

 *nu instruments*

Perspective
Stable Isotope Ratio Mass
Spectrometer

Getting Started Guide
Issue 1.0

Preface

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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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Task 1: Manually introducing reference gas

Aim of this task

This task gives an overview of the software schematic through which the user gains direct control of the system valves. The schematic also gives various pressure and pump speed readbacks. **Part A** of this task describes the setup for continuous flow, **Part B** describes the setup for the Dual Inlet.

A: Continuous flow setup

The Perspective can be fitted with between two and five reference gas valves. The principle of operation is the same in all configurations. The reference gas is introduced into a stream of helium carrier gas, flowing in a stainless steel tube. A length of silica capillary is inserted into the stainless steel tube, the other end of which is connected to the input of the main mass spec valve. The silica capillary samples a small fraction of the carrier/reference gas mixture and introduces it into the source. This arrangement is known as an open split.

The flow rates of the helium and carrier gases are controlled individually, using Electronic Pressure Controllers (EPC) operated from the system computer. These are accessed through the Gas Control window shown below.

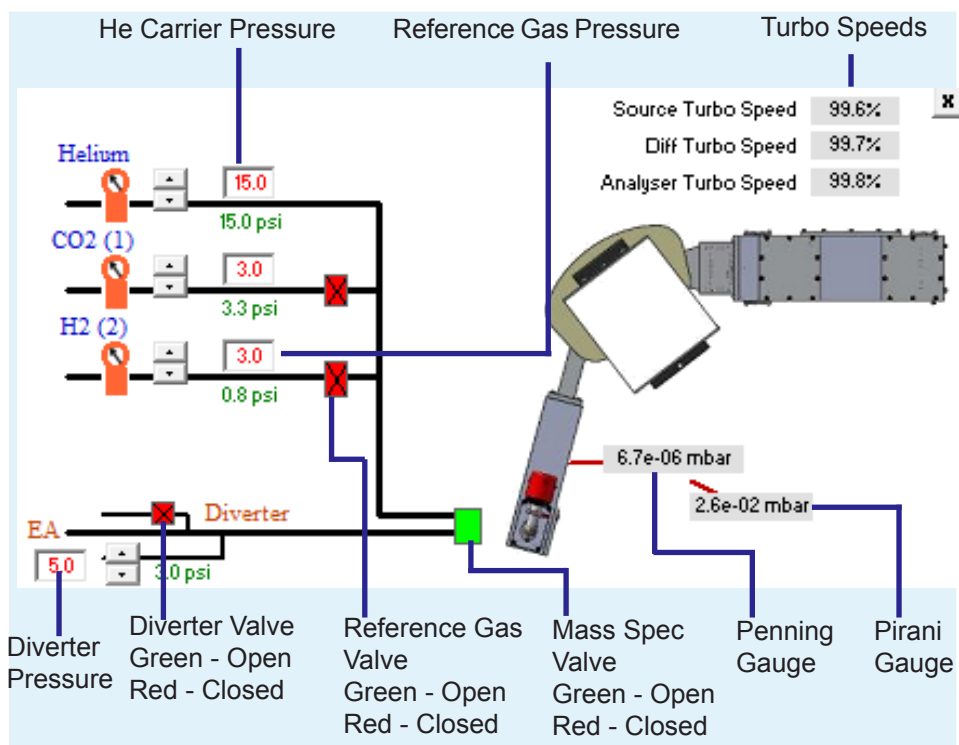


Figure 1.1: The Gas Control window.

When working in continuous flow mode, the helium carrier gas must be kept flowing at all times when the mass spec valve is open - otherwise air will enter the instrument via the open split. When the instrument is idle, the helium carrier gas can be conserved by reducing the flow rate to a few cm^3 per minute. Irrespective of the flow rate of the helium carrier, the pressure on the Penning gauge should be between 2×10^{-6} and 1×10^{-5} mbar.

The following instructions begin with no gas flows and the mass spec valve closed. If the helium carrier gas flow is already established and the mass spec valve is open, steps 1 and 2 can be omitted, but set the helium carrier EPC to 15 p.s.i. If a suitable reference gas flow is also established, point 3 can also be omitted.

1. Set the helium carrier EPC to 15 p.s.i. by clicking in the box at the top of the **Gas Control** window and typing 15, or by using the up and down arrows on the left box. The green numbers below the box indicate the actual pressure - after a few seconds this should match the set pressure. Before proceeding to step 2, wait for about 5 minutes, to flush air from the reference gas line.

2. Open the mass spec valve. If the source is operating, a prompt to turn off the HT and source will be displayed. This prevents damage to the source and source electronics, by the pressure burst when the mass spec valve opens. Once the mass spec valve has been open for about 1 minute, switch on the source and the HT, as long as the Penning gauge is reading in the 10^{-6} mbar region.

3. Set the **reference gas EPC** to 7 p.s.i. by either clicking in the appropriate box and typing 7 or by using the arrows to the left of the box. After a few seconds the actual EPC pressure (green numbers below the box) should match the set pressure. Wait for about 5 minutes for the reference gas to flush air from the capillaries between the EPC and the reference gas valve.

4. Open the reference gas valve by double clicking on the box shown in the figure above. Red indicates that the valve is closed. This should change to green, indicating that the valve is open.

Reference gas is now being admitted into the source, but the magnet may not be set correctly to measure any ion beams. The beams can be found using the magnet scan facility described in **Task 2**. Point 5 assumes that beams have been found.

5. Set the beams to the required current. This can be done either by adjusting the pressure of the helium carrier EPC or by adjusting the pressure of the reference gas EPC.

If the beam is too small, either decrease the helium carrier EPC pressure in small increments of 1 p.s.i. steps, or increase the reference gas EPC pressure, again in small increments, until the desired beam current is reached. Either of these actions will increase the fraction of reference gas in the carrier gas stream, leading to an increase in beam current.

If the beam is too large, either increase the helium carrier EPC pressure in small increments, or decrease the reference gas EPC pressure, again in small increments, until the desired beam current is reached. Either of these actions will decrease the fraction of reference gas in the carrier gas stream, leading to a decrease in beam current.

B: Dual Inlet setup

To protect the instrument from inadvertent damage, it is highly recommended that the Dual Inlet manual is understood, before attempting to admit gas into the Perspective from the Dual Inlet.

Gas can be admitted from the reference or sample bellows, but in most instances reference gas will be admitted from the reference bellows. A pressure of 20 to 50 millibar with the bellows fully open is usually suitable, though in special circumstances it can be higher. See the Dual Inlet manual for the introduction of an aliquot of gas into the sample or reference bellows.

The following description assumes that gas will be introduced from the reference bellows, but the procedure is easily adapted to apply to the sample bellows should that ever be necessary.

1. Admit gas to the reference bellows as described in the Dual Inlet manual.
2. A steady flow must be established in the reference capillary. This is to prevent a pressure burst, which may damage the source or the source electronics. Set the valve states to that shown in the figure below. Green indicates open, red closed. This allows gas to flow through the reference capillary to waste and not into the source. Wait for about 10 seconds. Steady flow will be established during this time period.

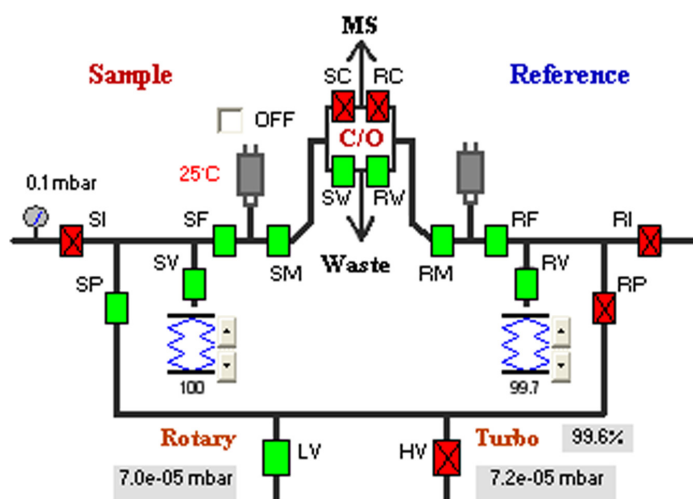


Figure 1.2: The Dual Inlet valves prior to admitting gas from the reference bellows.

3. Open valve **RC** and close valve **RW**. The change in valve states is shown in the figure below.

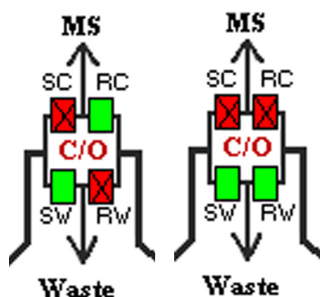


Figure 1.3: The change of state of valves RC and RW.

Gas from the reference bellows is now being admitted to the source. The beams can be found using the Magnet Scans facility described in **Task 2**. Point 4 assumes that the beams have been found and are being measured.

4. If the beam is smaller than that required with the reference bellows fully open, the position of bellows can be changed to achieve the required beam current. If the bellows reach the fully closed state and the beam is still too small, open **RW** and close **RC** and introduce a second aliquot of gas to the bellows and repeat points 1 to 3.

If the beam is larger than that required with the bellows fully open, the pressure in the bellows must be too high and needs to be reduced. Close **RV** to isolate the reference bellows, open **RW** and close **RC**. Now open **LV** and **RP** to evacuate the Dual Inlet, with the exception of the reference bellows, as shown in the figure below.

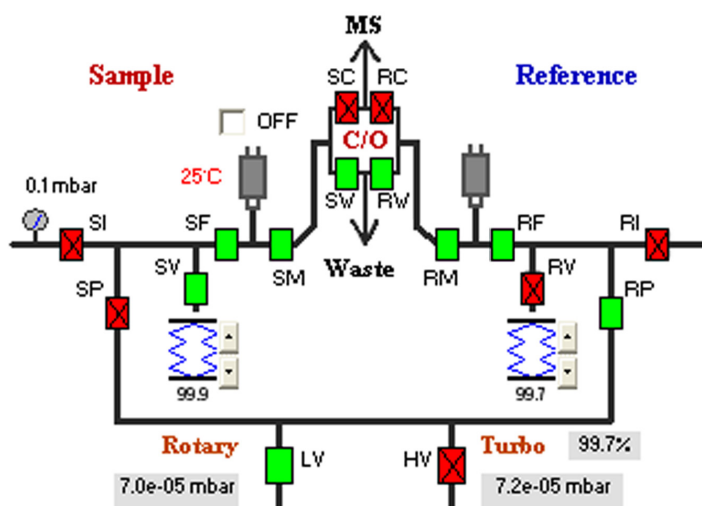


Figure 1.4: Evacuating the Dual Inlet apart from the reference bellows.

Wait until the pressure indicated by the Pirani Gauge next to **LV** in the figure above falls below 1×10^{-2} mbar then open **HV** and pump to high vacuum for 5 minutes. **HV**, **SP** and **RF** should then be closed as shown in the figure below.

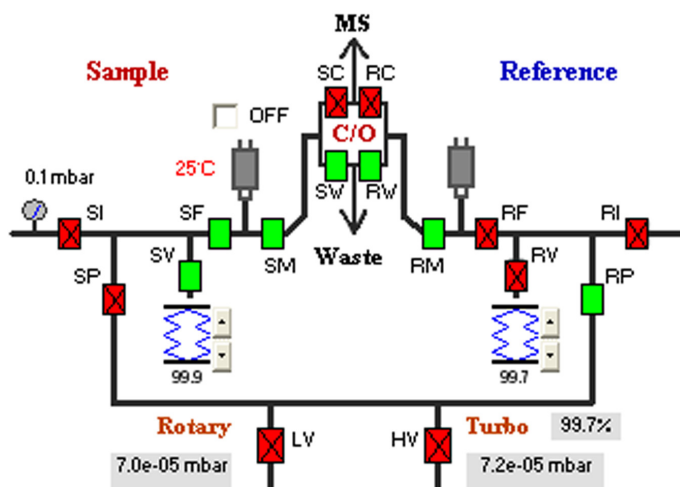


Figure 1.5: Isolating the volume between RF and SP.

Now open **RV** to expand the gas in the reference bellows into the additional volume between **RF** and **SP**, thus reducing the pressure. Repeat steps 2 to 4.

Task 2: Magnet Scans

Aim of this task

This task explains the functionality of the **Magnet Scans** window. All of the user definable scanning and display parameters will be described. This window can be used for tuning the instrument and for peak detection.

To start a magnet scan, go to **Mass Spec > Scan > Magnet**.

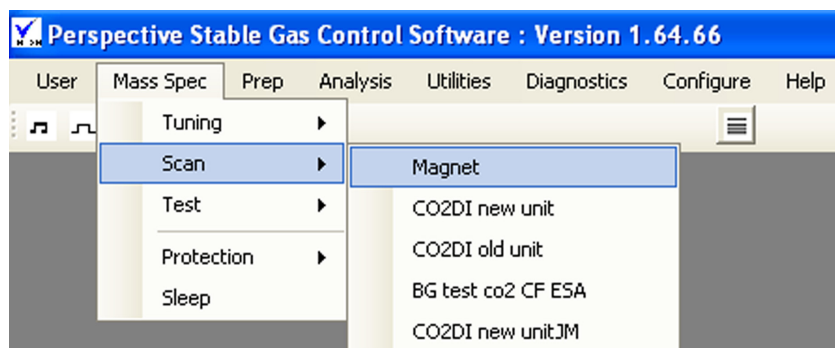


Figure 2.1: The Mass Spec/Scan Tabs.

Magnet will always appear in the box to the right. Clicking this will open the scan window with the parameters that were last used. The other options are filenames containing parameters that have been saved from previous magnet scans. Clicking one of these options will reset the scan window to use the parameters saved for that particular scan. The **Magnet Scans** window is shown in the figure below; the peaks in this example are the masses 44, 45 and 46 peaks from CO₂. The various control buttons are also labelled.

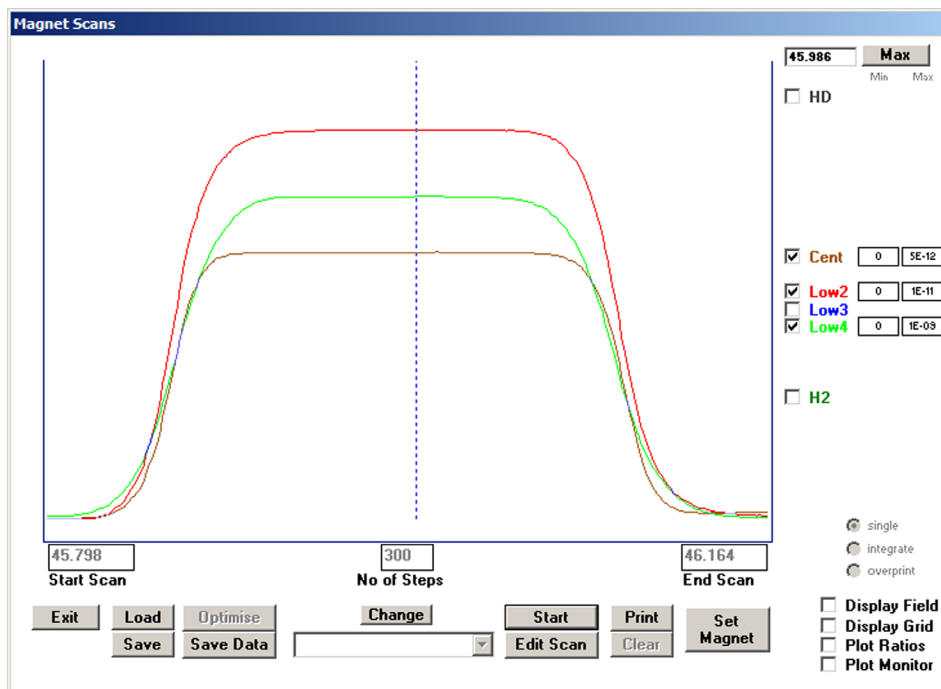


Figure 2.2: The Magnet Scans window.

The function of each control button is as follows, starting in the window itself.

The cursor

When a scan is in progress a blue dotted vertical line sweeps from one side of the screen to the other. This cursor indicates the current mass being collected in the collector labelled **Cent**. The mass scale is calibrated relative to this collector.

Mass Value Display

This is the small box at the top right hand corner of the Magnet Scans window. It displays the current mass position of the cursor.

Select and Scale Beams

Below the mass display, down the right hand side is a series of boxes. There is a check box, a label and to the right of the label, two more boxes. An example is shown below.

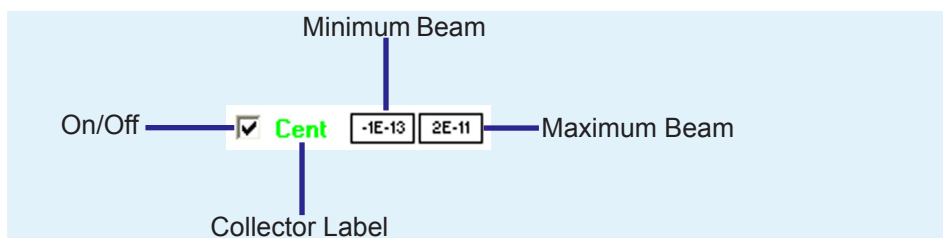


Figure 2.3: The Select and Scale Beams control.

If the box is checked, a trace corresponding to the beam in that collector will be displayed. The displays can be turned on and off by clicking in this box. The two boxes on the right show the maximum and minimum beam currents for the display. These can be edited by double clicking in the box and typing in a new value. The change takes place instantly. The minimum beam must, obviously, be less than the maximum beam. If this condition is not met, the box being edited turns red and no change occurs.

Depending on the collector set, there will be either six or ten sets of labels. If there are six, they will be labelled (from top to bottom) **HD**, **Cent**, **Low2**, **Low3**, **Low4** and **H2**. If there are ten the labels are (again, from top to bottom) **HD**, **Hi3**, **Hi2**, **Hi1**, **Cent**, **Low 1**, **Low 2**, **Low 3**, **Low 4** and **H2**.

Scan Mode Selection

These are the three round buttons in the lower right hand side of the display. Only one can be selected, if the scan mode is to be changed, click in the appropriate button - it will go dark - and that scan mode will start. The options are:-

- Single** This is single scan mode, in which the current scan replaces the previous scan as the cursor sweeps across the window.
- Integrate** This displays a cumulative average. The number of scans is shown adjacent to the mode selection buttons.
- Overprint** This displays the current scan, but retains all previous scans. Again, in this mode the number of scans is displayed adjacent to the mode selection buttons.

Display Options

These are the four options below the scan mode selection button in the lower right hand corner of the display. There is no limit to the number than can be selected. To turn on any particular option, click in the checkbox next to it. To turn it off, click the checkbox again. The options are:

- Display field** This displays the magnetic field. A box will appear in the top right corner of the scan window. This is only used during mass calibration.
- Display grid** This displays a horizontal grid across the scan window, each line is 1/10th the difference between the maximum and minimum values set by the scale buttons.
- Plot ratios** This displays the isotopic ratio across the scan range referenced to the major beam of whatever is being analyzed. So N_2 is referenced to mass 28, CO_2 to mass 44, etc. This will only be displayed after the first scan has been completed.

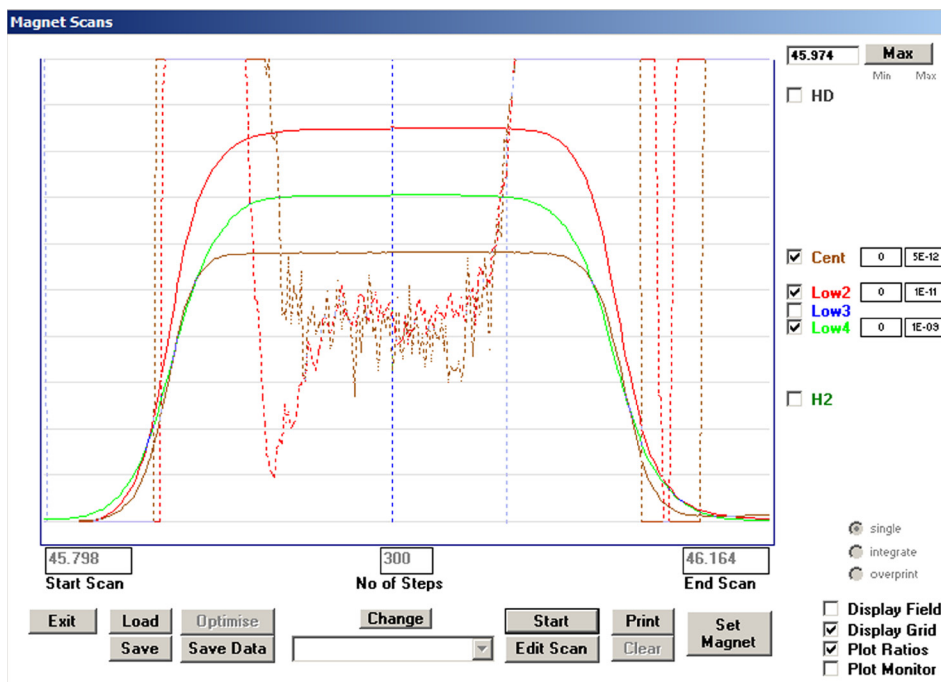


Figure 2.4: The display grid and beam ratios plot.

Plot monitor This allows the user to plot additional information in the scan window, such as Penning gauge pressure. The parameters to be displayed are selected by selecting **Configure > Monitor**, as shown in the figure below.

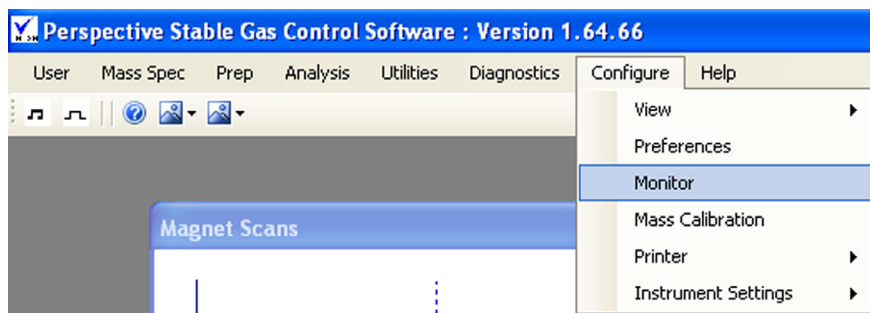


Figure 2.5: Selecting the Plot Monitor parameters.

The following panel will then appear.

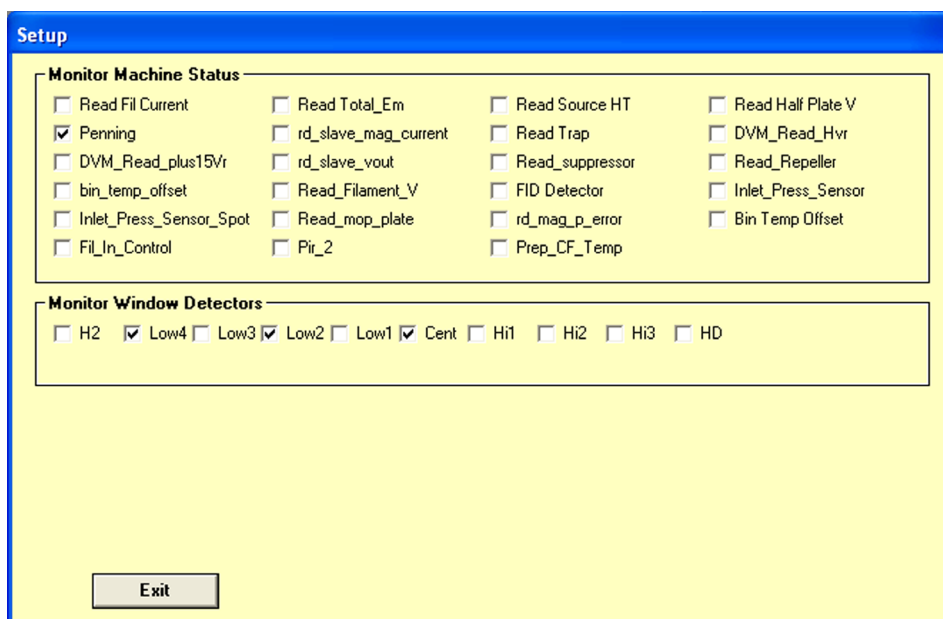


Figure 2.6: Plot monitor setup panel showing the list of parameters that can be displayed.

To select a parameter click in the checkbox next to it and a set of controls will appear in the lower right of the Magnet Scans window. These work in exactly the same way as previously described.

The name of the parameter being monitored and its value will also appear in the **Magnet Scans** window. Only one additional parameter can be displayed.

Click on **Set Magnet**.

Set Magnet

If the scan is stopped at any position in the scan window, using the **Start/Stop** command and the **Set Magnet** button clicked, the plot of mass against beam current will revert to a display of time against beam current, with the magnet fixed at the stop point. The Set Magnet label will change to **Restore Range**, clicking this will restart a normal magnet scan.

Print

This prints the **Magnet Scans** window to the default printer.

Clear

This clears the scan window and restarts the scan from the mass value set in the **Start Scan** box. If the scan was in **overprint** or **integrate** modes, everything is cleared, not just the last scan. The number of scans will start counting from zero.

Start/Stop

The label on this button toggles. When it reads **Stop**, clicking the button will stop the scan at the current position and the label will change to **Start**. Clicking the button when it reads **Start** will start the scan from the current cursor position and the label will change to **Stop**. Note that when the scan has been stopped, the cursor can be dragged to any position by right clicking on it. Starting the scan will start from the new position.

Edit Scan

Clicking this button activates the **Start Scan**, **End Scan** and **No. of Steps** boxes and the label on the button will change to **Save Edit**. Clicking in the Start Scan and End Scan boxes and typing a new mass in a.m.u. will enter new values. New values can also be entered by right clicking and dragging the cursor to a new position and then double clicking on it. If adjusting the scan parameters using this method, the Start Scan box will always update first.

The number of steps determines the time it takes for a scan, independent of the Start Scan and Stop Scan values. The scan time in seconds is approximately the number of steps divided by ten.

Save Data

Saves the scan data to a <.csv> file. All active beams are saved.

Load

This loads a previously saved magnet scan. Clicking this will open the window shown below.

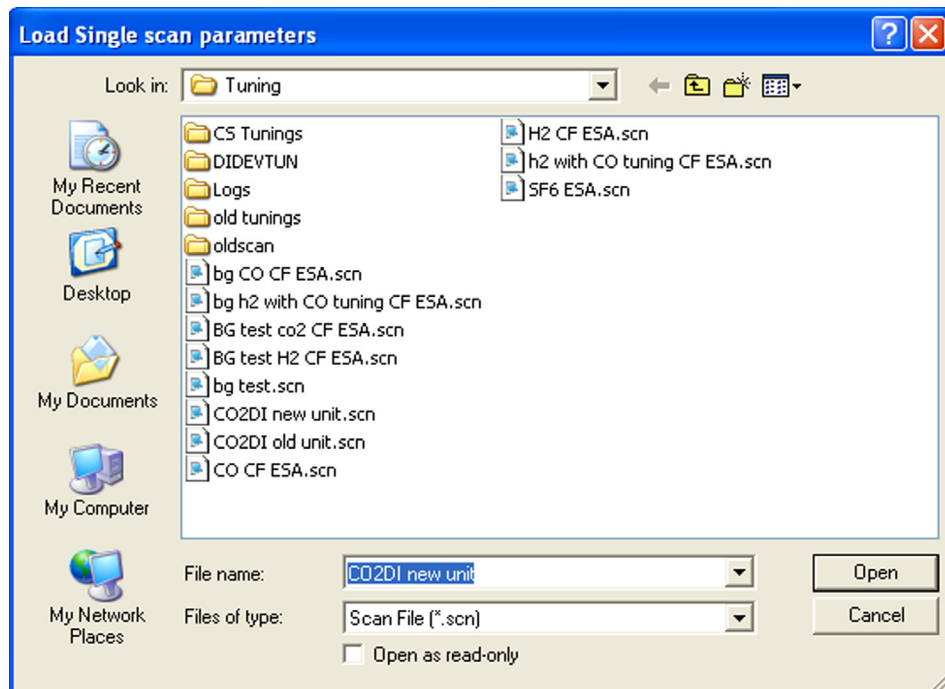


Figure 2.7: The magnet scan load window.

Select the required scan from the list and click **Open**.

Save

This saves the current scan parameters for future use. Clicking **Save** opens the **Save Single scan parameters** window.

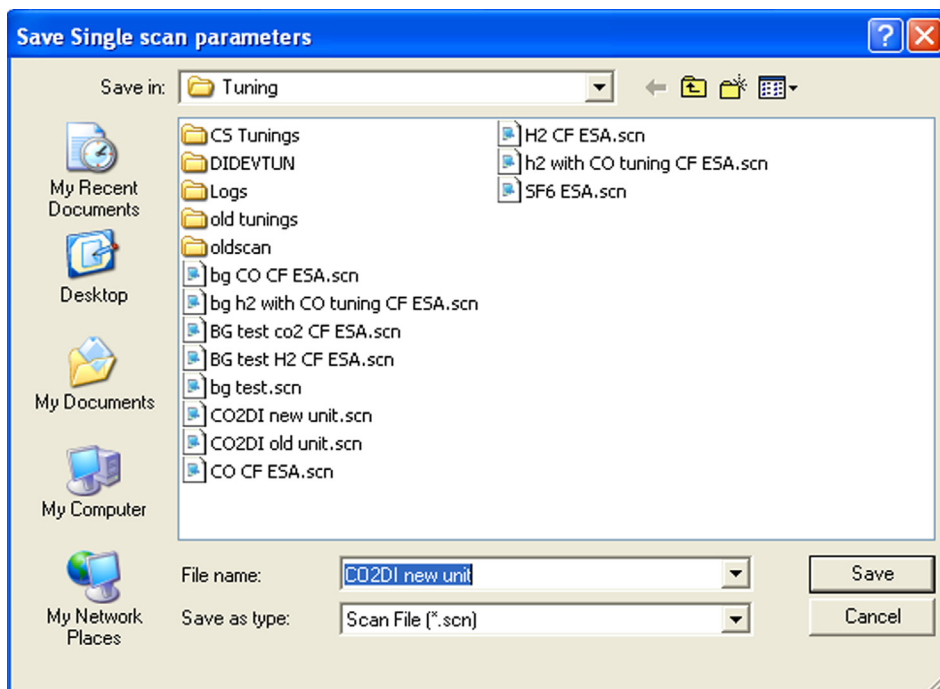


Figure 2.8: The magnet scan save window.

A prompt for entry of a new file name will appear. An existing file can be overwritten by selecting a file name from the list and clicking **Save**. If a file is being overwritten, a prompt will ask for confirmation.

Exit

Closes the Magnet Scans window at the current cursor setting.

Task 3: Tuning procedure

Aim of this task

This task explains in detail the optimization of the instrument tuning.

In the previous task, a peak was found in the **Magnet Scans** window and the correct collectors for each isotope were chosen. The Y-axis scale was adjusted, to allow viewing of the peaks in a single window. The source and the Quad lens voltages now need to be optimized.

Different tunings are required for;

- Different gases.
- The same gas at different beam voltages.
- DI and CF modes of operation.

The source may also need to be retuned if;

- The instrument has been moved to a new location.
- The instrument has been vented for a number of weeks.
- The filament has been changed.
- The instrument is not performing as expected.

Once a tuning condition has been found, it can be stored and reloaded when required. Some tunings are already stored and available at installation. If these are not suitable, a new tuning can be obtained using the following procedure.

STEP 1: Preparing the instrument for tuning

The instrument should be in the following state before attempting to retune the source and quad parameters.

- The instrument should be operating in either Continuous Flow or Dual Inlet mode.
- The source should be fully operational, with a previously determined tuning loaded.

1. To load the tuning file, go **Mass Spec > Tuning > Load**. The **Tuning Parameter** window opens, displaying all <*.tun> files. Select the appropriate file and click on **Open**.

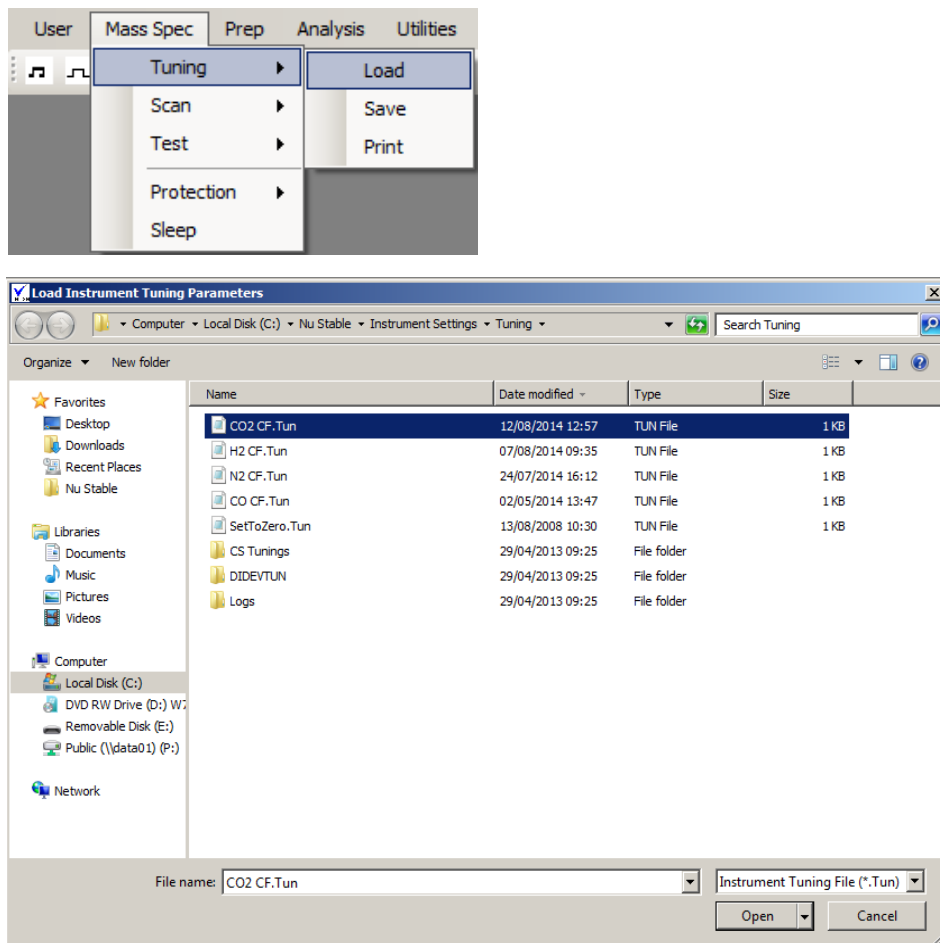


Figure 3.1: Loading the tuning file.

2. The voltages will now be automatically changed according to the selected tuning file.

STEP 2: Tuning the source

CO₂ is used in this example, but the same procedure can be used for any gas. There are minor differences if the tuning is for hydrogen. These are highlighted at the appropriate points.

1. If the beam voltage is to be changed, set it to its new value. This is done on the **Source** tab of the **Tuning** window. Also reset the Half Plate voltage to keep the ratio of Half Plate voltage to Beam voltage approximately constant.

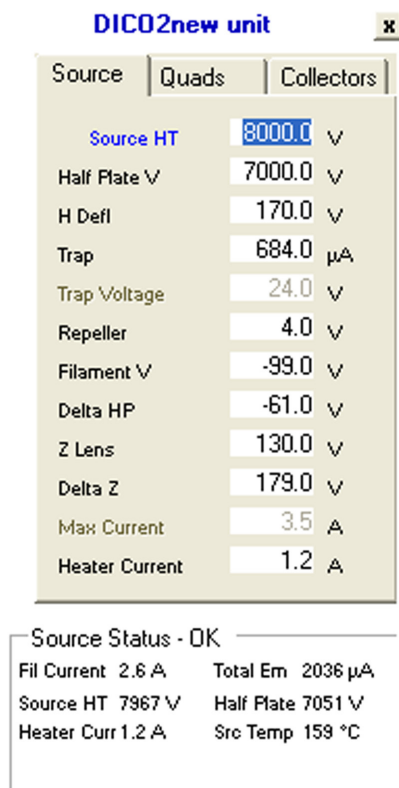


Figure 3.2: The Source tab in the Tuning window.

If working in Continuous Flow mode, make sure the CO₂ reference gas is connected and turned on at the cylinder with a regulator pressure of 4 Bar. Introduce the reference gas as described in Task 1 and start a magnet scan, with only the **Low4** collector selected. Locate the largest CO₂ peak. This is the mass 44 beam. Stop the scan when the cursor is in the middle of the peak.

If working in Dual Inlet mode, admit an aliquot of CO₂ into the reference bellows. Introduce CO₂ into the source as described in Task 1. Start a magnet scan with only the **Low4** collector selected. Locate the largest CO₂ peak. This is the mass 44 beam.

For hydrogen, select only the **H2** collector.

Note that if the **Source HT** voltage is changed, the current mass calibration will no longer be applicable. If the beam voltage has been increased, the range selected in the **Magnet Scans** window will need to cover a higher mass range. If the beam voltage has been decreased, a lower mass range will be necessary. The procedure to calibrate the mass scale is described in Task 4.

3. Once the beams have been located, set the magnet to measure the mass 44 beam in the **Low4** collector, by stopping the magnet scan on the top of the appropriate peak. Adjust the bellows or the reference gas EPC as appropriate, so that the beam current is between 10 nA and 20 nA. At this stage the peak shapes may be poor, which is not a problem at this stage.

For hydrogen, the beam is measured in the **H2** collector, not the **Low4** collector.

4. In the **Source** tab in the **Tuning** window set the **Trap Current** and **Heater Current** to the required values. These should now be kept fixed while the beam current is maximized, by varying the other source parameters shown on the Source tab. Maximise each parameter using the **Low4** beam current. Start with **Half Plate V** and work down the list of parameters. Several iterations will probably be required until the beam current is maximized for all the parameters.

For hydrogen, the reported beam current is the **H2** beam current. Note that the repeller voltage for hydrogen should be considerably higher than for any other gas, in the range 35 to 40 Volts. This is to minimize the production of H_3^+ ions in source reactions, but it does compromise beam current. For any other gas, the repeller setting is usually a few volts either side of zero.

Several of the source parameters interact with each other. This includes **Half Plate V** and the **Repeller Voltages**. **H Defl**, **Delta H P** and **Delta Z** lens Voltages also interact. It is a good idea to keep the Horizontal deflector as small as possible and to keep the Repeller Voltage within a few volts of zero, except for hydrogen as noted above. If the Repeller Voltage is too negative, the beam current will be reduced, if it is too positive poor peak shapes will result.

For Delta Z, there is a range of voltages at which the beam will be at maximum. It is recommended to choose somewhere close to the middle of this range.

Because of these interactions, there is no single correct tuning. If the beam is maximized following the general guidelines above, a reliable optimum will be obtained.

When tuning the source has been completed, continue to let the gas into the source, ready for tuning the quad lenses. Setting the quad lenses for hydrogen uses a different procedure, described in **Step 4**.

STEP 3: Tuning the Quad lenses for gases other than hydrogen

In tuning the quad lenses:

- Quadratic terms change the position of the focal plane.
- Cubic terms change the orientation of the focal plane.
- Linear terms act as beam deflectors and change the apparent mass.

The beam focus controls the steepness of the side of the peak. The aim is to obtain steep peak sides, with all of the beams entering their respective collectors at the same magnet setting, a condition known as coincidence.

1. Continuing with the use of CO₂ as the example, set all of the Quad lens voltages to zero. The current in the major beam should be between 10 and 20 nA.
2. Using only the **Cent** collector, set up a magnet scan over the mass 44 to mass 46 beams. This is to allow the identification of the correct beam for the focus step. An example is shown in the figure below.

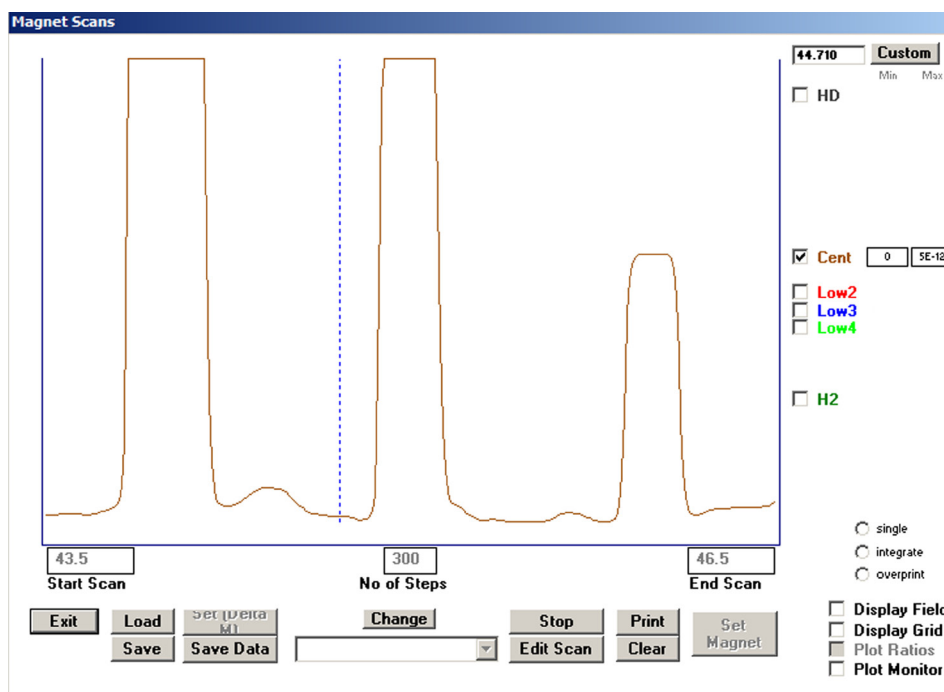


Figure 3.3: A magnet scan over the CO₂ beams using the Cent collector.

3. Using the magnet scan controls as described in **Task 2**, zoom in on the mass 46 beam, which is the mass normally collected in the **Cent** collector for CO₂ applications. The figure below shows the Quad settings before and after the tuning process. The right hand Quad values are the optimized settings.

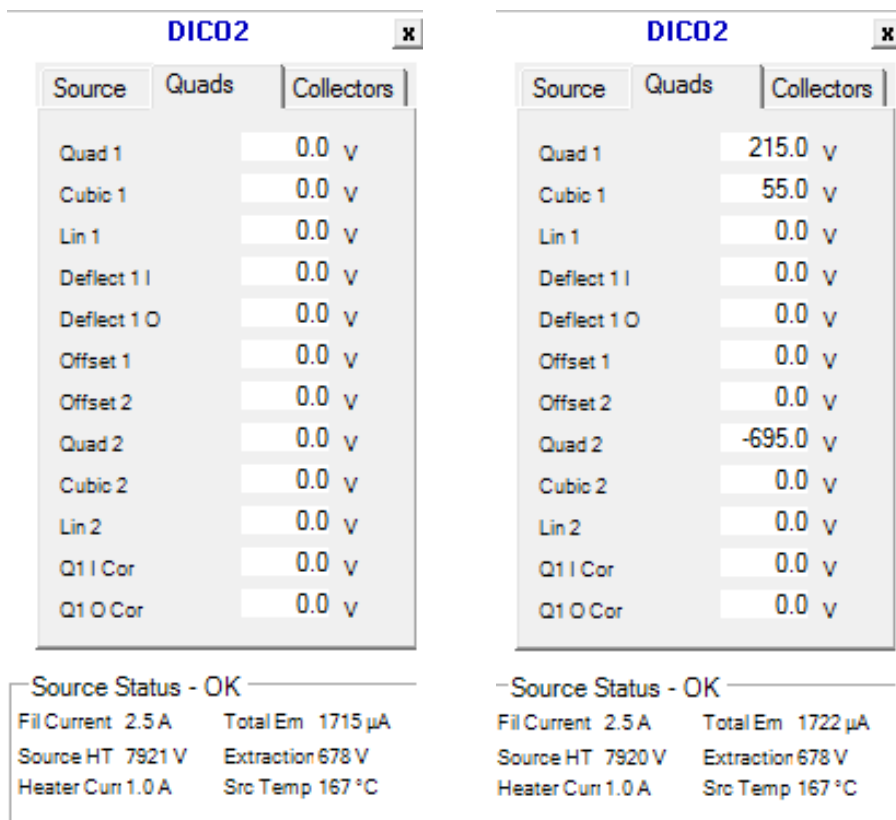


Figure 3.4: Quad values before tuning (left) and after optimization (right).

4. Using the magnet scan, scan over the peak in the **Cent** collector, and observe the effect on the peak shape on changing the **Quad 1** voltage. The aim is to obtain a flat top peak with steep sides. When this is achieved, the beam is in focus. The following figure shows the effect on the shape of the mass 46 CO_2 beam when the Quad 1 Voltage is changed from zero volts to -90 volts.

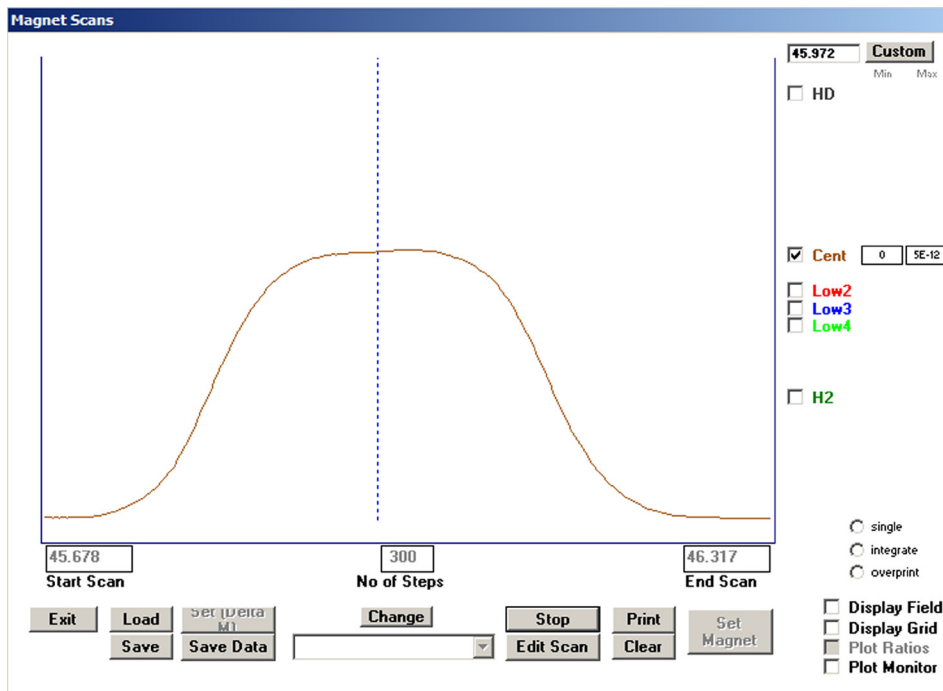


Figure 3.4a: Mass 46 peak shape at Quad 1 settings of Zero Volts (above) and -90 Volts (below).

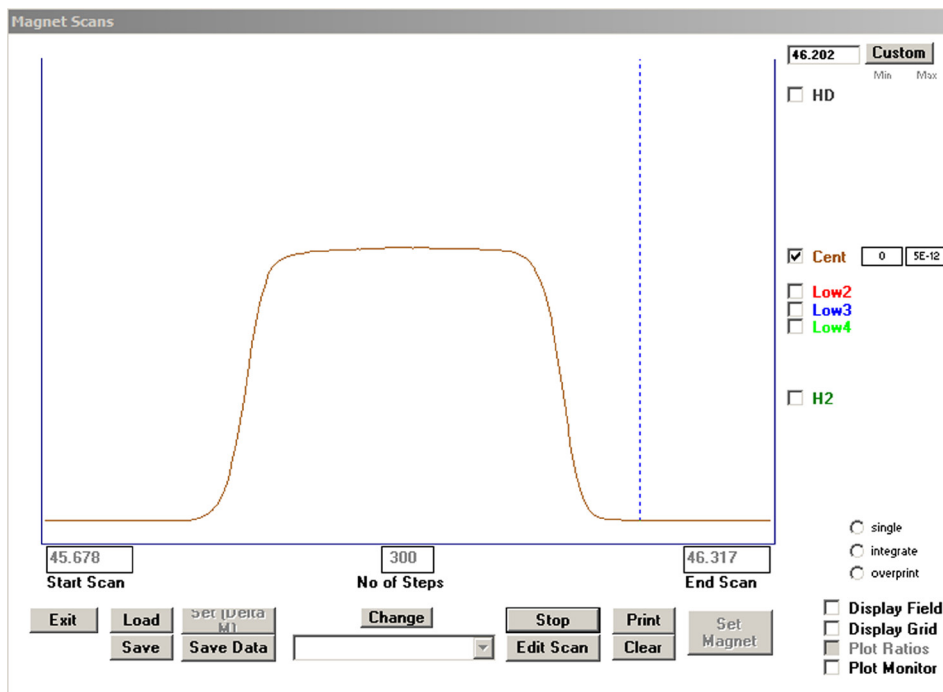


Figure 3.4b: Mass 46 peak shape at Quad 1 settings of Zero Volts (Figure 3.4a) and -90 Volts (above).

Changing the Quad 1 voltage is likely to cause a small shift in the peak position.

5. Turn on the displays for the **Low2** and **Low4** collectors. These will collect the mass 45 and mass 44 beams respectively. To see these beams in the **Magnet Scans** window, the range may have to be expanded and they will probably not be in focus or in coincidence. Beams falling into the **Hi** set of collectors, if required, will be dealt with later.

6. Adjust **Cubic1** until all beams are in focus. They will probably not yet be in coincidence. Re-check **Quad1**, which may be slightly dependent on Cubic1, and adjust it if necessary.

The focal plane is now parallel and coincident with the collector slit, with **Quad2** setting at zero. From this point, **Cubic1** should not be changed again.

7. Adjust the voltage on **Quad2** to bring the peaks closer together. As Quad2 is adjusted, the focus will deteriorate, but can be regained by re-adjusting **Quad1**. This process should be repeated until the beams are coincident and in focus. The figure below shows masses 44, 45 and 46 (from CO₂) before and after adjustments to Quad2, with the beams coincident and in focus. As the beams come closer to coincidence, the limits on the magnet scan can be reduced to obtain a more detailed view of peak shapes.

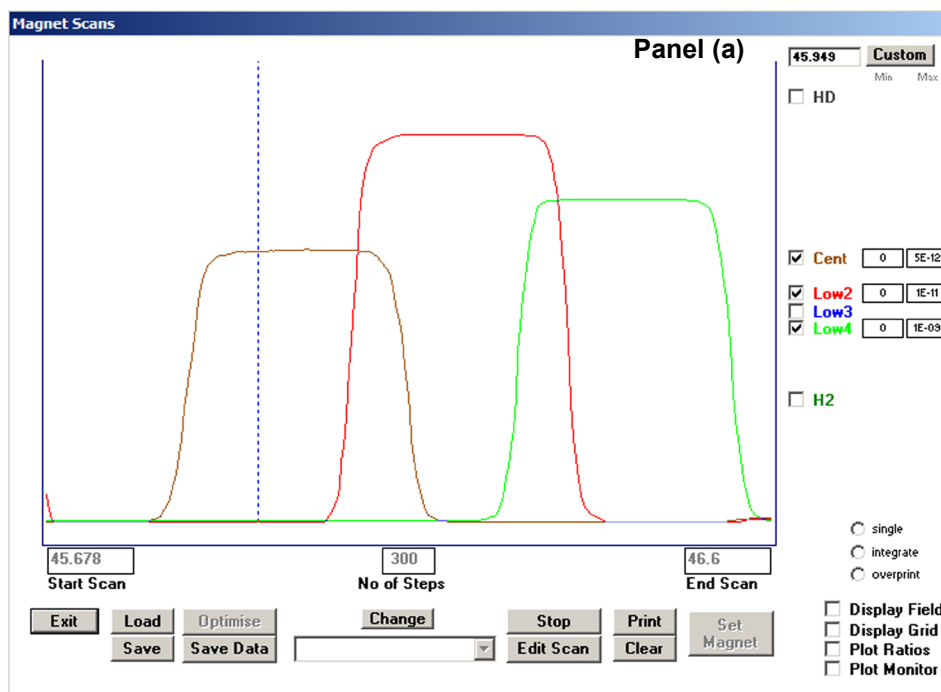


Figure 3.5: Mass 44, 45 and 46 peaks before and after Quad2 adjustment. Panel (a) shows the beams in focus, with Quad 2 at zero volts. Panel (b) shows the peaks closer together using Quad 2. Panel (c) shows the beams coincident and in focus.

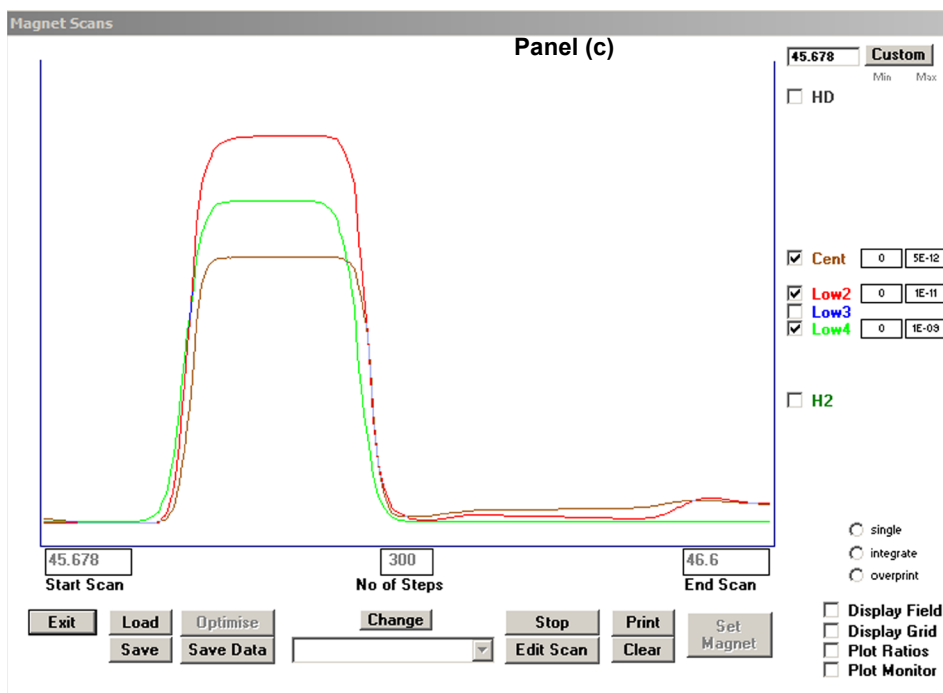
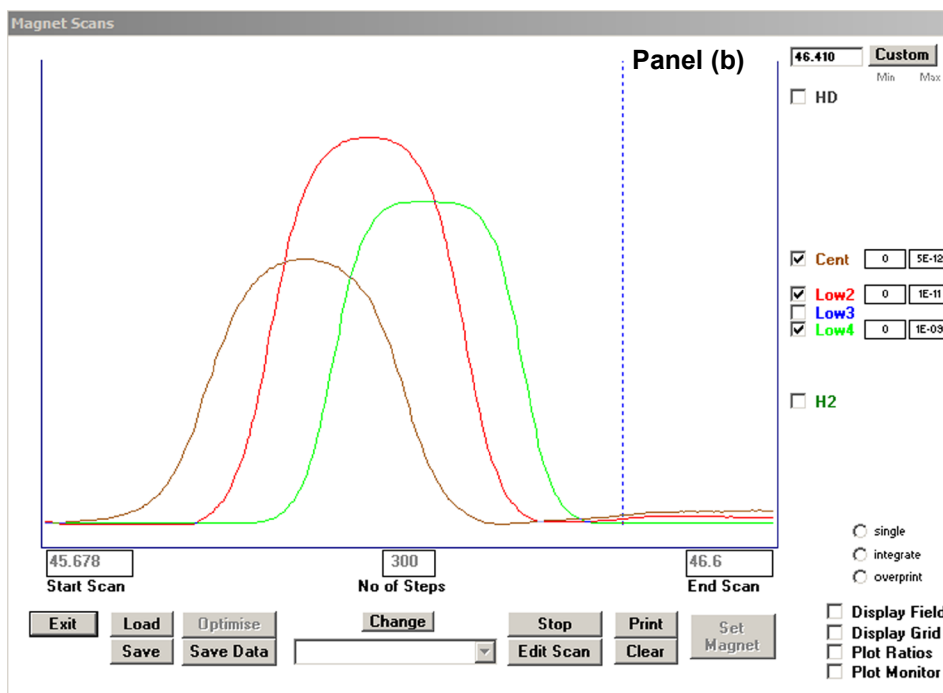


Figure 3.5 continued: Mass 44, 45 and 46 peaks before and after Quad2 adjustment. Panel (a) shows the beams in focus, with Quad 2 at zero volts. Panel (b) shows the peaks closer together using Quad 2. Panel (c) shows the beams coincident and in focus.

8. If beams collected in the **Hi** collector set are not required, go to point 9. If Hi collectors are required, the correct voltage on the ESAs must be found, using the **Collectors** tab of the **Tuning** window, as shown in the figure below.

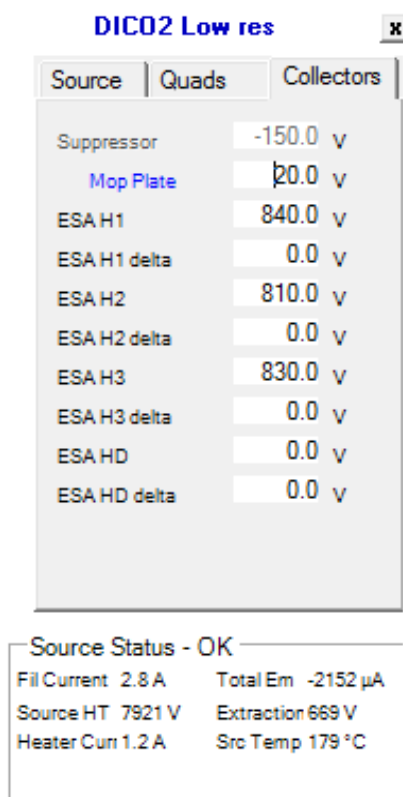


Figure 3.6: The Collectors tab in the Tuning window showing the ESA voltages.

The ion beam currents that are collected in the **Hi** collectors are usually much smaller than those collected in the **Low** collectors. To obtain reasonably sized beams, increase the amount of gas entering the source so that the major beam current is 30 to 40 nA.

On the right hand side of the **Magnet Scans** window, turn on the displays for the beams that fall into the **Hi** set of collectors. Having achieved coincidence for the **Low** beams, the high beams should also be in coincidence, or at least close to coincidence. If, however, the voltages on the ESA filters are not optimized, peaks may not be seen.

The ESA voltages must be adjusted until focus has been achieved and the peaks show a flat top. The ESA voltages are listed on the **Collectors** tab in the **Digital** window. The first one is the **H1** collector, followed by the **H2** collector, **H3** and **HD** collector.

Each ESA consists of a pair of curved parallel plates with equal voltages of opposing polarity applied to them. For example, if ESA H1 is given a value of 800 V in the **Digital > Collector** window, the outer ESA H1 plate will be at +800 V and the inner ESA H1 plate will be at -800 V. The opposing voltages cause the ion beam to follow a curved path through the ESA. Ions of lower and higher energy are filtered out. As a guide, for an 8kV source voltage, ESA settings of 800 V to 900 V are typically required.

The user can apply voltages of different magnitude to the inner and outer plates of the ESAs by using the **delta** value. For example, if **ESA H1 delta** is given a value of 20 V, the outer ESA H1 plate will be at 810 V and the inner ESA H1 plate will be at -790 V.

Delta values are not commonly used, only if the deflection of the beam has to be changed to finely adjust the focus.

9. Set the voltage on all unused ESA plates to zero.

10. Save the new tuning condition.

11. If the **Source HT** has been changed, a new mass calibration will be required - see **Task 4**.

STEP 4: Tuning the Quad lenses for hydrogen

1. Set all of the Quad lens voltages to zero. The major beam current should be about 10 nA.

2. Using the **H2** and **HD** collectors, set up a magnet scan to display the mass 2 and mass 3 beams. They will not be coincident and probably not in focus. An example is shown below.

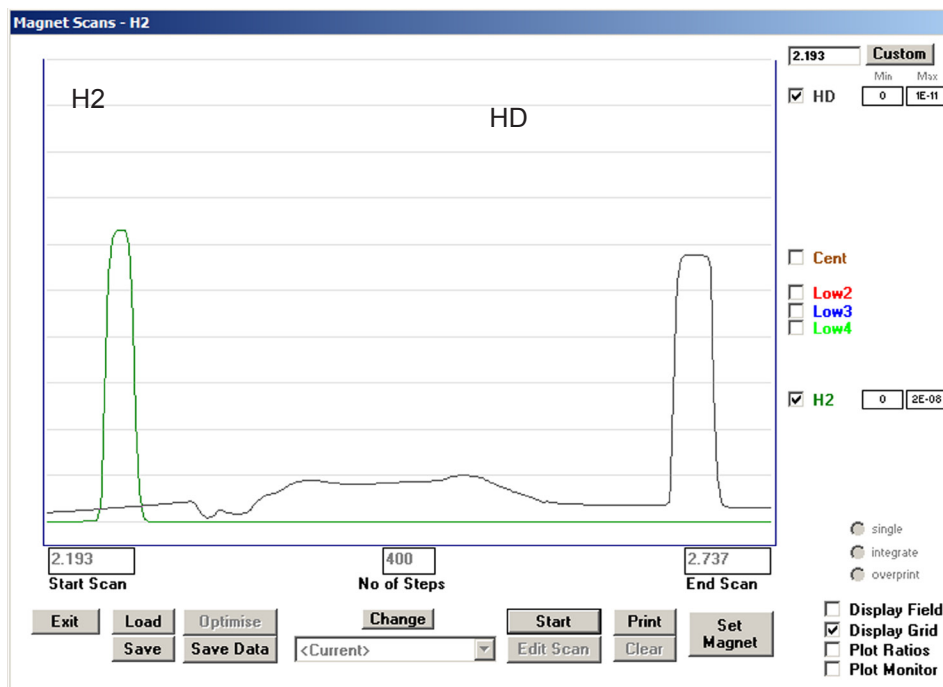


Figure 3.7: Mass 2 and mass 3 beams with the quad voltages set at zero.

3. Use **Q1 I Cor** to focus the **H2** beam and **Q1 O Cor** to focus the **HD** beam.
4. Apply +50 volts to **Deflect 1 O** and **Deflect 1 I**. This will cause the peaks to move towards each other in the display, but will also cause some defocusing. Repeat point 3.
5. Repeat point 4 until the beams are in coincidence and are in the best compromise focus.

This is usually found with different voltages on **Deflect 1 O** and **Deflect 1 I**. Typical settings are approximately 700 V on **Deflect 1 O** and 900 V on **Deflect 1 I**.

The effects of increasing the voltages on **Deflect 1 O** and **Deflect 1 I** are shown in the two figures below, leading to focused coincident beams.

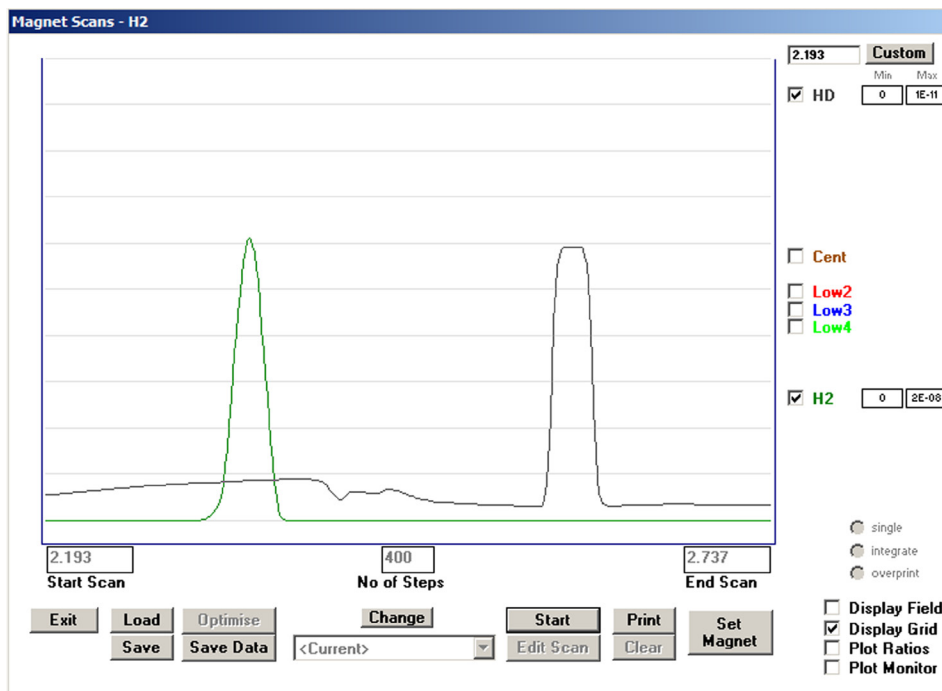


Figure 3.8: Peaks before increasing the voltage on Deflect 1 O and Deflect 1 I.

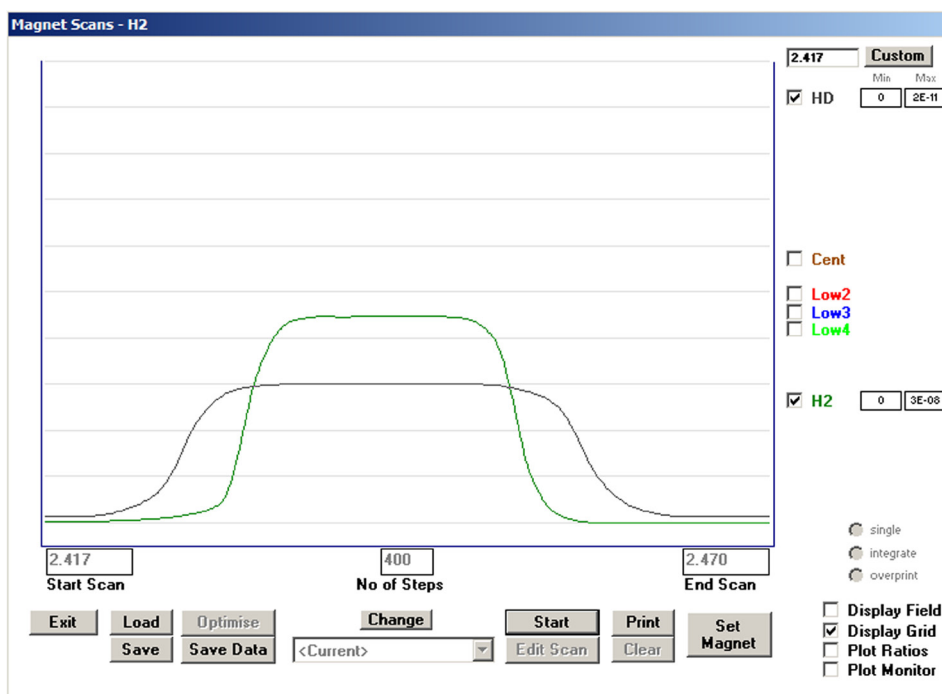


Figure 3.9: Coincident and focused H2 and HD beams.

6. Save the tuning condition, which completes the tuning of the Quad lenses for hydrogen.

Note - If the tuning is a little difficult using only **Q1 I Cor** and **Q1 O Cor**, adjust, by tens of Volts, the values of **Quad1** or **Cubic1**. **Quad2** is normally left at zero when tuning for hydrogen.

STEP 5: Thermal effects in the source

The source has a heater and a temperature sensor to give the user an option to run at approximately 200 °C. The aim is to prevent build up of any contaminant gases that may be introduced into the source along with the sample, or gases that may form from ion-molecule reactions in the source. This facility has proved particularly useful in the analysis of sulphur-containing compounds.

The source will work efficiently without the heater being switched on. In such cases, the normal operating temperature of the source is approximately 100 °C, due to radiative heating from the filament.

If the source is tuned from cold (when it has been off for a considerable time), it takes several hours for the source temperature to reach thermal equilibrium. The tuning is a little sensitive to the relative positions of the source components, which will shift by tiny amounts due to differential thermal expansion. Therefore, while the source temperature is stabilizing, it is normal to see a small drift in beam current. It is worth checking that the tuning is still optimal after the source has been running for several hours.

If a new tuning condition is being set up on an already operating source, these thermal effects are too small to be of any significance.

Task 4: Mass calibration

Aim of this task

This task describes the mass calibration procedure. A known sample will be analyzed and a correlation will be established between the magnet current and the mass of the peaks seen in the resulting spectrum. A mass calibration must be performed whenever the Source HT is changed. Even with a constant Source HT, the mass calibration should be carried out periodically. The mass range used for the mass calibration should correspond to the mass range to be analyzed in subsequent applications.

STEP 1: Mass calibration

HINT: Only perform a mass calibration after the source, magnet and HV supplies have been running for at least 3 hours. This will allow the temperature of the source and Hall probe to stabilize. Small changes in these temperatures can cause shifts in the apparent mass, especially the temperature of the Hall probe.

The mass calibration links the current in the magnet to the mass collected in the **Cent** collector. At a fixed source voltage, the square root of the mass collected in the Cent collector is proportional to the magnetic field. If the Source HT is changed, a new mass calibration will be needed.

The mass range over which the calibration is performed should span the normal working range and should contain as many peaks as possible. Hydrogen is treated separately to the other gases and is dealt with at the end of this section. If the instrument is configured for continuous flow, different reference gases can be introduced into the spectrometer to provide the peaks for mass calibration. These can be supplemented with gases from the background; some useful data is shown in the table below.

Gas	Mass	Comments
Nitrogen	14	From e ⁻ beam decomposition in the source $N_2 \rightarrow 2N$
Water vapour	18	Molecular ion H_2O^+ from background gas
Nitrogen	28	Molecular ion N_2^+ either reference or background gas
Carbon Monoxide	28	From e ⁻ beam decomposition in the source $CO_2^+ \rightarrow CO^+ + O$
Oxygen	32	Molecular ion O_2^+ from the background
Argon	40	Ar^+ from background gas (this is usually small but usable)
Carbon Dioxide	44	Molecular ion CO_2^+ either reference or background gas
Sulphur Dioxide	64	Molecular ion SO_2^+ from reference gas

Table 4.1: Gases that can be used for mass calibration.

Although introducing reference gas for the mass calibration does give large beams to work with, it is easy to perform a mass calibration using only background gases. This is particularly convenient if the instrument is configured for dual inlet mode only. Of course, if sulphur dioxide is not used this can be omitted from the calibration. Whether background or reference gases are used, the methodology is the same:-

- 1 - Find the peaks.
- 2 - Identify the peaks.
- 3 - Measure their position.
- 4 - Enter the data in the Mass Calibration section of the software.
- 5 - Save the new calibration.

The method described below uses background gases, but is easily adapted to use reference gases. A minimum of three data points are required, although six data points would normally be recommended.

HINT: 6 data points are recommended.

Note - if SO₂ needs to be included in the mass calibration, this will not normally be found in the background.

Since the mass calibration method is common to both dual inlet and continuous flow configured instruments, the mass scan can be performed from the background gases. The species that are going to be used are N (mass 14), H₂O (mass 18), N₂ (mass 28), O₂ (mass 32), Ar (mass 40) and CO₂ (mass 44).

1. If the instrument is configured for dual inlet mode only, valves **SC** and **RC** should be closed and **SW** and **RW** should be open.

If the instrument is configured for continuous flow mode only, the main mass spec valve should be closed. It is easier to identify peaks when it is closed.

2. Select a tuning for CO₂ at a suitable source voltage. If one already exists at the correct voltage and a retune is being performed, use this tuning, otherwise use the one closest in voltage to that required.

Since different species are being used, the tuning will not be optimal for them all, but it is the peak positions which are of importance, not the size of the beam.

3. Using only the **Cent** collector, perform a magnet scan that covers the mass range, for example 10 to 50 a.m.u. with 500 steps. If the source voltage has been increased and the old calibration is for a lower voltage, the displayed mass will be an underestimate of the actual mass and the maximum limit of the scan will have to be extended to cover the whole mass range. On the other hand, if the source voltage has been decreased and the old calibration is for a higher voltage, the displayed mass will be an overestimate of the actual mass and the lower limit of the scan will have to be decreased.

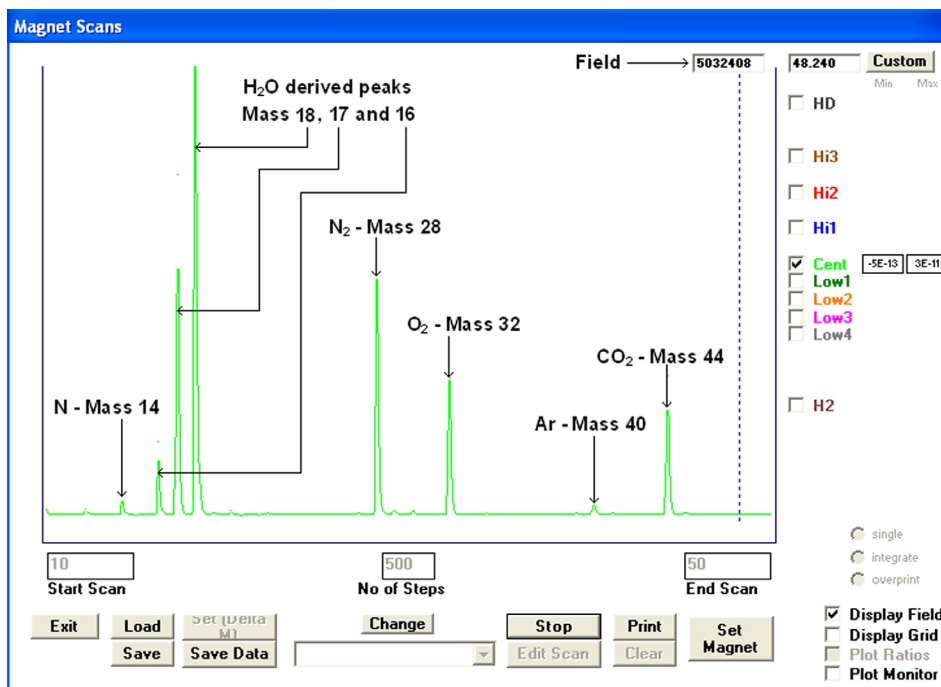


Figure 4.1: Wide magnet scan with background peaks identified.

A typical example of a wide scan over the background peaks is shown in the figure above.

4. Also shown in the bottom right hand corner of the scan window is a box labelled **Display Field**, check the box, as shown in the figure above. A box containing a seven digit number will appear in the top right hand corner of the scan window. The number in this box is proportional to the magnetic field at the current position of the blue dotted cursor that sweeps across display during a magnet scan. The numbers that appear in this box when the cursor is in the middle of each calibration peak are the data required for the mass calibration.

5. To accurately find the field at the centre of each peak, zoom in on each of the peaks in turn so that the scan starts just before the peak and ends just after it. If necessary, change the vertical scale so that the edges of the peak are clearly defined and let the cursor sweep across the screen once so that a clear representation of the peak is visible. On the second sweep, stop the cursor in the centre of the peak by pressing the **Stop** button. The label on the button will change to **Start** and the field setting at the cursor position will be displayed in the box at the top right of the scan window. The example below shows the scan zoomed in on the mass 32 peak, with the cursor stopped on the centre of the peak with a field setting of 4350041.

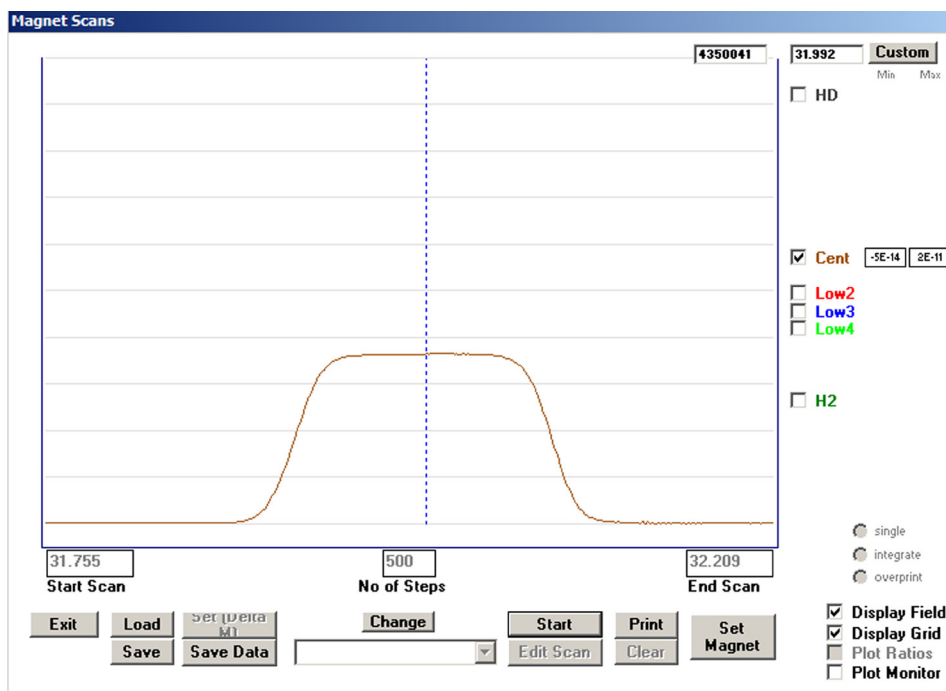


Figure 4.2: A magnet scan on the background mass 32 peak.

Make a note of the field reading and the mass to which it corresponds.

6. Repeat point 5 for all the other peaks in the mass calibration.

7. Once point 6 is complete, a list of field values for each mass used in the calibration should be known and if the calibration was performed at 5, 6 or 8 kV source voltage, they should be similar to, but not exactly the same as, the values in the table below. The values vary from instrument to instrument and with tuning. In practice, these slight variations do not affect the instrument performance.

Mass	Field at 5 kV	Field at 6 kV	Field at 8 kV
14	2128292	2332385	2693628
18	2413932	2646352	3057741
28	3014123	3305102	3819972
32	3222948	3534456	4087086
40	3606780	3955706	4574439
44	3872060	4247356	4913260

Table 4.2: Typical field values at 5, 6 and 8 kV source voltages.

On the main menu bar at the top of the screen, go to **Configure > Mass Calibration**, as shown in the figure below.

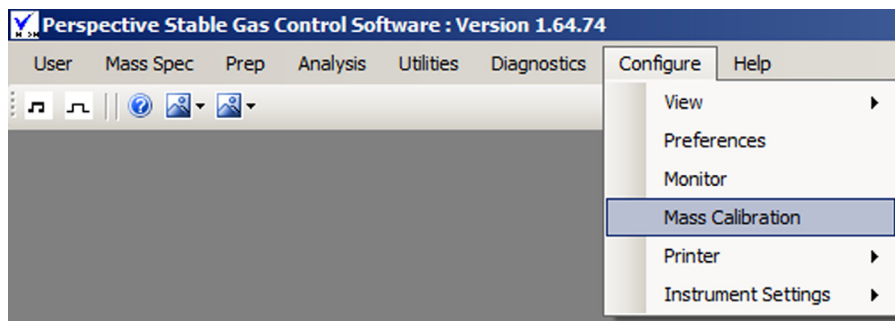


Figure 4.3: Selecting the Mass Calibration.

9. Clicking on **Mass Calibration** brings up the Linear least squares window.

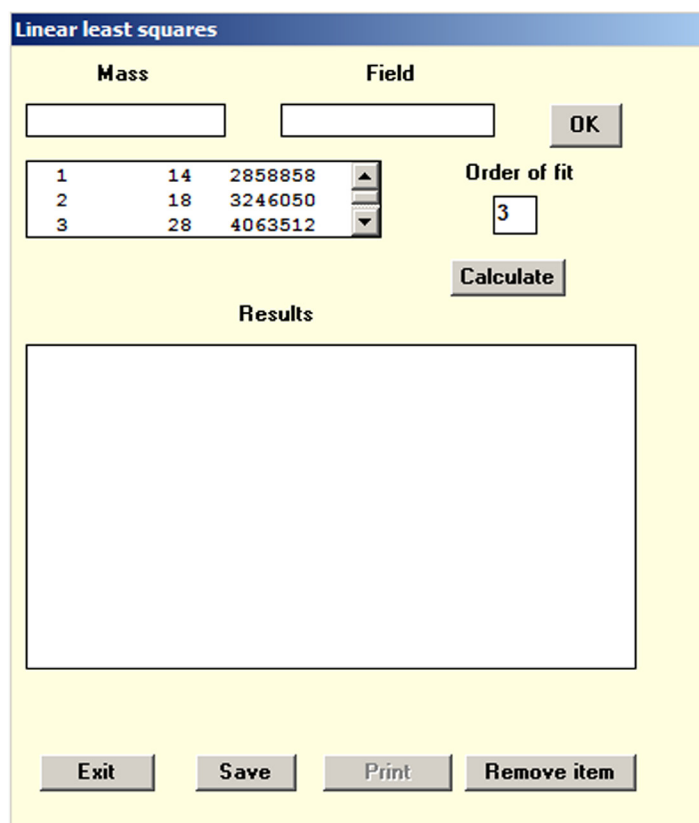


Figure 4.4: The Linear least squares window.

This is the window in which the new data is entered.

10. The data points used in the calibration are labelled with a sequential number. The order in which the data for the different masses are entered doesn't matter. Click **Remove Items** to delete the old mass calibration values, then enter the new values by entering the mass (in a.m.u) and field value. Click **OK** to enter the values.

11. Now select the **Order of fit** by typing a number in the box. The minimum is two, the maximum is 4. An order of fit of 2 uses a quadratic equation of the form $y = ax^2 + bx + c$ and requires a minimum of three data points. An order of fit of 3 uses a cubic equation of the form $y = ax^3 + bx^2 + cx + d$ and requires a minimum of four data points, and so on.

An order of fit of 2 or 3 is usually adequate.

12. Click the **Calculate** button. The data for the new fit will appear in the central window, as shown in the figure below.

Linear least squares

Mass Field

1	14	2858858	▲
2	18	3246050	▬
3	28	4063512	▼

Order of fit

Results

	Coefficient	Error
1	9.663901e+04	1.26e+05
2	7.009971e+05	7.53e+04
3	1.182260e+04	1.47e+04
4	-4.960831e+02	9.33e+02

Standard deviation = 1.26e+03

X	Y	Obs-Calc
1.400000e+01	2.858858e+06	-2.021e+02
1.800000e+01	3.246050e+06	4.098e+02
2.800000e+01	4.063512e+06	1.267e+01
3.200000e+01	4.349760e+06	-8.402e+02

Figure 4.5: Data for the new calibration.

12. Click **Save** to use the new calibration, followed by **Exit**. The new calibration will be saved and the Linear least squares window will close.

HINT: The old calibration is overwritten when the new one is saved. If a new calibration was performed because the source voltage was changed, it may be worth recording the old data as a reference for future use.

Mass Calibration for hydrogen

Because hydrogen does not use the **Cent** collector, the mass scale can not be defined in the same way for hydrogen. The peak center value is set to 2.4 a.m.u., which corresponds to the field value when the peaks are in the H₂ and HD collectors.

Task 5: Testing preamplifier noise and voltage offset

Aim of this task

The aim of this task is to perform the Faraday noise test. This test checks that the Faradays and preamplifiers give an acceptable level of noise and voltage offset.

STEP 1: Setting the parameters for the Faraday noise test

1. In order to perform the Faraday noise test, it is important that there is no ion beam present. The HT must therefore be turned off. Go to the **Digital** window and uncheck source **HT** to turn it off.
2. Close all other windows in the Perspective software. This is important, as otherwise the Faraday noise test will not run.

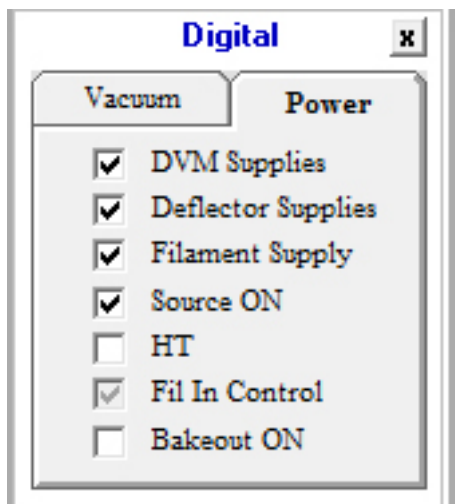


Figure 5.1: Unchecking HT in the Digital window.

3. Go to **Diagnostics > Beam Noise**.

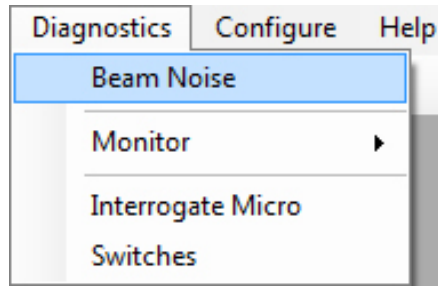


Figure 5.2: Opening the Farady Noise Test window.

4. This will open the **Faraday Noise Test** window.

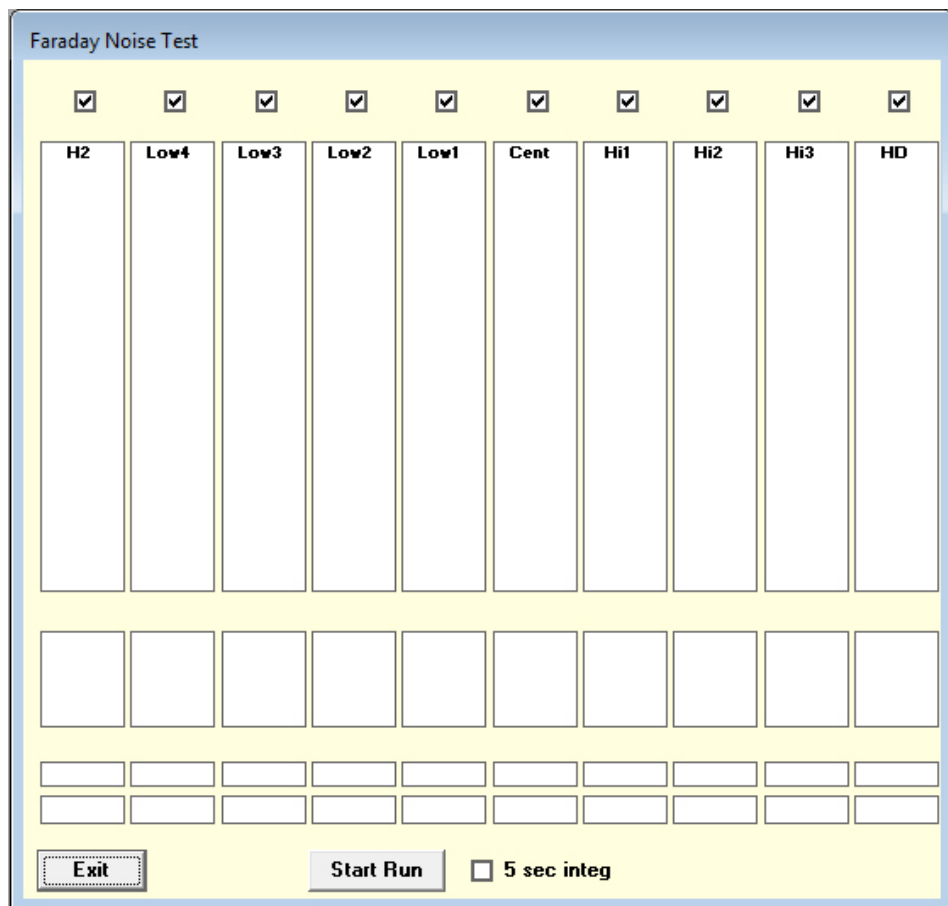


Figure 5.3: Faraday Noise Test window.

STEP 2: Running the preamplifier gain calibration

1. Click on **Start** to initiate the automated software routine. The sequence will now measure the voltage output of the preamplifiers integrated in 1 second intervals.

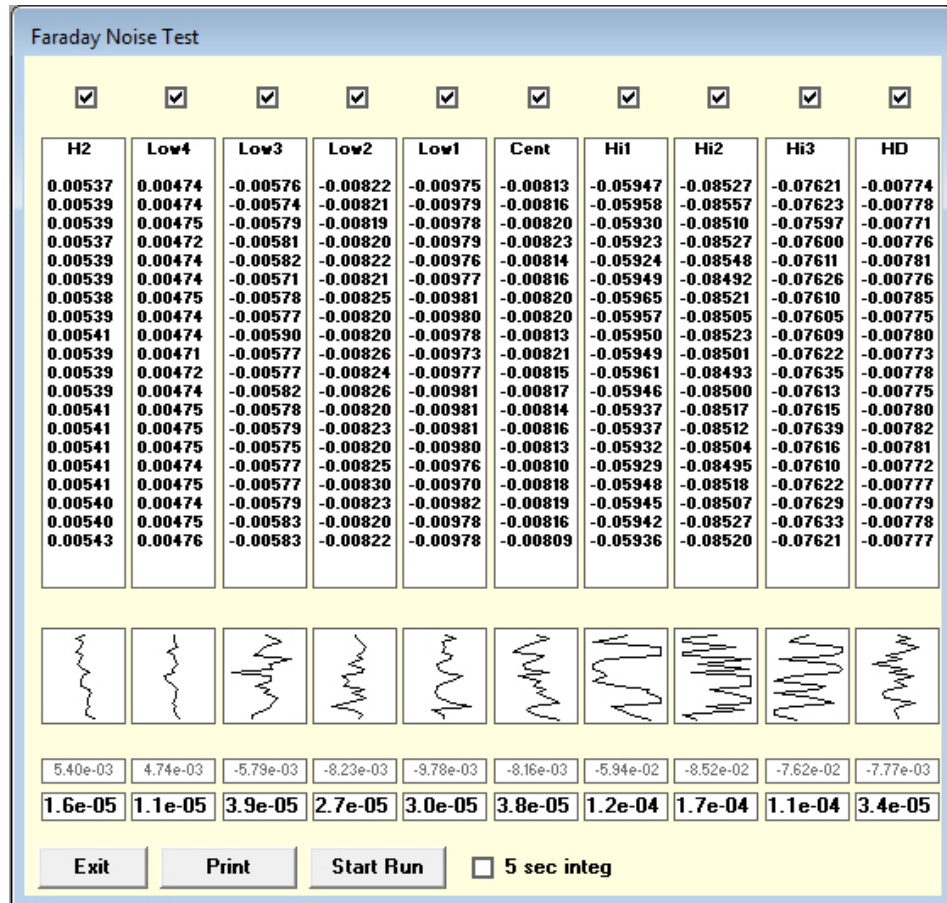


Figure 5.4: Running the Faraday noise test. The numbers at the base of each column show the average voltage offset (in gray) and the standard deviation of the voltage readings (in black).

STEP 3: Interpreting the results

1. Once the sequence is complete, the average voltage (in gray) and standard deviation of the voltage (in black), for each Faraday, is displayed at the bottom of the noise test window. The values should be within the specification shown in the table below.

Test	Conditions	Specification
Faraday Noise (Standard Deviation, V)	All electronics apart from HT ON, 1 second integration time	$HD < 2 \times 10^{-4}$ $Hi3 < 3 \times 10^{-4}$ $Hi2 < 3 \times 10^{-4}$ $Hi1 < 3 \times 10^{-4}$ $Cent < 2 \times 10^{-4}$ $Low2 < 2 \times 10^{-4}$ $Low3 < 2 \times 10^{-4}$ $Low4 < 5 \times 10^{-5}$ $H2 < 5 \times 10^{-5}$
Faraday Voltage Offset (V)	All electronics apart from HT ON, 1 second integration time	$HD < \pm 2 \times 10^{-2}$ $Hi3$ – not specified $Hi2$ – not specified $Hi1$ – not specified $Cent < \pm 2 \times 10^{-2}$ $Low2 < \pm 2 \times 10^{-2}$ $Low3 < \pm 2 \times 10^{-2}$ $Low4 < \pm 5 \times 10^{-3}$ $H2 < \pm 5 \times 10^{-3}$

Table 1: Faraday Noise value specifications.

Task 6: Linearity and H₃⁺ corrections

Aim of this task

This task describes the linearity test, which should be carried out before analyzing any data or before another inlet, such as the Nu Carb, GC or Dual Inlet is connected up to the instrument. Good source linearity ensures that the measured isotope ratio of a gas is not dependent on signal size.

STEP 1: Setup preparation

In all applications, the measured ratio depends, to some extent, on the beam size. In the Perspective, this factor is extremely small and in all cases, apart from hydrogen analysis, no corrections are necessary, as long the source is tuned correctly.

1. Load or set up the tuning for which the linearity correction is to be applied, as described in Task 3.
2. Let some gas into the instrument from either the dual inlet bellows or the reference gas injector, whichever is used for the linearity measurement. Run a magnet scan and stop the cursor on the flat region of the coincident peaks. This is best done on the second scan, since both edges of the peaks can be seen. Exit the magnet scan.
3. Adjust the amount of gas entering the source so that the mass 44 beam current is about 2 nA and note the EPC pressure if using a reference gas injector. If using the dual inlet bellows, the beam should be 2 nA with bellows fully open. Adjust the EPC parameters to obtain mass 44 beam currents in the ranges of 10 to 15, 25 to 30 and 35 to 40 nA. Close the bellows fully (0%) to get the highest beam possible (around 10 times higher than when fully open).
4. To determine the linearity, go to **Mass Spec > Test > Linearity**, as shown in the figure below.

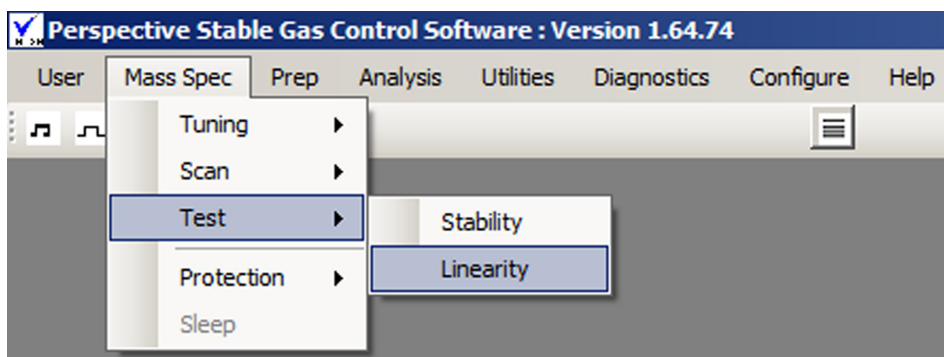


Figure 6.1: Selecting Linearity.

The **Source Linearity Measurement** window will appear. In this example, the software automatically selected CO₂, because it was the last linearity test to be run.

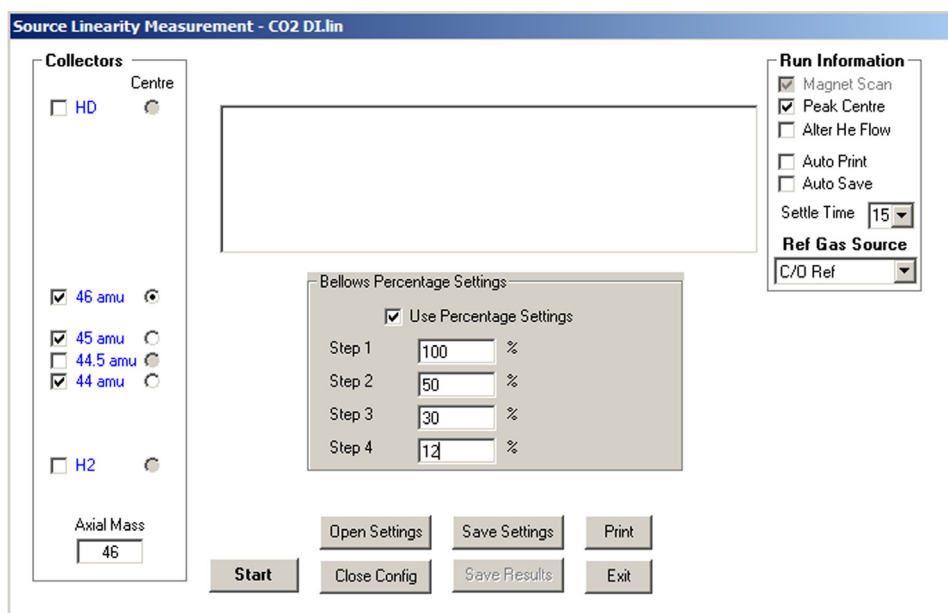


Figure 6.2: The source Linearity Measurement window.

5. Edit the settings as necessary. The settings that appear are those which were last used. These can be changed by either editing the parameters that are already there or by loading the settings that were saved earlier. To use previously saved settings, click **Open Settings**, select the required file and click **Open**.

If the configuration window **Bellows Percentage Settings** or **EPC Settings** are not visible, the button that should be marked **Close Config** will read **Open Config**. Click this and the window will appear.

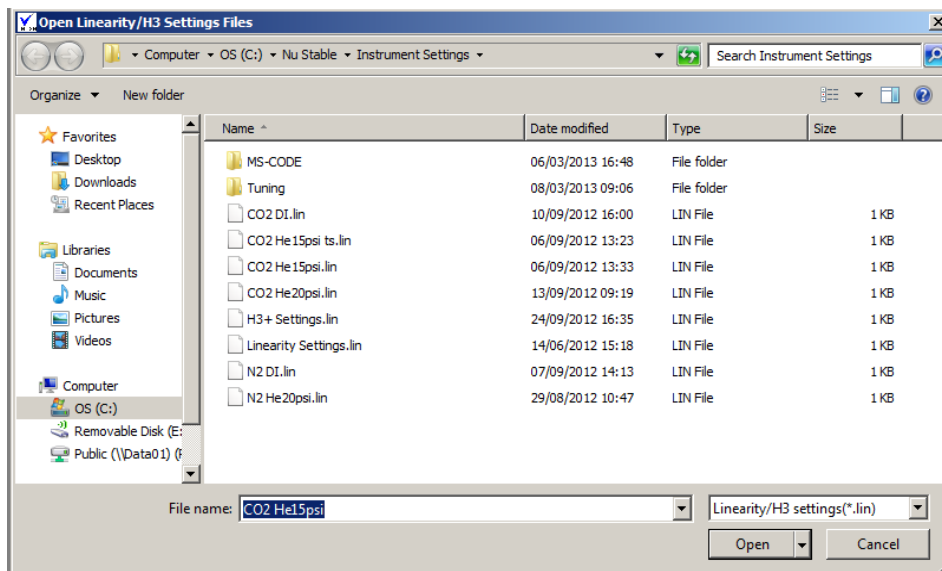


Figure 6.3: The Open Linearity / H₃⁺ Settings File window.

If the parameters are to be edited, firstly open the **Ref Gas Source** drop down menu in the top right hand panel of the **Source Linearity Measurement** window. All of the available choices will be shown, as illustrated below.

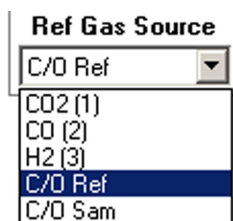


Figure 6.4: Reference gas sources.

C/O Ref and **C/O Sam** refer to the reference and sample bellows of a dual inlet, the others are continuous flow reference gas injectors. Click on the appropriate option to select it.

6. If either **C/O Ref** or **C/O Sam** were selected in Point 5, the configuration window will request input of **% bellows settings**. If one of the reference gas injectors was chosen, **EPC Pressures** will be requested. Enter the settings recorded at point 3 into the configuration window. The order of entry does not matter.

7. Edit the collectors to be used by clicking in the boxes on the left hand side of the **Source Linearity Measurement** window. The last collector selected will automatically be the one that is used for a peak centre. Any other can be selected instead in the column headed **Centre** to the right of the collector labels.

8. Enter the axial mass, the mass of the isotope collected in the **Cent** collector, in the box in the bottom left of the **Source Linearity Measurement** window.

9. Checking **Peak Centre** will perform a peak centre before the linearity run, using the collector selected in Point 7. If this box is not checked, a peak centre will not be performed.

The **Alter He Flow** option only refers to continuous flow. Instead of changing the reference gas pressure to change the beam size, checking this box causes the helium carrier pressure to be changed. The values in the configuration panel should now refer to the helium carrier pressure. This method is not commonly used.

The **Auto Print** and **Auto Save** buttons are self explanatory.

The **Settle Time** is the delay between a change in bellows setting or a change in EPC setting and the start of data collection. This allows beams to stabilise before any data is collected. The number in the box is the time in seconds.

10. Click **Start** and if a peak centre is required, a small window, shown below, will appear. If a peak centre is not required, the linearity routine continues directly to Point 11. The **Start** button will revert to the **Abort** button.

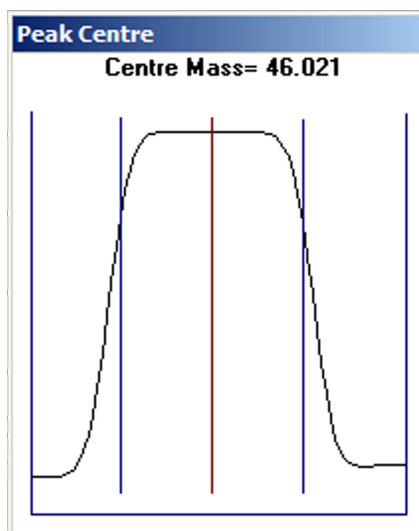


Figure 6.5: The peak centre window.

On completion of the peak centre routine, the magnet position will reset to the mass indicated at the top of the panel. This coincides with the dark vertical line at the centre of the peak. The linearity routine then automatically proceeds to the next point.

11. Before measuring ratios, the beam currents are first measured with the reference gas valve closed. These background values are later deducted from the measured beams before ratios are calculated. The linearity routine then measures the beam ratios using the data entered into the panel at points 4 to 8. The size of the major beam and the ratios are displayed as the measurement for each setting is completed. When the routine is complete a graph and the numeric results are plotted, as shown in the figure below.

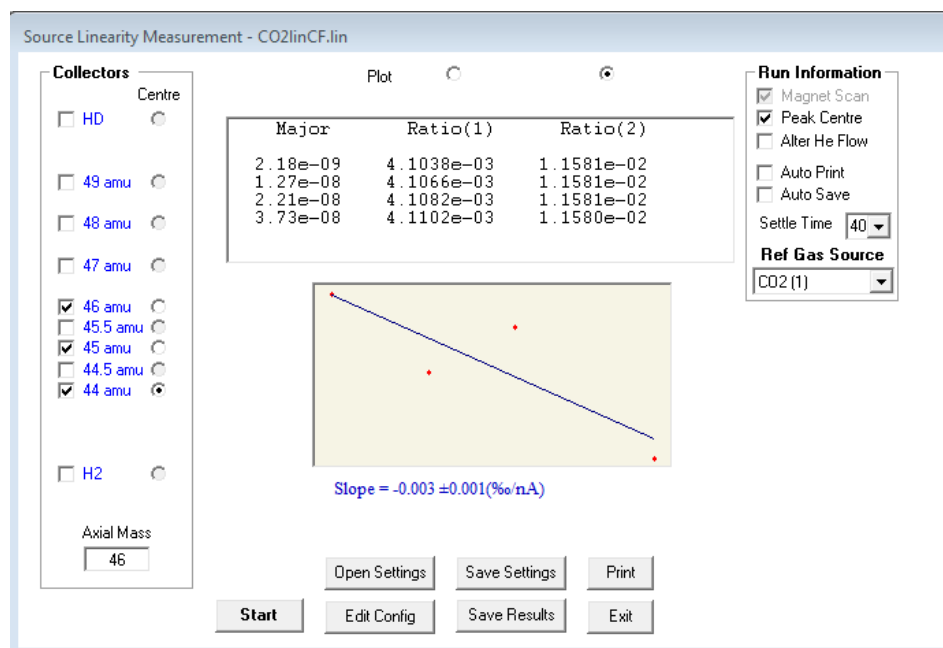


Figure 6.6: A completed linearity using CO₂.

The linearity is the reported slope. **Ratio (1)** corresponds to the mass 46/44 ratio and **Ratio (2)** corresponds to the mass 45/44 ratio. The displayed plot can be selected by clicking in the **Plot** button for the desired ratio.

12. Compare the results with the instrument specifications as listed below.

Perspective Specifications		
Linearity		
Type	Conditions	Specification
CO2 45/44 (C13)	2 to 40 nA Mass 44 beam	Linearity < 0.020 ‰/nA Error < ±0.010‰
CO2 46/44 (O18)	5 measurement average	Linearity < 0.020 ‰/nA Error < ±0.010‰
N2 29/28 (N15)	2 to 40 nA Mass 28 beam 5 measurement average	Linearity < 0.020 ‰/nA Error < ±0.010‰
CO 29/28 (C13)	2 to 40 nA Mass 28 beam 5 measurement average	Linearity < 0.030 ‰/nA Error < ±0.010‰
CO 30/28 (O18)		Linearity < 0.030 ‰/nA Error < ±0.010‰
H3+ correction		
Test	Conditions	Specification
H3+ correction	2 to 30nA Mass 2 beam 5 measurement average	H3+ correction < 10 ppm/ nA Error < ±0.050 ppm/nA

Table 6.1: Specifications

13. Click **Save Results** to store the linearity data. This will overwrite any previous linearity and will be automatically applied if this option is set in the **Configure/Preferences** window. This is accessed by clicking **Configure** on the main menu bar at the top of the screen; this will reveal the drop down menu shown below.

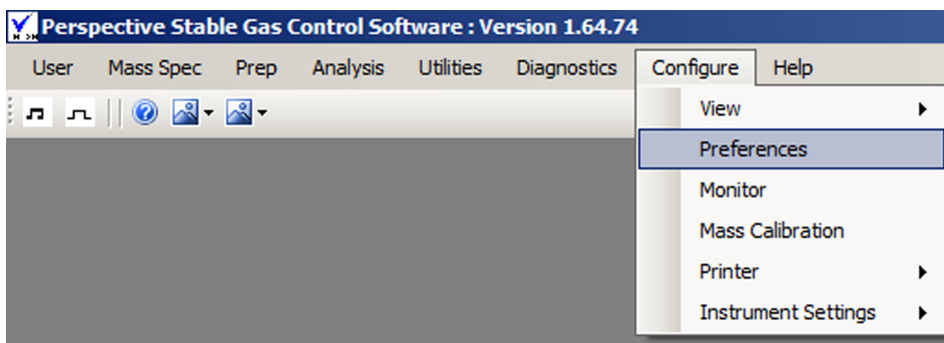


Figure 6.7: The Configure drop down menu.

Selecting **Preferences** will show the **Setup** window shown below.

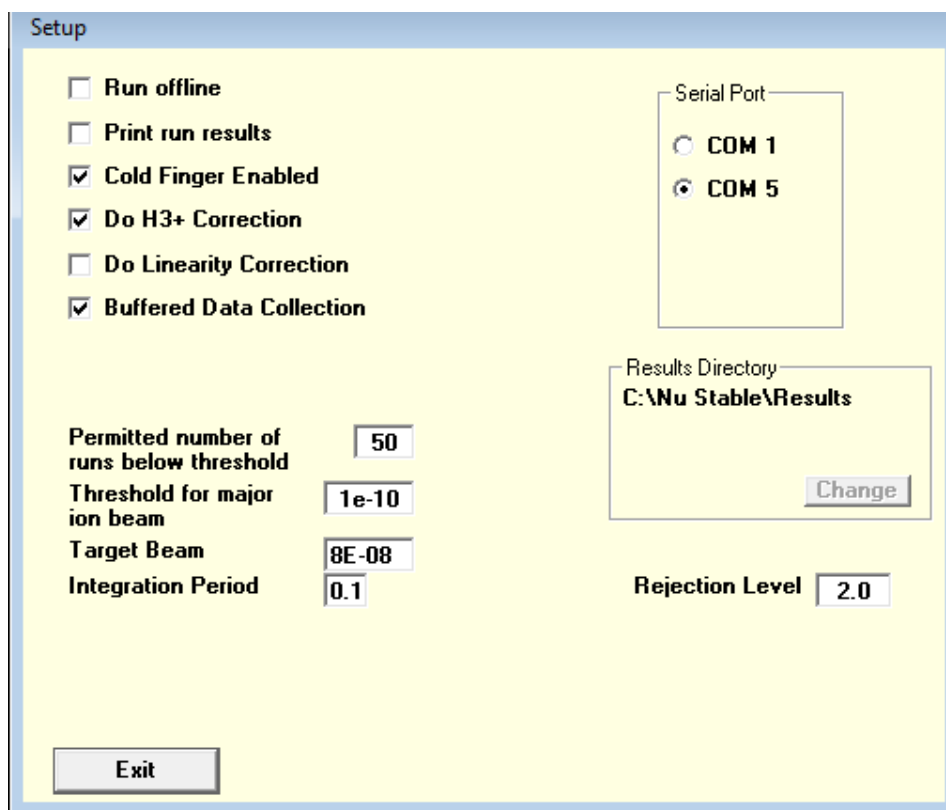


Figure 6.8: The Setup window.

Checking **Do H3+ Correction** and **Do Linearity Correction** will automatically apply the last stored linearity correction to any acquired data. It is recommended that the H3+ correction is always applied, whereas the linearity correction does not normally need to be applied.

Task 7: Stability test

Aim of this task

This task describes the continuous flow stability test, which should be run periodically to check the instrument's stability compared to the performance specifications. This test can not be used to check the dual inlet stability.

STEP 1: Setting the signal for the stability test

1. Before running the stability test, it is necessary to set the beam height required to run the stability test. This is set in the **Gas Control** window. Refer to **Task 1: Manually introducing the reference gas** for a detailed description.

Below is a brief summary of the steps to take:

- a. Open the **Magnet Scans** window and scan over CO₂ at mass 46 on the **Cent** collector.
- b. Once scanned, stop the scan and move the cursor to the peak center.
- c. By observing the **Monitor** window, set the beam height to a suitable value. For the stability test, 15 nA is recommended. This is set in the **Gas Control** window by changing the **EPC** value. Adjust this value until a 15 nA beam is achieved.

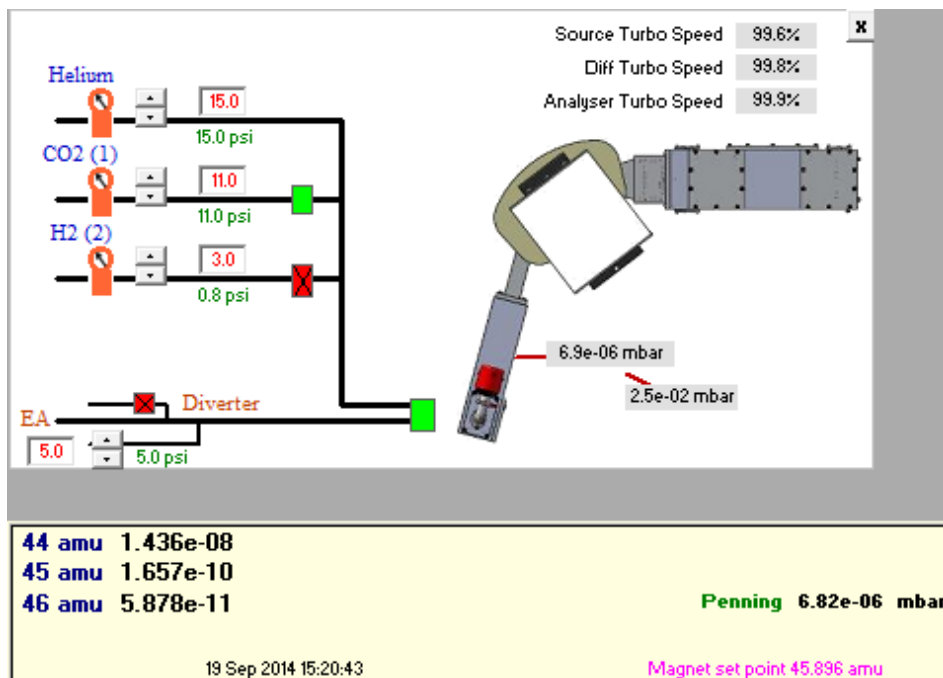
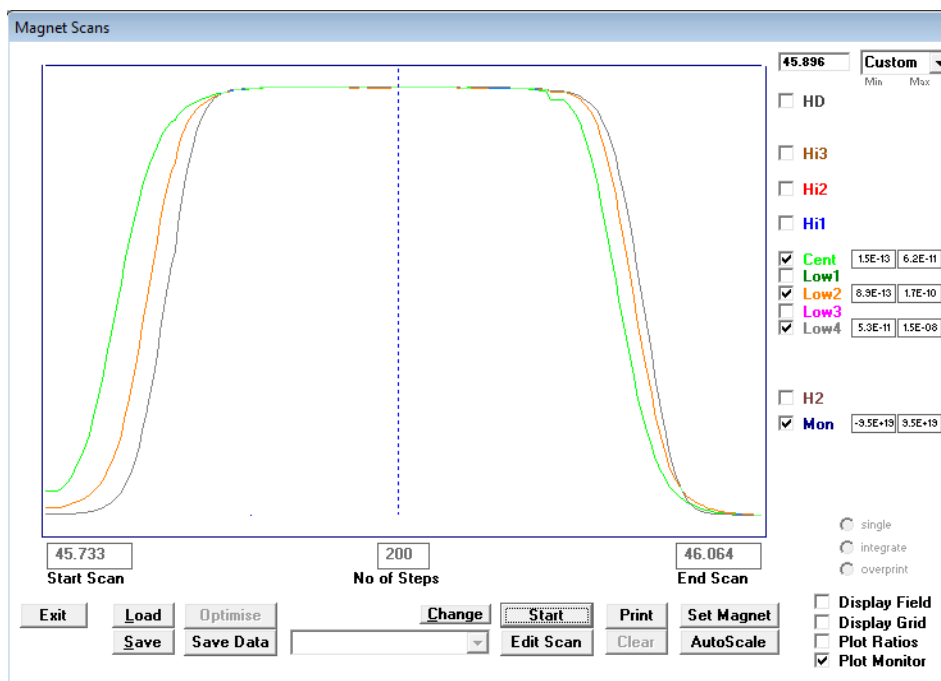


Figure 7.1: Setting the signal.

Figure 7.2: Scanning over CO₂ in the Magnet Scans window.

d. Exit the **Magnet Scans** window.

STEP 2: Opening the stability test

1. Ensure that the **EA** is selected in the **Prep** tab, in order to run the stability test. Go to **Prep > EA**.

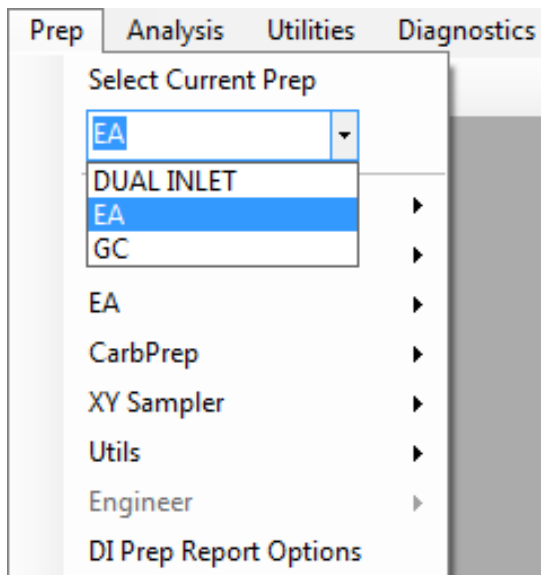


Figure 7.3: Selecting the EA prep system.

2. Go to **Mass Spec > Test > Stability** to open the stability test.

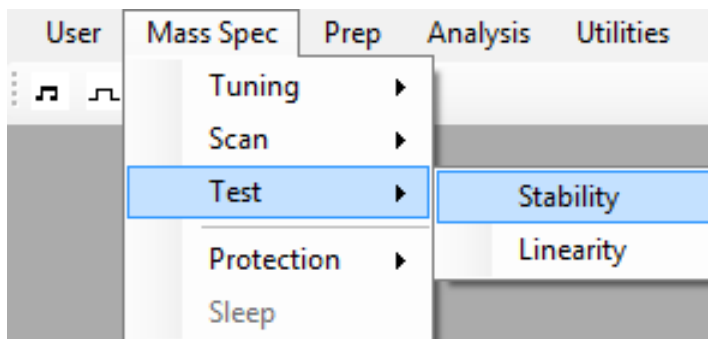


Figure 7.4: Opening the stability test.

3. In the **Continuous Flow Analysis Run Setup** window, click on **Select File** to open the **Nu Stable>Analyses** folder. There will be a pre-written stability file for each gas species. In this example, select **<CO2 Ref x10.rdf>**. Click on **Open**. The selected file will be now be displayed in the Continuous Flow Analysis Run Setup window.
4. Place a checkmark next to **Peak Centre before Analysis** in the Continuous Flow Analysis Run Setup window.

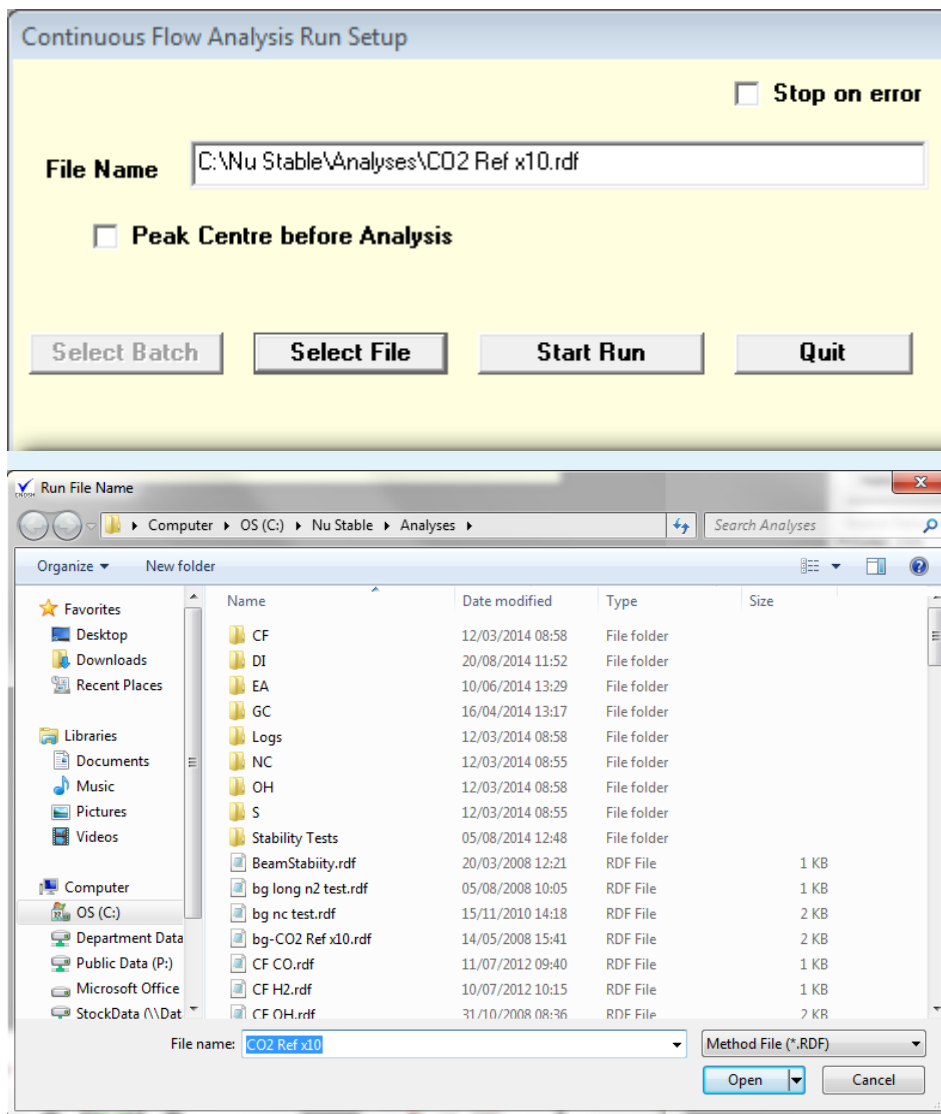


Figure 7.5: Selecting the file for the stability test.

STEP 3: Running the stability test

1. Click on **Start Run**. The **Data Acquisition** window will then open. Type the name of the test into the bar at the bottom of the window and then press **Start** to begin the stability test.

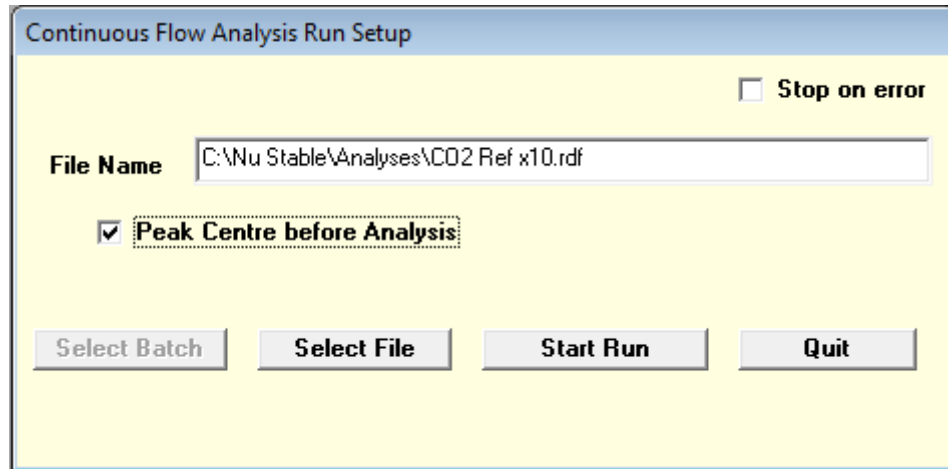


Figure 7.6: Initiating the stability test.

2. The **Data Acquisition** window for the stability test opens.

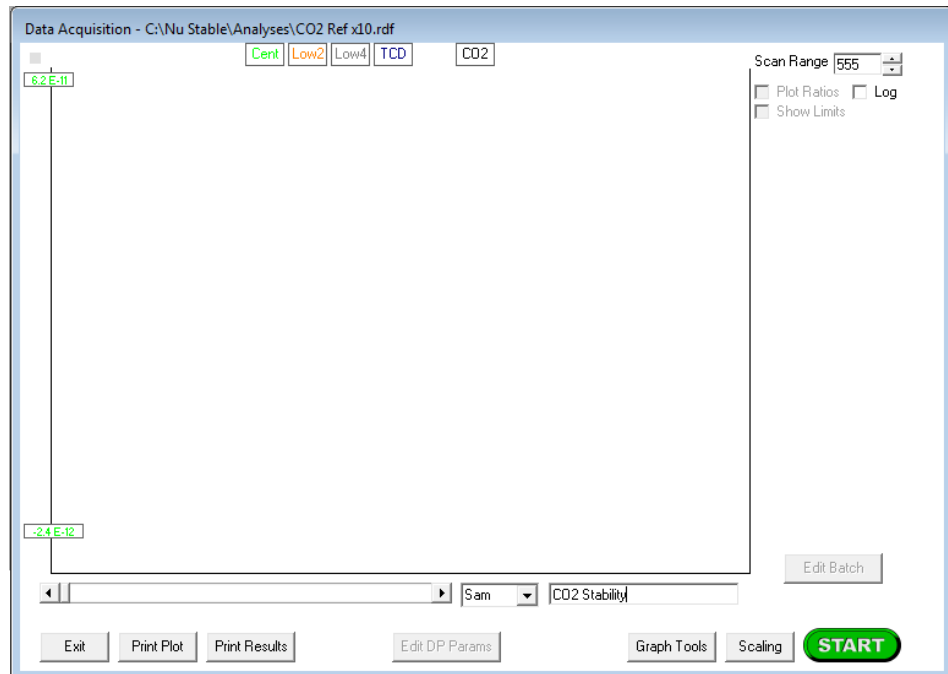


Figure 7.7: Starting the stability test.

3. The stability test will firstly measure the background signals with the reference valve shut, then it opens and closes the reference gas valve 10 times. When the test is completed, the ratio for each peak is measured and the standard deviation and the fit of the peaks are calculated.

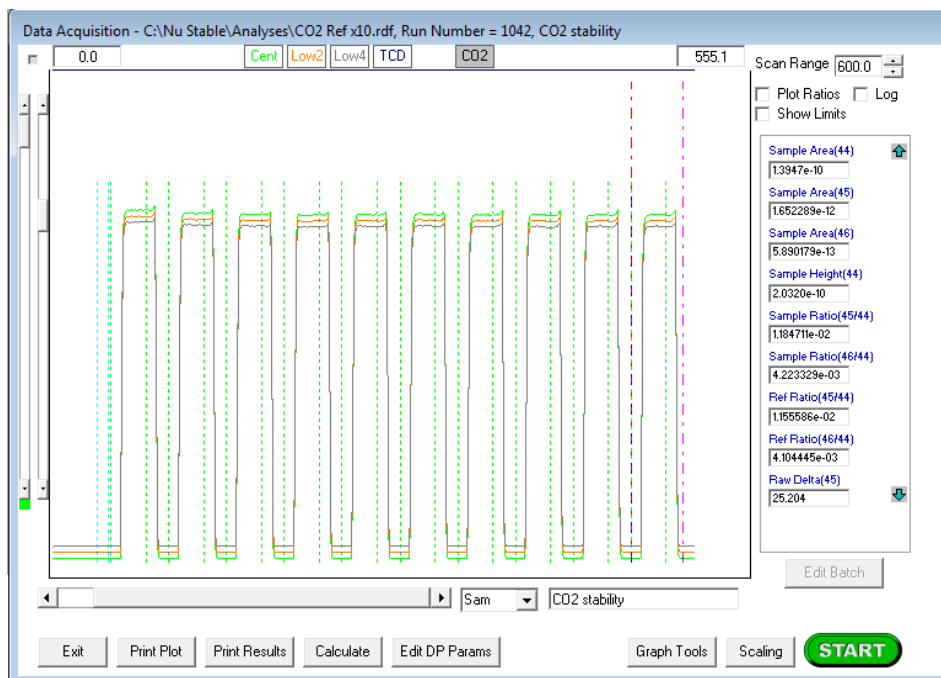


Figure 7.8: The completed stability test.

STEP 4: Interpreting the results

1. Once the stability test is completed, the results can be viewed to verify that performance is as expected. Go to **Prep > Utils > Explore Results**.

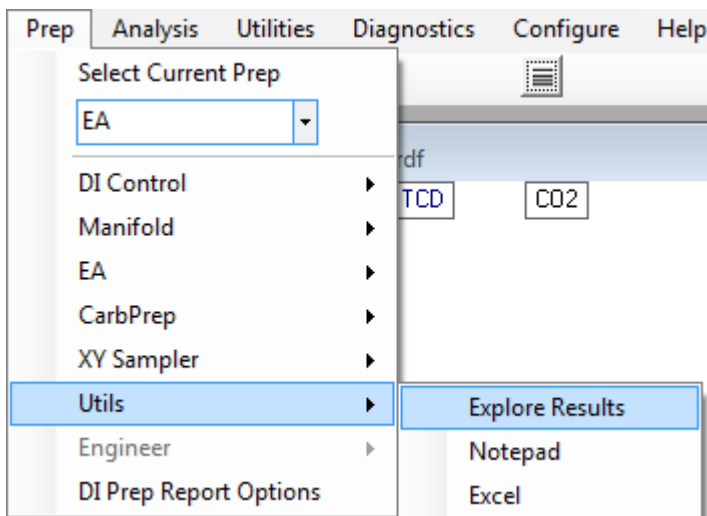


Figure 7.9: Opening the Results folder.

2. The **Nu Stable > Results** folder opens. Scroll down to locate the **Single Run** folder and double click on it to open it. Select the most recent stability report.

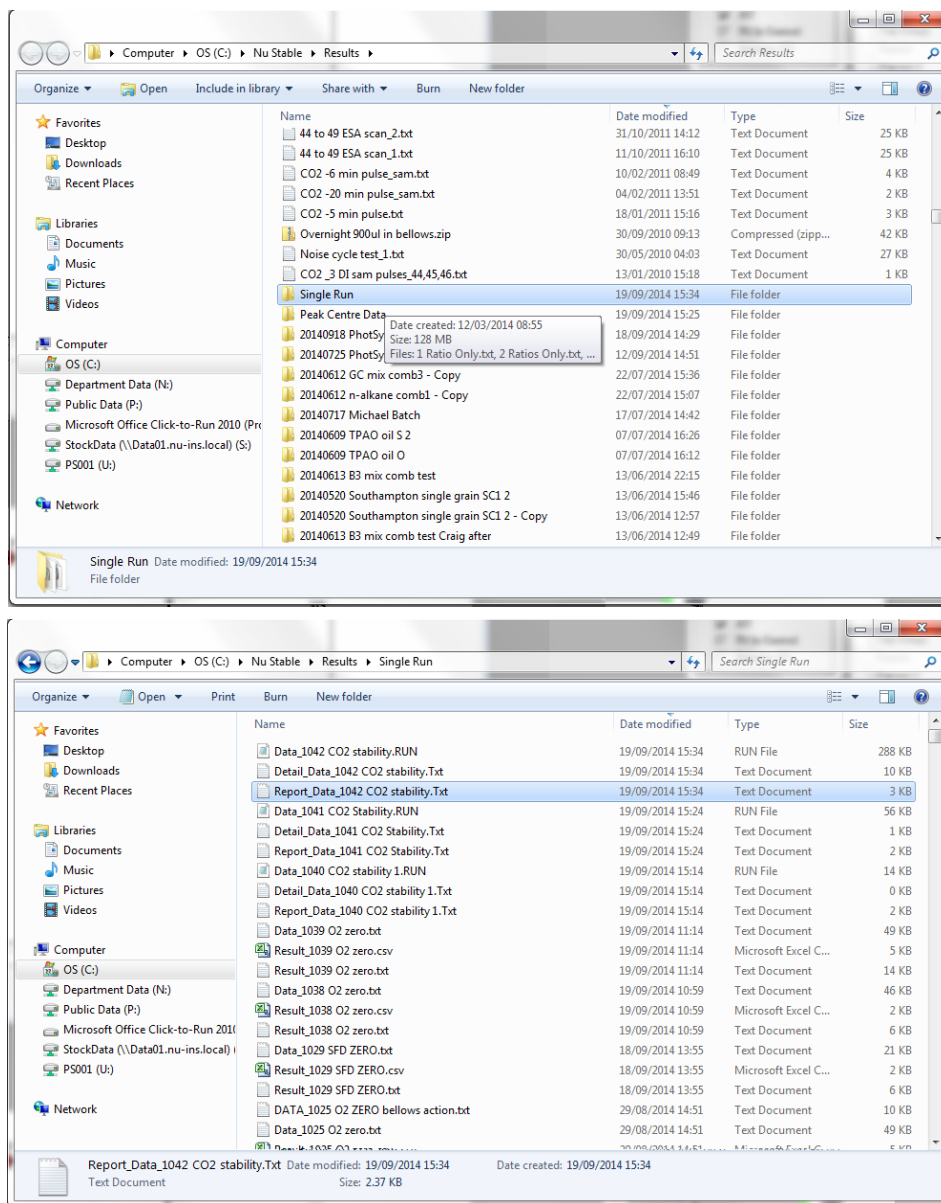


Figure 7.10: Opening the stability results.

3. The results of the stability test are now displayed. Compare the results achieved with the instrument specifications.

Observing the results, the fit and the range are of greatest interest. For example, for CO₂, the fit and the range of the 45/44 ratio and 46/44 ratio must meet the specification.

The fit is the standard deviation of the ratio in per mil (‰) notation. The standard deviation should be <0.06 ‰ for CO₂.
The range is the difference between the smallest and largest value and should be less than 0.2 ‰ for CO₂.

```

Report_Data_1042 CO2 stability.Txt - Notepad
Nu Stable Isotope Analysis Program

Data acquired on 19 September 2014 at 15:25 with stable 1.68.22 on PS001
Reanalyzed on 19 September 2014 at 15:34 with stable 1.68.22 on PS001

Run file : CO2 Ref x10
Sample   : CO2 stability

Raw data is stored in file: C:\Nu Stable\Results\Single Run\Data_1042 CO2 stability.RUN

Reference Gas Peaks

Time (s)   Major      Ratio 45/44  Ratio 46/44
-----
78.0       1.4545E-8    1.15644E-2   4.10665E-3
128.1      1.4408E-8    1.15641E-2   4.10647E-3
178.0      1.4369E-8    1.15644E-2   4.10640E-3
228.1      1.4346E-8    1.15644E-2   4.10642E-3
278.1      1.4350E-8    1.15644E-2   4.10642E-3
328.1      1.4362E-8    1.15642E-2   4.10643E-3
378.1      1.4352E-8    1.15640E-2   4.10648E-3
428.1      1.4326E-8    1.15640E-2   4.10645E-3
478.1      1.4349E-8    1.15640E-2   4.10630E-3
528.1      1.4348E-8    1.15636E-2   4.10642E-3

Average    1.15641E-2   4.10644E-3
Stdev      2.7E-7       8.7E-8
Fit        0.024        0.021
Range      0.073        0.085

Sample Area(44) = 1.3947e-10
Sample Area(45) = 1.652289e-12
Sample Area(46) = 5.890179e-13
Sample Height(44) = 2.0320e-10
Sample Ratio(45/44) = 1.184711e-02
Sample Ratio(46/44) = 4.223329e-03
Ref Ratio(45/44) = 1.155586e-02
Ref Ratio(46/44) = 4.104445e-03
Raw Delta(45) = 25.204
Raw Delta(46) = 28.965
Delta(13) wrt PDB = -13.97
Delta(18) wrt PDB = 3.27
  
```

Figure 7.11: Viewing the results.

Test	Conditions	Specification
CO ₂ 45/44 (¹³ C)	Standard Deviation 45/44 ratio in 10 CO ₂ pulses Mass 44 beam at 15nA	SD < 0.060 ‰ Range <0.2
CO ₂ 46/44 (¹⁸ O)	Standard Deviation 46/44 ratio in 10 CO ₂ pulses Mass 44 beam at 15nA	SD < 0.060 ‰ Range <0.2
N ₂ 29/28 (¹⁵ N)	Standard Deviation 29/28 ratio in 10 N ₂ pulses Mass 28 beam at 15nA	SD < 0.060 ‰ Range <0.2
CO 29/28 (¹³ C)	Standard Deviation 29/28 ratio in 10 CO pulses Mass 28 beam at 15nA	SD < 0.150 ‰ Range <1.0
CO 30/28 (¹⁸ O)	Standard Deviation 30/28 ratio in 10 CO pulses Mass 28 beam at 15nA	SD < 0.150 ‰ Range <1.0
H ₂ 3/2 (HD)	Standard Deviation 3/2 ratio in 10 H ₂ pulses Mass 2 beam at 15nA	SD < 0.500 ‰ Range <2.0

Table 7.1: Stability specifications.

3. Comparing the results to the specifications given above, the results achieved are well within the specifications.

The fit was 0.024 ‰ for the 45/44 ratio and 0.021‰ for the 46/44 ratio, both well below the 0.06 ‰ specification.

The range was 0.073 ‰ for the 45/44 ratio and 0.085 ‰ for the 46/44 ratio, again both well below the 0.2 ‰ specification.

The stability test was successful and the instrument is ready to run analyses.

Task 8: Data acquisition setup menus

Aim of this task

This short task describes how the prep system is selected. Data acquisition is described in the individual Prep System Getting Started Guides.

The setup of data acquisition is specific to the sample preparation system. To access the correct setup menu, click **Prep** on the main menu bar then click the down arrow just beneath **Select Current Prep**.

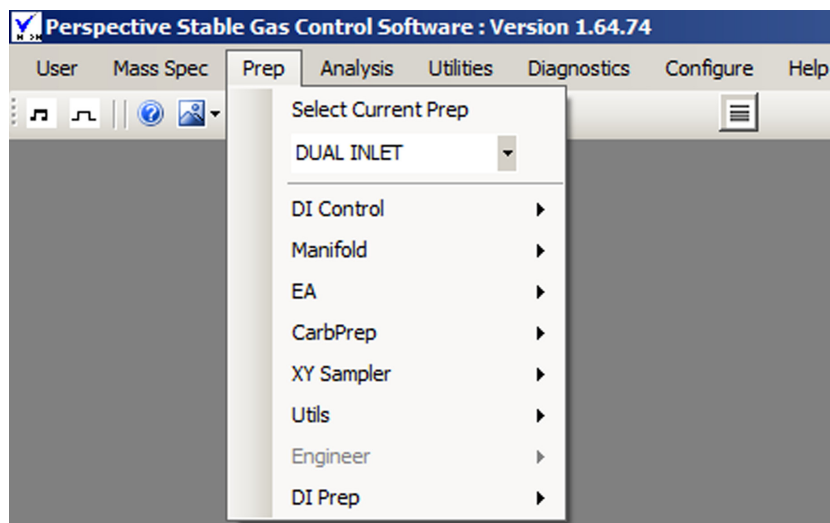


Figure 8.1: The Prep dialog box.

The prep system options will appear in a drop down menu, as shown in the example below.

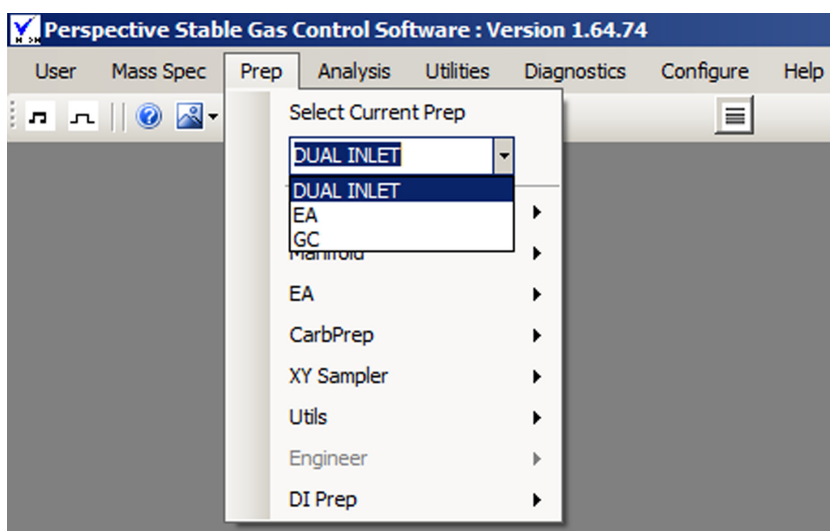


Figure 8.2: The Preparation System options.

Select the appropriate preparation system.

Task 9: Software sample calibration

Aim of this task

This task describes how to enter the isotopic and elemental compositions of reference standards and gases to give software calibration of measured samples.

STEP 1: Opening the Reference Data Input Form - Reference gas

1. To open the **Reference Data Input Form**, go to **Utilities > Calibrations > Reference Gas Values**.

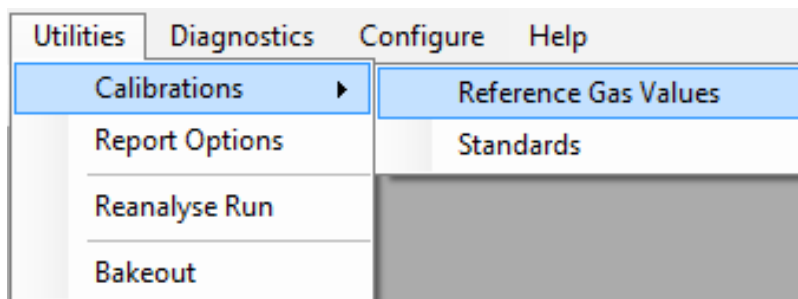
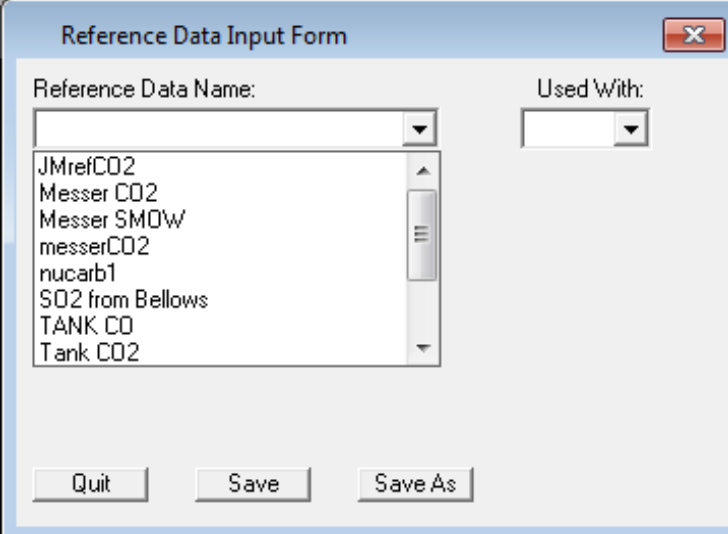


Figure 9.1: Opening the Reference Data Input Form.

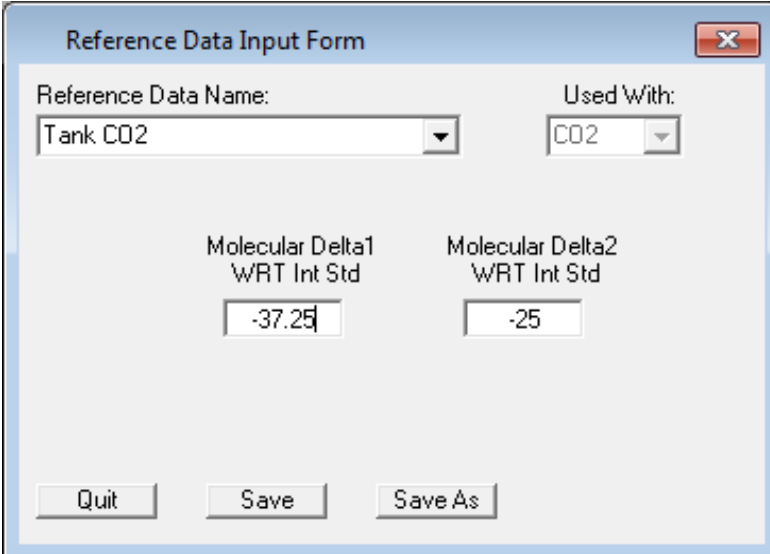
2. The **Reference Data Input Form** opens. Select the reference gas from the **Reference Data Name** dropdown list or enter a new name for the reference gas.



The image shows a software window titled "Reference Data Input Form". It contains two dropdown menus: "Reference Data Name:" and "Used With:". The "Reference Data Name:" dropdown is open, showing a list of options: JMrefCO2, Messer CO2, Messer SMOw, messerCO2, nucarb1, SO2 from Bellows, TANK CO, and Tank CO2. The "Used With:" dropdown is empty. At the bottom of the window are three buttons: "Quit", "Save", and "Save As".

Figure 9.3: Empty Reference Data Input Form upon opening.

3. Update the values if the reference gas has been changed or if a new reference gas bottle is being used. The values need to be measured with respect to known isotopic standards. Enter them and click **Save**. If CO₂ is selected, both delta values need to be with respect to PDB. SMOw can not be used for the oxygen value.



The image shows the same "Reference Data Input Form" window, but now it is filled with data. The "Reference Data Name:" dropdown is set to "Tank CO2" and the "Used With:" dropdown is set to "CO2". Below these are two input fields for "Molecular Delta1 WRT Int Std" and "Molecular Delta2 WRT Int Std". The first field contains the value "-37.25" and the second field contains the value "-25". The "Quit", "Save", and "Save As" buttons are still present at the bottom.

Figure 9.2: Values entered into the Reference Data Input Form.

STEP 2: Opening the Reference Data Input Form - Standards

1. To open the Reference Data Input form, go to **Utilities > Calibrations > Standards**.

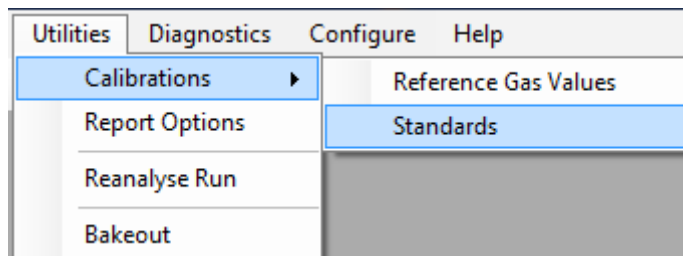


Figure 9.4: Opening the Standards Data Input Form.

2. The **Reference Data Input Form** opens. Select the standard from the dropdown list or enter a new name for the standard.

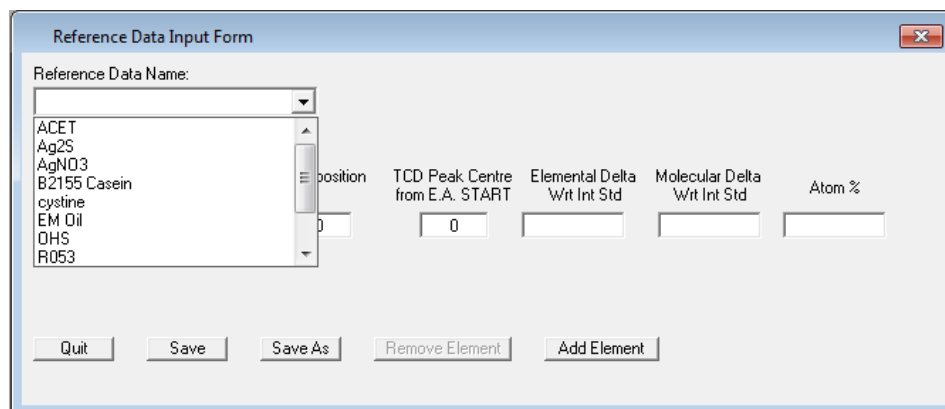
A screenshot of the 'Reference Data Input Form' dialog box. It features a 'Reference Data Name:' dropdown menu with a list of standards: ACET, Ag2S, AgNO3, B2155 Casein, cystine, EM Oil, DHS, and R053. To the right of the list are five input fields: 'Position' (with a value of 0), 'TCD Peak Centre from E.A. START' (with a value of 0), 'Elemental Delta W/it Int Std', 'Molecular Delta W/it Int Std', and 'Atom %'. At the bottom of the dialog are five buttons: 'Quit', 'Save', 'Save As', 'Remove Element', and 'Add Element'.

Figure 9.5: Opening the Standards Reference Data Input Form.

3. Enter the element of interest (either C, N, S, O or H).

To obtain elemental compositions of samples using the thermal conductivity detector (TCD) data obtained from the EA, enter the elemental composition of the standard. The time taken for the peak to be detected by the TCD after the EA start signal must be entered into the **TCD Peak Centre from EA START** box.

The elemental delta of the standard with respect to international standard and molecular delta of the standard with respect to international standard can be entered into the next two boxes.

Elemental Delta wrt Int Std is, for example, the isotopic delta ^{13}C value relative to PDB standard, or delta ^{18}O relative to PDB standard (PDB is an international isotopic standard).

The **Molecular Delta wrt Int Std** applies to CO_2 and SO_2 only. It is the raw delta value (e.g. $\delta 45$, $\delta 46$) with respect to international standard before the Craig / 'iterative' correction for ^{17}O is applied.

The screenshot shows a software window titled "Reference Data Input Form". At the top, there is a dropdown menu for "Reference Data Name" with "B2155 Casein" selected. Below this is a table with columns: Element, % Composition, TCD Peak Centre from E.A. START, Elemental Delta Wrt Int Std, Molecular Delta Wrt Int Std, and Atom %. The table contains three rows of data for elements S, C, and N. At the bottom of the window, there are buttons for "Quit", "Save", "Save As", "Remove Element", and "Add Element". A "Deconvolute" button is also present to the right of the table.

	Element	% Composition	TCD Peak Centre from E.A. START	Elemental Delta Wrt Int Std	Molecular Delta Wrt Int Std	Atom %
Element 1	S	0.751	170	6.32	0	0
Element 2	C	46.5	85	-26.98	0	0
Element 3	N	13.32	245	5.94	0	0

Figure 9.6: Values entered for the Standards Reference Data Input Form.



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