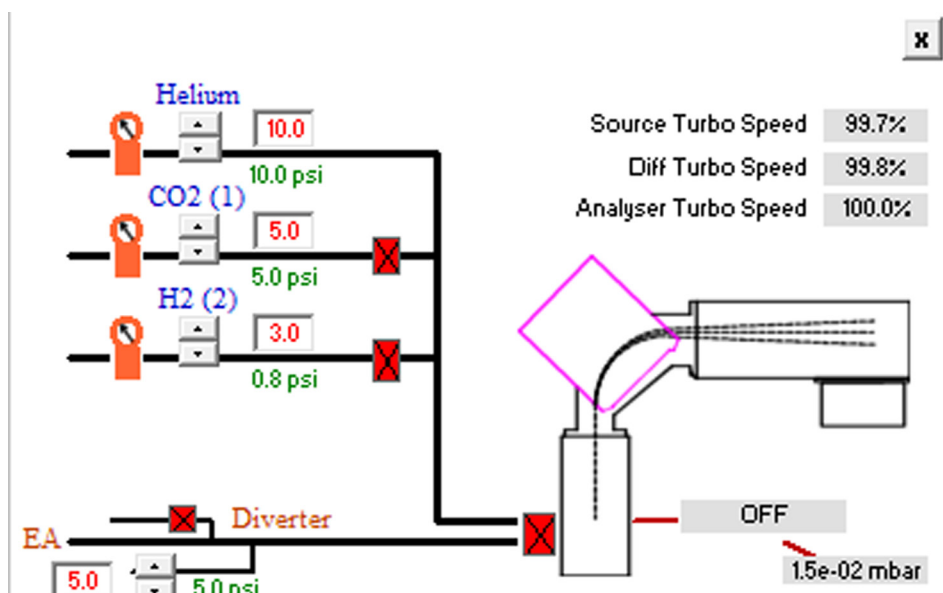


Figure 3.6-2: Closing continuous flow valves and main isolation valve.

4. The Perspective is now safely configured to change the helium cylinder.

HINT: Refer to the prep system manuals for procedures to make them safe for helium exchange before changing the helium cylinder.

5. Once the cylinder has been changed, thoroughly leak check the connection at the cylinder, using a suitable leak sniffer or liquid leak detector.

6. Atmosphere enters the pipework during the cylinder exchange, so the lines need to be purged after the leak check at the cylinder before returning the Perspective to operation. To do this, loosen the line connection where the line enters the rear of the instrument and purge for 15 to 30 seconds. If several instruments or systems are connected to the same line, start with the connection that is closest to the cylinder and work round to those furthest away. Typically, for a system connected directly to a cylinder with a 5 m line, 15 seconds of purging should be sufficient.

7. Once the lines are purged and the connections remade, thoroughly leak check the line using a suitable leak sniffer or liquid leak detector.

8. Return the helium pressures to their normal levels and open the main isolation valve.

9. Reload the relevant tuning file and turn on the source and HT.

3.6.2 Reference gas exchange

HINT: Any cylinder should be changed before it is completely empty. This reduces the risk of any contaminants on the internal walls of the cylinder leaching out and contaminating the gas lines or instrument.

1. Turn off the HT and Source by unchecking the relevant box in the **Digital** window.
2. Set all connected gas pressures to a low value of around 5 psi.
3. Close all of the continuous flow valves, including the reference valves and the main isolation valve.

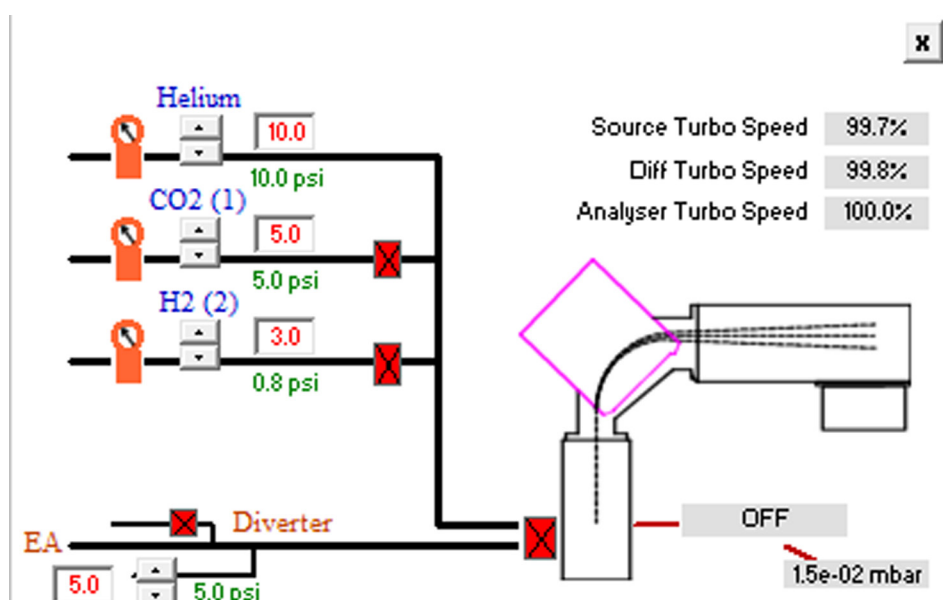


Figure 3.6-3: Closing continuous flow valves and main isolation valve.

4. Once the reference gas cylinder is closed, increase the set point in the software to 50 psi and allow it to fall slowly to below 10 psi before disconnecting the cylinder.
5. The Perspective is now safely configured to change the reference gas bottle.

HINT: Changing a reference gas bottle should not affect any attached prep system.

6. Once the bottle has been changed, thoroughly leak check the connection at the cylinder using a suitable leak sniffer or liquid leak detector.

**⚠ CAUTION**

Purging the reference gas line is very dependent on the reference gas being used. For flammable or toxic gases (H_2 , CO and SO_2), it is not recommended to purge into the laboratory environment. If the main vent line is connected to the instrument, these gases can be purged through the Perspective gas system itself using the following procedure;



- a) Leak check the entire line using a suitable leak sniffer or liquid leak detector.
- b) Set the reference EPC to a high value (>30 psi) and leave it for several hours (ideally overnight). This should safely purge the entire line.

7. Atmosphere enters the pipework during the cylinder exchange, so the lines need to be purged after the leak check at the cylinder, before returning the Perspective to operation. To do this, loosen the line connection where the line enters the instrument and purge for 15 to 30 seconds. Typically, for a system connected directly to a cylinder with a 5 m line, 15 seconds of purging should be sufficient.



Figure 3.6-4: Purging the reference gas line.

8. Return the reference pressure to its normal level and open the main isolation valve.
9. Reload the relevant tuning file and turn on the source and HT.

3.7 Prep system change

Certain prep systems are fixed to the Perspective (Dual Inlet, Nu Carb and GC). Other prep systems connect to the instrument sample line. Care must be taken when changing the prep system to minimise the risk of damaging the instrument.

1. Turn off the attached prep system, in accordance with the instructions given in its individual manual.
2. Turn off the HT and Source by unchecking the relevant box in the **Digital** window.

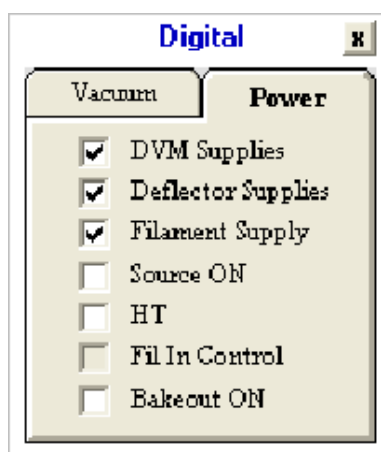


Figure 3.7-1: Turning off HT and Source in the Digital window.

3. Close all of the continuous flow valves, including reference valves and the main isolation valve.

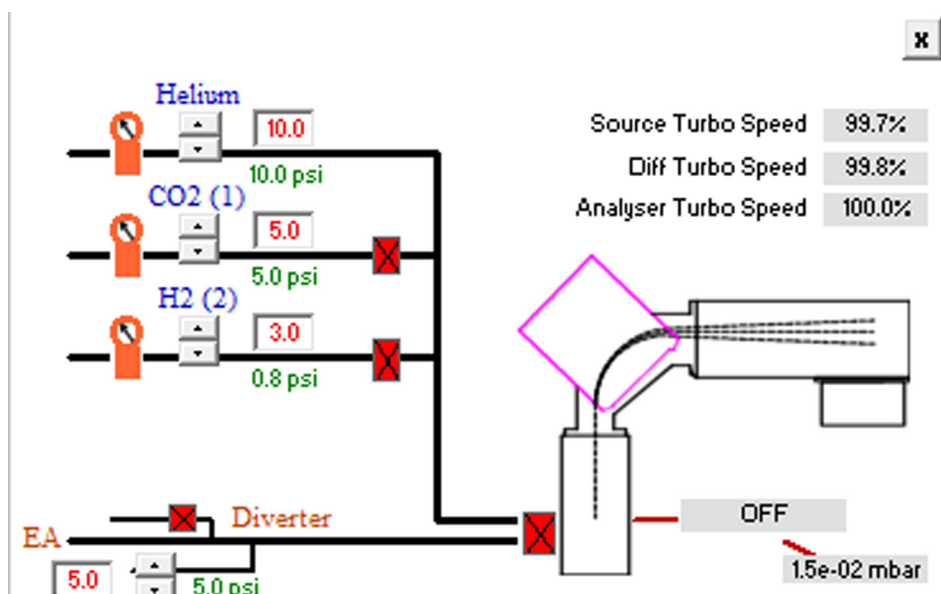


Figure 3.7-2: Closing continuous flow valves and main isolation valve.

4. Using two 1/4" spanners, break the 1/16" sample line connection on the rear of the Perspective.

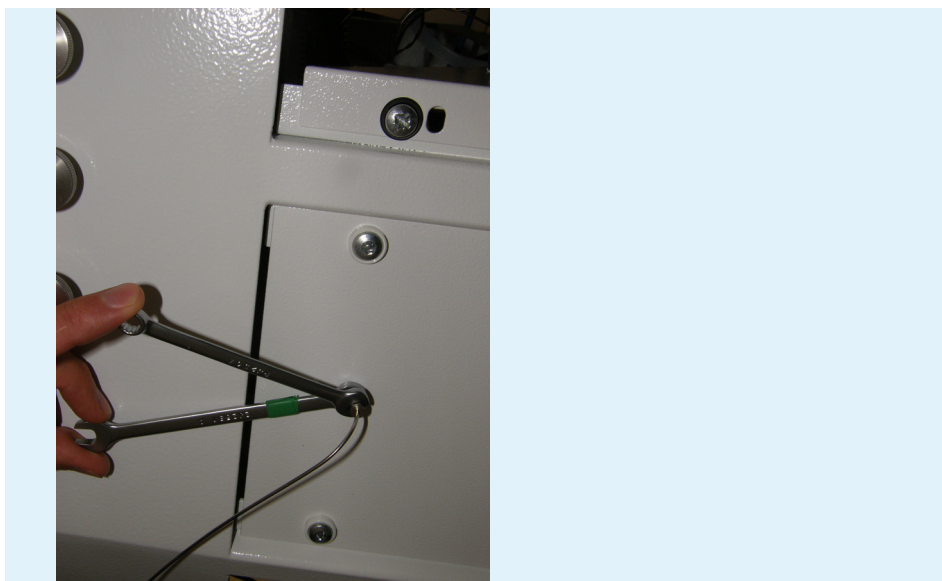


Figure 3.7-3: Line connections to Prep system.

5. Attach the 1/16" connection from the new prep system and tighten using two 1/4" spanners.
6. Turn on the prep system, in accordance with the instructions given in its individual manual and ensure that helium is flowing from the prep system.
7. Open the main isolation valve.
8. Load a relevant tuning file and turn on the source and HT.

Chapter 4: Troubleshooting

This chapter gives some guidance on the identification of possible instrument problems that may lead to unexpected results, poor performance or error indications. It is recommended to always start with the simpler options for the resolution of a particular problem. This chapter is written with this in mind, so that the user is guided to address possible solutions to problems in a logical sequence.

There are three general rules for fault finding:

- 1 Think clearly and logically. Do not jump to conclusions without evidence.
- 2 Use all of the information available.
- 3 Try to narrow the search by identifying the part of the instrument in which the problem lies.

The first step is to determine what has happened. An example is shown below.

Example 1: If the beam has vanished suddenly

This may be caused by;

- a) A spontaneous failure of an instrument component or subsystem (e.g. a power supply).
- b) Something done by the user, either unintentionally, or with unexpected consequences.

If the beam suddenly vanishes, try to retrace steps, starting with the most recent activity. It may be as simple as reloading the correct tuning file. Always try the simple solutions first, before making any changes to the system.

4.1 Problems with the observed beams

This section describes fault finding in the area of observed beam issues, including peak shape and sensitivity problems.

HINT: Peak shapes are a rich source of information.

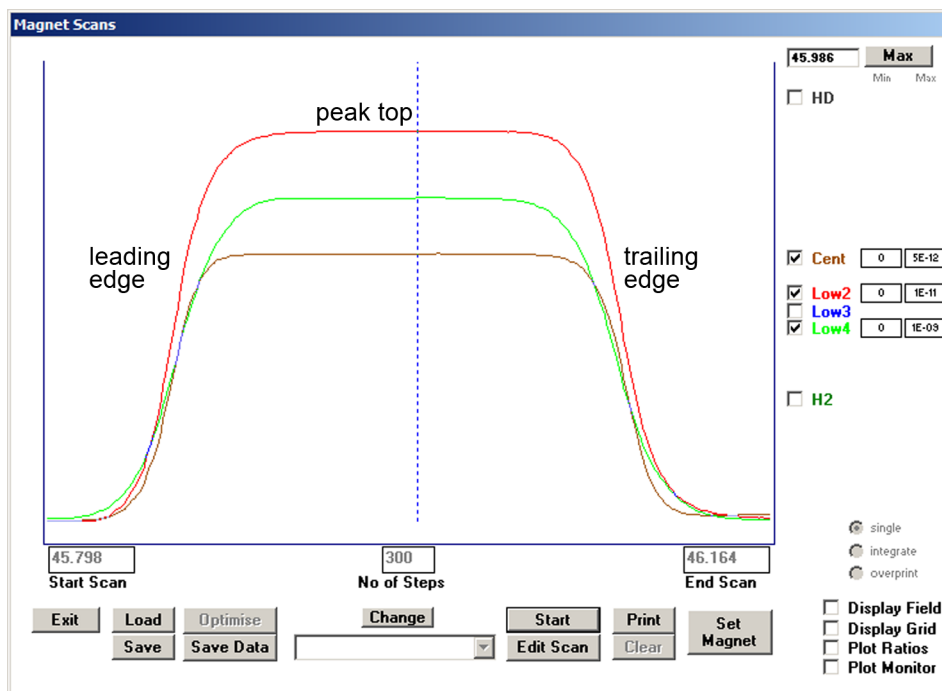


Figure 4.1-1: Exemplary peak shapes for CO₂ (masses 44, 45 and 46).

The above scan shows a typical CO₂ trace for masses 44, 45 and 46. It is always worthwhile taking screenshots after changing any component of the mass spectrometer or related parameters (e.g. after filament change, retuning etc). This screenshot can later be used as a reference to speed up fault finding.

If there are issues with the ion beam, then it is always worth checking the following.

1. Check if there are any error message flags (seen in the bar on the top of the software control window and in the message window). See **Section 4.4** for descriptions of error messages.
2. Check if the instrument parameters are as expected for that gas species. For example, is the correct tuning loaded and are the supplies giving out the correct voltages (see **Section 4.2.3** to see how to monitor the supply voltages).
3. Check that gas is going into the source and that it is the species to be measured. Check the main mass spectrometer mimic to ensure that the correct valves are open and that EPCs are supplying the correct pressures.

Below is a list of several common beam issues, with some hints about aspects to check.

Error	Possible fix
No beams in any of the collectors in the expected mass range.	<p>Visually inspect the position of the Hall probe and magnet.</p> <p>Check that HT voltages are as expected in the Monitor Supply dialog box. If not, reload the tuning file.</p> <p>Check the magnet monitor parameters.</p> <p>Scan over a wider mass range (e.g. from 10 to 50) to see if the peaks have moved. If so the mass calibration file may be incorrect.</p>
Beams no longer in coincidence.	<p>Check that the tuning parameters are as expected for that gas species.</p> <p>Verify quad parameters by checking the voltages in the monitor window (engineer monitor HV1 test, ...)</p> <p>Check if the magnet has been moved.</p>
Beam missing from one or more collectors but present in others.	<p>Check if the readback from the affected beam is frozen or is varying.</p> <p>If frozen, reset the DVM supplies by turning the checkbox off then back on in the Digital window.</p> <p>Scan over a wider range to see if the collector responds to any ion beam.</p> <p>Contact Nu Instruments for more information if problem persists.</p>
Sensitivity lower than expected.	<p>Are the tuning parameters as expected for that gas species.</p> <p>Is the reference gas EPC reading the correct pressure i.e. does the read back match the set point?</p> <p>Is the beam centered on the correct mass.</p> <p>Is the penning pressure as expected.</p>

Error	Possible Fix
Beam position moving during a scan.	<p>If the magnet has just been turned on, wait till the proportional error (found in magnet monitor) is stable, then recheck.</p> <p>Verify quad parameters by checking the voltages in the monitor window (engineer monitor HV1 test, ...)</p>
Beam sensitivity unstable.	<p>Check that the tuning parameters are as expected for that gas species.</p> <p>Check if the reference gas EPC or carrier EPC readings are unstable.</p> <p>Check the penning pressure stability. This helps identify if it is an electrical or internal issue. If the penning pressure is unstable then it is more likely an issue in the source (e.g. arcing) or gas supply into the source. If the pressure is stable then it is more likely to be an external electronic issue.</p> <p>Consider tuning H Defl and Delta Z values.</p>

Table 4.1-1: Common beam issues.

4.2 Checking parameter settings

The user will be familiar with the setting of instrument parameters through the various control windows. In addition, readbacks for many parameters are also available, for example when a requested voltage is measured at a power supply and returned to the computer. These can be useful if a failed or malfunctioning power supply is suspected.

Some of the main readback windows will now be described briefly.

HINT: It is recommended that the user gains some familiarity with these windows when the instrument is functioning correctly, before actually using them for fault finding.

4.2.1 Supplies window

In the **Supplies** window, the set parameters for the **Source**, **Quads** and **Collectors** are listed. Listed below are some of the source readbacks.



Figure 4.2-1: Source status in the Tuning window.

4.2.2 Main Monitor window

The **Main Monitor** window provides useful information. Up to six detector readbacks, three monitor values, and the magnet set point are available.

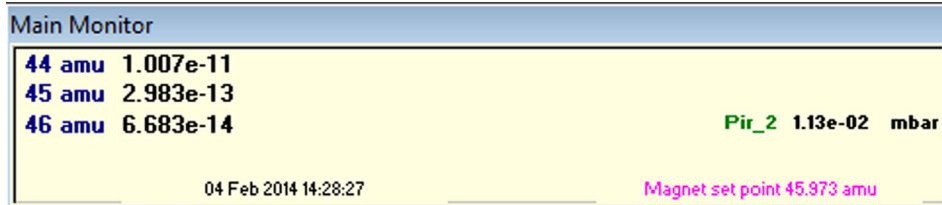


Figure 4.2-2: Monitor window.

The choice of which detector and monitor parameters to display is made through the **Setup** window, in which the **Monitor Window Detectors** and **Monitor Machine Status** status pane are available.

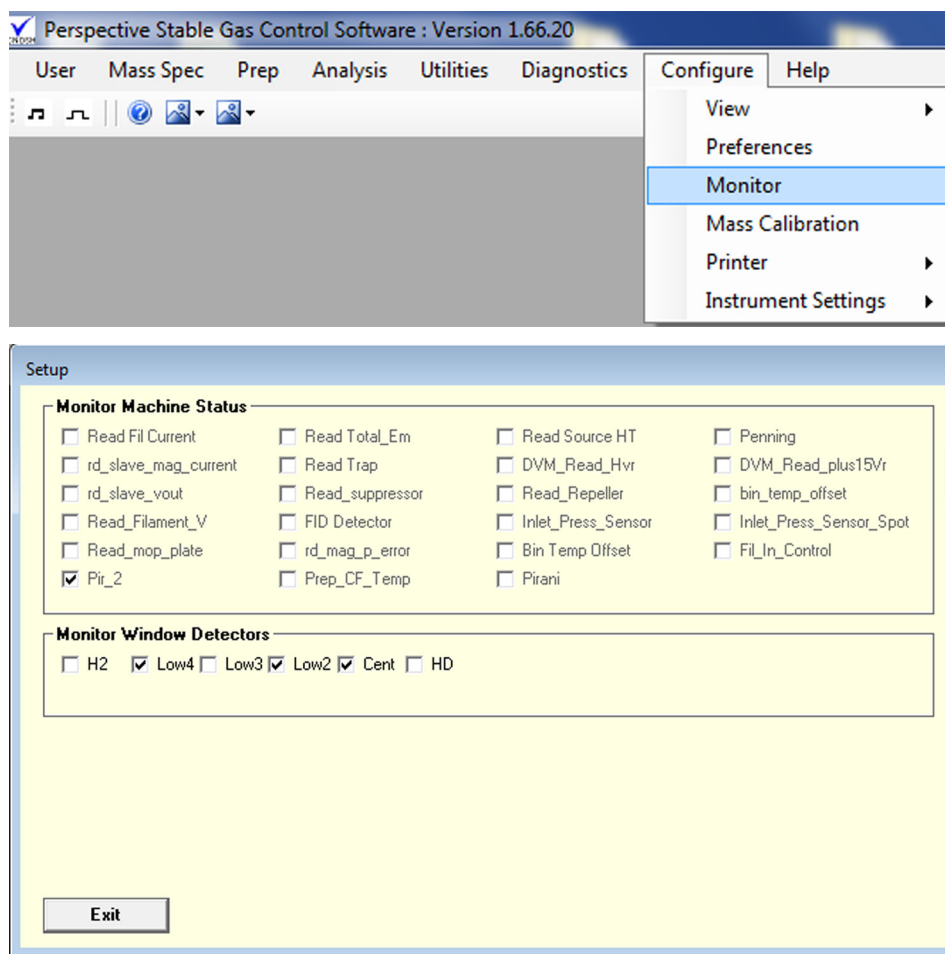


Figure 4.2-3: Setup window.

Up to three monitor values from the **Monitor Machine Status** pane can be selected and viewed in the **Main Monitor** window. However, if they are viewed in the **Magnet Scan** window, then only one should be selected.

4.2.3 Monitor windows

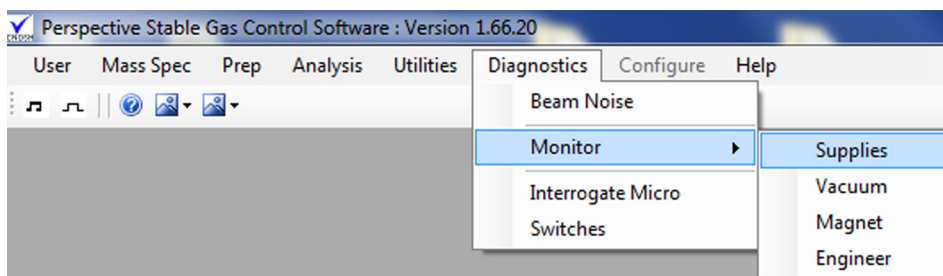


Figure 4.2-4: Opening the Monitor Supplies window.

1. Go to **Diagnostics > Monitor > Supplies**. The **Monitor Supplies** window shows readbacks for the source, various deflectors and the quad plates. For the quads, the voltages on individual plates are shown. These parameters are updated sequentially, over a period of several seconds. To see rapid changes in a parameter, use the **Setup** window and then display the chosen parameter using the plot monitor, in the **Magnet Scans** window.

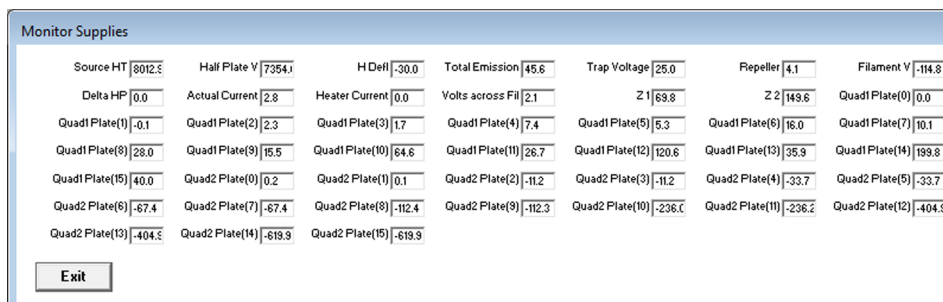


Figure 4.2-5: Monitor Supplies window.

2. Go to **Diagnostics > Monitor > Vacuum** to open up the Vacuum monitor. This monitor displays various vacuum parameters, including turbo-molecular pump temperature and speed.

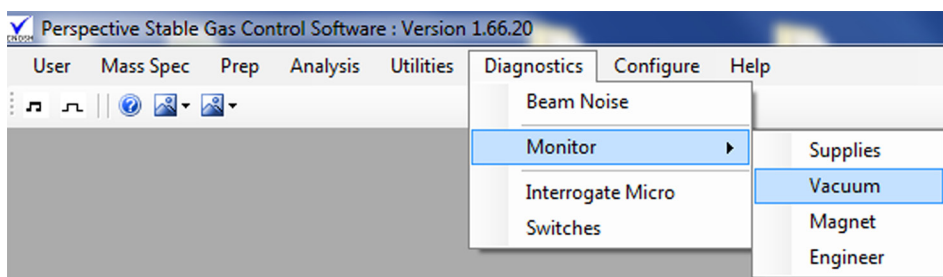


Figure 4.2-6: Opening the Vacuum Monitor window.

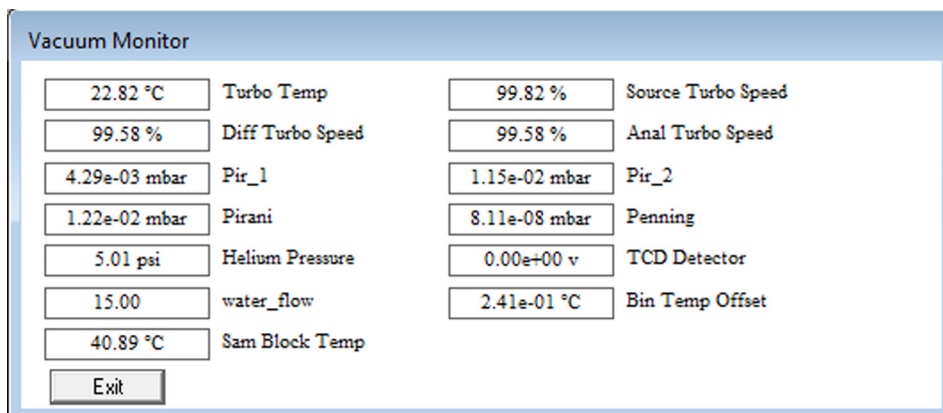


Figure 4.2-7: Vacuum Monitor window.

3. Go to **Diagnostics > Monitor > Magnet** to open the **Perspective Magnet Monitor** window. It displays useful information about the magnet parameters. The magnet current is particularly important, as it dictates the mass along with the accelerating voltage.

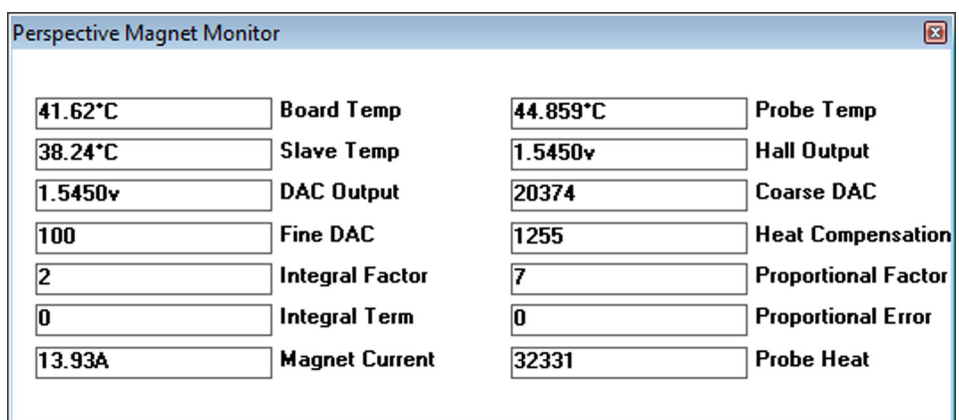


Figure 4.2-8: Magnet Monitor window.

4. Go to **Diagnostics > Monitor > Engineer** for a range of monitor windows. The most useful monitor windows are **HV1_TEST1**, **HV1_TEST2**, etc, which displays the set and read points for the various HV1000 outputs.

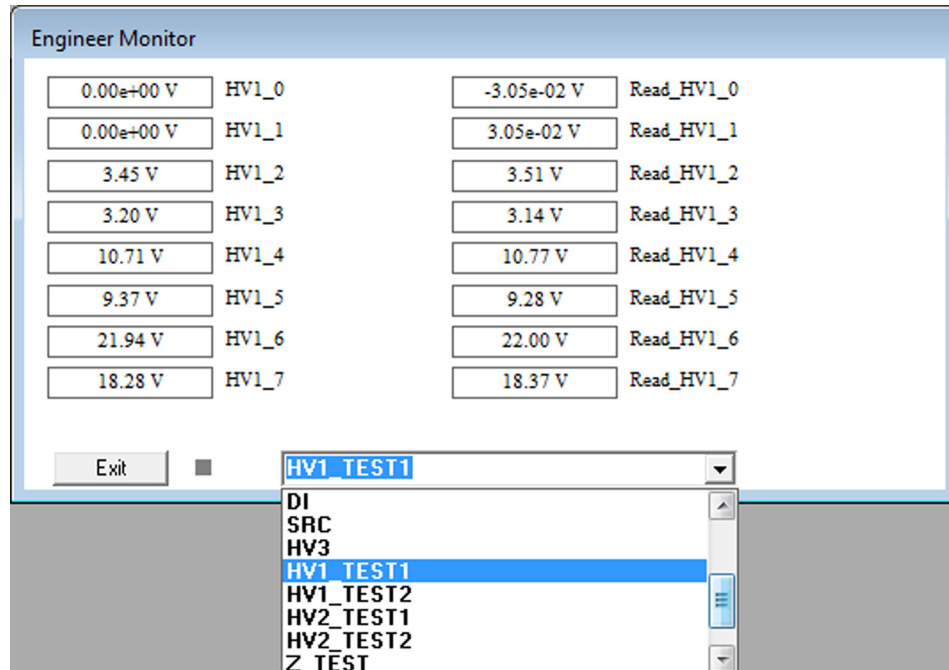


Figure 4.2-9: Engineer Monitor window.

4.2.4 Interrogate Micro window

This feature allows the user to read and write directly to and from the system controller. In normal operation, the user should rarely need to access the system controller directly. This feature is described here because the user may be asked to use it for fault finding, as advised by the factory. It can also be used to access certain readback values that are unavailable in the main control software <Stable.exe>.

⚠ CAUTION

This feature allows the direct control of System Controller parameters without any protection to keep them within a certain range. Use this facility with care.

Interrogate Micro is accessed from **Diagnostics > Interrogate Micro**.

1. Go to **Diagnostics** in the menu bar to open the **Interrogate Micro** window. The communication with the Micro can now be demonstrated.

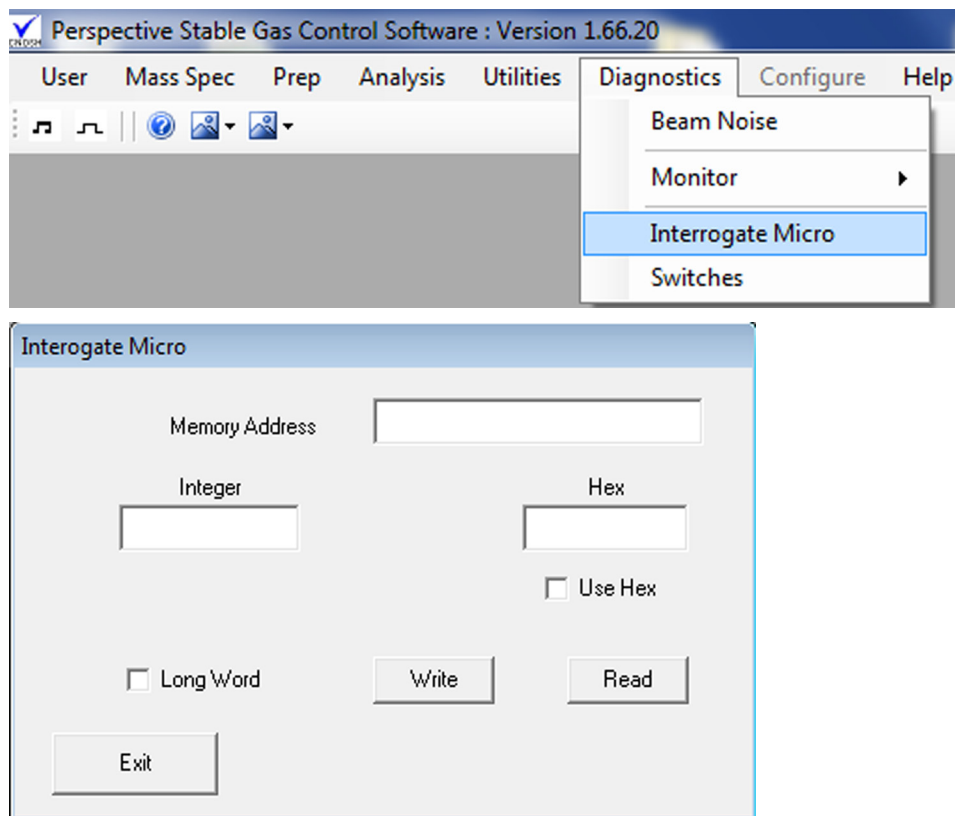


Figure 4.2-10: IC Interrogate Micro.

This feature will be demonstrated by means of a simple example, using the parameter **slot_cf1**. This parameter defines the position in the electronics rack in which continuous flow card 1 should be.

Enter the parameter name into the box labelled **Memory Address**, then press the **Read** button. In this example, continuous flow card 1 should be situated in slot 5.

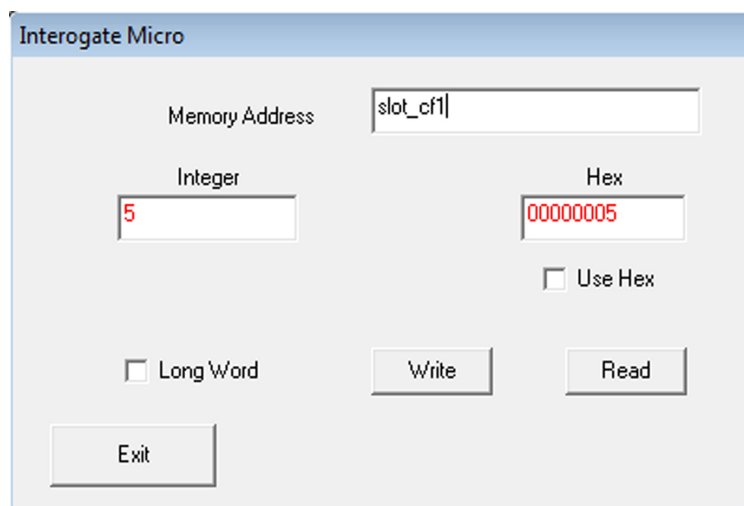


Figure 4.2-11: Interrogate Micro.

4.3 Software not communicating with the instrument

When first starting the software, the following message may be displayed.

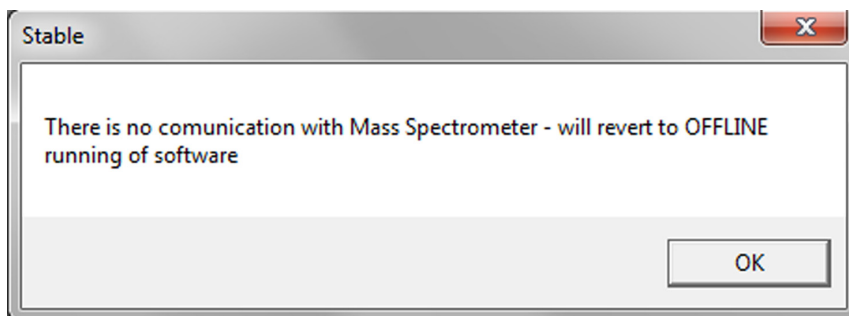


Figure 4.3-1: Communications error message.

If this message is seen, firstly try restarting the software. If the message still appears, there is likely to be a problem with the cable setup or the system micro is turned off.

Check that the system micro switch on the MDU is turned on.

Check that the communications cable is plugged into the connector on the back of the computer labelled **RS485**.

Next, check that the cable is plugged into the connector on the instrument labelled **Computer**.

If the above steps fail to remedy the problem, try resetting the electronics, as explained in **Section 4.5**.

4.4 Error codes

If the system is or has been in an error state, this is flagged up in the main status bar and also in the message log. Sometimes errors are temporary and can be cleared by going to **Mass Spec > Protection > Reset Trips**.

Listed below are the most common error messages, with a description and some possible causes.

Error	Description	Possible issues and fixes
Source out of regulation	Source is switched off or not in regulation.	<p>Source turned off intentionally - check parameters and attempt to turn on the source again.</p> <p>Source inoperable - check filament.</p>
Penning trip - Source OFF	Overpressure in source causing automatic switching off of the Penning gauge, source supply and HT.	<p>Leak on sample line or blockage.</p> <p>Large inrush into source or sample from prep system too large.</p> <p>For CF instruments, helium supply failed or compressed air failed.</p> <p>Backing pressure poor.</p> <p>Turbo-molecular pump faulty/turned off.</p> <p>System leak.</p>
Penning trip - Turbo <50%	Turbo less than 50% speed causing Penning, source supply and HT to automatically switch off.	<p>Backing rotary not pumping - check pump and backing pressure.</p> <p>Turbo molecular pump over-temperature - check turbo temperature in Monitor.</p> <p>Reset power.</p>
Turbo Overheating >45°C	Turbo-molecular pump too hot. The pump may automatically switch off to protect itself.	<p>Chiller switched off/ blocked.</p> <p>Turbo working too hard due to poor backing pressure.</p> <p>System baked with chiller turned off.</p>
Magnet slave trip	Magnet in protection mode.	Turn off software, reset the magnet by turning off and on the switch labelled Magnet on the MDU and then restart software.

		Hall probe knocked/ moved out of position.
Water Cooling Flow Low (<10) (only applicable if a manual flow meter is not fitted).	Water flow from chiller low.	Turbo chiller turned off. Flow sensor/water lines blocked.
Source voltage errors - each source voltage has its own error monitor e.g. Trap Voltage, Repeller, Source HT.	Voltage from unit does not match expected value.	Usually occurs if a large change is applied suddenly e.g. when tuning. Try resetting system trips to see if the problem disappears.

Table 4.4-1: Listing of error codes.

4.5 System hardware reset

Occasionally the electronics may need to be reset. The following steps will ensure that the system is protected during the reset.

1. In the **Digital** window turn off the **HT**, **Source Supply**, **Filament Supply**, **Deflector supplies** and **DVM supplies**.
2. Open the main isolation valve.
3. Open the source isolation valve and ensure that the gas is flowing from reference and sample. If a **Dual Inlet** is fitted, close the pump valves (LV, HV, PC, PH, WP, PF) and open all other valves.
4. Turn off the penning gauge.
5. Close the software.
6. On the rear of the instrument, turn off the switch labelled **Micro Rack**.
7. Wait 5 seconds and turn back on. If a **Dual Inlet** is fitted there will be a burst (10-15 s) of compressed air as the Venturi valves reset.
8. Restart the software. An error message will appear informing the user that the electronics have been reset. Allow the software to load, then restart again.
9. Return the system to operation following the normal procedure.

4.6 Unexpected pressure readings

The following section deals with pressure issues in the Pirani and Penning gauge.

4.6.1 Pirani errors

If the Pirani reads a value below 10^{-3} mBar, then it has lost calibration. Use a small screw driver to press the calibration button, labeled CAL. At the same time, monitor the Pirani pressure reading. When the reading reaches a value above 10^{-3} mBar, release the pressure on the button.

4.6.2 Penning errors

If the software switches off the Penning gauge as soon as it is switched on, then there could be a leak in the vacuum system. Check for possible significant leaks.

If the Penning gauge indicates an unrealistic vacuum value (e.g. pressure in the 10^{-10} or 10^{-11} mBar region), check if the cable is correctly plugged in at both ends. If the problem persists, switch the Penning off and on again and wait for a few minutes. If there is still an unexpected reading, then the penning anode may need to be cleaned. Over time, deposits can form on the anode, resulting in the pressure misreading. Full details of the service of the Penning gauge is given in the manufacturer's manual.

4.6.3 Hardware issues

The following section describes common hardware issues and how to solve them.

4.7 Valves cannot be operated

First of all, the compressed air system should be checked. Open the panel of the instrument with the words **Perspective** written on it and check the compressed air regulator gauge. The gauge should show a pressure of approximately 7 Bar.

If the compressed air pressure is correct, there may be a lack of communications from the electronics. Follow the steps of section 4.5 to perform a hardware reset.

If a specific valve cannot be operated, check the compressed air line for leaks.

There is a red light on the solenoid valves above the compressed air regulator, which illuminates when the valve is opened and off when the valve is closed (opposite for the source isolation valve). Check that this is operating correctly.

4.7.1 Leak checking using argon

CAUTION

Do not spray argon on the source region for long periods of time. Argon ionizes easily and may arc the source or source cabling to ground, potentially damaging components.

Firstly, perform the following test with the source valve closed, checking for leaks in the analyzer. If no leak is found, open the source valve and check for leaks in the pipework.

Scan the magnet from 39 to 41 amu, using only the **Cent** collector.

Adjust the scale of the **Cent** collector to accommodate the argon peak.

Stop the magnet in the centre of the beam and click on **Set Magnet**.

Set the 'Number of Steps' in the scan window to 500.

Set the scale to 10%.

Spray short bursts of argon around any junctions where leaks could occur, whilst observing the magnet scan. If the height of the beam increases, then a leak has been found.

4.7.2 Water cooling flow problems

If the error message **Water cooling flow too low (<10)** appears, go to **Diagnostics > Monitor > Vacuum**.

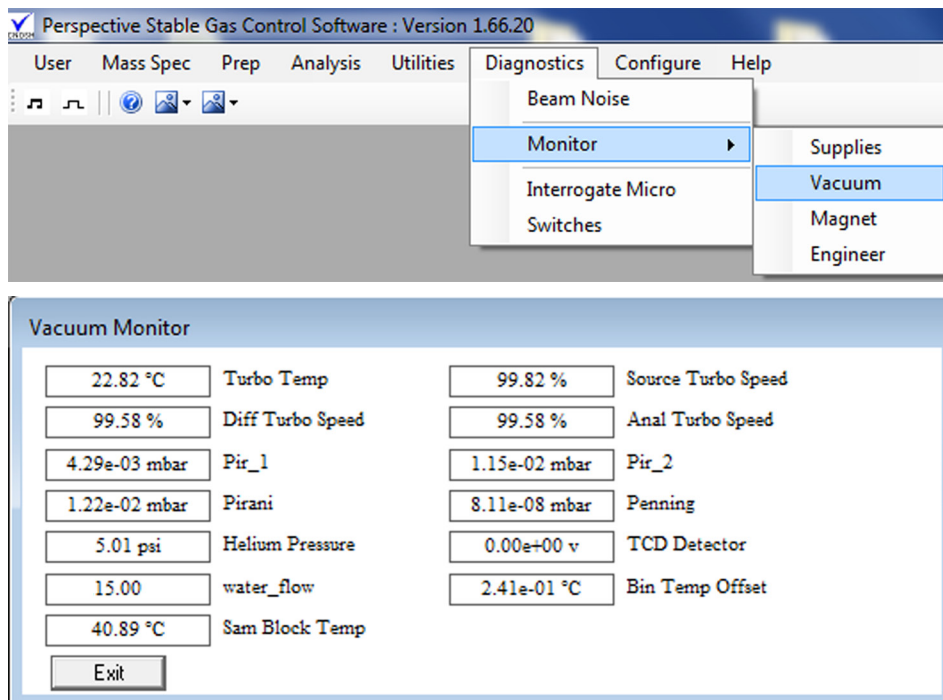


Figure 4.7-1: Vacuum Monitor window.

The **Vacuum Monitor** dialogue box will indicate the water flow level. If the indicated flow reading is below 10, then the software will flag up an error.

If the water flow monitor reads zero, tap the sensor a few times. It is located at the rear of the instrument, on the water out line. This usually fixes the problem.

If the water flow sensor is reading above 10, reset the trips by going to **Mass Spec > Protection** and click on **Reset trips**.

If the water flow monitor is still reading below 10, try tapping the sensor a few times again. If the problem persists, then there could be a blockage, most likely in the sensor.

Carefully avoiding spillages, switch off the water re-circulating chiller and disconnect both water lines at the chiller.

Reconnect them the opposite way around to try to flush the system through. If this does not work, it may be necessary to empty the system of water and blow through with compressed air (set to a low pressure to avoid damage to pipework).

Reconnect the water lines and switch on the chiller. Refill with water if required.

Appendix

I Aim of this chapter

The aim of this chapter is to give the user a broad introduction to some of the key principles and ideas in mass spectrometry, which relate to the design and use of the Perspective.

Mass spectrometry is used to perform an extraordinarily broad range of analytical tasks and a corresponding diversity of instrument configurations can be found in the field.

II Ionization source

Neutral species can not be accelerated, separated or detected by a mass spectrometer, so an ionization source is required. Its function is to generate ions from the received sample.

A number of ionization source designs are employed in the field of mass spectrometry. The choice of source depends on the sample form and the analytical task. Mass spectrometer systems such as the Perspective, in which gaseous samples are analyzed, use a Nier-type source, which generates ions through electron impact. A hot tungsten filament produces electrons, which are accelerated towards the ionization region. Small permanent magnets on either side of the source provide a vertical magnetic field in the ionization region. This helps to collimate the electron beam and causes the beam to follow a spiral path, thus increasing the path length and the chances of collision with gas molecules. After leaving the ionization region, the electron current is sampled by an electron trap, and the measured current, known as the trap current, is used to regulate the filament heating current, so as to ensure a stable ion beam. A schematic diagram of the source is shown in the figure below.

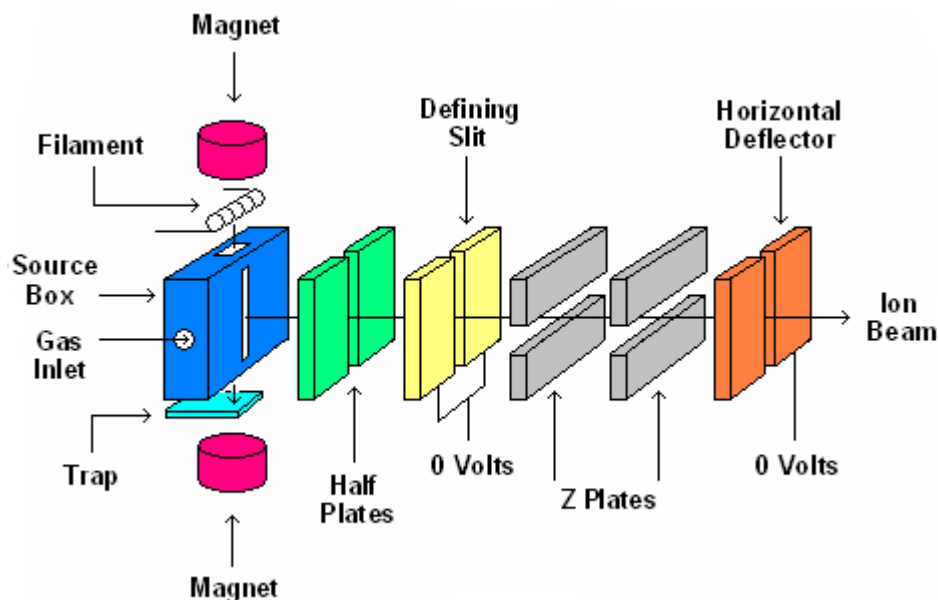


Figure A-1: Schematic diagram of the Perspective Nier-type source.

III Acceleration region

Ionization of species in the source does not impart the required trajectory of the ions towards the mass spectrometer. In fact, ions in the source will initially have a relatively low velocity, with a very wide cone of trajectory, so they need to be accelerated towards the mass analyzer. This is achieved using electrostatic fields, produced by plates at very high negative potential, with respect to the ionization region (in the case of positively charged ions). In practice, the source is often held at high positive potential, and the potential difference between source and ground provides the acceleration. If a high accelerating voltage is used, the divergence of the resulting beam of positive ions will be made as low as possible, which enhances the efficiency of transmission through the mass spectrometer entrance aperture. The kinetic energy of the ions accelerated by the accelerating voltage (E_{acc}) is given by equation 1.

$$\frac{1}{2}mv^2 = zeE_{acc} \quad (\text{equation 1})$$

Where m is the mass of the ion, v the velocity of the ion, e the electronic charge and z the number of electronic charges the ion carries.

IV Analyzer region

To visualize what happens to ions in the magnetic field of the analyzer region, the discussion will assume a population of ions of uniform ion energy. The beam of ions is steered towards the analyzer, in which they encounter a magnetic field \mathbf{B} , perpendicular to their velocity \mathbf{v} . In this magnetic field, each ion experiences a force \mathbf{F} perpendicular to both \mathbf{v} and \mathbf{B} , whose magnitude is given by equation 2.

$$F = zevB \quad (\text{equation 2})$$

Since the force F is always perpendicular to v , it acts as a centripetal force, and imparts a circular component of radius r_m to the ion's trajectory, shown in the figure below.

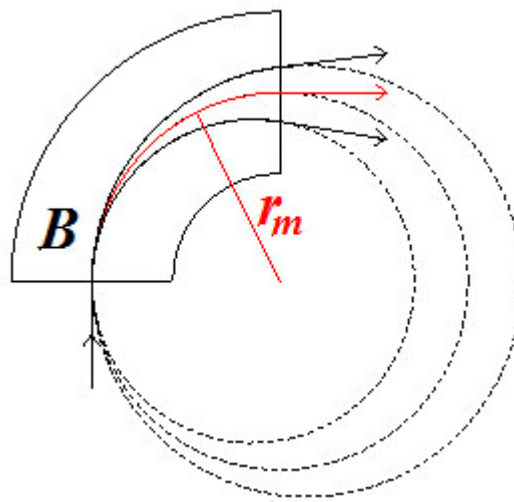


Figure A-2: Circular ion trajectories in a magnetic field.

The centripetal force is given by equation 3:

$$F = \frac{mv^2}{r_m} \quad (\text{equation 3})$$

Equating equations 2 and 3 gives:

$$zevB = \frac{mv^2}{r_m} \quad (\text{equation 4})$$

Eliminating v between equations 1 and 4 gives the radius r_m as:

$$r_m = \frac{1}{B} \sqrt{\frac{2mE_{\text{acc}}}{ze}} \quad (\text{equation 5})$$

For ions of unit charge, equation 5 approximates to:

$$r_m = \frac{144}{B} \sqrt{mE_{acc}} \quad \text{(equation 6)}$$

Where m is expressed in atomic mass units, E_{acc} in Volts and B in Gauss. With these units, r_m is expressed in centimeters.

Equation 5 has a number of important implications. For a particular magnetic field and for ions of the same energy, it can be seen that the magnet disperses ions according to their mass/charge ratio. Under these conditions, B and E become constants and equation 5 simplifies to:

$$r_m \propto \sqrt{\frac{m}{z}} \quad \text{(equation 7)}$$

This is the fundamental basis of the instrument. Ions with a smaller m/z ratio travel on a smaller radius. For singly charged ions, this simplifies further so that:

$$r_m \propto \sqrt{m} \quad \text{(equation 8)}$$

Therefore, in a mass spectrometer, for a fixed magnetic field, the circular trajectory of lighter ions has a smaller radius than the trajectory of heavier ions. Thus ions are separated according to their mass.

Considering ions with multiple charge, equation 7 indicates that these appear at a fraction of their true mass. For example, the doubly charged molecular ion $^{12}\text{CO}_2^{2+}$ appears at mass 22, rather than mass 44, where the $^{12}\text{CO}_2^+$ ion is observed.

In the above discussion, the magnet field was considered to be fixed. In practice, there is only a small range of radii on which ions can travel as they pass through the analyzer region. In order to analyze a range of masses, the magnetic field is varied so that an ion of a given mass travels on the correct radius to pass through the analyzer region. This situation is best described by rearranging equation 5 to:

$$B = \frac{1}{r_m} \sqrt{\frac{2mE_{acc}}{ze}} \quad \text{(equation 9)}$$

Taking the magnet radius as a fixed ion trajectory around the magnet and assuming that all ions of all masses have the same charge and energy, that is r_m , z , e and E_{acc} constant for all ions. Under these conditions, equation 9 becomes:

$$B \propto \sqrt{m} \quad (\text{equation 10})$$

Therefore, the magnetic field required to collect ions of mass m is proportional to the square root of the mass. This is the basis of the magnet mass calibration. (Note that the magnetic field and the Hall Probe output are proportional to magnet current).

For ions of the same mass, assuming a fixed trajectory around the magnet and that all ions have the same charge, r_m , z , e and m all have fixed values. Equation 9 simplifies to:

$$B \propto \sqrt{E_{acc}} \quad (\text{equation 11})$$

This means that by increasing the accelerating voltage, the magnetic field also needs to increase to keep ions of the same mass on the same trajectory and hence in a particular collector. That is to say, a different mass calibration is required.

For a single, unchanged mass calibration, equations 10 and 11 give:

$$m \propto E_{acc} \quad (\text{equation 12})$$

It may be useful to remember this when considering a change in the accelerating voltage. As an example, imagine that a mass calibration is firstly established at an accelerating voltage of 6 kV and the $^{12}\text{CO}_2^+$ ion at mass 44 is initially incident at the collector. If the accelerating voltage is then increased from 6 kV to 7.5 kV, the $^{12}\text{CO}_2^+$ beam would now appear to be at a higher mass of $44 \times (7.5 / 6) = 55$ amu.

A further important property of the magnet needs to be appreciated. Ions pass through the mass spectrometer source slit at an essentially singular position, since the source defining slit is narrow. However, they emerge from the magnet at a range of angles, forming a slightly diverging beam. In order to be counted, these ions must pass through another narrow slit immediately in front of the collectors, so they must be focused onto this slit. This focussing can be achieved by paying careful attention to the boundary of the magnetic field - that is by carefully shaping the magnet pole pieces.

As the ions pass through and leave the analyzer region, they are focused towards the detection system, essentially forming an image of the source defining slit at the slit in front of the collectors. This is the principle of the **single focusing** mass spectrometer, in which ions from a source slit are brought into focus on the collector slit. Without this focusing by the magnetic field, an image of the entrance slit could not be formed at the collector and so mass spectrum peaks, resolved by mass, would not be formed. The Perspective is a single focusing mass spectrometer.

V The effect of ion energy

From equation 5, it can be seen that for a particular magnetic field strength **B** and radius r_m ;

$$m \propto \frac{1}{E_{acc}} \quad \text{(equation 13)}$$

Therefore, by fixing the magnetic field, but changing the accelerating voltage and consequent ion energy, the mass following the trajectory with radius r_m changes. An alternative expression of this idea is that the mass of an ion measured at a particular collector changes if the energy of the ions is changed.

Alternatively, by considering ions of a particular mass, it can be seen from equation 5 that;

$$r_m \propto \sqrt{E_{acc}} \quad \text{(equation 14)}$$

So changing the ion energy changes the trajectory through the magnet, which gives the same result, that a particular mass may no longer be collected by the detector. In practice, this means that the source voltage needs to be very stable, otherwise the mass calibration will be unstable. Additionally, if the source produces ions with a significant spread in energy, a peak from ions of a single mass will be appear to be smeared or blurred.

Different types of ionization source produce different ion energy distributions. Glow discharge and plasma sources produce a wide range of energies. Consequently, these instrument types are double-focussing, incorporating an electrostatic analyzer (ESA) to compensate for this energy spread. On the other hand, thermal ionization or gas sources, such as the Nier source used by the Perspective, yield ions with a very narrow range of energy. These instruments are usually single focussing, and do not require an ESA.

VI Mass resolution and mass resolving power

The resolution of an instrument is a measure of the ability to separate two adjacent mass peaks. A higher resolution setting will be able to separate, or resolve, peaks which are closer together in mass.

A simple definition of mass resolution is;

$$\text{Mass Resolution} = \frac{M}{dM} \quad \text{(equation 15)}$$

where **M** is the mass being measured and **dM** is the smallest achievable mass difference between resolved peaks.

In practice, several different parameters can be used to describe peak shapes, and these are sometimes a source of confusion. These are outlined below, in order that the user may understand the various conventions used by mass spectrometer users.

Definition using two adjacent peaks

Often, **dM** is defined as the peak width at 10 % of the peak height. In this case, two identical peaks separated by **dM** will have a 20% valley between them:

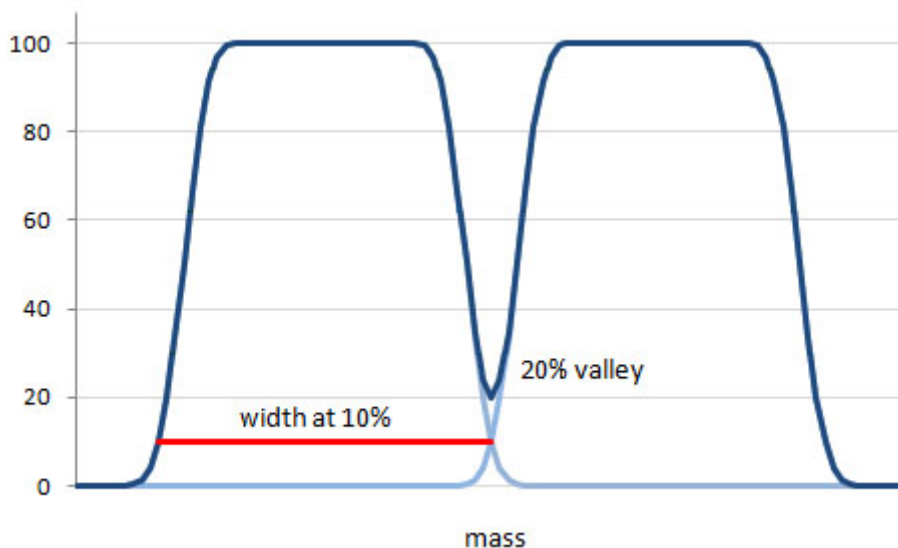


Figure A-3: Peaks resolved at 10 % of peak height.

In this definition, **dM** depends on both the collector slit width and the ion beam width.

Definition using the width of a single peak

An alternative definition describes **dM** as the full width of a single peak at half the maximum height of the peak (FWHM):

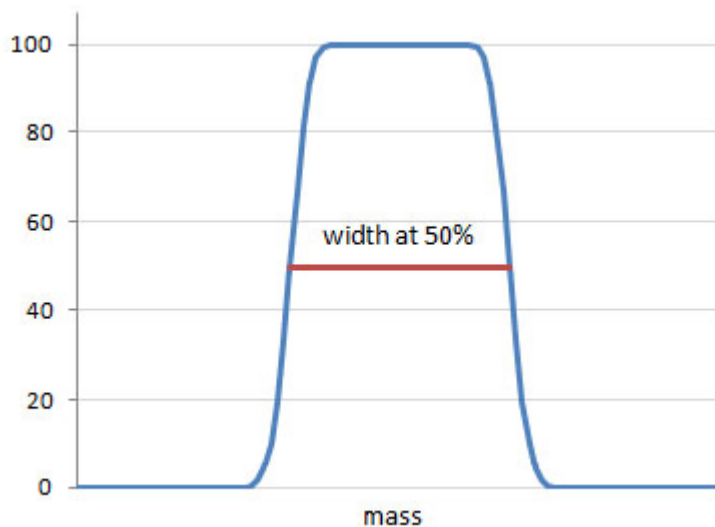


Figure A-4: Resolution at full width at half maximum height.

In this case, dM depends only on the collector slit width, but two peaks separated by dM will not be resolved (i.e. will not show a valley between them).

Definition using the width of the side of a peak

The width of the side of the peak corresponds to the beam being swept over the side of the collector slit. This can be used to define the mass resolving power or **MRP**:

$$\text{MRP} = \frac{M}{dM_{5-95}} \quad (\text{equation 16})$$

where dM_{5-95} is the mass difference between the points on the side of the peak, at which intensity is 5% and 95% of the maximum intensity:

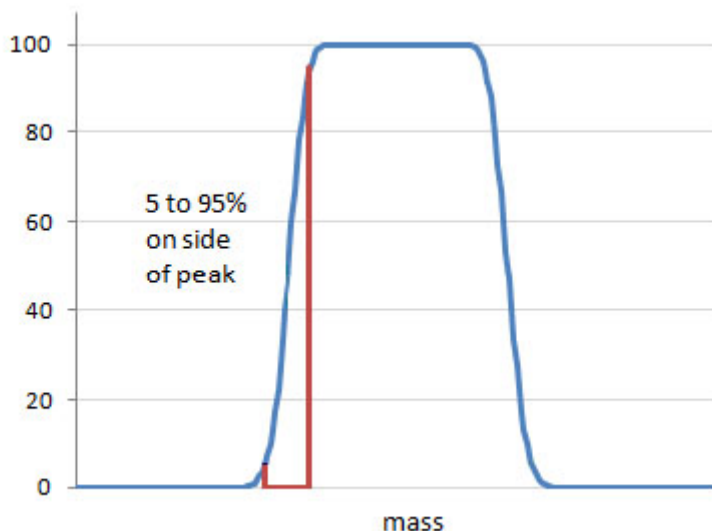


Figure A-5: Mass resolving power measured using the side of the peak.

A higher MRP corresponds to a narrower beam width, and a steeper side to the peak.

For precision measurements, it is conventional that the collector slit is wider than the beam (i.e. image of the source). This produces a flat topped peak, in which the entire ion beam for that mass is collected by the detector (in practice, this implies that MRP is at least several times greater than the mass resolution). Minor mass instabilities then have little or no effect on intensity, since if the image of the source moves slightly across the detector, the whole of the peak is still collected and measured intensity is unaffected. On the other hand, if the beam was the same width, or wider than the collector slit, the recorded signal would be sensitive to mass instability.

VII Mass dispersion

Mass dispersion quantifies the physical separation between adjacent masses in terms of a physical distance, called the dispersion length, **D**. This is fixed property of the mass spectrometer design. For peaks at mass **M** and **(M + ΔM)**, having a physical distance between them of **X**.

$$\frac{D}{X} = \frac{M}{\Delta M} \quad (\text{equation 17})$$

For peaks at adjacent masses, $\Delta M = 1$ amu and X is equal to the separation between adjacent collectors of the multi-collector array at that mass. Since D is a fixed property of the instrument design, equation 17 indicates that as the mass decreases, the separation between adjacent masses will increase.

VIII Abundance Sensitivity

This describes the tailing of one peak into its neighbor. There are two types of peak tailing in mass spectrometry, symmetrical and asymmetric and both can be observed at the same time.

Symmetrical peak tailing

Ions are accelerated by a high voltage, so they are travelling significantly faster than the mean velocity of residual gas molecules in the vacuum system. This means that collisions between fast ions and residual gas molecules will lead to only a small change in trajectory of the ion. Even so, a small number of these collisions will cause the ion to be diverted into an adjacent collector. When it arrives at one of the neighboring collectors, the ion is recorded at the wrong mass. This is the main cause of the symmetrical component of tailing from one mass onto its two neighbors, and can be significant when measuring very low abundance peaks next to masses of much higher abundance. Collisions after the magnet produce this symmetric tailing, since the change in trajectory is equally likely in any direction.

The cleanliness of the inside surfaces of the mass spectrometer is paramount in maintaining vacuum performance, but can also have another direct effect on peak shape. Any small nonconducting particulate contamination close to the ion beam path can gain a static charge, which will deflect a very small, but perhaps measurable, percentage of the total ion beam. The net effect can be the production of an elevated background of scattered ions.

Asymmetric peak tailing

The second type of tailing is asymmetric tailing to the low mass side of a peak. Almost all collisions will reduce the energy of an ion, since most residual gas molecules are slower-moving than the accelerated ions. When collisions occur before the magnet, resulting in the loss of energy of the ion, the magnetic field will deflect the ion more than it otherwise would if there had been no collision. In some cases, the extra deflection means that the ion is detected by the collector below the correct one. This is seen as asymmetric tailing, from the high mass peak across the lower mass peak. There is no tailing to the higher mass side, since energy is almost always lost in collision, not gained. This tailing is only significant when a high intensity peak is present one mass above a much lower intensity peak of interest.

The abundance sensitivity is conventionally defined as the amount of the tail from a peak at mass M , recorded in one of the adjacent collectors. The abundance sensitivity is normally expressed in units of parts per million (abbreviated to ppm), i.e. the number of counts due to mass M recorded in one of the adjacent collectors per million counts of mass M in the correct collector. Since the main cause of both types of tailing is scattering of ions by collision with the residual gas molecules, reducing the mass spectrometer operating pressure will improve the abundance sensitivity. There is a diminishing return for further and further pressure reductions, so other methods such as the use of retarding potential filters are required to meet the highest specifications.

IX Faraday Collectors

All of the collectors in the Perspective are of the Faraday type. The Faraday collector is a long thin receptacle into which the ions pass and collide with the base of the collector. On impact, the ion charge transfers to the collector and contributes to the current detected by the electronic circuitry. Faraday signal is amplified using a feedback resistor, typically in the gigaohm range. As an example, assuming that each ion carries a single electronic charge of 1.6×10^{-19} Coulombs, then 10^9 ion impacts per second would transfer $10^9 \times (1.6 \times 10^{-19})$ Coulombs per second (Amperes) to the collector, equal to 1.6×10^{-10} A. The rate of transfer of charge to the collector (current measured by the electronics) is proportional to the rate of impacts of incoming ions.

On impact of a high-energy ion, however, a number of other events can also occur. The initial impact will eject secondary ions and electrons from the surface of the collector. If one of these electrons were to escape from the collector, the electronic charge transferred would no longer be 1.6×10^{-19} Coulombs, but 3.2×10^{-19} Coulombs, since the collector has not only gained one positive ion, but has also lost a negatively charged electron. Similarly, if a secondary positive ion were to escape, the final recorded signal would become zero for that impact event, since one positive ion has entered the Faraday and one positive ion has exited the Faraday. The designs of these originally simple devices have become quite sophisticated, to eliminate these effects.

The base material is specially selected to minimize the yield of secondary species, and is now normally some form of highly porous carbon, which allows the incoming ion to penetrate into the bulk of the collector base, reducing the production of secondary ions. The collector "buckets" themselves are made as long as possible, to minimize the escape angle for any ejected species. Some form of magnetic field is also often present, so as to place the electrons into a spiral path, which makes it more likely that they will impact the side walls of the collector bucket, instead of escaping, increasing the trapping efficiency.

From early work on detectors, it was noticed that many systems showed anomalous negative dips, at the sides of the recorded peaks. These were identified as coming from secondary electrons formed as the ion beam strikes the detector slit (i.e. the metal shield in front of the actual detector device) and subsequently entering the collector mouth. This was especially prevalent as the ion beam struck the slit sides, even the use of very thin slit material failed to prevent the problem.

This effect was overcome by placing an extra electrode behind the defining slit. This is known as the suppressor and is held at between -50 V and -100 V. This repels the electrons scattered from the slit. As a bonus, electrons formed within the bucket itself are also repelled back towards the base of the collector.

X Signal noise

(i) Faraday noise

It is worthwhile to consider the origins of noise in conventional Faraday detectors. Rather than measure current from a Faraday collector, it is conventional to convert the small detected current to a voltage, using a high value resistor across a high impedance operational amplifier. The resistance is typically 10^9 to 10^{11} Ohms. There is a random noise associated with this high resistance (Johnson noise) given by:

$$\sigma(I) = \sqrt{\frac{4kT\Delta f}{R}} \quad \text{(equation 18)}$$

Where k	=	Boltzmann constant (1.38×10^{-23} JK ⁻¹)
T	=	Absolute temperature (Kelvin)
Δf	=	Measurement bandwidth
R	=	Resistance

For a 1 second measurement period, using a 10^{11} Ohm gain resistor and making the measurements at room temperature, there is a noise current limit of 4×10^{-16} amps rms, and for a 5 second integration period, this improves to 1.8×10^{-16} amps rms.

This is the absolute limit of this detection method and assumes that all other sources of noise have been eliminated.

(ii) Beam noise

There is a fundamental limit to the precision with which an ion beam can be measured. Since the beam consists of a stream of individual particles, which arrive at the detector randomly, statistical fluctuations are described by Poisson statistics: If n ions are detected, the expected standard deviation is $n^{1/2}$. (This is valid provided n is not too small, say $n < 100$). In Faradays, this gives rise to an effect known as shot noise and is described by equation 19 below.

$$\rho(I) = \sqrt{eI\Delta f} \quad \text{(equation 19)}$$

Where e = electronic charge (1.6×10^{-19} C)
 I = beam current (Amps)
 Δf = measurement bandwidth (s^{-1})

As a simple example, assume a beam current of 1×10^{-10} amps, a gain resistor of 10^{11} Ohms and a 1 second integration period. This gives a figure of 1.3×10^{-15} amps rms for the shot noise. In a Faraday system, the current is ($10^9 \times 1.6 \times 10^{-19}$) i.e. 1.6×10^{-10} amps. This is about an order of magnitude greater than the Johnson noise. This is fairly typical. In most practical situations shot noise dominates and to a first approximation can be taken to be the minimum expected noise level - assuming no other sources of noise.

If a full noise analysis is required, the Johnson noise and shot noise are combined as the square root of the sum of the squares.

XI Peak shapes

(i) Resolving adjacent peaks

Suppose that the source slit is uniformly illuminated by ions, and that a perfect image of this is formed at the collector slit. The following diagram shows signals observed by sweeping this beam profile over two different collector slits. The left example has the collector slit equal in width to the beam. On the right, the collector slit is double the width of the beam. The yellow blocks represent the ion beam, the blue lines are to indicate alignment and the red trace represents the measured signal.

peak shapes - single ion beam

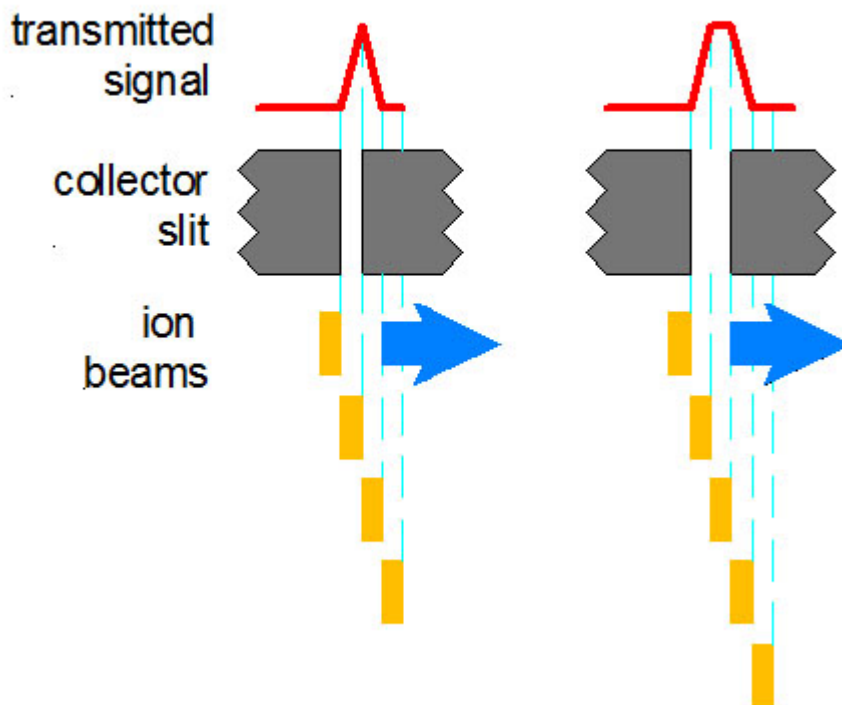


Figure A-6: Peak shapes for a single ion beam.

Key points:

The width of the side of the peak corresponds to the width of the ion beam. The straight edge to the side of the peak comes from the idealized ion beam profile in this example. In practice, the ion beam will not have perfectly sharp edges, giving rise to peak shapes more like those shown in section VI.

When the beam is the same width as the source slit (left example), the beam only just fits through the slit and there is no flat top to the measured peak.

With the collector slit wider than the beam, the peak shows a flat top.

Now consider an example with two ion beams. In this example, the ion beams are identical, and the distance between them is equal to the ion beam width.

peak shapes - composite peak

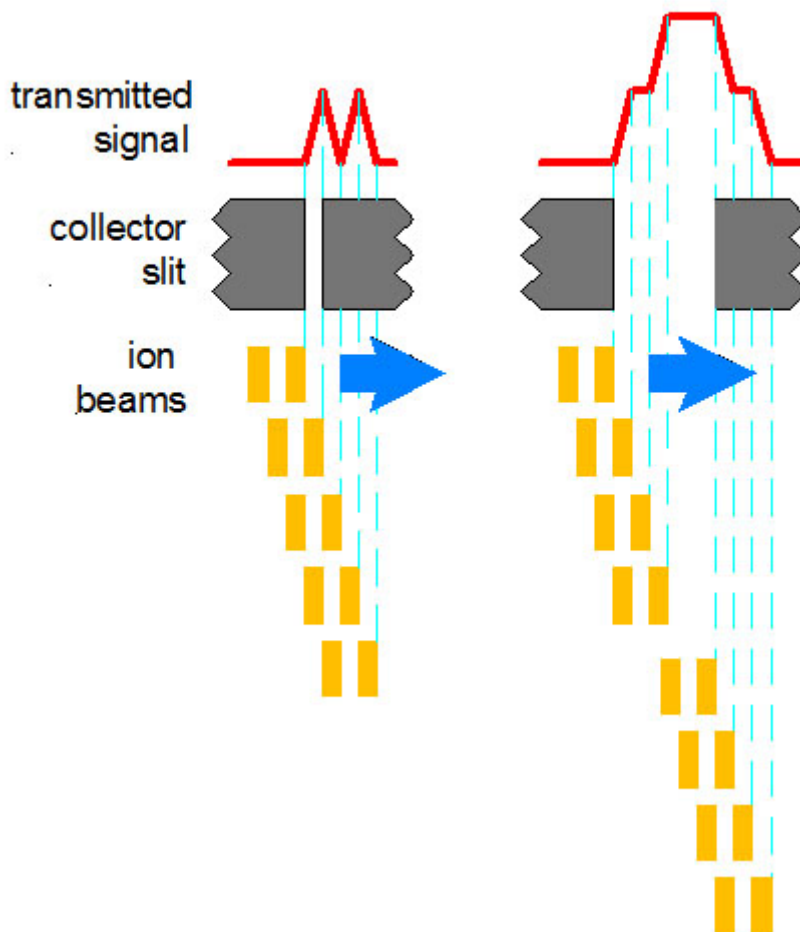


Figure A-7: Composite peak shapes.

In the left example, a valley is seen between the peaks. This is called true resolution, similar to the case discussed previously (section VI). Now consider what happens if this pair of ion beams is scanned over a wide slit. The first peak enters the collector and the signal is seen to rise to the plateau on the left - as in the case of a single ion beam. But now the second peak enters the collector before the first one has left, so the signal rises again. The central plateau corresponds to the situation where both ion beams enter the collector. The first ion beam then leaves the collector and the signal drops to the plateau on the right and finally the second beam leaves the collector and the signal drops to zero. This is known as a composite peak, and is shown in the right hand diagram. It is still possible to measure the ion beams independently, by sitting on the plateau on the side of the composite peak. This is referred to as **pseudo high resolution**. In fact, pseudo high resolution offers an advantage, in that some peak flat has been obtained, which generally gives greater signal stability, though it is preferable to have well resolved peaks.

The only time that this is likely to happen in stable isotope mass spectrometry is in the analysis of hydrogen. It is possible to tune the source in such a way that a measurable amount of He^{2+} is produced, which is very close in mass to the H_2^+ peak.

(ii) Aberrations

A simple description of the trajectory of the ion beam through the mass spectrometer will give an idealistic impression of what is really occurring, with the final image being an exact copy of the source, but life is never so simple. It is actually a distorted copy, and the degree of this distortion is a consequence of the aberrations introduced by the ion optical elements in the path, through which the ions have to pass.

First, a simple example is considered, in which the source slit is uniformly illuminated, producing the following profile, scanning across the beam immediately after the slit. In this example it is assumed that the source slit has a width of 1 mm;

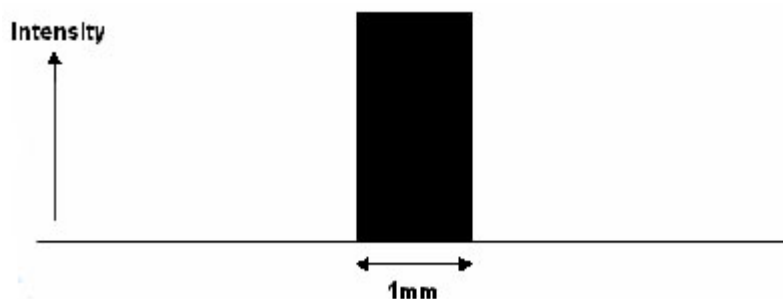


Figure A-8: Idealized image of a uniformly illuminated source slit.

The following description considers what occurs if this beam is scanned across a collector slit of, as an example, 3 mm width, in the absence of any distortions. This is the case for an ideal spectrometer.

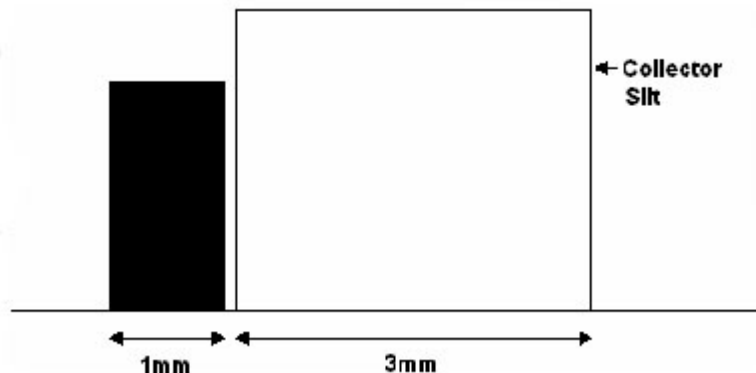


Figure A-9: The ion beam completely to one side of the collector slit.

In the previous figure, the magnetic field is set to a value that is just too low to allow any part of the beam to enter the collector slit. Nothing will be recorded on the detector. As the magnetic field is slowly increased, the image of the source slit will move from left to right. When the image of the source slit begins to overlap with the left hand edge of the collector slit, the detector begins to record some signal. The intensity of this signal will be proportional to the fraction of the source image overlapping the collector slit. This is shown in the figure below.

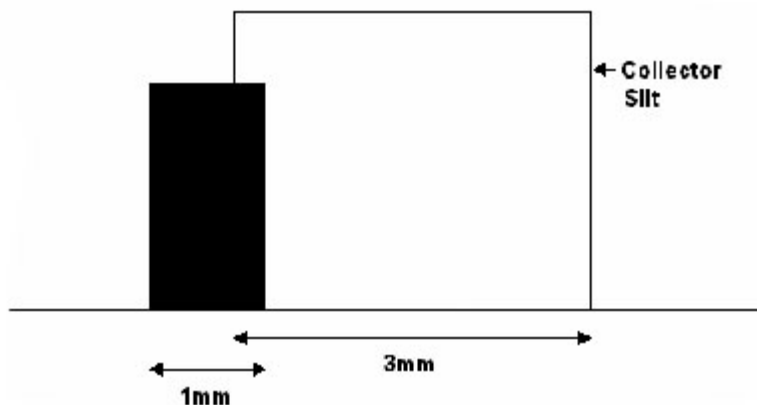


Figure A-10: The ion beam begins to scan across the collector slit.

The detector does not record the maximum possible signal until the magnet has moved the image of the source slit so that the whole of the image is just inside the collector slit. This is shown in the figure below.

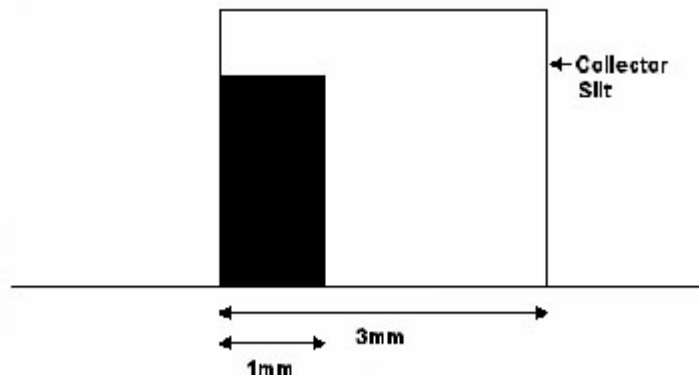


Figure A-11: Ion beam falling completely within the collector slit.

As the magnet moves the image of the source slit further to the right, the recorded signal will stay at its maximum value until the right hand edge of the image begins to leave the collector slit. Now the signal will begin to fall and reach zero when the left hand edge of the image leaves the collector slit.

Using this model, it is clear that as the magnet moves the image across the collector slit, the recorded signal profile will have the trapezoidal form shown in the figure below.

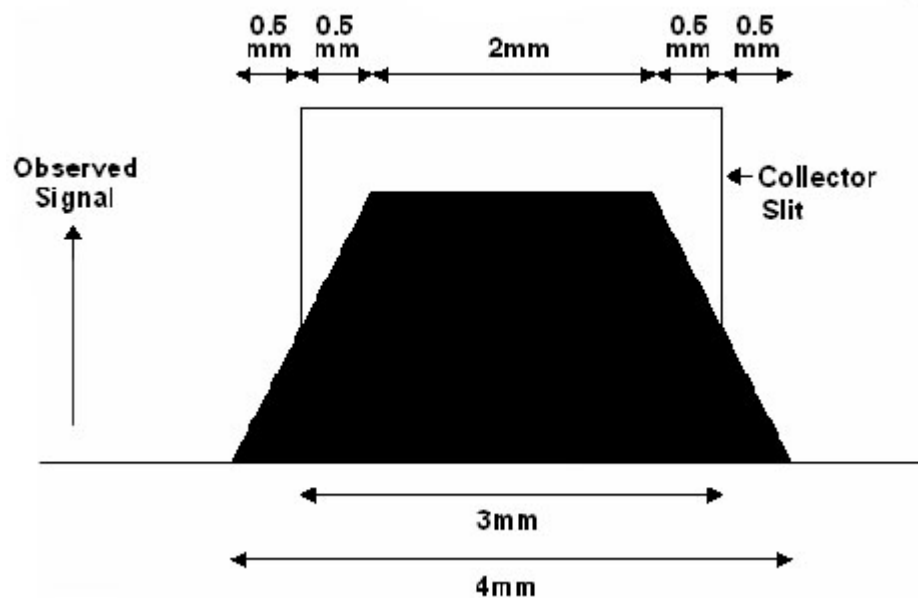


Figure A-12: The idealized trapezoid peak shape at the collector.

The width of the peak flat region of this ideal peak shape is equal to;

$$\text{Peak flat} = (\text{Collector slit width}) - (\text{Width of the image of the source slit})$$

The total width of the recorded peak base is equal to;

$$\text{Peak base width} = (\text{Collector slit width}) + (\text{Width of the image of the source slit})$$

The width of the peak at the half height position is equal to the collector slit width. With no aberrations, a perfect image of the source slit is formed, so in this instance the width of the image of the source slit and the actual source slit are equal.

In practice, the beam incident on the collector slit is not the ideal profile, but distorted.

These distortions can be simply grouped into two types. The most common is asymmetric distortion, in which the beam profile shows a distortion on one side.



Figure A-13: Asymmetric ion beam profile.

This is shown, greatly exaggerated, in the figure above. This figure also uses a narrower source slit width than in the previous example, in order to emphasize the effects. The observed detector signal from a beam profile shown above would look something like that shown in the figure below.



Figure A-14: The detector response from an asymmetrically distorted beam profile.

A symmetric distortion of the ion beam could produce an image of the source slit shown in the figure below.

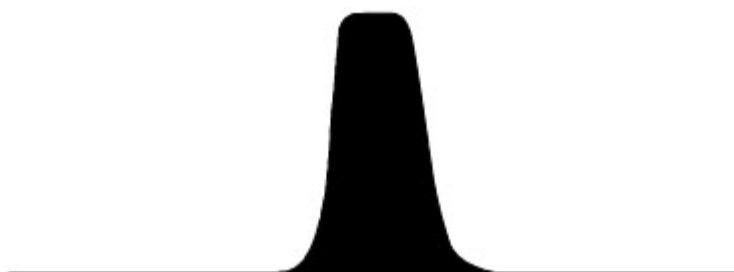


Figure A-15: The detector response from a symmetrically distorted beam profile.

Without going into detailed ion optical theory, it is generally the case that if there is a symmetric distortion, this can be corrected with the application of an evenly powered electrostatic field. The most common example of this would be the “normal” out of focus beam. Adjusting the zoom settings will bring the beam into focus. The asymmetric recorded beam intensity can often be corrected by the use of a cubic term to the zoom elements, but this may only work with the axial beam, and may add other distortions on the off-axis collectors.



Unit 74, Clywedog Road South,
Wrexham Industrial Estate, Wrexham, LL13
9XS, UK

Tel: +44 (0)1978 661304
Fax: +44 (0)1978664301
Email: sales@nu-ins.com
www.nu-ins.com