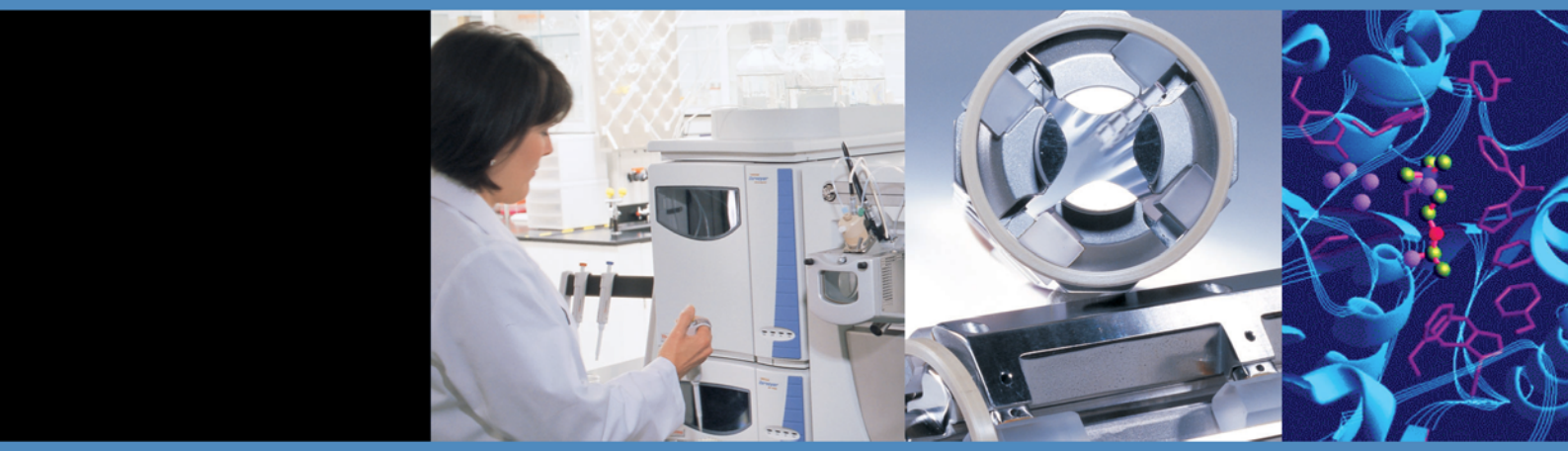
XSERIES 2 ICP-MS

Getting Started Guide

Revision B - 121 9590



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ThermoFisher SCIENTIFIC

XSERIES 2 ICP-MS

Getting Started Guide

Revision B - 121 9590

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EMC (emissioni)

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Bremen, Germany, 11. Dezember 2006

ThermoFisher
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Director of Operations
Direttore fabrizione

Jörg Behrens

Read This First

Welcome to the Thermo Fisher Scientific, XSERIES 2 system! The XSERIES 2 is a member of the Thermo Scientific family of advanced mass spectrometer (MS) detectors.

Who uses this Guide

This *XSERIES 2 Getting Started Guide* is intended for all personnel that needs to operate the XSERIES 2, especially the key operator. This manual should be kept near the instrument to be available for quick reference.

Scope of this Guide

This *XSERIES 2 Getting Started Guide* provides you with information on how to set up, calibrate, and tune the XSERIES 2. In addition, this manual provides step-by-step instructions for cleaning and maintaining your instrument.

This manual includes the following chapters:

- Chapter 1: "Optimizing the Signal" describes how to switch on the instrument, obtain the first signal and optimize the instrument.
- Chapter 2: "Initial Performance Checks" describes how to set up a short term stability test and how to perform a survey scan.
- Chapter 3: "Instrument Calibrations" describes how to calibrate the instrument for optimum performance.
- Chapter 4: "Sample Analysis" describes how to set up an experiment for the analysis of standards and unknown samples.
- Chapter 5: "Operating PlasmaScreen" describes how to optimize and use PlasmaScreen in cool plasma mode and hot screen mode.
- Chapter 6: "CCT Operation" describes how to use Collision Cell Technology with different gases.
- Chapter 7: "Maintenance" describes how to clean the body of the XSERIES 2 and how to optimize pump performance.
- Chapter 8: "Troubleshooting" describes how to diagnose and resolve the most typical trivial problems.

Intended Use of the Equipment

The equipment is intended to be used by trained personnel within a laboratory environment, for the purpose of chemical analysis. It should not be used for any other purpose or within any other environment. The instrument must not be used in an unspecified manner.

Reference *Thermo Fisher Scientific Pre-Delivery Pack* for intended use of the equipment including technical specifications, environmental conditions, laboratory preparation requirements and other health and safety issues.

Changes to the Manual

To suggest changes to this manual, please send your comments to:

Editors, Technical Documentation
Thermo Fisher Scientific
Hanna-Kunath-Str. 11

28199 Bremen

Germany

documentation.bremen@thermofisher.com

You are encouraged to report errors or omissions in the text or index. Thank you.

Typographical Conventions

Typographical conventions have been established for Thermo Fisher Scientific manuals for the following:

- Data input
- Admonitions
- Topic headings

Data Input

Throughout this manual, the following conventions indicate data input and output via the computer:

- Messages displayed on the screen are represented by capitalizing the initial letter of each word and by italicizing each word.
- Input that you enter by keyboard is identified by quotation marks: single quotes for single characters, double quotes for strings.
- For brevity, expressions such as "choose File > **Directories**" are used rather than "pull down the File menu and choose Directories."
- Any command enclosed in angle brackets < > represents a single keystroke. For example, "press **<F1>**" means press the key labeled *F1*.
- Any command that requires pressing two or more keys simultaneously is shown with a plus sign connecting the keys. For example, "press **<Shift> + <F1>**" means press and hold the <Shift> key and then press the <F1> key.
- Any button that you click on the screen is represented in bold face letters. For example, "click on **Close**".

Admonitions

Admonitions contain information that is important, but not part of the main flow of text.

Admonitions can be of the following types:

- **Note** – information that can affect the quality of your data. In addition, notes often contain information that you might need if you are having trouble.
- **Caution** – information necessary to protect your instrument from damage.
- **Warning** – hazards to human beings. Each Warning is accompanied by a Warning symbol.

Topic Headings

The following headings are used to show the organization of topics within a chapter:

Chapter Name

The following headings appear in the left column of each page:

Second Level Topics

Third Level Topics

Fourth Level Topics

Safety and EMC Information

In accordance with our commitment to customer service and safety, these instruments have satisfied the requirements for the European CE Mark including the Low Voltage Directive.

Designed, manufactured and tested in an ISO9001 registered facility, this instrument has been shipped to you from our manufacturing facility in a safe condition.

Caution This instrument must be used as described in this manual. Any use of this instrument in a manner other than described here may result in instrument damage and/or operator injury. ▲

Safety Warning

Due to design changes and product improvements, the information in this document is subject to change without notice. Thermo Fisher Scientific reserves the right to change hardware and software designs, which may subsequently affect the contents of this customer information document. Thermo Fisher Scientific assumes no liability for any errors that may appear in this guide.

The methods and analytical procedures described in this and other manuals supplied with the XSERIES 2 are designed to be carried out by properly trained personnel in a suitably equipped laboratory. In common with many laboratory procedures, the methods described may involve hazardous materials or substances of unknown toxicity. For the correct and safe execution of the methods, it is essential that laboratory personnel follow standard safety procedures for the handling of hazardous materials.

Whilst the greatest care has been exercised in the preparation of this document, Thermo Fisher Scientific expressly disclaims any liability to users of these procedures for consequential damages of any kind arising out of or in connection with use of these procedures.

There are many sources of safety information available. Consult your chemical and laboratory supply catalogues. You may also refer to the *CRC Handbook of Laboratory Safety*, published by CRC Press Inc., Boca Raton, Florida.

Identifying Safety Information

The *XSERIES 2 Getting Started Guide* contains precautionary statements that can prevent personal injury, instrument damage, and loss of data if properly followed. Warning symbols alert the user to check for hazardous conditions. These appear throughout the manual, where applicable, and are defined in Table i.

Table i. Warning Symbols

Symbol	Description
	**General** This general symbol indicates that a hazard is present, which if not avoided, could result in injuries. The source of danger is described in the accompanying text. ▲
	**Electric Shock** Voltages above 50 V ac are present on the instrument. The instrument must be shut down and disconnected from line power before service or repair work is performed. ▲
	**Dangerous Chemicals** Chemicals are used in the normal operation of the instrument that may present a hazard. Ensure safety precautions are taken when dealing with hazardous liquids. ▲
	**Flammable Liquid** Some applications may use flammable solvents. Ensure safety precautions are taken when using flammable liquids. ▲
	**Hot Surface / Heat** Heat is generated by internal components within the XSERIES 2. Ensure that the cooling outlets on the back panel of the XSERIES 2 remain unobstructed at all times. Allow heated components to cool down before servicing them! ▲

Instrument-Specific Hazards

Every instrument has specific hazards, so be sure to read and comply with the following general warnings and safety precautions. They will help ensure the safe, long-term use of your system. More specific warnings and recommendations are given in the corresponding chapters of the *XSERIES 2 Getting Started Guide*.

1. The system should be operated by trained personnel only. Read the manuals before starting the system and make sure that you are familiar to the warnings and safety precautions!

2. Accurate results can be obtained only, if the system is in good condition and properly calibrated.

3. Service by the customer should be performed by trained qualified personnel only and should be restricted to servicing mechanical parts!

4. Service on electronical parts should be performed by Thermo Fisher Scientific Service Engineers only!

5. Before plugging in any of the instrument modules or turning on the power, always make sure that the voltage and fuses are set appropriately for your local line voltage.

6. Only use fuses of the type and current rating specified. Do not use repaired fuses and do not short-circuit the fuse holder.

7. The supplied power cord must be inserted into a power outlet with a protective earth contact (ground). When using an extension cord, make sure that the cord also has an earth contact.

8. Do not change the external or internal grounding connections. Tampering with or disconnecting these connections could endanger you and/or damage the system.

Caution The instrument is properly grounded in accordance with regulations when shipped. You do not need to make any changes to the electrical connections or to the instrument's chassis to ensure safe operation. ▲

9. Never run the system without the housing on. Permanent damage can occur.

10. Do not turn the instrument on if you suspect that it has incurred any kind of electrical damage. Instead, disconnect the power cord and contact a Service Representative for a product evaluation. Do not attempt to use the instrument until it has been evaluated. (Electrical damage may have occurred if the system shows visible signs of damage, or has been transported under severe stress.)

11. Damage can also result if the instrument is stored for prolonged periods under unfavorable conditions (e.g., subjected to heat, water, etc.).

12. Always disconnect the power cord before attempting any type of maintenance.

13. Capacitors inside the instrument may still be charged even if the instrument is turned off.

14. Never try to repair or replace any component of the system that is not described in this manual without the assistance of your service representative.

15. When leaving the system, make sure, that all protective covers and doors are properly connected and closed, and that heated areas are separated and marked to protect for unqualified personnel!

16. Do not dismantle the pump head. There are no serviceable parts inside. Warranty will be void if the pump has been opened.

17. Particulate material present in the samples can cause serious damage to the XSERIES 2. Ensure that your samples do NOT contain any particulate material before using them with the XSERIES 2.

18. Prolonged exposure to mineral acids at concentrations above 10% can cause degradation of the material used in the XSERIES 2's high precision piston pump. Avoid the use of samples containing high concentrations of mineral acid if possible; otherwise, ensure that you wash the XSERIES 2 with clean water between each measurement and at the end of the analysis.

Contents

Chapter 1 Optimizing the Signal

Objective

This exercise will lead you through the first basic operational steps from switching the instrument on, obtaining the first signal and optimizing the instrument.

This chapter treats the following steps:

Solution required for this practical

10 ppb tuning solution (Li, Be, Bi, Ce, Co, In, Ba, Pb, Tl, U) in 2% HNO_3.

Step 1 - Instrument Pre Startup Status

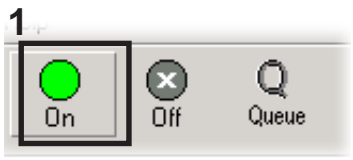
Perform the following checks before switching the instrument into operational state:

1. Ensure that glassware and cones are in position.

2. Check that the peristaltic pump tubing is in good condition and that tubing is clamped.

3. Ensure that the Faraday cage is in its forward position and that the Faraday cage door is closed.

Step 2 - Switching on the Instrument



1. Click once on the **On** button. The instrument will pass automatically through the start-up cycle which lasts approximately 90–120 s.

During the sequence, the sample introduction system is purged with argon. The neb gas is then switched off before the RF power is started and the ICP ignited. The interface valve is opened, and the slide valve separating the interface from the high vacuum region is opened (this occurs when the expansion pressure has reached less than 3 mbar). Once On, check the Peltier chiller temperature (if required).

Step 3 - Configuration Setup

1. Click on the **Instrument** icon to open up the *instrument control*.

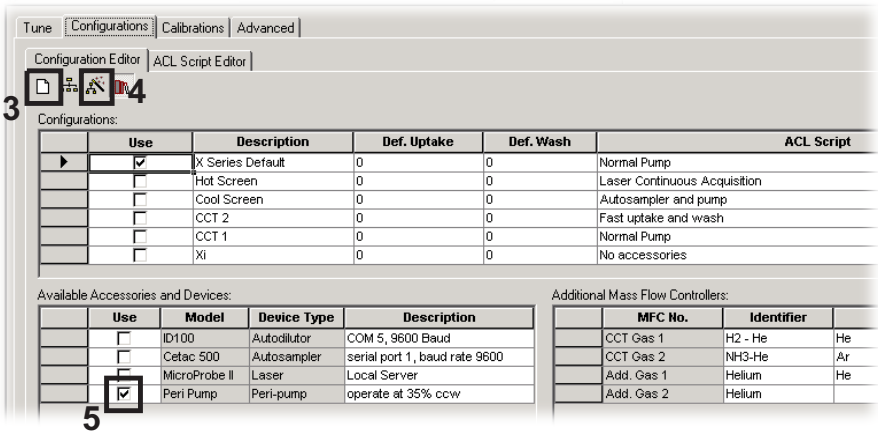
2. Open the **Configuration** page, which displays the *Configuration Editor*. From this page, the correct configuration can be selected.

   Configurations are usually set up for different sample introduction setups, that is Autosamplers, Low flow nebulizers, Laser Ablation, USN6000 etc.

   The *Accessories* and *Devices* that are to be used can be selected by ticking the appropriate box or setup using the accessory wizard.

   The instruments settings can be selected here, if a particular set of tuning parameters is required, or left unticked, if the current instrument settings are to be used.

3. Click on **New Configuration**, if different settings other than default or those already displayed are required. The *Description* is editable in order to indicate which options are being used. Each configuration has its own related tune settings which when saved appear under *Instrument* settings. Click on the required configuration to use it.



4. Click on **Accessories Wizard**, if a required accessory is not available for selection. The wizard will guide you through the selection and configuration of new accessories.

5. Tick the **Use** box on the *Available Accessories and Devices* to select which accessory is to be used for future experiments (that is, if an autosampler is selected here, when a new experiment is opened, it will automatically configure the experiment for autosampler sample positions).

Step 4 - Accessories Control

Use the standard tuning solution containing 10 ppb of Li, Be, Co, In, Ba, Ce, Pb, Bi, Tl and U. Place the sample uptake tube into the 1 ppb tune solution and ensure that the solution is taken up smoothly. Ensure that the solution uptake and the drainage are working correctly.

1. Click on **Tune** page in the instrument section.

2. Ensure that **Pulse Counting** is selected for Real Time Display.

3. Click on the **Accessories** window in the **Tune** page to display the Accessories control.
   The peristaltic pump or autosampler can be initialized and pump speeds optimized. Also autosamplers can be manually set to go to different positions from here, if required, e.g. to send the autosampler to the tune solution via an autosampler position.

Step 5 - Optimizing the Signal Manually

You can optimize the signal either manually or automatically. Usually the **Autotune** option is used, but it is good practise to be familiar with the manual tuning process, which will be explained briefly. Optimize for the highest sensitivity while obtaining low levels of oxides and doubly charged species.

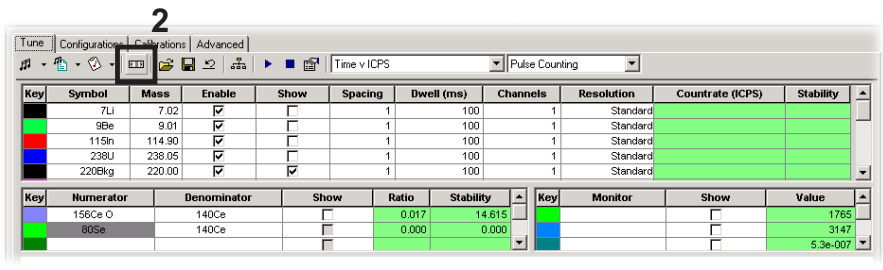
1. Click on **Tune** in the Instrument section, which is already open. Otherwise, click on the **Instrument** icon to open this section.

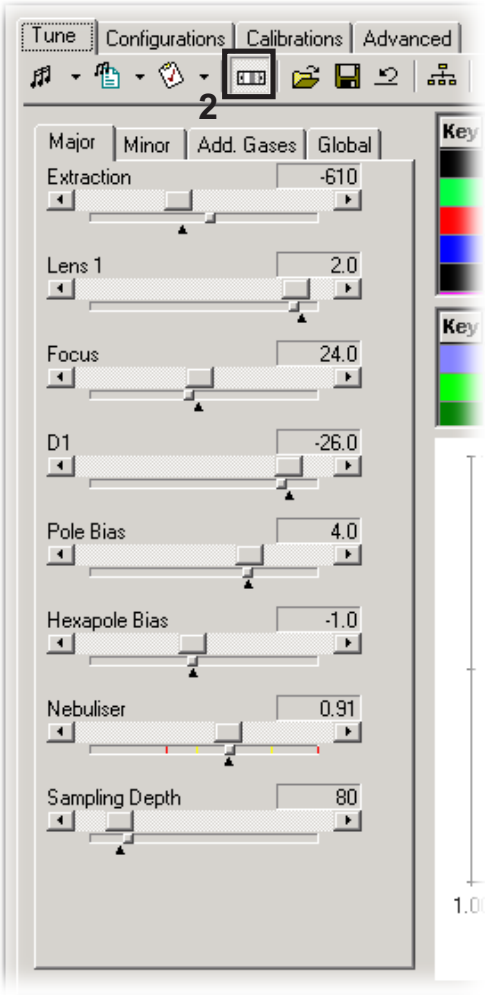
2. Click on the **Hide/Show** button to either hide or show the manual slide controls. For users who only use the *Autotune* functions, the manual slider controls are usually hidden. The setting of the **Hide/Show** button is remembered on a per user basis from the Windows logon.

3. Click on **Major**. These parameters are the most common ones for tuning and have the greatest effect on signal stability and sensitivity.

4. Click here to start the *Real Time Display (RTD)*.

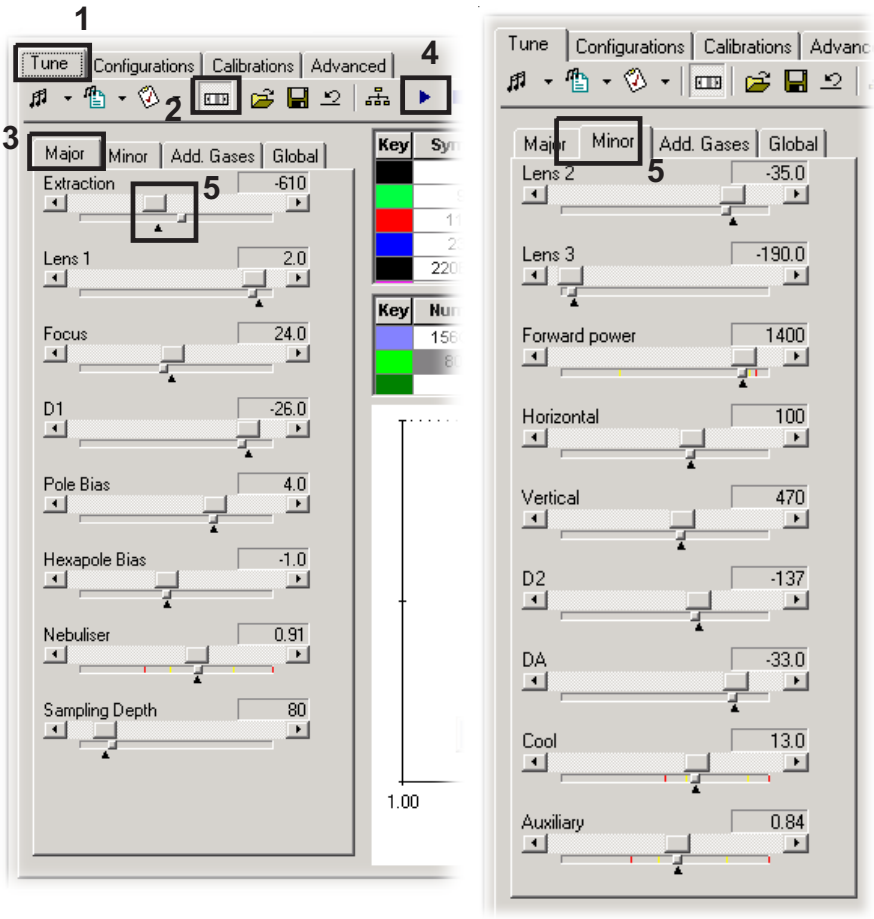
5. Use the slider to select the *default settings* as shown. The large button is the slider bar to change the voltage setting. The small reset button underneath the slider resets the voltage to the last saved setting. The red color underneath the slider indicates the voltage region which should not be used, the yellow indicates caution and the gray range is the recommended settings for voltage tuning. Gently drag the slider by moving the mouse pointer to the gray rectangle, and while holding down the left button, move it along.





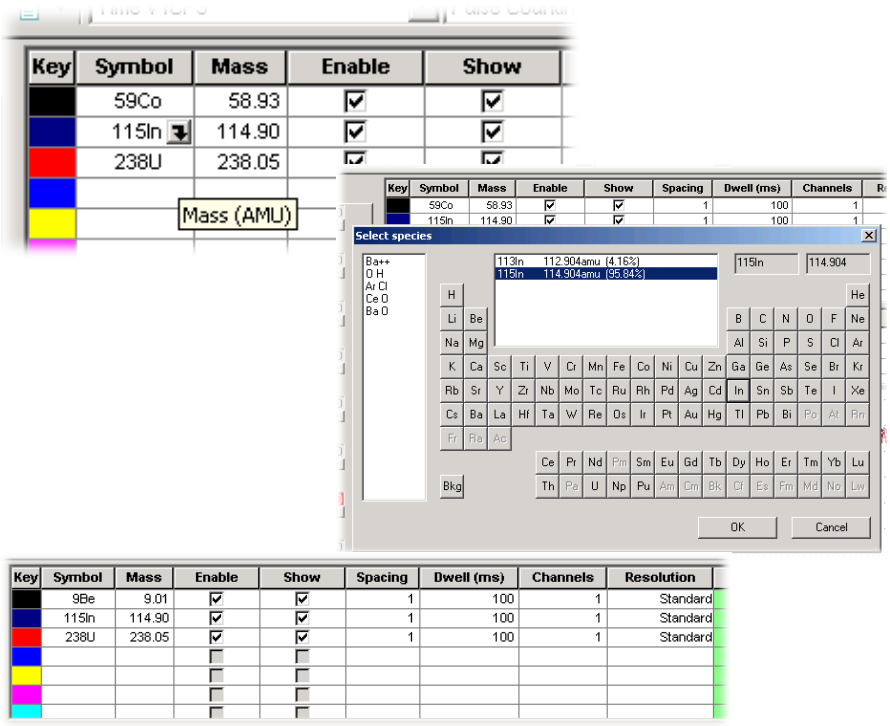
The voltage will be changed when the slider is released. Assuming that the torch is generally centered on the sample cone, start with *D1*, followed by *Extraction*, *Lens 1*, *Focus*, then *Pole Bias*, monitoring how the signal changes with each adjustment. Continue adjusting each of the lenses in sequence, until the optimum signal has been obtained. Click in the light gray region for coarse tuning and the arrows at the ends of the slider bar for fine tuning.

Note The figure below shows the default settings. ▲



6. Click on **Minor** for fine adjustment of the tuning. The horizontal and vertical positions of the torchbox might need re-positioning slightly when using different torch types or if the torch has been replaced.

7. It is possible to change the masses to be displayed in the RTD at any time. **Select the masses** Co59, In115 and Bi209 by selecting the **Enable** check box next to their mass. To change the monitored masses, click into the **Symbol** field and then on the **downward arrow**, which will display a periodic table. Select the analyte or interference you wish to monitor from the periodic table.

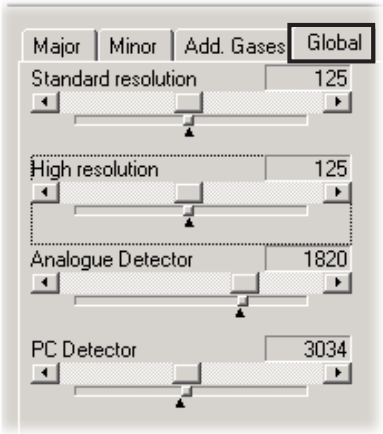


8. Enter a *Dwell time* of 100 ms. Drag and drop can be used to fill down.

9. Select CeO (156) and Ce^{++}(70) as *Numerators* ratioed to Ce(140) as *Denominator*, to monitor the oxide and doubly charged species levels (levels of < 2% CeO and < 3% Ce^{++} are acceptable).

Note These must first be added to the mass list before *Numerator* and *Denominator* can be selected. ▲

10. Monitor the masses selected in the *RTD*. When tuning the ion optics, a decrease and increase of the signal in CPS can be observed, when changing the voltage to the lenses. Tune for the maximum sensitivity, while obtaining a flat mass response across the whole mass range. Ensure that the oxides and doubly charged species do not exceed the acceptable levels. The responses measured depend on the interface type used and the mass monitored (that is, Be does not ionize as well as Li and therefore will not be as sensitive).



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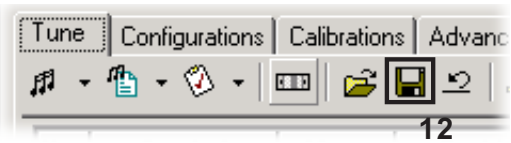
11. In the **Global** tune page, the settings for the resolution and detector voltages are displayed. This needs to be changed seldom, as for most purposes the resolution is factory-set and the detector voltages can be set up automatically using the *instrument calibration wizard* (see Chapter 3: "Instrument Calibrations".



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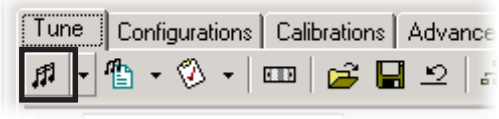
12. Save the optimized parameters.

Step 6 - Autotune Procedure

The **Tune** page contains a further icon to perform the Autotune. This function replaces the manual tuning procedure explained in "Step 5 - Optimizing the Signal Manually" on page 1-4. The Autotune procedure is fully user-definable to allow tuning for specific application needs. A list of Autotune procedure templates is supplied in the software and each procedure indicates which tuning solution should be used.

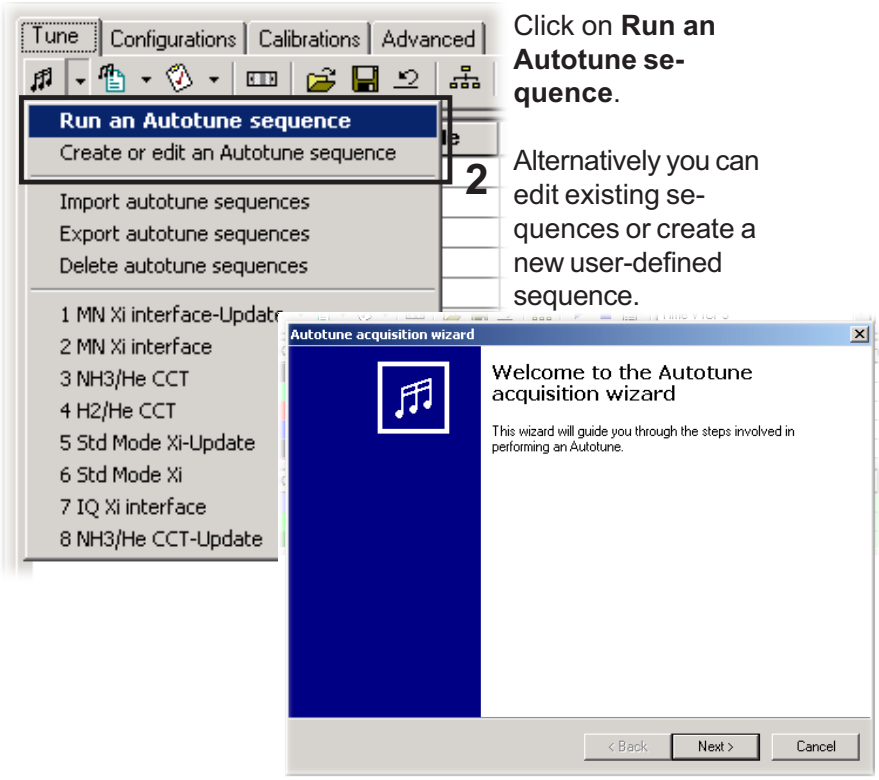
The Autotune procedure is subdivided into different stages. For each stage individual minimum and maximum criteria can be defined.

1. The Autotune can be started manually by clicking on the **Start the Autotune Wizard** icon.



1

2. Click on either the **Autotune** icon or the arrow next to it. Both will initiate the *autotune wizard*. The autotune wizard will now guide you step by step through the procedure. Click on **Next** after each step.

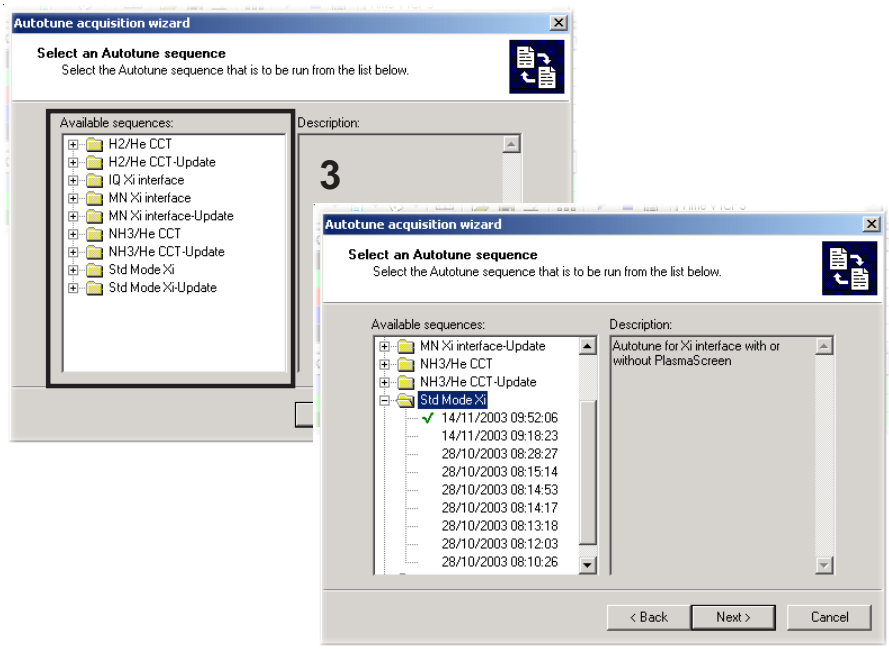


Click on **Run an Autotune sequence**.

Alternatively you can edit existing sequences or create a new user-defined sequence.

3. This page allows you to select an existing Autotune sequence and if more than one version of the sequence exists, a specific version.

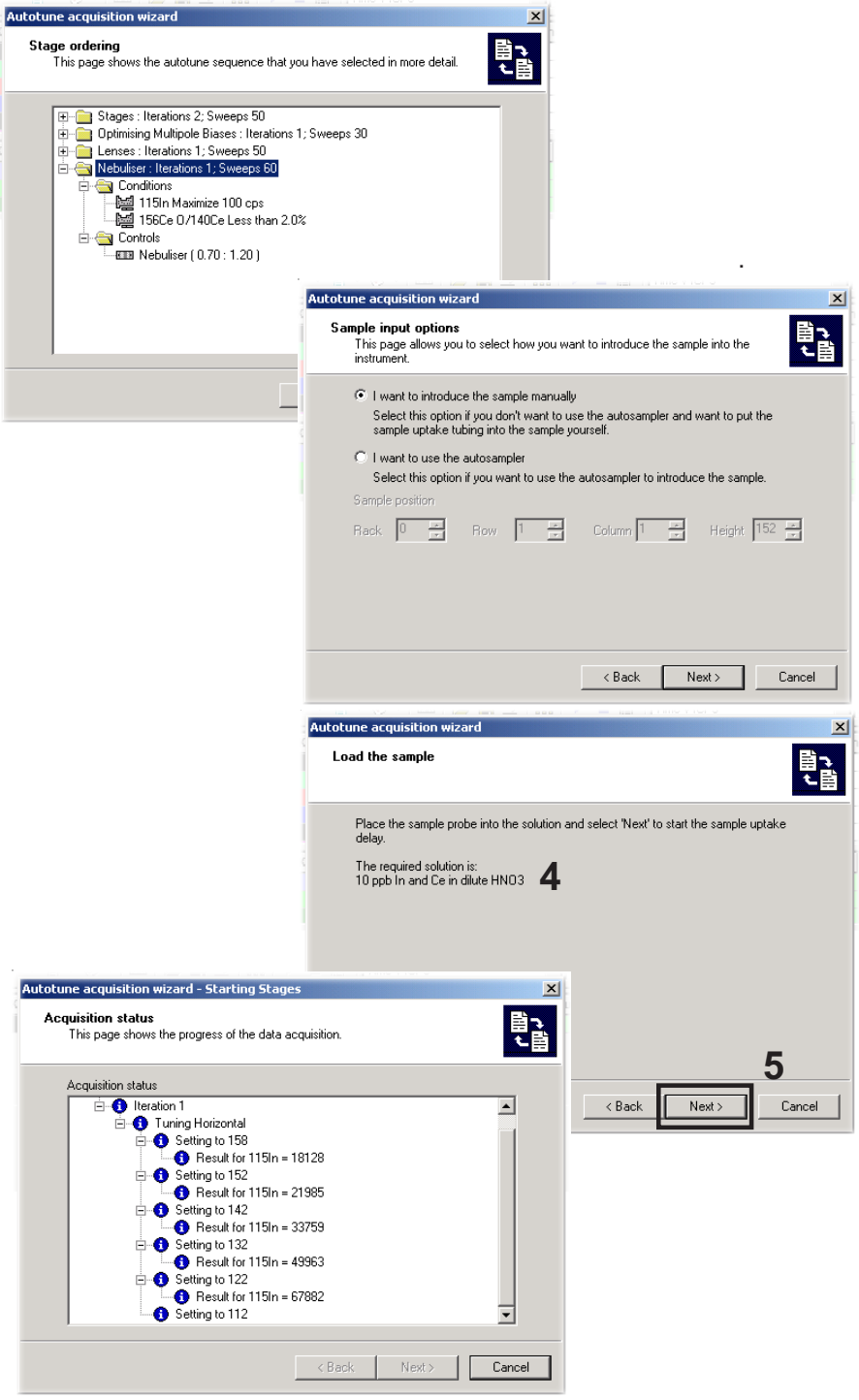
   Select the *sequence* from the list which suits your current application and configuration. E.g. a higher sensitivity specification for the high mass end and/or lower oxide levels could be defined for specific applications.



Each Autotune sequence is listed as a folder. Clicking on the + sign next to the folder will list the archived versions of the Autotune sequence. The green tick mark shows the currently active version of the sequence. Clicking on the folder will automatically select the currently active version. To select any of the archived versions, expand the folder and click on the required version.

The text box on the right of the page shows the description of the currently selected sequence.

If the wizard is being used to run a sequence, pressing the **Next** button will show the stage ordering for the Autotune sequence allowing the user to confirm that this is the correct Autotune sequence to use.



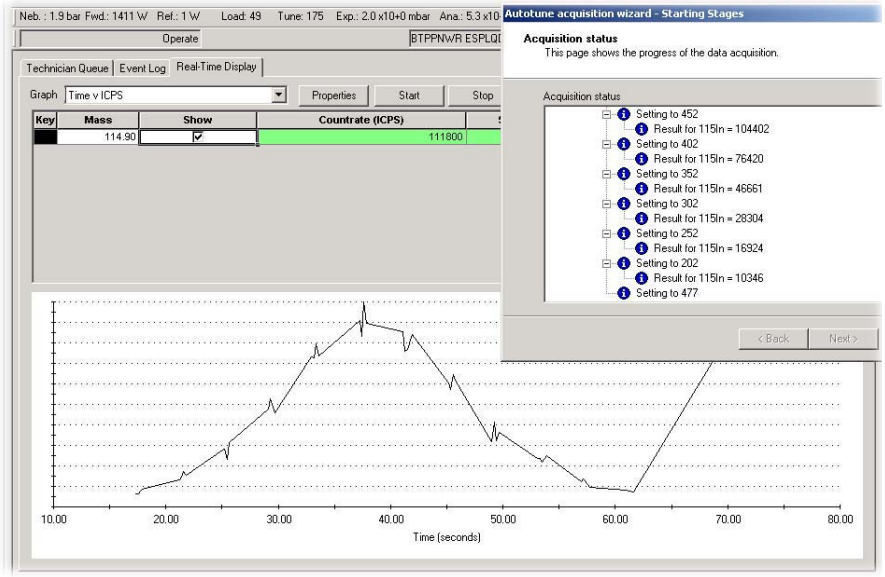
The detailed steps of the Autotune sequence are listed. This page allows the user to review the order and contents of the stages for the Autotune sequence before running and editing it. Each Autotune stage is listed as a folder.

The user can choose to introduce the sample manually or via the autosampler. If the autosampler is selected, the rack, row, column and height of the tune solution can be specified.

4. For each sequence, a tuning solution is recommended, which you should aspirate.

5. Click on **Next** to start the Autotune.

6. The progress of the Autotune process is displayed in-situ in the dialog box. The Autotune process can also be viewed in the **Real Time Display**. A full Autotune procedure will take approximately 10 min. The range for each parameter can be pre-defined by the user or by the default template. By editing the parameters appropriately, the duration of the Autotune process can be shortened.



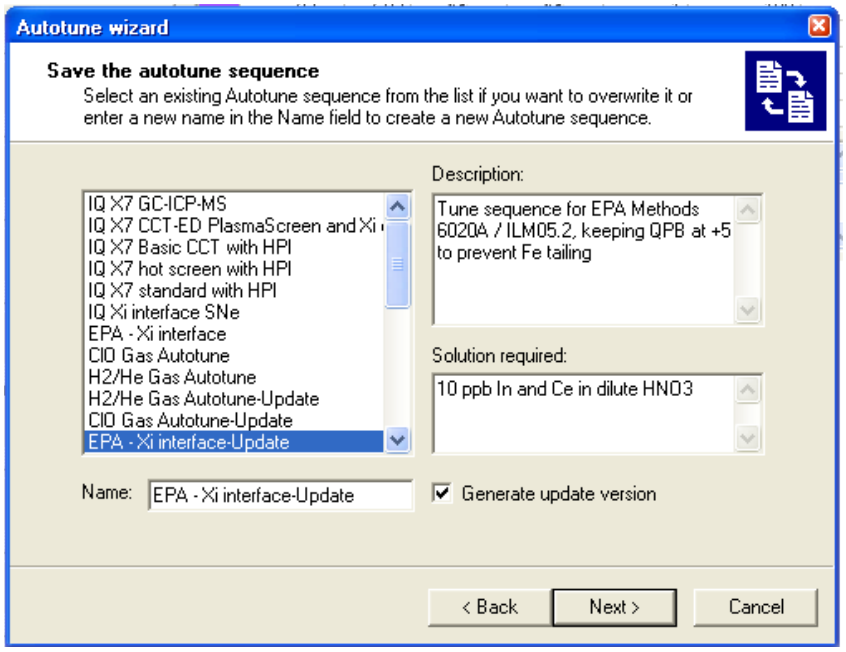
The Autotune script selected for this example is divided into three stages:

Stage 1: It will optimize the horizontal and vertical position of the torch while maximizing the In signal.

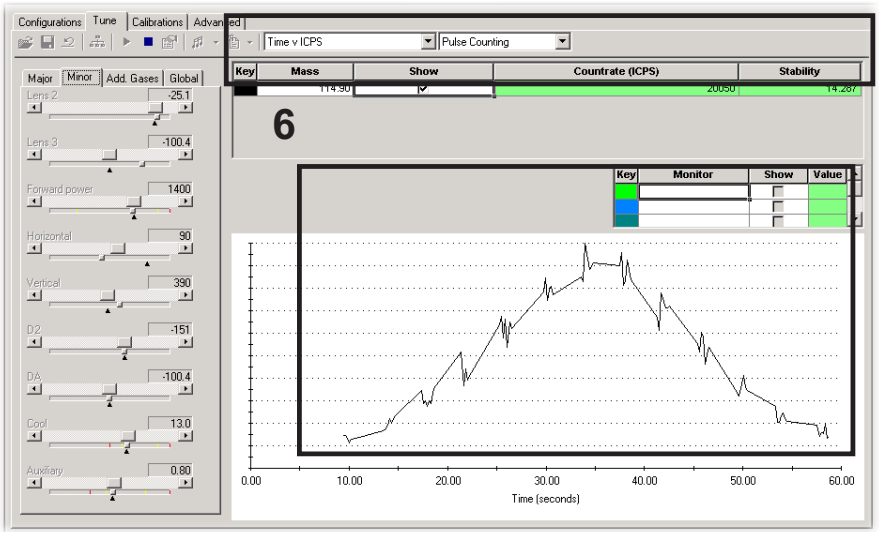
Stage 2: It will then optimize all the lenses one at a time, while maximizing the signal for a defined mass.

Stage 3: Tuning the nebulizer gas flow. It will go through maximizing the signal as well as minimizing the oxides at the same time.

Note It is recommended that you create an Autotune update sequence once the instrument is fully optimized. This option generates a new sequence that uses the new parameters as the default setting and automatically narrows the range over which each parameter is adjusted. Thus, the Autotune time can be significantly reduced. An update is created automatically, if the sequence has the **Create Update** check box marked at the end of the editing process. ▲



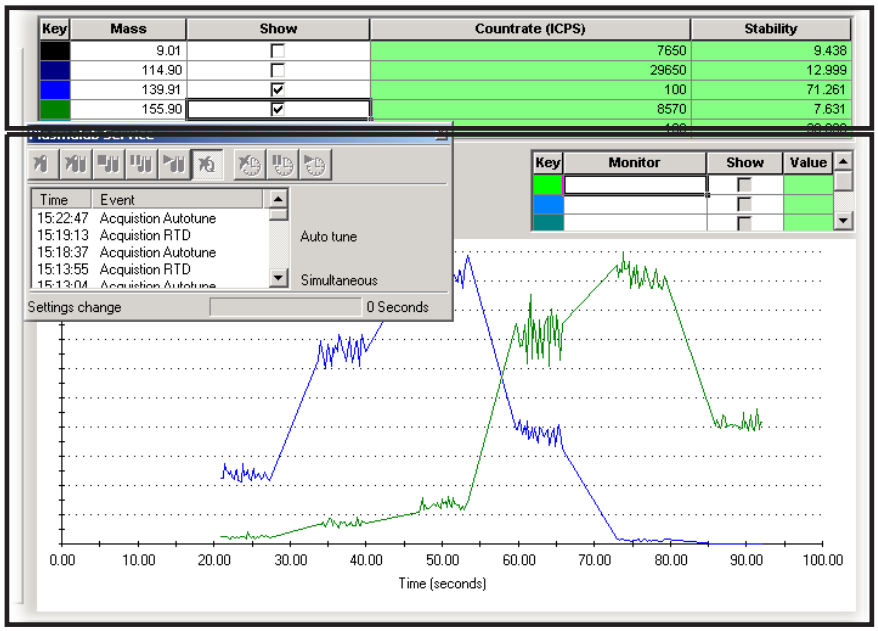
During torch optimization, the In signal is tuned for maximum sensitivity.



During the horizontal alignment procedure, the torch is changed between pre-defined settings.

While the torch is moved between the horizontal pre-defined positions, the In signal is plotted over time. The software will then define the horizontal position, where the maximum signal was determined.

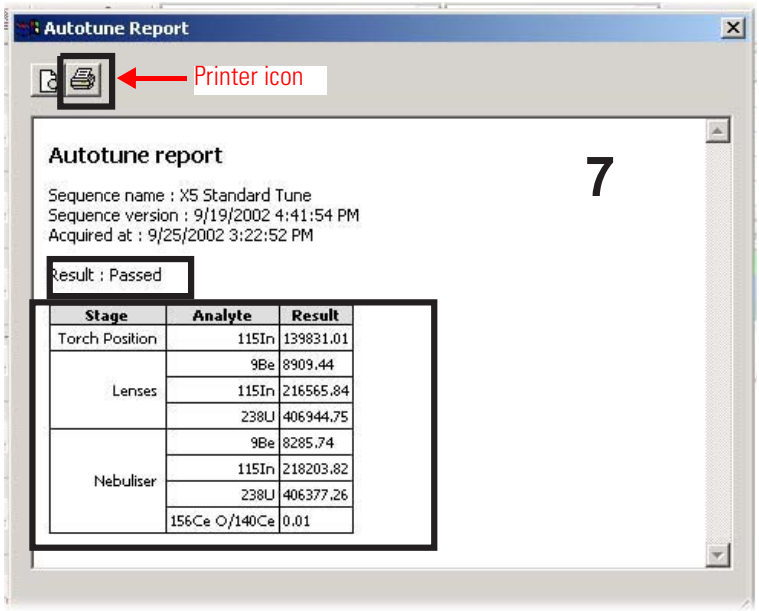
While the nebulizer gas setting is changed, not only In is monitored for maximum sensitivity, but also the CeO/Ce ratio (blue line) is monitored in order to minimize it.



When tuning the nebulizer gas flow, the effect is only visible in the RTD after approximately 5 s, due to the time taken for the nebulizer back pressure to adjust. For this reason, a default setting change is set in the software between each change in the gas flow rate.

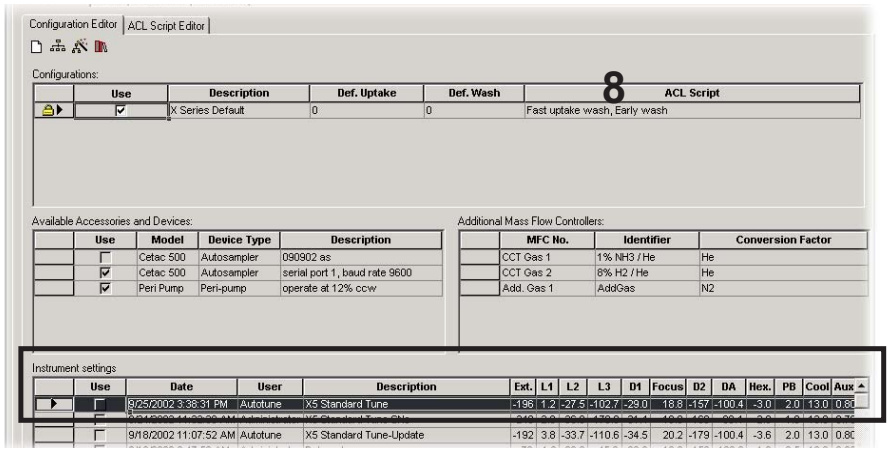
The RTD display can show both the analyte signal (black) and the CeO/Ce ratio (blue). The nebulizer gas flow is slowly increased or decreased and the effect on the analyte and oxide signal can be illustrated in the RTD display.

7. Once the Autotune procedure is completed, an *Autotune report* is generated which shows the chosen setting as well as the results for each stage of the tuning process. The Autotune report can be printed by simply clicking on the **Printer** icon. In this example, all set criteria were passed. If any criteria were not passed, it would be highlighted in red. The three stages *torch position*, *lenses* and *nebulizer* are listed as well as the analytes and/or oxides monitored.



As can be seen, during stage 1, (*torch position*) only the In signal was optimized, whereas during stage 2 (*lenses*), the signal was optimized for Be, In and U. In stage 3 (*nebulizer*), the sensitivities for the three analytes were monitored, while the CeO/O ratio was minimized.

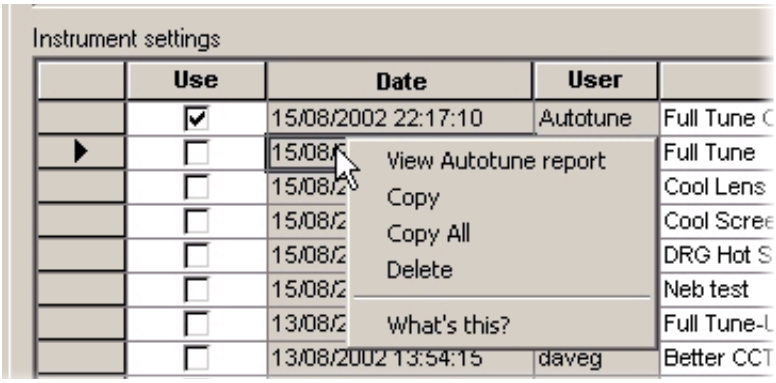
8. After completion, the settings found during the Autotune procedure are automatically saved in the **Configuration** page.



PlasmaLab allows you to view the results of successful Autotune sequences. Right mouse clicking on the instrument settings grid on the **Configuration** page will bring up the following menu.

The top menu item *View Autotune report* only appears for settings that have a *User of Autotune*. Selecting this menu item will bring up a dialog showing the stage by stage progress of the Autotune sequence used to create the settings.

To print this report, click on the **Printer** icon, or click on the **Print Preview** button to preview what the print will look like.



Chapter 2 Initial Performance Checks

Objective

To set up a short term stability test with monitoring the oxides and doubly charged species as isotope ratios. This is the daily checking routine prior to commencing the analysis of unknowns. A survey scan is also performed to check for good peak shapes and check mass calibration and peak resolution.

This chapter treats the following topics:

Solution required for this practical

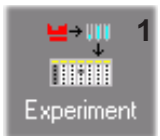
10 ppb tuning solution (Li, Be, Bi, Ce, Co, In, Pb, U)

Short term stability experiments can provide a great deal of useful information on the performance of your instrument. They should be undertaken as a daily checking routine every morning after optimizing the instrument, and to archive the data so that day to day performance can be monitored.

The *short term test* lasts only 10 min. and should be undertaken as part of the standard startup routine by all users. The instrument should be allowed to stabilize for 15 min. after starting up before attempting the

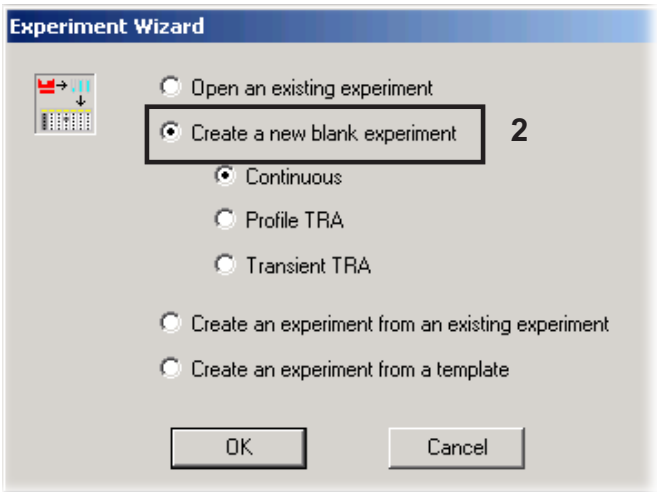
short term stability test. A *Survey Scan* can be performed as part of this test to give a great deal of useful information about peak shapes, resolution and mass calibration.

Step 1 - Creating a New Experiment

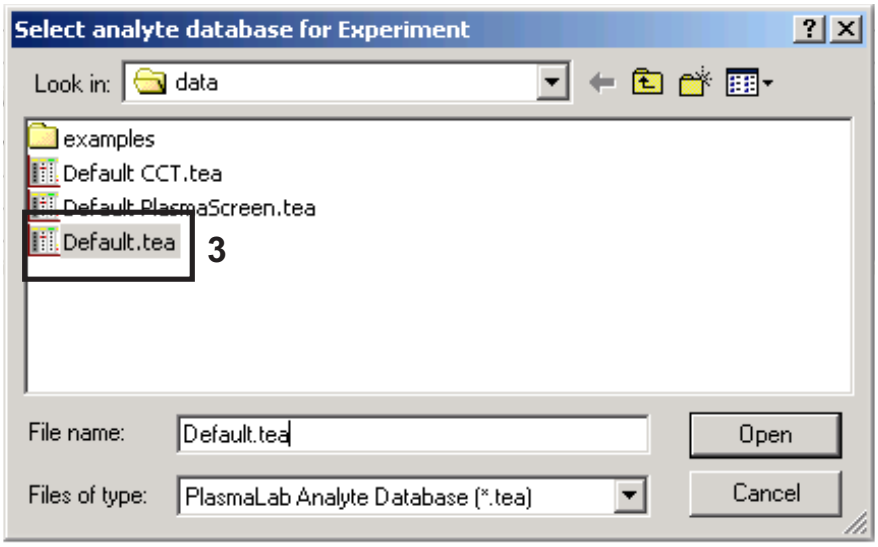


1. Click on the **Experiment** icon.

2. In the *experiment wizard*, click on **Create a new blank experiment**.



3. You are automatically prompted to choose a database.
   Select **Default.tea** as the analyte database for this experiment.

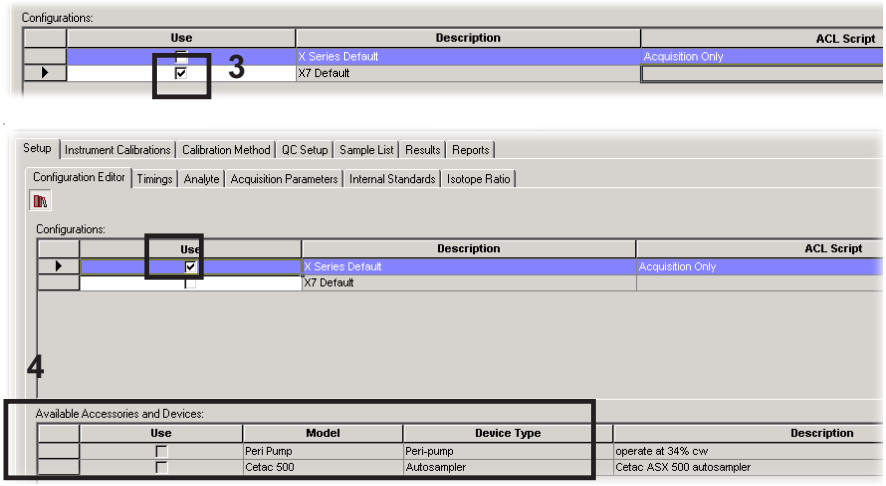


Step 2 - Experiment Setup

1. If the *Setup* pages are not displayed, click on the **Setup** tab to show them.

2. Click on the **Configuration Editor** to select the correct configuration for the experiment.

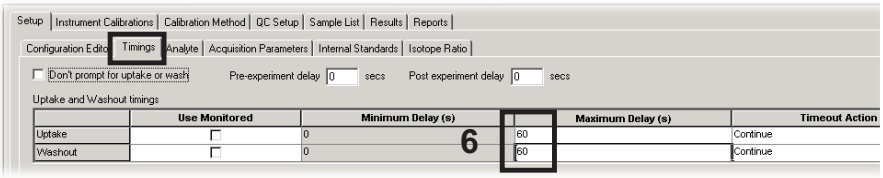
3. By default, the ICP-MS will use the current configuration and tune settings, which are shown in purple. If different conditions are required, select the *correct configuration* for the experiment by ticking the **Use** box. The software will now display the selected configuration in purple. By selecting a configuration for an experiment, it allows multiple experiments to be queued with different conditions.



4. The *Accessories* used with this configuration are shown. Should these be incorrect, the configuration will need to be edited in the *Main Instrument Section* of the software before continuing.

5. The *instrument settings* box can remain unticked, if only one set of instrument settings is being run. This will enable the experiment to use the last set of saved instrument settings.

   If different settings are used (that is, *Standard mode* and *PlasmaScreen mode*), the relevant instrument settings should be ticked in each experiment.

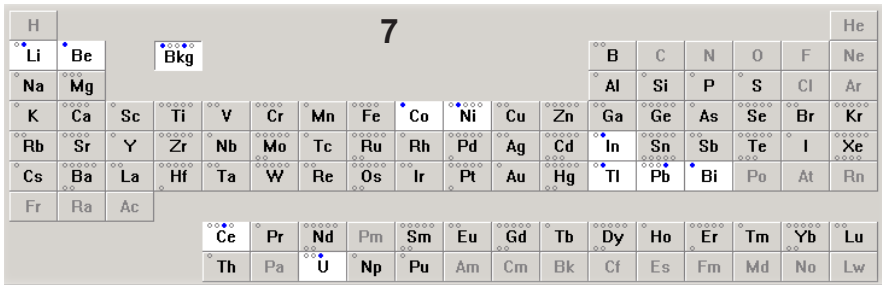


6. Click on the **Timings** tab to set up the sample introduction timings. Select the *Uptake time* for the time taken for the sample to reach the plasma and *Washout times* as shown above. Monitored uptake and wash will allow masses to be used to automatically determine when a sample has been introduced or washed out. Consult the *online help* for further information on this function.

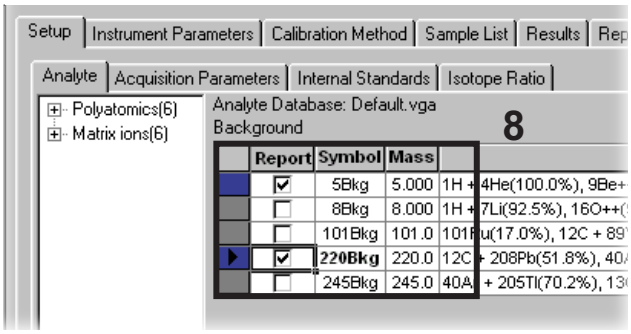
7. Click on the **Analyte** tab. Select the following *analytes* by double clicking to select the default isotope: Li, Be, Co, In, Ce, Pb, Bi, U and Bkg.

   The masses Bkg and Ce are monitored for checking the background

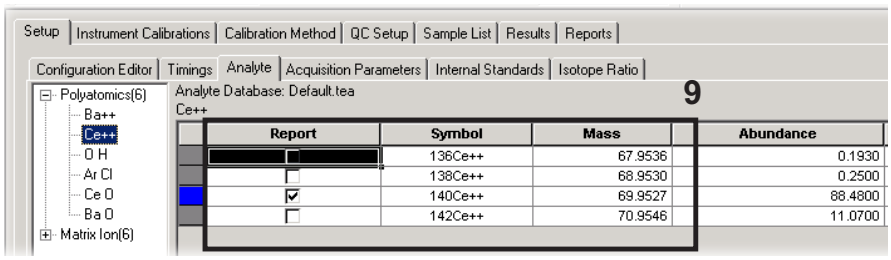
noise, oxides and doubly charged species. The other analytes are monitored for the short term stability test.



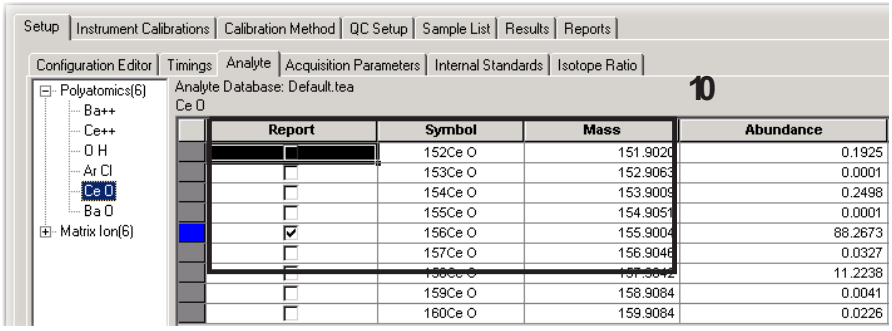
8. Click once on **Bkg** to manually select the masses 5 and 220 in order to monitor the background noise for the low and the high mass. Two blue dots will now appear for Bkg, indicating that two isotopes have been selected.



9. To monitor the *doubly charged species*, select **Ce^{++}** from the polyatomic list and select **$^{140}Ce^{++}$**.

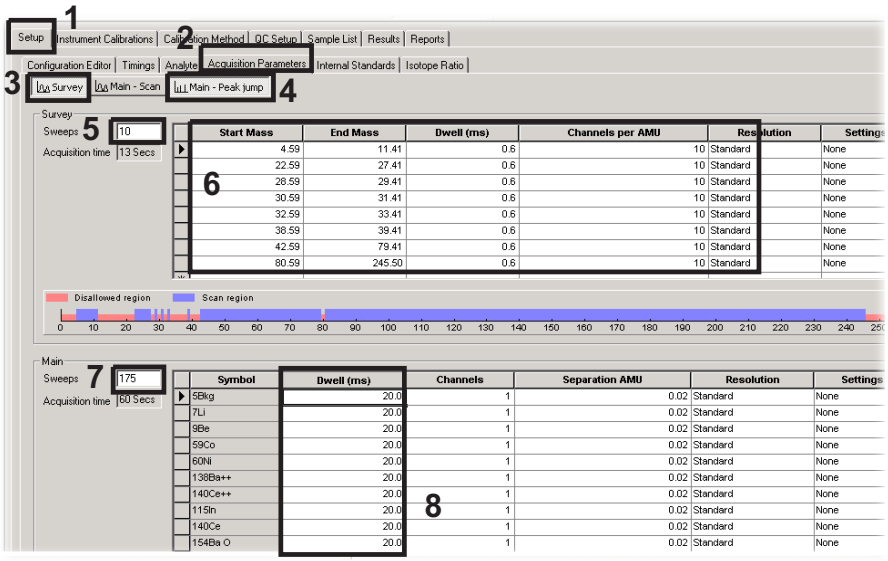


10. To monitor the *oxides*, select **CeO** from the polyatomic list and select **156CeO**.



Step 3 - Setting Acquisition Parameters

1. Now the *Acquisition parameters* can be defined. Select from the experiment section: **Setup**.



2. Select **Acquisition parameters**.

3. Select **Survey Run** (light gray when selected).

4. Select the main run to be **Peak jump**. This will just run the elements selected in the analyte menu.

5. Enter the same value for the number of **Sweeps** as shown above.

6. Select the **Start** and **End Mass** for the skip regions and **Dwell time**, **Channels per AMU** as shown for the Survey run. These parameters only become available when the Survey run has been selected. The Acquisition time will be automatically calculated. The survey run is

always performed in *Scanning mode*. The parameters shown are default and should already be present.

7. For the Main Peak jump run, select the number of sweeps to be 110.

8. As the Main run is normally performed in *Peak Jumping mode*, the number of *Sweeps*, *Dwell time* and *Channels per AMU* are different to the survey run.

   Enter the **Dwell times** for the analytes previously defined in the *element* menu. Use a Dwell time of 30 ms for the analytes, oxides and doubly charged species and 100 ms for the background masses. The acquisition time will be automatically calculated. It should be approximately 60 s for a short term stability test.

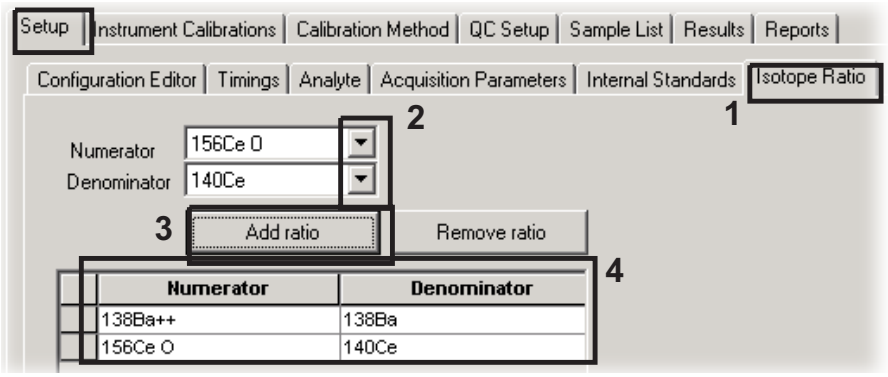
Step 4 - Isotope Ratio Selection

1. To define the Isotope Ratios, click on **Setup** and **Isotope Ratio**.

2. The ratios for the *oxides* and *doubly charged species* can now be defined. Click on the **downward arrow** to display the list of analytes to choose the *Numerator* and *Denominator*.

3. After selecting first the Numerator and then the Denominator, click on the **Add ratio** button.

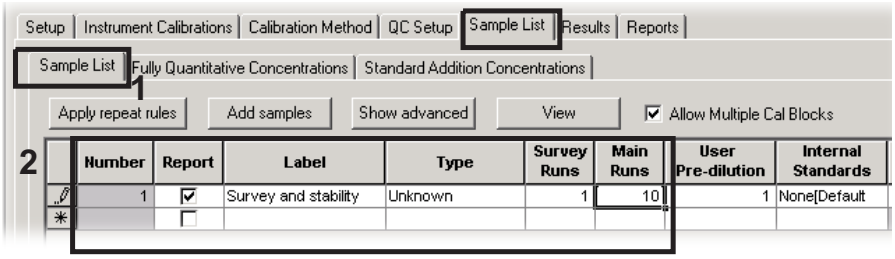
4. After clicking the **Add ratio** button, both analytes will be displayed in the table. Select the ratios as shown below for Ce^{++}/Ce and CeO/Ce. If a ratio is incorrectly added, it can be removed by clicking the **gray box** next to the ratio in the grid and press the **Delete ratio** button.

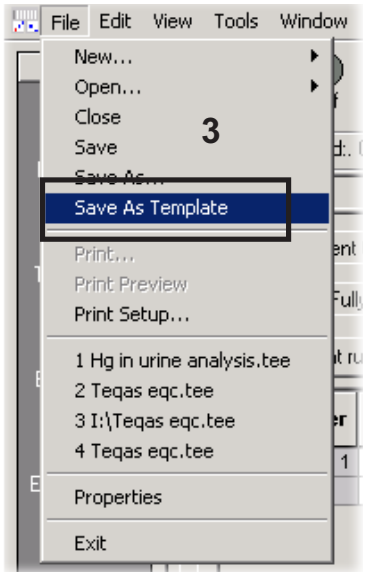


Step 5 - Creating Sample List

1. Now the sample list can be defined. Click on **Sample List** and then again **Sample List**.

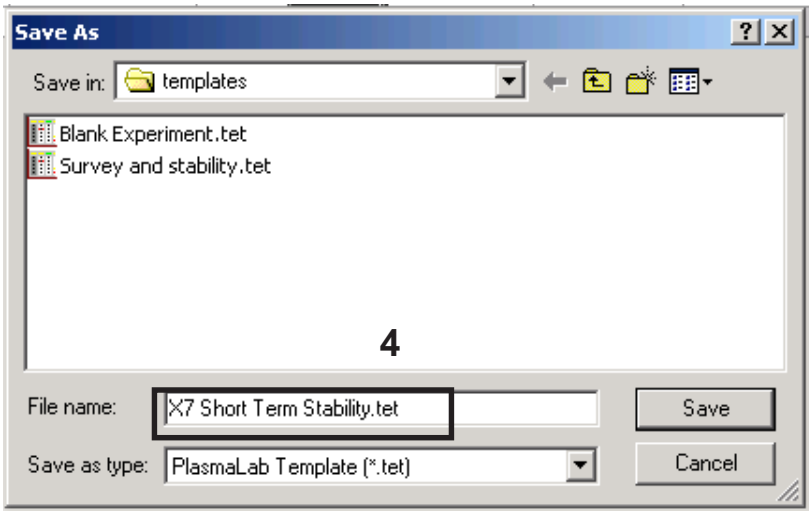
2. Enter all fields as shown below. The *Survey Run* will be used to check the mass calibration and peak shapes for the full mass range. Then the ten *Main Runs* will be used for the short term stability test.



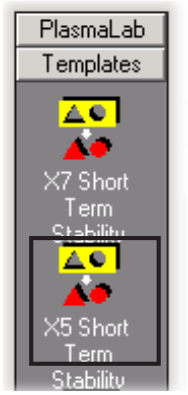


3. It is recommended to build up a library of templates for routine analysis types, e.g. for tuning, short term stability test etc.

   To create a template at any stage of building an experiment or even post acquisition, choose the **File > Save As Template** option when the experiment is open.



4. **File Save as Template**, file name *"Survey and stabilities"*.

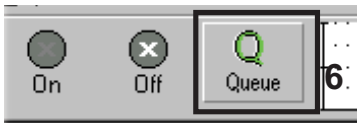


5. The list of templates available can be viewed by clicking on **Template** at the lower left corner of the screen. The currently defined templates will be shown.

   To perform the short term stability test in the future, click on **Template** at the lower left corner and select **Survey and stabilities**.

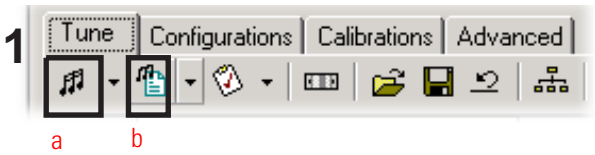
   Alternatively, click on the **Experiment** icon, create an experiment from a template and select **Survey and stabilities.tet**. It is also possible to add extra samples or analytes to the original experiment, if desired.

6. When the sample list is complete, click on the Experiment **Queue** button to start the Acquisition.



Step 6 - Running Performance Report Test

The *Tune* page contains two further icons to either perform the *Autotune* (a) or to start the *performance report wizard* (b). Those two functions replace the manual tuning procedure and the short term stability test as explained in the earlier steps of this practical.



The Autotune and performance report functions are designed to replace the manual tuning procedure and the short term stability test. A user-defined performance report can be set up similar to the short term stability test, in which the user will define the daily performance specifications for the instrument. If the criteria are met, the instrument is ready to commence sample analyses. If the criteria are not met, an Autotune must be performed after verifying that all other possible sources of the performance test failure have been excluded.

The performance report is a user-definable procedure, although the software is supplied with pre-defined examples. It is generally set up to run a short term stability test, in which the user can define the pass/fail criteria. These criteria could entail different sensitivity specifications for the analytes, RSD limits, and/or limits on oxide levels, doubly charged species, background counts etc. These limits can be set to either minimum or maximum values.

The user can specify the action to be taken, if any of the criteria are not met. The user can also define what analytes to analyze in the short term stability test, which could be the tune solution or it could contain analytes closer to the analytes in the samples. After the analysis, the software will evaluate the pass criteria.

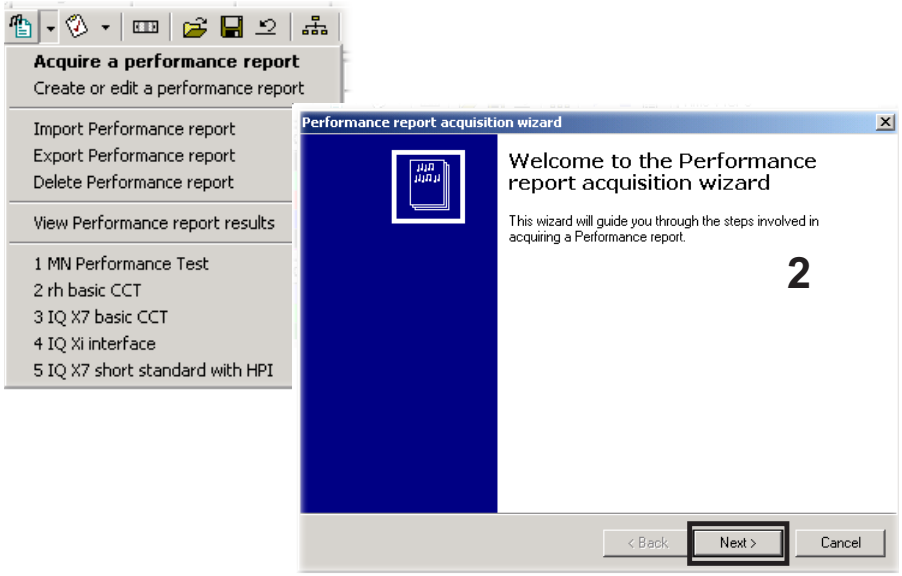
If it passes, the user can carry on with the analyses of standards and unknown samples. If it fails, the Autotune procedure should be performed. If this passes, the performance test should be repeated.

The performance report and Autotune routines can be fully automated in an experiment. Refer to the online help for further information about this option.

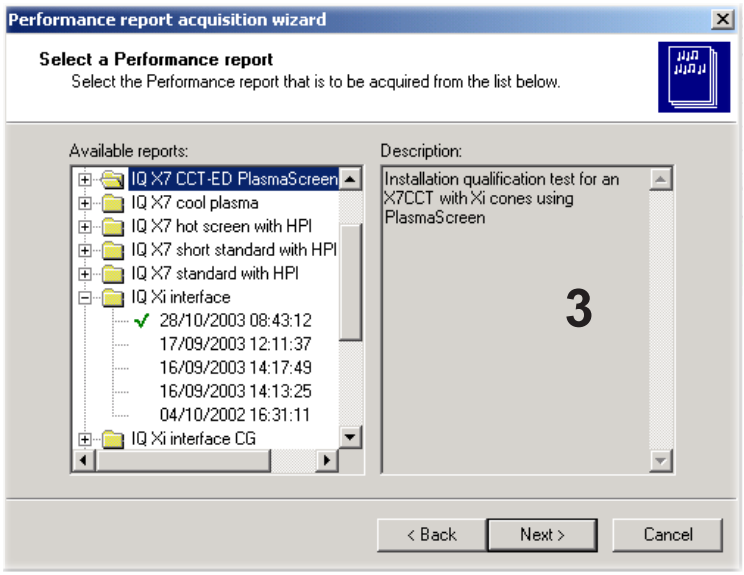
1. After the instrument has been switched on and has warmed up, click on the icon for the **Performance Report**, and a wizard will automatically open up. The *toolbar button* provides access to the *Performance report configuration* and *execution functions*. Clicking on the icon starts the *performance report acquisition wizard*.

   The first item in the menu allows the user to start the *performance report acquisition wizard*, which is the same action as clicking on the **toolbar button**. Up to eight of the last Performance reports that have been run are listed at the bottom of the menu. Clicking on one of these items will start the currently active version of the Performance report.

2. Follow the steps of the *performance report wizard* as shown below. Click on **Acquire a Performance Report**. Click on **Next** after each step.



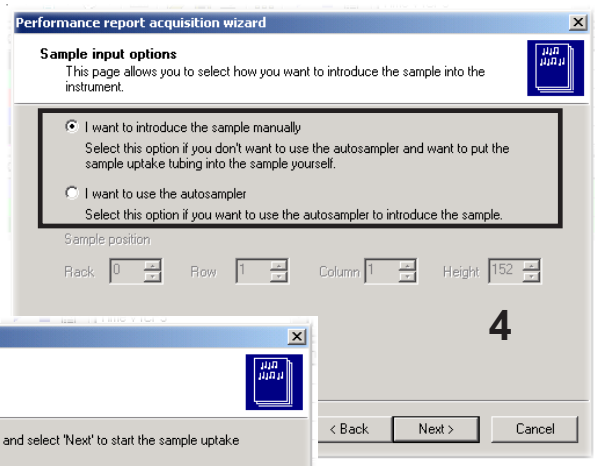
3. Select the report that matches your configuration from the list. If no report suitable for your configuration is available, **cancel** the wizard and either edit an existing report or create a new one at the start of the performance report wizard.

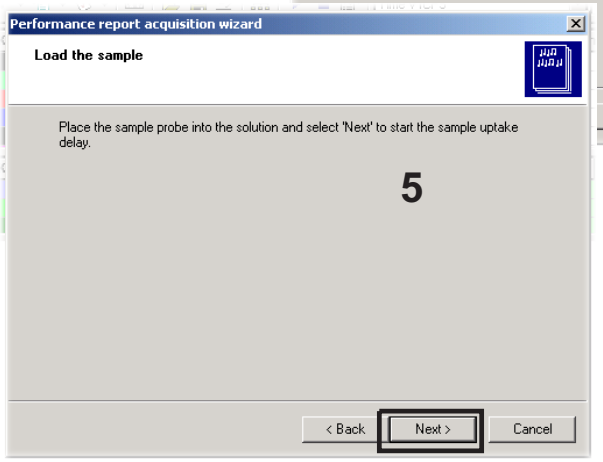


Each Performance report is listed as a folder. Clicking on the **+ sign** next to the folder will list the archived versions of the Performance report. The green tick mark shows the currently active version of the report.

Clicking on the folder will automatically select the currently active version. To select any of the archived versions, expand the folder and just click on the required version. The text box on the right of the page shows the description of the currently selected report.

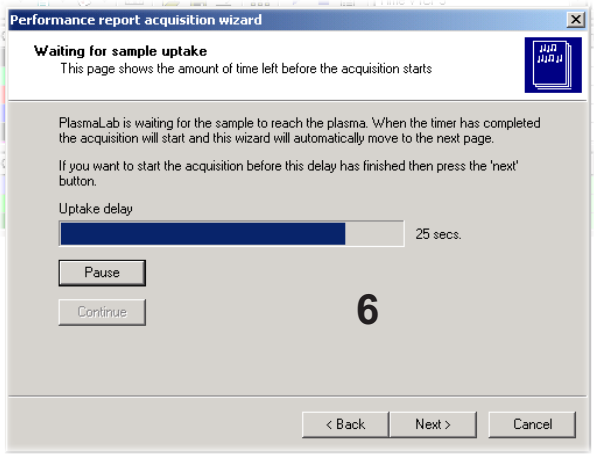
4. Select whether you wish to introduce the sample manually or via the autosampler.



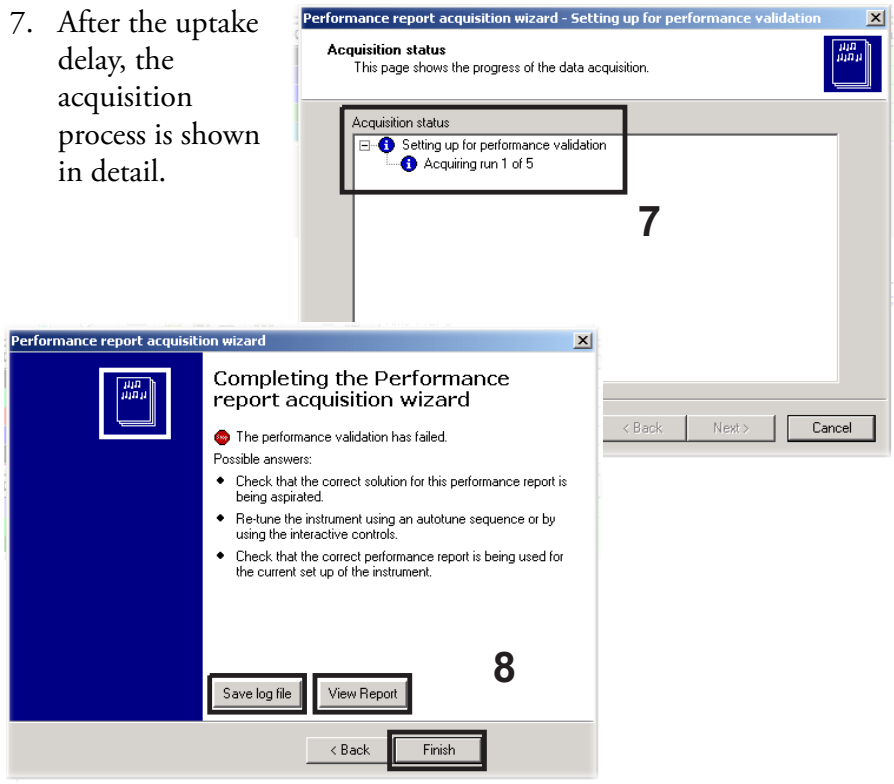


5. Load the sample and click on **Next** to start the performance report sample update.

6. The performance report acquisition wizard displays every step of the process, e.g. uptake delay in seconds.



7. After the uptake delay, the acquisition process is shown in detail.



8. The results of the performance report acquisition wizard are displayed. In this example, the criteria were not met, and the performance validation has failed. The wizard will list possible explanations for the failure.

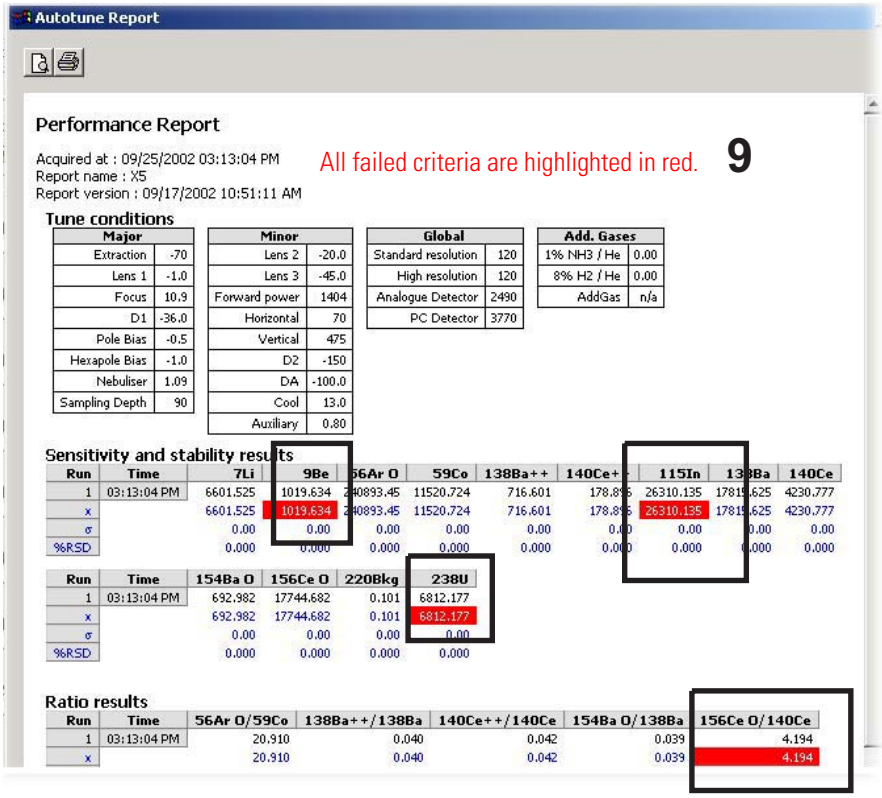
Note If the correct solution and performance report for the current instrument configuration were chosen, it is recommended to re-tune the instrument. ▲

This is the final page for the performance report wizard. Clicking on the **Finish** button will finish the wizard. The **View Report** button will display the report and allow it to be printed. This report can also be viewed using the *View Performance Report Results* of the menu of the **Performance Report** button.

The **Save Log File** button allows the progress of the performance report to be saved to a file for diagnostic purposes, if a failing performance report test needs troubleshooting.

9. The performance report is automatically displayed. The *tuning parameters* are listed at the top of the page. The report can be printed by clicking on the **Print** icon.

In this example, some of the criteria failed, such as the sensitivity specification, as well as the oxide level. The failed criteria are highlighted in red.



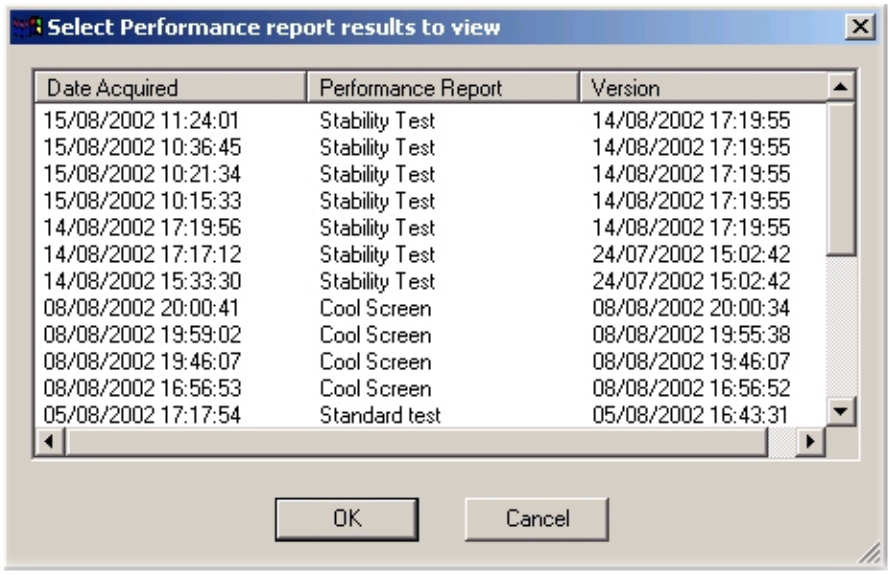
Note Generally, the performance test will be passed first time. It is then possible to commence with the sample running immediately afterwards. ▲

Note It is also possible to run the *Autotune* prior to the performance test by clicking its icon. ▲

PlasmaLab allows you to view the results of performance reports that are acquired from the *Instrument Tune* page. Selecting the **View Performance report results** menu item from the **Performance reports** drop down menu shows the following dialog.

Each performance report result is listed in the *list* window. Clicking on the header of each column will sort that column, toggling between ascending and descending for each click.

To select a result to view, click on the name in the list. If you wish to view more than one result, hold down the **Ctrl** key whilst using the mouse to click on each of the required results in the list.


Select Performance report results to view
Date Acquired
Performance Report
Version
15/08/2002 11:24:01 Stability Test 14/08/2002 17:19:55
15/08/2002 10:36:45 Stability Test 14/08/2002 17:19:55
15/08/2002 10:21:34 Stability Test 14/08/2002 17:19:55
15/08/2002 10:15:33 Stability Test 14/08/2002 17:19:55
14/08/2002 17:19:56 Stability Test 14/08/2002 17:19:55
14/08/2002 17:17:12 Stability Test 24/07/2002 15:02:42
14/08/2002 15:33:30 Stability Test 24/07/2002 15:02:42
08/08/2002 20:00:41 Cool Screen 08/08/2002 20:00:34
08/08/2002 19:59:02 Cool Screen 08/08/2002 19:55:38
08/08/2002 19:46:07 Cool Screen 08/08/2002 19:46:07
08/08/2002 16:56:53 Cool Screen 08/08/2002 16:56:52
05/08/2002 17:17:54 Standard test 05/08/2002 16:43:31
OK
Cancel

Chapter 3 Instrument Calibrations

Objective

This section will take you through all steps of instrument calibration. The XSERIES 2 ICP-MS periodically needs to be calibrated for optimum performance. The *instrument calibration wizard* allows you to adjust the mass calibration, as well as the detector calibration to ensure a good wide dynamic range with a cross calibration of the pulse counting and analog modes of the detector.

This chapter treats the following topics:

General Remarks

There are two types of calibration routines to be performed on a regular basis:

- *Detector calibration* and
- *Mass calibration*

These calibrations are made through the instrument calibration wizard in the *Instrument* section or an experiment using an *instrument setup sample*. Whenever an instrument setup sample is analyzed, the data is always copied back to the *instrument database* for subsequent experiments to use as well as a copy being kept in the experiment itself.

The *Instrument Calibrations* page also shows the *Detector Monitoring* page which allows the user to track the lifetime of the detector currently installed. For each new detector, a new lifetime dataset is created as well as a new section for the *detector calibrations*.

Solutions required for instrument calibration

It is recommended to use a ME solution in the 50-100 ppb range, containing low, mid and high mass elements.

The sample used for the *detector setup wizard* needs to be a multi-element solution which meets the following criteria:

- At least one analyte peak between masses 100-140 amu must have a count rate between 500,000 and 2,000,000 cps. This may be a minor isotope that is not normally used for measurement in an experiment (that is, 117Sn). The software will use as many peaks as it can between 100 and 140 amu to set up the voltage on the analog detector section. It will normally use more than one peak, but unless it can find one peak that meets the criteria, the calibration will fail. An appropriate message will be given in the wizard dialog.

- If possible, all the analyte elements that are to be measured using analog mode for any part in the experiment should be present in the solution with count rates exceeding 500,000 cps. It does not matter if any (or most) of the peaks exceed the maximum count rate allowed for the pulse counting detector section, as the software will automatically detect this during the calibration and use points at the side of the peak to perform the cross calibration.

- To ensure a reasonable interpolation of the cross calibration when no analytes are present in a mass range, it will be better, if both very low mass and very high mass analytes are present at count rates exceeding 500,000 cps in the calibration solution.

Step 1 - Detector Calibration

The XSERIES 2 ICP-MS can operate over a wide dynamic range, allowing the user to analyze trace, minor and major element concentrations. Trace elements produce very low signals, which in turn require a large amount of amplification. These elements are detected in the pulse counting part of the detector. Large concentrations are measured with the detector operating in analog mode.

In order to achieve a continuous and wide dynamic range, the pulse counting and analog acquisition modes operate together.

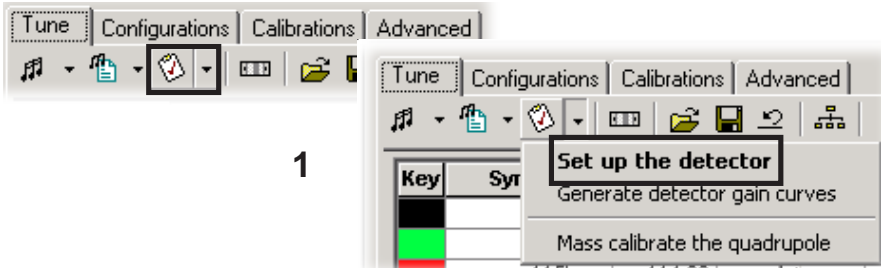
Note It is important to have the correct correlation between the two modes of operation to ensure continuity over the complete dynamic range. The correlation between pulse counting and analog detection is mapped by the detector cross calibration. ▲

A conversion factor is calculated for each of the valid masses. This is used to calculate a polynomial equation which is then used to generate a lookup table of correction factors for each integer mass over the whole mass range. Before being stored in the experiment and the instrument database, for any masses where a factor has been measured, the lookup table is replaced by the measured value.

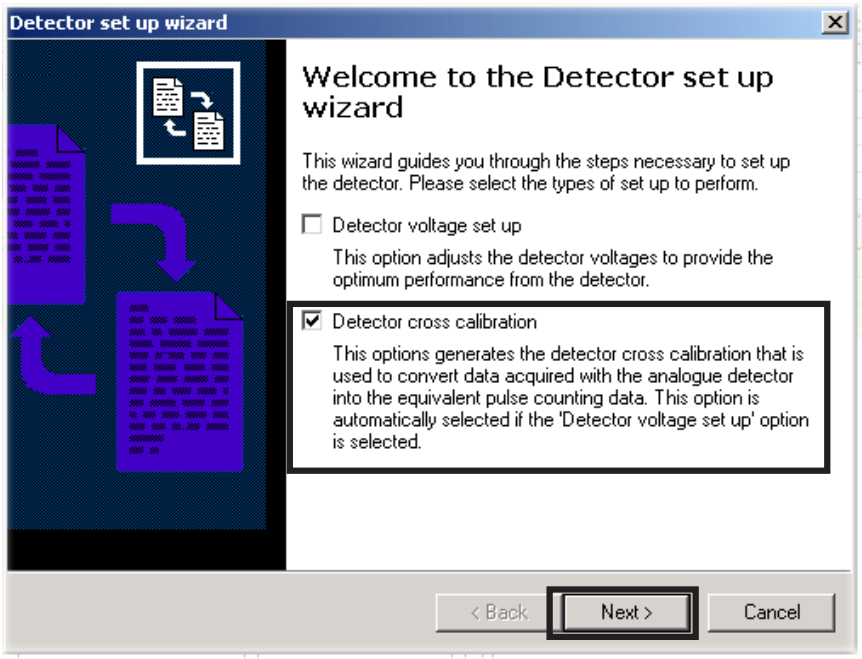
As the instrument calibrations are automated procedures, the user is not required to input any analyte information. The software automatically detects the analytes present in the solution and chooses the most appropriate ones to use.

The detector calibration software is designed to work with solutions that have high analyte concentrations which collect data in the analog detector mode. The software will automatically measure points at the side of a peak which trips the detector gate at its maximum.

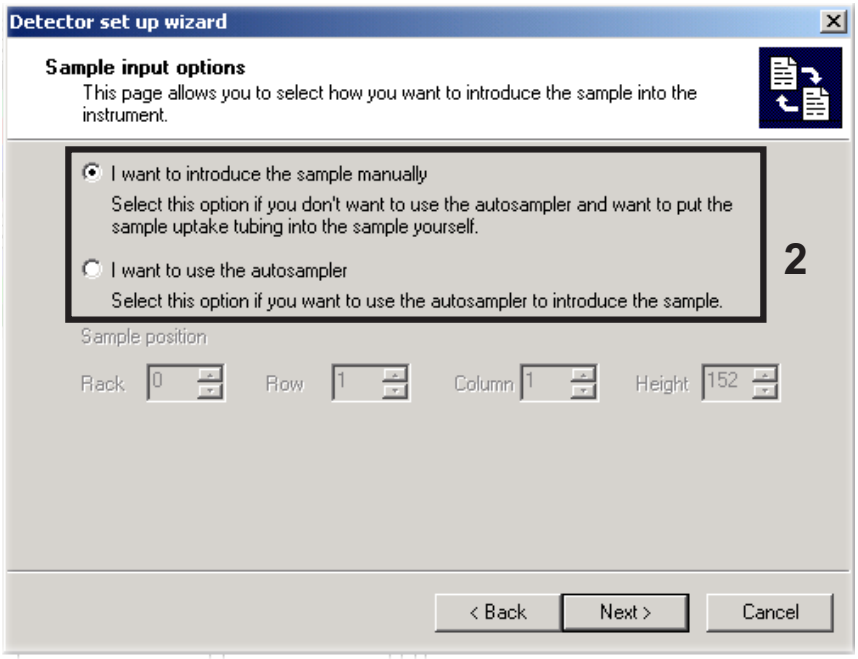
The *instrument calibration wizard* will perform all the steps necessary to set up the detector.



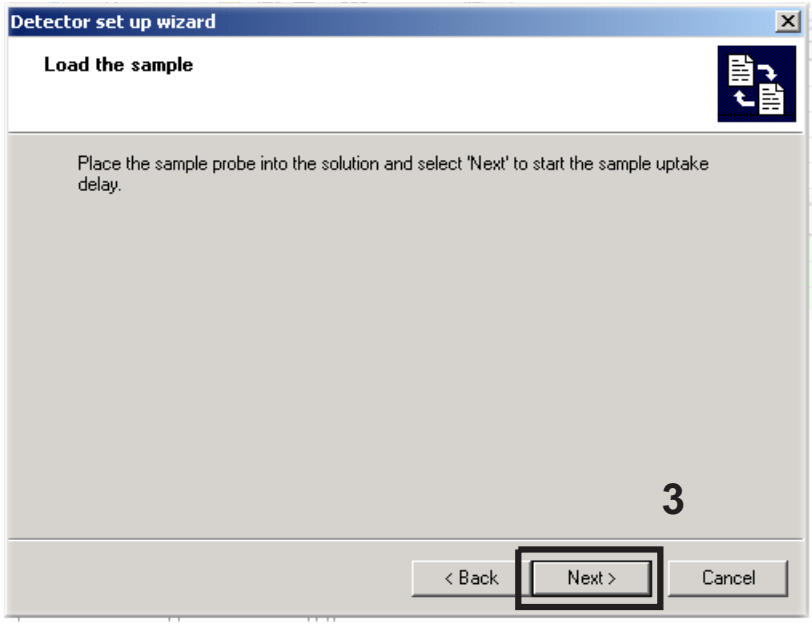
1. Click on **Set up the detector**. A dialog will open to present the user with options to perform a calibration between the pulse counting and analog detector modes (cross calibration) or to fully set up the detector voltages and then do a cross calibration. Click on **Next**.



2. The user can either present the solution manually or via the autosampler.

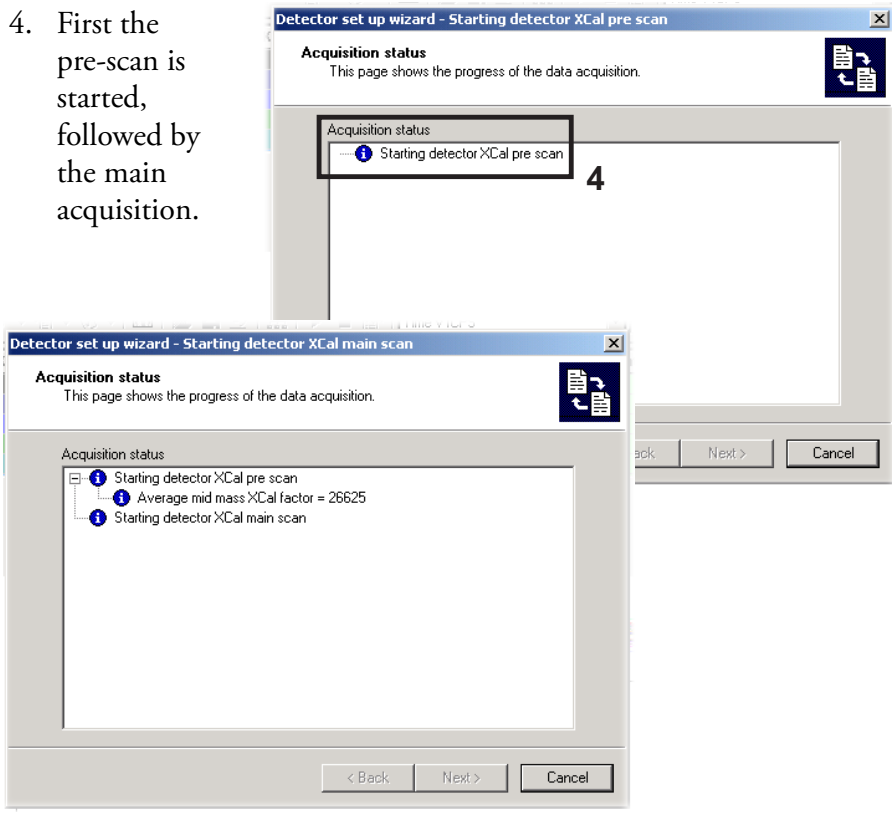


3. Click on **Next** to start the sample uptake.

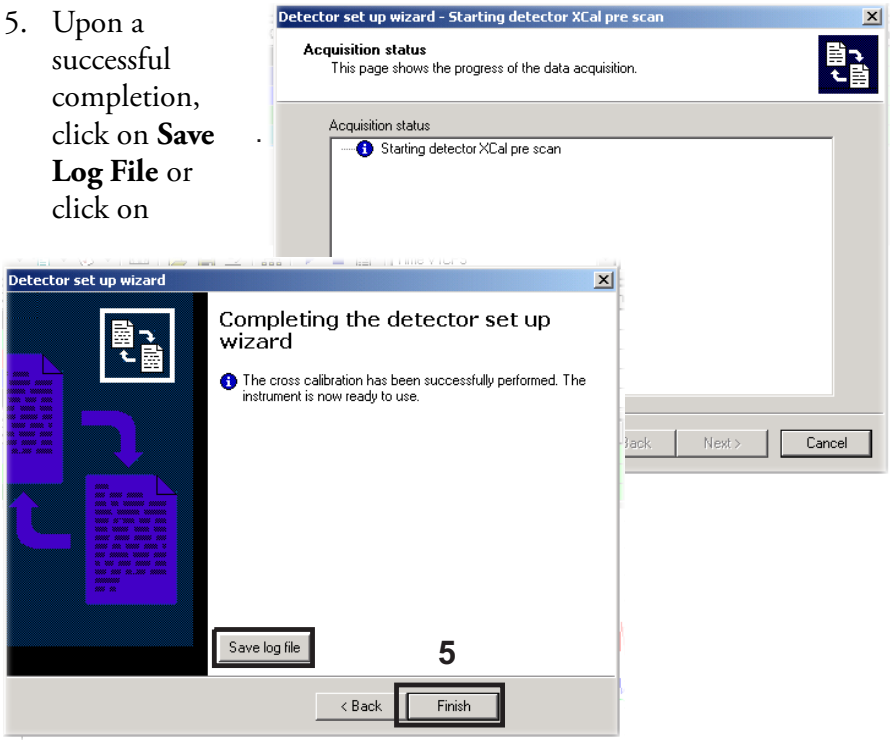


Start the calibration process by pressing the **Next** button. This will give a sample uptake timer which can be paused, if required. To begin the calibration before the timer completes, press the **Next** button. During the data acquisition for the detector calibration, the wizard will show the steps being undertaken whilst the Real Time Display will show the actual data acquired.

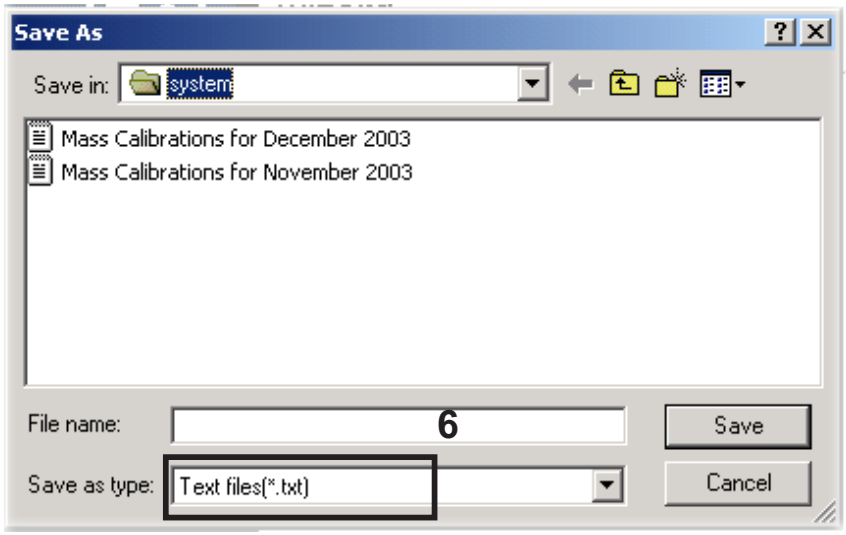
4. First the pre-scan is started, followed by the main acquisition.



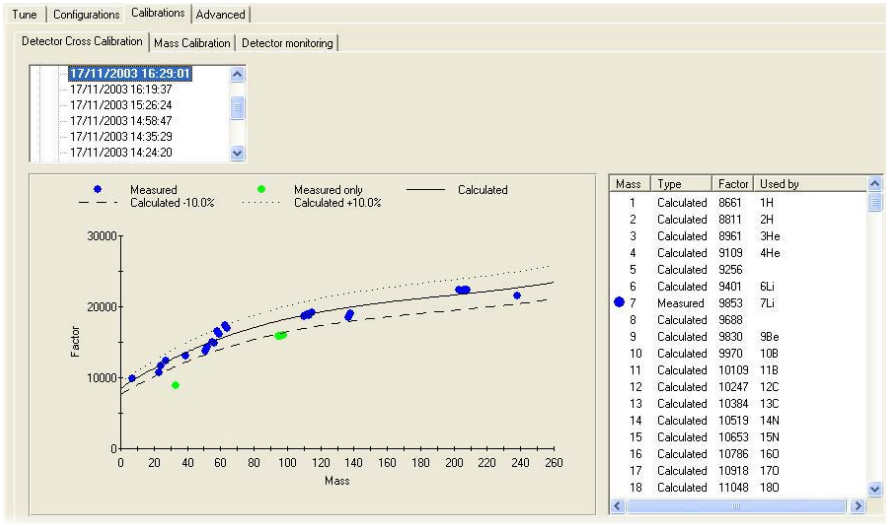
5. Upon a successful completion, click on **Save Log File** or click on .



6. Enter a filename to save the instrument calibration log file.



7. The *Detector Cross Calibration* can be viewed in the *Instrument* section.

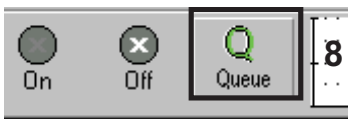


The detector cross calibration is used to convert the data acquired using the analog detector mode into the equivalent pulse counting data. The results of a successful calibration are shown on the *Detector Cross Calibration* page. This page is shown in both the *experiment* and the *instrument* views.

The current cross calibration is shown in bold at the top of the tree-list. If a new detector has been fitted, the old cross calibrations are archived under the “old calibrations” part of the tree. To view a cross calibration graph and its data, click on the **date and time** for the calibration in the list.

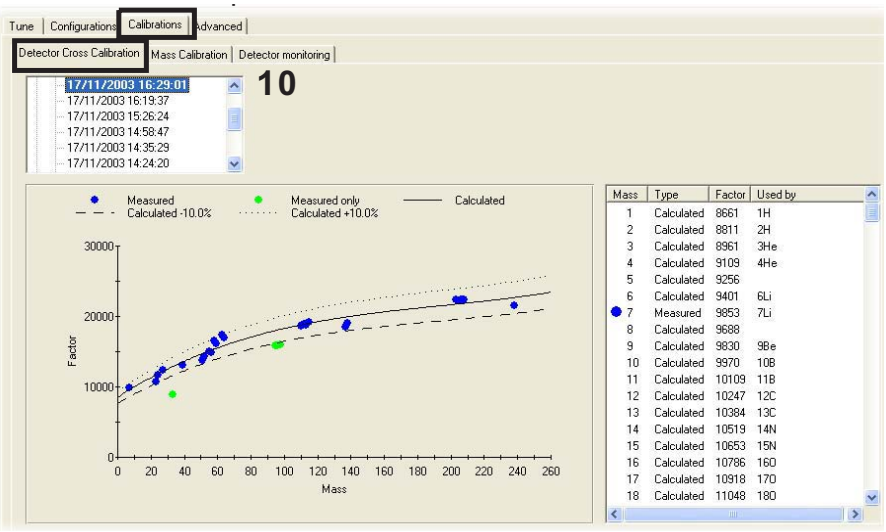
The graph shows the measured data points with error bars showing the +/- acceptable errors from the calculated curve. The raw data for each point can be seen by clicking on the point in the graph or on the row for a measured analyte in the table.

8. Before queueing the experiment, aspirate an appropriate solution (see solution required).



9. To view the results of the detector calibration, click on the **Instrument** icon.

10. Select **Calibrations** and **Detector Cross Calibration**. Click on the detector cross calibration that has just been performed, shown by the date and time stamp.



11. The graph will show the calculated cross calibration factor for each mass found on the y-axis against the mass on the x-axis. A polynomial fit will be applied through the enabled points. The cross calibration factor should be approximately 20,000–40,000 at mid mass.

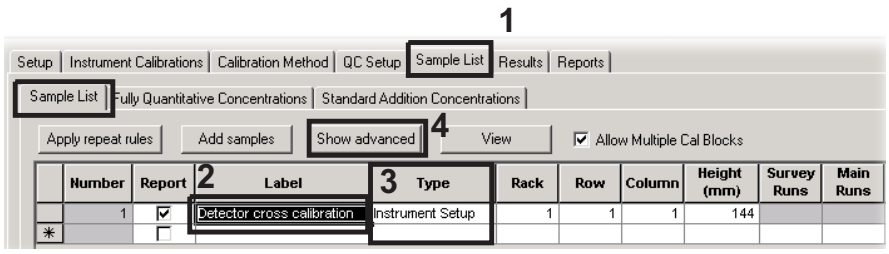
Note A non-matching cross calibration point can be viewed and edited by clicking on the point. All the values used to determine the point are shown, and the values can be edited to improve the fit, if required. More information is available in the online help. ▲

Alternately, the detector calibration can be performed via the *instrument setup sample*.

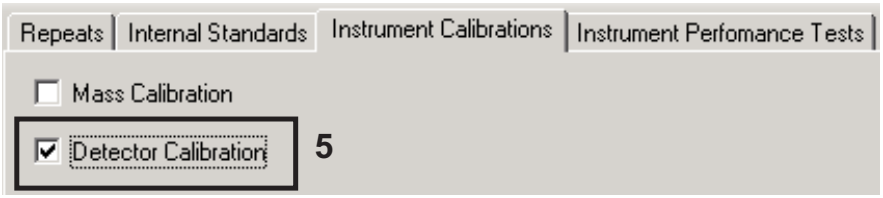
1. Click on **Sample List > Sample List** in an Experiment.

2. Enter a sample **Label** for the detector cross calibration.

3. Click in the **Type** column and then on the **downward arrow** and select **Instrument Setup**.



4. Click on **Show Advanced** to view the *Advanced Properties*.



5. To perform a *detector calibration*, check the appropriate box.

Note More diagnostic information on calibration is provided by using the *instrument calibration wizard* rather than the *Instrument Setup Sample* in an experiment. ▲

Step 2 - Mass Calibration

The software uses a mass calibration equation so that when a mass is selected for measurement, the control electronics can set the quadrupole to transmit that mass. The mass calibrations are stored such that any experiment can access them when the experiment is being run.

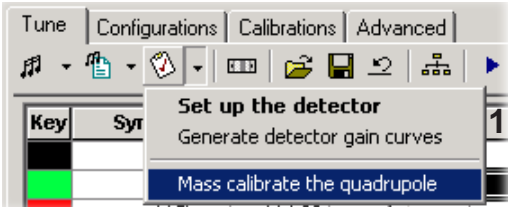
PlasmaLab uses the following default set of acquisition parameters for the mass calibration:

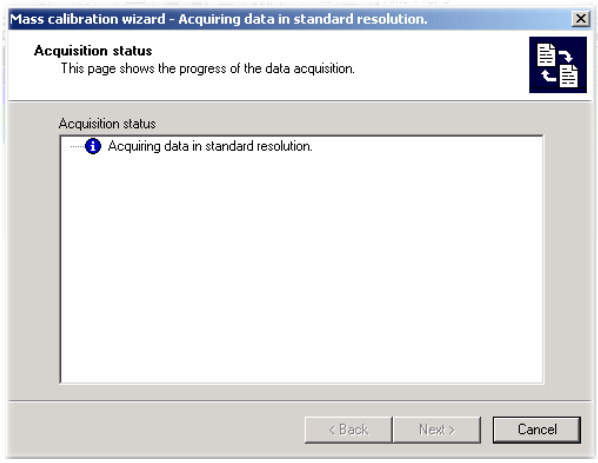
Acquisition Parameter	Value
Dwell	1000 µs
Channels per AMU	20
Scan Regions	4.5 to 10.5 23.5 to 27.5 50.5 to 78.5 81.5 to 245.5

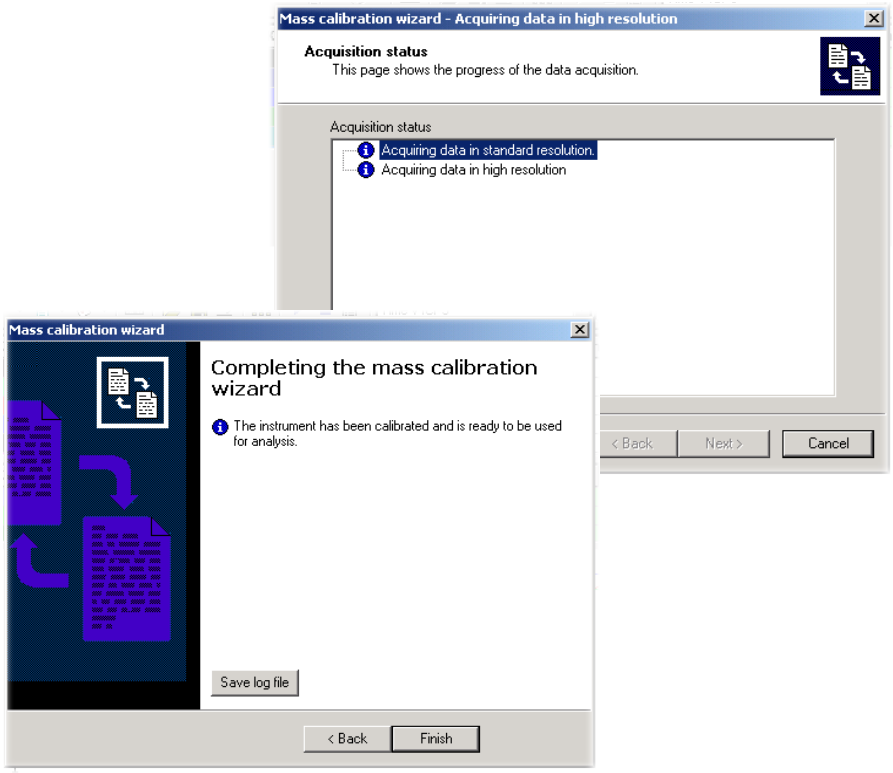
PlasmaLab converts the mass ranges into DAC values using the currently selected mass calibration. These parameters are then sent to the instrument which acquires 10 sweeps.

A mass calibration set comprises the mass calibrations for both the standard and high resolution quadrupole modes which are automatically done at the same time by the *mass calibration wizard* or as part of an *Instrument Setup Sample* in an experiment. Follow the mass calibration wizard step by step.

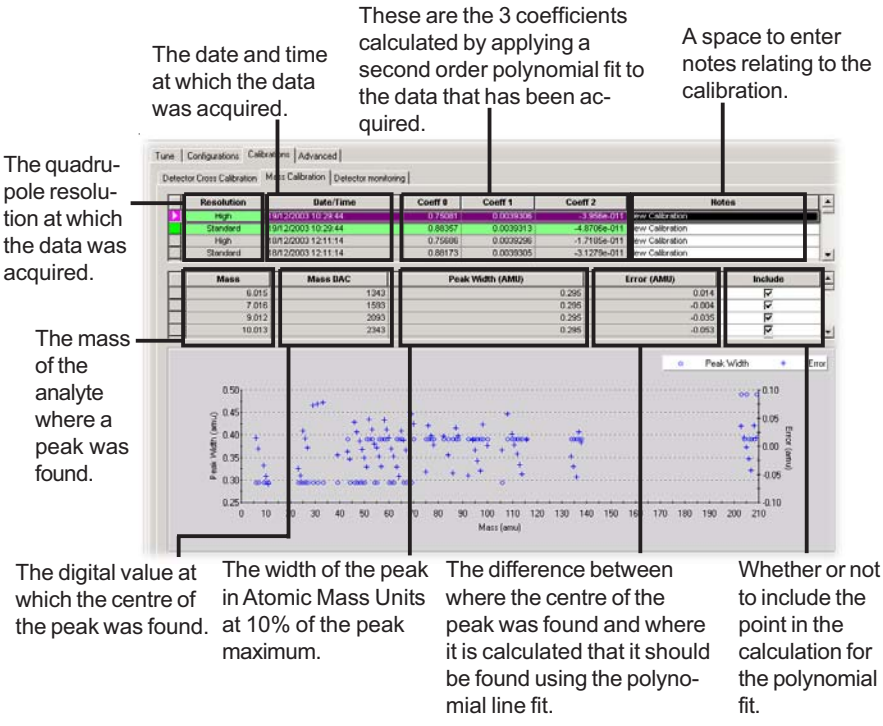
1. Click on the **Instrument Calibration Wizard** icon, then on **Mass calibrate the quadrupole**.







The mass calibration can be viewed in **Instrument > Calibration > Mass Calibration**.



The top grid lists all of the mass calibrations that have been successfully acquired and calculated. Clicking the right mouse button on the grid will show a menu that allows a mass calibration to be set as the current one.

The current mass calibration set is highlighted in green in the top grid. Clicking on any row will show the data for the mass calibration in the lower grid and the relevant graph.

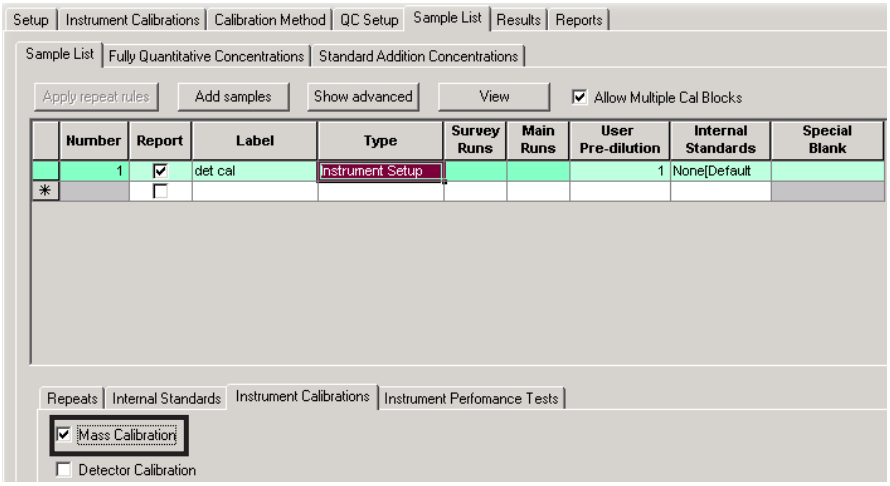
The graph will show the peak width and error between the found peak center and the calculated peak center on the y-axis against the mass on the x-axis for each mass at which a peak has been found.

Data points can be included or excluded by either clicking on the point on the graph or changing the state of the *include box* for the mass in the lower grid.

Every time an experiment is queued, the current mass calibration as selected in the *instrument view* is copied from the instrument database. The top grid lists all of the mass calibrations that have been copied into the experiment. Clicking the right mouse button on the grid will show a menu that gives the option to import a mass calibration from the instrument database and to apply a mass calibration to all of the data in an experiment.

Instrument setup samples can be placed anywhere in the sample list and they can also have repeat rules applied. However for most applications, only one will be required at the start of the experiment. The data collected by an instrument setup sample for the instrument calibrations or Autotune is also copied to the instrument database for use by subsequent experiments.

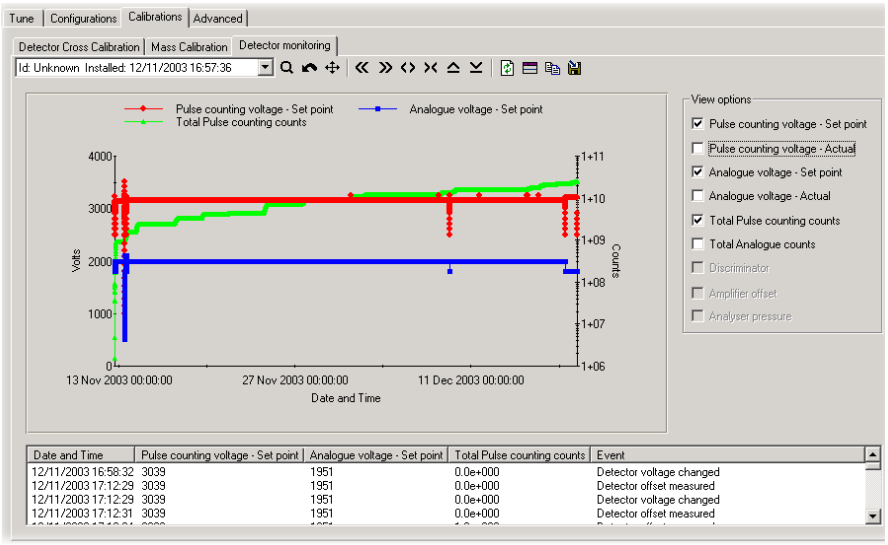
The *Instrument Setup* for the *Instrument Setup Sample* is divided into two parts: the first enables the mass calibration and detector setup, whereas the second part enables the performance report and Autotune.



Once the calibrations have been completed, the data can be viewed either in the experiment that has just used the Instrument Setup Sample under the *Instrument Calibrations tab*, or a complete list of all calibrations can be viewed in the Instrument section of the software under the *Calibrations* tab.

Step 3 - Detector Monitoring

After a new detector has been installed and the automatic outgas procedure has been completed (see Chapter 7: “Maintenance”), the detector will be monitored throughout its lifetime. The *Detector Monitoring* page will show the information for the new detector. The various parameters can be displayed using the *Options* on the right of the page.



The software will track various events during the lifetime of a detector for diagnostic purposes. The various parameters measured can be viewed on the *Calibrations* pages of the *Instrument* section. Should a detector lifetime issue arise, this information will be able to help track the cause of the problem.

Chapter 4 Sample Analysis

Objective

The objective is to set up an experiment for the analysis of standards and unknown samples. Calibration graphs will be created showing the quantitative and semi-quantitative results.

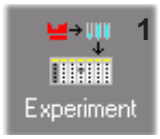
Solutions required for this practical

- 2% HNO_3 blank and unknown in 2% HNO_3 matrix.
- A series of standards at concentrations of 5 ppb, 10 ppb and 20 ppb from a tuning solution (e.g. CLMS-2) containing 1 ppm of the following analytes: Li, Be, Co, Ni, In, Ba, Tl, Pb, Ce and U.

  Add internal standard solution containing Ga, Rh and Bi at 10 ppb.

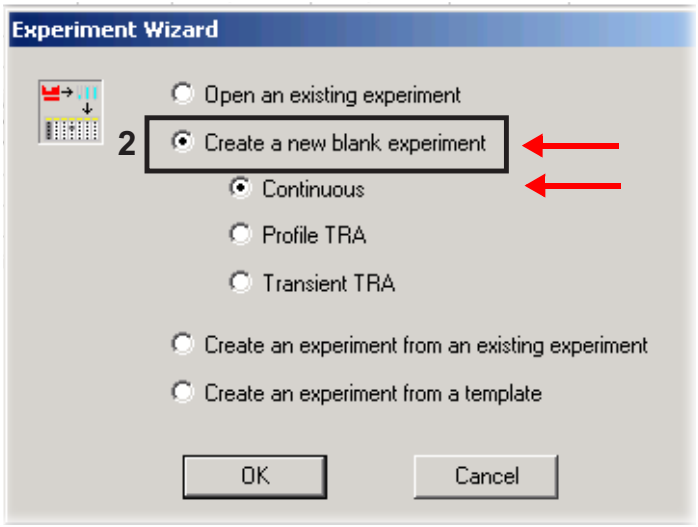
This chapter treats the following topics:

Step 1 - New Experiment Creation



1. Click on the **Experiment** icon to open the *Experiment Wizard*.

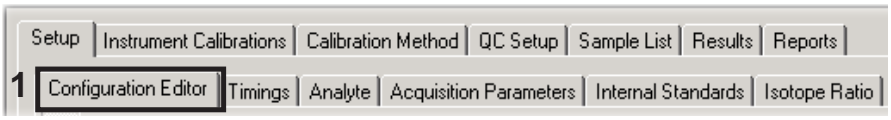
2. From the Experiment Wizard select **Create a new blank experiment**. Select **Continuous** mode of operation.



3. You will be automatically prompted to choose a database. Select **Default.tea** as the analyte database for this experiment.

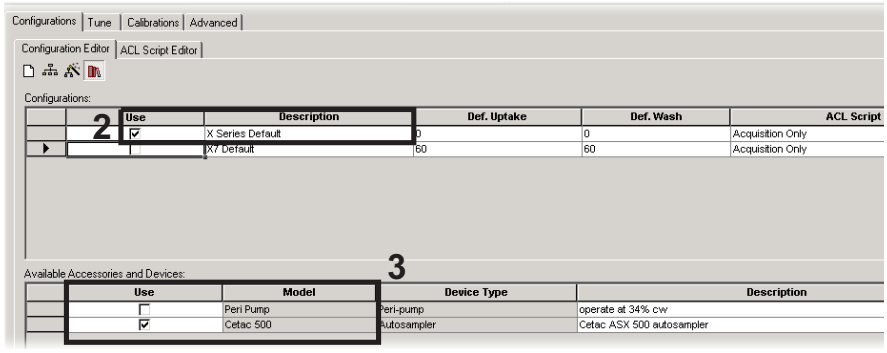
Step 2 - Experiment Setup

1. In the selected new experiment click on the **Configuration Editor** in the **Setup** page to select the correct configuration.



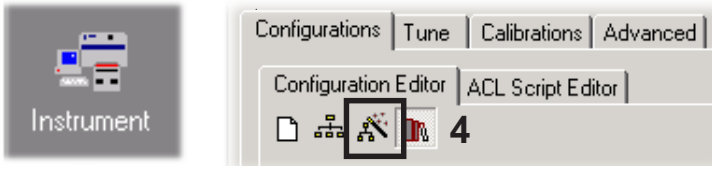
2. Click on the **Use** box to select the *standard default configuration*, if not already selected.

3. Ensure that the correct *accessories* for the experiment are visible as shown (e.g. for a peristaltic pump for manual operation or an autosampler for unattended operation).



4. If an accessory is not shown for the experiment, it must first be set up using the **Instrument** icon and the *Accessory Wizard* in the **Configuration Editor** page.

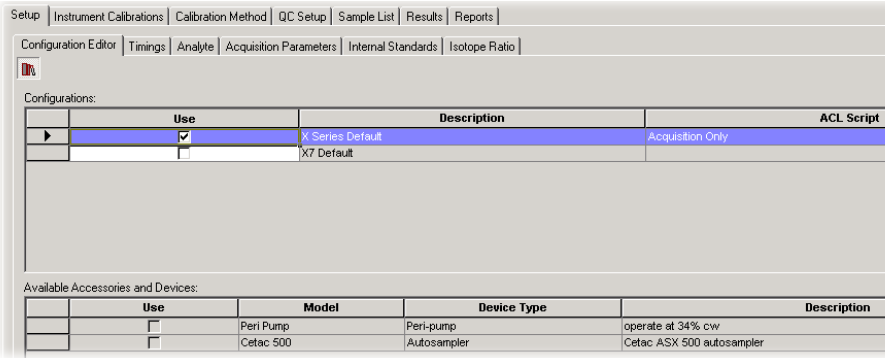
   This will help you to select and configure the available accessories, which will then be available when opening new experiments. The accessories are setup in the instrument and shown in the *Experiment* section only for reference purposes.



5. The **Instrument settings** box can remain unticked, if only one set of instrument settings is being run. This will enable the experiment to use the last set of saved instrument settings.

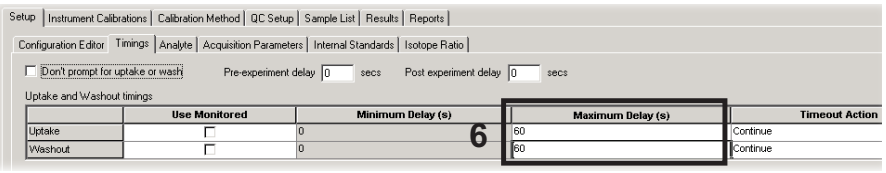
   If different settings are used (that is, *Cool Plasma* and *Hot Plasma* mode), the relevant instrument settings should be ticked in each experiment. If not available, these can be set up and edited in the *Instrument Control*.

Note A **Delay time** can be inserted to allow the instrument to settle between settings. ▲



6. Select the **Timings** tab in the *Experiment Setup* to select the **Uptake time** as the time taken for the sample to reach the plasma and **Washout time**, as appropriate.

Note Timings can be shortened considerably by the use of monitored uptake and wash (which can be selected here by ticking the **Use Monitored** check boxes and selecting a mass, such as the internal standard, whose signal can be monitored) and rabbit (fast pumping) on uptake and wash. The *rabbit* option is set in the *Instrument* section using accessory control language (ACL) scripts. See the online help about editing ACL scripts. ▲



7. To select the analytes which are required to be measured, click on **Analyte** on the **Experiment Setup** page.

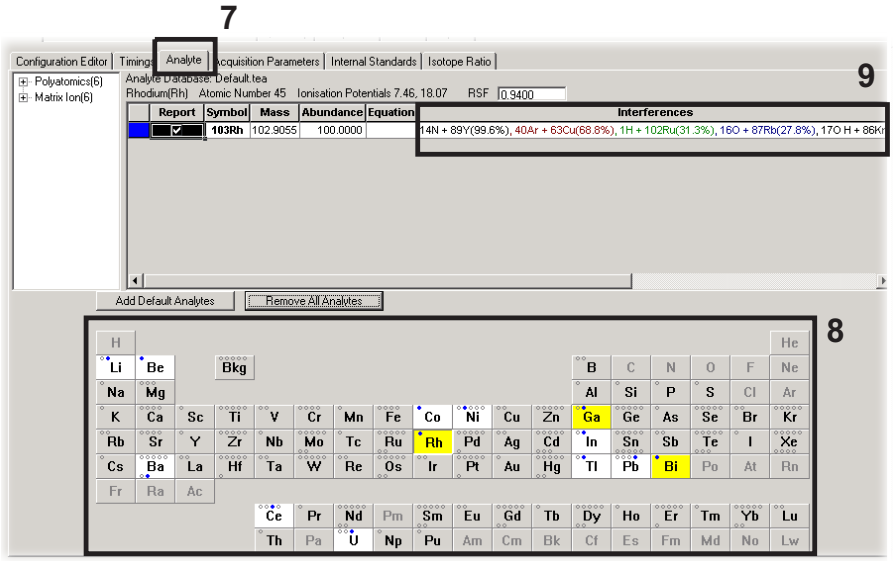
8. Select all analytes required by double clicking the left mouse button on the analytes required. This selects the default isotope with the least interference. These default isotopes are setup in the analyte databases.

   Alternatively, a single click will display a choice of isotopes which can be manually ticked for selection. The number of isotopes selected will be highlighted as dark blue dots above the analytes. The light blue dots automatically appear, if analytes are required for an interference correction equation.

   Select all analytes that are required for use as internal standards. These will appear as yellow when selected as internal standards (see

step 4). To remove an analyte, use the right hand mouse button menu over the analyte in question.

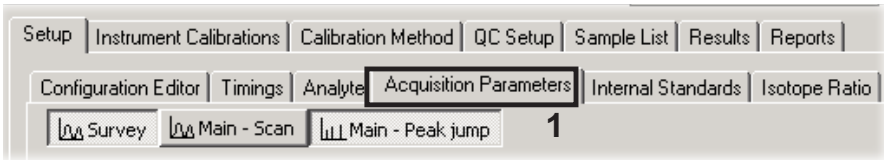
9. To disable any interference correction equations not required, right hand mouse click on the row in the grid with the equation and choose the **Disable Equation** option.



Step 3 - Acquisition Parameters

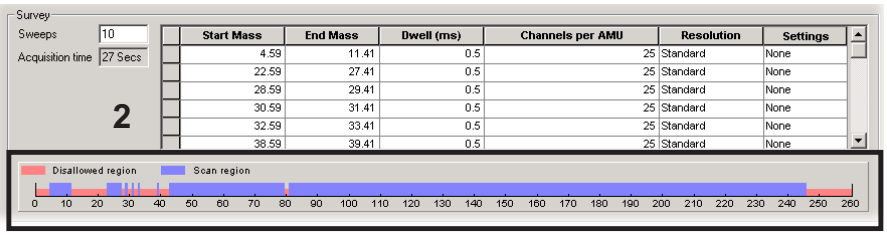
1. In **Experiment Setup** click on **Acquisition Parameters** to set up the survey and main run for the acquisition. The *Main run* selection can be either *peak jump* or *scanning*, depending on the application being run. The light gray areas show the selection made.

   Survey can be performed on all samples as a backup to the Main run, from which semiquantitative data can be obtained. It can also be used to identify interferences. The Main run can be selected either as *Peak Jump* for best detection of a small number of isotopes (< 20) in the shortest time or as *Scan* for full spectral information.

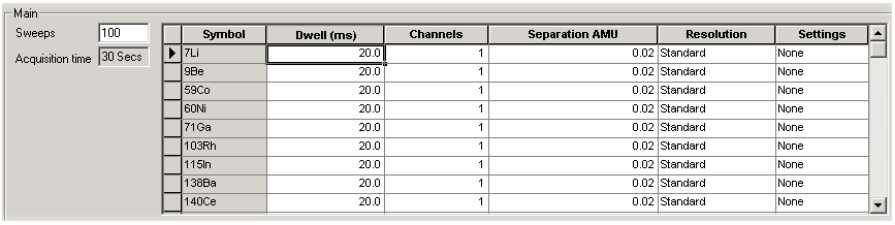


2. The default parameters for the *Survey* are shown below. If required, the scan region can be adjusted by a right mouse click to add or remove scan regions on the mass scan graphic.

Alternatively, the values in the numerical grid can be edited, and the changes will be repeated in the graphic.



3. The Main run *Peak jump* default parameters are shown below. *Peak jump* mode is the preferred method for fast acquisition with best detection. All the analytes selected in the *Analyte* menu will automatically appear in the symbol column.



4. If multiple tune settings have been chosen for the experiment, they can be associated with different analytes or scan regions here. The order of the settings and any stabilization times between tune sets is set up on the *Configuration* page of the experiment.

   It is possible here to choose which of the quadrupole's two *Resolution* settings are to be used for each analyte or region. Typically the resolutions are set to 0.8 and 0.4 amu at 10% peak height. The narrower peak (higher resolution) will improve interference removal for small peaks next to very large ones and will also extend dynamic range even further for high concentration analytes.

Step 4 - Internal Standard Selection

1. In the **Setup** menu click on **Internal Standards** to select internal standards for the experiment.

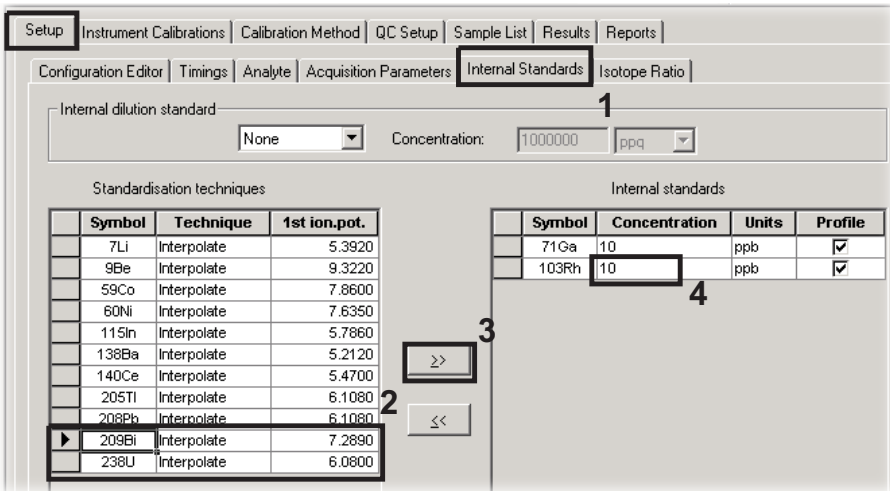
Note It is recommended to select an internal standard at low, middle and high mass for a multi-element analysis. This ensures that any changes in response due to drift or matrix suppression/enhancement are compensated for. ▲

Internal standards are selected that are (if possible) close in ionization potential to the analyte masses selected, near to 100% abundance and not present in the samples. To select an internal standard, these must be present in the *Analyte* menu for them to appear under the *Symbol* column.

2. To select an internal standard, highlight the required internal standard as shown above for Bi (Be and In have already been selected).

3. Click on the forward arrows to move the analyte across to the *Internal Standard selected* box on the right hand side.

4. Enter the concentration for the internal standard.



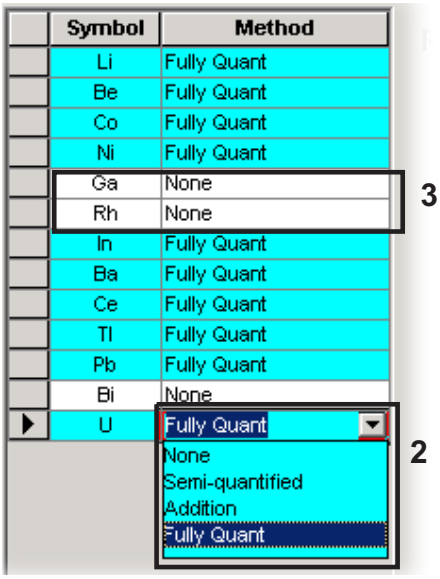
5. The default internal standardization method is *Interpolate*. Interpolate takes the response of two internal standards (that is, ^{9}Be and ^{115}In) and interpolates a linear response between the two masses. That is, if ^{9}Be is 80% of its reference value and ^{115}In is 90% of its reference value, then an analyte at mass 62 amu (Ni) would have an interpolated value of 85% for the correction.

   By default, all internal standards are added to the profile for interpolation. It is possible to remove internal standards from the profile so they may be used only as reference points for specific analytes (e.g. ^{197}Au for ^{202}Hg correction). Even internal standards that are used in interpolation can also be used for specific references.

   To specifically reference an internal standard to an analyte, choose it in the drop down menu in the *Technique* column.

Step 5 - Calibration Method Setup

1. From the menu, select **Calibration Method**. It is possible to select different calibration methods for each analyte.



2. Click on the drop down arrow next to each analyte to display the dropdown list.
   Select the *calibration method* of choice as shown below for each analyte.

3. For the internal standard analytes select **None** in the method column.
   By selecting *None*, a concentration file and calibration will not be assigned to these analytes.

4. In the right hand table under the *Calibration Method* menu, select in the **Forcing** column the option **Through blank** by clicking on the downward arrow to display the dropdown list. Select the **Forcing method** of choice for each isotope. The *Through blank* option is recommended.

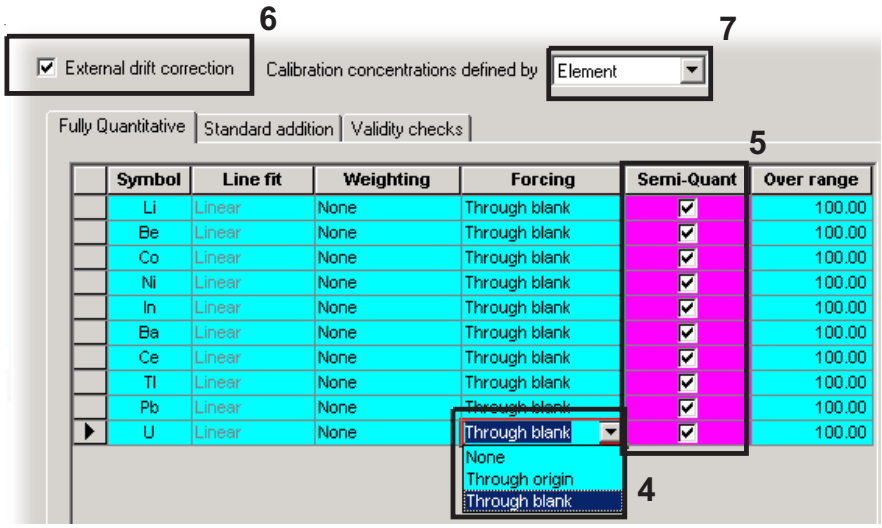
5. Tick the boxes in the **Semi-Quant** column to select the isotopes that will be used to create the response curve. This will then be used for semi-quantitative analysis, if required.

   To build a reasonable *semi-quant curve*, you need only choose 3–5 analytes that cover the mass range of interest and are easily ionized in the ICP (e.g. Li, Co, In, Tl and U would build a good curve, representative of the mass response of the XSERIES 2 ICP-MS).

6. Select the **External drift correction**. Calibration blocks are set up, typically every 10–20 samples, and a drift correction can be applied between them, if this check box is ticked.

Note To enable external drift correction to be performed on all samples, there must also be a calibration block at the end of the sample list. ▲

7. Select the Calibration concentrations by **Element**. They can also be defined by **Isotope**, but this is only for use when non-natural isotopic abundances occur.

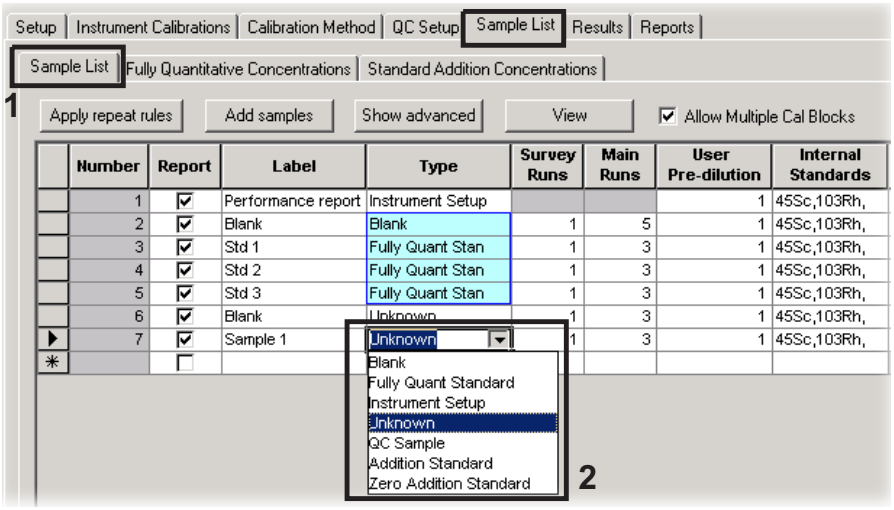


Step 6 - Creating a Sample List

1. Select the **Sample List** and then again **Sample List**.

2. Define the **Blanks**, **Standards** and **Unknowns** using the dropdown list as shown. The blank and standards will turn light blue, this is the *Calibration block* and can be copied at the end of the sample list and used for *External drift correction*.

   Highlight any sample in the calibration block to be copied and right mouse click to *append copy* - a copy of the whole block will be made.

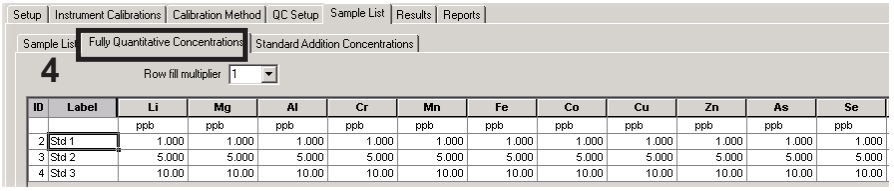


3. Select the number of **survey runs** and **main runs** as shown.

4. Select the **Fully Quantitative Concentrations** for all analytes in all standards. Enter **standard concentrations**, use drag and drop to copy across the columns.

   The *row fill multiplier* is very useful when working with multi-element standard stocks. Define the concentrations for standard 1 and then change the multiplier to 10. Select all the cells in the first row and then move to the end of the row.

   Click on the bottom right corner of the selected cells and drag the box down to standard 3. The rows will then be filled as x10 and x100 of standard 1.

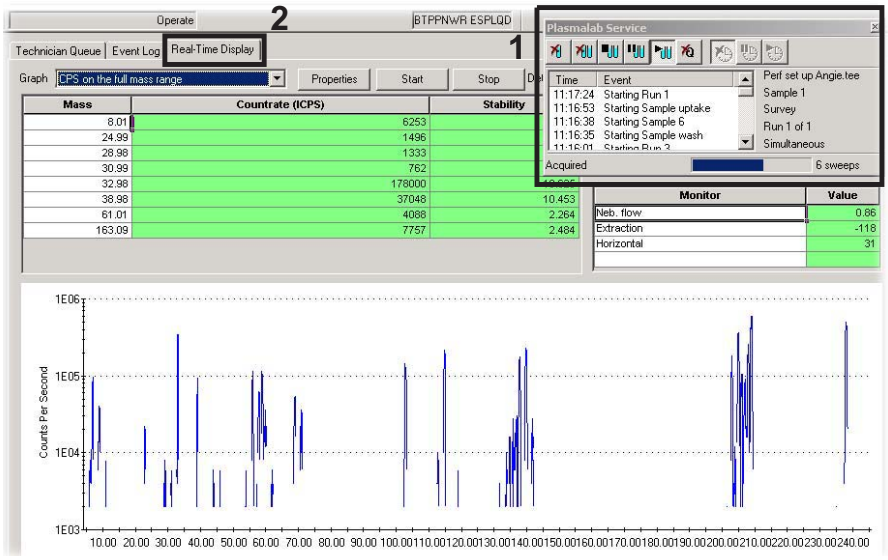


5. Click on **File** in the *Menu* bar and select **Save as** to save the current experiment.
   Alternatively, click on the **Queue** button to start the acquisition, and for any unsaved files, it will ask for the experiment to be saved.

Step 7 - View Data Acquisition

1. To monitor data acquisition, double click on the **MS** icon in the lower right corner of the screen. This will show the *PlasmaLab Service* window.

1. The *Real Time Display* can be monitored during the acquisition via the **Technician** page.

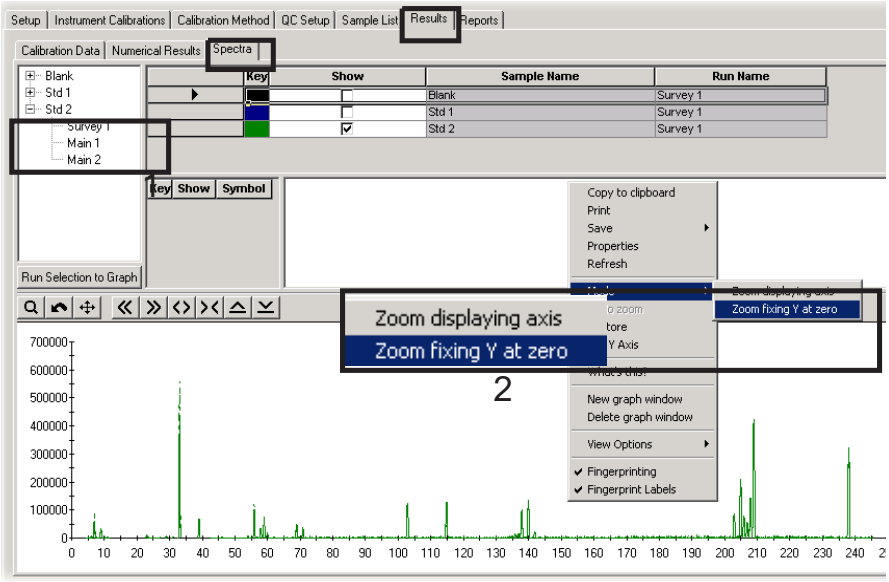


Step 8 - Viewing Spectra

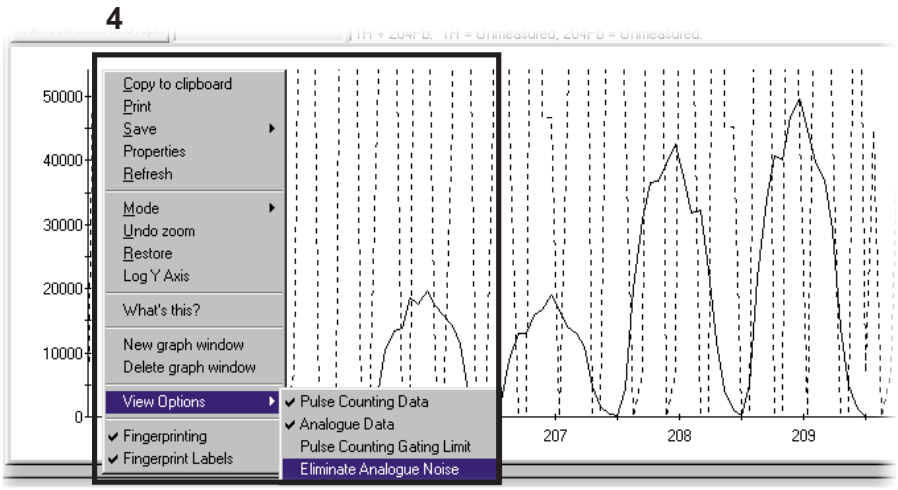
1. After the Survey run is completed, the results can be viewed. Go to **Results > Spectra**. A spectrum can be viewed by clicking on the cross next to the sample (that is, *survey and stability run* to enlarge the menu). Then double click on the **Survey run**. The graph below shows the complete mass range for the Survey run.

2. Select a *zoom option* by clicking the right hand mouse button on the graph and choosing **Mode** from the menu. Then choose **Zoom fixing y at zero**.

3. Zoom in on an analyte you wish to view more closely (e.g. Pb peaks). Hold down the left mouse button and drag over the Pb peaks (204–210 amu).



4. Now the mass spectrum shows a zoom-in of the mass region. The spectrum displays both the pulse counting and the analog data. Click once using the right mouse key to show the dropdown menu.

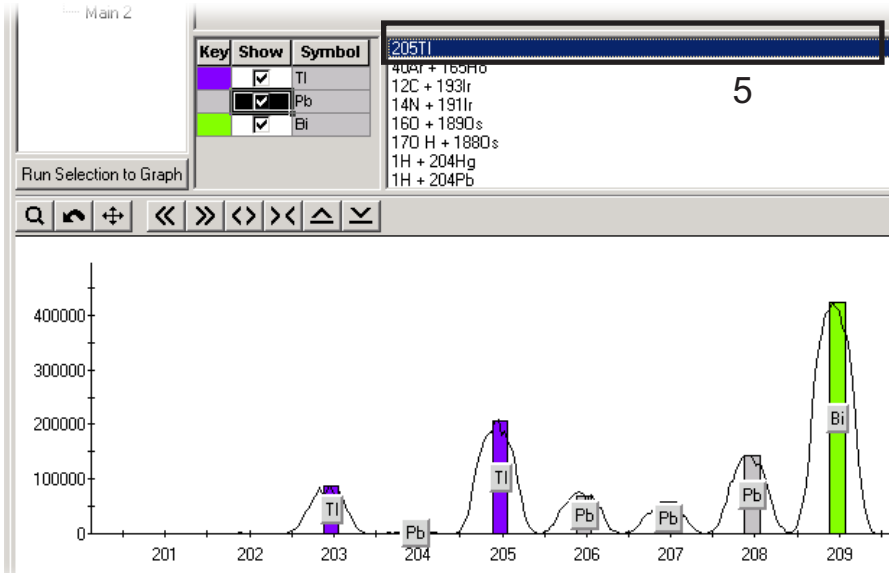


The dropdown menu gives a list of options. You can either **Undo zoom**, this will step back to the previous zoom or **Restore** to return to the original full mass region.

The spectrum can be viewed for either detector mode, or the detector analog noise can be eliminated. To eliminate the analog noise, place the cursor over **View Options**. Another dropdown menu will be displayed. Therein select **Eliminate Analogue Noise**.

5. To identify the analytes with respect to their isotopic fingerprint, click on the peak of interest. Double click on the element of interest or most likely interferent.

Note Fingerprinting only works effectively, if the analog noise has been removed. ▲



Step 9 - Semi-Quantitative Results

Any standard entered for fully quantitative analyses can also be used to establish the semi-quantitative response curve. Semi-quantitative results can be obtained for any analyte found in the Survey or Main runs that does not have a standard calibration, by using the response curve.

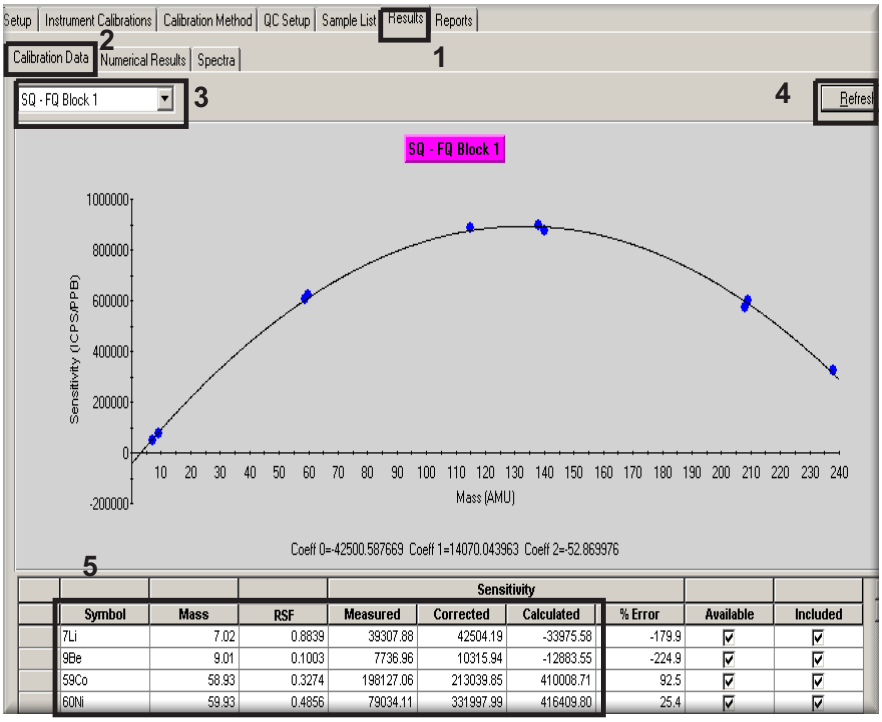
1. Click on **Results**.

2. Click on **Calibration Data** to view the results.

3. Select the **SQ-FQ Block** from the dropdown menu.

4. The *response curve* is displayed. Any point of the curve can be excluded by clicking on it (when excluded, it will displayed in red). The response curve values in the grid can then be recalculated by clicking on **Refresh**.

5. The *results* of the mass response are displayed in the table.



6. The accuracy of semiquant values can be refined by adjusting the *RSF value* of the analyte. This *Relative Sensitivity Factor* is the measure of the analyte's response in the ICP-MS compared to the theoretical response. The RSF will change for different plasma and sample introduction conditions. It can be useful to build a library of values for different conditions.

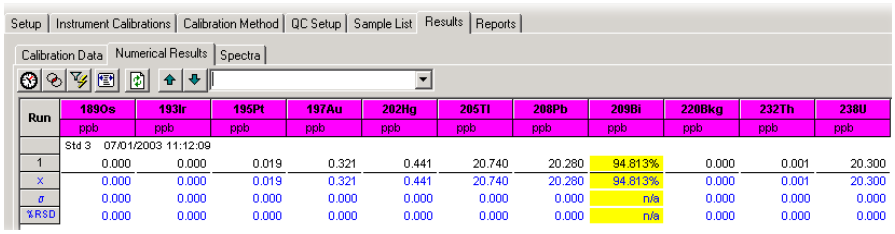
   If a calibrated analyte has not been used to build the semiquant curve, it is still displayed and, if necessary, its correct RSF value can be recalculated.

   To recalculate a RSF value, first **deselect the point** to be recalculated by clicking on it with the left mouse button. Then click on the right hand mouse button to select the *dropdown list* and select **Calculate RSF**. The corrected RSF values can be exported to any analyte database for later use with the *Analyte Database Wizard* on the tool menu.

7. Click again in the RSF cell with the left mouse button to display the corrected RSF value.

8. The results of the semi-quantitative analyses of the entire mass range are shown in purple (selected from **Numerical Results** and then **Survey Analyte Dilution Concentration**). 20–30% accuracy can

be expected for semi-quantitative analysis, when appropriate RSF values are used (see step 7).



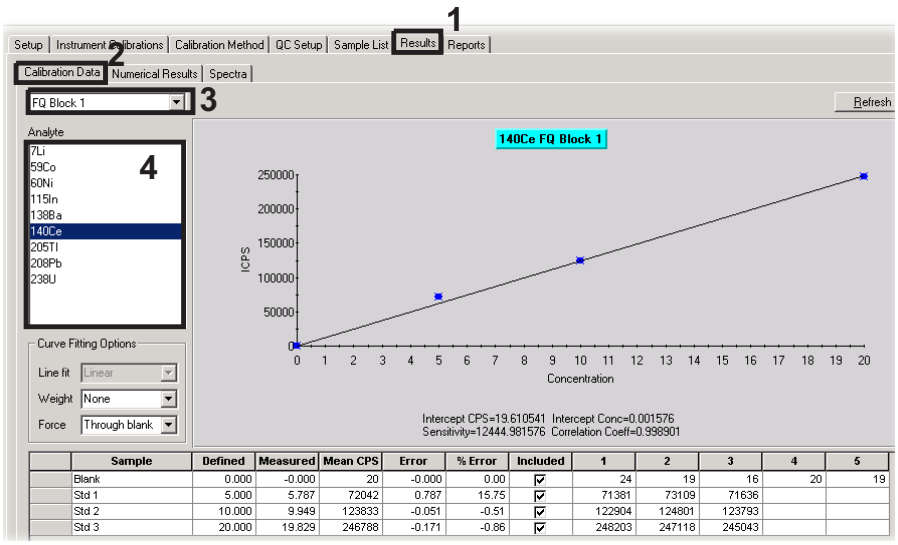
Step 10 - Fully Quantitative Results

1. Click on **Results**.

2. Click on **Calibration Data** to view the results.

3. Select the **fully quantitative calibration block** for viewing.

4. Select the **analyte** to view its calibration graph.

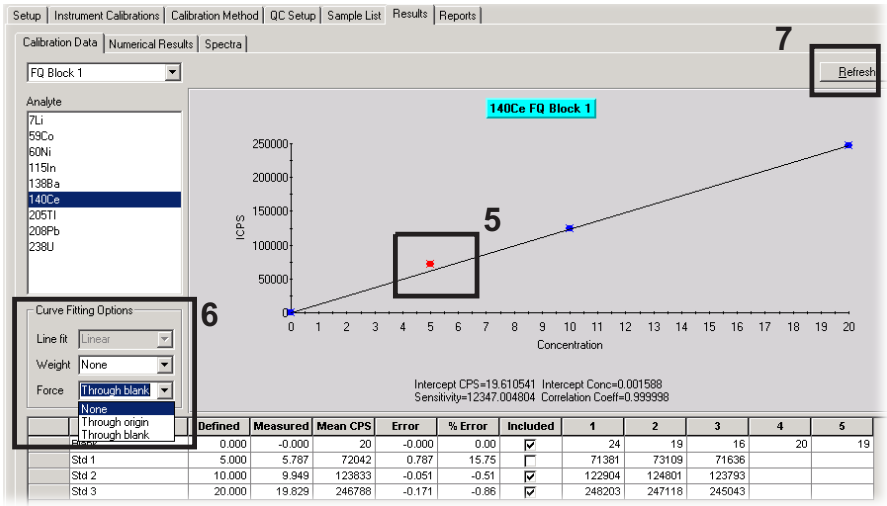


5. Erroneous points can be excluded from the calibration by clicking with the left hand mouse button on the respective point as illustrated for Ba Std 1. The point changes red when excluded.

6. Using the dropdown menus in the **Curve Fitting Options** box, a choice of weighting the calibration can be made. Default is none.

The calibration can also be modified to force through zero, through origin or through blank. The latter is recommended.

7. Changes to weighting or forcing can be seen immediately on graph. Click on the **Refresh** button to change the grid underneath and *recalculate* the whole database.

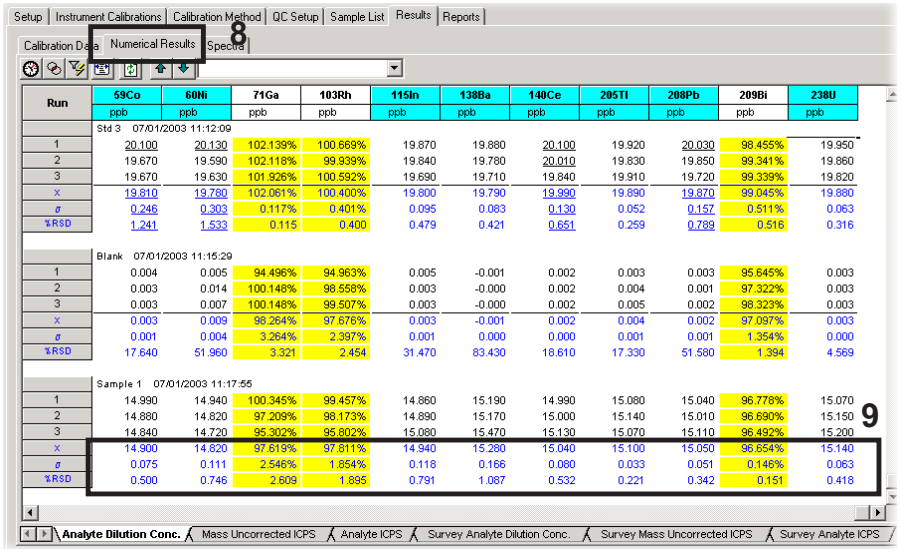


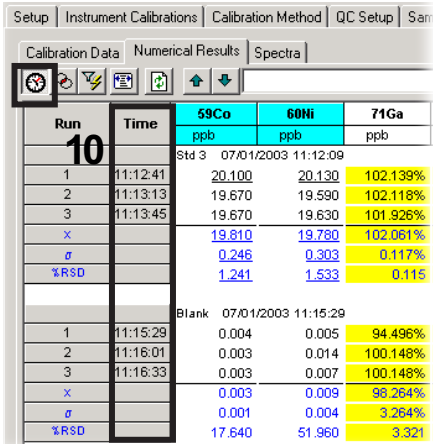
8. Click on **Results** and then on **Numerical Results** to view the results. The concentrations can be viewed in the *analyte dilution concentration* file. The data, mean, SD and %RSD are displayed for all standards and unknown samples.

9. Check the accuracy of the data.

Note The %RSD should in general be less than 2% (where the concentration is equivalent to approximately > 300 times the standard deviation of the blank). ▲

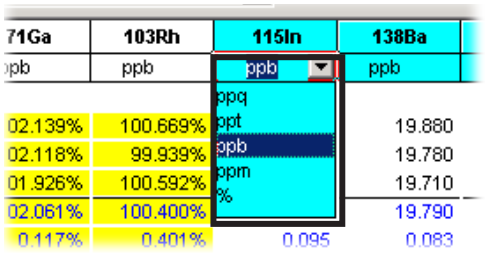
Any points excluded from the calibration are shown as shaded regions. To exclude erroneous data points, right mouse click over the cell to be excluded and select **Exclude**.





10. The results display can be modified to display the *run times*.

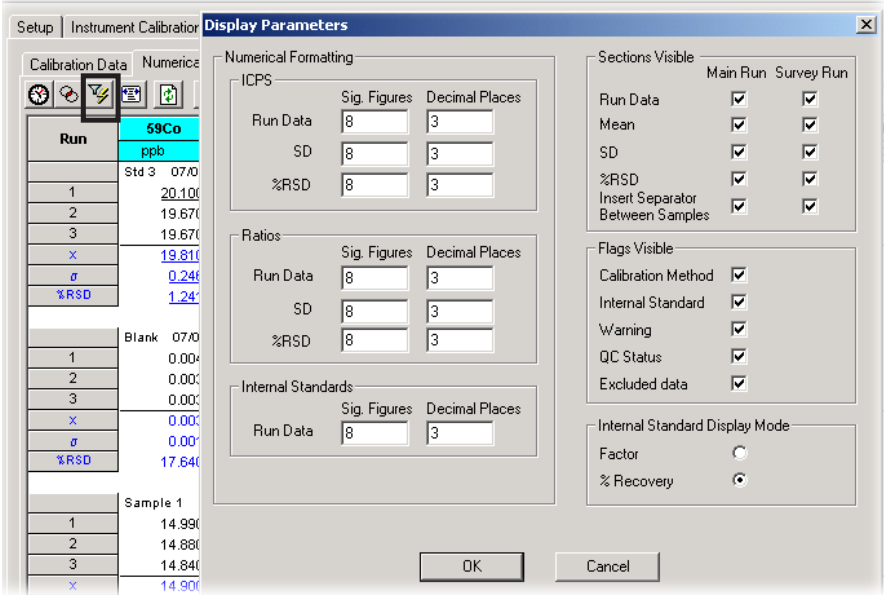
    For this purpose, click on the icon shown.



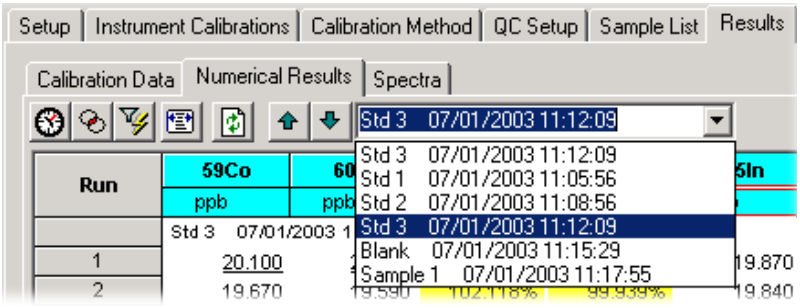
11. Unit sets can be adjusted for individual elements by using the dropdown menu in the cell below the element.

12. The display parameters can be modified using the **Edit display parameters** icon.

12

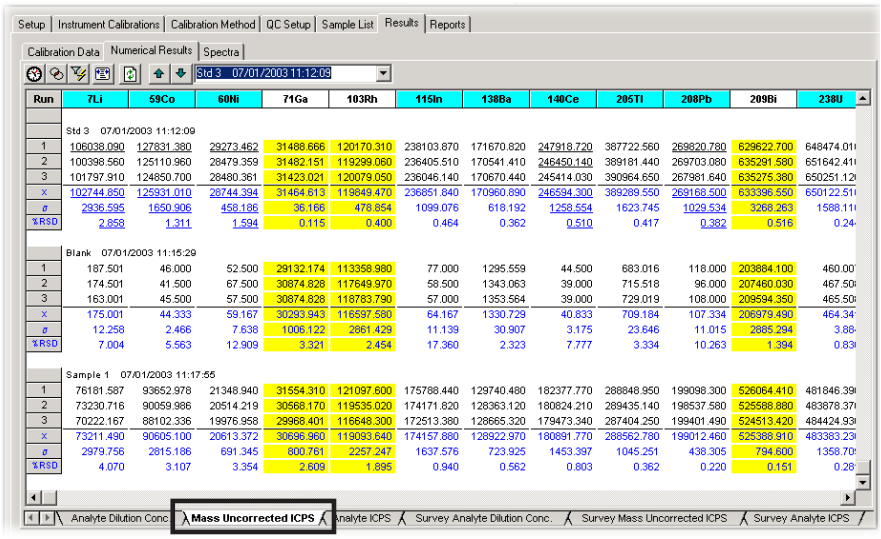


13. Using the drop down menu as shown below, the sample list is displayed, and sample results can be selected.



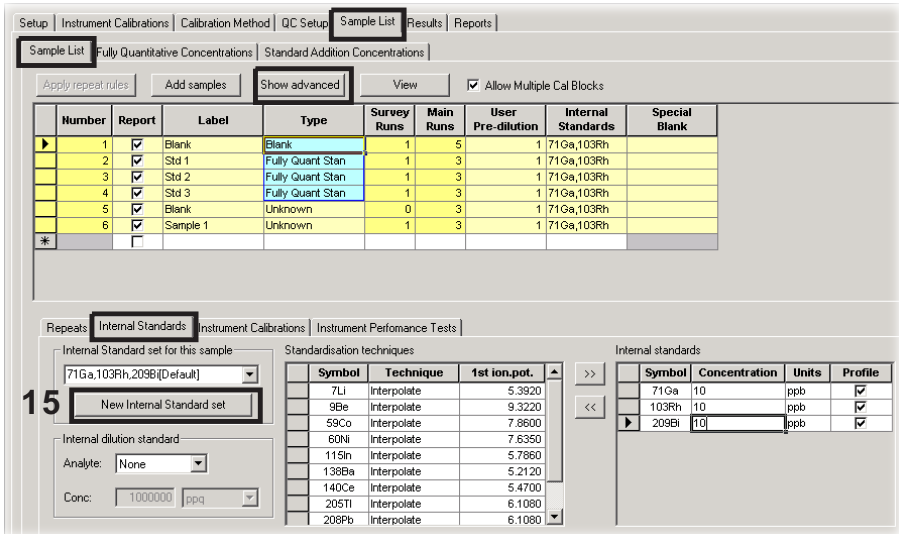
14. The *internal standard* is shown in yellow. The table below shows the mass-uncorrected ICPS and can be used to check that the response does not change significantly throughout the run.

    The internal standard can be displayed as the Internal Response Factor (used in the calculations) or % internal standard recovery (which is the reciprocal of the ISR, expressed as a percentage). The % recovery is the value most popularly monitored by analysts throughout the world.



15. To make any changes to the internal standard (that is if the internal standard is at a higher concentration than expected or unexpectedly in a sample and needs to be deselected), these changes can be made in the *Sample list*.

    Click on the sample that needs correcting and select **Show advanced**. In the *Internal standard* folder select **New Internal Standard set for this sample**. Any changes in concentration or selecting different internal standards or deselecting internal standards can be made. A new name will be set for the internal standard for this sample.

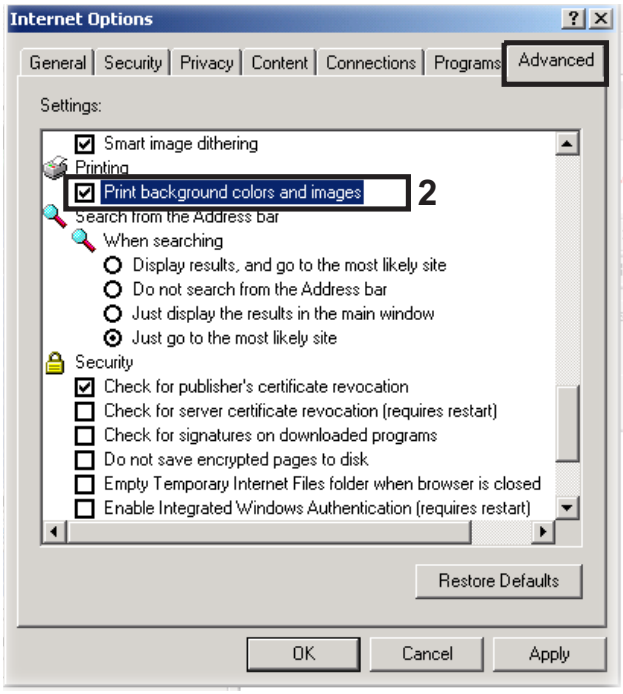


Step 11 - Printing Report

Microsoft Internet Explorer 6 is required to preview and print reports. Internet Explorer should be set up to enable printing of colored backgrounds.

1. This is set up in **Internet Explorer > Tools > Internet Options**...

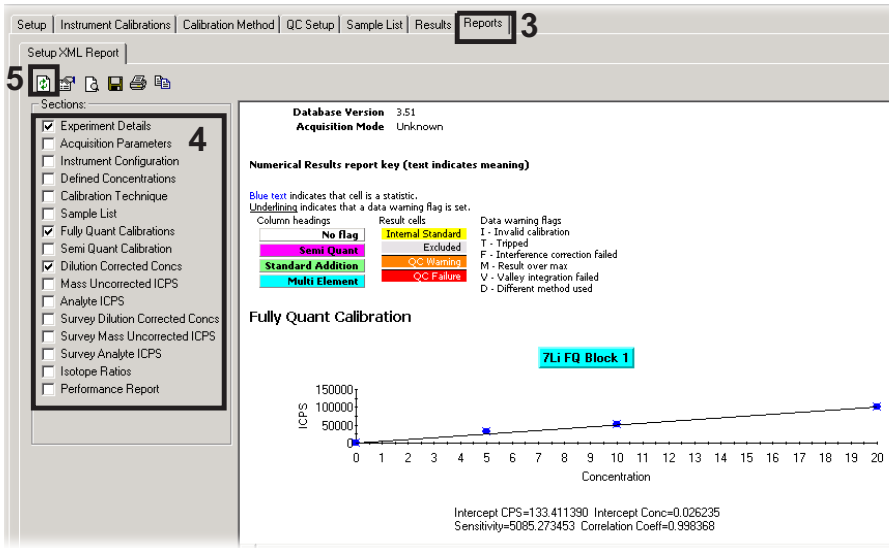
2. In **Internet Options**, select **Advanced**. Scroll down to **Printing**. Ensure that the box **Print background colors and images** is ticked.



3. To display the results for printing, click on **Reports**.

4. Select the *sections* which require reporting by ticking the relevant boxes.

5. Click on the **Refresh** button to display the report on screen.



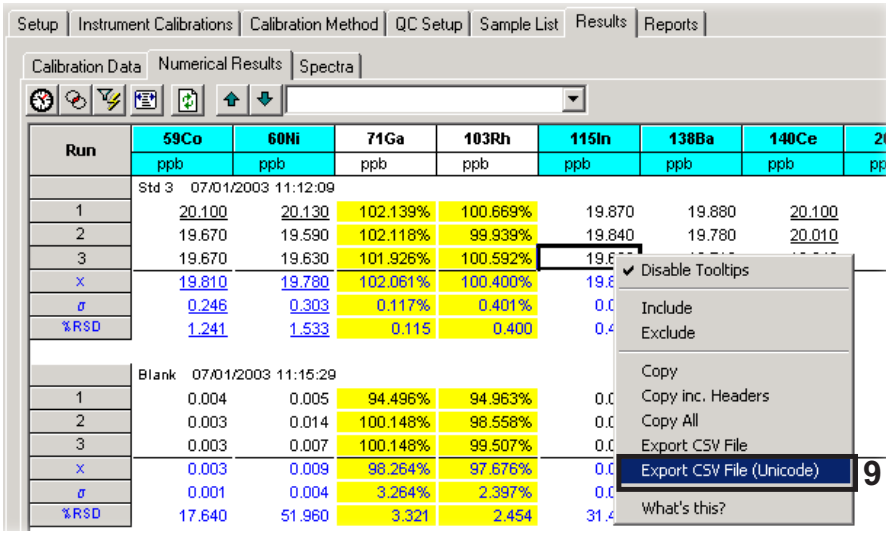
6. Click on the **Configure the reports settings** icon to enable a preview in PlasmaLab. Alternatively, open the report in a Windows-associated package and click on **Refresh** to view the report.

7. Use the **Save** icon to save the report in the appropriate folder.

8. Results can also be copied and pasted from the *Results* page: highlight the results and right mouse click to copy with headings. Then paste into a Windows-associated package, that is Microsoft Excel or Microsoft Word.

9. It is possible to export the data directly to a *.csv file (comma separated variable) from the *results grid*'s right hand mouse menu. This can be used for LIMS or Microsoft Excel data processing.

   The data is exported as Unicode characters to allow international characters to be recognized. Import into Microsoft Excel requires the Excel wizard to be used to choose comma as the variable separator.


Setup
Instrument Calibrations
Calibration Method
QC Setup
Sample List
Results
Reports
Calibration Data
Numerical Results
Spectra
Run
59Co
60Ni
71Ga
103Rh
115In
138Ba
140Ce
ppb
Std 3 07/01/2003 11:12:09
1 20.100 20.130 102.139% 100.669% 19.870 19.880 20.100
2 19.670 19.590 102.118% 99.939% 19.840 19.780 20.010
3 19.670 19.630 101.926% 100.592%
x 19.810 19.780 102.061% 100.400%
σ 0.246 0.303 0.117% 0.401%
%RSD 1.241 1.533 0.115 0.400
Blank 07/01/2003 11:15:29
1 0.004 0.005 94.496% 94.963%
2 0.003 0.014 100.148% 98.558%
3 0.003 0.007 100.148% 99.507%
x 0.003 0.009 98.264% 97.676%
σ 0.001 0.004 3.264% 2.397%
%RSD 17.640 51.960 3.321 2.454
Disable Tooltips
Include
Exclude
Copy
Copy inc. Headers
Copy All
Export CSV File
Export CSV File (Unicode)
What's this?
9

Chapter 5 Operating PlasmaScreen

Objective:

To familiarize yourself working with the *PlasmaScreen mode* of operation. Optimization and usage of PlasmaScreen in *cool plasma* and *hot screen modes* will be explained in detail.

Cool plasma allows selective reduction of molecular interferences (e.g. Ar, ArO) to aid the analysis of trace analytes whose nominal mass is identical to that of the interfering species such as K, Ca and Fe. It is also useful for the analysis of easily ionizable elements such as Li and Na.

Hot Screen mode allows higher sensitivities to be obtained compared to normal plasmas with the plasma screen fitted. Tuning is similar to normal plasma except that a little more care is required on the nebulizer flow rate to obtain good oxide levels (< 6% for hot screen mode).

Sample Preparation

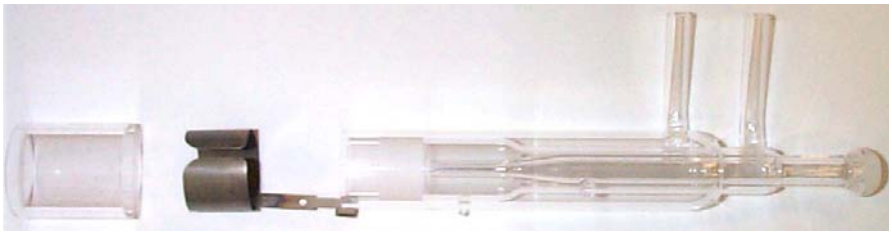
Prepare a 10 ppb Co and In solution in deionized water. It is used for cool plasma tuning. Prepare the solution just prior to the analysis:

- Blank DI water + 0.05% HNO_3
- 10 ppb Co and In in fresh Milli-Q water
- 1 ppb screen tune containing Li, Na, K, Ca and Fe

This chapter treats the following topics:

Step 1 - Installing PlasmaScreen

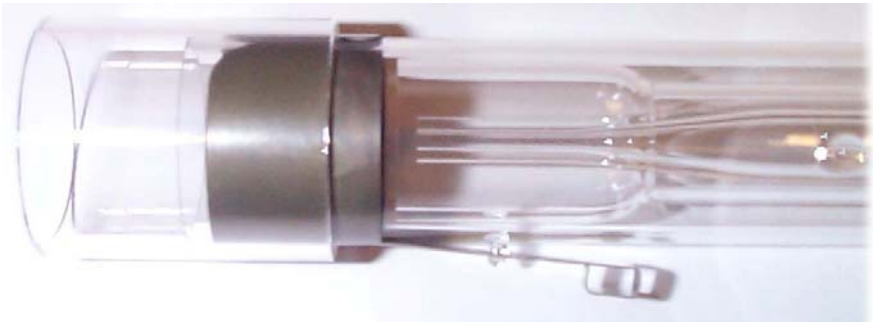
1. The torch has been removed for cleaning and is ready for re-fitting.



Note The correct alignment of the screen is absolutely vital to performance! ▲

The torch, screen and bonnet are designed so that the screen location is fixed in the optimum position by way of a quartz pip on the torch body and a corresponding hole in the screen leg. The pip fixes the screen position and also serves to fix the bonnet position when this item is placed over the screen and pushed back until it touches the pip.

Caution It is important that the screen is located correctly via the pip! If it is not, and is set up such that the screen is protruding from the end of the torch, the plasma is likely to fail to ignite as the ignition spark will arc to the protruding tip of the nickel screen. This will also damage the screen.
The quartz bonnet must completely cover the screen. If the bonnet does not cover the screen correctly, that is it is not pushed fully back to the screen locating pip, or if the bonnet is not fitted at all, the plasma will again fail to ignite and the screen will be damaged. ▲



Note The grounding tab of the screen must be situated completely squarely over the grounding pin. A good connection between the grounding tab and pin is essential for PlasmaScreen Plus to work effectively. The grounding pin itself should not be visible, as the tab should cover it completely. ▲

Ensure that the tab on the screen is fitted correctly into the holder in the block. The cool gas push fit connector is connected to the torch and lined up with the groove in the cartridge, for accurate alignment of the torch.

Ensure that the cool and auxiliary gas lines are connected securely to the torch. Then close the PTFE locking block over the torch and clip it into place.

The torch is then connected back to the spray chamber, and the torch box area is closed ready for operation.

Step 2 - Plasma Screen Operation and Configuration Setup

1. Switch on the plasma and allow to stabilize for approximately 15 min.

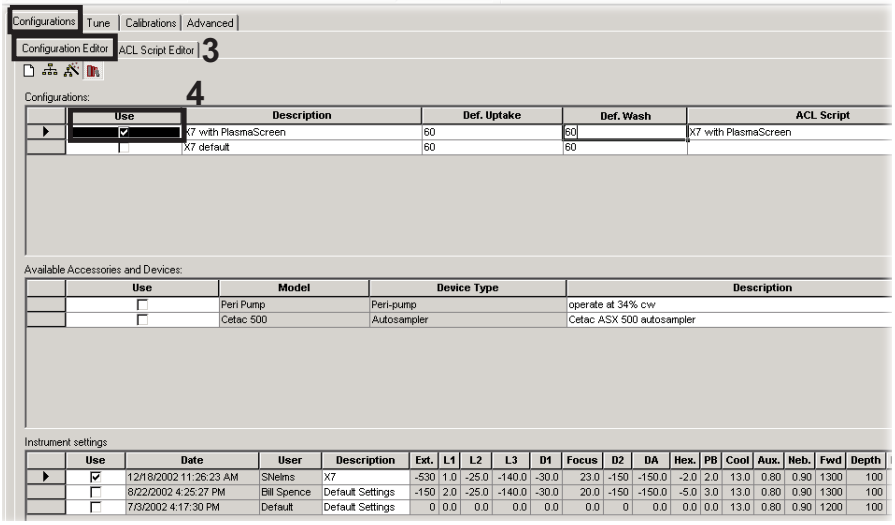
2. The PlasmaScreen Configuration is setup from the *Instrument Control*. Click on the **Instrument** icon.

   The configuration relates to the hardware manually fitted to the XSERIES 2 ICP-MS. A new configuration should be set up for each hardware configuration. The PlasmaScreen is physically removed or

fitted by the user. Thus it should have its own configuration, or multiple configurations for different sample introduction setups, if required (that is glass concentric nebulizer or ultrasonic nebulizer).

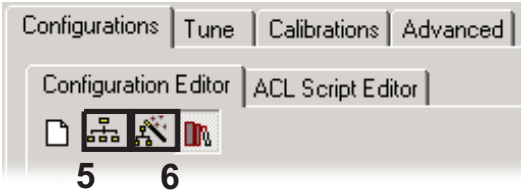
3. Click on the **Configurations** page and **Configuration Editor**. Setting up the configuration allows selecting available accessories and particular pre-set instrument settings. Future saved tune parameters will be stored here as well.

4. Click on the required **PlasmaScreen configuration**. Select the required *accessory*.



5. If the required configuration is not available, create a new configuration from the *icon* shown and edit the description.

6. If a required accessory is not available, a new accessory can be selected by following the *Accessory Wizard*s instructions.



Step 3 - Tuning PlasmaScreen for Cool Plasma Mode

The aim when optimizing the tuning parameters for Cool Plasma is to obtain the maximum analyte signal (typically 10 ppb ^{59}Co in deionized water) with minimum $^{40}Ar^{16}O$ signal.

The main changes in tuning parameters compared to *Standard* mode are summarized in the table below.

Tuning Parameter	Range or Value
Reduced RF Power	range 480 W to 600 W
Negative Pole Bias	-10 V
Reduced Extraction	range -80 V to -150 V
Hexapole Bias	-5 V
Increased Sampling Depth	range 100 to 300 (100 steps back from normal operating position)
Reduced Focus	range 2 V to 15 V
Increased Neb gas flow	1.0 L/min - 1.1 L/min
Increased Aux gas flow	1.0 L/min - 1.2 L/min
Increased Coolgas flow	14 L/min - 15 L/min

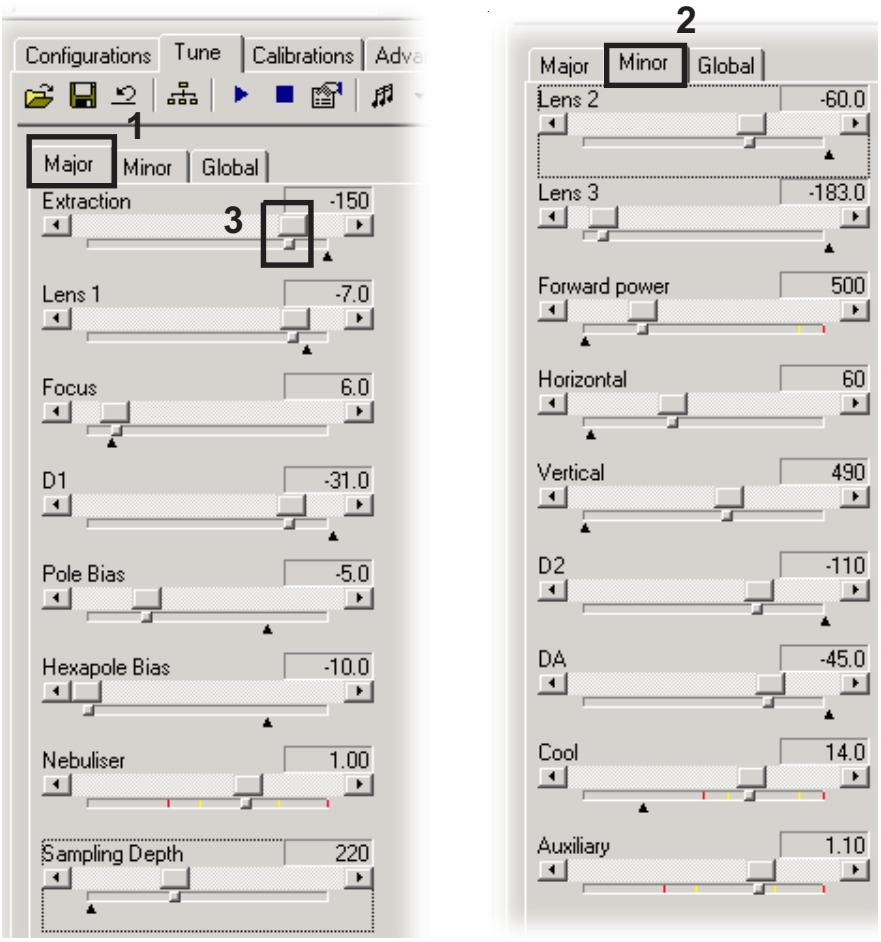
Aim for a Co signal of approximately 10 Mcps/ppm and a $^{40}Ar^{16}O$ contribution of approximately 100 cps.

The ratio of $^{59}Co/^{40}Ar^{16}O$ should be a minimum of 100. If it is proving difficult to reduce the $^{40}Ar^{16}O$ to 100 cps whilst aspirating the 10 ppb Co solution, record the Co signal and aspirate the blank solution (deionized water) to remove the possibility of ^{56}Fe contamination in the Co stock solution.

1. Click on **Tune** in the **Instrument** section. Click on **Major**. These parameters have the greatest effect on the tuning.

2. Use the slider to select the **default settings** as shown.

3. Click on **Minor**. These parameters are generally for fine tuning, however, for *Cool Plasma* mode of operation ensure that the **Forward Power** is set to **low power** as shown.

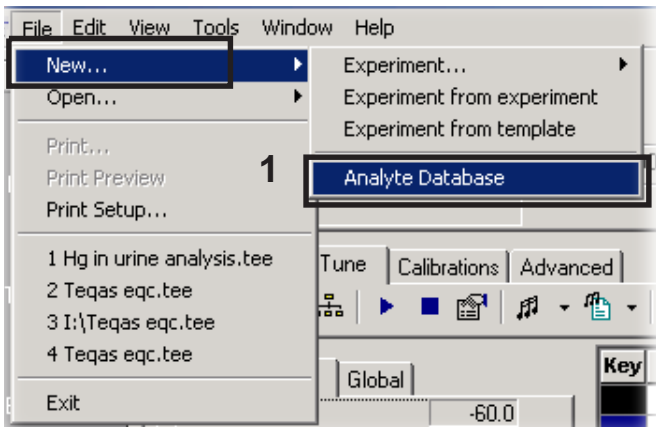


4. Aspirate the 10 ppb Co solution to optimize the tuning. Adjust the RF power, lens settings and nebulizer flow rate to obtain the required ratio.

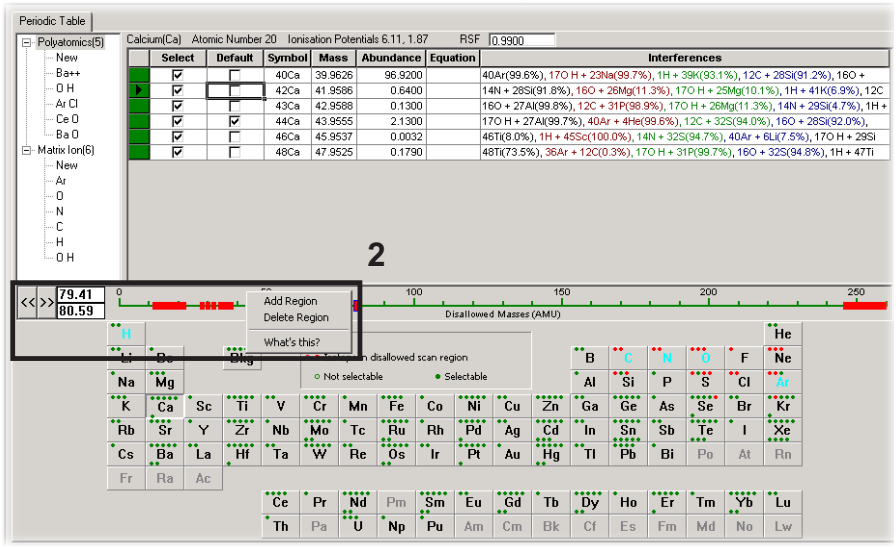
   Once the required $^{59}Co/^{40}Ar^{16}O$ ratio has been achieved, save the instrument settings. The ratio can be monitored in the *real time display*.

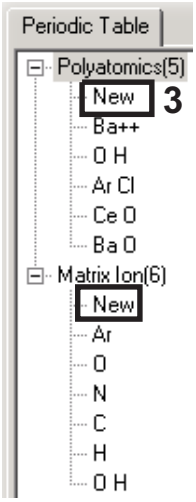
Step 4 - Edit New Analyte Database

1. For the use of PlasmaScreen with Cool Plasma, an *Analyte Database* is required to enable monitoring of ^{40}Ar for the major Ca isotope. A default Analyte Database for cool plasma operation is provided. It can be edited or a new one be created, if required. Click on **File > New > Analyte Database** to create a new one.



2. The database *scan regions* have to be altered to enable the analysis of ^{40}Ar.
   Select the *scan and skip regions* by clicking on them in the graphic.
   Click the right hand mouse button on the graphic to *add or remove* scan regions.
   Alternatively, type in values in the box on left hand side for each individual scan region.



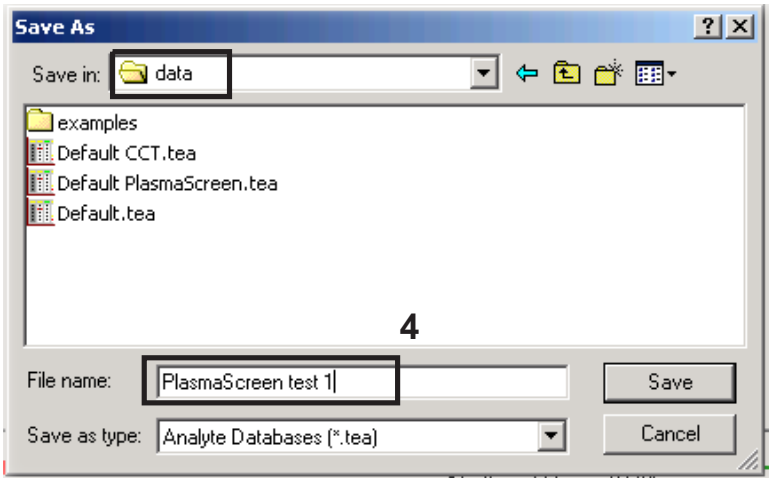


3. Add *matrix* and *polyatomics* in the database as shown:

   Click on **New** to add any polyatomics or matrix ions common to the samples being analyzed. The species shown below are defaults to all databases.

   Any unwanted ones can be removed by right hand mouse clicking on the highlighted species in the list and choosing **Delete**.

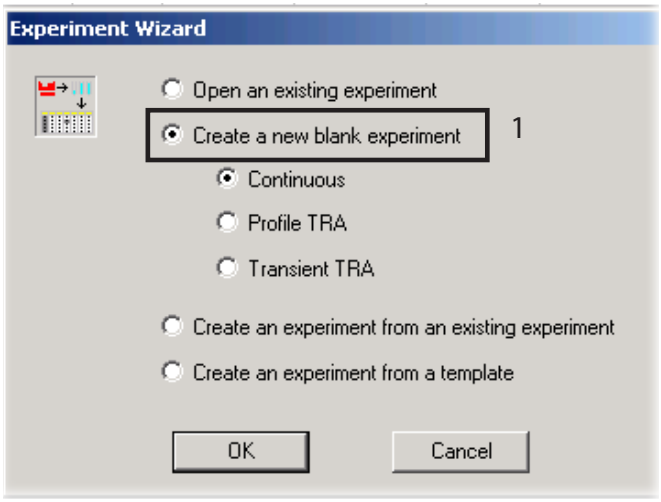
4. **Save As** a new Analyte Database in the **Data** folder as **Default PlasmaScreen.tea**.



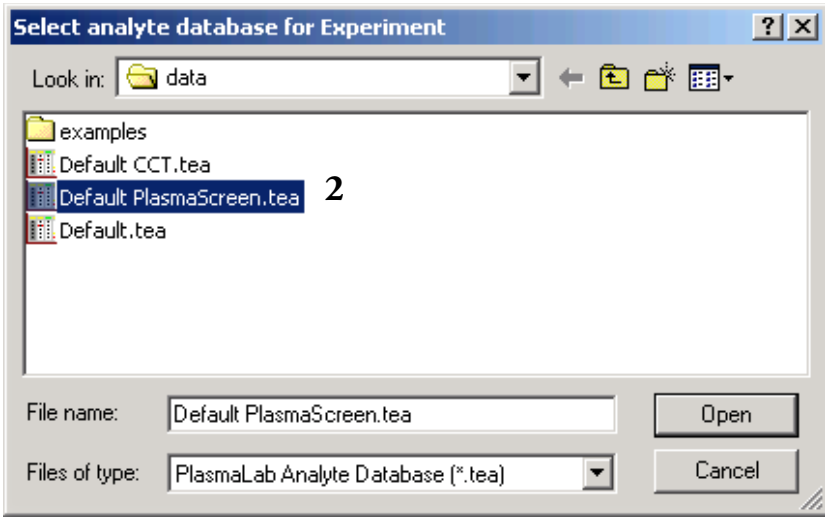
Step 5 - Create New Experiment

Aim: detection limit determination of PlasmaScreen elements.

1. Select **Create a new blank experiment** from the *Experiment Wizard* by clicking on the **Experiment** icon.



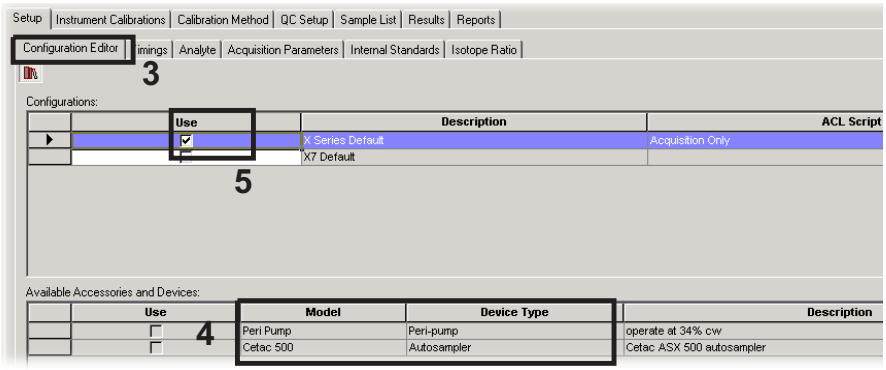
2. Select the *PlasmaScreen Analyte Database* previously created for this experiment, or the default database for cool plasma provided with PlasmaLab.



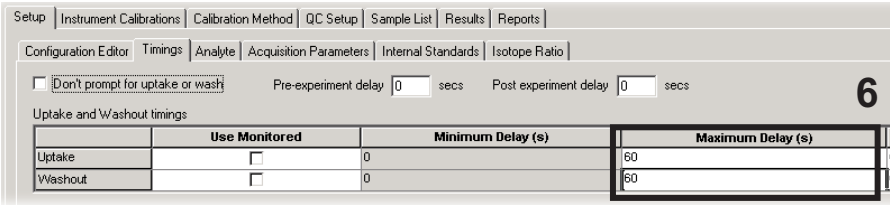
3. In **Setup > Configurations Editor** select the *configuration* for PlasmaScreen by ticking the **Use** box. This can be left, if PlasmaScreen is the current configuration anyway.

4. The *Available Accessories and Devices* are pre-selected in *Instrument Control*. If an accessory is not available, select the accessory using the **Instrument** icon and the *Accessory Wizard* as described in Chapter 1: "Optimizing the Signal", before continuing to edit the *Experiment*.

5. Select the *current instrument settings* by ticking the **Use** box. This can be left deselected, if the current instrument settings are to be used. However, instrument settings must be selected, if multiple settings sets are to be used (that is, *Cool Plasma* and *Hot Screen* mode in the same acquisition).



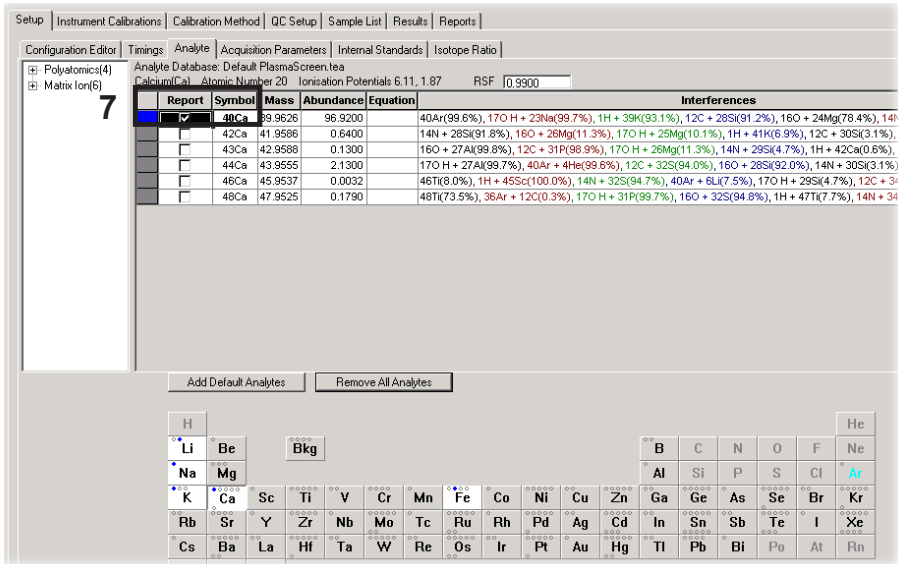
6. In **Setup > Timings** select the time for the *uptake* (measured by the time taken for the sample to reach the plasma) and *washout*. It is typically set between 30 s and 90 s depending on the sample matrix and concentration.



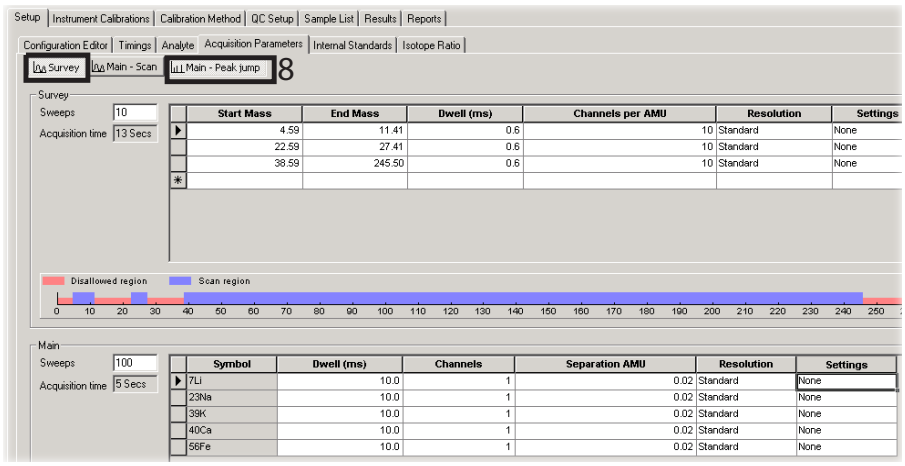
7. Select the analytes via **Setup > Analyte** menu as shown. For the *detection limit* determination, the analytes ^{7}Li, ^{23}Na, ^{39}K, ^{40}Ca and ^{56}Fe are selected.

Note ^{40}Ca can now be selected, which is the most abundant isotope of calcium. ▲

Caution Take care not to use ^{40}Ca in normal or hot screen plasma conditions as the high levels of ^{40}Ar produced will cause high count rates to be seen on the detector. Even though the XSERIES 2 ICP-MS has a mechanism to handle trips on the detector, the lifetime of the detector will suffer when exposed to high levels for prolonged periods. ▲

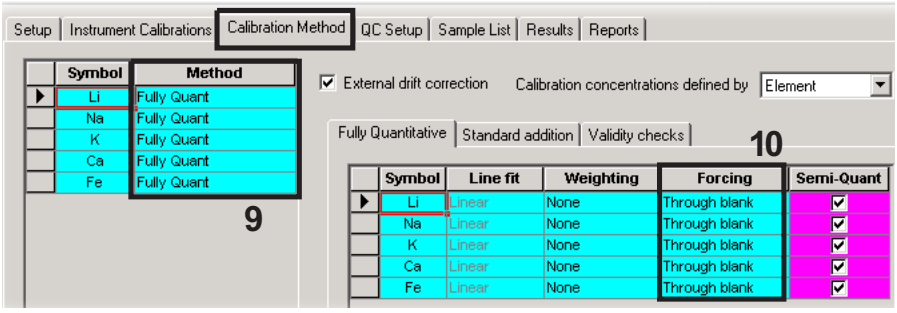


8. Set up the *Acquisition Parameters* for a *Survey* run and *Peak Jump Main* run. The default parameters are shown below.



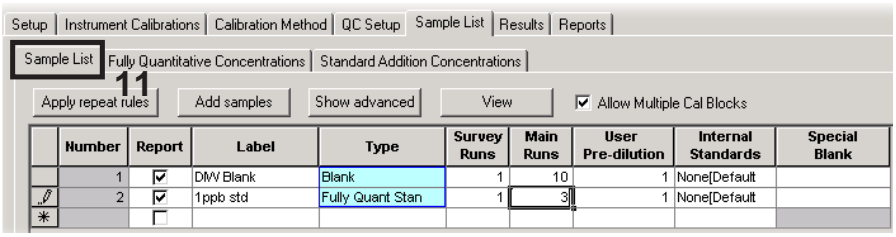
9. Select **Calibration Method**. Select the *Method* from the drop down menu as **Fully Quant** for all analytes.

10. The remaining parameters are set as default as shown, with the exception of *Forcing* which is recommended to be set as **Force Through Blank** selected from the drop down menu.

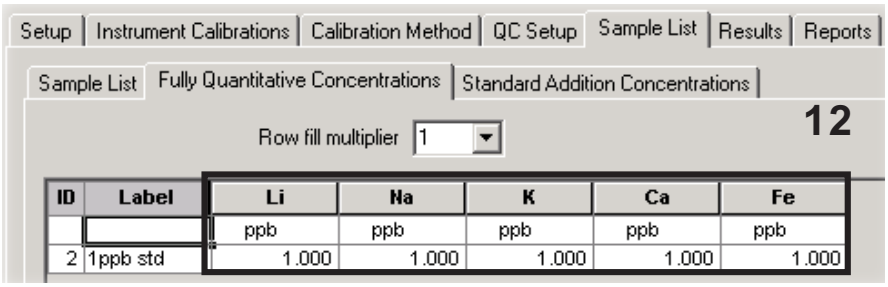


11. Enter the *Sample list* as shown for the DIW Blank and the 1 ppb Std.

Note 10 repeats are selected for the blank for detection limit determination. ▲



12. Add the Fully Quantitative concentration of 1 ppb.



13. **Save** and **Queue** the experiment.

14. Calculate the *detection limits* via the **Analyte Dilution Conc** page, by using a *user pre-dilution factor* of 3. This will show a blank standard deviation of 3 times the actual blank standard deviation (that is, equal to the 3 σ detection limit).

Chapter 6 CCT Operation

Objective:

To familiarize yourself working with the CCT option. The optimization using Collision Cell Technology and the use of different gases will be explained in detail.

Solution required for this practical:

Prepare a 10 ppb Tuning Solution containing Co, In, Ce and U in 2% HNO_3. Recommended gas for multi-element analysis is an 8% H_2 in He mix.

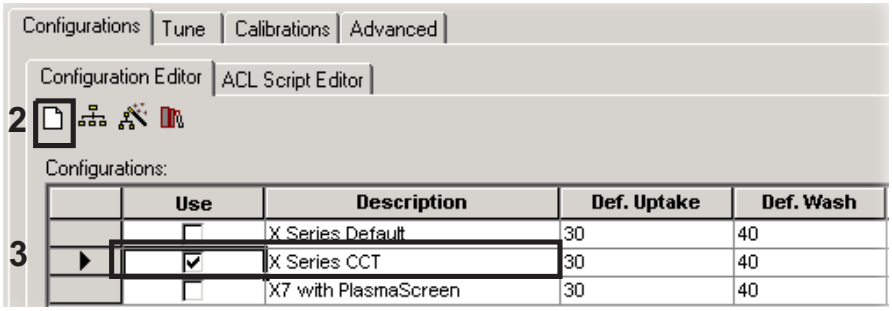
This chapter treats the following topics:

Step 1 - CCT Setup

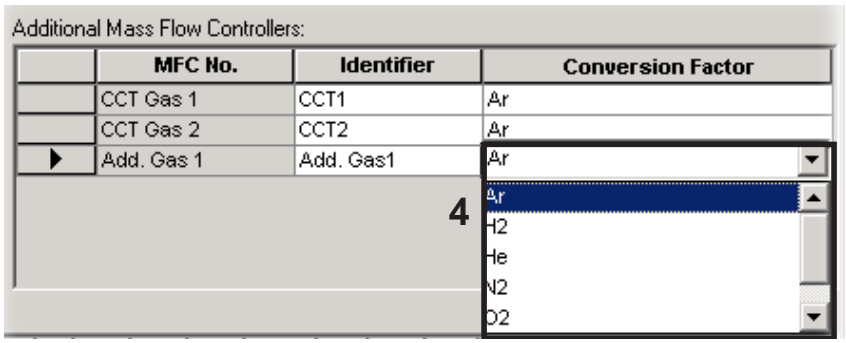
1. The gases required to run CCT are set up in *Instrument Control*. The accessories required for the experiment can also be configured here. Click on the **Instrument** icon to open Instrument Control.

2. A new configuration can be created using the *Configurations* menu and the *Configuration Editor* page, in which all the new instrument settings will be stored. Click on the **New Configuration** icon.

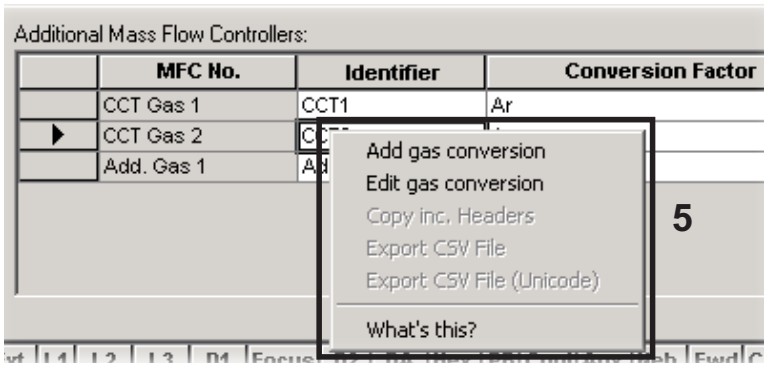
3. Enter a configuration description as shown (that is, CCT Default) and tick the **Use** box for the new configuration.



4. The *Additional Mass Flow Controllers* required to control the gases for CCT must be set up within the Configuration Editor. Type in the name of the CCT gas in the **Identifier** box and select the correct gas in the **Conversion Factor** box by clicking on the downward arrow in the right hand side of the box to display the menu. The conversion is related to helium gas (that is, He factor equals 1.0). For mixed gases choose the setting for the bulk gas.



5. To add or edit a gas conversion, right mouse click on the row to be edited, to display the options.

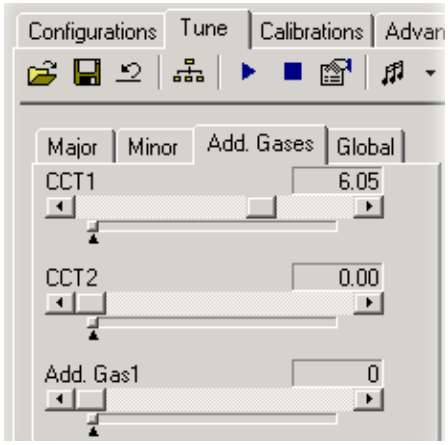


Step 2 - Tuning CCT

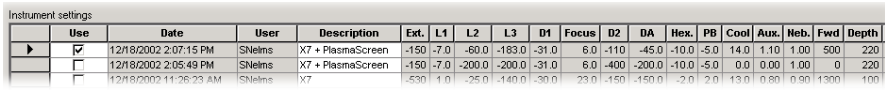
1. Before tuning the CCT, it is recommended to have good *standard mode tuning* first.

2. Set the chosen *CCT Mass Flow Controller(s)* to 6 mL/min for a minimum of 30 s to purge the lines before tuning.

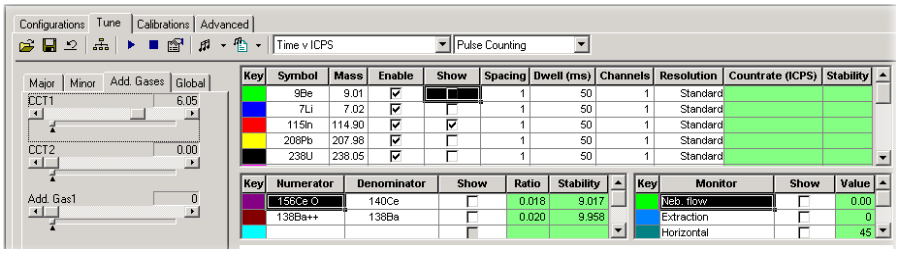
   If the CCT option has not been used for several days, if the gas supply has been changed, or if a leak in the lines has just been fixed, the gas lines will need to be purged overnight to remove entrained water vapor. This increases oxide formation and affects CCT performance.



3. The typical tuning parameters for CCT are shown below. The main differences between *standard mode* and *CCT tuning* are that the *Pole Bias* is set to negative (usually -10 V), the *Hex Bias* is also set to negative (typically about -5 V) and the *Focus* drops from around +19 V to approximately 0 to +5 V, depending on the type of interface cones being used.

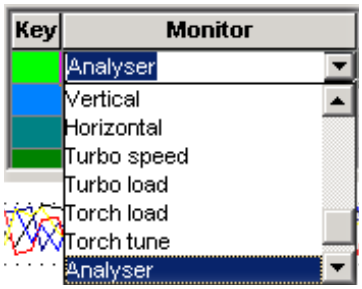


4. In **Instrument Control > Tune**, monitor the elements ^{59}Co, ^{115}In (target 300,000 cps), and ^{238}U for maximum signal. Also maximize the ratio 59/56 (target > 200).



5. Tune the gas flow rate and lenses to give low counts at mass ^{56}ArO, while keeping good transmission at ^{59}Co, ^{115}In, and ^{238}U.

   Typical gas flows when using 8% H_2 in He mixture are in the range 4-7 mL/min.

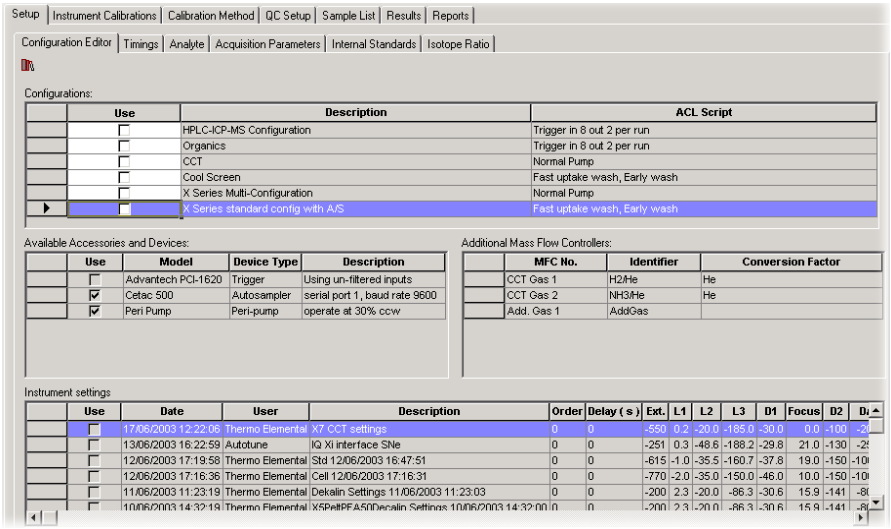


6. Extra instrument parameters can also be monitored when tuning, by clicking on the **downward arrow key** in the **Monitor** box.

Step 3 - Tuning CCT^{ED}

The purpose of CCT^{ED} is to allow the interferences from the plasma to be removed whilst screening out or preventing the formation of new interferences in the collision cell.

1. Before tuning the *CCTED mode*, there must already be a good *CCT* tuning (see "Step 2 - Tuning CCT" on page 6-2). Select a saved *CCT tune file* as shown below:



2. Starting with the *Hex Bias*, reduce the differential between it and the Pole Bias towards zero, while monitoring ^{115}In for maximum signal (target 50,000 cps) and minimizing the CeO/Ce ratio (target 0.03).

   The *Pole Bias* can be increased slightly (typically to -9.5 V) and the Hexapole bias set to -10 V to achieve CCT^{ED} conditions. This sets up a small energy discrimination barrier between the hexapole and

the quadrupole. By changing the magnitude of this barrier, oxide and other polyatomic interference transmission can be dramatically reduced, but at the expense of sensitivity.

Example: CCT Experiment

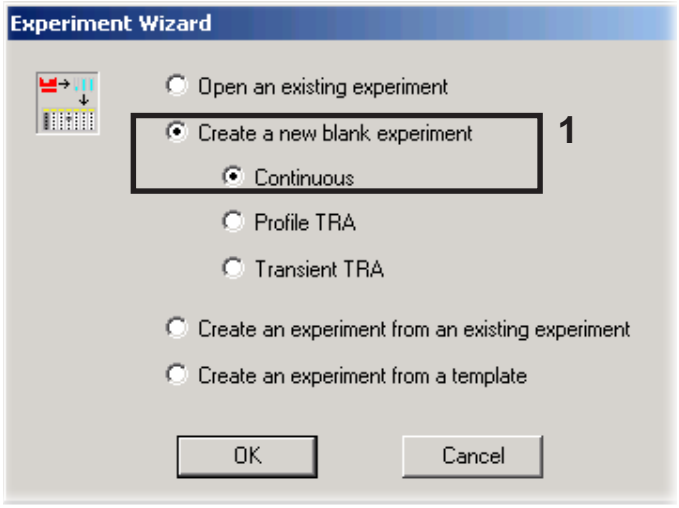
Now that the signal has been optimized, the system is ready for analysis. An experiment can be set up to scan over the 70 to 90 mass region and use the PlasmaLab fingerprint function to place the isotopic fingerprint of Se on mass 80 and As mass 75. To demonstrate the removal of Ar_2 (mass 80) and ArCl (mass 75).

Solution required for this practical:

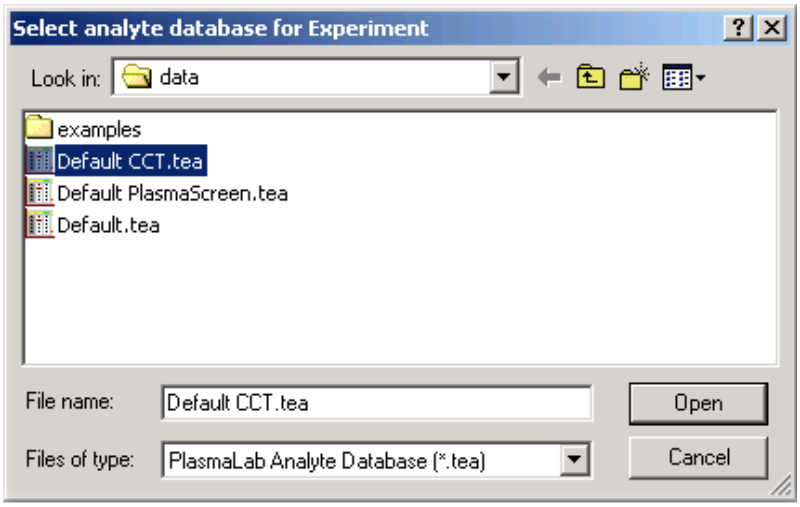
Prepare a 10 ppb solution of Co, As and Se in 5%HCl and a 5% HCl blank.

Step 4 - Create an Experiment

1. Select **Create a new blank experiment**.

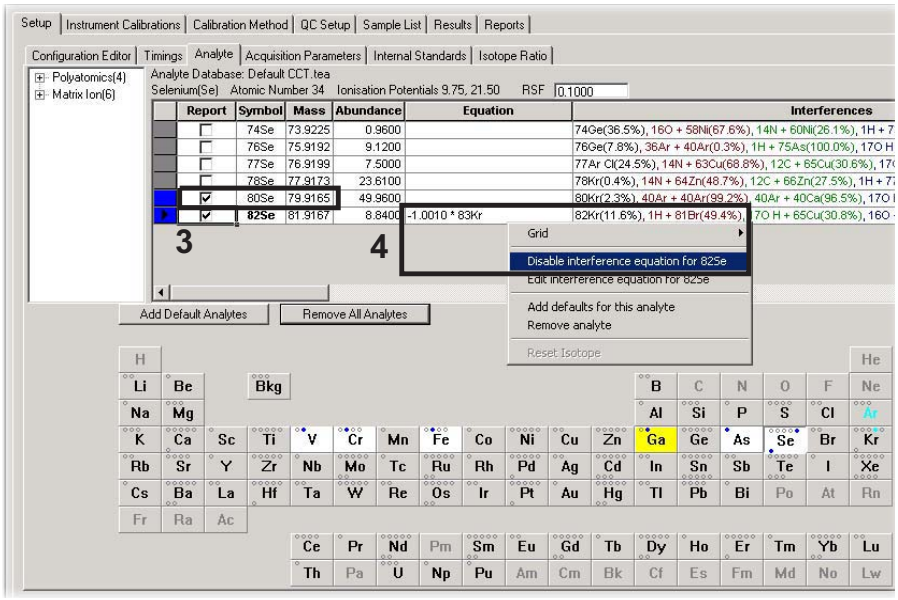


2. Select the **Default CCT.tea** database. This database allows scanning over regions, such as ^{40}Ar (^{40}Ca), $^{75}ArCl$ (^{75}As), and $^{80}Ar_2$ (^{80}Se). These were previously skipped due to interference attenuation of argon-based polyatomics.

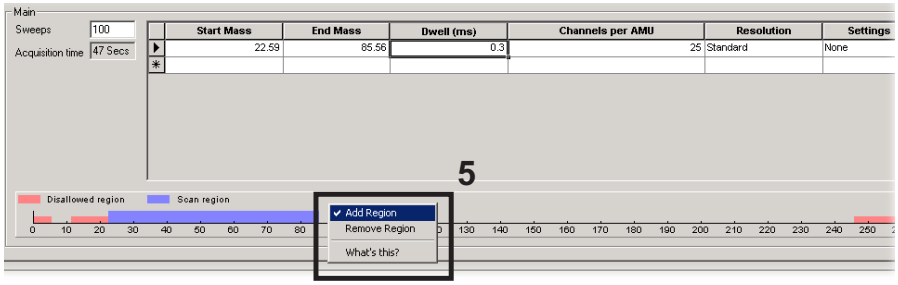


3. Define the *Analyte* menu for Co, As and Se. The major Se isotope at mass 80 can now be selected.

4. Any default interference equations that were present for interferences on As and Se can be removed by right mouse clicking on the **Equation** box to view the drop down menu. Select **Disable interference equation**.



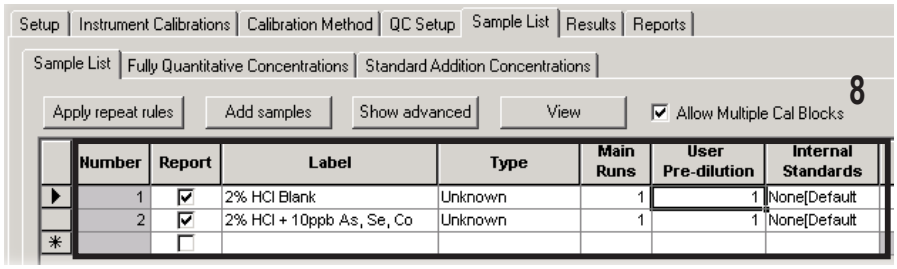
5. Select the **Acquisition Parameters** as shown below for **Main Scan** only.



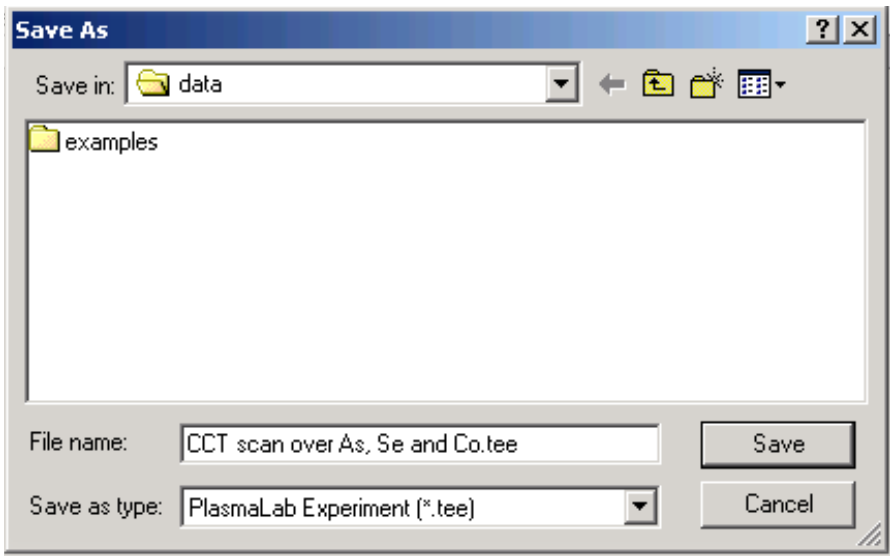
6. Click the right mouse button to view the menu to change between *Add Regions* or *Remove Regions*, if required on the graphic of the mass range.

7. Set the scan region to include the range from mass 23 to 85. Point the mouse over the scan region. Left mouse click and drag over the region to be included.

8. Define the sample list for the analysis.



9. Save the experiment as **CCT scan As, Se, Co**.

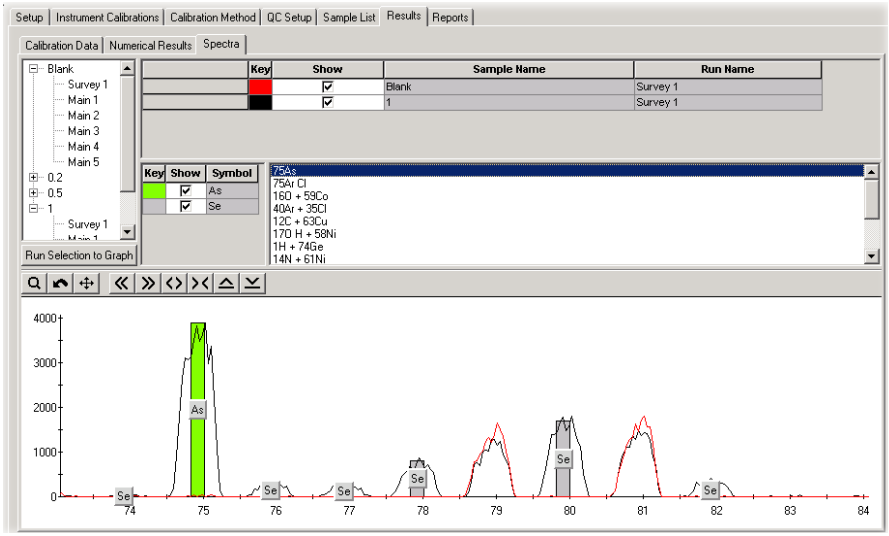


10. Queue the experiment.

Step 5 - Data Interpretation

View the spectra in **Results > Spectra**, and double click on both *blank* and *sample survey*.

Zoom in to region between mass 70–85 amu to show removal of ArCl (mass 75) and Ar_2 (mass 80), enabling 10 ppb As (mass 75) and Se (mass 80) to be measured easily, in a high chloride matrix.



Chapter 7 Maintenance

Objective:

This chapter contains general maintenance instructions for the XSERIES 2 ICP-MS. If you follow these instructions, you will obtain the best possible performance with the instrument. The XSERIES 2 ICP-MS is a precision instrument which will yield high performance for a long time, if it is properly and regularly maintained. You will be taken step by step through all aspects of routine maintenance.

This chapter treats the following topics:

Caution Take care to wipe up any spillages that occur when handling acids or organic solvents near the instrument. Otherwise, the enamel coating may corrode. Before using any cleaning or decontamination methods except those specified by the manufacturer, the user should check together with the manufacturer that the proposed method will not damage the equipment. ▲

Warning Pay particular attention to the area inside the torch box where the sample introduction glassware is mounted. As acids tarnish the surface finish, wipe up spills. Wear protective gloves while doing this! If for any reason, you want to clean the instrument panels, use a diluted soap solution and paper towels to wipe down the panels. ▲

Step 1 - Peristaltic Pump Tubing

Note The condition of the peristaltic pump tubing can affect the stability of the ion beam. Therefore, the peristaltic pump tubing should be replaced regularly: for a typical 40 h working week, it is recommended to replace it weekly. ▲

1. Remove the sample uptake tube from the sample and drain the sample introduction glassware and all tubing. Stop the peristaltic pump by using *Accessories Control*.

2. Wipe the surface of the peristaltic pump rollers and tension arms with a dry cloth to remove any dust.

3. Replace the sample uptake tubing with a sufficiently long 0–2 mL/min Tygon® tubing with orange/yellow collars (P\N 1200211). If it proves difficult to insert the PTFE tubing in the ends, widen them by inserting the end of a clean micro pipette tip.

4. Replace the drain tubing with a piece of 0–5 mL/min Tygon® tubing with white/black collars (P/N 1200212).

5. Place the two pieces of Tygon® around the barrel rollers. Take care that they are not twisted and secure them in place using the collars and brackets. Attach the tension arms. Ensure that the Tygon® tubes are fitted for correct sample and drain flow direction when the peristaltic pump is operating.

6. Switch the peristaltic pump on. Check that sample is being taken up into the instrument and being drained efficiently. If needed, adjust the tension on the Tygon® tubing using the thumbscrews on the peristaltic pump.

Note Do not over-tighten, as this would reduce the tubing lifetime! ▲

7. If an autosampler is being used, renew the wash station tubing whenever the sample uptake and drain tubing are changed. If the sample to be analyzed contains a high percentage of organic solvent, the pump tubing should to be replaced with solvent-resistant isoversinic pump tubing.

   The uptake tube has an inner diameter of 0.5 mm (P\N 1200092). The wider drain tubing has an inner diameter of 2 mm (P\N 1200093).

Step 2 - Spray Chamber

Due to the acidic nature of most sample types in ICP-MS, the O rings at the rear side of the impact bead spray chamber, which secure the nebulizer in place, can degrade and become brittle over time. This is observed as a discoloration of the O rings. It can lead to leakage of the nebulizer gas from the rear side of the impact bead spray chamber and poor signal stability.

Note Old O rings should be carefully removed using a set of tweezers and be replaced with two Kalrez O ring seals (P\N 1201833). Rinse adequately with a non-aggressive solvent at the end of a sample set to prolong the lifetime of the O rings. ▲

Step 3 - Removing Glassware

Removal and cleaning of ICP glassware and fittings is carried out to reduce blank levels for elements which tend to accumulate on the glassware. For higher productivity, Thermo Fisher Scientific recommends having two sets of glassware available for use, one set in the XSERIES 2 ICP-MS and the other set being cleaned in preparation for use.

Alternatively, if the analytical program alternates between ultra trace determination and relatively high level/complex matrix work, a complete set of glassware and cones should be assigned to each.

1. Switch the XSERIES 2 ICP-MS into **Operate** mode. Rinse the sample tubing and glassware by aspirating deionized water into the impact bead spray chamber.

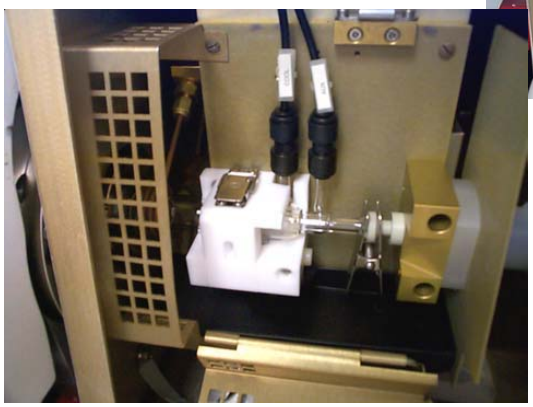
2. Switch the XSERIES 2 ICP-MS **off**, into **Vacuum ready**. Drain the complete sample introduction system.

3. Release the push-fit connector on the nebulizer gas line.

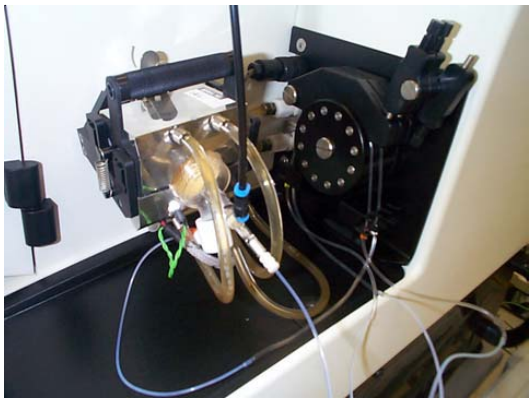
Caution With great care, gently remove the nebulizer from the rear side of the impact bead spray chamber. ▲

Note To remove the push-fit connectors, push the collar towards the connector body whilst gently pulling the tube away from the connector. ▲

4. Open the torch box cover. Then open the torch box RF shield. Remove the metal clip that fixes the transfer tube (the elbow) to the rear side of the torch.



5. Lift up the slider that holds the Peltier cooler in place around the impact bead spray chamber and place it into the higher rest position. Gently remove the spray chamber from the XSERIES 2 ICP-MS.



6. Release the two push-fit connectors that supply the coolant and auxiliary gases to the torch. Release the metal clip on the PTFE block that holds the torch in place. Slide the transfer tube (elbow) backwards away from the torch and gently pull the torch clear of the block and coil.

Release the push-fit for the Aux and Cool gas.

Release the metal clip.

Step 4 - Cleaning Glassware

Warning When handling the corrosive acids required to perform the cleaning procedure described below, take the following precautions:

Wear protective clothing.

Use acid resistant laboratory gloves and glasses.

Make yourself aware of all safety procedures in case of spillage or exposure to skin or eyes before commencing the cleaning.

The acid cleaning of the glassware should take place in a fume hood. ▲

1. The torch, impact bead spray chamber, transfer tube and nebulizer may be cleaned by soaking them in a laboratory glassware cleaner (e.g. 5% Decon™ 90 or similar) for 30 min. Raising the temperature will speed up the process.

Caution Do not boil the liquid when cleaning polypropylene components as the plastic will degrade! 50–60 °C are sufficient. ▲

2. Rinse at least three times with deionized water. Insert the impact bead spray chamber in 10-20% Analar grade nitric acid. Leave it there until the glassware is required for use. For a thorough cleaning, the soaking period should exceed 6 h. Alternatively, items can be cleaned in less than 1 h with an ultrasonic bath and the same cleaning solution.

3. Prior to re-installing the glassware, remove it from the nitric acid and wash it at least three times in fresh deionized water. Finally, allow it to air dry completely. If any components are to be stored for some time before use, they should be placed in metal-free sealed plastic bags.

4. The torch should be replaced (P\N 3600969), if significant devitrification of the injector tip is evident, as this can produce high blanks for sodium and other analytes.

5. Inspect the tip of the nebulizer using a magnifying glass. If the tip appears damaged in any way, it should be replaced (P\N 4600294).

   Damaged nebulizers can give rise to poor stability and sample transport efficiencies. Replace the O rings in the rear side of the impact bead spray chamber, if the nebulizer seal is not good. If there is any damage evident to the impact bead spray chamber, it should be replaced.

Step 5 - Nebulizer Maintenance

The XSERIES 2 ICP-MS is supplied with a glass concentric type nebulizer. With use, these nebulizers may become blocked. A blockage is typically caused by particulate matter present either in the supply gas or in the sample. To prevent nebulizer blockage, follow the general rules below:

- Filter the supply gas by installing a low impedance gas filter inline. This prevents particulate matter present in the gas or gas lines from lodging in the nebulizer.

- Filter the sample. This is recommended provided that the solid material present in the sample is not of analytical importance. Typically, the nebulizer will not lose efficiency when analyzing samples containing small particle size matter because the design of the sample capillary is sufficiently robust for the analysis of such samples.

- Rinse the nebulizer.

Note This is extremely important when switching the XSERIES 2 ICP-MS off at the end of a sample analysis. ▲

Solids may deposit in the nozzle, restricting the nebulizer and causing a loss in performance. Allow the nebulizer to dry before turning off the gas supply. Make sure that the sample uptake tube is disconnected or arranged so as to prevent siphoning into the nebulizer when the gas is switched off. This procedure also extends the lifetime of the O rings fitted to the impact bead spray chamber.

Should the nebulizer become blocked, apply compressed gas to the nozzle and backflush the blockage. Alternatively, flush the nebulizer with a 3–5% solution of aqua regia.

Warning When handling corrosive acids such as aqua regia take the following precautions:

Wear protective clothing.

Always use acid resistant laboratory gloves and glasses.

Make yourself aware of all safety procedures in case of spillage or exposure to the skin or eyes before commencing the cleaning.

Preferably the acid cleaning of the glassware should take place in a fume hood.

Follow local health and safety procedures. ▲

If the nozzle is coated in crystalline deposits, soaking in dilute nitric acid (2%) is sufficient.

Caution Do not use an ultrasonic bath to remove blockages or during nebulizer cleaning as cavitation and ultrasonic vibrations can cause irreparable damage to the nebulizer tip! ▲

Step 6 - Refitting Glassware

1. Attach the push-fit connectors containing the auxiliary and cool gas lines to the arms of the torch. The cool gas arm of the torch is lined up with the groove in the cartridge. In this way, the torch is always aligned in the same place.

2. The PTFE block cartridge is closed over the torch, and the metal clip is secured in place.

3. Replace the transfer tube and then the impact bead spray chamber. Secure the transfer tube to the torch and spray chamber using the clips provided. If using a Peltier cooler, lift the top half of the Peltier cooler into place around the spray chamber from its rest position.

4. Attach the nebulizer to the nebulizer gas line using the push-fit connector.

5. Plug the push-fit connector of the nebulizer sample uptake tube into the nebulizer supply. Gently push the nebulizer into the rear of the impact bead spray chamber.

Caution Take care not to damage the tip! ▲

Attach the drain tube to the drain adapter. Secure onto the impact bead spray chamber. Replace the torch box RF shield and close the torch box door.

Step 7 - Cleaning Cones

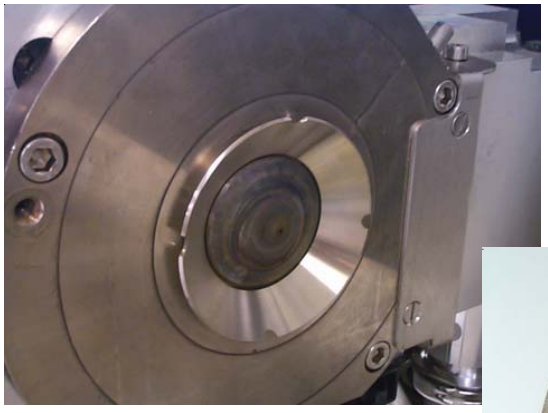
The condition of the cones affects sensitivity, blank levels and the magnitude of signals from some background species (particularly oxide species). The actual intervals at which cleaning is required depend on the analytical workload and the nature of the samples being analyzed (particularly the level of dissolved solids in the samples being aspirated).

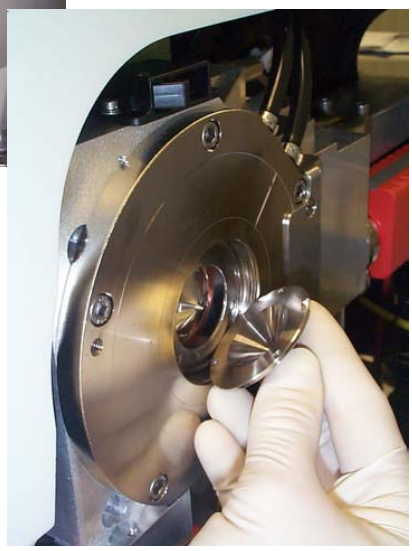
Note The cones are manufactured to high precision from pure nickel and should be handled with care, particularly the skimmer. The tip of the skimmer cone is fragile and as such is easily damaged. ▲

1. Open the torch box door to release the clip between the torch box turret and the interface region. Push the torch box enclosure away from the rest of the instrument to reveal the interface region.

2. Using the *sample cone tool* provided, unscrew the locking collar holding the sampler in place.

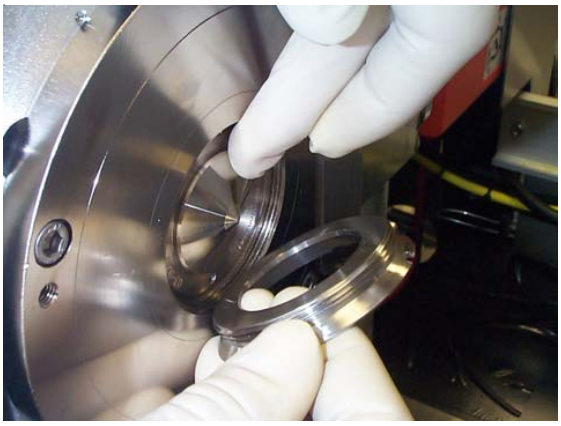
Note Should the sample cone be stuck, carefully insert a small flat screwdriver into the edge of the sampler to gently prize it away from the interface front plate. If the graphite gasket is damaged, it should be replaced (P\N 3004382). ▲





Remove the sample cone first.

Use the *sample cone tool* to unscrew the locking collar of the sample cone.



When removing the locking collar, hold the skimmer cone in place with one finger as illustrated.

Then remove the skimmer cone for cleaning.

3. Inspect the sample cone for excessive wear around the orifice. Replace, if necessary (P\N 3004661 for X7/HPI interface or P\N 3600812 for X5/Xi interface).

4. To clean the sample and skimmer cones, place in an ultrasonic bath in ~5% Decon 90 (or similar laboratory detergent) for approximately 15 min. Rinse with deionized water, then ultrasonicate for 15 min. with deionized water. Dry carefully with a non-fibre shedding laboratory wipe, or leave to air dry.

5. If further cleaning is required (that is, if performance expectations cannot be met), clean the front and rear surfaces of the sample cone by gently rubbing from the center outwards with an abrasive pad such as Scotchbrite™ (P\N 1026048). Alternatively, apply a fine abrasive metal polish such as Polaris (P\N 1041300) made into a paste with deionized water.

Note Pay most attention to the area around the orifice. All polish or abrasive fibers must be removed before refitting. ▲

Place the sample cone in 2% nitric acid in an ultrasonic bath for 2 min. Then rinse thoroughly with deionized water. Finally, dry carefully with a non-fibre shedding laboratory wipe, or leave to air dry.

Caution Do not rub abrasive material over the tip of the cones! Their edges must be sharp and regular. ▲

6. The skimmer cone is less mechanically robust than the sample cone (particularly at the tip). The edges of the cone must be sharp and regular. If there is evidence of damage to the tip, it should be replaced (P\N 3200860 for X7/HPi interface or P\N 3600811 for X5/Xi interface).

Caution The skimmer cone must therefore be handled with extreme care! ▲

The procedure for removal is as follows:

- Remove the skimmer cone through the sample cone hole in the front plate (by inserting the *recessed cone tool* and aligning the two lugs in the cone tool in the outer edge of the cone surface; then turning counterclockwise, it will be held in place magnetically).
- Inspect the skimmer and reject it, if damage is apparent.
- Clean the skimmer cone by placing it in an ultrasonic bath in ~5% Decon 90 (or similar laboratory reagent) for approximately 15 min. Rinse with deionized water. Then sonicate for 15 min with deionized water. Dry carefully with a non-fibre shedding laboratory wipe, or leave to air dry.

- If further cleaning is required, clean the front and rear surfaces of the cone by gently rubbing from the center outwards using an abrasive pad such as Scotchbrite™ (P\N 1026048), or alternatively apply a fine abrasive metal polish such as Polaris (P\N 1041300) made into a paste with deionized water.

Caution Pay most attention to the area around the orifice and clean with extreme care! ▲

The rear of the orifice may be reached using a cotton bud and Polaris. All polish or abrasive fibres must be removed before refitting. Place the cone in 2% nitric acid in an ultrasonic bath for 2 min. Then rinse thoroughly with deionized water. Finally, dry carefully with a non-fibre shedding laboratory wipe, or leave to air dry.

7. Inspect the 28×1.5 mm O ring which is located behind the skimmer cone for damage. Replace it, if necessary (P\N 1201240).

8. Reassemble the interface in reverse order. Ensure that the skimmer is seated properly and that the front plate and sample cone seals are in position and clean.

   Tighten the sample and skimmer cones finger-tight to give a good thermal and vacuum seal. Move the torch box back into position and close the torch box door.

Caution Be careful to push the torch box enclosure into position with the door open to avoid damage to the interlock key! ▲

Step 8 - Replacing Multiplier

This section details the procedure for replacing the *AF214 Simultaneous Multiplier* used in the XSERIES 2 ICP-MS. The XSERIES 2 has been specifically designed by Thermo Fisher Scientific to enable easy access and replacement of this part.

Note When replacing a multiplier, you will be entering the vacuum system of the instrument. In order to ensure that the vacuum system does not get contaminated during this process and the optimum performance of your XSERIES 2 is maintained, it is important that you follow the precautions below. ▲

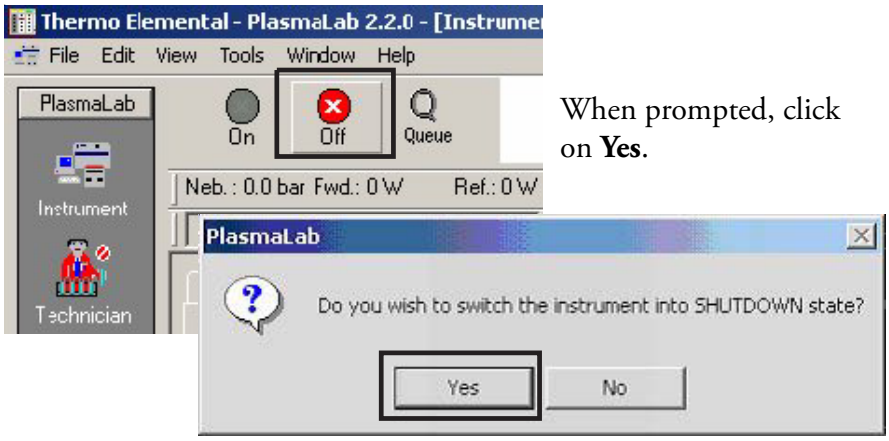
Caution Always wear powder- and lint-free gloves when handling components inside the vacuum system! ▲

Note Close the lid of the analyzer chamber whenever you are not actively working inside it! ▲

Switching to Shutdown Mode

Switch to *Shutdown* mode as follows:

1. With the XSERIES 2 in **Vacuum Ready** mode, click on the **Off** button.



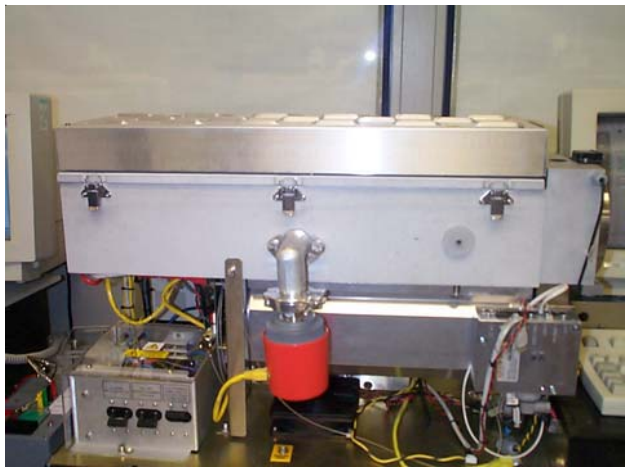
When prompted, click on **Yes**.

2. Leave the instrument for 5 min. to vent fully.

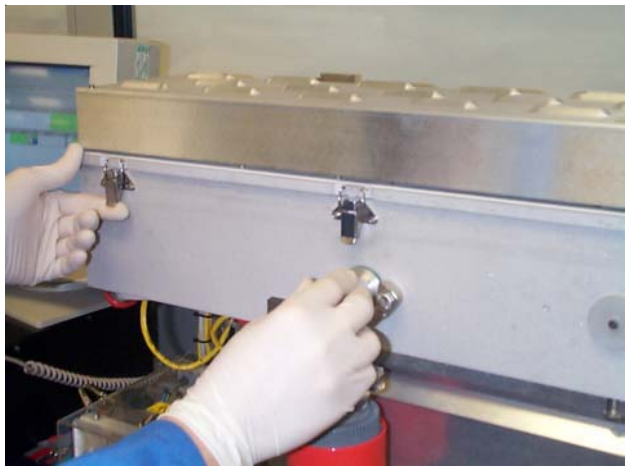
Removing Existing Multiplier

Remove the existing multiplier as follows:

1. Remove the main left hand side cover and store it carefully.

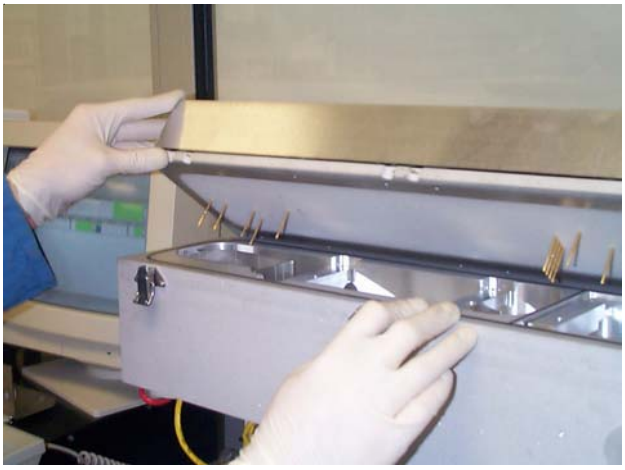


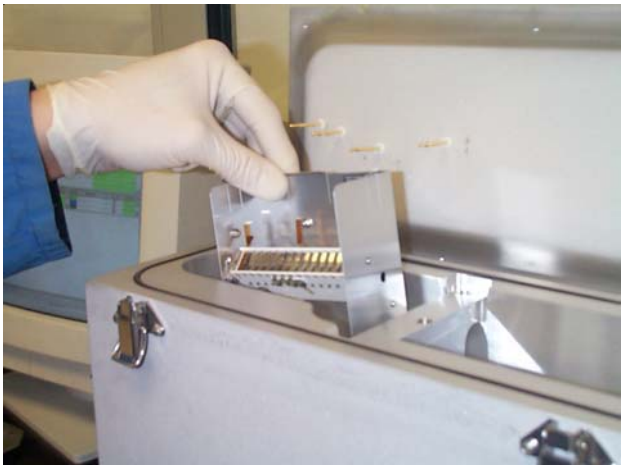
2. Undo the three clips holding the lid of the analyzer chamber.



3. Open the lid and remove the existing multiplier.

   Close the lid until you are ready to fit the new multiplier, making sure that the O ring seal around the chamber top is located fully in its groove.





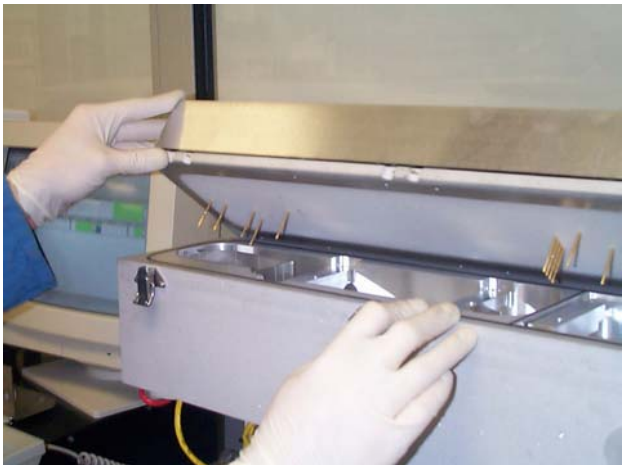
Fitting New Multiplier

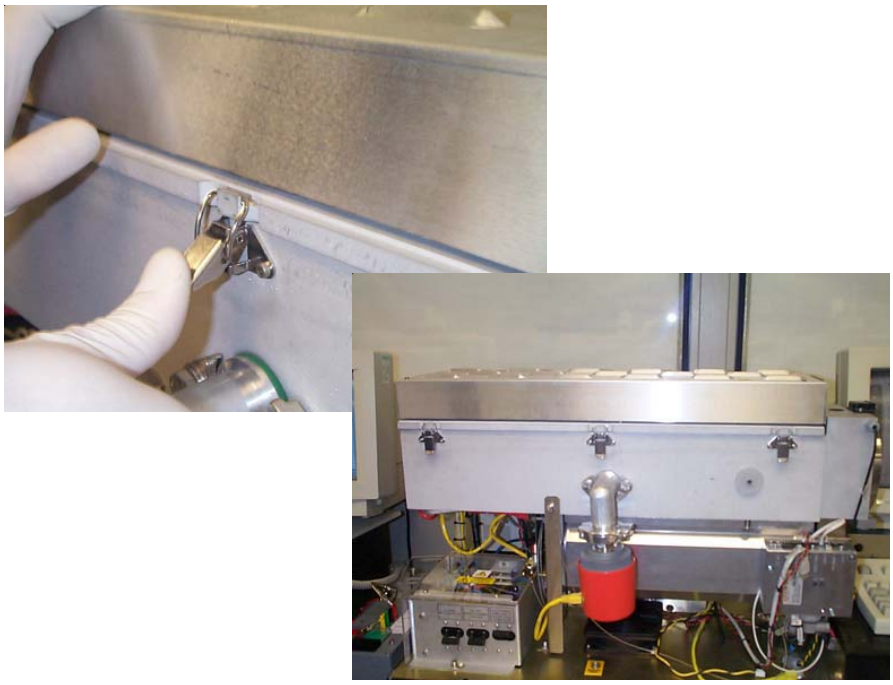
Fit the new multiplier as follows:

Caution Only unpack the multiplier immediately before fitting. Install immediately. Do not expose the multiplier to air unnecessarily. ▲

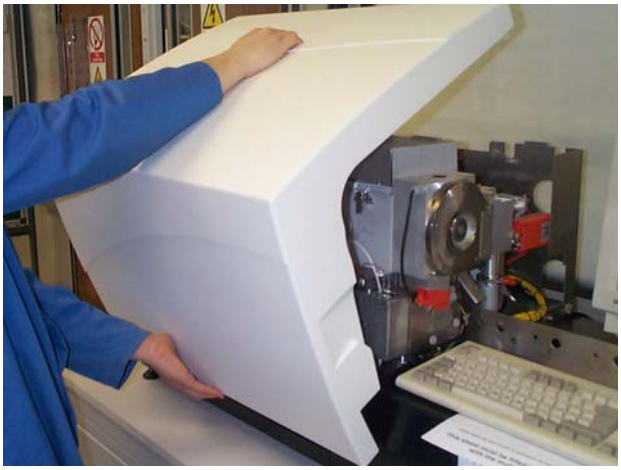
1. Remove the new multiplier from its packaging and place with care in the locating slots provided in the base of the analyzer chamber.

2. Carefully close the lid of the analyzer chamber, making sure that the O ring around the chamber top is located fully in its groove. Secure the three clips which hold the lid down.

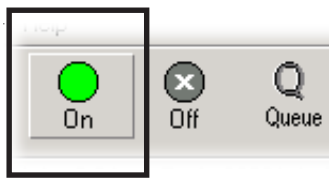




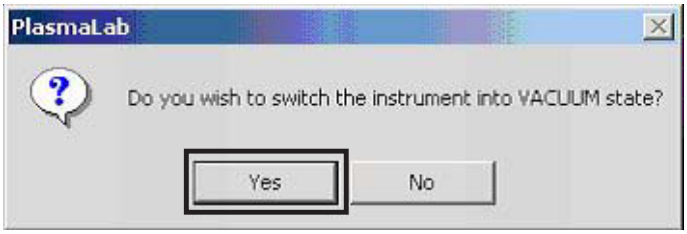
3. Replace the main left hand side cover.



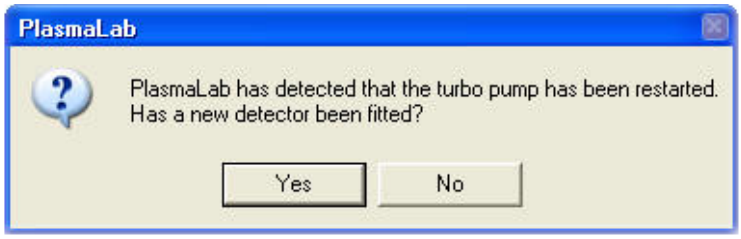
Switching to Vacuum Ready Mode



In *PlasmaLab*, click on **On**. At the prompt, select **Yes**.

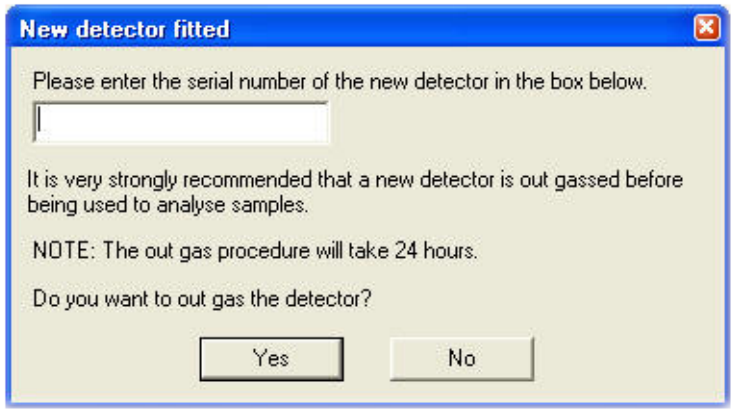


Each time the turbo pump is started, the user will be prompted to state, whether a new detector has been fitted.



- If **No** is selected, no further action will be taken. All information for *detector monitoring* and *cross calibration* continues to be stored for the current detector.

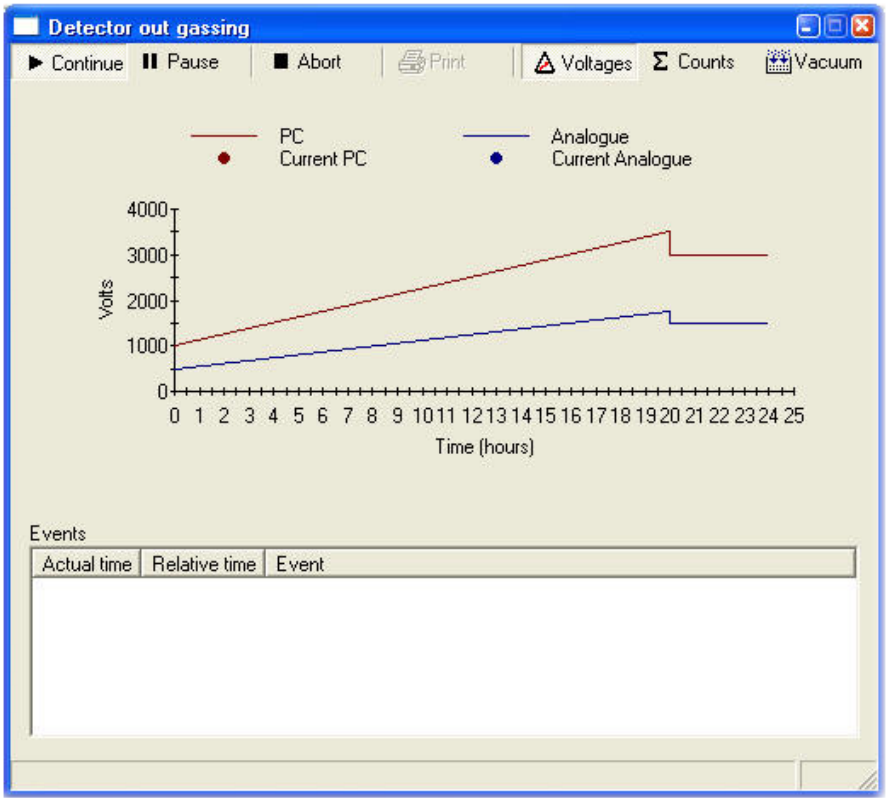
- If **Yes** is selected, the user will be prompted to enter the serial number for the new detector. The number can be found on the side of the box containing the new detector.



Caution It is highly recommended that new detectors go through the burn-in procedure. Failure to do this could seriously impact the lifetime of the new detector! ▲

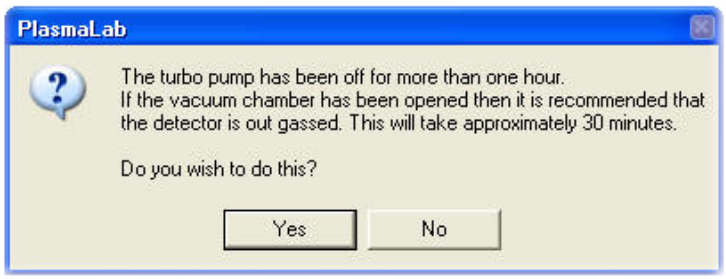
- If **No** is selected for outgassing the new detector, the old detector data is archived and new records created for *detector monitoring* and *cross calibration*.

- If **Yes** is chosen for outgassing, the new records are created, and the outgassing dialog is shown.



This dialog shows the progress of outgassing and any events (e.g. outgassing has not started because the vacuum has not yet reached the trip point). The dialog can be minimized and PlasmaLab can be used to process data. However, while the detector is outgassing, no analysis can be performed. Outgassing can be aborted or paused, and a note will be made in the event log for that detector. Once the automatic outgassing is finished, a message will be shown and analysis can begin.

If the turbo pump has been switched off for more than 1 h, when it is restarted, if the user chooses **No** to the *New Detector* dialog, the following dialog is presented.

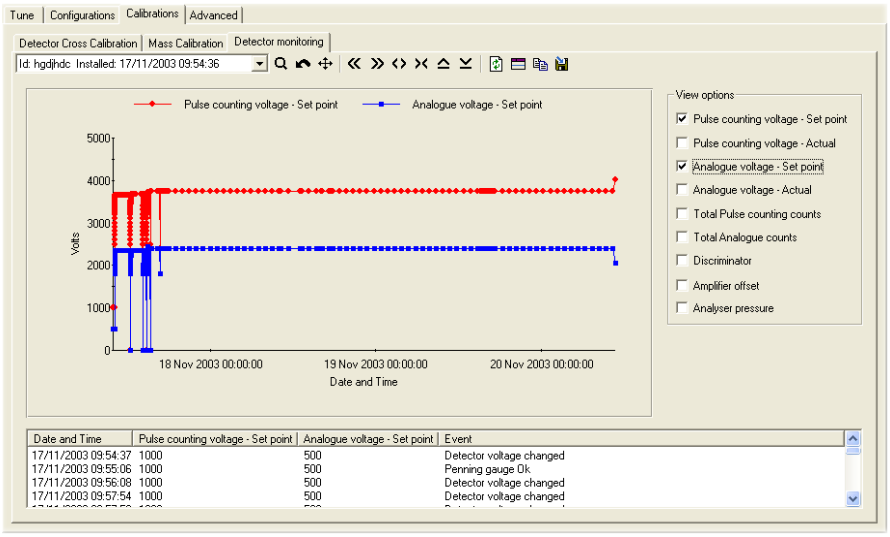


Caution It is highly recommended that all detectors go through the burn-in procedure! Failure to do this could seriously impact the lifetime of the new detector. ▲

Detector Monitoring

On the *Detector Monitoring* page, the changes that have taken place on the instrument during the lifetime of the detector can be viewed. The main purpose is for diagnostic reasons, allowing any problems to be tracked that may contribute to the lifetime of the detector.

The *Detector Monitoring* page will show the information for the new detector. The various parameters can be displayed using the *Options* on the right of the page.



This data will show every voltage change made on the detector whether automatically by the instrument calibration software or manually via the *Tune* page. Monitoring data for older detectors can be viewed by using the drop down dialog box at the top of the page.

It is possible to export the monitoring data to a file using the **Export** button.

To view parts of the data in more detail, use the *zoom tools*:

To highlight any events that occurred in the zoomed region of the graph, the *highlighting tools* can be used to show the information in the table below the graph.

Routine Maintenance Checklist

Note The six-monthly and annually procedures listed below should be carried out by a qualified service engineer. ▲

Interval	Maintenance Procedure
daily	Inspect and clean cones, if necessary (that is, if performance is not acceptable)
	Complete instrument log
weekly	Prepare fresh performance monitoring solution
	Replace sample and drain peristaltic pump tubing
	Exchange ICP glassware (optional)
	Clean removed glassware ready for next exchange (optional)
	Clean spray chamber drain plug
	Clean nebulizer
	Check air filters and clean, if necessary
	Check multiplier voltages and perform cross calibration
monthly	Replace sample uptake tubing
	Check rotary pump oil
	Check oil mist filters
	Check chiller water level in reservoir
six-monthly*	Examine lens system and clean, if necessary
	Examine penning gauge and clean, if necessary
	Change rotary pump oil
annually	Replace any worn O rings

*Thermo Fisher Scientific engineer servicing

Chapter 8 Troubleshooting

Objective:

This chapter goes through the most typical trivial problems that might occur with the instrumentation. It will help you to diagnose a particular problem and will recommend remedial action. Many problems in high performance instrumentation are the result of trivial concerns, usually after some routine maintenance, or if another operator has left the instrument in a non-standard configuration.

The two rules of diagnosing potential problems are to:

- examine the recent history of use of the instrument
- consider the simple causes first
  (e.g. sample introduction is more likely than quadrupole failure)

The single most useful 'tool' for probing analytical concerns and hardware problems is the daily instrument log. Once a day (when in use) following the routine tuning procedure, the lens, gas and RF settings should be documented, and a short term stability test and a scan be performed. Hard copies of settings, stabilities, and once a week spectra, should be put into the log book.

If you have any doubts about the performance of your instrument, trained Thermo Fisher Scientific Technical Support personnel are always available to assist you, however trivial you think the problem may be.

This chapter deals with the following topics:

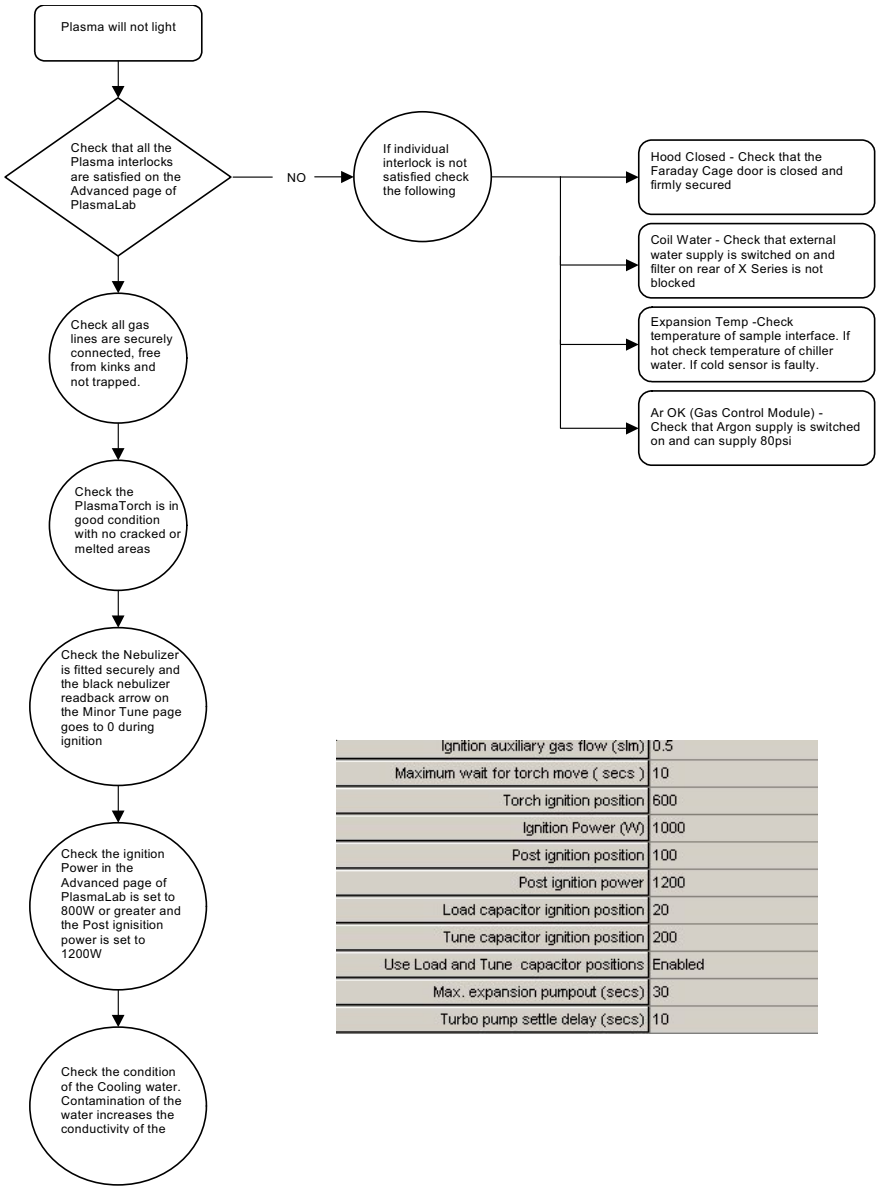
Step 1 - Vacuum Ready and Operator Mode

Instrument will not go from *Vacuum Ready* to *Operate* mode

If the instrument does not respond to any command, check that the instrument and data system are communicating via the *PlasmaLab Service* icon. If required, stop and restart the service.

When PlasmaLab fails to start the instrument successfully, a fault message is placed in the Technician/Event Log. View this message first as it is the quickest and easiest way to pinpoint the problem.

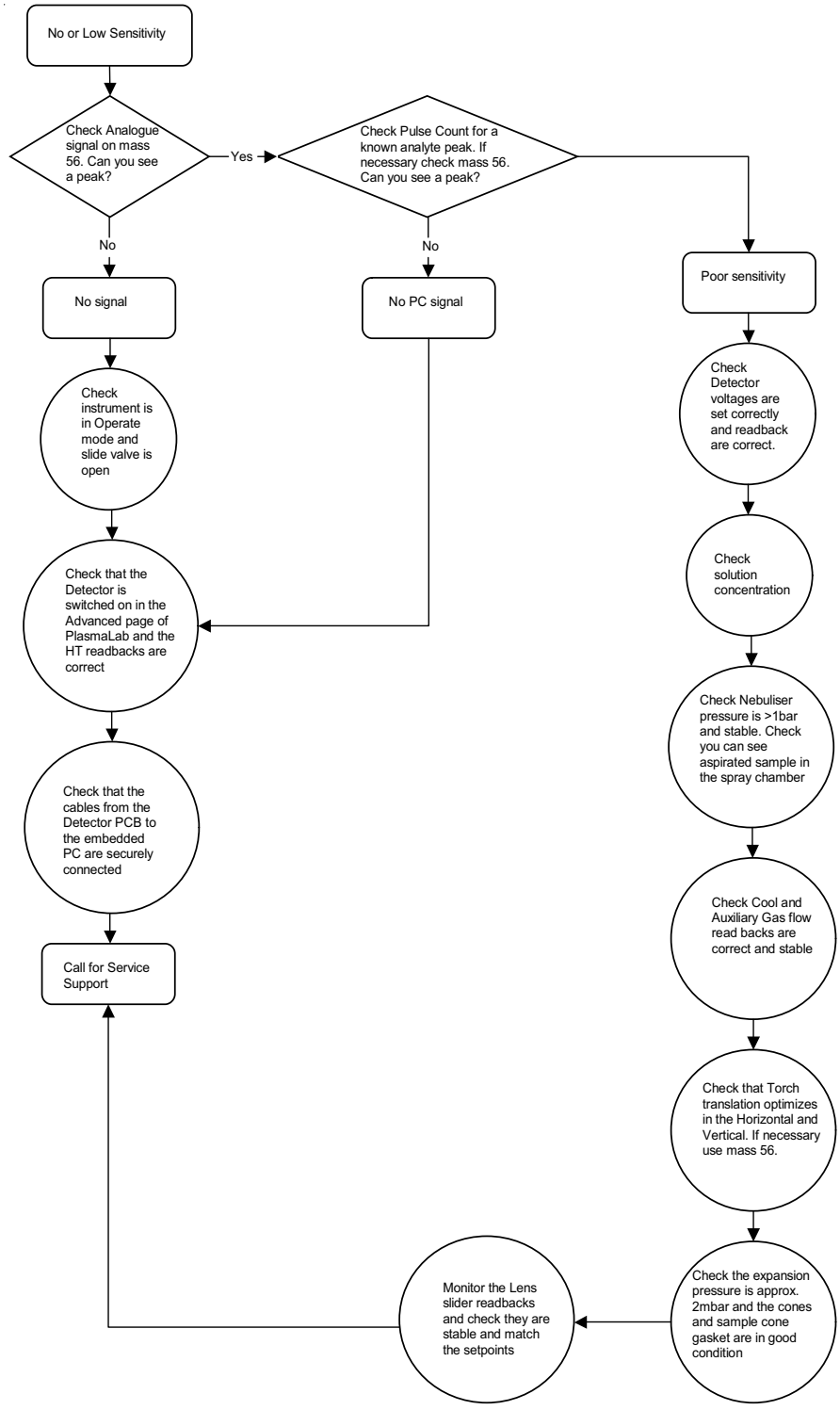
Note The *Technician* icon changes from yellow to red, if a problem is detected. ▲



Ignition auxiliary gas flow (slm)	0.5
Maximum wait for torch move (secs)	10
Torch ignition position	600
Ignition Power (W)	1000
Post ignition position	100
Post ignition power	1200
Load capacitor ignition position	20
Tune capacitor ignition position	200
Use Load and Tune capacitor positions	Enabled
Max. expansion pumpout (secs)	30
Turbo pump settle delay (secs)	10

One of the most common causes for the instrument not to start is a failure to light the plasma. The flowchart above shows a simple step-by-step guide which covers the most frequent causes of this failure.

Step 2 - Sensitivity Problems



The majority of concerns about performance surround an aspect of signal sensitivity.

In most cases, the problems are trivial, caused by a recent change in the sample introduction system or tuning, and can be addressed with ease. Only occasionally are they caused by failure of a critical component.

The daily log of instrument performance is most useful. Check the previous days log. Make sure that none of the instrument parameters has significantly changed. Use the guide above to help to diagnose the problem.

Step 3 - Signal Drifts with Time

The usual cause of signal drift is deposition of matrix on the sample and skimmer cones. As material builds up, the sampling and skimmer orifices become smaller, and the total ion beam is reduced. Removal and cleaning of the cones will resolve this problem.

However, if you suspect an instrumental drift, perform a short term stability test and examine the % RSD's obtained. Use the guide below to diagnose the problem:

1. Ensure the total dissolved solids level of the solution being aspirated is below the recommended levels (that is, less than 0.2% m/v).

2. Check that the laboratory temperature is stable.

3. Check that the temperature of the coolant water being supplied to the instrument is stable.

4. Monitor the temperature of the Peltier control (where fitted), and check that it is stable.

5. Monitor the *nebulizer slider control* read back. Check that it is stable and matches the *nebulizer slider set point*.

6. Monitor the *nebulizer pressure* value. It should be stable and consistent with values previously recorded for the same nebulizer/flow conditions.

7. Inspect the sample introduction system. Ensure that all components are correctly fitted and all connections are secure.

8. Check that the tension on the peristaltic pump tubing is correct and that the pump tube is in good condition.

9. Monitor the *lens slider control* read backs and check that they are stable. Set the *real time display* to monitor a low, mid and high mass peak (e.g. Be, In, U). Check that the mass calibration is accurate.

Step 4 - Poor Stability

Stability is interpreted as the ability of the instrument to produce good % RSD values on repeats of the same sample (sometimes defined as *internal precision*). Poor stability is often the reason behind a set of data being discarded. Sample matrix can affect stability, especially if the nebulizer cannot form a stable aerosol. System-related stability problems will occur, if short term stability test % RSD's worse than 2 to 3% are obtained when running a standard tune solution.

1. Ensure the total dissolved solids level of the solution being aspirated is below the recommended levels (that is, less than 0.2% m/v).

2. Check that the laboratory temperature is stable.

3. Check that the temperature of the coolant water being supplied to the instrument is stable.

4. Monitor the temperature of the Peltier control (where fitted) and check that it is stable.

5. Monitor the *nebulizer slider control* read back. Check that it is stable and matches the *nebulizer slider set point*.

6. Monitor the *nebulizer pressure* value. It should be stable and consistent with values previously recorded for the same nebulizer/flow conditions.

7. Inspect the sample introduction system. Ensure that all components are correctly fitted and that all connections are secure.

8. Check that the tension on the peristaltic pump tubing is correct and that the pump tube is in good condition.

9. Monitor the *lens slider control* read backs and check that they are stable. Set the *real time display* to monitor a low, mid and high mass peak (e.g. Be, In, U). Check that the mass calibration is accurate.

Glossary

This section lists and defines terms used in this manual. It also includes acronyms, metric prefixes, symbols, and abbreviations.

A ampere

AA Atomic Absorption

ac alternating current

ACX Accessory Control Language

ADC analog-to-digital converter

ASCII American Standard Code for Information Interchange

b bit

B byte (8 b)

baud rate data transmission speed in events per second

°C degrees Celsius

cm centimeter

cm^3 cubic centimeter

CPU central processing unit (of a computer)

<Ctrl> control key on the terminal keyboard

d depth

Da dalton

DAC digital-to-analog converter

dc direct current

Diluent The solution used to dilute the sample from the XSERIES 2 before it enters the ICP-MS.

EI electron ionization

<Enter> Enter key on the terminal keyboard

EPA Environmental Protection Agency. A part of the US federal government that enforces environmental laws and provides information and guidance to policy makers.

ESD electrostatic discharge

eV electron volt

f femto (10^{-15})

°F degrees Fahrenheit

ft foot

g gram

G Gauss; giga (10^9)

GC gas chromatograph; gas chromatography

GC/MS gas chromatograph / mass spectrometer

GND electrical ground

GPIB general-purpose interface bus

GUI graphical user interface

h hour

Host Computer The computer used to control the XSERIES 2 ICP-MS.

HPLC high-performance liquid chromatograph

HV high voltage

Hz hertz (cycles per second)

ICP inductively coupled plasma

ID inside diameter

IEC International Electrotechnical Commission

IEEE Institute of Electrical and Electronics Engineers

in inch

ISF internal standard factor

k kilo (10^3, 1000)

K kilo (2^{10}, 1024)

kg kilogram

l length

L liter

LAN local area network

lb pound

LC liquid chromatograph; liquid chromatography

LC/MS liquid chromatograph / mass spectrometer

LED light-emitting diode

LIMS Laboratory Information Management Systems

µ micro (10^{-6})

m meter

m milli (10^{-3})

M mega (10^6)

$M^{+\bullet}$ molecular ion

MB Megabyte (1 048 576 bytes)

MH^+ protonated molecular ion

min minute

mL milliliter

mm millimeter

MS mass spectrometer; mass spectrometry

MS MS^n power: where n = 1

MS/MS MS^n power: where n = 2

MS^n MS^n power: where n = 1 through 10

m/z mass-to-charge ratio

n nano (10^{-9})

NIST National Institute of Standards and Technology (USA)

OD outside diameter

Ω ohm

p pico (10^{-12})

Pa pascal

PCB printed circuit board

Peristaltic Pump The pump on the ICP-MS used to control flow of solution into the instrument.

PID proportional / integral / differential

PlasmaLab The Thermo Fisher Scientific software package used to control and acquire data from the XSERIES 2 ICP-MS.

P/N part number

P/P peak-to-peak voltage

ppm parts per million

psig pounds per square inch, gauge

PTFE Polytetrafluoroethylene

RAM random access memory

RF radio frequency

RMS root mean square

ROM read-only memory

RS-232 industry standard for serial communications

RSD The % Relative Standard Deviation. This figure is widely used to quote the stability of repeat readings taken using an ICP-MS.

RSF relative sensitivity factors

s second

T Tesla

TCDD 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin

TCP/IP transmission control protocol / Internet protocol

TIC total ion current

Torr torr

u atomic mass unit

V volt

V ac volts alternating current

V dc volts direct current

vol volume

w width

W watt

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