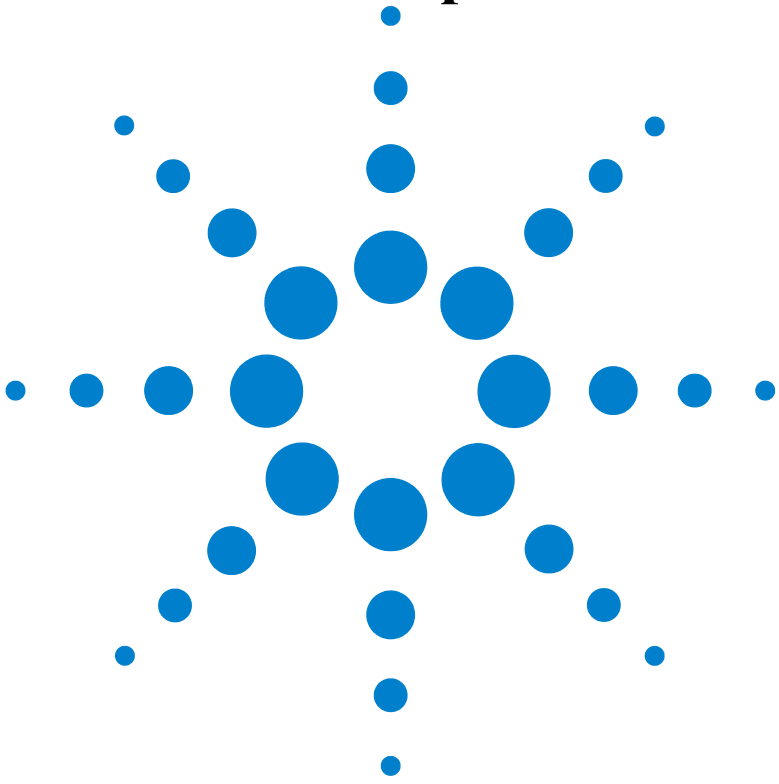
Agilent 7500 ICP-MS ChemStation (G1834B) Operator's Manual



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Agilent 7500 ICP-MS ChemStation(G1834B) Operator's Manual

G3270-90112

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Preface

Preface

The *Agilent 7500 ICP-MS ChemStation Operator's Manual* explains the procedures necessary to use the Agilent 7500 for both sample and data analysis. It also provides information about accessing the software and ensuring optimal instrument performance.

Who Should Read This Book

The primary audience for the *Agilent 7500 ICP-MS ChemStation Operator's Manual* consists of chemists and instrument operators in a laboratory. To use this manual effectively, you should have a strong knowledge of chemistry, at least a basic level of computer experience.

How to Use This Book

The *Agilent 7500 ICP-MS ChemStation Operator's Manual* contains the following chapters:

Chapter 1, "Using the Agilent 7500 ICP-MS ChemStation Software", explains how to access and exit the software and provides information about basic Microsoft® Windows™ tools.

Chapter 2, "Configuration" explains how to configure the ChemStation software.

Chapter 3, "Startup, Shutdown and Status", explains how to start and shut down the instrument, as well as, check instrument status.

Chapter 4, "Tuning", explains how to tune the instrument mainly using the standard torch.

Chapter 5, "Creating a Method", explains how to create a method for data acquisition.

Chapter 6, "Setting Up a Sequence", explains how to arrange samples when analyzing samples automatically.

Chapter 7, "Chained Sequence", explains how to edit and use chained sequence.

Chapter 8, "Running a Sample Analysis", explains how to analyze an unknown sample.

Chapter 9, "Viewing Spectra", explains how to interpret the spectra from an unknown sample to determine what elements are present in the sample.

Chapter 10, "Viewing a Time Chart", explains how to analyze the results from an unknown sample acquired using time resolved or time program mode to determine what elements are present in the sample.

Chapter 11, "Creating Custom Reports Database", describes how to create a template to use for generating custom reports and how to set up databases using the results of quantitative and semiquantitative analysis.

Chapter 12, "Performing a Quantitative Analysis", explains how to determine concentrations of elements in unknown samples by comparing with calibration curves.

Chapter 13, "Performing a Semiquantitative Analysis", explains how to determine concentrations of all elements based on a previously stored element response file.

Chapter 14, "Performing an Isotope Ratio Analysis", explains how to measure and quantitate isotope ratios.

Chapter 15, "Performing an Isotope Dilution Analysis", explains how to determine the concentration of elements using an isotope dilution technique.

Chapter 16, "Tools Menu", explains how to generate multiple types of reports for multiple data files continuously using DoList.

Chapter 17, "Database Editor", explains how to edit the ICP-MS databases which are supplied with the ChemStation.

Chapter 18, "Installing the Agilent 7500 ChemStation Software (Windows XP)", explains how to set up the computer and install the ChemStation software.

Chapter 19, "Installing the Agilent 7500 ChemStation Software (Windows 2000)", explains how to set up the computer and install the ChemStation software.

Appendix A, provides a reference of Agilent 7500 ChemStation menus. You can quickly determine which menu to access for a specific purpose.

Appendix B, provides a reference of description and equations for Calibration, Quantitation, SemiQuantitaiton and Averaging Repetition Files.

Appendix C, explains how to measure the Dead Time of the detector. For normal use, the Dead time Calibration is not necessary.

Appendix D, explains file compression and decompression.

Appendix E, explains precautions for installing the ICP-MS ChemStation and the LC/GC ChemStation on a single PC and using the two in combination.

Conventions Used in This Book

This book uses certain conventions to distinguish different types of information easily. This section describes these conventions.

Instructions

All step-by-step instructions are numbered and in bold, as in the following example.

1 Select *Instrument*>>Tune from the *ICP-MS Top* Window.

The **Sensitivity** tuning window appears.

Alternatives

Lines beginning with a bullet (•) in step-by-step instructions indicate alternative steps, as in the following example:

2 Change the parameter value in one of the following ways:

- Click the right or left arrow at the end of the scroll bar, which changes the parameter value in small increments.
- Click the scroll bar, which changes the parameter value in larger increments.
- Double-click the box above the scroll bar and enter a new parameter value.

Menu Items

This book uses the following format for referring to menu items:

Instrument>>Tune

The text before the arrow symbol is the name of the menu; the text after the arrow symbol is the menu choice. This example refers to the Tune menu choice in the Instrument menu.

Terminology

This book frequently uses the following terms:

Convention	Information
Press	To hold down a button on the keyboard.
Click	To press and release the mouse button.
Double-click	To click the mouse button twice in rapid succession.
Drag	To press and hold the mouse button while moving the pointer.
Active Window	The window in which the cursor is currently located.
Radio Buttons	Choices where you can select only one item from a list. The selected radio button contains a solid dot. A grayed out radio button is a choice that is unavailable at a given time.
Check Box	Choice where you can select or clear the named item. Multiple items can be chosen simultaneously and are each marked with an X in a square. You cannot use a grayed out check box.
Push Button	Button used to initiate an immediate action. Cancel and Help are examples of push buttons. Push buttons are labeled on the buttons themselves.
ICP-MS	An inductively coupled plasma mass spectrometer.
Agilent 7500	The Agilent Technologies 7500 ICP-MS.
ChemStation	The ChemStation software for Agilent Technologies ICP-MS.

Notes

Notes contain a reminder about the effect of particular actions. They are indicated as follows:

NOTE This example shows how a note is displayed.

Cautions

Cautions indicate situations that could overwrite data or require immediate attention to prevent harm to the instrument. Cautions are indicated as follows:

CAUTION 

This example shows how a caution is displayed.

Warnings

Warnings indicate situations that could cause bodily harm. Warnings are indicated as follows:

WARNING 

This example shows how a warning is displayed.

Where to Go for More Information

In addition to the *Agilent 7500 ICP-MS ChemStation Operator's Manual*, you can reference the following resources:

- *Agilent 7500 ICP-MS Hardware Manual*
- *Agilent 7500 ICP-MS Option Instruction Manual*
- *Agilent 7500 ICP-MS Application Handbook*
- *Agilent 7500 ICP-MS Customer Maintenance Parts List*
- *Online Help*

Your computer system has additional manuals that document the software and hardware. For more information on using or maintaining your computer, printer, or peripheral equipment, consult the respective hardware user's guide. Microsoft® Windows™ have a user's guide that explains how to install, use, and troubleshoot the software.

Agilent Technologies on Internet

For the latest information on products and services visit our worldwide web site on the internet at:

http://www.agilent.com/chem/icpms

1

Using the Agilent 7500 ICP-MS ChemStation Software

Using the Agilent 7500 ICP-MS ChemStation Software

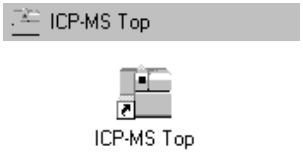
The Agilent 7500 inductively coupled plasma-mass spectrometer (ICP-MS) is capable of measuring trace elements at levels as low as one part per trillion or quickly scanning more than 70 elements to determine an unknown sample's composition. Controlling this instrument through the ChemStation, you can safely analyze each sample and accurately interpret the resulting data.

Accessing and Exiting the Software

Accessing the Software

Click on ***Start*** on the Windows Task bar and select ***Programs>>ICP-MS ChemStation***.

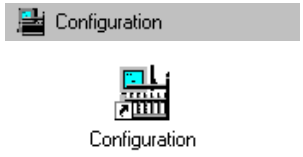
Several menus appear. Each menu accesses a different function of the software, as described in the following list. Select the menu you want to use.



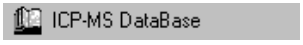
The ICP-MS Top icon starts the software and allows you to access ChemStation functions pertaining to the instrument, data acquisition, and data analysis. It also enables you to create and run methods and sequences. This manual contains information about these ChemStation functions.

The Offline Data Analysis icon starts the offline analysis function. With this function, you can manipulate data that has already been acquired, while at the same time acquiring new data. For information about this function, see Chapter 9 through 15.

The Offline Edit Sample Log Table icon allows you to edit the sample log table offline. It can be used while a sequence or method is running. For more information about this function, see Chapter 6, "Set Up a Sequence" and the appropriate sections of the Intelligent Sequence Manual.



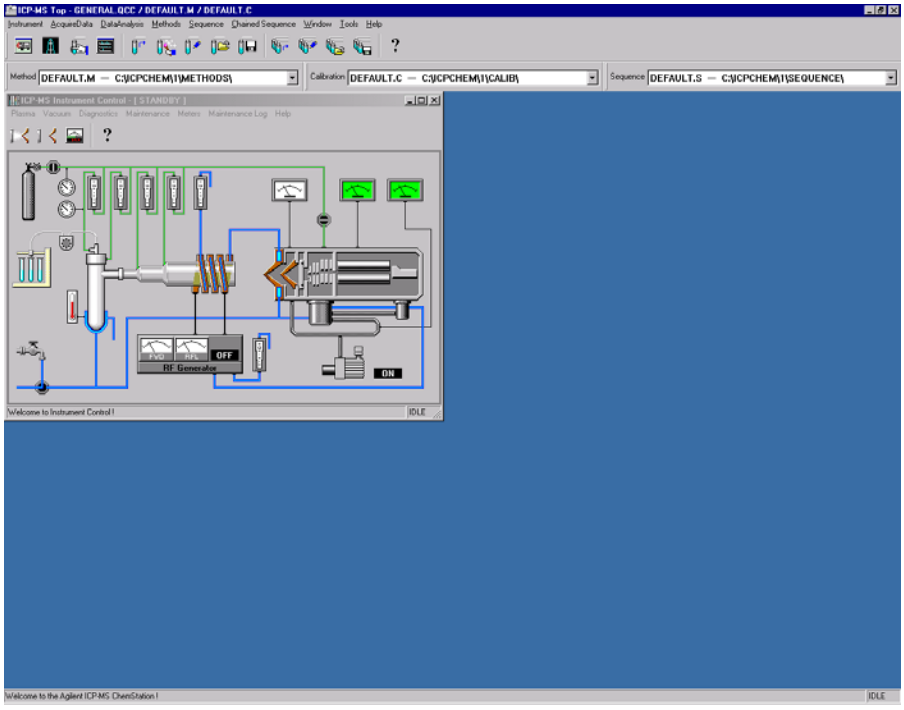
The Configuration icon provides access to software configuration and remote access. For more information about configuration, see Chapter 2, "Configuration".



The ICP-MS DataBase icon opens the ICP-MS DataBase, which provides element, AMU, and interference information. For more information about the ICP-MS DataBase, see Chapter 5, "Creating a Method".

The DataBase Editor icon opens "Select database" dialog box, which allows the creation and editing of the above ICP-MS database. For more information about the DataBase Editor, see Chapter 17, "Editing the Database"

The ***ICP-MS Top*** window appears when the ***ICP-MS Top*** is selected
The ***ICP-MS Top*** window shows the last window displayed when ChemStation was closed. To use a function of ChemStation, select it on the menu bar or click on the icon. The drop lists for ***Methods***, ***Calibration***, and ***Sequence*** each show the most recently used files. These lists provide easy access to files.



ICP-MS Top Window

NOTE

Please start the ChemStation either from the shortcut key which are automatically displayed upon installation, or through the ***Start>>Programs>>ICP-MS ChemStation*** sequence. Starting ChemStation from Windows NT Explorer or the shortcut key which is made peculiarly can cause a software mal-function.

Exiting the Software

Use any one of the following methods to exit ChemStation. Be sure to save any changes before exiting.

- Double-click the Control Box Menu in the upper left corner of the ***ICP-MS Top*** window, or click the Control Box Menu and then click ***Close*** in the pop-up menu that appears.
- Click on the "close" button in the upper right corner of the ***ICP-MS Top*** window.
- Select ***Instrument>>Exit*** from the ***ICP-MS Top*** window.

All of these choices caution you to ensure your work is saved and then ask if you want to exit now. Click ***Yes*** to exit or ***No*** to return to the program and save your work or make more changes.

NOTE Exiting ChemStation does not shut off the plasma. Ensure that the plasma is off before you exit ChemStation. For more information, see Chapter 3, "Startup, Shutdown and Status"

Using Online Help

Online help is available from ***Help*** menu in the windows or ***Help*** buttons in the dialog boxes. The help menu/button opens the Online Help window.

Moving within the Software

The ChemStation Top window remains open while you use ChemStation. To open other ChemStation windows, select items from the Top drop down menus or the tool bar. This section discusses how to use the mouse and how to move between windows.

Using the Mouse

Use the mouse to select functions or text, to push buttons and to move scroll bars. For example, you can select a word in a list box by double-clicking it. You can also click ***OK*** to accept changes made in a dialog box.

Use the left mouse button when clicking or double-clicking unless using the right mouse button is specified.

To move from one field to the next when entering information in a dialog box, click the field you are moving to. You can also press the ***Tab*** key unless otherwise indicated. To move to a previous field, click the field, or while holding down the ***Shift*** key, press the ***Tab*** key.

Moving Between Windows

In the ***ICP-MS Top*** window, multiple task windows can be open at the same time. A different task can be selected by clicking in any one of the windows or a menu command can be executed in the selected window. However, a new task cannot be selected and a new menu command cannot be executed if a dialog box is opened or a command is being executed in the actual task window, unless the dialog box is closed or the command execution is completed.

To activate a non-active window, click on any part of that window. The window then moves to the front of the screen.

To minimize a window, click on the "Minimize" button in the upper right corner of the menu bar. Only the button on the Windows Task bar remains. To change a button to a window, double-click the button, or click the button and click ***Maximize*** in the pop-up menu that appears.

For More Information

For more information on using the mouse and on other Windows features, refer to your Microsoft Windows documentation and the online Windows tutorial.

Using the Command Line

The command line is used to create and edit macros to customize ChemStation. You can turn on the command line in two ways:

- Click the Control Box Menu in the upper left corner of any of the main windows and click ***Cmdline on***.
- Enter ***CMD OFF*** in the command line.

When not using the command line, you may want to turn it off. You can turn off the command line in two ways:

- Close the window you are currently in and then reopen it.
- Enter ***CMD OFF*** in the command line.

You can view a list of available commands by typing ***COM*** at the command line in ***ICP-MS Instrument Control***, ***ICP-MS Tuning***, and ***ICP-MS Data Analysis*** windows.

Supplemental Information

Listed in this section is useful information for using the ICP-MS ChemStation software:

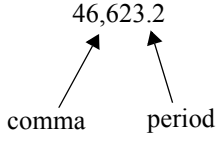
- File names should not be any longer than 8 letters and/or numbers.

  It must not contain the following characters:

Period (.)	Slash (/)	Brackets ([ ])
Comma (,)	Backslash (\ )	Vertical bar (|)
Semicolon (;)	Equal sign (+)	Space ( )
Colon (:)	Quotation mark (")	

- If the ICP-MS ChemStation hangs up and you need to shutdown the software using Windows task manager, you must select ***ICPACQ.EXE, ICPDA.EXE, ICPTUNE.EXE***, and ***MSTOP.EXE*** and click on the ***End Process*** button to completely shutdown ChemStation.
- Please use ". (periods)" for decimal points, and ", (commas)" for separating digits.

e.g. forty six thousand six hundred and twenty three point two:



2

Configuration

Configuration

If you have changed the system configuration, change the software settings accordingly. When you start the ***Configuration*** task, the ***ICP-MS Configuration*** dialog box appears, enter sample introduction settings such as whether to use a peristaltic pump, autosampler, etc.

This chapter explains how to configure the ChemStation software.

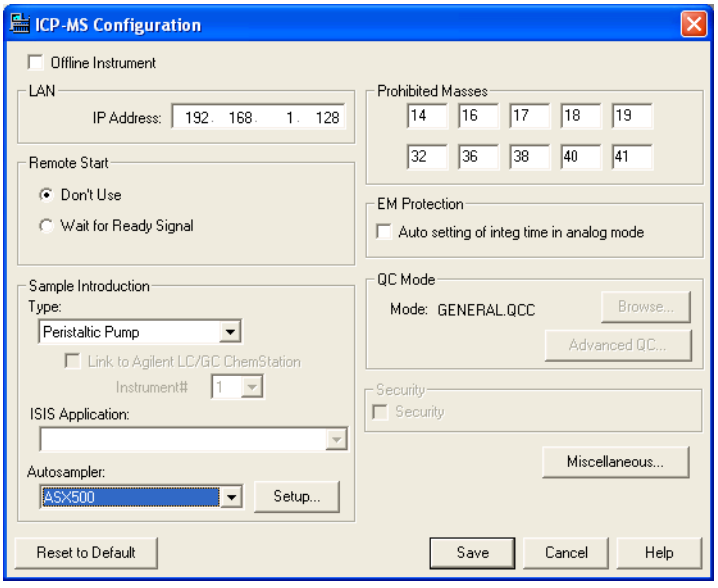
Configuring the ChemStation Software

To configure the ChemStation, complete the following steps:

1 **Click the *Configuration* short cut key, or click the *Start* button and select *Programs>>ICP-MS ChemStation>>Configuration* from the *Task bar.***

The ICP-MS Configuration dialog box appears.

NOTE The ***Configuration*** task cannot be started while other tasks are executing, so end all other tasks first.



ICP-MS Configuration Dialog Box

2 Set the *Offline Instrument* check box to OFF (do not check this box).

On-line is the state in which data is transferred via the LAN or GPIB interface. In this mode, you use ChemStation to control the operation , tuning and data analysis

Off-line, on the other hand**,** is the state in which ChemStation is used alone without transferring data between ChemStation and the instrument, for example, to analyze existing data.

To use ChemStation off-line, set the ***Off-line Instrument*** check box to **ON** (Check this box).

3 Set the ICP-MS address in the GPIB area.

The initial value is set to 20. If the ***Offline Instrument*** box is checked, this value is not used.

4 Set the Remote Start to start the data acquisition by an external signal.

Setting this mode allows the start of data acquisition using an external signal, e.g. Laser Ablation System.

- Don't Use

Select this if you do not want to use the remote start mode.

- Wait until Ready Signal

Select this if you want to use the remote start mode.

5 Set the sample introduction.

CAUTION 

RS-232 Configuration is necessary when ISIS or Autosampler are used. For the setting method, see "I/O Library Setup" in Chapters 18 or 19.

- Type

None :	Select if you are not using the peristaltic pump, ISIS, or LC/GC/Laser Ablation.
ISIS :	Select if you are using the ISIS for data aqusition
Peristaltic Pump :	Select if you are using only the instrument pump for sample uptake and/or drain
LC/GC/laser abrasion:	Uses LC/GC/laser abrasion.

- Link to Agilent LC/GC ChemStation checkbox/Instrument# combo box: Selecting ***LC/GC/Laser Ablation*** as ***Type*** enables the ***Link to Agilent LC/GC ChemStation*** checkbox. Select this checkbox to use the LC/GC ChemStation. Selecting the checkbox also enables the ***Instrument#*** combo box. Select the number of the instrument you wish to control.

NOTE Only one LC/GC ChemStation can synchronize with the ICP-MS ChemStation, and you can control only a single instrument from the LC/GC ChemStation.

CAUTION To use the LC/GC ChemStation, you must enable Remote Start. Selecting the Link to Agilent LC/GC ChemStation checkbox prevents selection of ***Don't Use*** in ***Remote Start***.

- ISIS application

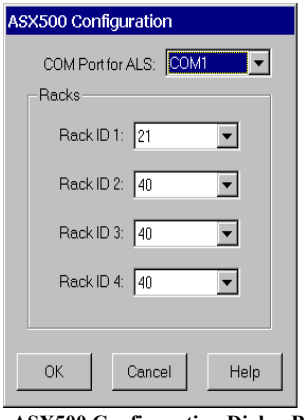
  When ISIS is selected as Type, an appropriate ISIS application, which are either standard or use defined application, should be selected in the pull-down menu.

- Autosampler

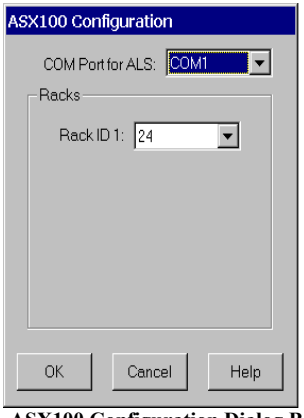
  If you want to use the autosampler (ASX500, ASX100, or I-AS), select the autosampler type in the pull-down menu. If an Autosampler is not being used, select ***None.***

  If an Autosampler type is selected, set the communication port and the sample rack size for the autosampler.

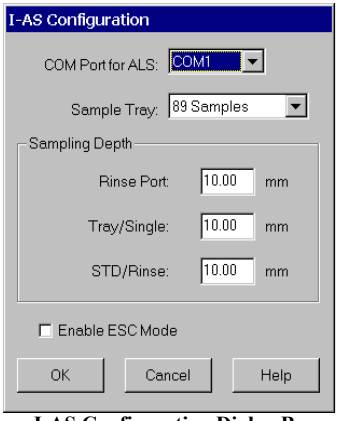
  The ***ASX500***, ***ASX100***, or ***I-AS configuration*** dialog box appears when you click ***Setup***.



ASX500 Configuration Dialog Box



ASX100 Configuration Dialog Box



I-AS Configuration Dialog Box

- COM Port for ALS

  Specify the COM port to which you want the Autosampler connected. Normally this is COM1.

- Racks (ASX500 or ASX100)

  For the ASX500, the racks are, from left to right, identified as Rack ID 1, Rack ID 2, Rack ID 3, and Rack ID 4.

  Note that if the setting is wrong the Autosampler needle will not go to the correct position.

- Sample Tray (I-AS)

  Select the sample tray that will be used.

- Sampling Depth (I-AS)

  In the locations below, set the travel distance for the I-AS arm from its highest to lowest positions. For more information about the Sapmling Depth, refer to the Integrated Autosampler Manual.

- Enable ESC Mode (I-AS)

  Check this to enter the ESC (escape) Mode. For more information about the ESC Mode, refer to the Integrated Autosampler Manual.

Click ***OK*** when the configuration of the Autosampler has been completed.

6 Set *prohibited masses*.

Insert prohibited masses to protect the detector (EM) from large signals caused by Oxygen, Argon, Nitrogen, Hydrogen, and other polyatomic ions.

CAUTION 

You should not modify the prohibited masses setting except for specific applications (For example, measurement of 40Ca with cool plasma condition). If under normal plasma conditions, large signals will cause the rapid deteriation of the detector (EM).

7 Check EM Protection

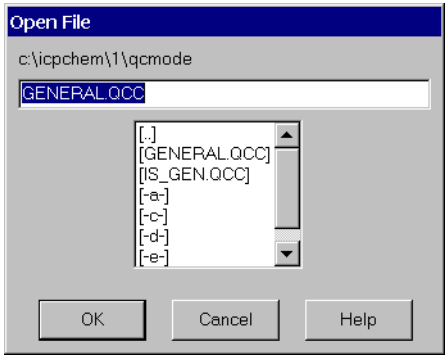
Normally, leave ***Auto setting of integ time in analog mode*** selected. If the detector mode is set to auto, the dwell time in analog mode is fixed at 100 microseconds so that large signals do not damage the detector, regardless of the dwell time value specified in the method.

In Spectrum and Isotope analysis, output counts are calculated and displayed as cps. In Time Resolved analysis, output counts are calculated to get counts for the integration time set in the acquisition parameters of the method. When ***Auto setting of integ time in analog mode*** isn't selected and the detector mode is set to AUTO in the acquisition parameters, the acquisition will be executed according to the integration time; the integration time doesn't change

8 Select a QC mode

Click ***Browse***, and select a QC mode file. QC mode files have qcc extensions. User-defined QC modes can be created by selecting ***Advanced QC***.

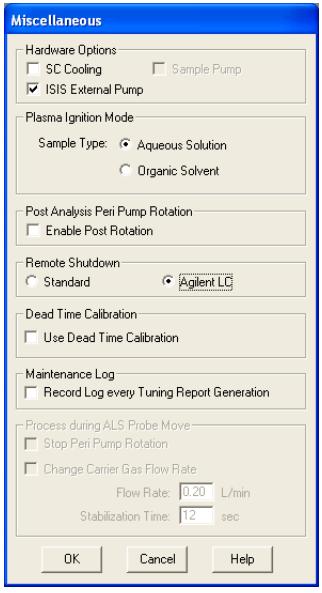
For more information about the QC configuration, refer to the ***Intelligent Sequence manual***.



Open File Dialog Box

9 Match the ChemStation configuration with the Instrument Hardware.

The ***Miscellaneous*** dialog box appears when the ***Miscellaneous*** button is clicked.



Miscellaneous Dialog Box

- Hardware Option

  SC Cooling:

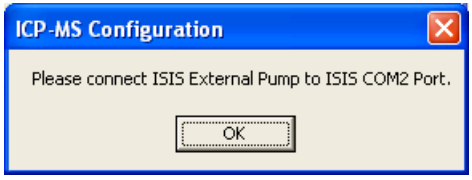
  In normal operation when introducing a liquid sample, the spray chamber is cooled to stabilize the fine particles supplied to the plasma. For other sample introduction systems like laser ablation, the connection is made directly to the torch and the spray chamber is not used. When cooling the spray chamber, select ***SC Cooling.*** When not cooling the spray chamber, the selection is not required.

  Pump2 (Option):

  It is selected when a second (optional) peristaltic pump is installed on the ICP-MS. When there is no option pump this box is not selected.

ISIS External Pump:

To enable this checkbox, select ***ISIS*** for ***Type*** in ***Sample Introduction*** in the ***ICP-MS Configuration*** dialog box, then select ***Discrete Sampling*** as the ***ISIS Application***.
Select this checkbox to use ISIS with an external pump. Selecting the checkbox displays the following dialog box. Confirm that ISIS and the external pump are connected.



Connection Confirmation dialog box

NOTE The only compatible pump is the Metrohm 818 IC Pump. Do not attempt to connect any other pumps.

- Plasma Ignition Mode

  Optimal Plasma ignition parameters differ depending upon whether the sample is an aqueous solution or an organic solvent.

  Aqueous Solution: Selected when the sample is an aqueous solution.

  Organic Solvent: Selected when the sample is an organic solvent.

- Post Analysis Peri Pump Rotation

  When the ***Enable Post Rotation*** is selected, the peristaltic pump will periodically rotate when the ICP-MS is in standby mode. This function is to protect the peristaltic pump tubing when you cannot remove the peristaltic pump tubing immediately after turning off the plasma. For example, the keyword is selected in the sample log table to turn off the plasma automatically.

- Remote Shutdown

  Standard (Default)

  The Standard option must be selected when the ICP-MS is connected to the ISIS.

  Agilent LC

  The Agilent LC option must be selected when ISIS is used with an external pump.
  The Agilent LC option can be selected when the ICP-MS is connected to any modular Agilent LC (eg. Agilent 1100, Agilent 1050) using an APG remote cable. This option will enable bidirectional communication between the LC and the ICP-MS in the event of a hardware error on either the LC or the ICP-MS. If there is a hardware error on the ICP-MS, or if the plasma is turned off via sequencing, a shutdown command will be sent to the LC. Also, if there is any hardware error on the LC, a shutdown command will be sent to the ICP-MS, which will turn off the ICP-MS plasma and switch the instrument from Analysis mode to Standby mode.

- Maintenance Log

  Record Log Every Tuning Report Generation

  When the option "Record Log Every Tuning Report Generation" is selected, printing a tuning report or selecting a tuning report in Autotune will cause the tuning parameters (as well as all parameters associated with meter indications and the status of devices) to be recorded in a maintenance log.

- Processing during ALS probe movement

  Stop PeriPump rotation

  The peristaltic pump will stop when the auto sampler probe is in motion, this will prevent air from being taken in from the sample probe.

  Change Carrier Gas Flow Rate

  Flow rate: Initially 0.2 l/min; set in a range from 0.0 to 2.00 l/min Stabilization time: Initially 12 sec; set in a range from 0 to 999 sec

  While the auto sampler probe is in motion, the carrier gas flow rate is changed to the set value. If a stabilization time is set, the probe is moved to the specified cleaning port after expiration of the set time.

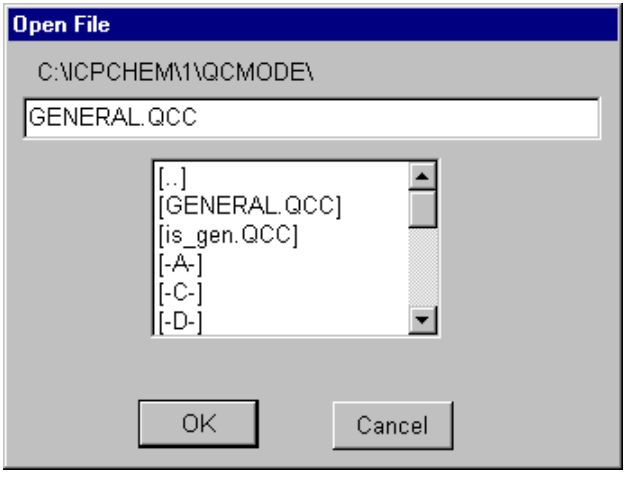
NOTE In normal use, there is no need to select Use Dead Time calibration. For more information, see Appendix C, "Dead Time Calibration".

Click ***OK*** when you complete the detail setting.

10 Click *Save.*

Save the settings and end the configuration task.

NOTE The Reset to Default, Save, Cancel, and Help buttons are displayed in the ICP-MS Configuration dialog box. If Reset to Default is clicked, it restores the factory settings (Except the setting of the toolbar and the QC configuration). If Cancel is clicked, it cancels the change of setting, and ends the ICP-MS Configuration dialog box.



Open File Dialog Box

11 Click *Save.*

Save the settings and end the configuration task.

NOTE

The ***Reset to Default, Save***, ***Cancel***, and ***Help*** buttons are displayed in the ICP-MS Configuration dialog box. If ***Reset to Default*** is clicked, it restores the factory settings (Except the setting of the toolbar and the QC configuration). If ***Cancel*** is clicked, it cancels the change of setting, and ends the ICP-MS Configuration dialog box.

3

Startup, Shutdown and Status

Startup, Shutdown and Status

The Agilent Technologies, Agilent 7500 is designed so that it is easy to start up and shut down. The instrument has three states of operation that are displayed in the Agilent 7500 ChemStation instrument control software:

- Analysis

  During tuning and sample analysis, the Agilent 7500 is in ***Analysis*** mode.

- Standby

  At the end of the working day, place the Agilent 7500 in ***Standby*** mode.

  In this mode both the turbo pumps and the backing rotary pumps remain on, but the quadrupole, detector, and RF (plasma) are switched off.

- Shutdown

  To maintain or move the Agilent 7500, put it into ***Shutdown*** mode. In an emergency, you can flip the main power switch to stop the instrument.

This chapter explains how to start and shut down the Agilent 7500, as well as, check instrument status.

Starting the Instrument

There are three states from which the Agilent 7500 can be started:

- Unplugged
- Shutdown mode
- Standby mode

This section explains how to start the instrument from all three states.

Starting the Instrument from Cold

Use the following procedure to start up the instrument after it has been unplugged for a move, maintenance, or a long-term shutdown.

1 **Ensure that the instrument is plugged in.**

Plug the instrument into a NEMA L15-30R power receptacle.

2 **Ensure that the hoses and power cables of the rotary pumps, the Ar gas hose, the computer GPIB cable to the Agilent 7500, and the water hoses are all properly connected.**

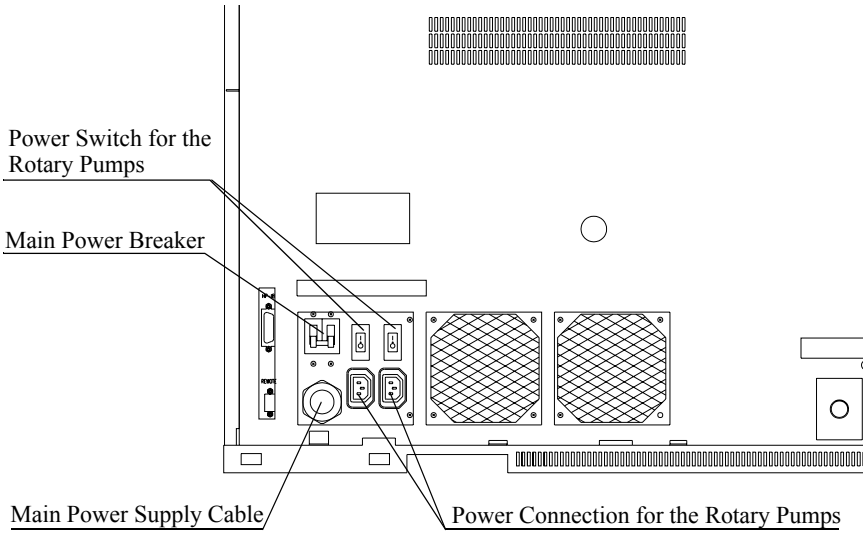
The hoses and the cables are connected on the rear side of the instrument. For more information, refer to the ***Hardware Manual***.

3 **Turn on the rotary pump power switches.**

A manual power switch located on each of the rotary pumps must first be switched on before attempting to turn on the pumps. Always check this switch before starting the vacuum of the instrument. When the switches are in the on position the pumps can then be turned on from the ChemStation. The pumps will remain off until the vacuum is turned on by the ChemStation software.

4 **Turn on the main power breaker and the pump switch(es) on the Agilent 7500.**

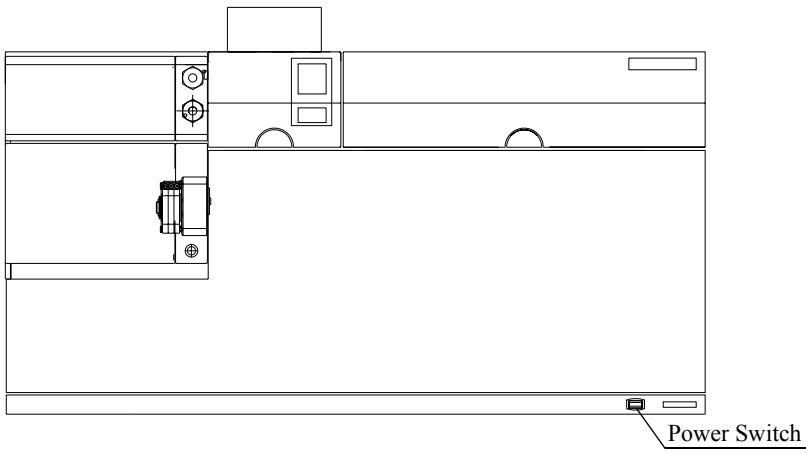
These three switches are located on the rear side.



Rear View and Control Switches

5 Turn on the instrument power switch.

The power switch is located on the front panel. The instrument now has power. And the green light on the power switch is turned on.



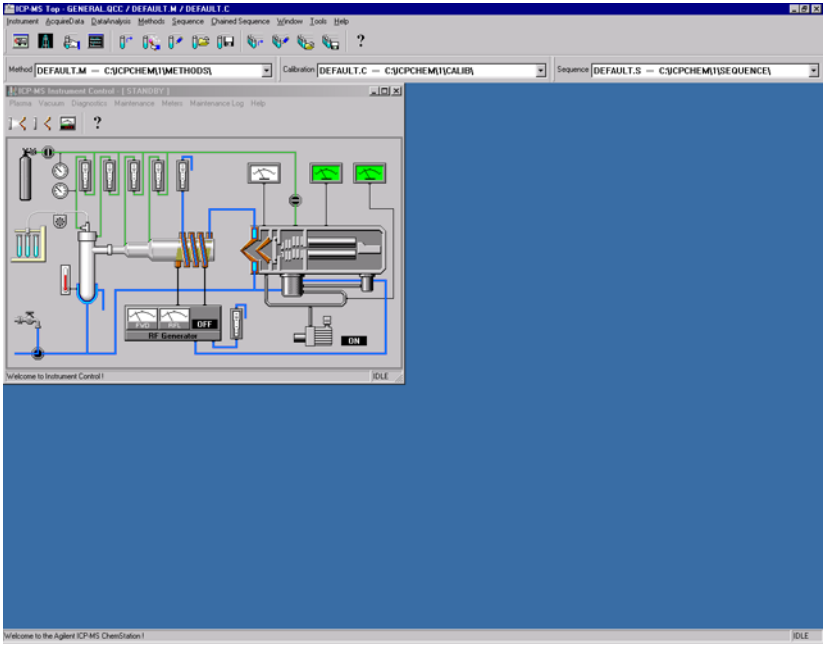
Front View of the Agilent 7500

6 Start up the printer and computer.

Refer to the printer and computer user manuals for more information.

7 Start the ChemStation software.

Refer to Chapter 1, "Using the Agilent 7500 ICP-MS ChemStation Software" for information on exiting the software. The ***ICP-MS Top*** window appears.



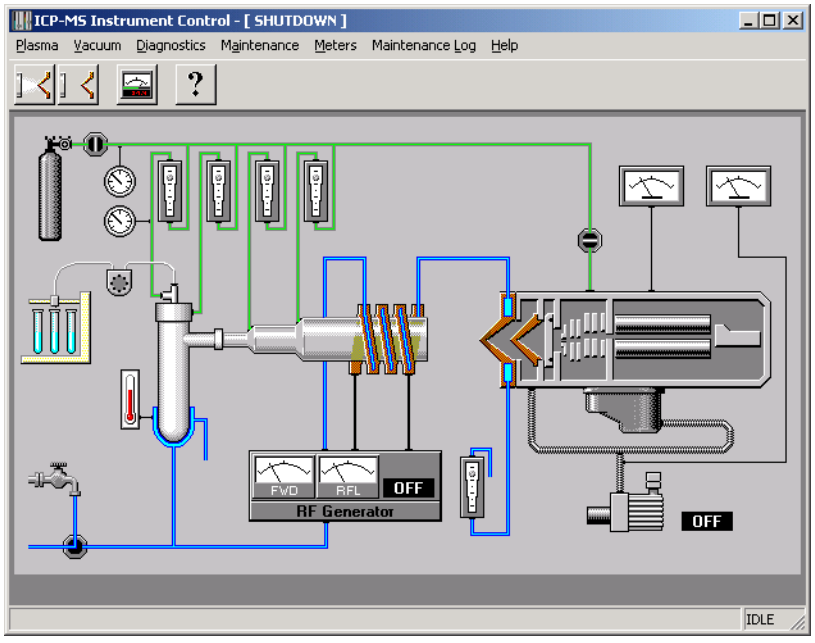
The ICP-MS Top Window

CAUTION

When the ChemStation software starts running immediately after turning on the main switch of the ICP-MS instrument, the mainboad of the instrument sometimes hangs up and becomes unavailable to control the instruments. ChemStation software should be started more than 40 seconds after turning on the main switch of the instrument.

8 Select *Instrument>>Instrument Control.*

The ***Instrument Control*** window appears. The title bar shows that the instrument is in Shutdown mode. The LED located on the top right corner of the right top cover is off during Shutdown mode. Continue with "Starting from Shutdown Mode".



Instrument Control Window in Shutdown Mode
(Picture may differ depending on ICP-MS Model/Mode)

Starting from Shutdown Mode

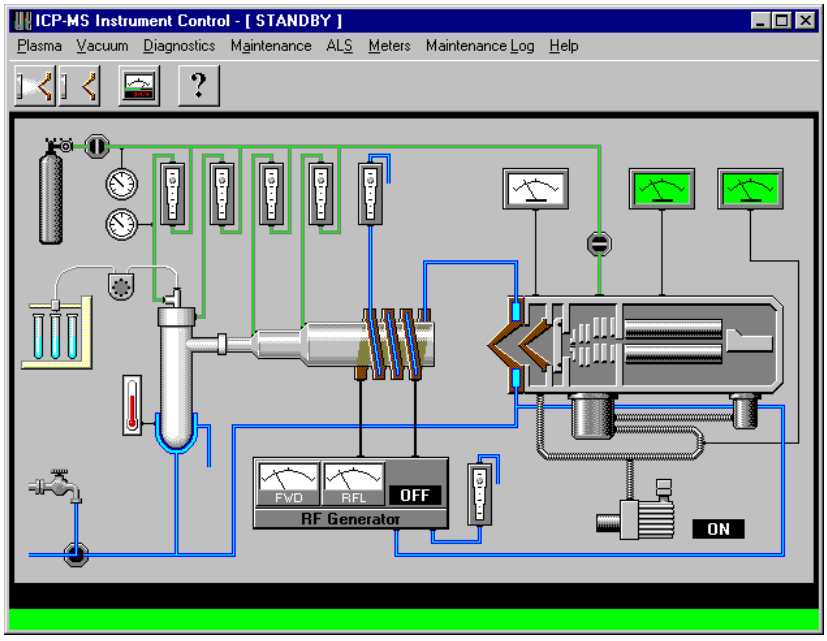
The following instructions assume that the instrument is turned on and the ***Instrument Control*** window is displayed. See "Starting the Instrument from Cold" to perform those tasks. Then, complete the following steps:

1 Select *Vacuum>>Vacuum On*.

A dialog box appears, asking if you want to turn the vacuum on.

2 Click *Yes*.

The instrument will turn on the rotary pump, open the backing line valve and turn on the turbo pump. The ***Instrument Control*** diagram displayed on the ChemStation will indicate that the rotary pump is on. It can take from 15 minutes to 2 hours for the vacuum chamber to attain its correct pressure of 5 x 10^{-4} Pa. The instrument is now in ***Standby*** mode, as displayed in the title bar. The process takes longer when the vacuum chamber has been open to the atmosphere for any length of time.



Instrument Control Window in Standby Mode
(Picture may differ depending on ICP-MS Model/Mode)

Starting from Standby Mode

The following instructions assume that the instrument is in ***Standby*** mode and the analyzer vacuum chamber is evacuated to a partial or high vacuum. See "Starting from Shutdown Mode" to evacuate the vacuum chamber.

The plasma is off when the Agilent 7500 is in ***Standby*** mode. At this point the Plasma can be switched on. Once the interface vacuum has reached 4.5 x 10^2 Pa and the plasma is on, the instrument will switch to ***Analysis*** mode and you can perform tuning and sample analysis. To start the instrument from ***Standby*** mode, complete the following steps:

1 **Ensure that the instrument is in Standby mode.**

The title bar shows that the instrument is in the ***Shutdown*** mode. Also the LED on the top cover displays an orange light. (A blinking orange LED indicates that the mode is changing).

2 **Close the hoods.**

Close both the hoods tightly. The safety function is tripped if either hood is open. The plasma will not ignite and go to the analysis mode if the covers are open.

3 **Ensure that the vent's air flow is acceptable.**

The exhaust air flow should be 5 m^3/min. See Chapter 2, "Precautions" in the "*Agilent 7500 ICP-MS Hardware Manual*", for information on "Checking the Exhaust System".

4 **Supply the instrument with cooling water and argon gas.**

The cooling water chiller must be turned on.

The primary argon gas pressure should be:

100 ± 2.8 psi (700 ± 20 kPa)

5 **Ensure that the drain vessel is empty.**

See Chapter 2, "Precautions" in the "*Agilent 7500 ICP-MS Hardware Manual*", for information on emptying the drain vessel.

6 **Ensure that the gas tubing is not pinched and that the torch box and sample introduction gas connections seal correctly.**

Check the gas tubing and make adjustments if necessary. See Chapter 4, "Maintenance" in the "*Agilent 7500 ICP-MS Hardware Manual*", for information on connecting the gas hoses.

7 **Insure that all peristaltic pump (peripump) tubes are in good condition and correctly clamped into the peristaltic pumps.**

CAUTION 

Be sure clamp the peripump tubing for the drain in the peripump. The drain from the spray chamber is provided by this peripump. If the spray chamber is not drained properly, it will fill with solution causing solution to flow to the torch which will cause the plasma to turn off.

8 Select *Plasma>>Plasma On*.

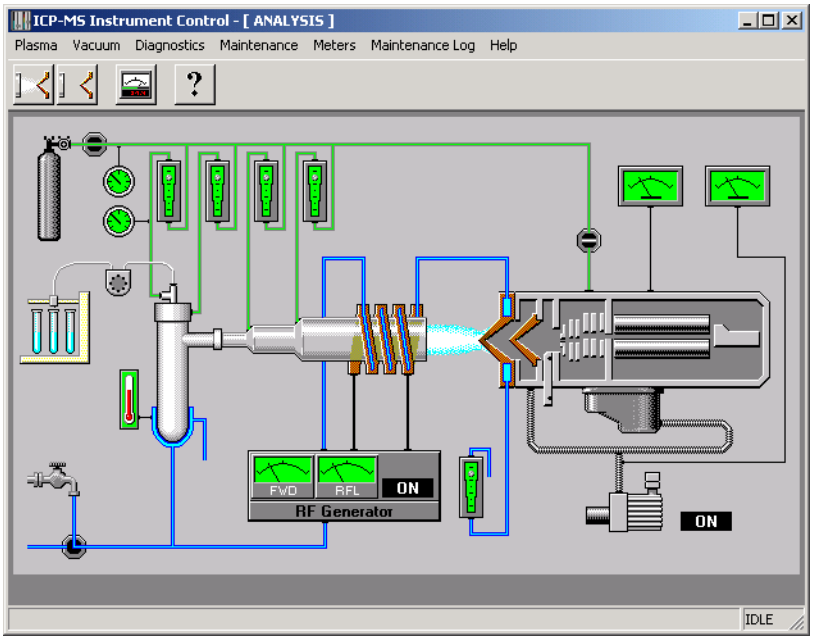
A dialog box appears, asking if you want to turn the plasma on.

9 Click *Yes*.

The gases for the plasma torch are switched on and the gas lines are purged. The plasma ignites and the interface rotary pump starts to evacuate the interface vacuum chamber. When changing to the Analysis mode is completed, the title bar indicates that the instrument has changed from the Standby to Analysis mode.

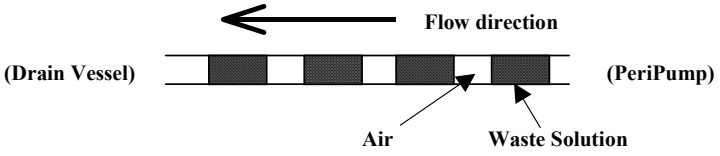
NOTE

The Agilent 7500 returns to the Standby mode when the plasma is off and remains there unless it is being shut down for major maintenance or for relocation.



Instrument Control Screen in Analysis Mode
(Picture may differ depending on ICP-MS Model/Mode)

WARNING



Confirm that the drain is flowing. Check that the tube from the drain peristaltic pump to the drain bottle is properly connected. You should see a regular pattern of liquid and air as shown above.

Shutting Down the Instrument

The Agilent 7500 is in ***Analysis*** mode during tuning and sample analysis. When not being used for these procedures, the instrument remains in ***Standby*** mode. To maintain or move the instrument, it should be placed in ***Shutdown*** mode and turned off.

The following sections explain how to return the Agilent 7500 to ***Standby*** mode at the end of each day and how to put the instrument in ***Shutdown*** mode when necessary.

Putting the Instrument in Standby Mode

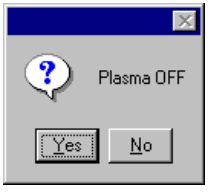
To return the instrument to ***Standby*** mode after running samples, complete the following steps:

1 **Select *Instrument>>Instrument Control* from the Top task.**

The ***Instrument Control*** window appears.

2 **Select *Plasma>>Plasma Off***

The dialog box appears.



3 **Click Yes.**

The Instrument Diagram shows that the plasma is off. The Agilent 7500 automatically turns off all gases, the torch RF generator, the peripump, and the cooling water flow, after cooling the torch and interfaces. Only the vacuum pumps remain on.

CAUTION Ensure that positive extraction remains operative while the instrument is in Standby mode. The rotary pump exhaust continues to pass into the exhaust duct when the instrument is in Standby mode.

NOTE While the instrument is in the Standby mode, turning off the computer and the printer will not cause any problems.

Putting the Instrument in Shutdown Mode

CAUTION  Except in emergency situations, you should put the Agilent 7500 into the Shutdown mode before turning off the main power.

To return the instrument to Shutdown mode, complete the following steps:

1 **Put the Instrument into Standby Mode**

Follow the instructions in "Putting the Instrument in Standby Mode"

2 **Supply argon gas by opening the argon gas valve.**

When the instrument is changing to ***Shutdown*** mode, argon gas is introduced into the vacuum chamber. For this reason, the argon gas supply needs to be opened until the instrument has switched to ***Shutdown*** mode.

3 **Select *Vacuum>>Vacuum Off***

The dialog box appears.

4 **Click *Yes*.**

The instrument starts to go to the ***Shutdown*** mode. After a short time, argon gas is introduced into the vacuum chamber. This is done to prevent pump oil from going up into the vacuum chamber. The turbo and rotary pumps are turned off. Only the main power to the instrument remains on. The title bar of the instrument control task shows that the instrument changes to ***Shutdown*** mode.

NOTE If argon gas is not supplied, air will be introduced into the vacuum chamber instead of argon. In this case, it may take more time to go back to the Standby mode.

Turning the Instrument Completely Off

Turn the Agilent 7500 completely off when you want to perform maintenance on electrical components or to move the instrument. To achieve a total shutdown, complete the following steps:

1 **Put the Instrument into Shutdown Mode**

Follow the instructions in "Putting the Instrument in Shutdown Mode"

2 **Exit the software and turn off the computer and printer**

Refer to Chapter 1, "Using the Agilent 7500 ICP-MS ChemStation Software", and the printer and computer user's manuals for more information.

3 **Turn off the power switch**

The switches are located on the front side. The power indicator light is now off.

4 **If needed, unplug the instrument**

Checking the Instrument Status

The Agilent 7500 ChemStation software (ChemStation) provides two methods for checking the instrument status:

- Instrument Control Diagram
- Meter Control Panel

This section explains how to use these monitoring screens.

Checking the Instrument Control Diagram

The ***Instrument Control*** window is a real-time display that enables you to quickly assess the current status of the instrument. It is a simplified representation of Agilent 7500 components. The window includes gauges, meters and on/off indicators, each of which corresponds to an instrument component. You can use this window when the instrument is in ***Shutdown***, ***Standby***, or ***Analysis*** mode.

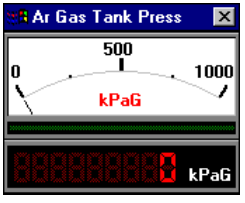
To access and interpret the ***Instrument Control*** window, complete the following steps:

1 **Select *Instrument>>Instrument Control* from the Top menu.**

The ***Instrument Control*** window appears. The title bar shows what mode the instrument is in.

2 **Starting from the upper left corner of the window, place the cursor on the circular gauge. Press the left mouse button when the cursor changes to a small meter box.**

The ***Argon Tank Gas Pressure*** meter box appears, showing the value in kilopascals (kPa). The value depends on the type of nebulizer used.



Argon Gas Tank Pressure Meter Box

3 **Moving the cursor clockwise, place it on the next gauge and press the left mouse button when the cursor changes to the small meter box.**

The ***Carrier Gas Flow*** meter box appears. An acceptable range of values for this meter is application specific.

4 **Repeat Step 3 until you have checked all 14 system components on the *Instrument Control* screen.**

Checking the Meter Control Panel

ChemStation also provides a ***Meter Control Panel*** for checking the instrument's status. Selected parameters appear on the screen in the meter boxes that provide real-time operating values for the Agilent 7500 components. The ***Meter Control Panel*** enables you to monitor more components than you can access from the ***Instrument Control*** window. Also the meters remain on the screen until you choose to remove them or close the ***Instrument Control*** window.

NOTE Up to five meter boxes can be displayed at one time. If you select a sixth meter to monitor, you must deselect one already displayed.

To monitor the Agilent 7500 internal and external environments, complete the following steps:

1 **Select *Instrument>>Instrument Control*.**

The ***Instrument Control*** screen appears.

2 **Select *Meters>>Meter Control Panel*.**

The ***Meters*** panel appears.

3 **Select up to five components to monitor by clicking the appropriate check boxes and then clicking *OK*.**

Meter boxes for the selected components appear on the right side of the screen. Check the following table to ensure that values displayed are within acceptable limits.

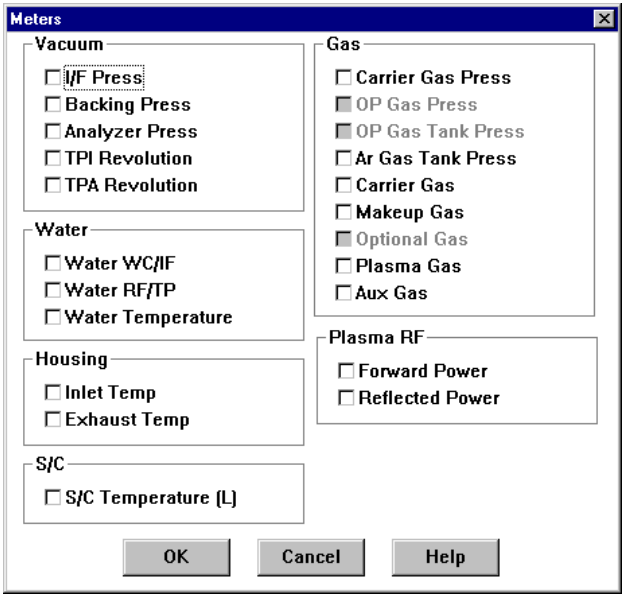
NOTE A delay in displaying the value on the meter in the can occur due to processing time required by PC.

NOTE Meter boxes are displayed until you close them individually or close the Instrument Control window. Meter boxes that are open when the Instrument Control window is closed will redisplay when this window is reopened

4 To monitor other components, deselect the current ones by clicking the appropriate boxes. Then click the boxes next to the additional components you want to monitor.

Meter boxes for the newly selected components appear on the right side of the screen.

5 Repeat Step 4 until all necessary components have been checked.



Meters Panel

Meter Control Panel Values

Meter	Typical Range		
	Shut Down Mode	Standby Mode	Analysis Mode
IF Press	---	---	250 to 490 (1 RP) * 180 to 300 (2 RPs)
Backing Press	---	0.3 to 5	250 to 490 (1 RP) * 180 to 300 (2 RPs)
Analyzer Press	---	$3x10^{-5}$ to $6x10^{-4}$	$3x10^{-4}$ to $2x10^{-3}$
TPI Revolution	0	65 to 100	95 to 100
TPA Revolution	0	65 to 100	95 to 100
Water WC/IF	0.00	0.00	1.1 to 2.0
Water RF/TP	0.00	0.00	1.1 to 3.0
Water Temp	15 to 25	15 to 25	15 to 25
Inlet Temp	Room Temperature	Room Temperature	25 to 35
Vent Temp	Room Temperature	Room Temperature	45 to 55
S/C Temp (L)	Room Temperature	Room Temperature	2
Carrier Gas Press	0	0	450 to 600 (BB) ** 300 to 400 (CF) 200 to 300 (CN)
OP Gas Press	0	0	Depends on application
OP Gas Tank Press	0	0	Depends on application
Argon Gas Tank Press	700	700	700

(continued)

Meter	Typical Range			Unit
	Shut Down Mode	Standby Mode	Analysis Mode	
Carrier Gas Flow	0	0	0.8 to 1.3	L/min
Makeup Gas Flow	0	0	0 to 1.0	L/min
Optional Gas Flow	0	0	Depends on application	%
Plasma Gas Flow	0	0	15	L/min
Aux Gas Flow	0	0	0 to 1.0	L/min
Forward Power	0	0	700 to 1600	W
Reflected Power	0	0	< 20	W

***RP: Rotary Pump**
****BB: Babington Nebulizer, CF: Cross-flow Nebulizer, CN: Concentric Nebulizer**

Ion Lenses Test

This test will check to determine if any of the ion lenses are shorted.

1 **Verify that the ion lenses are connected to the equipment.**

If the ion lenses are not connected, the test results will not be valid because the ion lenses will not be detected as being shorted.

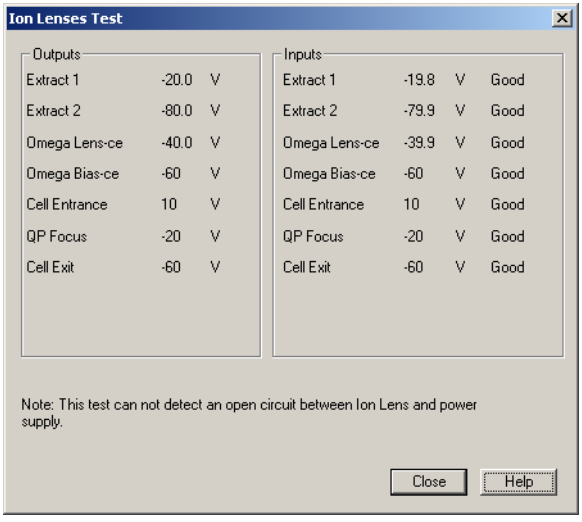
2 **Place the equipment in standby mode and verify that the cover switch is closed.**

3 **Select *Instrument>> Instrument Control* from the the Top menu.**

The ***ICP-MS Instrument Control*** window will be displayed.

4 **Select *Diagnostics >> Ion Lenses Test.***

The ***Ion Lenses Test*** dialog box will be displayed.



Ion Lenses Test dialog box

The ion lenses will be checked and the measured values displayed in the Inputs area five seconds later.

Compare the set value in the Outputs area with the measured value in the Inputs area. If the measured value is within +/-1 V of the set value, "OK" will be displayed to the right of the measured value. Measured values out of this range will be marked with "NG"

5 Click *Close*.

The ***Ion Lenses Test*** dialog box will be closed.

Nebulizer Test

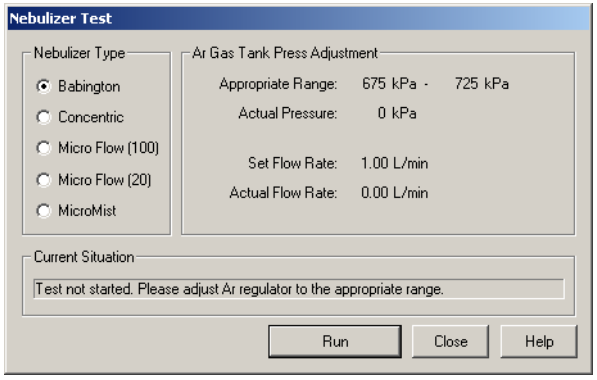
This test checks whether the nebulizer pressure is in the appropriate range.

1 Select *Instrument* >> *Instrument Control* from the Top menu.

The ***ICP-MS Instrument Control*** window will be displayed.

2 Select *Diagnostics* >> *Nebulizer* Test.

The ***Nebulizer Test*** dialog box will be displayed.



Nebulizer Test dialog box

3 Select the type of nebulizer from the *Nebulizer Type* area.

4 Adjust the source pressure of the argon gas to fit within the appropriate range using a regulator.

The appropriate range will vary with the type of nebulizer and main unit.

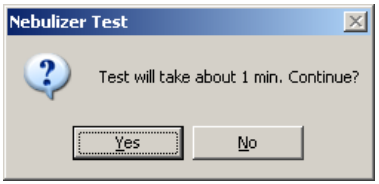
(x denotes a numeral)

Nebulizer type	Main unit type G327xA	G315xA/B	Flow rate for pressure adjustment
Babington	675 - 725 kPa (700 kPa + - 3.5%)	675 - 725 kPa (700 kPa +- 3.5%)	1.0 ml/min
Concentric		482 - 518 kPa (500 kPa +- 3.5%)	1.0 ml/min
Micro Flow(100)		337 - 363 kPa (350 kpa +/- 3.5%)	0.8 ml/min
Micro Flow(20)		337 - 363 kPa (350 kpa +/- 3.5%)	0.8 ml/min
MicroMist		675 - 725 kPa (700 kPa +- 3.5%)	1.0 ml/min

5 Click *Run*.

The source pressure of the argon gas will be checked.

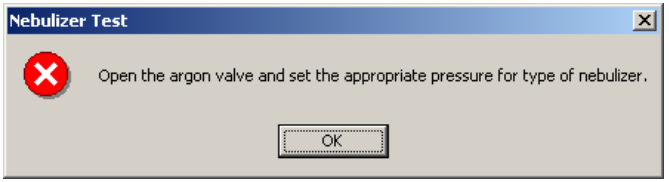
The following message appears when the source pressure is within the appropriate range.



This dialog box appears when the source pressure is within the appropriate range

Click ***Yes*** to execute the nebulizer test.

The following message appears when the source pressure is outside of the appropriate range.

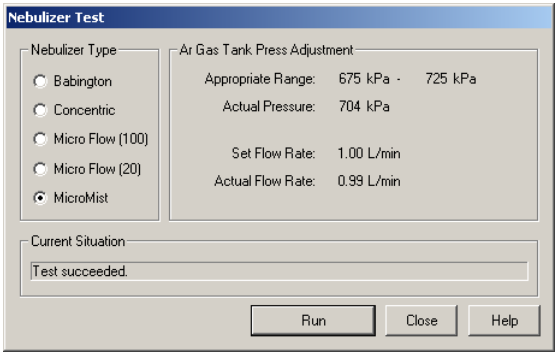


This dialog box appears when the source pressure is outside of the appropriate range

If the source pressure is outside of the appropriate range, a message will be 5 displayed telling the user to adjust the source pressure. Click ***OK*** and perform the procedure again beginning with step 4.

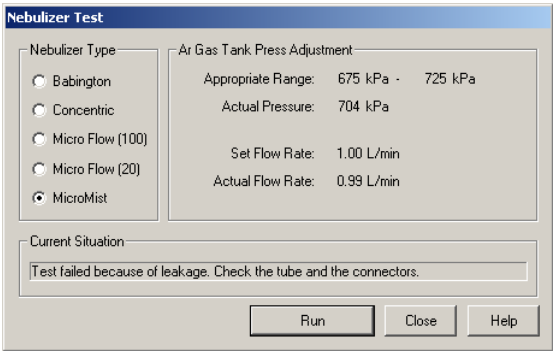
6 The test result will be displayed when the nebulizer test is completed.

If there are no problems with the measured values, a message indicating that the test has been successfully conducted will be displayed in the ***Current Situation*** area.



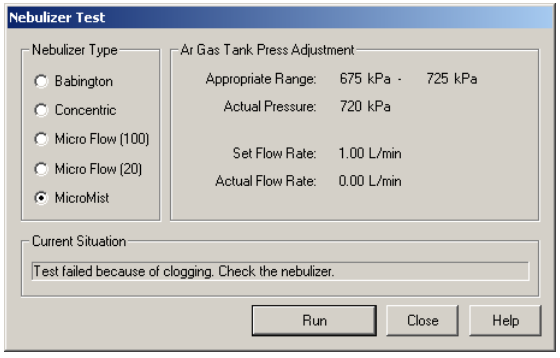
This dialog box appears when there are no problems with the measured values

If the average value of the source pressures of the argon gas is 100 kPa or less, a message indicating that the test has failed will be displayed in the ***Current Situation*** area. Check to see whether the nebulizer is leaking.



This dialog box appears when there is a leak in the nebulizer.

If the average value of the measured flow rates is "set flow rate value - 0.1" or less or the average value of the carrier gas pressures exceeds "argon tank pressure by 0.95," a message indicating that the test has failed will be displayed in the ***Current Situation*** area. Check to see whether the nebulizer is clogged.



This dialog box appears when there is a clog in the nebulizer.

7 Click *Close*.

The ***Nebulizer Test*** dialog box will be closed.

Typical Meter Values for the Vacuum System

Refer to the following meter values when the error occurs.

1 After the vacuum is ON

Parameters	Limit	Conditions
Backing pressure (BK)	> 100 Pa	if BK is higher, even 60 seconds after the vacuum is ON.
Backing pressure (BK)	> 25 Pa	if BK is higher, even 70 minutes after the vacuum is ON.
Analyzer pressure (AN)	> 5 x 10^{-4} Pa	if AN is higher, even 5 hours after the vacuum is ON.

The Agilent 7500 goes back to Shutdown mode.

2 After the plasma is OFF

Parameters	Limit	Conditions
Interface pressure	> 450 Pa	if IF is higher, even 2 minutes after the IF pump is ON.
Analyzer pressure (AN)	> 1.2 x 10^{-3} Pa	if AN is higher, even 3 minutes after the gate valve is opened.

The Agilent 7500 goes back to Shutdown mode.

3 After the plasma is OFF

Parameters	Limit	Conditions
Analyzer pressure (AN)	> 5 x 10^{-4} Pa	if AN is higher, even 15 seconds after the gate valve is closed.

The Agilent 7500 goes back to Shutdown mode.

4 In Standby mode

Parameters	Limit	Conditions
Analyzer pressure (AN)	> 2 x 10^{-3} Pa	Agilent 7500 goes back to Shutdown mode.
Backing pressure (BK)	> 40 Pa	Agilent 7500 goes back to Shutdown mode.

5 In Analysis mode

Parameters	Limit	Conditions
Interface pressure (IF)	> 530 Pa	Agilent 7500 goes back to Standby mode.
Analyzer pressure (AN)	> 2 x 10^{-3} Pa	Agilent 7500 goes back to Standby mode.
Backing pressure (BK)	> 40 Pa	Agilent 7500 goes back to Shutdown mode.

4

Tuning

Tuning

Tuning allows you to monitor and adjust Agilent 7500 performance before beginning sample analysis. Tuning and sample analysis should be performed in analysis mode. You can change tuning parameters to adjust the instrument's condition for a particular application. The tuning parameters can be saved to a file and used for the next tuning session.

Optimization enables autotune, tuning for a specific purpose, or resolution of a tuning problem to achieve optimum sensitivity. The Agilent 7500 ChemStation software simplifies this task. Within the tuning windows, you can adjust parameters for the sample introduction, torch box, analyzer and detector system components. You tune the Agilent 7500 by monitoring the signal produced from running a tuning solution through the instrument. Then, if necessary, you adjust the operating parameters until the instrument's signal meets performance requirements.

For most applications, use a tuning solution of four elements covering the whole range of masses. This ensures good Agilent 7500 performance when analyzing all masses. For special applications requiring ultimate sensitivity at a certain mass, you can tune the instrument using an element in that mass range. For routine use, the Agilent 7500 has ample sensitivity at all masses simultaneously.

In this chapter the basic tuning method at the time of mainly using the standard torch has been explained. Please see the "Option Instruction Manual" for the details of tuning when the ShieldTorch is used.

Overview

Tuning involves the following steps:

- Introducing and ionizing samples efficiently in the plasma and introducing the ions into the vacuum systems

  See "Tuning for Sensitivity (Agilent 7500a)" on page 4-4.

  It is especially important to set the plasma condition for reducing the oxide and/or doubly Charged Ions. See "Reducing Oxide Ions" on page 4-12 and "Reducing Doubly Charged Ions" on page 4-15.

  If you are using the Agilent 7500ce, refer to "Tuning for Sensitivity (Agilent 7500ce)" on page 4-39.

  If you are using the Agilent 7500cs, refer to "Tuning for Sensitivity (Agilent 7500cs)" on page 4-62.

- Setting the ion lens voltage to introduce the ions into the Q-pole

  See "Tuning for Sensitivity (Agilent 7500a)" on page 4-4.

  If you are using the Agilent 7500ce, refer to "Tuning for Sensitivity (Agilent 7500ce)" on page 4-39.

  If you are using the Agilent 7500cs, refer to "Tuning for Sensitivity (Agilent 7500cs)" on page 4-62.

- Setting the parameter for the Q-pole to recognize the mass number (m/z) correctly

  See "Tuning for Resolution and Mass Axis" in this chapter.

- Setting the detector to count the ions correctly and to convert analog into pulse

  See "Tuning for the Detector" in this chapter.

- Generating a Tune Report

  See "Generating a Tune Report" in this chapter.

Tuning for Sensitivity (Agilent 7500a)

Tune the Agilent 7500 for sensitivity to ensure that the instrument produces the best results for the masses being analyzed. Achieve good sensitivity by running a recommended tuning solution of 10 parts per billion (ppb) of Li, Y, Ce and Tl.

If you are using the Agilent 7500ce, refer to "Tuning for Sensitivity (Agilent 7500ce)" on page 4-39.

If you are using the Agilent 7500cs, refer to "Tuning for Sensitivity (Agilent 7500cs)" on page 4-62.

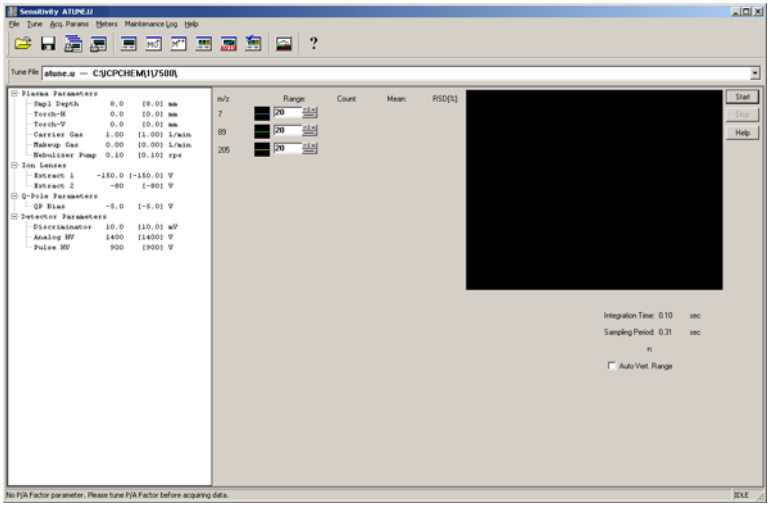
NOTE

When an internal standard solution is added to a sample by the peristaltic pump and the internal standard solution overlaps tuning elements, it might cause wrong tuning parameters. To avoid this situation, the sample tube for the internal standard solution must be placed in pure water while tuning.

To tune for sensitivity, complete the following steps:

1 **Select *Instrument>>Tune* in the *ICP-MS Top* window.**

The ***ICP-MS Tuning-Sensitivity*** window for sensitivity appears.



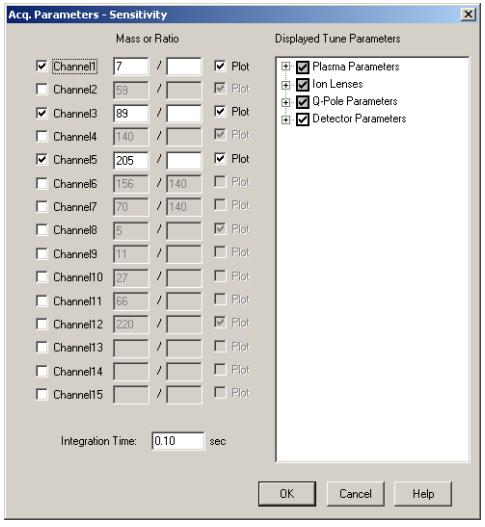
The ICP-MS Tuning-Sensitivity Window

2 Select *Tune>>Acq. Params.*

The ***Acquisition Parameters - Sensitivity*** dialog box appears.

Use this dialog box to set or change the masses you want to view, the integration time, and the ratio of any two channels. You can also display the ratio of signal strengths of any two selected masses.

This dialog box also allows you to select the channels to be displayed on the ***ICP-MS Tuning - Sensitivity*** window, the tuning parameters to be indicated in the tree display, and the channels to be plotted on the graph.



Acquisition Parameters-Sensitivity Dialog Box

NOTE To use tuning parameter settings from a previous tuning session as a starting point for this session, see "Creating and Using a Tune File".

3 Select the check boxes of the channels to be displayed on the *ICP-MS Tuning - Sensitivity* window.

4 Enter the values of the masses in the left-side boxes under "Mass or Ratio."

5 To set a strength ratio, type the mass values in the right-side boxes under "Mass or Ratio."

6 Enter the data collection time (integration time) for each mass in the Integration Time: text box

Generally, set 0.1 seconds per one mass value.

7 Select the *Plot* check boxes of the channels to be displayed on the graph in the *ICP-MS Tuning - Sensitivity* window.

8 In the Displayed Tuning Parameters tree display, select the check boxes of the tuning parameters to be displayed in the *ICP-MS Tuning - Sensitivity* window.

Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Clicking on a parameter check box will alternately select or deselect the parameter.

A parameter in gray indicates that only some of the parameters contained within are selected.

9 Click *OK*.

Clicking ***OK*** closes the ***Acquisition Parameters - Sensitivity*** window and displays the entered settings in the ***ICP-MS Tuning - Sensitivity*** window.

10 Put the sample uptake tube into the tuning solution.

Wait for uptake of the sample to the nebulizer.

11 Click *Start* to monitor the Agilent 7500's signal and view the numerical values in the real-time display.

As the signal appears, ChemStation inserts numerical values for the count, mean, RSD, and ratio.

The window includes a box for a real-time display of the instrument's signal. The box contains up to 200 data points. The current counts of each mass are given, and the mean and RSD (relative standard deviation) of the signal are given for the data points shown in the display.

NOTE When you check ***Auto Vert. Range*** check box, the vertical range of the real-time display is adjusted automatically.

NOTE You can print a copy of the real-time display at any time during the tuning procedure. See "Generating a Tune Report" later in this chapter.

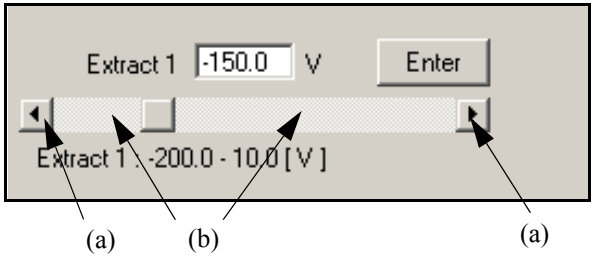
12 Adjust the tuning parameters if necessary.

Tuning parameters are shown in the tree display on the left side of the window. Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Indicated on the right side of each parameter in [ ] are the adjustment value and the pre-adjustment numerical value (value stored in the tune file).

Clicking on a parameter will cause a text box or a scroll bar to appear at the lower center section of the window. Use one of the following methods to set a parameter value.

- Enter a new parameter value in the text box (next to the parameter you want to change or above the scroll bar).
- Click the right or left arrow at the end of the scroll bar (a), which changes the parameter value by small increments.
- Click the scroll bar (b), which changes the parameter value by larger increments.
- Click ***Apply*** to set the adjusted parameter value.
  Clicking ***Cancel*** will cause the pre-adjustment parameter value (value stored in the tune file) to be set.



Adjust the tuning parameters until the signal appears constant and the numerical values meet performance requirements. The performance specification depends on the application.

CAUTION

Use care when adjusting values by clicking the scroll bar or entering a new value in the box above the scroll bar. Changing parameter values too quickly can cause extreme changes in the Agilent 7500 and can alter its performance over time. In most cases, you should adjust the tuning parameters by clicking the arrows at the end of the scroll bar.

Typical value of Sensitivity and RSD (Using the normal torch)

Mass	Counts / 10 ppb Integ. Time = 0.1sec	RSD
^{7}Li	>6400	<15%
^{89}Y	>16000	<15%
^{205}Tl	>9600	<15%

Plasma Condition

- RF Power

  Controls RF power supplied to the load coil. A higher RF power increases the sensitivity and reduces oxide and doubly charged ions. However, the sensitivity of lighter masses may be decreased when the RF power is too high.

- RF Matching

  Adjusts reflected RF returned from the load coil.
  To reduce the reflected power, use of the automatic adjustment function is recommended. To use the automatic adjustment function, select **Tune>>RF Matching** on the Tuning window, and then follow the displayed dialog box.

NOTE

If you adjust the RF Matching manually, use only the scroll arrows to change this value. If you click on the scroll bar itself, the instrument will turn off the plasma. Adjust until the RF reflected power meter reads as low as possible. Standard value for RF matching is approximately 1.6 to 2.0 V.

- Smpl Depth

  Controls distance between the edge of the load coil and the tip of the sampling cone. A shorter sampling depth increases sensitivity, but oxide level increases as well. If the torch is too far from the interface opening for the plasma flow to sustain the interface pressure, the plasma is turned off.

- Torch-H, Torch-V

  Controls torch position. The Torch-H and Torch-V parameters measure the horizontal and vertical movement of the torch relative to the interface. Adjusts to get the highest sensitivity for all masses. When the sampling depth is changed,

adjustment of Torch-H/V will be required. If these parameters are changed using the software scroll bar, the plasma may turn off. Use only the scroll arrows.

- Carrier Gas

  Controls nebulization efficiency of sample and sample uptake rate when self-aspiration is applied. A higher gas flow rate increases the sensitivity of the analysis for low masses, but too high a carrier gas flow rate will result in increased oxide and doubly charged levels.

- Makeup Gas

  Makes up Ar gas into the spray chamber is mixed with the carrier gas. When the Ar gas supply pressure is not high enough to get a sufficient carrier gas flow, the makeup gas is used. (e.g. cool plasma condition)

- Optional Gas (option)

  When the optional gas line is added, it is possible to introduce gas other than argon. (e.g. introduction of oxygen etc. when measuring organic solvent)

- PeriPump 1

  Controls a sample uptake rate and drain from the spray chamber. It affects sensitivity, stability of signal, oxide and doubly charged formation. Higher speed increases sensitivity, but too high speed decreases sensitivity. Higher speed increases oxide and doubly charged ions.

CAUTION 

Even when self-aspiration is used for sampling, the PeriPump 1 must be operated to drain the sample from the spray chamber, if this is not optimized the spray chamber will fill with sample solution.

- PeriPump 2 (option)

  It can be used when a second (optional) peristaltic pump is installed on the ICP-MS. It is possible to shorten the rinse time or the sample uptake time.

- S/C Temp

  Controls the temperature of the spray chamber. Lowering the spray chamber temperature lowers the sample vapor pressure and removes more water, reducing oxide levels. It should be set at 2 ºC for aqueous sample to avoid ice formation. When aspirating organic solvents run the chamber at -5 ºC.

Ion lenses

- Extraction 1 & 2
  It extracts ions from the plasma to accelerate them toward the Einzel lens. Adjust to get enough sensitivity for all masses. This is lens that needs the most frequently adjusted everyday.

  In order to get maximum sensitivity, adjust between 0 V and 6 V. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass gets maximum sensitivity at high voltage. Therefore, select the appropriate voltage. Even with the voltage around -100V, high sensitivity can be obtained. However, the background of polyatomic ions may be high in the reaction mode (H_2 mode or He mode)

- Einzel 1,3
  It focuses the ion beam from the extraction lens. A typical value is -80V. If the voltage becomes lower, sensitivity will go up, but the off-mass background will also be high.

- Einzel 2
  It focuses the ion beam from the extraction lens. An element of low mass number gets maximum sensitivity at low voltage. On the other hand, an element of high mass number gets maximum sensitivity at high voltage. Therefore, set a value to get enough sensitivity for all masses.

- Omega Bias

  Provides the same potential at the entrance and exit of the Omega lens block. It must be used at voltages more positive than -40V. The more negative voltage, the higher the sensitivity, but the background might increase.

- Omega (+), Omega(-)

  Separating the ions from photon and introduce the ions to the Q-pole. Adjusts to get good sensitivity for all masses.

- QP Focus

  Decelerates and focuses the lenses and introduces the ions to the Q-pole. Adjusts to get good sensitivity for all masses.

- Plate Bias

  Re-focuses the ion beam. A typical value is 0 V without the ShieldTorch system. It may be used several voltage when the peak shape and resolution are not good. This voltage should be the same as the QP Bias to avoid higher background.

- QP Bias

  Controls the speed of ions as they pass through the Q-pole. A typical value is 0 V without the ShieldTorch system. It may be used up to 6 V when the peak shape and resolution are not good. This voltage should be the same as the Plate Bias to avoid higher background.

13 Click *Stop* to stop the display.

ChemStation freezes the real-time display until you click ***Start***. When you click ***Start***, the display clears and then starts again.

Typical values of tuning parameters

Parameter	Typical Conditions	Adjustment
RF Power (W)	1300	1200 to 1600
Sampling Depth (mm)	6	4 to 8
Carrier gas (L/min)	1.2	0.8 to 1.3
Makeup gas (L/min)	0	0 to 0.4
Peri-pump 1 (rps)	0.1	0.06 to 0.15
S/C Temp (°C)	2	Normally used at 2 °C
Extraction 1 (V)	-150	-200 to -100
Extraction 2 (V)	-70	-150 to -10
Einzel 1, 3 (V)	-100	-130 to -40
Einzel 2 (V)	7	-20 to +70
Omega Bias (V)	-35	-40 to 0
Omega (+) (V)	5	0 to +30
Omega (-) (V)	-5	-30 to +10
QP Focus (V)	3	0 to +10
Plate Bias (V)	0	0 to +6
QP Bias (V)	0	0 to +6

Reducing Oxide Ions

When changing tuning parameters to achieve greater sensitivity, you can inadvertently increase the level of oxide ions. The ***Sensitivity*** tuning window can be used to check for oxide ions, but ChemStation provides another window with the mass values already set for the standard tuning solution, 10 parts per billion (ppb) of Li, Y, Ce and Tl. This procedure explains how to identify and reduce oxide ions using the ***Oxide Ion*** tuning window.

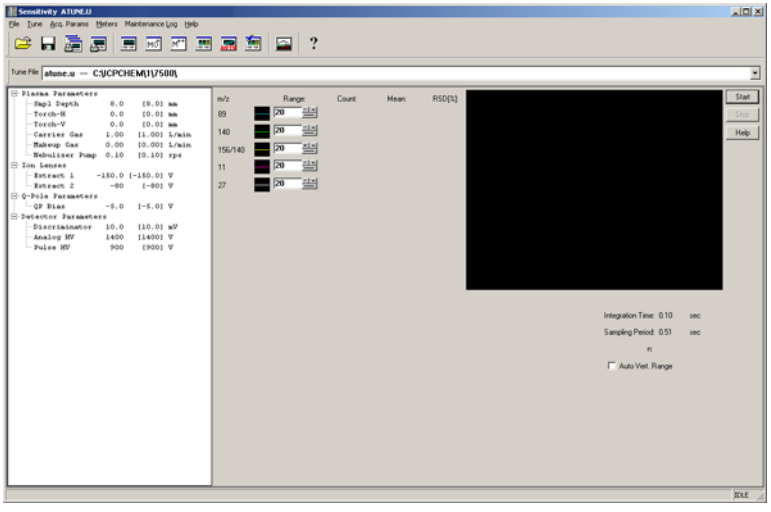
To identify and reduce oxide ions, complete the following steps:

1 **If measuring sensitivity or resolution, click *Stop* in the *Tuning* window.**

The real-time display is stopped.

2 **Select *Tune>>Oxide Ion*.**

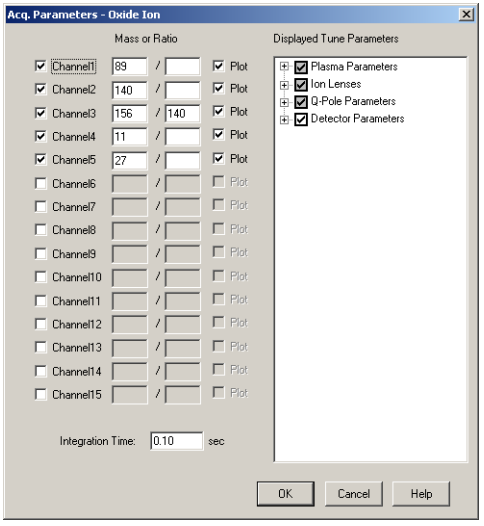
The ***ICP-MS Tuning-Oxide Ion*** window appears showing the real time display for an oxide ion. The window is similar to the ***Sensitivity*** tuning window, but the m/z channels are different.



ICP-MS Tuning-Oxide Ion Window

3 Select *Tune* >> *Acq.* Params.

The ***Acquisition Parameters - Oxide Ion*** dialog box will appear.



Acquisition Parameters - Oxide Ion Dialog Box

4 Select the check boxes of the channels to be displayed in the *ICP-MS Tuning - Oxide Ion* window.

5 When using a tuning solution different from the standard tuning solution, change the mass value in the Mass or Ratio text box.

Enter the mass (m/z) of the element used for tuning and the mass (m+16) of its oxide.

Since Ce has a high oxide bond strength, it will show a high oxide ion generation rate. Therefore, the use of Ce is recommended for tuning applications in which suppression of oxide ion generation is desired. When using Ce, set the mass values to 140 amu and 156 amu so that the strength ratio becomes 156/140 amu.

6 Enter the data collection time (integration time) for each mass in the Integration Time: text box.

Generally, set 0.1 seconds per one mass value.

7 Select the *Plot* check boxes for the channels to be displayed on the graph in the *ICP-MS Tuning - Oxide Ion* window.

8 In the *Displayed Tuning Parameters* tree display, select the check boxes of the tuning parameters to be displayed in the *ICP-MS Tuning - Oxide Ion* window.

Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Clicking on a parameter check box will alternately select or deselect the parameter. A parameter in gray indicates that only some of the parameters contained within are selected.

9 Click *OK*.

Clicking ***OK*** will close the ***Acquisition Parameters - Oxide Ion*** window and displays the entered settings in the ***ICP-MS Tuning - Oxide Ion*** window.

10 Click *Start* to monitor the Agilent 7500 signal and view the numerical values in the real-time display.

The window includes a box for a real-time display of the instrument's signal. The box contains up to 200 data points. The current counts of each mass are given, and the mean, RSD (relative standard deviation) of the signal, and the ratio of oxide are given for the data points shown in the display.

11 If the ratio of the oxide mass to the element mass is equal to or greater than 1.0% (2.0% if you use a concentric nebulizer), we recommend adjustment for the following parameters to reduce the signal of the oxide ion.

RF Power, Smpl Depth, Carrier Gas, Makeup Gas, and PeriPump 1

Click each box and use the arrows to change the value.

To reduce the level of oxide ions, a key factor is plasma temperature. Higher plasma temperature accelerates the decomposition of oxides. Promoting the decomposition of oxide species,

- Increase the Sampling depth
- Decrease the Carrier gas (or Makeup gas) flow
- Increase the RF Power
- Decrease the Sample flow rate

Reducing Doubly Charged Ions

When tuning parameters are changed to achieve greater sensitivity, this can inadvertently increase the level of doubly charged ions. You can use the ***Sensitivity*** tuning window to check for doubly charged ions, but ChemStation provides another window with the mass values already set for the standard tuning solution. This procedure explains how to identify and reduce doubly charged ions using the ***Doubly Charged Ion*** tuning window.

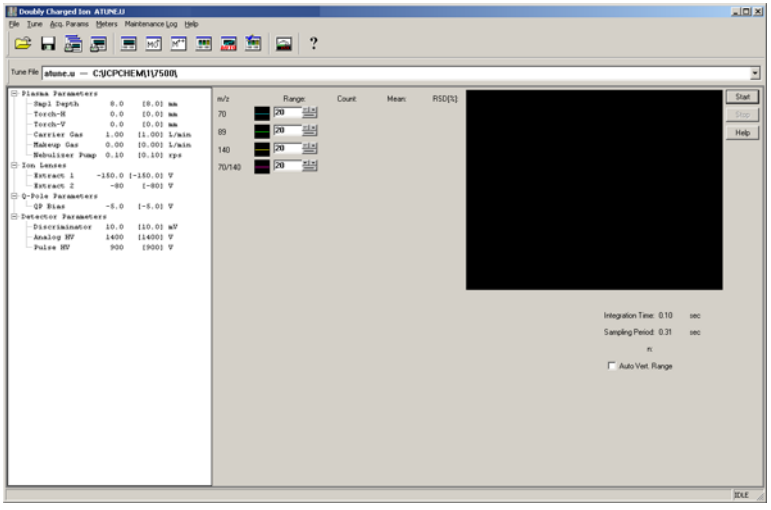
To tune for doubly charged ions, complete the following steps:

1 **If measuring sensitivity or resolution, click *Stop* in the *Tuning* window.**

The real-time display is stopped.

2 **Select *Tune>>Doubly Charged Ion*.**

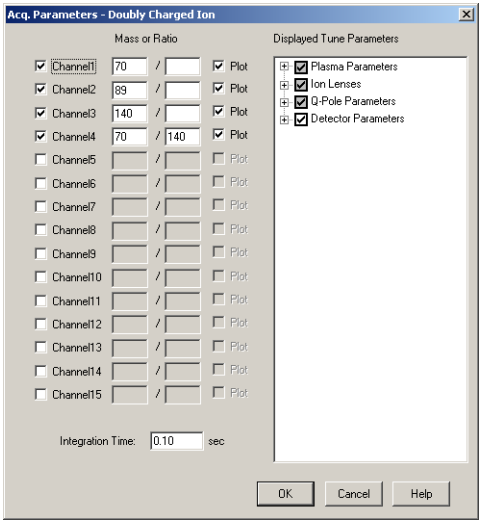
The ***ICP-MS Tuning- Doubly Charged Ion*** window appears showing the real time display for a doubly charged ion. The window is similar to the ***Sensitivity*** tuning window, but the m/z channels are different.



ICP-MS Tuning-Doubly Charged Ion Window

3 Select *Tune >> Acq. Params*.

The ***Acquisition Parameters - Doubly Charged Ion*** dialog box will appear.



Acquisition Parameters - Doubly Charged Ion Dialog Box

4 Select the check boxes of the channels to be displayed in the *ICP-MS Tuning - Doubly Charged Ion* window.

5 When using a tuning solution different from the standard tuning solution, change the mass value in the Mass or Ratio text box.

Enter the mass (m/z) of the element used for tuning and the mass (m/2) of its oxide.

If Ce is not present in the tuning solution, use Ba. Ba also readily generates doubly charged ions and is often used in tuning in which suppression of doubly charged ion generation is desired.

When using Ce, set the mass values to 70 amu and 140 amu so that the strength ratio becomes 70/140 amu.

When using Ba, set the mass values to 69 amu and 138 amu so that the strength ratio becomes 69/138 amu.

6 Enter the data collection time (integration time) for each mass in the Integration Time: text box.

Generally, set 0.1 seconds per one mass value.

7 Select the *Plot* check boxes of the channels to be displayed on the graph in the *ICP-MS Tuning - Doubly Charged Ion* window.

8 In the *Displayed Tuning Parameters* tree display, select the check boxes of the tuning parameters to be displayed in the *ICP-MS Tuning - Doubly Charged Ion* window.

Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Clicking on a parameter check box will alternately select or deselect the parameter. A parameter in gray indicates that only some of the parameters contained within are selected.

9 Click *OK*.

Clicking ***OK*** will close the ***Acquisition Parameters - Doubly Charged Ion*** window and display the entered settings in the ***ICP-MS Tuning - Doubly Charged Ion*** window.

10 Click *Start* to monitor the Agilent 7500 signals and view the numerical values in the real-time display.

The window includes a box for a real-time display of the instrument's signal. The box contains up to 200 data points. The current counts of each mass are given, and the mean, RSD (relative standard deviation) of the signal, and the ratio of doubly charged ion are given for the data points shown in the display.

11 If the ratio of the signal of the doubly charged ion to the element signal is equal to or greater than 2.0% (3.5% in the case of Ba), then adjust the following parameters to reduce the signal of the doubly charged ion.

RF Power, Smpl Depth, Carrier Gas, Makeup Gas, and PeriPump 1

Click each box and use the arrows to change the value.

The ratio of doubly charged ions mainly depends on the tuning conditions of the sample introduction system. In order to reduce the ratio of doubly charged ions,

- Increase the Sampling depth
- Decrease the Carrier gas (or Makeup gas) flow
- Increase the RF Power
- Decrease the Sample flow rate

ShieldTorch System

The ShieldTorch system is very powerful with features such as a Cool plasma for Fe, K, Ca analysis at the ppt level, a High sensitivity mode and a Soft extraction mode.

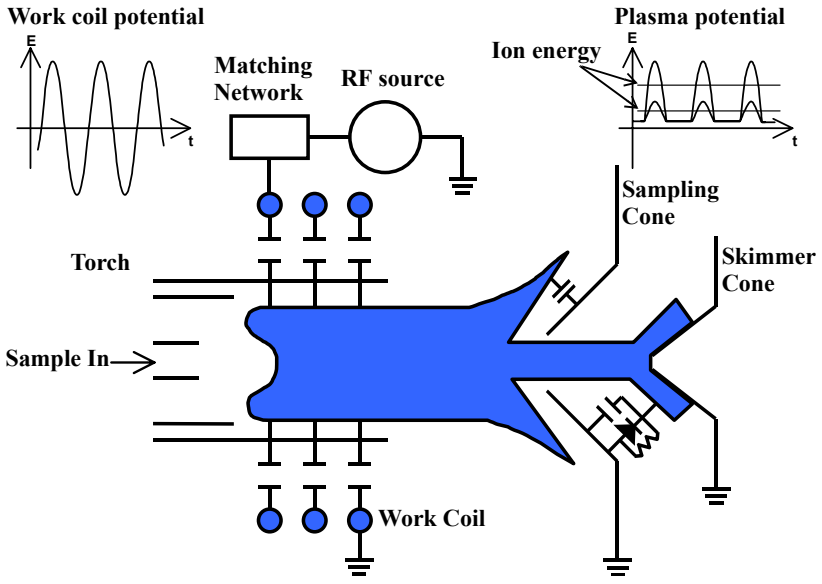
The ShieldTorch is not applicable to all sample types and it must be carefully considered whether it is appropriate for the particular samples and elements that are to be analyzed.

Theory of the ShieldTorch System

The polyatomic ion, such as ArO, is formed behind the interface and plasma.

At behind the interface

The potential in the plasma is grounded through the interface and the secondary discharge occurs at this time. The secondary discharge ionizes molecules such as ArO, ArH, and ArAr, and atoms behind the sampling cone. When the ShieldTorch system is used, the shield plate inserted between the torch and the RF coil eliminates the capacitive coupling between the plasma and the RF coil, so that the potential in the plasma is lower than that without the ShieldTorch system. As a result, there is no longer a secondary discharge and polyatomic ions such as ArO, ArH and ArAr, are not ionized behind the sampling cone.



Electrical Model of the Plasma and the Interface Region

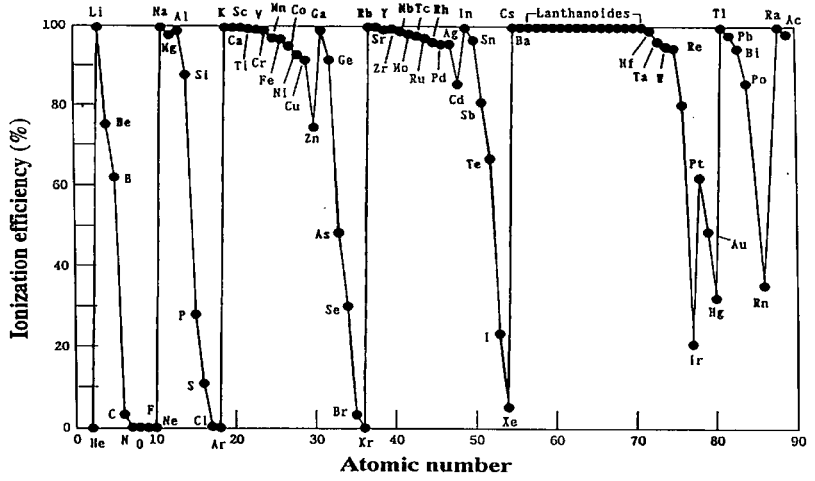
In the plasma

The ionization efficiency of elements in the Ar plasma can be calculated using the Saha-Egggert Equation given below:

$$\frac{a}{1-a} = \frac{(2\pi mkT)^{\frac{3}{2}}}{Neh^3} \times \frac{2Z^+}{Z} \times e^{\frac{-V}{kT}}$$

a:	**Ionization efficiency**
Ne:	**Electron density**
m:	**Mass of an ion**
V:	**Ionization potential of an element**
k:	**Boltzman constant**
T:	**Electron temperature**
h:	**Plank constant**
Z:	**Partition function**

If the electron density is 1.47×10^{14} cm^{-3} and the electron temperature is 6,680K, the ionization efficiency of elements will be as shown in the next figure. Elements that have lower ionization potentials such as Li, Na, and Lanthanoids can be ionized with nearly 100 % efficiency in the plasma. On the other hand, the elements that have relatively higher ionization potentials such as As, Se, and Hg are ionized about 30 to 50 %. Ar can be ionized only about 0.009%. So, it is assumed that polyatomic ions exist in the Ar plasma under normal plasma conditions, and they are detected even when the ShieldTorch system is used.

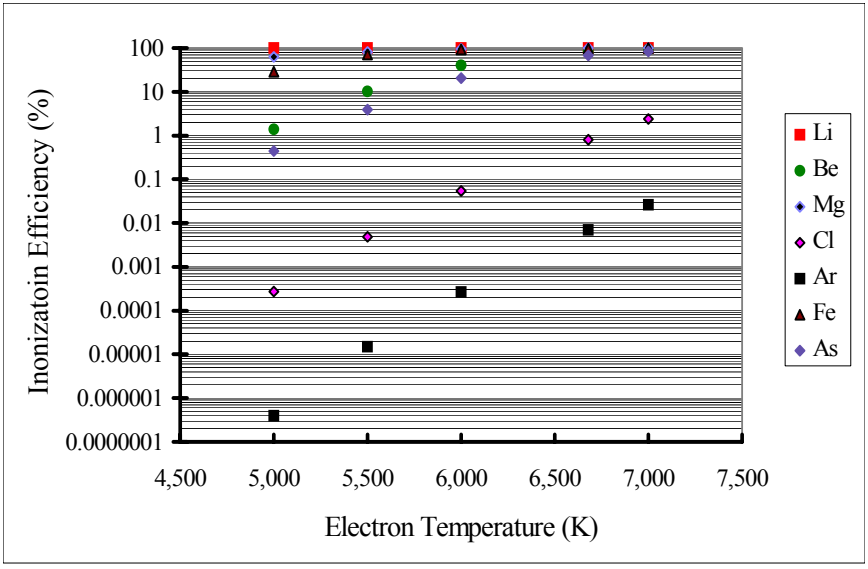


Ionization Efficiency of Elements in an Ar Plasma

However, the polyatomic ions are observed under normal plasma conditions with the ShieldTorch system: 1200W RF power and about 1.2 l/min carrier and blend gas flow. To reduce the level of polyatomic ions the plasma temperature must be reduced.

When reducing the RF power cools the plasma temperature or increasing the carrier gas flow, the ionization efficiency of elements changes as shown in the next figure. Upon decreasing the electron temperature (by decreasing the plasma temperature), lower ionization potential elements such as Li, Mg and Fe are still ionized at nearly 100%. On the other hand, the ionization efficiencies of elements with higher ionization potentials such as Cl and Ar are drastically reduced. So, the ionization

potentials of polyatomic ions are relatively high, probably higher than 10 eV and similar to that of Cl.



Ionization Efficiency of Elements as a Function of the Electron Temperature

In conclusion, by using the combination of the ShieldTorch system and cool plasma conditions, polyatomic ions such as ArH, ArC, and ArO can be drastically reduced while analytes such as Li, Na, Mg, K, Ca, Cr, and Fe are still ionized efficiently.

Feature of the ShieldTorch system

There is a clear difference between the standard system (without the ShieldTorch) and the ShieldTorch system. Each has advantages and disadvantages, therefore it must be considered which system is better for the samples and elements to be analyzed.

Effect of Carrier Gas Flow Rate

Standard system

- The optimal carrier gas flow rate, which gives the maximum sensitivity for analytes depends on the mass; the optimum flow rate for heavier elements is lower than that for lighter elements.
- The signals of polyatomic ions such as ArH, ArO, ArAr, and CO_2 increase with carrier gas flow rates higher than the optimum flow rate, and then gradually decrease.
- The behavior of Ba^{2+}, BaO, and BaOH is similar to the polyatomic ions; increasing the carrier gas flow increases the ratio of Ba^{2+}/Ba, BaO/Ba, and BaOH/Ba.

Increasing the carrier gas flow reduces the residence time of analytes in the plasma, which leads to insufficient decomposition. The elements that form a strong bond with oxygen, such as Ba and REEs (Rare Earth Elements), form oxides, some of which can be ionized as oxide ions.

The ShieldTorch system (Cool plasma)

The optimal carrier gas flow rate, which gives the maximum sensitivity for analytes depends on ionization potential, boiling point and bond strength. The optimal carrier gas flow rates are different for Be and Li because of ionization potential, or Pb and W because of boiling point and the bond strength with oxygen, even though the mass numbers are close. In contrast, Co, In, and Pb show similar behavior, despite having very different masses.

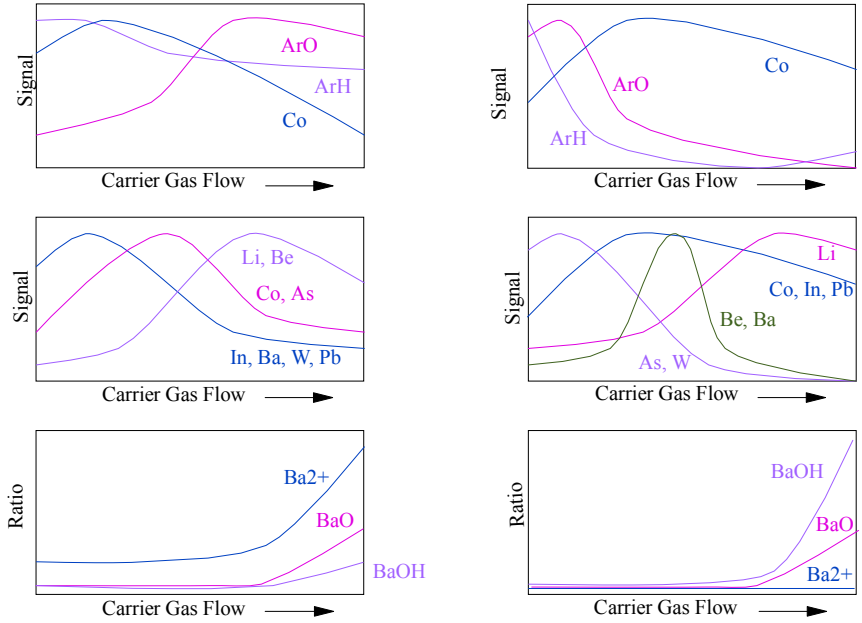
- The signal of ArO drastically decreases as a function of higher carrier gas flow. The signal of ArH also drastically reduces at first, and then gradually increases. The behavior of Ar is almost the same as that of ArH.
- Levels of BaO and BaOH increase (especially, BaOH) at higher carrier gas flow

rates, while Ba^{2+} is very low.

As an example, the relationship between carrier gas flow rate and signal using the elements in the table is shown in the next figure.

Chemical / Physical Characterisisties of Selected Elements

Element	Main isotope (AMU)	Ionization potential (eV)	Boiling point (°C)	Oxygen bond strength (kcal / mol)
Li	7	5.39	1327	81.4
Be	9	9.32	2399	105.4
Co	59	7.86	2747	88
As	75	9.81	-	115
In	115	5.785	2000	86
Ba	138	5.21	1639	130.4
W	184	7.98	5927	156
Pb	208	7.415	1750	90.3
La	139	5.61	3469	?



Normal system (without the ShieldTorch) **The ShieldTorch system**

Relationship between Carrier Gas Flow Rate and Signal

Each of the features for the three plasma conditions

There are 3 combinations to be considered: the standard system with normal plasma conditions, the ShieldTorch system with normal plasma conditions, and the ShieldTorch system with cool plasma conditions. Some features of each follow:

Standard system (normal plasma)

Advantage

- Relatively resistant to matrix effects.

Disadvantage

- Difficult to analyze those elements which suffer interferences by polyatomic ions containing Ar and C, e.g. K, Ca, and Fe.

The ShieldTorch system (High Sensitivity mode)

With the ShieldTorch system, the pattern for extracting ions from plasma at the interface varies. The Plasma gets into the backside of the Skimmer and the interaction between the electric field from the extraction lens and the plasma decides the extraction condition. At this time, the condition for the space charge effect and ion focus will be quite different from those with no shield plate.

Under this condition, two ion lens modes are available to select. The Extraction mode realizes the ion extraction under far advantageous conditions and higher sensitivity. The Soft extraction mode reduces the background.

Advantages

- It gives a higher sensitivity than when it is under the Standard torch and Normal plasma condition. The sensitivity can be especially high in the Extraction mode.
- In the Soft extraction mode, background caused by the interface will be reduced. Especially, it is effective in decreasing the alkaline background with elements, such as Li and Na, which are increased by the memory at the interface. Thus, the detection limit of the element will be expanded.
- In the Soft extraction mode, low mass sensitivity is lowered in use and this will reduce the random background. Then, the BEC(Background Equivalent Concentration) will be substantially improved in mid mass or high mass.

Disadvantage

- Matrix effects are more severe when the shield plate is fitted.

The ShieldTorch system (Cool plasma)

Advantage

- Analysis of elements which suffer interference from polyatomic ions due to Ar and C.

  Ar --- Ca (Ar), K (ArH), Cr (ArC), and Fe (ArO)

  C --- Mg (C_2), Cr (ArC)

- Analysis of some lighter elements, such as Li, Na, Mg, and Al. Under cool plasma conditions, the sensitivity of lighter masses increases and the background due to the sampling cone is minimized due to the elimination of secondary discharge.

Disadvantage

- Matrix effects are more severe.
- Difficult to analyze the elements, which have higher boiling points, higher ionization potentials or higher bond energies with oxygen. The cool plasma conditions prevent decomposition of sample in the plasma. Some elements, which easily form oxides are not decomposed well. Bond energy is a good indicator of whether oxide formation will occur, if the bond energy with oxygen is high and the ionization potential of the oxide is low, you will see the oxide ions, e.g. bond energies of REEs with oxygen are higher than 150 kcal/mol. Si-O bond energy is 193 kcal/mol, so it is also difficult to decompose. Bond energy for Ba-O is 130 kcal/mol and the BaO and BaOH ions are commonly found even at lower concentrations. For higher boiling point elements such as W, Ta and Mo, ionization might be difficult under cool plasma conditions. The bond energy of these elements with oxygen is also quite high.
- Generation of polyatomic ions. Although the polyatomic ions, which have higher ionization potentials, can be decreased by the ShieldTorch system with cool plasma conditions, the polyatomic ions, which have lower ionization potentials, are drastically increased. In particular, the polyatomic ions due to Ca are generated more than without the ShieldTorch, and these interfere with many isotopes for transition metals such as Fe and Ni. Therefore, **the ShieldTorch should not be used for environmental samples**, which normally contain a lot of Ca.

Notes for the use of the ShieldTorch system

Plasma ignition

- Igniting the plasma is a little more difficult compared to that of the standard torch because the Shield plate is between the torch and the coil.
- it is recommended to tune the instrument 30 minutes after igniting the plasma.
- When adjusting the EM, the Discriminator should be done under normal plasma conditions.

NOTE When analyzing with the Normal Torch after analyzing with the ShieldTorch for a long time, sometimes sensitivity becomes low. Cleaning of the ion lenses can rectify this.

Tuning the ShieldTorch (High Sensitivity mode)

This mode gives the highest sensitivity at the normal plasma condition.

NOTE The Agilent 7500s model (discontinued model) is constructed prestigiously with two rotary pumps. The two rotary pumps give the best performance with the highest sensitivity. To achieve maximum performance of the two rotary pumps, make sure the valve on the top of the second rotary pump is open. (Rotate it counterclockwise.)

NOTE When the cool plasma condition is activated, only one rotary pump is used. This avoids generating cluster ions. To do this, close the valve on the top of the second rotary pump. (Rotate it clockwise.) The sensitivity will be lower than when using two rotary pumps, but the operating condition characteristics are the same. Also, the adjustments are the same.

Tuning solution

Mg, Y, Ce, and Tl, solution at 1 ppb

NOTE For the high sensitivity mode, Li is not used because it is difficult to reduce the background. Li remains on the interface as memory.

Tuning procedure

To tune the sensitivity, complete the following steps:

1 **Install the Shield plate**

2 **Select *Tune>>Acq. Params* on the Tuning window.**

The Acquisition Parameters - Sensitivity dialog box appears. Change masses to **24, 89** and **205** and click ***OK***.

3 **Aspirate a tuning solution (1 ppb of Mg, Y and Tl).**

4 **Set the tuning parameters as typical values.**

Set the parameters as follows:

Typical values for the ShieldTorch (High Sensitivity mode)

Tuning parameters	Typical	Range	Note
RF Power (W)	1500	1200 ~ 1600	
Smpl Depth (mm)	6	4 ~ 8	Set lower than the normal value.
Carrier Gas (L/min)	1.2	0.8 ~ 1.3	
Makeup Gas (L/min)	0	0 ~ 0.4	Set as to make the total flow rate, including the carrier gas, to be about 1.2 L/min.
PeriPump (rps)	0.2	0.06 ~ 0.20	Set higher to get a high sensitivity, but making it too high doesn't always increase further sensitivity.
Extract 1 (V)	-180	-200 ~ -100	
Extract 2 (V)	-35	-150 ~ -10	
Einzel 1,3 (V)	-100	-130 ~ -40	Even if the value of the Einzel lens is decreased, the background may hardly be increased and only the sensitivity may be increased.
Einzel 2 (V)	10	0 ~ 15	
Omega Bias (V)	-30	-100 ~ -25	If this value is lowered, sensitivity may be increased. Even if the value of the Omega lens is decreased, the background may hardly be increased and only the sensitivity may be increased.
Omega (+) (V)	8	0 ~ 30	
Omega (-) (V)	5	-30 ~ 0	
QP Focus (V)	0	0 ~ 10	Because the ion energy is low, it is necessary to make this value smaller than that of the Standard torch. Be sure to use with 0 or (+). In the case of (-), the background will sharply rise.
Plate Bias (V)	-10	-10 ~ -5	Because the ion energy is low, it is necessary to add negative voltage.
QP Bias (V)	-5	-10 ~ -5	Because the ion energy is low, it is necessary to add negative voltage.

5 Adjust Torch-H and Torch-V position.

6 Adjust the RF Power.

7 Adjust the Smpl Depth (Sampling depth).

8 Adjust the Carrier Gas and Makeup Gas flow rate.

9 Adjust the PeriPump (sample flow rate).

10 Adjust lens parameters.

1) Adjust the extraction lenses.

Adjust the Extract 1 in 10V increments until reaching a plateau sensitivity, and then adjust the Extract 2 in 1V increments until reaching a plateau sensitivity. Repeat this until no more increase in sensitivity is achieved.

2) Adjust the Omega(+). Only small increments will be necessary.

3) Adjust the Omega(-). Only small increments will be necessary.

4) Adjust the QP Focus. Only small increments will be necessary.

5) Adjust the Einzel 2. Only small increments will be necessary.

Adjust the tuning parameter until the signals are stable and the sensitivity is high. The Following are examples of sensitivity:

Example of Sensitivity (High sensitivity tuning)

Element	Counts / 1 ppb (Integ. : 0.1sec)
Mg (24 amu)	> 13,000
Y (89 amu)	> 45,000
Tl (205 amu)	> 18,000

Tuning the ShieldTorch (Soft Extraction Mode)

The Soft Extraction mode gives the lowest background.

NOTE The Agilent 7500 model is constructed prestigiously with two rotary pumps. The two rotary pumps give the best performance with the highest sensitivity. To achieve maximum performance of the two rotary pumps, make sure the valve on the top of the second rotary pump is open. (Rotate it counterclockwise.)

NOTE When the cool plasma condition is activated, only one rotary pump is used. This avoids generating cluster ions. To do this, close the valve on the top of the second rotary pump. (Rotate it clockwise.) The sensitivity will be lower than when using two rotary pumps, but the operating condition characteristics are the same. Also, the adjustments are the same.

Tuning solution

Li, Y, and Tl solution at 1 ppb

Tuning procedure

To use the Soft extraction mode, complete the following steps:

1 **Install the shield plate.**

2 **Introduce the tuning solution.**

3 **Set the tuning parameters to the typical values shown in the following table.**

Typical values for the ShieldTorch (Soft extraction mode)

Tuning parameter	Typical	Range	Note
RF Power (W)	1500	1200 ~ 1600	In the Soft extraction mode, a higher plasma temperature setting is necessary. The Carrier gas flow rate and Sampling depth also affect the plasma temperature. So it is necessary to adjust by combining these three parameters.
Smpl Depth (mm)	7	4 ~ 8	If the Sampling depth is too great, it is the same effect as increasing the RF power. For the correct effect, adjust the Sampling depth and the RF power together. However, making the sampling depth too small, may worsen the oxide ion ratio and making the sampling depth too great may cause a reduction in sensitivity.
Carrier Gas (L/min)	1.15	0.8 ~ 1.3	When the Carrier gas is increased, it has the same effect of decreasing the RF power. Adjust the Carrier gas and the RF power together.
Makeup Gas (L/min)	0	0 ~ 0.4	
PeriPump (rps)	0.2	0.06 ~ 0.20	Set it higher to get a high sensitivity, but setting it too high doesn't always increase sensitivity.
Extract 1 (V)	4	3 ~ 5	
Extract 2 (V)	-50	-60 ~ -20	
Einzel 1,3 (V)	-50	-100 ~ -40	Even if the value of the Einzel lens is decreased, the background may hardly be increased compared with the standard torch. Only sensitivity may be increased.
Einzel 2 (V)	10	0 ~ 15	
Omega Bias (V)	-35	-100 ~ -25	Decreasing this value may increase sensitivity. Even if the value of the Omega lens is decreased, the background may hardly be increased compared with the standard torch. Only sensitivity may be increased.
Omega (+) (V)	5	0 ~ 30	
Omega (-) (V)	0	-30 ~ 0	
QP Focus (V)	3	0 ~ 10	As ion energy is low, it is necessary to make the QP focus lower than the standard torch. Make sure to use Positive voltage. Using negative voltage will sharply increase the background.
Plate Bias (V)	0	-10 ~ 0	
QP Bias (V)	-10	-10 ~ -5	As ion energy is low, it is necessary to add negative voltage.

4 Adjust the torch position (Torch-H and Torch-V).

5 Adjust the flow rates of the Carrier gas and the Makeup gas.

6 Adjust the Peristaltic pump speed (Sample flow rate).

7 Adjust the RF power.

8 Adjust the Sampling depth.

9 Adjust the Ion lens parameter.

1) Adjust the Extraction lens.

Depending on the mass number of Li, Y, and Tl, the value for the highest sensitivity differs. Therefore adjust the lens according to your purpose.

To reduce random background, increase the voltage of the Extraction lens 1 (about +4.5V). Simultaneously adjust the Omega(+) voltage and Omega (-) voltage to decrease Li and increase Y and Tl. Then make the sensitivity of Li several Mcps/ppm. (If it's set integration time of 1ppb and 0.1seconds, it is equivalent to several hundred count.). At this time, do not adjust the sensitivity of Y and Tl too low. The guideline is more than 100Mcps/ppm. If checking the off mass background on the tuning report, it is found that the background is restricted lower than usual.

2) Adjust other lens parameters.

How to adjust the other lens parameters is almost the same as with the High Sensitivity mode under the ShieldTorch.

Ensure not to set a too negative voltage when tuning for a reduced whole background. As shown in the table, set -50 V instead of -100V to the Einzel 1,3, and don't make the Omega bias lower than -35V. As for the Plate bias, it is necessary not to make it too negative than necessary.

Adjust the Tuning parameter until signals are stable and high sensitivity is achieved. The Following are examples of sensitivity:

Element	Count/ 1 ppb(Integ.: 0.1 sec)	Back ground (cps, off mass)*
Li (7 amu)	~600	~1
Y (89 amu)	>30,000	~1
Tl (205 amu)	>20,000	~1

*Shown on the tuning report.

NOTE

If the background is compromised, it is easy to increase low mass sensitivity to more than100 MCPS/ppm. Adjust the Extraction lens1, Extraction lens 2, and Omega(+) and Omega(-), depending on your purpose.

Tuning the ShieldTorch (Cool plasma)

Ca (40 AMU) can be analyzed with the cool plasma conditions not the normal plasma conditions. To do that, delete the 40 AMU in the Prohibited Masses of the Configuration task.

CAUTION 

When you return to the normal plasma conditions from the cool plasma condition, you must reset the 40 AMU in the Prohibited Masses because detector gets damage.

Tuning solution

Li, Y, Ce, and Tl solution at 1 ppb: for tuning the mass resolution/axis

Co solution at 1 or 10 ppb: for tuning the sensitivity

NOTE In the cool plasma condition, Y cannot be used because it generates the oxide ion (YO), which decreases the sensitivity of 89amu(Y).

Tuning procedure

To use the ShieldTorch in the cool plasma condition, perform the following steps:

1. **Install the Shield plate.**
2. **Aspirate a tuning solution containing 10 ppb (or 1 ppb) Co and select.**
3. **Set the tuning parameters as the typical values shown in the table.**

Typical values for the ShieldTorch (Cool plasma)

Tuning parameter	Typical	Range	Note
RF Power (W)	900	600 ~ 1000	Adjust the RF power lower than the standard torch.While monitoring the RF reflection power from the Tuning window, make the RF power lower from the normal plasma (1200-1400 W) to 900 W. If the RF reflection power increases, adjust the RF matching to prevent the RF reflection power from increasing. In the case of 600W, plasma may turn out. If this happens, set it higher than 800W and turn on the plasma again.
Smpl Depth (mm)	13	10 ~ 20	Keep it apart from the standard torch position.
Torch-H (mm) Torch-V (mm)	-	-	Changing the Sampling depth may slip the torch axis, it is necessary to adjust frequently.
Carrier Gas (L/min)	1.0	0.8 ~ 1.3	The supply pressure of the Ar gas restricts the carrier gas flow rate. Make up the loss by introducing the Makeup gas.
Makeup Gas (L/min)	0.6	0 ~ 1.0	For the standard torch (injector internal diameter is 2.5 mm), the standard value is about 1.6 L/min including the carrier gas. In the case of using a smaller injector torch, the best flow rate will be lower than 1.6 L/min. (about 1.2L/min if the injector's internal diameter is about 1.5mm). If the Makeup gas flow rate is increased, the signals of ArO (56amu) will be decreased.
PeriPump 1 (rps)	0.1	0.10 ~ 0.20	To lower the plasma temperature, increase the number of pump rotation.
Extract 1 (V)	-50	-100 ~ -20	
Extract 2 (V)	-10	-100 ~ -10	
Einzel 1,3 (V)	-100	-200 ~ -100	Even if the value of the Einzel lens is decreased, the background may hardly be increased and only sensitivity may be increased, compared to that of the standard torch.
Einzel 2 (V)	0	0 ~ 15	
Omega Bias (V)	-30	-100 ~ -25	If this value is lowered, sensitivity may be increased. Even if the value of the Omega lens is decreased, the background may hardly be increased and only sensitivity may be increased, compared to that of the standard torch.
Omega (+) (V)	0	0 ~ 30	
Omega (-) (V)	0	-30 ~ 0	

Tuning parameter	Typical	Range	Note
QP Focus (V)	0	0 ~ 5	Because the ion energy is low, it is necessary to make this value smaller than the standard torch. Make sure to use with 0 or (+). In the case of (-), the background will sharply rise.
Plate Bias (V)	-10	-10 ~ -5	Because the ion energy is low, it is necessary to add negative voltage.
QP Bias (V)	-10	-10 ~ -5	Because the ion energy is low, it is necessary to add negative voltage.

4 Monitor 39(ArH), 56(ArO), and 59(Co) AMU.

5 Adjust the torch position (Torch-H, Torch-V).

Adjust the torch position to which the mass 56 signal is low and the mass 59 signal is high.

6 Adjust the Carrier gas and the Makeup gas.

Adjust the Carrier gas and the Makeup gas to which the mass 59 signal is high.

7 Adjust the sampling depth (Smpl Depth).

Adjust the sampling depth to which the mass 39 and 56 signals are low.

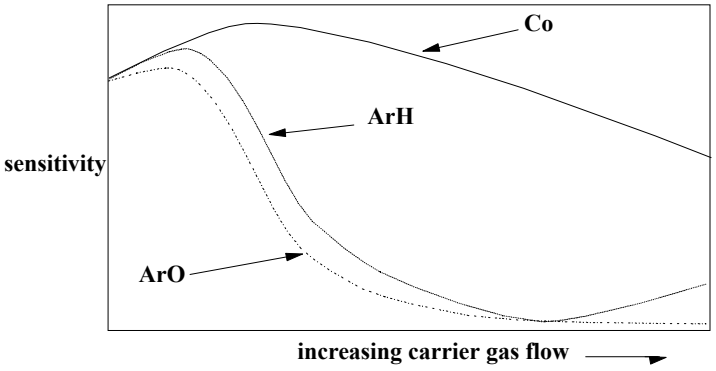
8 Adjust the lens parameters.

Adjust to the mass 59 higher and the mass 39 and 56 lower. If the mass 39 and 56 signal behave the same as the mass 59 signal, there may be Fe and K contamination in the Co tuning solution. In such case, monitor the mass 76 (ArAr) instead of the mass 39.

9 Adjust the plasma condition.

Adjust to the parameters related to the plasma condition (RF power, Carrier gas, Makeup gas, Sampling depth). Adjust to the mass 59 higher and the mass 39 and 56 lower.

The behavior of 39 m/z (ArH) is different from 56 m/z (ArO). Decreasing the plasma temperature decreases ArO sensitivity asymptotically. However, ArH sensitivity reaches a minimum and then increases at very low plasma temperatures. Polyatomic ions at mass 40 (Ar) and 55 (ArOH or KO) behave the same as ArH. When K, Ca, Mn and Fe are analyzed, check that you are getting low background values for both masses 39 and 56 simultaneously.



Behavior of Co, ArH and ArO

10 Check the background level

If the tuning solution contains impurities and it is difficult to check the background of 39amu(ArH) or 56amu(ArO). Use fresh ultra pure water and check if the signal is decreased. The following are standard levels:

39amu(ArH)--less than 1/10~1/50 of 59 amu signal (Co 10 ppb).

56amu(ArO)--less than 1/100~1/500 of 59amu signal (Co 10 ppb).

NOTE Easily, there may Fe and K contamination from the environment. Even in the clean room, there is a possibility of contamination if samples are exposed to wind; therefore, use fresh ultra pure water.

Example

Element	Count/ 1 ppb (Integ.: 0.1 sec)
Co (59 amu)	> 1000 ~ 3000
ArO (56 amu)	< Signal (59) x $\frac{1}{100}$

NOTE Compared to the normal plasma, the cool plasma reacts more sensitively to the matrix effect. Therefore, it is recommended to use the matrix matched tuning solution when analyzing high matrix samples with the ShieldTorch system.

Tuning for Sensitivity (Agilent 7500ce)

Adjust sensitivity of elements to be measured obtain high stable signals over the whole mass range. The standard tuning solution is a 1% nitric acid solution containing 1 ng/mL (ppb) of each of the following: Li, Co, Y, Ce, and Tl.

When using the Agilent 7500a refer to "Tuning for Sensitivity (Agilent 7500a)" on page 4-4.

When using the Agilent 7500cs, refer to "Tuning for Sensitivity (Agilent 7500cs)" on page 4-62.

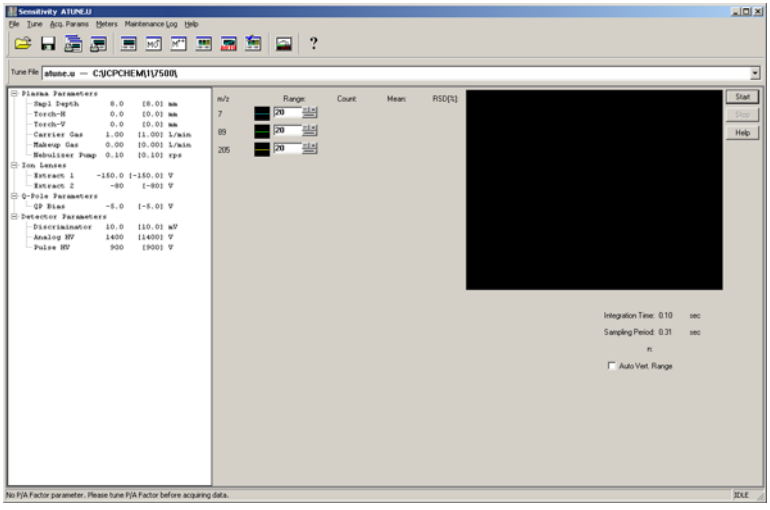
CAUTION

Whenever the Agilent 7500cs is used, the ShieldTorch must be installed in it. For installation of the ShieldTorch, refer to the *Agilent 7500 ICP-MS Hardware Manual*.

Adjust sensitivity using the following steps:

1 **Select *Instrument>>Tune* in the ICP-MS Top Window.**

The **ICP-MS Tuning-Sensitivity** Window will appear.



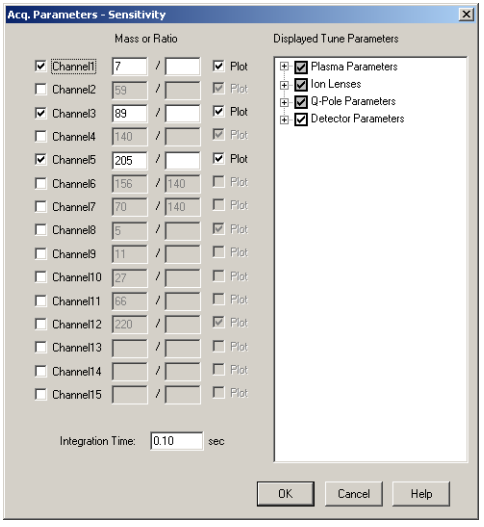
ICP-MS Tuning-Sensitivity Window

2 Select *Tune>>Acq. Params.*

The ***Acquisition Parameters-Sensitivity*** Dialog Box will appear.

Use this dialog box to set or change the masses you want to view, the integration time, or the ratio of any two channels.

You can also display the ratio of signal strengths of any two selected masses. This dialog box also allows you to select the channels to be displayed on the ***ICP-MS Tuning - Sensitivity*** window, the tuning parameters to be indicated in the tree display, and the channels to be plotted on the graph.



Acquisition Parameters-Sensitivity Dialog Box

NOTE To use tuning parameter settings from a previous tuning session as a starting point for this session, refer to "Creating and Using a Tune File" on page 4-99.

3 Select the check boxes of the channels to be displayed on the *ICP-MS Tuning - Sensitivity* window.

4 Enter the values of the masses in the left-side boxes under "Mass or Ratio."

5 To set a strength ratio, type the mass values in the right-side boxes under "Mass or Ratio."

6 Enter the data collection time (integration time) for each mass in the Integration Time: text box

Generally, set 0.1 seconds per one mass value.

7 **Select the *Plot* check boxes of the channels to be displayed on the graph in the *ICP-MS Tuning - Sensitivity* window.**

8 **In the Displayed Tuning Parameters tree display, select the check boxes of the tuning parameters to be displayed in the *ICP-MS Tuning - Sensitivity* window.**

Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Clicking on a parameter check box will alternately select or deselect the parameter.

A parameter in gray indicates that only some of the parameters contained within are selected.

9 **Click *OK*.**

Clicking ***OK*** closes the ***Acquisition Parameters - Sensitivity*** window and displays the entered settings in the ***ICP-MS Tuning - Sensitivity*** window.

10 **Put the sample uptake tube into the tuning solution.**

Wait for the uptake of the sample to the nebulizer.

11 **Click *Start* to monitor the Agilent 7500's signal and view the numerical values in the real-time display.**

As the signal appears, ChemStation inserts numerical values for the count, mean, RSD, and ratio.

The window includes a box for a real-time display of the instrument's signal. The box contains up to 200 data points. The current counts of each mass are given, the mean and RSD (relative standard deviation) of the signal are given for the data points shown in the display.

NOTE When you check **Auto Vert. Range** check box, the vertical range of the real-time display is automatically adjusted.

NOTE You can print a copy of the real-time display at any time during the tuning procedure. See "Generating a Tune Report" on page 4-97.

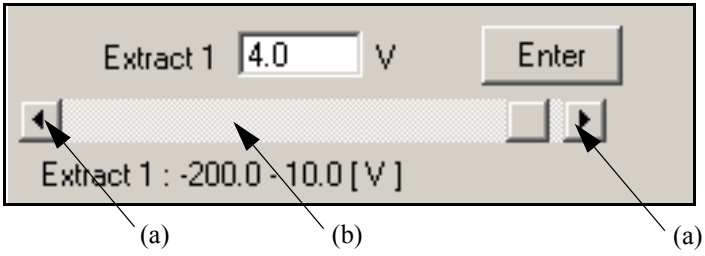
12 **Adjust the tuning parameters if necessary.**

Tuning parameters are shown in the tree display on the left side of the window. Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Indicated on the right side of each parameter in [ ] are the adjustment value and the pre-adjustment numerical value (value stored in the tune file).

Clicking on a parameter will cause a text box or a scroll bar to appear at the lower center section of the window. Use one of the following methods to set a parameter value.

- Enter a new parameter value in the text box (next to the parameter you want to change or above the scroll bar).
- Click the right or left arrow at the end of the scroll bar (a), which changes the parameter value by small increments.
- Click the scroll bar (b), which changes the parameter value by larger increments.
- Click ***Apply*** to set the adjusted parameter value.
  Clicking ***Cancel*** will cause the pre-adjustment parameter value (value stored in the tune file) to be set.



Adjust the tuning parameters until the signal appears constant and the numerical values meet performance requirements. The performance specification depends on the application.

For the workings of each tuning parameter, refer to "Each Parameter's Function" on page 4-66.

CAUTION

Use care when adjusting values by clicking the scroll bar or entering a new value in the box above the scroll bar. Changing parameter values too quickly can cause extreme changes in the Agilent 7500 and can alter its performance over time. In most cases, you should adjust the tuning parameters by clicking the arrows at the end of the scroll bar.

NOTE

To display all the tuning parameters, check **Advanced Setting** to ON.

13 Click *Stop* to stop the display.

ChemStation freezes the real-time display until you click **Start**. When you click **Start**, the display clears and starts again.

Each Parameter's Function

The working of each parameter used for sensitivity adjustment is described below.

NOTE The Agilent 7500ce has three modes: Standard Mode, which does not use a reaction gas, Reaction Mode, which uses hydrogen (H_2) gas or helium (He) gas as a reaction gas. For information on sensitivity adjustment in each mode, refer to "Information on Sensitivity Adjustment" on page 4-46.

Plasma Condition

- RF Power
  It controls the RF power supplied to the load coil. A higher RF power increases the sensitivity and reduces oxide and doubly charged ions. However, the sensitivity of lighter masses may be decreased when the RF power is too high.
- RF Matching
  Adjusts reflected RF returned from the load coil.
  To reduce the reflected power, use of the automatic adjustment function is recommended. To use the automatic adjustment function, select **Tune>>RF Matching** on the Tuning window, and then follow the displayed dialog box.

NOTE If you adjust the RF Matching manually, use only the scroll arrows to change this value. If you click on the scroll bar itself, the instrument will turn off the plasma. Adjust until the RF reflected power meter reads as low as possible. Standard value for RF matching is approximately 1.6 to 2.0 V.

- Smpl Depth
  It controls the distance between the edge of the load coil and the tip of the sampling cone. A shorter sampling depth increases sensitivity, but the oxide level increases as well.
- Torch-H, Torch-V
  It adjusts the torch position. The Torch-H and Torch-V parameters measure the horizontal and vertical movement of the torch relative to the interface. Adjust to get the highest sensitivity for all masses. When the sampling depth is changed, adjustment of the Torch-H/V will be required. If these parameters are changed using the software scroll bar, the plasma may turn off. Use only the scroll arrows.
- Carrier Gas
  It controls the nebulization efficiency of a sample and the sample uptake rate when self-aspiration is applied. In general, a gas flow of 0.9 L/min when using the Micro Mist Nebulizer.

- Makeup Gas
  Makes up Ar gas into the spray chamber and is mixed with the carrier gas. When the Ar gas supply pressure is not high enough to get a sufficient carrier gas flow, the makeup gas is used.
- Optional Gas (option)
  When the optional gas line is added, it is possible to introduce a gas other than argon. (e.g. introduction of oxygen etc. when measuring organic solvent)
- PeriPump 1
  Controls a sample uptake rate and the drain from the spray chamber. It affects sensitivity, stability of signal, and oxide and doubly charged formation. Higher speed increases sensitivity, but too high speed, decreases sensitivity. Higher speed increases oxide and doubly charged ions.

CAUTION 

Even when self-aspiration is used for sampling, the PeriPump 1 must be operated to drain the sample from the spray chamber, if this is not optimized the spray chamber will fill with sample solution.

- PeriPump 2 (option)
  It can be used when a second (optional) peristaltic pump is installed on the ICP-MS. It is possible to shorten the rinse time or the sample uptake time.
- S/C Temp
  Controls the temperature of the spray chamber. Lowering the spray chamber temperature causes the sample vapor temperature to lower and removes more water, thus, reducing the oxide levels. It should be set at 2 ºC for aqueous sample to avoid ice formation. When aspirating organic solvents run the chamber at -5 ºC.

Ion Lens

- Extract 1
  It extracts ions from the plasma to accelerate them toward the Einzel lens. Adjust to get enough sensitivity for all masses. This is lens that needs the most frequently adjusted everyday.

  In order to get maximum sensitivity, adjust between 4 V and 8 V. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass number gets maximum sensitivity at high voltage. Therefore, select the appropriate voltage. Even with the voltage around -100V, high sensitivity can be obtained. However, the background of polyatomic ions may be high in reaction mode (H_2 mode or He mode)

- Extract 2
  It focuses the ion beam from the extraction lens. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass gets maximum sensitivity at high voltage. Therefore, set a value to get optimum sensitivity foe the whole mass range.
- Omega Lens-ce
  Separating the ions from photon and introduce the ions to the Reaction Cell. Adjusts to get good sensitivity for all masses.
- Omega Bias-ce
  Provides the same potential at the entrance and exit of the Omega lens block. It must be used at voltages more positive than -40V. The more negative voltage, the higher the sensitivity, but the background might increase.Cell Focus
  Locates on the first lens of the Reaction Cell. The voltage is set to 0V at all times. This lens is not displayed as a tuning parameter.
- Cell Entrance
  Adjust to get enough sensitivity for all masses. It is necessary to set a lower voltage than the OctP bias. If the voltage is set higher than the OctP bias, polyatomic ions may increase on some specific masses.
- QP Focus
  Decelerates and focuses the ions and introduces the ions to the Q-pole. Adjust to get good sensitivity for all masses.
- Cell Exit
  Adjust to get enough sensitivity for all masses in standard mode. There is an optimum value near the OctP bias. If the OctP bias voltage is changed, the cell exit value also needs to be simultaneously changed by the same degree.
- Plate Bias
  It re-focuses the ion beam. This lens is set to the same voltage of Cell Exit automatically.
- OctP RF
  Adjust to get enough sensitivity for all masses. A typical range is from 150 V to 200 V. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass gets maximum sensitivity at high voltage.
- OctP Bias
  Adjust to get enough sensitivity for all masses. The optimum value in standard mode is different from that in reaction mode and cool mode.
- QP Bias
  It controls the speed of ions when an element ion passes through the Q-Pole and prevents polyatomic ions of low energy from passing. When peak shape and

resolution are not good, adjust it up to -2 V.

QP bias needs to be more positive than the OctP bias by the value shown as below:

- In standard mode: 2 to 4 V (Block polyatomic ions produced from the residual gas in the ion lens chamber.)
- In reaction mode: 1 to 2 V (Block polyatomic ions produced from the residual gas in the ion lens chamber and the Ar-related polyatomic ions, which lost energy by reaction. If the QP Bias is set to a high voltage, the sensitivity will also decrease.)

Reaction Cell

- Reaction Mode
  Switch between standard mode, cool mode and reaction mode. When a check box is ON, it is in reaction mode and when a check box is OFF, it's in standard mode, cool mode
- H_2 Gas
  Adjust the flow rate of hydrogen gas (H_2) used as the reaction gas. The higher the flow rate of H_2 gas, the lower the sensitivity of a sample and the intensity of the background. Adjust the flow rate in order to make the background low enough and to get the necessary level of sensitivity.
- He Gas
  Adjust the flow rate of helium gas (He) used as the reaction gas. The higher the flow rate of He gas, the lower the sensitivity of a sample and the intensity of the background. Adjust the flow rate in order to make the background low enough and to get the necessary level of sensitivity.
- Optional Gas (option)
  On the Agilent 7500ce, if an optional gas line is added other than H_2 gas and He gas, another type of gas can be introduced.

Information on Sensitivity Adjustment

The Agilent 7500ce can be used with no gas (standard mode) or may use a reaction gas (reaction mode). In reaction mode, either hydrogen gas (H_2 mode) or helium gas (He mode) is used as a reaction gas.

Even using reaction mode (H_2 mode and He mode) is used, if tuning is done in the standard mode beforehand, tuning can be completed easily just by a partial change

of the lens voltage. Adjustment of the reaction gas flow rate is done by measuring the flow rate correlation of the BEC (background equivalent concentration) with a blank solution and a standard solution. This is described below.

It is described for adjusting the sensitivity of the Agilent 7500ce for the next 3 modes in this section.

- Standard Mode
- Reaction Mode (H_2 Mode)
- Reaction Mode (He Mode)

NOTE Once a tuning parameter is determined, and a matrix sample is not introduced, only a fine adjustment of voltage, flow rate, etc. is needed for daily tuning.

NOTE It is good practice for tuning parameter to be saved under different file names after tuning in each mode.

Standard Mode (When Reaction Gas is Not Used)

Tune according to the function of each parameter above (Page 4-48) and the table of each parameter's value.

It is also possible to tune in standard mode by using Autotune (Page 4-102).

Typical values of Tuning Parameters in the Standard Mode
(with ShieldTorch, Micro Mist Nebulizer)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.9	Normally used 0.9
Makeup Gas (L/min)	0.15	0.1 to 0.3
PeriPump 1 (rps)	0.1	
S/C temp (ºC)	2	Normally used at 2ºC
Extract 1 (V)	4.0	2 to 6
Extract 2 (V)	-140	-140 to -110
Omega Bias-ce (V)	-22	-30 to -16
Omega Lens-ce (V)	1.2	-2 to 3
Cell Entrance (V)	-26	-40 to -20
QP Focus (V)	2	-2 to 4
Cell Exit (V)	-30	-45 to -20
OctP RF (V)	150	100 to 200
OctPBias (V)	-6	-12 to -6
QP Bias (V)	-3	-5 to -3 (3V positive than OctP Bias)

Typical values of Sensitivity (Standard Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{7}Li	> 2000	< 15%
^{89}Y	>6000	< 15%
^{205}Tl	>3000	< 15%

H_2 Mode (When Hydrogen is used as Reaction Gas)

It is recommended to tune in H_2 mode after tuning in standard mode.

Autotune cannot be used in H_2 mode

The following is an example of the sensitivity tuning method in the H_2 mode. This is assuming that tuning in the standard mode is completed. First, tune the lens voltage under the appropriate H_2 flow rate, and then adjust the H_2 flow rate.

1 Adjustment of Lens Voltage

a. Save the tuning parameters in standard mode.

b. Aspirate the tuning solution and confirm that Co (59 amu), Y (89 amu), and Tl (205 amu) show normal 7500ce counts on the **ICP-MS Tuning-Sensitivity** Window.

c. Turn **Reaction Mode** Check Box ON and set the value to 5 mL/min. for the **H_2 Gas** flow.

d. Set the QP Focus to -15V, the OctP Bias to -18V, and the QP Bias to -16V. Use the values from Standard Mode for the following parameters;

- RF Power
- Sampling Depth
- Carrier Gas
- Makeup Gas
- Torch-H, V
- Extract 1
- Extract 2
- Omega Bias -ce
- Omega Lens-ce
- OctP RF

e. Maximize the sensitivity of Y (89 amu) by adjusting Cell Entrance, QP Focus, and Cell Exit. If the sensitivity is low, change the Cell Exit to a lower voltage. In this case, sometimes, the background may increase. Monitor the background when adjusting.

f. Adjust the Makeup Gas and choose a value that gives maximum sensitivity.

g. Readjust the Cell Entrance, QP Focus, and Cell Exit to obtain best sensitivity.

h. (Background Check) Aspirate fresh DI water and measure the background of mass (56 amu and 78 amu), which is to be measured in H_2 mode. If the count of 56 amu and 78amu is too high, increase 0.4 mL/min of H_2 flow rate, and then repeat step e to step g.

Typical values of Tuning Parameters in the H_2 Mode
(with ShieldTorch, MicroMist Nebulizer)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.9	Normally used 0.9
Makeup Gas (L/min)	0.15	0.1 to 0.3
PeriPump 1 (rps)	0.1	
S/C temp (ºC)	2	Normally used at 2ºC
Extract 1 (V)	4.0	2 to 6
Extract 2 (V)	-140	-140 to -110
Omega Bias-ce (V)	-22	-30 to -16
Omega Lens-ce (V)	1.2	-2 to 3
Cell Entrance (V)	-26	-40 to -20
QP Focus (V)	-15	-20 to 0
Cell Exit (V)	-30	-80 to -20
OctP RF (V)	150	100 to 200
OctPBias (V)	-18	Normally used -18
QP Bias (V)	-3	-Normally used -16
H_2 Gas (ml/min)	5	3 to 6

Typical values of Sensitivity (H_2 Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{89}Y	>2000	< 15%

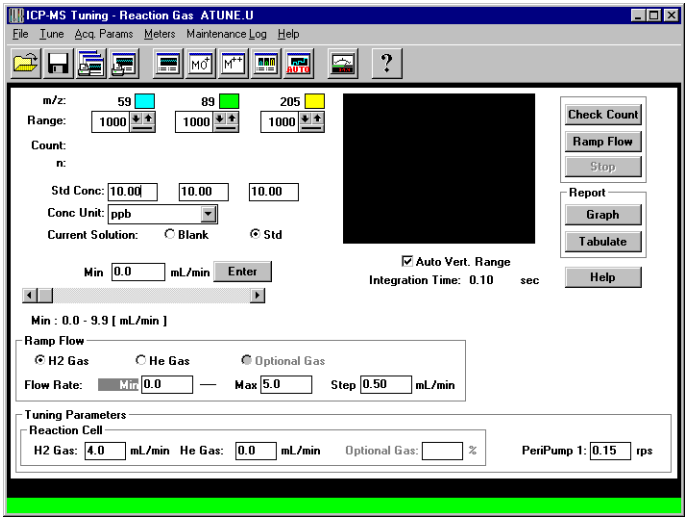
2 Adjustment of H_2 gas flow

The higher the flow of H_2 gas, the lower the intensity of the sample and the background. Adjust the flow in order to make the background low enough and to get a necessary level of sensitivity. To optimize the flow rate of H_2 gas use the **ICP-MS Tuning-Reaction Gas** Window as follows:

1) Select **Tune>>Reaction Gas** in the **ICP-MS Tuning** Window.

   The **ICP-MS Tuning-Reaction Gas** Window will appear.

   In this window, the data of the tuning and blank solutions are collected as the gas flow change. The **Graph** button will produce a graph of the sensitivity and BEC (Background Equivalent Concentration). This graph will help to determine the flow rate of H_2 gas.



Reaction Gas Tuning Window

2) Aspirate the tuning solution.

3) Set the mass number and integration time by selecting Acq.Params.

4) Input the concentration of each mass number of the tuning solution in **Std Conc** Text Box and select the concentration unit in **Conc Unit**.

5) Select **Std** (standard solution) in **Current Solution**.

6) Select **H_2 Gas** in the **Ramp Flow** group and input the minimum value, maximum value, and the step size of the H_2 gas flow rate to adjust the **Flow Rate**.

7) Display the signals by clicking the **Check Count** button and confirm that the signal is stable. At this time, the flow rate of the H_2 gas doesn't change. Click the **Stop** button after confirming signal stability.

8) Click the **Ramp Flow** button. Sensitivity data is collected by changing the H_2 gas within the flow rate range that was previously set. The message will be displayed on the message line after the data collection is complete.

9) When the previous step has finished, aspirate fresh DI water or nitric acid solution (blank solution) whose concentration is the same as the tuning solution.

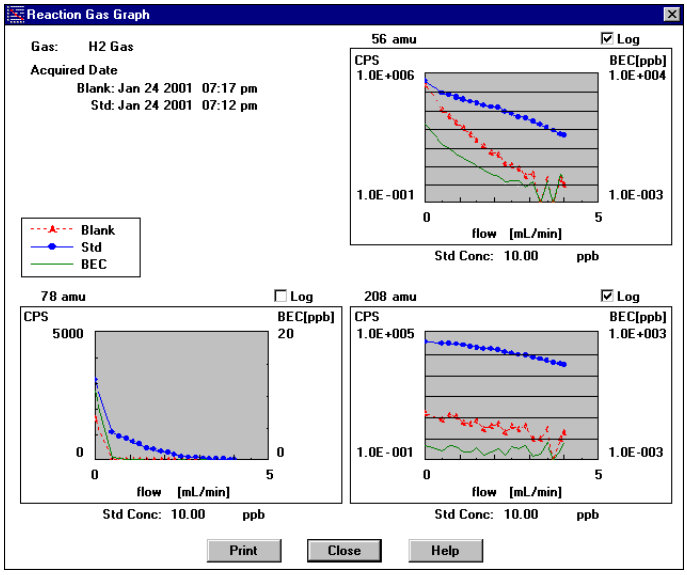
10) Select **Blank** (blank solution) in **Current Solution**.

11) Display the signal by clicking the **Check Count** button and confirm that the signal is at a normal background level replacing the solution. If it's not low enough, wait until it falls. At this time, the flow rate of the H_2 gas doesn't change. Click the **Stop** button once confirming an acceptable background level.

12) Click the **Ramp Flow** button. The background data is collected by adjusting the H_2 gas within the flow rate range that was previously set. A message will be displayed on the message line after the data collection is complete.

13) After collecting data from both the tuning and blank solutions, click the **Graph** button to view the result.

14) The **Reaction Gas Graph** Dialog Box will appear. In the dialog box, the sensitivity and the BEC (Background Equivalent Concentration) of the tuning and blank solution, for the different flow rates of H_2 gas, are plotted each mass measured. Examine this graph and determine the best sensitivity and flow rate of the H_2 gas while satisfying the BEC.



Reaction Gas Graph Dialog Box

NOTE The **Reaction Gas Report** will appear when you click the **Tabulate** button in the **ICP-MS Tuning-Reaction Gas** Window.

15) Click the **Close** button, close the **Reaction Gas Graph** Dialog Box, and return to the **ICP-MS Tuning-Reaction Gas** Window.

16) Input the flow rate of H_2 gas determined in step 14) in the **H_2 Gas** Text Box in the **Tuning Parameters**. The optimization of H_2 gas is now complete.

NOTE The blank solution can be measured before the tuning solution.

NOTE Previous data will be overwritten once the **Ramp Flow** starts. To keep the previous data, print the graph and save the table beforehand.

NOTE It is good practice for the tuning parameter to be saved under a different file name from a standard mode tuning file.

NOTE Once the parameter of the reaction gas flow rate is tuned; only a fine adjustment of the flow rate is enough for daily tuning.

He Mode (When Helium is used as the Reaction Gas)

It is recommended to tune in He mode after tuning in H_2 mode.

Autotune cannot be used in He mode.

The following is an example of the sensitivity adjustment method in He mode: This assumes that adjustment in H_2 mode is complete. First, tune the lens voltage with an appropriate He flow, and then adjust the He flow for optimum conditions.

1 Adjustment of lens voltage

a. Save the tuning parameters from H_2 mode.

b. Aspirate the tuning solution and confirm that Co (59 amu), Y (89 amu), and Tl (205 amu) show normal counts in the **ICP-MS Tuning-Sensitivity** Window.

c. Turn **Reaction Mode** Check Box ON and set the value 3.5 mL/min. for the **He Gas** flow rate.

d. Check the sensitivity. If the sensitivity is not enough, tune same as H_2 mode.

Typical values of Tuning Parameters in the He Mode
(with ShieldTorch, Micro Mist Nebulizer)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.9	Normally used 0.9
Makeup Gas (L/min)	0.15	0.1 to 0.3
PeriPump 1 (rps)	0.1	
S/C temp (°C)	2	Normally used at 2°C
Extract 1 (V)	4.0	2 to 6
Extract 2 (V)	-140	-140 to 0-110
Omega Bias-ce (V)	-22	-30 to -16
Omega Lens-ce (V)	1.2	-2 to 3
Cell Entrance (V)	-26	-40 to -20
QP Focus (V)	-15	-20 to 0
Cell Exit (V)	-30	-80 to -20
OctP RF (V)	150	100 to 200
OctPBias (V)	-18	Normally used -18
QP Bias (V)	-16	-Normally used -16
He Gas (ml/min)	3.5	2.5 to 5.5

Typical values of Sensitivity (He Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{59}Co	> 1000	< 15%

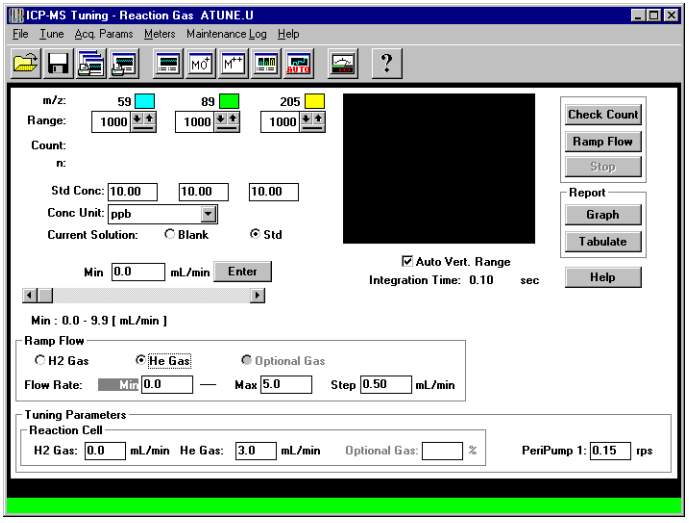
2 Adjustment of He gas flow rate

The higher the flow of He gas, the lower the intensity of the sample and background. Adjust the flow in order to make the background low enough and to get a necessary level of sensitivity. To optimize the flow of He use the **ICP-MS Tuning-Reaction Gas** Window as follows:

1) Select **Tune>>Reaction Gas** in the **ICP-MS Tuning** Window.

   The **ICP-MS Tuning-Reaction Gas** Window will appear.

   In this window, the data of the tuning and blank solutions are collected as the gas flow rates change. The **Graph** button will produce a graph of the sensitivity and BEC (Background Equivalent Concentration). This graph will help to determine the flow rate of He gas.



Reaction Gas Tuning Window

2) Aspirate the tuning solution.

3) Set the mass number and integration time by selecting Acq.Params.

4) Input the concentration of each mass number of the tuning solution in the **Std Conc** Text Box and select the concentration unit in **Conc Unit**.

5) Select **Std** (standard solution) in **Current Solution**.

6) Select **He Gas** in the **Ramp Flow** group and input the minimum value, maximum value, and the step size of the He gas flow rate to adjust the **Flow Rate**.

7) Display the signals by clicking the **Check Count** button and confirm that the signal is stable. At this time, the flow rate of He gas doesn't change. Click the **Stop** button after confirming signal stability.

8) Click the **Ramp Flow** button. Sensitivity data is collected by changing the He gas within the flow rate range that was previously set. A message will be displayed on the message line after the data collection is complete.

9) When the previous step has finished, aspirate fresh DI water or nitric acid solution (blank solution) whose concentration is the same as the tuning solution.

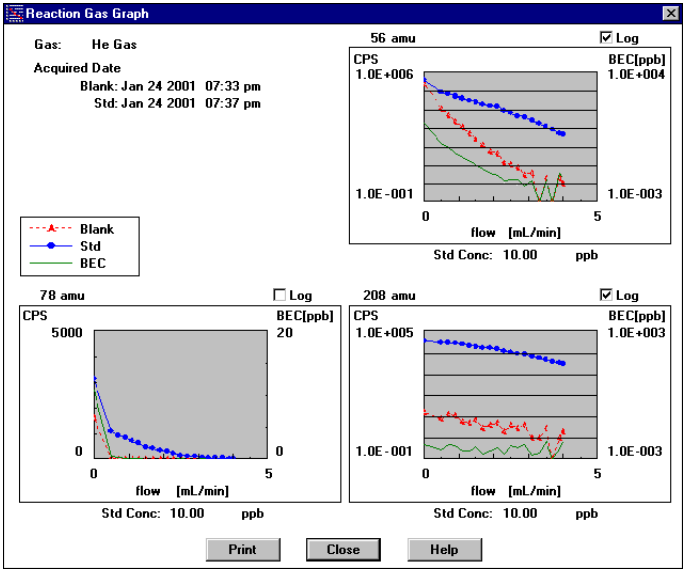
10) Select **Blank** (blank solution) in the **Current Solution**.

11) Display the signal by clicking the **Check Count** button and confirm that the signal is at a normal background level after replacing the solution. If it's not low enough, wait until it falls. At this time, the flow rate of He gas doesn't change. Click the **Stop** button after confirming an accepted background level.

12) Click the **Ramp Flow** button. The background data is collected by adjusting the He gas within the flow rate range that was previously set. A message will be displayed on the message line once the data collection is complete.

13) After collecting data from both the tuning and blank solutions, click the **Graph** button to view the result.

14) The **Reaction Gas Graph** Dialog Box will appear. In the dialog box, the sensitivity and BEC (Background Equivalent Concentration) of the tuning solution and blank solution, for the different flow rates of He gas, are plotted for each mass measured. Examine this graph and determine the best sensitivity and flow rate of He gas while satisfying the BEC.



Reaction Gas Graph Dialog box

NOTE The **Reaction Gas Report** will appear when you click the **Tabulate** button in the **ICP-MS Tuning-Reaction Gas** Window.

15) Click the **Close** button, close the **Reaction Gas Graph** Dialog Box, and return to the **ICP-MS Tuning-Reaction Gas** Window.

16) Input the flow rate of He gas determined in step 14) in the **He Gas** Text Box in the **Tuning Parameters**. The optimization of He gas is now complete.

NOTE The blank solution can be measured before the tuning solution.

NOTE Previous data will be overwritten once the **Ramp Flow** starts. To keep the previous data, print the graph and save the table beforehand.

NOTE He mode is effective to decrease polyatomic ions produced from a sample matrix such as ClO, NaAr, and ArCl.

NOTE It is good practice for the tuning parameter to be saved under a different file name from a standard mode or H_2 mode tune file.

NOTE Once the parameter of the reaction gas flow rate is tuned; only a fine adjustment of the flow rate is needed for daily tuning.

Reducing Oxide Ions

The reason for tuning to reduce Oxide ions is the same as other Agilent 7500 series. For details, refer to "Reducing Oxide Ions" on page 4-12.

The typical value of oxide ions is less than 10% in standard mode.

Reducing Doubly Charged Ions

The reason tuning to reduce Doubly Charged ions is the same as other Agilent 7500 series. For details, refer to "Reducing Doubly Charged Ions" on page 4-15.

The typical value of doubly charged ions is less than 10% in standard mode.

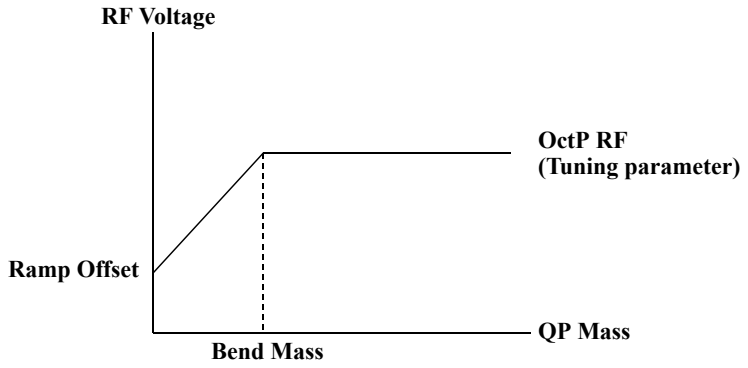
Scan of the Octopole

Generally, the RF voltage of the Octopole is fixed at a certain value, this has no effect for selecting the mass of an element to be measured. However, when an element of low mass is measured with higher sensitivity, the RF voltage of the Octopole can be scanned by working with the Q-pole Scan. Be careful about the stability of signals because the scan of Octopole may increase the factors to make the instrument unstable.

Scan line of RF voltage

Scan of the Octopole RF voltage will have a scan line as shown in the following figure. The RF voltage will rise in proportion to the mass on the low mass side the voltage will be constant on masses number larger than the bend mass number.

If the Octopole is not scanned, the voltage will be constant (OctP RF) on all the mass numbers.



Scan line of Octopole

Three parameters are used to determine this scan line:

- Ramp Offset: The Offset voltage of the RF voltage in the mass portion area.
- Bend Mass: The Mass number moving from the mass portion area to the constant voltage area.
- OctP RF: The RF voltage in the constant mass area.

OctP RF can be set in tuning parameters in the **ICP-MS Tuning** Window.

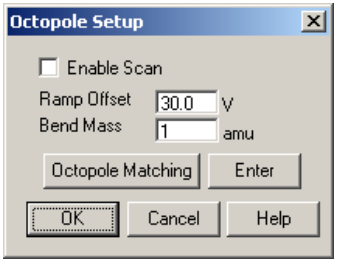
To enable the scan and set the two remaining parameters you use the **Octopole Setup** Dialog Box.

To set these parameters, carry out the following steps:

1 **Select *Instrument>>Instrument Control* in the *ICP-MS Top* Window, and display the *ICP-MS Instrument Control* Window**

2 **Select *Maintenance>>Octopole* in the *Instrument Control* Window.**

The **Octopole Setup** Dialog Box will appear.



Octopole Setup Dialog Box

3 Set each parameter necessary to scan the Octopole

- **Enable Scan:** To scan, input a check mark in this check box. If there is no check mark in the check box, Octopole will not be scanned. Usually, leave the check box blank.
- **Ramp Offset:** Input the offset voltage of the RF voltage in the mass portion area. The range is 30V to 200V. Input a value lower than the OctP RF in the tuning parameter.
- **Bend Mass:** Input the mass number moving from the mass portion area to the constant voltage area. The range is 1 amu to 260amu. The Mass number input in the bend mass will be included in the constant voltage area. To make all the mass range the mass portion area, input 260 amu.

4 Click *OK* after finishing the parameter setting.

The setting is complete and the **Octopole Setup** Dialog Box closes.

Tuning for Sensitivity (Agilent 7500cs)

Adjust sensitivity of elements to be measured obtain high stable signals over the whole mass range. The standard tuning solution is a 1% nitric acid solution containing 1 ng/mL (ppb) of each of the following: Li, Co, Y, Ce, and Tl.

When using the Agilent 7500a refer to "Tuning for Sensitivity (Agilent 7500a)" on page 4-4.

When using the Agilent 7500ce, refer to "Tuning for Sensitivity (Agilent 7500ce)" on page 4-39.

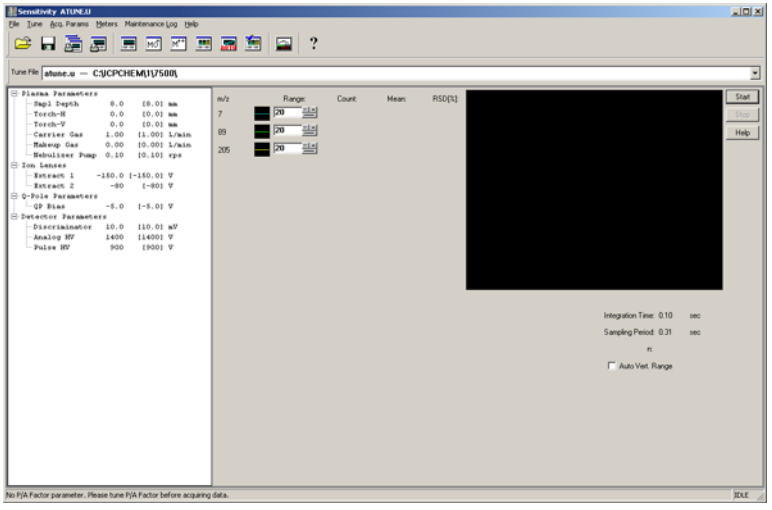
CAUTION

Whenever the Agilent 7500cs is used, the ShieldTorch must be installed in it. For installation of the ShieldTorch, refer to the *Agilent 7500 ICP-MS Hardware Manual*.

Adjust sensitivity using the following steps:

1 **Select *Instrument>>Tune* in the ICP-MS Top Window.**

The **ICP-MS Tuning-Sensitivity** Window will appear.



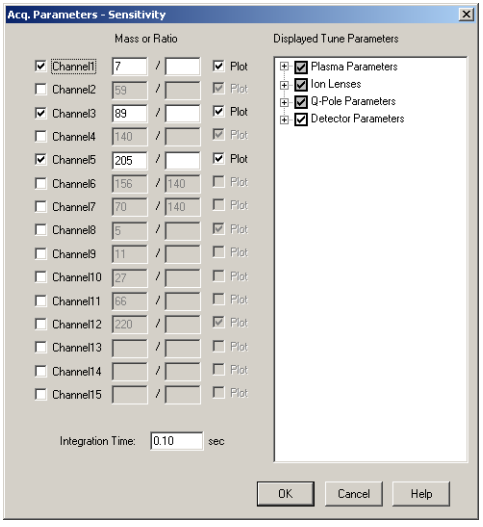
ICP-MS Tuning-Sensitivity Window

2 Select *Tune>>Acq. Params.*

The ***Acquisition Parameters-Sensitivity*** Dialog Box will appear.

Use this dialog box to set or change the masses you want to view, the integration time, or the ratio of any two channels. You can also display the ratio of signal strengths of any two selected masses.

This dialog box also allows you to select the channels to be displayed on the ***ICP-MS Tuning - Sensitivity*** window, the tuning parameters to be indicated in the tree display, and the channels to be plotted on the graph.



Acquisition Parameters-Sensitivity Dialog Box

NOTE To use tuning parameter settings from a previous tuning session as a starting point for this session, refer to "Creating and Using a Tune File" on page 4-99.

3 Select the check boxes of the channels to be displayed on the *ICP-MS Tuning - Sensitivity* window.

4 Enter the values of the masses in the left-side boxes under "Mass or Ratio."

5 To set a strength ratio, type the mass values in the right-side boxes under "Mass or Ratio."

6 Enter the data collection time (integration time) for each mass in the Integration Time: text box

Generally, set 0.1 seconds per one mass value.

7 Select the *Plot* check boxes of the channels to be displayed on the graph in the *ICP-MS Tuning - Sensitivity* window.

8 In the Displayed Tuning Parameters tree display, select the check boxes of the tuning parameters to be displayed in the *ICP-MS Tuning - Sensitivity* window.

Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Clicking on a parameter check box will alternately select or deselect the parameter.

A parameter in gray indicates that only some of the parameters contained within are selected.

9 Click *OK*.

Clicking ***OK*** closes the ***Acquisition Parameters - Sensitivity*** window and displays the entered settings in the ***ICP-MS Tuning - Sensitivity*** window.

10 Put the sample uptake tube into the tuning solution.

Wait for the uptake of the sample to the nebulizer.

11 Click *Start* to monitor the Agilent 7500's signal and view the numerical values in the real-time display.

As the signal appears, ChemStation inserts numerical values for the count, mean, RSD, and ratio.

The window includes a box for a real-time display of the instrument's signal. The box contains up to 200 data points. The current counts of each mass are given, the mean and RSD (relative standard deviation) of the signal are given for the data points shown in the display.

NOTE When you check **Auto Vert. Range** check box, the vertical range of the real-time display is automatically adjusted.

NOTE You can print a copy of the real-time display at any time during the tuning procedure. See "Generating a Tune Report" on page 4-97.

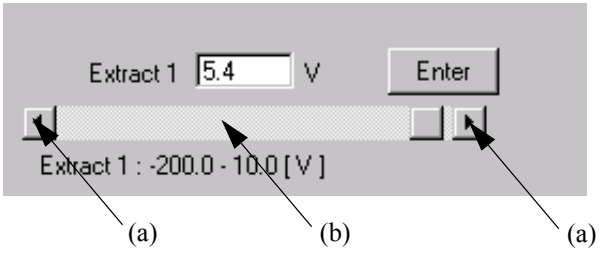
12 Adjust the tuning parameters if necessary.

Tuning parameters are shown in the tree display on the left side of the window. Parameters can be displayed or hidden by clicking on the [+]/[-] box.

Indicated on the right side of each parameter in [ ] are the adjustment value and the pre-adjustment numerical value (value stored in the tune file).

Clicking on a parameter will cause a text box or a scroll bar to appear at the lower center section of the window. Use one of the following methods to set a parameter value.

- Enter a new parameter value in the text box (next to the parameter you want to change or above the scroll bar).
- Click the right or left arrow at the end of the scroll bar (a), which changes the parameter value by small increments.
- Click the scroll bar (b), which changes the parameter value by larger increments.
- Click ***Apply*** to set the adjusted parameter value.
  Clicking ***Cancel*** will cause the pre-adjustment parameter value (value stored in the tune file) to be set.



Adjust the tuning parameters until the signal appears constant and the numerical values meet performance requirements. The performance specification depends on the application.

For the workings of each tuning parameter, refer to "Each Parameter's Function" on page 4-66.

CAUTION

Use care when adjusting values by clicking the scroll bar or entering a new value in the box above the scroll bar. Changing parameter values too quickly can cause extreme changes in the Agilent 7500 and can alter its performance over time. In most cases, you should adjust the tuning parameters by clicking the arrows at the end of the scroll bar.

NOTE

To display all the tuning parameters, check **Advanced Setting** to ON.

13 Click *Stop* to stop the display.

ChemStation freezes the real-time display until you click **Start**. When you click **Start**, the display clears and starts again.

Each Parameter's Function

The working of each parameter used for sensitivity adjustment is described below.

NOTE The Agilent 7500cs has four modes: Standard Mode, which does not use a reaction gas Reaction Mode, which uses hydrogen (H_2) gas or helium (He) gas as a reaction gas and Cool Mode For information on sensitivity adjustment in each mode, refer to "Information on Sensitivity Adjustment" on page 4-70.

Plasma Condition

- RF Power
  It controls the RF power supplied to the load coil. A higher RF power increases the sensitivity and reduces oxide and doubly charged ions. However, the sensitivity of lighter masses may be decreased when the RF power is too high.
- RF Matching
  Adjusts reflected RF returned from the load coil.
  To reduce the reflected power, use of the automatic adjustment function is recommended. To use the automatic adjustment function, select **Tune>>RF Matching** on the Tuning window, and then follow the displayed dialog box.

NOTE If you adjust the RF Matching manually, use only the scroll arrows to change this value. If you click on the scroll bar itself, the instrument will turn off the plasma. Adjust until the RF reflected power meter reads as low as possible. Standard value for RF matching is approximately 1.6 to 2.0 V.

- Smpl Depth
  It controls the distance between the edge of the load coil and the tip of the sampling cone. A shorter sampling depth increases sensitivity, but the oxide level increases as well.
- Torch-H, Torch-V
  It adjusts the torch position. The Torch-H and Torch-V parameters measure the horizontal and vertical movement of the torch relative to the interface. Adjust to get the highest sensitivity for all masses. When the sampling depth is changed, adjustment of the Torch-H/V will be required. If these parameters are changed using the software scroll bar, the plasma may turn off. Use only the scroll arrows.
- Carrier Gas
  It controls the nebulization efficiency of a sample and the sample uptake rate when self-aspiration is applied. Both high gas flow and the low gas flow causes signal instability when using the Micro Flow Nebulizer (100 uL/min). In general,

a gas flow of 0.8 L/min.

- Makeup Gas
  Makes up Ar gas into the spray chamber and is mixed with the carrier gas. When the Ar gas supply pressure is not high enough to get a sufficient carrier gas flow, the makeup gas is used.
- Optional Gas (option)
  When the optional gas line is added, it is possible to introduce a gas other than argon. (e.g. introduction of oxygen etc. when measuring organic solvent)
- PeriPump 1
  Controls a sample uptake rate and the drain from the spray chamber. It affects sensitivity, stability of signal, and oxide and doubly charged formation. Higher speed increases sensitivity, but too high speed, decreases sensitivity. Higher speed increases oxide and doubly charged ions.

CAUTION 

Even when self-aspiration is used for sampling, the PeriPump 1 must be operated to drain the sample from the spray chamber, if this is not optimized the spray chamber will fill with sample solution.

- PeriPump 2 (option)
  It can be used when a second (optional) peristaltic pump is installed on the ICP-MS. It is possible to shorten the rinse time or the sample uptake time.
- S/C Temp
  Controls the temperature of the spray chamber. Lowering the spray chamber temperature causes the sample vapor temperature to lower and removes more water, thus, reducing the oxide levels. It should be set at 2 ºC for aqueous sample to avoid ice formation. When aspirating organic solvents run the chamber at -5 ºC.

Ion Lens

- Extract 1
  It extracts ions from the plasma to accelerate them toward the Einzel lens. Adjust to get enough sensitivity for all masses. This is lens that needs the most frequently adjusted everyday.

  In order to get maximum sensitivity, adjust between 4 V and 8 V. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass number gets maximum sensitivity at high voltage. Therefore, select the appropriate voltage. Even with the voltage around -100V, high sensitivity can be obtained. However, the background of polyatomic ions

may be high in reaction mode (H_2 mode or He mode)

- Extract 2
  It focuses the ion beam from the extraction lens. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass gets maximum sensitivity at high voltage. Therefore, set a value to get optimum sensitivity foe the whole mass range.
- Omega Lens-cs
  Separating the ions from photon and introduce the ions to the Reaction Cell. Adjusts to get good sensitivity for all masses.
- Omega Bias-cs
  Provides the same potential at the entrance and exit of the Omega lens block. It must be used at voltages more positive than -40V. The more negative voltage, the higher the sensitivity, but the background might increase.Cell Focus
  Locates on the first lens of the Reaction Cell. The voltage is set to 0V at all times. This lens is not displayed as a tuning parameter.
- Cell Entrance
  Adjust to get enough sensitivity for all masses. It is necessary to set a lower voltage than the OctP bias. If the voltage is set higher than the OctP bias, polyatomic ions may increase on some specific masses.
- QP Focus
  Decelerates and focuses the ions and introduces the ions to the Q-pole. Adjust to get good sensitivity for all masses.
- Cell Exit
  Adjust to get enough sensitivity for all masses in standard mode. There is an optimum value near the OctP bias. If the OctP bias voltage is changed, the cell exit value also needs to be simultaneously changed by the same degree.
- Plate Bias
  It re-focuses the ion beam. This lens is set to the same voltage of Cell Exit automatically.
- OctP RF
  Adjust to get enough sensitivity for all masses. A typical range is from 150 V to 200 V. An element of low mass gets maximum sensitivity at low voltage. On the other hand, an element of high mass gets maximum sensitivity at high voltage.
- OctP Bias
  Adjust to get enough sensitivity for all masses. The optimum value in standard mode is different from that in reaction mode and cool mode.

- QP Bias
  It controls the speed of ions when an element ion passes through the Q-Pole and prevents polyatomic ions of low energy from passing. When peak shape and resolution are not good, adjust it up to -2 V.

  QP bias needs to be more positive than the OctP bias by the value shown as below:

  - In standard mode: 2 to 4 V (Block polyatomic ions produced from the residual gas in the ion lens chamber.)
  - In reaction mode: 1 to 2 V (Block polyatomic ions produced from the residual gas in the ion lens chamber and the Ar-related polyatomic ions, which lost energy by reaction. If the QP Bias is set to a high voltage, the sensitivity will also decrease.)

Reaction Cell

- Reaction Mode
  Switch between standard mode, cool mode and reaction mode. When a check box is ON, it is in reaction mode and when a check box is OFF, it's in standard mode, cool mode
- H_2 Gas
  Adjust the flow rate of hydrogen gas (H_2) used as the reaction gas. The higher the flow rate of H_2 gas, the lower the sensitivity of a sample and the intensity of the background. Adjust the flow rate in order to make the background low enough and to get the necessary level of sensitivity.
- He Gas
  Adjust the flow rate of helium gas (He) used as the reaction gas. The higher the flow rate of He gas, the lower the sensitivity of a sample and the intensity of the background. Adjust the flow rate in order to make the background low enough and to get the necessary level of sensitivity.
- Optional Gas (option)
  On the Agilent 7500cs, if an optional gas line is added other than H_2 gas and He gas, another type of gas can be introduced.

Information on Sensitivity Adjustment

The Agilent 7500cs can be used with no gas (standard mode, cool mode) or may use a reaction gas (reaction mode). In reaction mode, either hydrogen gas (H_2 mode) or helium gas (He mode) is used as a reaction gas.

Even using reaction mode (H_2 mode and He mode) is used, if tuning is done in the standard mode beforehand, tuning can be completed easily just by a partial change of the lens voltage. Adjustment of the reaction gas flow rate is done by measuring the flow rate correlation of the BEC (background equivalent concentration) with a blank solution and a standard solution. This is described below.

In the cool mode, tuning is executed with the cool plasma condition which has the cooler plasma temperature.

It is described for adjusting the sensitivity of the Agilent 7500cs for the next 4 modes in this section.

- Standard Mode (Page 4-70)
- Reaction Mode (H_2 Mode) (Page 4-72)
- Reaction Mode (He Mode) (Page 4-77)
- Cool Mode (Page 4-82)

NOTE Once a tuning parameter is determined, and a matrix sample is not introduced, only a fine adjustment of voltage, flow rate, etc. is needed for daily tuning.

NOTE It is good practice for tuning parameter to be saved under different file names after tuning in each mode.

NOTE For the ShieldTorch and Cool Plasma, refer also to the "Agilent 7500 ICP-MS Option Instruction Manual".

Standard Mode (When Reaction Gas is Not Used)

Tune according to the function of each parameter above (Page 4-66) and the table of each parameter's value.

It is also possible to tune in standard mode by using Autotune (Page 4-102).

Typical values of Tuning Parameters in the Standard Mode
(with ShieldTorch, Micro Flow Nebulizer, self-aspiration)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.8	0.6 to 1.0
Makeup Gas (L/min)	0.45	0.3 to 0.7
PeriPump 1 (rps)	0.1	
S/C temp (°C)	2	Normally used at 2°C
Extract 1 (V)	5.4	4 to 8
Extract 2 (V)	-100	-150 to -60
Omega Bias-cs (V)	-32	-45 to -25
Omega Lens-cs (V)	8.6	5 to 10
Cell Entrance (V)	-26	-40 to -20
QP Focus (V)	2	-2 to 5
Cell Exit (V)	-30	-45 to -20
OctP RF (V)	150	100 to 200
OctPBias (V)	-6	-12 to -6
QP Bias (V)	-3	-5 to -3

Typical values of Sensitivity (Standard Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{7}Li	> 4000	< 15%
^{89}Y	>12500	< 15%
^{205}Tl	>6000	< 15%

H_2 Mode (When Hydrogen is used as Reaction Gas)

It is recommended to tune in H_2 mode after tuning in standard mode.

Autotune cannot be used in H_2 mode

The following is an example of the sensitivity tuning method in the H_2 mode. This is assuming that tuning in the standard mode is completed. First, tune the lens voltage under the appropriate H_2 flow rate, and then adjust the H_2 flow rate.

1 Adjustment of Lens Voltage

a. Save the tuning parameters in standard mode.

b. Aspirate the tuning solution and confirm that Co (59 amu), Y (89 amu), and Tl (205 amu) show normal 7500cs counts on the **ICP-MS Tuning-Sensitivity** Window.

c. Turn **Reaction Mode** Check Box ON and set the value to 4.5 mL/min. for the **H_2 Gas** flow.

d. Enter the typical tune value for each parameter. But use the values from Standard Mode for the following parameters;

- RF Power
- Sampling Depth
- Carrier Gas
- Makeup Gas
- Torch-H, V
- Extract 1
- Extract 2

e. Maximize the sensitivity of Y (89 amu) by adjusting Omega Lens-cs, QP Focus, and OctP RF.

f. Adjust the Makeup Gas and choose a value that gives maximum sensitivity.

g. Readjust the Omega Lens-cs, QP Focus, and OctP RF to obtain best sensitivity.

h. (Background Check) Aspirate fresh DI water and measure the background of mass (56 amu), which is to be measured in H_2 mode. If the count of 56 amu is too high, increase 0.4 mL/min of H_2 flow rate, and then repeat step e to step g.

i. If the sensitivity is low, change the Omega Bias-cs and the Cell Exit to a lower voltage. In this case, sometimes, the background may increase. Monitor the background when adjusting.

Typical values of Tuning Parameters in the H_2 Mode
(with ShieldTorch, Micro Flow Nebulizer, self-aspiration)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.8	0.6 to 1.0
Makeup Gas (L/min)	0.45	0.3 to 0.7
PeriPump 1 (rps)	0.1	
S/C temp (°C)	2	Normally used at 2°C
Extract 1 (V)	5.4	4 to 8
Extract 2 (V)	-100	-150 to -60
Omega Bias-cs (V)	-40	-50 to -30
Omega Lens-cs (V)	8.6	5 to 10
Cell Entrance (V)	-30	-50 to -30
QP Focus (V)	-12	-20 to 0
Cell Exit (V)	-40	-80 to -20
OctP RF (V)	180	100 to 200
OctPBias (V)	-18	Normally used -18
QP Bias (V)	-3	-Normally used -16
H_2 Gas (ml/min)	4.5	3 to 6

Typical values of Sensitivity (H_2 Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{59}Co	> 1500	< 15%
^{89}Y	>8000	< 15%
^{205}Tl	>7000	< 15%

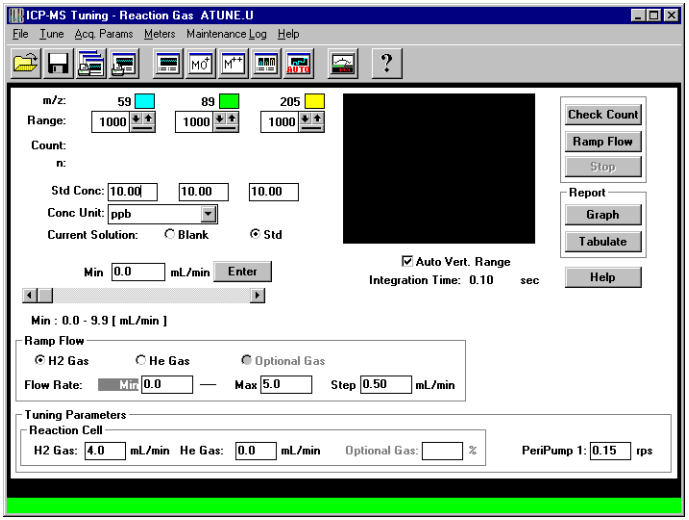
2 Adjustment of H_2 gas flow

The higher the flow of H_2 gas, the lower the intensity of the sample and the background. Adjust the flow in order to make the background low enough and to get a necessary level of sensitivity. To optimize the flow rate of H_2 gas use the **ICP-MS Tuning-Reaction Gas** Window as follows:

1) Select **Tune>>Reaction Gas** in the **ICP-MS Tuning** Window.

   The **ICP-MS Tuning-Reaction Gas** Window will appear.

   In this window, the data of the tuning and blank solutions are collected as the gas flow change. The **Graph** button will produce a graph of the sensitivity and BEC (Background Equivalent Concentration). This graph will help to determine the flow rate of H_2 gas.



Reaction Gas Tuning Window

2) Aspirate the tuning solution.

3) Set the mass number and integration time by selecting Acq.Params.

4) Input the concentration of each mass number of the tuning solution in **Std Conc** Text Box and select the concentration unit in **Conc Unit**.

5) Select **Std** (standard solution) in **Current Solution**.

6) Select **H_2 Gas** in the **Ramp Flow** group and input the minimum value, maximum value, and the step size of the H_2 gas flow rate to adjust the **Flow Rate**.

7) Display the signals by clicking the **Check Count** button and confirm that the signal is stable. At this time, the flow rate of the H_2 gas doesn't change. Click the **Stop** button after confirming signal stability.

8) Click the **Ramp Flow** button. Sensitivity data is collected by changing the H_2 gas within the flow rate range that was previously set. The message will be displayed on the message line after the data collection is complete.

9) When the previous step has finished, aspirate fresh DI water or nitric acid solution (blank solution) whose concentration is the same as the tuning solution.

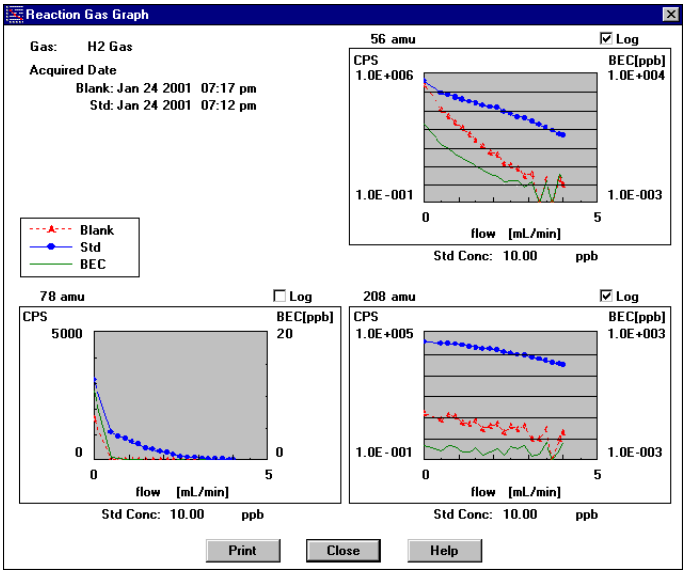
10) Select **Blank** (blank solution) in **Current Solution**.

11) Display the signal by clicking the **Check Count** button and confirm that the signal is at a normal background level replacing the solution. If it's not low enough, wait until it falls. At this time, the flow rate of the H_2 gas doesn't change. Click the **Stop** button once confirming an acceptable background level.

12) Click the **Ramp Flow** button. The background data is collected by adjusting the H_2 gas within the flow rate range that was previously set. A message will be displayed on the message line after the data collection is complete.

13) After collecting data from both the tuning and blank solutions, click the **Graph** button to view the result.

14) The **Reaction Gas Graph** Dialog Box will appear. In the dialog box, the sensitivity and the BEC (Background Equivalent Concentration) of the tuning and blank solution, for the different flow rates of H_2 gas, are plotted each mass measured. Examine this graph and determine the best sensitivity and flow rate of the H_2 gas while satisfying the BEC.



Reaction Gas Graph Dialog Box

NOTE The **Reaction Gas Report** will appear when you click the **Tabulate** button in the **ICP-MS Tuning-Reaction Gas** Window.

15) Click the **Close** button, close the **Reaction Gas Graph** Dialog Box, and return to the **ICP-MS Tuning-Reaction Gas** Window.

16) Input the flow rate of H_2 gas determined in step 14) in the **H_2 Gas** Text Box in the **Tuning Parameters**. The optimization of H_2 gas is now complete.

NOTE The blank solution can be measured before the tuning solution.

NOTE Previous data will be overwritten once the **Ramp Flow** starts. To keep the previous data, print the graph and save the table beforehand.

NOTE It is good practice for the tuning parameter to be saved under a different file name from a standard mode tuning file.

NOTE Once the parameter of the reaction gas flow rate is tuned; only a fine adjustment of the flow rate is enough for daily tuning.

He Mode (When Helium is used as the Reaction Gas)

It is recommended to tune in He mode after tuning in standard mode.

Autotune cannot be used in He mode.

The following is an example of the sensitivity adjustment method in He mode: This assumes that adjustment in standard mode is complete. First, tune the lens voltage with an appropriate He flow, and then adjust the He flow for optimum conditions.

1 Adjustment of lens voltage

a. Save the tuning parameters from standard mode.

b. Aspirate the tuning solution and confirm that Co (59 amu), Y (89 amu), and Tl (205 amu) show normal counts in the **ICP-MS Tuning-Sensitivity** Window.

c. Turn **Reaction Mode** Check Box ON and set the value 3.5 mL/min. for the **He Gas** flow rate.

d. Enter the typical tune value of each parameter. But use the value from the Standard Mode tune;

- RF Power
- Sampling Depth
- Carrier Gas
- Makeup Gas
- Torch-H, V
- Extract 1
- Extract 2

e. Maximize the sensitivity of Co (59 amu) by adjusting the Omega Lens-cs, QP Focus, and OctP RF.

f. Adjust the Makeup Gas and choose a value that gives maximum sensitivity.

g. Readjust the Omega Lens-cs, QP Focus, and OctP RF to obtain best sensitivity.

h. (Background Check) Aspirate fresh DI water and measure the background of mass (58 amu), which is to be measured in He mode. If the count of 58 amu is too high, increase 0.4 mL/min of He flow rate, and then repeat step e to step g.

i. If the sensitivity is low, change the Omega Bias-cs and the Cell Exit to a lower voltage. In this case, sometimes, the background may increase. Monitor the background when adjusting.

Typical values of Tuning Parameters in the He Mode
(with ShieldTorch, Micro Flow Nebulizer, self-aspiration)

Parameter	Typical Value	Adjustment
RF Power (W)	1500	Normally used 1500
Sampling Depth (mm)	8	7 to 10
Carrier Gas (L/min)	0.8	0.6 to 1.0
Makeup Gas (L/min)	0.45	0.3 to 0.7
PeriPump 1 (rps)	0.1	
S/C temp (ºC)	2	Normally used at 2ºC
Extract 1 (V)	5.4	4 to 8
Extract 2 (V)	-100	-150 to -60
Omega Bias-cs (V)	-40	-50 to -30
Omega Lens-cs (V)	8.6	5 to 10
Cell Entrance (V)	-30	-50 to -30
QP Focus (V)	-12	-20 to 0
Cell Exit (V)	-40	-80 to -20
OctP RF (V)	180	100 to 200
OctPBias (V)	-18	Normally used -18
QP Bias (V)	-16	-Normally used -16
He Gas (ml/min)	3.5	2.5 to 5.5

Typical values of Sensitivity (He Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{59}Co	> 3000	< 15%

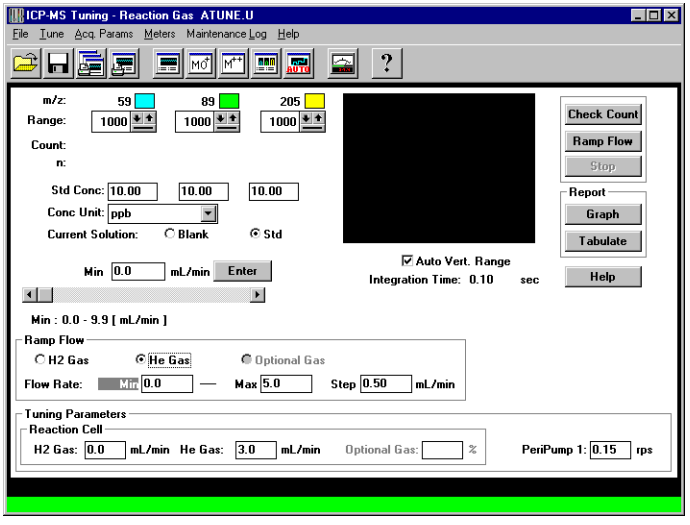
2 Adjustment of He gas flow rate

The higher the flow of He gas, the lower the intensity of the sample and background. Adjust the flow in order to make the background low enough and to get a necessary level of sensitivity. To optimize the flow of He use the **ICP-MS Tuning-Reaction Gas** Window as follows:

1) Select **Tune>>Reaction Gas** in the **ICP-MS Tuning** Window.

   The **ICP-MS Tuning-Reaction Gas** Window will appear.

   In this window, the data of the tuning and blank solutions are collected as the gas flow rates change. The **Graph** button will produce a graph of the sensitivity and BEC (Background Equivalent Concentration). This graph will help to determine the flow rate of He gas.



Reaction Gas Tuning Window

2) Aspirate the tuning solution.

3) Set the mass number and integration time by selecting Acq.Params.

4) Input the concentration of each mass number of the tuning solution in the **Std Conc** Text Box and select the concentration unit in **Conc Unit**.

5) Select **Std** (standard solution) in **Current Solution**.

6) Select **He Gas** in the **Ramp Flow** group and input the minimum value, maximum value, and the step size of the He gas flow rate to adjust the **Flow Rate**.

7) Display the signals by clicking the **Check Count** button and confirm that the signal is stable. At this time, the flow rate of He gas doesn't change. Click the **Stop** button after confirming signal stability.

8) Click the **Ramp Flow** button. Sensitivity data is collected by changing the He gas within the flow rate range that was previously set. A message will be displayed on the message line after the data collection is complete.

9) When the previous step has finished, aspirate fresh DI water or nitric acid solution (blank solution) whose concentration is the same as the tuning solution.

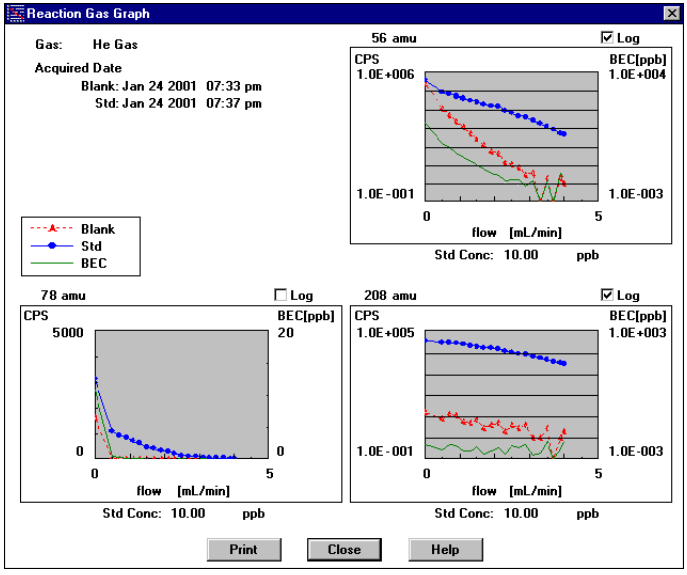
10) Select **Blank** (blank solution) in the **Current Solution**.

11) Display the signal by clicking the **Check Count** button and confirm that the signal is at a normal background level after replacing the solution. If it's not low enough, wait until it falls. At this time, the flow rate of He gas doesn't change. Click the **Stop** button after confirming an accepted background level.

12) Click the **Ramp Flow** button. The background data is collected by adjusting the He gas within the flow rate range that was previously set. A message will be displayed on the message line once the data collection is complete.

13) After collecting data from both the tuning and blank solutions, click the **Graph** button to view the result.

14) The **Reaction Gas Graph** Dialog Box will appear. In the dialog box, the sensitivity and BEC (Background Equivalent Concentration) of the tuning solution and blank solution, for the different flow rates of He gas, are plotted for each mass measured. Examine this graph and determine the best sensitivity and flow rate of He gas while satisfying the BEC.



Reaction Gas Graph Dialog box

NOTE The **Reaction Gas Report** will appear when you click the **Tabulate** button in the **ICP-MS Tuning-Reaction Gas** Window.

15) Click the **Close** button, close the **Reaction Gas Graph** Dialog Box, and return to the **ICP-MS Tuning-Reaction Gas** Window.

16) Input the flow rate of He gas determined in step 14) in the **He Gas** Text Box in the **Tuning Parameters**. The optimization of He gas is now complete.

NOTE The blank solution can be measured before the tuning solution.

NOTE Previous data will be overwritten once the **Ramp Flow** starts. To keep the previous data, print the graph and save the table beforehand.

NOTE He mode is effective to decrease polyatomic ions produced from a sample matrix such as ClO, NaAr, and ArCl.

NOTE It is good practice for the tuning parameter to be saved under a different file name from a standard mode or H_2 mode tune file.

NOTE Once the parameter of the reaction gas flow rate is tuned; only a fine adjustment of the flow rate is needed for daily tuning.

Cool Mode (When Reaction Gas is Not Used)

In cool mode, tuning is executed with cool plasma condition.

a. Change the measurement masses to 7 amu (Li), 59 amu (Co), and 80 amu (background), and then introduce tuning solution.

b. Enter the typical value from the table on the next page for each parameter. When changing the RF Power, decrease the power gradually (for example 100 W increments). If you changing the RF Power suddenly, the plasma may turn off. If the RF Reflection Power increases, re-adjust RF Matching.

c. Adjust the Torch-H,V to maximize the sensitivity of 59 amu (Co).

d. Maximize the sensitivity of 59 amu (Co) by adjusting Extract 2, Omega Lens-cs, QP Focus, OctP RF.

e. Make sure the background (80 amu) is low. The typical value is 50 counts at 0.1 second Integration time. If the count is too high, increase the Makeup Gas and then repeat step d.

f. If the sensitivity is not enough, adjust the parameters as follows;

- Adjust Extract 1 and Extract 2
- Adjust Omega Lens-cs and Omega Bias-cs
- Decrease QP Bias by 1 V (If the voltage becomes lower, sensitivity will go up, but the background will also be high.)

g. (Background Check) Introduce fresh DI water and measure 56 amu instead of 59 amu, and make sure the background (56 amu) is low enough.

NOTE Environmental elements Fe and K can easily contaminate sample solutions, even in clean room conditions. You should always use fresh DI water.

Typical values of Tuning Parameters in the Cool Mode
(with ShieldTorch, Micro Flow Nebulizer, self-aspiration)

Parameter	Typical Value	Adjustment
RF Power (W)	600	Normally use 600
Sampling Depth (mm)	18	Normally use 18
Carrier Gas (L/min)	0.7	Normally use 0.7
Makeup Gas (L/min)	0.75	0.6 to 1.2
PeriPump 1 (rps)	0.1	
S/C temp (°C)	2	Normally use at 2°C
Extract 1 (V)	-120	-200 to -60
Extract 2 (V)	-6	-30 to 5
Omega Bias-cs (V)	-80	-120 to -40
Omega Lens-cs (V)	8	5 to 10
Cell Entrance (V)	-30	Normally use -30
QP Focus (V)	-4	-10 to 5
Cell Exit (V)	-50	Normally use -50
OctP RF (V)	140	100 to 200
OctPBias (V)	-18	-30 to -10
QP Bias (V)	-5	-6 to -3

Typical values of Sensitivity (Cool Mode)

Mass	Count/1 ppb Integration time = 0.1 sec.	RSD
^{59}Co	> 2000	< 15%

NOTE

When the plasma is 600 W, the plasma may turn off when the tune file is loaded during the transition between Standby to Analysis mode. If this happens increase the RF power in the tune screen to 800 W and then re try plasma ignition

It is also possible to tune in cool mode by using Autotune (Page 4-102).

NOTE It is recommended that the 2 tuning items, "EM" and "Resolution / Axis", are executed in standard mode. Autotune is executed with higher sensitivity in standard mode. In cool mode, execute Autotune without "EM" and "Resolution / Axis".

Reducing Oxide Ions

The reason for tuning to reduce Oxide ions is the same as other Agilent 7500 series. For details, refer to "Reducing Oxide Ions" on page 4-12.

The typical value of oxide ions is less than 10% in standard mode.

Reducing Doubly Charged Ions

The reason tuning to reduce Doubly Charged ions is the same as other Agilent 7500 series. For details, refer to "Reducing Doubly Charged Ions" on page 4-15.

The typical value of doubly charged ions is less than 10% in standard mode.

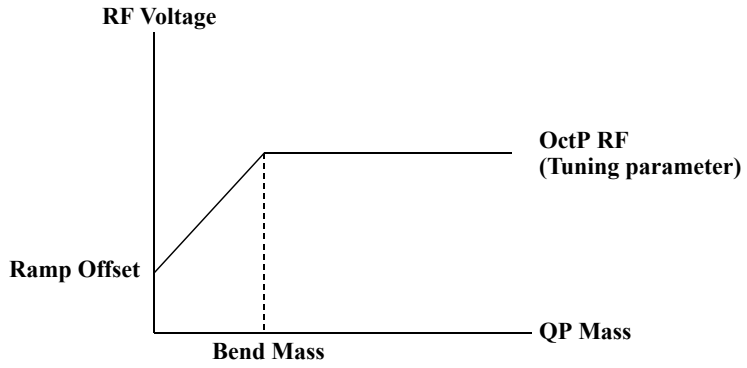
Scan of the Octopole

Generally, the RF voltage of the Octopole is fixed at a certain value, this has no effect for selecting the mass of an element to be measured. However, when an element of low mass is measured with higher sensitivity, the RF voltage of the Octopole can be scanned by working with the Q-pole Scan. Be careful about the stability of signals because the scan of Octopole may increase the factors to make the instrument unstable.

Scan line of RF voltage

Scan of the Octopole RF voltage will have a scan line as shown in the following figure. The RF voltage will rise in proportion to the mass on the low mass side the voltage will be constant on masses number larger than the bend mass number.

If the Octopole is not scanned, the voltage will be constant (OctP RF) on all the mass numbers.



Scan line of Octopole

Three parameters are used to determine this scan line:

- Ramp Offset: The Offset voltage of the RF voltage in the mass portion area.
- Bend Mass: The Mass number moving from the mass portion area to the constant voltage area.
- OctP RF: The RF voltage in the constant mass area.

OctP RF can be set in tuning parameters in the **ICP-MS Tuning** Window.

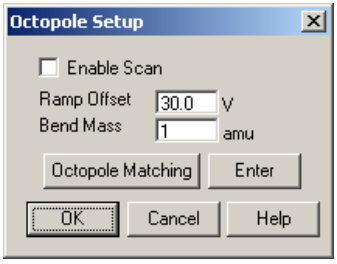
To enable the scan and set the two remaining parameters you use the **Octopole Setup** Dialog Box.

To set these parameters, carry out the following steps:

1 **Select *Instrument>>Instrument Control* in the *ICP-MS Top* Window, and display the *ICP-MS Instrument Control* Window**

2 **Select *Maintenance>>Octopole* in the *Instrument Control* Window.**

The **Octopole Setup** Dialog Box will appear.



Octopole Setup Dialog Box

3 Set each parameter necessary to scan the Octopole

- **Enable Scan:** To scan, input a check mark in this check box. If there is no check mark in the check box, Octopole will not be scanned. Usually, leave the check box blank.
- **Ramp Offset:** Input the offset voltage of the RF voltage in the mass portion area. The range is 30V to 200V. Input a value lower than the OctP RF in the tuning parameter.
- **Bend Mass:** Input the mass number moving from the mass portion area to the constant voltage area. The range is 1 amu to 260amu. The Mass number input in the bend mass will be included in the constant voltage area. To make all the mass range the mass portion area, input 260 amu.

4 Click *OK* after finishing the parameter setting.

The setting is complete and the **Octopole Setup** Dialog Box closes.

Tuning for Resolution and Mass Axis

Resolution and mass axis tuning is important because when the Agilent 7500 acquires data, it will scan each mass according to the tuning parameters. To tune the resolution and mass axis, you use the recommended tuning solution of 10 ppb of Li, Y, and Tl to ensure acceptable responses over a wide range of masses.

NOTE It is also possible to tune for resolution and mass axis by using "Autotune" on page 4-102.

To tune for resolution and mass axis, complete the following steps:

1 **Select *Top>>Instrument*.**

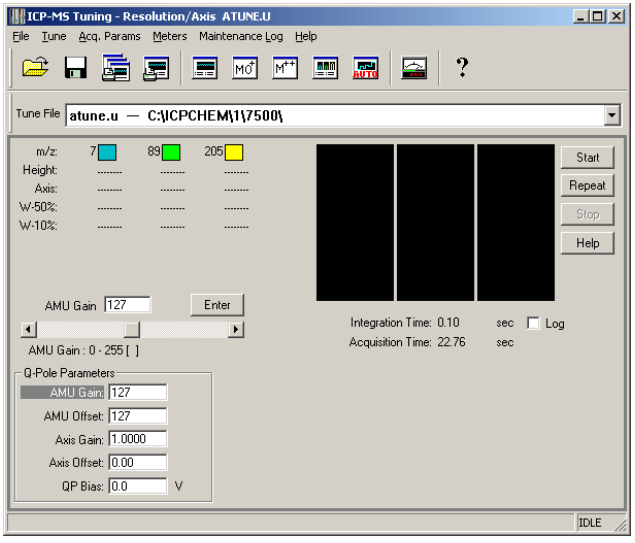
The Instrument menu appears.

2 **Select *Instrument>>Tune*.**

The ***Tuning*** window appears showing the real-time display screen for sensitivity.

3 **Select *Tune>>Resolution/Axis*.**

The ***Resolution/Axis*** window appears.



ICP-MS Tuning-Resolution/Axis Window

The window includes three boxes. Each box is 3 AMU wide and shows the peak shape for one mass.

4 Click *Start* to monitor the Agilent 7500 signals and view the spectra and numerical values in the real-time display.

Three spectra will be displayed one in each box. The spectra are obtained by 20 points per mass, three masses per window, for 100 scans. To adjust the resolution and mass calibration in a wide range of mass scale, three masses should be selected from lighter mass to heavier mass, 0.1 or 0.05 seconds integration time is appropriate. Using the standard tuning solution, ^{7}Li, ^{89}Y, and ^{205}Tl.

ChemStation inserts numerical values for the following items when the real-time display starts:

- Height

  Shows intensity of the peak top in each box.

- Axis

  Shows mass axis of the peak top in each box. Mass axis should be within ± 0.05 of the selected mass.

- W-50%

  Shows peak width (AMU) at 50 percent of the peak height.

- W-10%

  Shows peak width (AMU) at 10 percent of the peak height. W-10% should be 0.65-0.8 AMU.

5 Adjust the following parameters manually if necessary.

Click each text box and use the arrows to change the parameter value. Adjust the tuning parameters until the resolution and mass calibration are within targeted value.

- AMU Gain

  Adjusts peak width. The higher the value, narrower peak widths for heavier masses.

- AMU Offset

  Adjusts peak width. The higher the value, narrower peak widths for all masses.

- Axis Gain

  Adjusts mass calibration. The higher the value, shifts the peak position of heavier

mass toward higher mass.

- Axis Offset

  Adjusts mass calibration. The higher the value, shifts the peak position of all masses toward higher mass.

- QP Bias

  Controls the speed of ions as they pass through the Q-pole. A typical value is 0 V without the ShieldTorch system. It may be used up to 6 V when the peak shape and resolution are not good. This voltage should be the same as the Plate Bias to avoid higher background. A typical value with the ShieldTorch installed will be -10 to 0 V.

Tuning the Detector

An electron multiplier (EM) is used as the detector. There are 2 modes for detecting signals and it is possible to measure samples with concentrations ranging from sub ppt up to hundreds of ppm.

- **Pulse counting mode**

  Pulse mode is selected automatically when the counts are lower than 1Mcps. If using the normal torch, this mode covers the range from sub ppt to 100 ppb.

- **Analog mode**

  Analog mode is selected automatically when the range of the counts are from approximately 1Mcps to the corresponding signal of 4Gcps. If using the normal torch, this mode covers the range from 100 ppb to hundreds of ppm.

When data acquisition starts, pulse mode at low concentration range and analog mode at high concentration range are automatically switched. When the signal is too high, the mass number is skipped to protect the detector.

Each EM parameter is adjusted in autotune, you must not adjust the voltages manually. This will effect the gain and correlation between pulse and analog counts.

Discriminator

The discriminator is a threshold for determining the difference between general electrical noise from power supplies etc. and analyte signal. If it is set too low, the noise becomes high. If it is set too high, it cuts not only the noise, but also decreases the sensitivity. Only use autotune to adjust the discriminator.

EM Voltage (Analog HV, Pulse HV)

The ions are converted into secondary electrons and the output of the signal is an amplification of these electrons. A higher EM voltage gives higher sensitivity, but there is a possibility that the EM deteriorates faster due to excessive signals. Only use autotune to adjust the EM voltage (do not adjust manually).

NOTE Tuning of the EM voltage will be required frequently when you analyze samples with a wide concentration range where both the pulse and analog modes are used., it is required every day when higher signals are analyzed for a long time the EM deteriorates faster.

NOTE You must do the P/A factor adjustment after tuning the EM voltage. For information about P/A factor settings, see "Setting P/A Factors".

Prevention of EM deterioration

When higher signals are analyzed for a long time with very high sensitivity, the EM may deteriorate fast. EM deterioration causes drift of the signal and the lifetime of the EM may be shortened. The following methods will minimize EM deterioration:

- **Set suitable integration time**

  Shorten the integration time according to the suitable setting required for the production of reproducible data. The following table shows the recommended value of integration time for the prevention of EM deterioration. Please refer to it and set the integration time for each mass.

Recommended Value for Integration Time

The Standard of concentration *	Counts	Integration Time per Point	Detector Mode
< a few 10 ppb	0 to 500Kcps	> 0.1sec	Pulse
10 ppb to approx 100ppb	500kcps to 1Mcps	0.01sec	Pulse
> approx 100ppb	1Mcps to 4Gcps	0.01sec	Analog

* the sensitivity is 10,000 cps/ppb

  These values are only for reference, so please set longer integration times for your application, if necessary. For more information regarding the settings for integration time, please refer to Chapter 5, "Creating a method" and the Application Handbook.

- Decrease the intensity

  When the concentration of signals that you want to analyze is large, high sensitivity is not required. It is recommended to decrease the sensitivity. If you want to decrease the intensity, adjust the ion lens voltage (not the EM voltage). To adjust the ion lens voltage, see "Tuning for Sensitivity" in this chapter.

Setting P/A Factors

ChemStation automatically switches between pulse and analog mode. For linear calibration curves, these two modes should be adjusted by using ***P/A Factor*** tuning.

Usually a standard solution that includes all analytes in your sample and will be used as one of the standard solutions to make calibration curves is used for tuning P/A Factors. If you don't need accurate quantitative data, you can use a solution of Li, Co, Y and Tl as the P/A Factor tuning solution. The counts of each element must range from 400,000 to 4,000,000 cps to get accurate P/A Factors (when using normal torch, the concentrations is normally around 100 ng/mL (ppb)). Prior to performing P/A Factor tuning, EM tuning should be performed using Autotune.

NOTE The P/A Factor adjustment must be performed after EM tuning. At this time, cancel the check of the ***Merge in the current data*** box in the P/A Factor Tuning dialog box.

NOTE The P/A factor adjustment must be performed everyday to get accurate results in a sample with a wide concentration range where both the pulse and analog mode are used.

For P/A Factor tuning, complete the following steps:

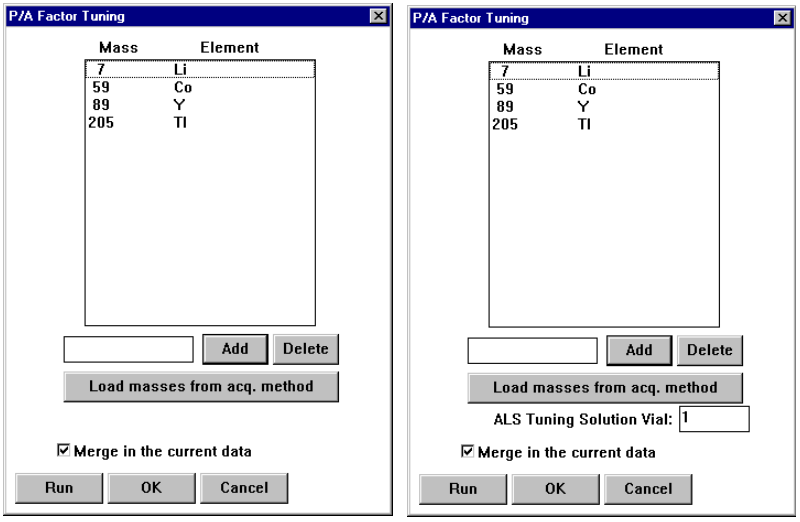
1 **Aspirate tuning solution.**

2 **Select *Tune>>P/A Factor.***

The ***P/A Factor Tuning*** dialog box appears.

3 **Set the mass number and/or elements to be used for tuning.**

The mass number and/or element can also be loaded from the current method by clicking ***Load masses from Acq. method***. Having a method specifically for P/A Factor tuning is recommended. For information about creating a method, see Chapter 5, "Creating a Method".



Without Autosampler **With Autosampler**

P/A Factor Tuning Dialog Box

4 **Cancel check of the *Merge in the current data* box, if necessary.**

The ***Merge in the current data*** box is disabled when adjusting the P/A factor for the first time. It is usually checked for the following times. Cancel the check when the EM adjustment has been carried out.

5 **Enter the vial number of the autosampler in the ALS Tuning Solution Vial.**

When the autosampler is set in the Configuration task, this box appears. Enter the vial number of the tuning solution. When "0" is entered, the tuning begins without

moving the sample probe of the autosampler. Uptake time and Stabilization time of the solution can be shortened when the solution washout time is sufficient.

NOTE When "0" is entered in the ALS tuning solution vial box, the sample probe **does not move to the Rinse Port** after the tuning.

6 Click *Run*.

P/A factor tuning starts. After tuning, the P/A Factor Tune Report appears.

7 The message is displayed.

When ***Yes*** is clicked, the set P/A factor is saved as the current tuning parameter.

When ***No*** is clicked, the set P/A factor is not saved as the current tuning parameter.

NOTE When the counts of an element are not within the range of 400,000 to 4,000,000 cps, the P/A factor of that mass will not be set correctly and the P/A factor of the P/A Factor Report may be reported as follows:
- EM error: need to reduce the sensitivity
(The sensitivity is higher than EM Protection)
- Sensitivity too low: need to increase the sensitivity
- Sensitivity too high: need to decrease the sensitivity

If the P/A factor is used as it is, the P/A factor of that mass is derived by a linear interpolation between the P/A factors of the two adjacent masses.

NOTE If the P/A factor of several elements have error values, please make sure of the following:

•The signal of the element is within 400,000 to 4,000,000 cps.
•***Only*** select the mass of the major isotope (the element may have more than one isotope).

When the signal is too high, decrease the sensitivity by adjusting the voltage of the ion lenses or by using a different concentration for the tuning solution. Tune the P/A factor again. If the voltage of the ion lenses is changed, please restore these voltages after P/A factor tuning (the P/A factor is not affected by changing these voltages).

NOTE It is also possible to continue the P/A factor tuning until there is no "-1" in the P/A Factor Report. If you select ***Merge in the current data*** and tune the P/A factor again, the previous P/A factor is adopted even if the mass gives an error this time. When

there is no error in the P/A factor, it is updated to the latest P/A factor.

Cancel the check of the ***Merge in the current data*** box when adjusting a new P/A factor.

NOTE When the tuning task is completed, the ***Merge in the current data*** box is disabled and the previous value of the P/A factor is not used. Do not finish the tuning task till the P/A adjustment is completed.

NOTE When the P/A factor's error still appears from tuning using a low concentration solution, it may be caused by a high background due to a polyatomic ion, etc. In this case, this element will be analyzed in analog mode. Remove this element from the ***P/A Factor tuning*** dialog box.

Generating a Tune Report

You can generate a tune report to keep a record of tuning results and tuning parameter values, which are lost when you exit the Tune window. The tune report provides data about Sensitivity, Resolution/Axis numerically and graphically, and all tuning parameters. It also gives the name of the tune file used for the tuning session. See the next section for more information about tune files.

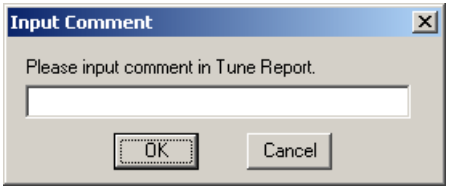
To generate a tune report, complete the following steps from a real-time display screen:

1 **If measuring sensitivity or resolution, click *Stop* in the *Tuning* window.**

The real-time display is stopped.

2 **Select *File>>Generate Report*.**

The ***Input Comment*** dialog box will appear..



Input Comment Dialog Box

3 **Enter comments on tuning reports and click *OK*.**

The results you had on the screen will disappear and the tune program will generate a new graphics display. ChemStation generates the report to your printer, the report will show Sensitivity data, Resolution/Axis data, and the current lens element settings. The acquire date and print date are also stamped.

NOTE

If you want to print out the current display, simply click on the ***Stop*** button and select ***File>>Print***.

If the ***Record log for each tuning report*** check box is checked on the ***Detail Setting*** dialog box, opened from the ***ICP-MS Configuration*** dialog box, the tuning results will be automatically recorded in the maintenance log.

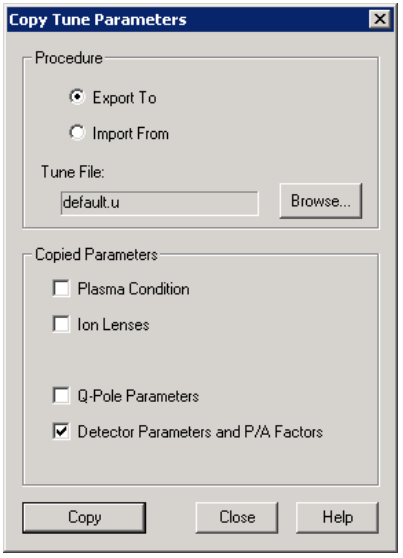
Copying a Tune Parameters

You can use the **Copy Tune Parameter** dialog box to copy the tune parameters from the current file to another tune file or from another tune file to current setting.

To copy the tune parameters, complete the following steps:

1 **Select *File>>Copy Tune Parameters*.**

The Copy Tune Parameters dialog box appears.



Copy Tune Parameters Dialog Box

2 **Set the parameters as follows:**

1) In the **Procedure** section, select the direction of copy and select the file where to copy to or where to copy from.

2) In the **Copied Parameters** section, select the parameters you want to copy.

3 **Click *Copy* to copy the parameters.**

4 **After coping, click *Close*.**

Creating and Using a Tune File

ChemStation automatically saves the tuning parameters to a tuning file. Exiting the Tune window overwrites the most recent tune values to the default tune file, atune.u. However, you may want to save a set of parameters to use as a starting point for future tuning procedures, especially if you are tuning the instrument for a special type of analysis. To do so, you create a separate tune file.

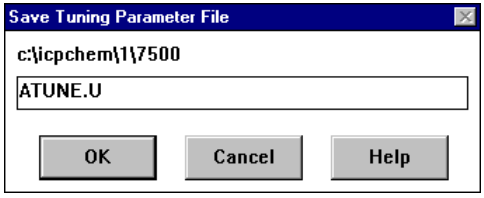
The following sections explain how to save and load tuning parameters.

Saving the Tuning File

To create a tune file, save the tuning parameters by completing the following steps from the Tune window:

1 Select *File>>Save Tune Values*.

The ***Save Tuning Parameter File*** dialog box appears.



Save Tuning Parameter File Dialog Box

2 Type a name for the tune file you want to save.

The name can be up to eight characters long. Do not use the following characters in the file name:

Period (.)Slash (/)Brackets ([])

Comma (,)Backslash (\)Vertical bar (|)

Semicolon (;)Equal sign (=)Space ( )

Colon (:)Quotation mark (“)

The file will have a.u extension, which is automatically added by the software to indicate that it is a tune file.

3 Click *OK* to save the file.

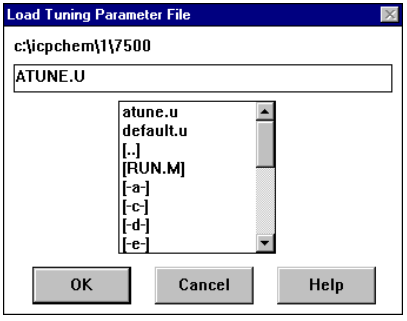
ChemStation automatically saves the file in the directory c:\icpchem\1\7500.

Loading a Tune File

To use a saved tune file for a tuning procedure, load the file by completing the following steps in the Tune window:

1 Select *File>>Load Tune Values*.

The ***Load Tuning Parameter File*** dialog box appears.



Load Tuning Parameter File Dialog Box

2 Select the tune file in one of the following ways:

- Type the name of the tune file and click ***OK***.
- Double-click a tune file in the displayed list.
- Click a tune file in the displayed list and click ***OK***.

Agilent 7500 ChemStation will immediately change tuning parameters according to the selected tune file.

NOTE If you change parameter values during the tuning procedure and want to save the new values, save the tune file again. Otherwise, the changes are saved in atune.u only. When you save, you can either overwrite or make a new tune file by renaming it.

Autotune

Autotune automates the optimization of various tunable lens and voltage settings to optimize the instrument. In Autotune it is possible to change the targeted sensitivity along with the range for each tuning parameter.

NOTE RF matching, Optional Gas (in case the optional gas line is installed), PeriPump, S/C temp, and QP Bias are not optimized with ***Autotune***. Optimize these parameters manually before ***Autotune***.

NOTE To adjust the RF Matching, use of the automatic adjustment function is recommended. To use the automatic adjustment function, select **Tune>>RF Matching** on the Tuning window, and then follow the displayed dialog box.

NOTE During ***Autotune***, do not use other software functions.

NOTE If you use an autosampler, set ***Autosampler*** in the ***Configuration*** task. For more information about the configuration task, see Chapter 2, "Configuration". When you set the configuration for an autosampler, you will need to turn on the autosampler before running ***Autotune***.

To perform Autotune, complete the following processes:

1 **Introduce a tuning solution.**

The standard tuning solution containing 10 ppb of Li, Y, Ce, Tl is recommended when you use the standard torch system.

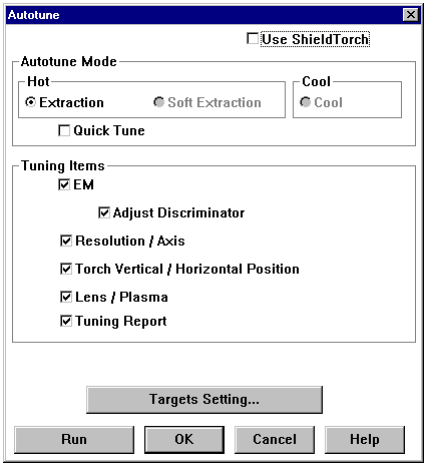
2 **Click *Start* in ICP-MS Tuning-Sensitivity Window and check that the signal is output.**

NOTE

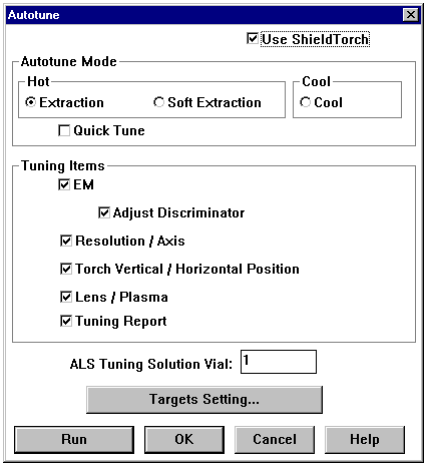
In order to start ***Autotune,*** the sensitivity and the RSD in the ***Sensitivity Tuning*** should be higher or better than 5,000 cps and 10%. If the sensitivity is less than 5,000 cps, adjust the tuning parameters to achieve a sensitivity of 5,000 cps. If the sensitivity is extremely low or the signal is not stable, autotune will not run correctly.

3 **Select *Tune>>Autotune*.**

The ***Autotune*** dialog box appears.



Without Autosampler



With Autosampler

Autotune Dialog box

4 **When using the standard torch, cancel the *Use ShieldTorch* box.**

When using the ShieldTorch, select the ***Use ShieldTorch*** box.

5 **Select the Autotune Mode from the Autotune Mode area.**

- Extraction

  Select when using ShieldTorch or when running hot plasma without the ShieldTorch. When selected, Extract 1 lens is set to a negative value for tuning.

- Soft Extraction

  This is the autotune mode for reducing background when using the Shield-Torch. In this setting Extract 1 lens is set to a positive value during tune.

- Cool

  This is the autotune mode for cool plasma.

NOTE The Soft Extraction mode and Cool mode become available when the ***Use ShieldTorch*** box is selected. See the "Option Instruction Manual" for the details of tuning when using the ShieldTorch.

NOTE Quick Tune is useful when there are few changes in the tuning parameters and a complete autotune is not needed. Quick Tune is not recommended when changing the autotune mode. (The time taken for autotune when Quick Tune is selected can be shortened up to 1/3 to 2/3, as compared to the usual mode.)

6 Select Tuning Item from the Item area.

- EM

  When selected, autotune automatically optimizes the detector (EM) voltage. In addition if ***Adjust Discriminator*** is selected, Autotune will optimize both the EM and Discriminator.

  Adjusted parameters:

  Analog HV, Pulse HV,
  Discriminator (when Adjust Discriminator is selected)

- Resolution/Axis

  When selected, autotune automatically adjusts the mass axis within a range of +/- 0.05 amu and a resolution at 10% of peak height to between 0.65-0.80 amu.

  Adjusted parameters:

  AMU Gain, AMU Offset, Axis Gain, Axis Offset

- Torch Vertical/Horizontal Position

When selected, autotune automatically optimizes the torch vertical and horizontal position.

Adjusted parameters:

Torch-H, Torch-V

- Lens/Plasma

When selected, autotune automatically optimizes the ion lens parameters in accordance with the target values.

Adjusted parameters:

Extraction Lenses, Einzel Lenses, Omega Lenses, QP Focus, Plate Bias, RF Power, Smpl Depth, Carrier Gas, Makeup Gas

- Tuning Report

Check this box to generate a tuning report after autotune is completed.

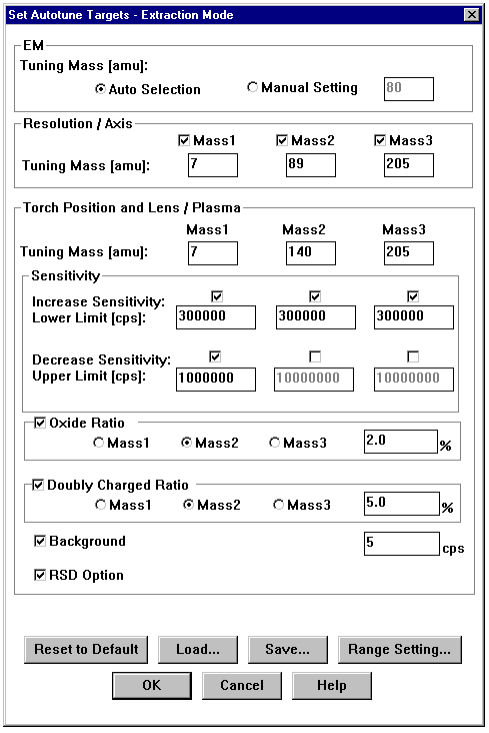
If the ***Record log for each tuning report*** check box is checked on the ***Detail Setting*** dialog box, opened from the ***ICP-MS Configuration*** dialog box, the tuning results will be automatically recorded in the maintenance log.

7 Enter the vial number of the tuning solution in the ALS Tuning Solution Vial text box.

When the autosampler is set in the Configuration task, this text box appears. When "0" is entered, the tuning starts without moving the sample probe of the autosampler.

8 Set the target values for tuning, if necessary.

When you click ***Target Setting***, the ***Set Autotune Targets*** dialog box is displayed. The ***Set Autotune Targets*** dialog box allows the selection of target values used by autotune.



Set Autotune Targets Dialog Box

- EM

  Set the mass number that will be used for gain adjustment in the detector (EM) tuning. When ***Auto Selection*** is selected, it automatically selects the mass used in the detector (EM) tuning. ***Auto Selection*** is recommended. When the EM tuning is needed during sample analysis, select ***Manual Setting*** and type the mass number. For EM tuning, select the mass number that is adjacent to the target mass number. It must range from 500,000 to 1,000,000 cps.

- Resolution/Axis

  A maximum of 3 masses used for resolution/mass axis tuning can be set.

- Torch Position and Lens/Plasma

  A maximum of 3 masses used for torch position and lens/plasma tuning can be

selected.

- Sensitivity

  For the lower limit, select the ***Increase Sensitivity*** box and type the target value of the lower limit. For the upper limit, select the ***Decrease Sensitivity*** box and type the target value of the upper limit. (e.g. reducing the ArO(56amu) with the cool mode) If you want to set the sensitivity within a certain range, select both ***Increase Sensitivity*** and ***Decrease Sensitivity***, then set the respective target values.

- Oxide Ratio

  Set the target value, which becomes the upper limit of the oxide ion ratio. Select the element, which becomes the base of the oxide generation from the mass set in **Torch Position** and **Lens/Plasma**.

- Doubly Charged Ratio

  Set the target value, which becomes the upper limit of the doubly charged ion ratio. Select the element, which becomes the base of the doubly charged ion generation from the mass, set in **Torch Position** and **Lens/Plasma**.

- Background

  Set the target value for the upper limit of the background signal that appears even though the mass filter (Q-pole) stops the ion transmission.

NOTE Oxide Ratio, Doubly Charged Ratio, and Background cannot be selected in the Cool mode.

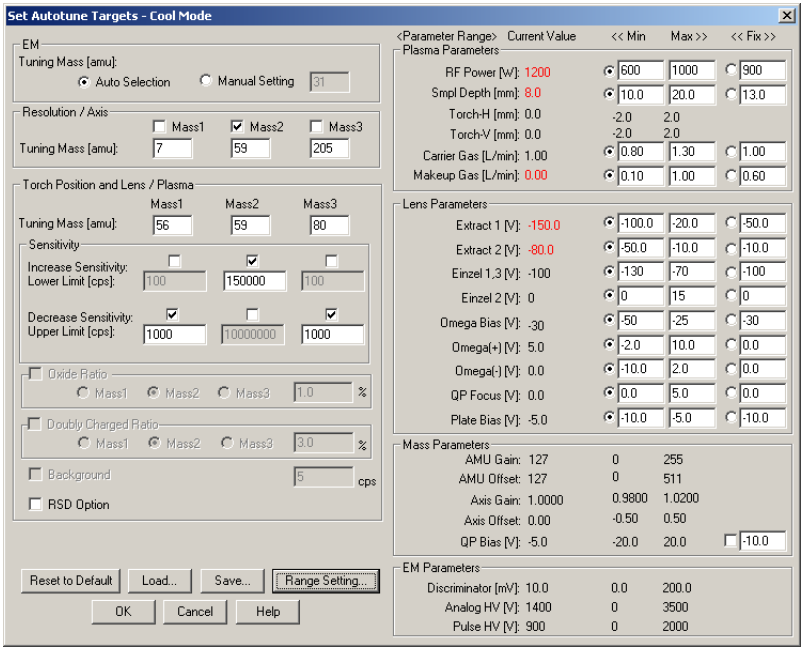
- RSD Option

  When you select the ***RSD Option***, Autotune will tune the instrument while trying to minimize the RSDs.

NOTE The control method of the peripump or the sample introduction system has a large effect on the RSD. When a sufficient performance can not be obtained, please check them.

NOTE Please ensure that the target values, which are set here, do not exceed the performance capabilities of the system. In case of setting a value, which exceeds the performance at times, it may not be possible to obtain sufficient performance.

When you click the ***Range Setting***, the Parameter Range appears at the right side of the ***Set Autotune Target*** dialog box. In autotune it is possible to set a range for each variable tuning parameter value.



The Set Autotune Targets Dialog Box

Each parameter has been assigned into one of 4 categories: ***Plasma Parameters, Lens parameters, Mass Parameters,*** and ***EM Parameters***. There are variable ranges or fixed values parameters which can be set for the categories.

In the ***Plasma Parameters*** and ***Lens Parameters***, it is possible to select the variable/fixed parameter (the variable range in the case of selecting variable and the fixed value in the case of selecting fixed). When the present value is shifted from the variable range, the present value will be displayed in red.

NOTE It is possible to carry out tuning effectively by providing an appropriate fixed value.

NOTE If you click ***Reset to Default***, The factory default values are reset.

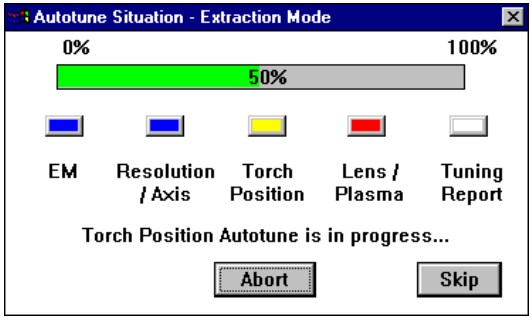
On completion of the target value setting, click ***OK*** to return to the ***Autotune*** dialog

box.

NOTE Autotune tuning parameter settings can also be saved as an Autotune Target File. For the details, see the chapter "Creating and Using a Autotune Target File".

9 Click *Run* in the *Autotune* dialog box.

The selected items in tune will be executed in the Autotune Mode. During autotune, ***Autotune Situation*** dialog box will be displayed.



The Autotune Situation Dialog Box

The status of each tuning item is displayed in color. The colors have the following meaning:

- Gray

  This tuning item was not selected in the Autotune dialog box.

- White

  This item was selected in the Autotune dialog box, but has not been executed yet.

- Yellow

  Auto tuning for this item is currently being executed. The progress of tuning is being displayed in the status bar.

- Red

  Autotune was unable to tune this item within set targets. In some cases Autotune may executed this item again after the optimization of other parameters.

- Blue

  Tuning is complete (shows that it has been executed as per target).

The Status Bar shows a graphical representation of the progress of each item as

tuning progresses. The current tuning item will have a yellow colored status lamp indicating that it is the item being optimized. The Status Bar is meant to give a rough indication of the tuning progress.

10 On completion of the autotune, click *Close* and exit the *Autotune Situation* dialog box.

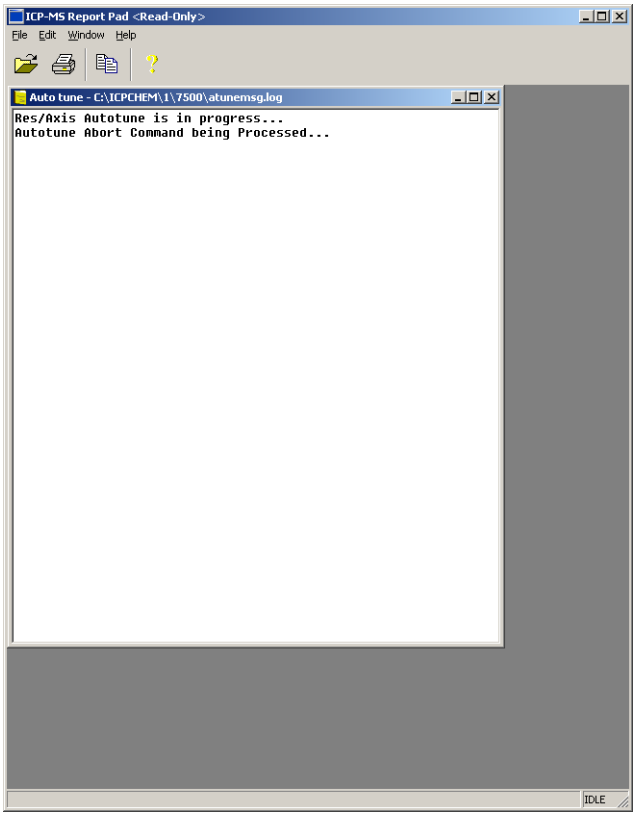
The message seeking confirmation is displayed. To save tuning parameters, click ***Yes***. Tuning parameters are saved to the tuning file **Autotune.u.**

NOTE When the peak profile is bad (peak splitting or high noise at the top of the peak), the autotune for resolution/mass axis may not successfully complete. In this case, try increasing or lowering the value of the QP Bias by several volts. Check for improved peak profiles and re-run Autotune.

NOTE The ***Skip*** button causes the executed item to be skipped and causes autotune to move to the next tune item. If the skipped item is resolution/mass axis or lens/plasma tuning, the current values are saved. If the skipped item is EM or torch position tuning, the values used are returned to their initial value.

NOTE The Tuning Report is generated when autotune is completed, and continue generating if you click ***Close*** in the ***Autotune Situation*** dialog box and close it. If you want to abort generating a report, click the Control Box Menu in the upper left corner of the ***ICP-MS Tuning*** window and click ***Abort.*** Tuning parameters are returned to their initial value (file name: Atuneorg.u). Tuning parameters after autotuning, however, are saved to the tuning file "Autotune.u". For more information about loading a tuning file, see the chapter, "Loading a Tune File".

When autotune is completed, any message displayed during the execution of autotune will be indicated on the report pad. If an error was generated during the execution of autotune, autotune will be aborted and the details of the error will be displayed.



Details of Error

Aborting Autotune

Autotune is suspended with the ***Abort*** button during the execution of autotune. A confirmation dialog is displayed that allows 3 selections for "Saving the Current Tune Status": ***Yes***, ***No*** or ***Cancel***. ***Cancel*** will continue the Autotune. ***No*** will close Autotune with no change in values and ***YES*** will save the current tune state.

NOTE

Tuning parameters will not be changed if you do not save and close the dialog box. Unchanged parameters are saved to the tuning file "Atuneorg.u".

Creating and Using a Autotune Target File

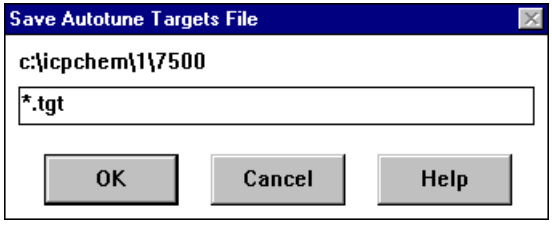
The target values set in the **Autotune Targets** dialog box can be saved as an autotune target file.

Saving the Autotune Target File

To create an Autotune Target file, save the target values by completing the following steps from the ***Set Autotune Targets*** dialog box:

1 Click *Save* in the *Set Autotune Targets* Dialog box.

The ***Save Autotune Targets File*** dialog box appears.



The Save Autotune Target File Dialog box

2 Type the name for the autotune target file you want to save.

The name can be up to eight characters long. Do not use the following characters in the file name:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (=)	Space ( )
Colon (:)	Quotation mark (")	

The file will have a.tgt extension, which is automatically added by the software to indicate that it is an autotune target file.

3 Click *OK* to save the file.

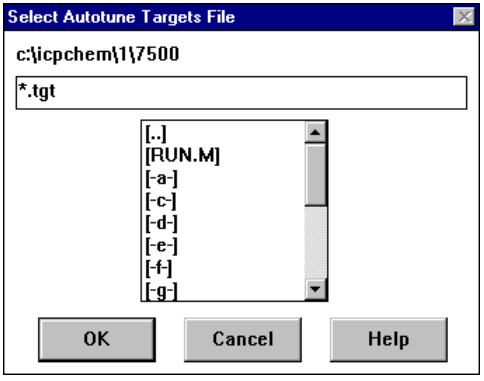
ChemStation automatically saves the file in the directory c:\icpchem\1\7500.

Loading an Autotune Target File

To use a saved Autotune Target file, load the file by completing the following steps from the ***Autotune Targets*** dialog box:

1 Click the *load* button in the Set Autotune Targets Dialog box.

The ***Select Autotune Targets File*** dialog box appears.



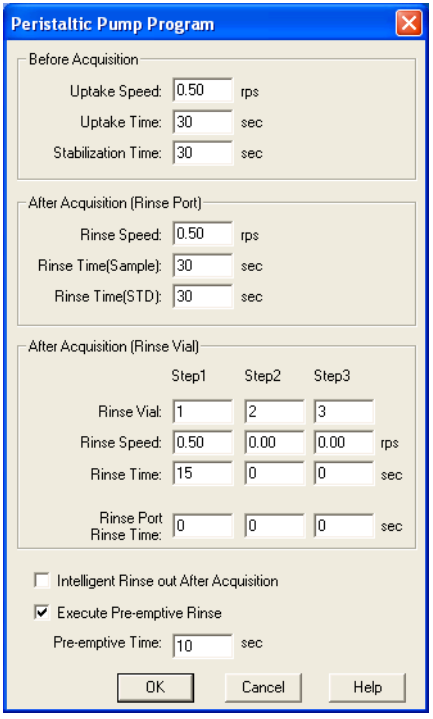
The Select Autotune Targets File Dialog Box

2 Select the autotune target file in one of the following ways:

- Type the name of the autotune target file and click *OK*.
- Double-click an Autotune Target file in the displayed list.
- Click an Autotune Target file in the displayed list and click ***OK***.

Peristaltic Pump Program for Autotune

When ***Autosampler*** is selected in the ***Configuration*** task, setting of the peristaltic pump program is required. Select ***Peri Pump Program for Autotune and the Peristaltic Pump Program*** dialog box appears. You can set the peristaltic pump speed or the stabilization time before and after ***Autotune***. For more information about peristaltic pump program, see the chapter "Setting the Peristaltic Pump Program" in Chapter 5, "Creating a Method".



The Peristaltic Pump Program Window

5

Creating a Method

Creating a Method

A method encompasses data acquisition and data analysis. Use them to define parameters for acquiring and analyzing data.

The last method used before closing ChemStation is loaded into memory, next time you access the software. The three most recently loaded methods are listed as numbered menu items, with the first entry corresponding to the most recently loaded method. Selecting any one of these numbered menu items will cause that method to be loaded. The name of the current method is displayed in the title bar at the top of the window. You can create a new method by editing any existing method and saving the changes to disk under a new name.

This chapter explains how to create a method. There are four types of acquisition modes for which you can create a method:

- Spectrum acquisition accumulates a signal for the entire data acquisition period, 100 scans. The signal displays as an accumulated intensity versus mass plot (mass spectrum) for the masses previously selected in the method. You can acquire both qualitative and quantitative data using Spectrum acquisition.
- Time Resolved Analysis acquisition (TRA) produces a transient signal that appears as an intensity versus time plot (time chart). This acquisition mode is usually used when the signal changes with time. You can monitor and display the signals obtained for each selected mass, in real time. It is useful for applications which use the Agilent 7500 combined with peripheral equipment such as the Liquid Chromatography, Laser Ablation System, etc.
- Time Program acquisition is basically the same as TRA but more powerful. You can change tuning parameters and the analyte mass automatically during TRA acquisition, allowing complex chromatographic programs to be developed.
- Isotope Analysis is similar to Spectrum analysis, but the number of scan is 1000. This allows for better precision when determining isotope ratios, for example in geological studies.

Creating a Method Using the Method Wizard

The method wizard is an easy way to create a method, even for beginners. Simply select an application and sample type, and a method suitable for the system configuration will automatically be created. With the wizard, a limited number of application and sample types are available.

A method created using the method wizard may be used immediately or edited using the ***Edit Default Method*** command. A method can also be created using the default method, rather than using the method wizard, as in the preceding model of ChemStation. For details on editing the default method, see Editing Default Method below.

The instrument types are defined as follows.

- ORS model: A model with an octopole reaction cell
  Agilent 7500ce and 7500cs
- Non-ORS model: A model without an octopole reaction cell
  Agilent 7500a

Follow the procedure described below to create a method using the method wizard.

1 **Specify the method filename.**

2 **Select a sample.**

3 **Edit the element information.**

4 **Edit the report information.**

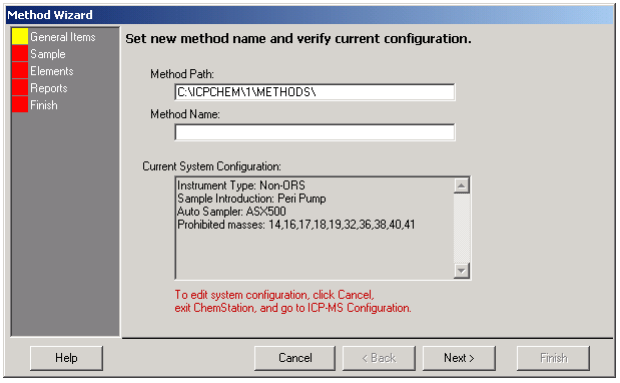
5 **Check the created method.**

Setting Method Filename

Set the name of a method file by the following procedure.

1 Select *Methods>>Run Wizard….*

The method wizard starts up. The list view appears on the left and the ***General Items*** page appears on the right.



General Items Page

The list view shows the five page names that make up the method wizard. Click on a page name, and the page appears, allowing setting of the items required for the creation of a method. The square ■ to the left of each page name indicates whether the items on the page have been set. Upon completion of setting on the page, the square is shown in green. When the page is on screen and setting is underway, the square is shown in yellow. When setting hasn't been yet performed, the square is shown in red.

NOTE The method wizard can also be run by clicking on the ***Method Wizard*** icon.

Method Wizard Icon

2 Enter a method name and check the method path and system configuration.

Enter a method name using up to 8 characters in the ***Method Name*** field. Add the extension .m. If the extension is not added, it will be automatically added to the filename. If the same filename exists in ***Method Path***, the existing file will be replaced by the new file. ***Method Path*** shows a location for the method to be saved. To change it, enter another location.
System Configuration shows the current system configuration. Be sure to set the system configuration in which you will use the method, as the method wizard creates a method based on the current system configuration. To change the settings, click on ***Cancel*** to exit ChemStation, and set the system configuration using ***ICP-MS Configuration***.

3 Click on *Next*.

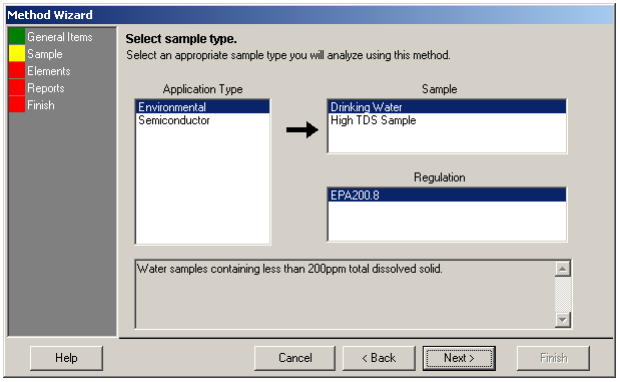
The ***Sample*** page appears.

Selecting a Sample

Select a sample on the ***Sample*** page by the procedure described below.

1 Select an application from *Application Type*.

Application Type shows only the applications supported by the current system configuration. The available options (***Sample*** and ***Regulation***) vary depending on the selected application.



Sample Page

2 Select a sample.

The available options (***Regulation*** and ***Guideline***) vary depending on the selected sample. An explanation of the sample appears at the bottom of the screen.

3 Click on *Next*.

The ***Elements*** page appears.

NOTE

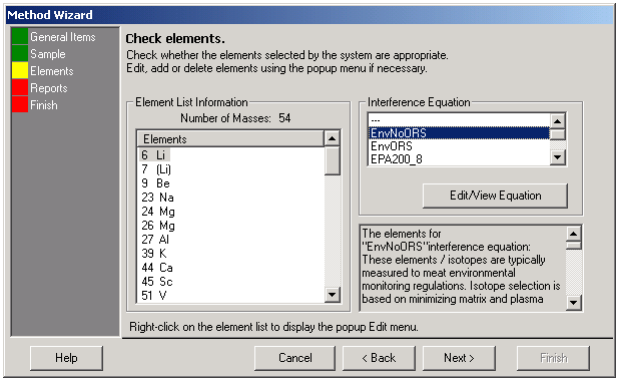
If the settings on the ***Sample*** page are re-edited after the ***Element*** or ***Reports*** page is viewed, the settings on the ***Element*** or ***Reports*** page will be reset to the default values.

Editing Element Information

Element List Information on the ***Elements*** page shows the mass numbers and names of the elements for which data is to be acquired. When the acquisition mode is set to "Multi Tune," the tune mode also appears.

In the Multi Tune mode, tune files with the same names as the tune names shown on this page shall be prepared before the method is actually used. The tune names represent the modes specified below. As the modes using a reaction gas, "HE" is helium-gas mode, "H2" is hydrogen-gas mode, "H2MOD" is modified-hydrogen-gas mode. "NOGAS" and "NORM" are modes that do not use a reaction gas. "COOL" is cool plasma and "HOT" is hot plasma. Depending on the instrument type, application, and sample, only the appropriate tune names are shown. For details on tuning, see Chapter 4, "Tuning."

Interference Equations show the library names of all interference correction equations. An explanation of the selected item appears at the bottom of the screen.

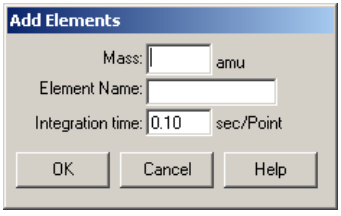


Elements Page

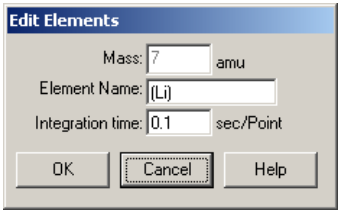
Edit the element information by the following procedure.

1 Add, edit, or delete an element.

- When adding or editing, right-click on the element in ***Element List Information***, and choose an option from the drop-down menu. Set the mass number (only when adding), the element name, and the integration time. In the Multi-Tune mode, set the tune step as well. The detector mode is set to "Auto" when an element is added, and is not changed when an element is edited.



Adding an Element

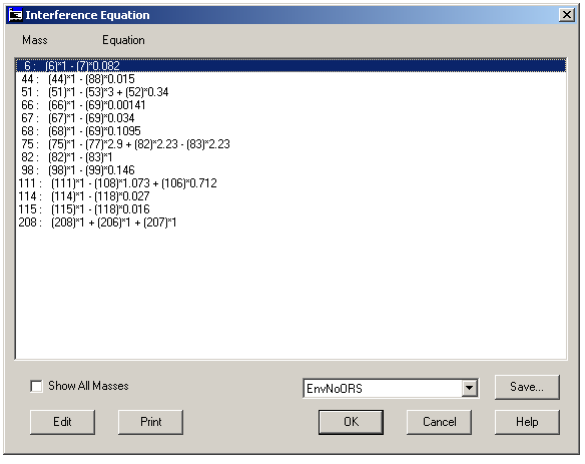


Editing an Element

- When deleting, select an element in ***Element List Information*** and right-click on it. Choose ***Delete*** on the menu.

2 View or edit an interference correction equation.

- When viewing, select the interference correction equation in ***Interference Equations*** and click on ***Edit/View Equation***.
- When selecting another equation, select the interference correction equation in ***Interference Equations***.
- When editing an equation, select the interference correction equation in ***Interference Equations*** and click ***Edit/View Equation***. For the procedure for editing interference correction equations, see "Creating and Editing Interference Correction Equations" in "Using the Default Method as a Template" below. The change will be reflected in the list box.



Interference-Equation Dialog Box

NOTE When an interference correction equation has been edited, the change is also reflected in the mass number in ***Element List Information***.

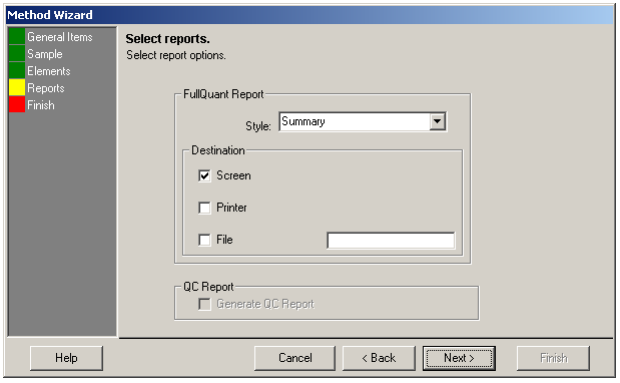
3 Click on *Next*.

The ***Reports*** page appears.

Editing Report Information

Edit the report information by the procedure described below.

1 Select a report style in *Style*.



Reports page

2 Select the destination to which the report is to be output in *Destination*.

- Screen
  Creates a report and displays it in the report pad.
- Printer
  Creates a report and outputs it to the connected printer. For a detailed report, the output destination is set to the printer only.
- File
  Creates a report and saves it as a file. Enter the filename.

3 When creating a QC report, check the *Generate QC Report* box.

The QC database must be edited after the method wizard is closed.

NOTE When the QC mode doesn't include a QC check, the ***Generate QC Report*** check box will be grayed out.

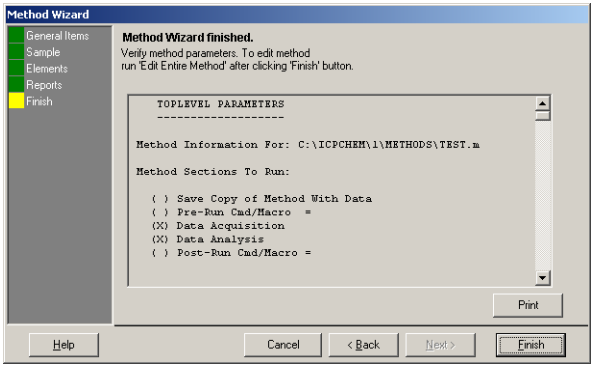
4 Click on *Next*.

The ***Finish*** page appears.

Checking the Set Method

The ***Finish*** page shows the details of the set method. Follow the procedure described below to check or edit the set method.

1 Check the details of the method and click on *Finish*.



Finish Page

The ***Finish Method Wizard*** dialog box opens.

NOTE Clicking ***Print*** allows printing of the entire method.

2 When editing the method, click on *Run 'Edit Entire Method.'* If the set method shown on the *Finish* page is OK, click on *Close*.

Finish Method Wizard Dialog Box

When ***Run 'Edit Entire Method'*** is clicked, ***Methods>>Edit Entire Method*** starts up, allowing the method to be viewed/edited. For details on ***Edit Entire Method***, see "Using the Default Method as a Template" below.

Using the Default Method as a Template

You can create a new method using the default method as a template. To create a method by editing the default method, you perform the following functions.

1 **Enter method information.**

2 **Create or edit interference correction equations.**

3 **Set the acquisition mode.**

4 **Set acquisition parameters.**

- Spectrum

  Set isotopes/elements, peak pattern, integration time, repetition

- Time Resolved Analysis

  Set isotopes/elements, peak pattern, integration time, acquisition time, real time plot, time window, repetition

- Time Program

  Set time program; set isotopes/elements, pattern, integration time, acquisition time, real time plot, time window, repetition, select tuning files

- Isotope Analysis

  Set isotopes/elements, peak pattern, integration time, repetition

- Spectrum (Multi Tune)

  Set isotopes/elements, peak pattern, integration time, repetition

- Isotope Analysis

  Set isotopes/elements, peak pattern, integration time, repetition

5 **Set the peristaltic pump program.**

6 **Specify whether background subtraction and interference corrections are to be used.**

7 **Select report types.**

8 **Select report options.**

9 **Edit calibration table, semi-Q parameters, etc. (when the sequence will run).**

10 Save the method and calibration.

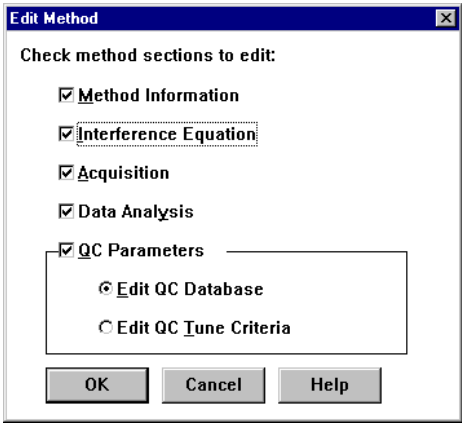
This section includes a detailed explanation of each of these functions. Unless otherwise indicated, each function applies to all four acquisition types.

Editing the Default Method

To edit the default method, check the title bar of the Top window to be sure that the default method, DEFAULT.M, is currently in memory. Then, complete the following steps:

1 Select *Methods>>Edit Entire Method*.

The ***Edit Method*** dialog box has five method sections you can edit: Method Information, Interference Equation, Acquisition, Data Analysis and QC Parameters.



Edit Method Dialog Box

NOTE If your QC configuration does not include any QC parameters, the QC Parameters section will be grayed out.

2 Click all of the check boxes to edit all method sections and then click *OK*.

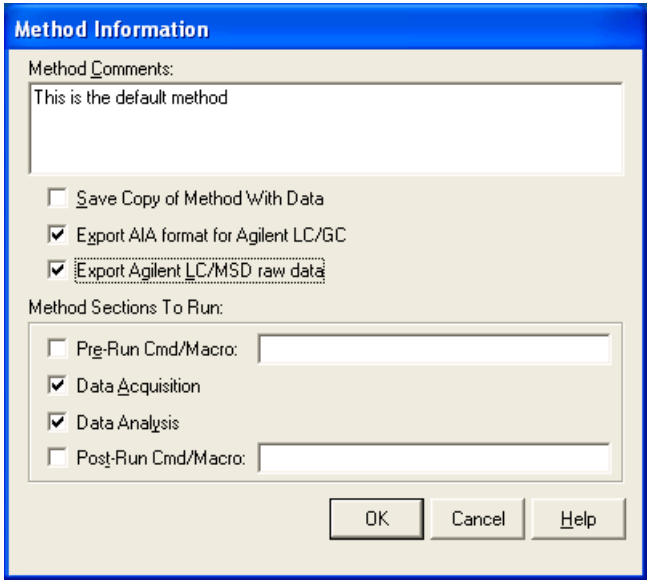
The Method Information dialog box appears. To continue creating the method, see "Entering the Method Information" in the next section.

When you are creating a new method, you may want to check all of the edit options contained in a method. However if you want to modify a method, you can edit the

necessary method sections without going through the entire method. For more information, see "Modifying a Method" in this chapter.

Entering the Method Information

The Method Information dialog box enables you to choose which method sections to run, and also to write comments about the method. If you want to save the method with the data, you can also designate it here.



Method Information Dialog Box

To enter method information, complete the following steps:

1 **Type the comments you want to save with the method in the Method Comments field.**

This field enables you to add identifying information to the method. You can write anything here.

2 **Click the *Save Copy of Method With Data* check box.**

This check box ensures the method is saved with the data files each time the method is run. This step is optional. Although this option requires more disk space, it provides a record of the conditions existing at the time of sample analysis.

3 You can select the data file output format.

- Export AIA format for Agilent LC/GC checkbox
  Selecting this checkbox will cause the data file to be saved in AIA format as well. This file is saved to the data file folder. Use the Agilent LC/GC ChemStation to import the file for display or analysis.
- Export Agilent LC/MSD raw data checkbox
  Selecting this checkbox will cause the data file to be saved as LC/MSD raw data as well. This file is saved to the data file folder. Use the Agilent LC/MSD ChemStation to load, display, or analyze the file.

4 Click the *Data Acquisition* check box.

The default method should show this check box already marked. For data acquisition to occur, you must click the check box if it is not already marked.

5 Click the *Data Analysis* check box.

Clicking this box tells ChemStation to run the data analysis section of the method following data acquisition.

NOTE

The pre-and post-run macro command check boxes enable you to execute actions before or after acquisition. Macros included with ChemStation allow automatic instrument startup and shutdown. Contact Agilent Technologies engineers for information about these as well as additional customized macros.

6 Click *OK* to enter the changes.

The Interference Equation dialog box appears. To continue creating the equation, see "Creating/Editing Interference Correction Equations" in the next section.

Creating/Editing Interference Correction Equations

Interference correction equations, if used in your method, should be edited prior to setting the acquisition parameters. This will ensure that all masses necessary for the interference corrections will be acquired. ChemStation enables you to modify or edit the Interference Equation. Interference Equations are very effective for elements that are subject to interference from polyatomic ions, and elements that only have one isotope, such as ^{75}As.

To edit and modify the interference equation, complete the following steps:

1 **Select *Methods>>Edit Interference Equations*.**

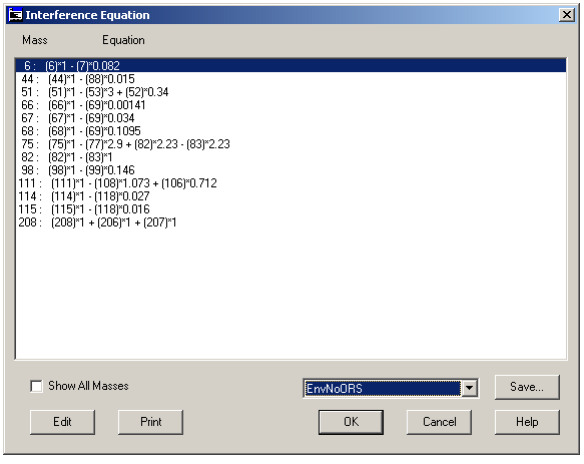
The ***Interference Equation*** dialog box appears. If no interference correction equations have been entered, the Interference Equation panel displays the message "No interference equation has been edited".

2 **Display interference correction equations.**

- When the ***Show All Mass Numbers*** box is checked, all interference correction equations are displayed.
- When a library is selected in the drop-down list box, the interference correction equations in the library are displayed.

The libraries below exist as defaults.

- EnvNoORS: Library for general environment analysis (for non-ORS models)
- EnvORS: Library for general environment analysis (for ORS models)
- EPA200.8: Library for EPA200.8
- EPA6020: Library for EPA6020
- FoodORS: Library for food analysis (for ORS models)



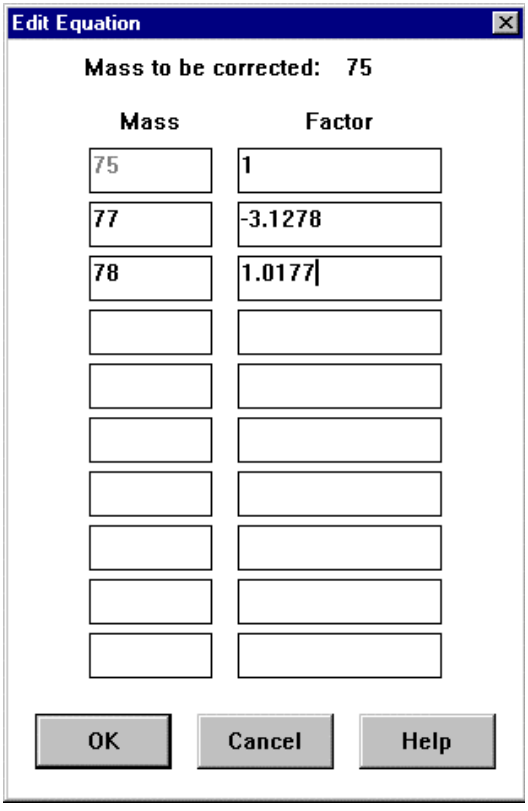
Interference Equation Dialog Box

3 Select the mass for which you want to edit the interference equation by clicking on the mass number.

The selected mass number will be highlighted.

4 Click the *Edit*.

The ***Edit Equation*** dialog box will appear.



Edit Equation Dialog Box

5 Edit the equation in the table.

For example, if the interference equation for As is:
$^{75}As = {}^{75}M \times 1 + {}^{77}M \times (-3.1278) + {}^{78}M \times 1.0177$

(where ^{75}M represents 75 AMU), it would be edited.
This equation is taken from EPA Method 6020 (CLP-M Ver.9).

NOTE The edited interference correction equation is a user-defined interference correction equation to be used only with the currently created method. The default library file will not be updated. To save a user-defined interference correction equation, click on *Save* and enter the name. The equation is saved as a library file. The library file is added to the drop-down list and can be used for other methods.

6 Click *OK* when you are finished editing for that mass.

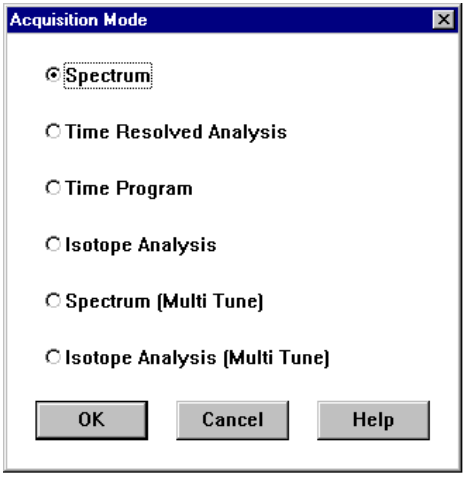
ChemStation will return to the ***Interference Equation*** dialog box, where you can select the next mass for which you want to edit the interference equation.

7 Click *OK* after you are finished editing the interference equations.

The Acquisition Mode dialog box appears. To continue creating the method, see "Setting the Acquisition Mode" in the next section.

Setting the Acquisition Mode

There are six acquisition modes available. You can choose which type of analysis you want to perform according to your application. ChemStation chooses Spectrum mode as default.



Acquisition Mode Dialog Box

Setting Acquisition Parameters

The following sections explain the six acquisition modes:

Spectrum

Using the ***Spectrum*** mode, you can perform ***qualitative***, ***semiquantitative***, and ***quantitative*** analysis. This section will explain how to set parameters for all three types of analysis.

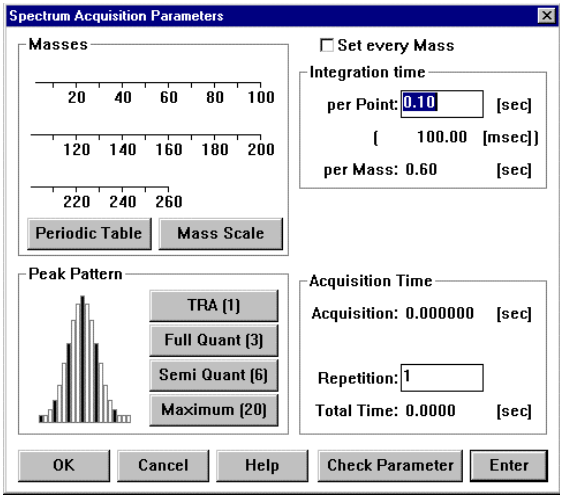
To set the Acquisition Mode to Spectrum, complete the following steps:

1 **Click the *Spectrum*.**

The button is marked and the selected mode is outlined.

2 **Click *OK*.**

ChemStation sets Spectrum as the acquisition mode for the method and the ***Spectrum Acquisition Parameters*** dialog box appears.



Spectrum Acquisition Parameters Dialog Box

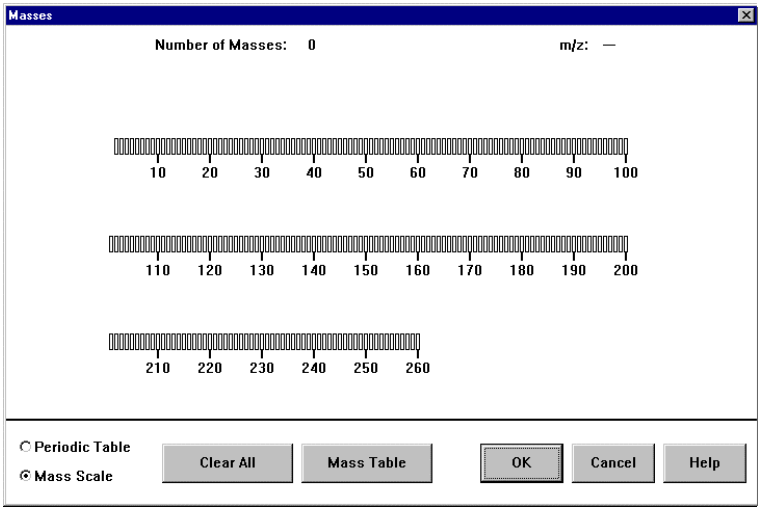
Setting Parameters for Qualitative Analysis

Qualitative analysis provides information about which elements are included in the sample. It is very useful for pre-screening unknown samples. ChemStation scans through all masses (2-260AMU), and gives you a quick visual indication of what elements are present in the sample. It is useful when choosing internal standards, because you can easily confirm that your internal standard choices are not already present in the sample.

To create a method for qualitative analysis, complete the following steps:

1 Click the *Mass Scale* from the Masses (left top) box.

The ***Masses*** dialog box screen will appear.



Masses Dialog Box (Mass Scale)

2 Click *Clear All.*

ChemStation clears any masses selected in the default method. The mass scale display now appears empty.

Double-clicking the right mouse button on a row also clears all masses for that row.

3 Double-click on each of the three rows.

ChemStation will select all masses (2-260 AMU) excluding masses 14, 16 to 19, 32, 36, 38, 40, and 41 AMU. You can tell that the masses are selected by the green vertical bars above the points on the mass scale.

NOTE You can select a range of masses by dragging the mass scale with the mouse, using the left button. You can deselect a range of masses by dragging the mass scale with the mouse, using the right button.

NOTE Masses that have high continuous background count rates, such as 14, 16 to 19, 32, 36, 38, 40, and 41 are automatically skipped since scanning these masses would damage the detector.

4 Click *OK*.

ChemStation returns to ***Spectrum Acquisition Parameters*** dialog box. You can see the masses selected on this screen as well.

5 Click the *Semi Quant (6)* in the Peak Pattern (left bottom) box.

ChemStation will display which 6 points in a peak it will scan. The width of each bar is 0.05 AMU.

6 Double-click or drag the mouse on the Text box in the *Integration time* (right top) box.

Input the integration time e.g. 0.10 (sec), and then click ***Enter***. ChemStation will automatically calculate and show the integration time per mass in seconds, inside the Integration Time box.

7 Input 1 for the number of repetitions in the Acquisition Time (right bottom) box and click *Enter.*

ChemStation will automatically calculate the acquisition time and show how much time it will take to acquire data using this method in seconds, inside the Acquisition Time box above the repetition.

8 Click *Check Parameters*.

Clicking Check Parameters is just to make sure you haven't made any major errors, such as not selecting some of the isotopes to be acquired, etc. If there are no errors, a dialog box will pop up indicating there is no error in the method. If there are errors, some description telling you what the mistake is, will pop up. Click ***OK*** on the pop

up box.

Check Parameter Dialog Box

9 Click *OK*.

All data acquisition parameters are now set. The screen goes on to Peristaltic Pump Program, explained later in this chapter.

Setting Parameters for Semiquantitative Analysis

Semiquantitative analysis is a very powerful feature of ICP-MS, since it provides reasonable accuracy (±10 - 30%) without the need to run external standards. It scans through all masses (or selected masses) and reports concentrations for all elements, based on a stored elemental response database. It is especially useful for screening unknown samples, or for evaluating new matrices sample types. It is also useful for determining the concentrations of calibration standards to be prepared for quantitative analysis.

Because Semiquantitative Analysis is similar to Qualitative Analysis, acquisition parameters are normally the same. Refer to "Setting Parameters for Qualitative Analysis", earlier in this chapter.

Setting Parameters for Quantitative Analysis

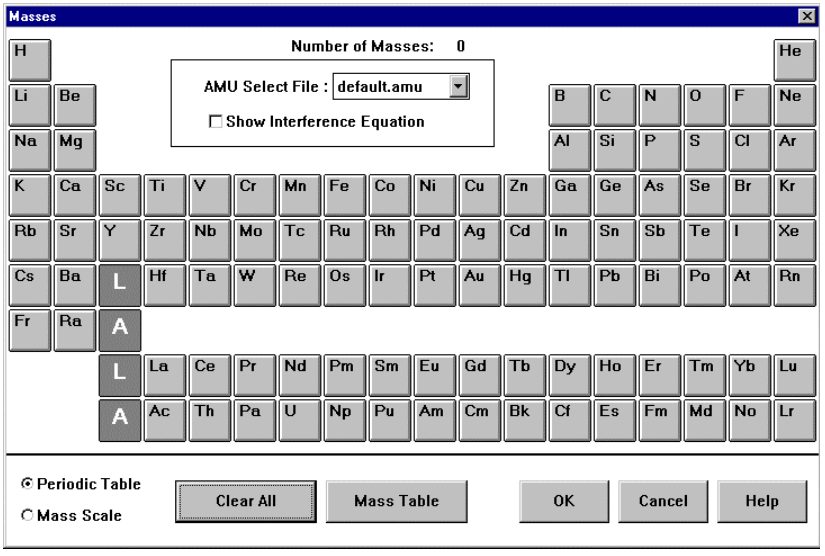
Quantitative analysis provides information about specific elements in a sample. You can quantitate the elements you selected by using calibration curves.

The Masses box in the Spectrum Acquisition Parameters dialog box can display either the periodic table or the mass scale. You can use either to select masses and elements. The periodic table display is useful to select the elements for quantitative analysis, so the following section will explain the operation of the periodic table display. To use the mass scale for selecting masses, refer to "Setting Parameters for Qualitative Analysis", in this chapter.

To set parameters for quantitative analysis, complete the following steps:

1 Click the *Periodic Table* from the Masses (left top) box.

The Masses dialog box (Periodic Table Masses) will appear.



Masses Dialog Box (Periodic Table)

2 Click *Clear All.*

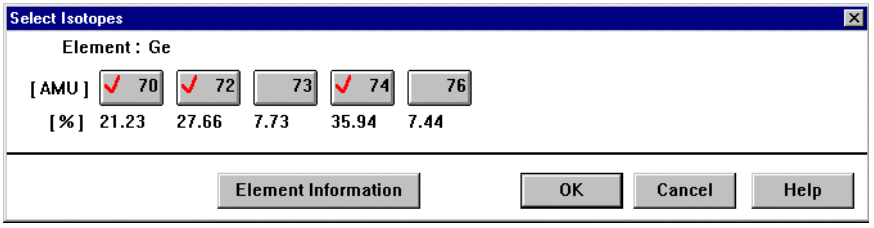
ChemStation clears any masses or elements selected in the default method. The periodic table display now appears empty.

3 Select elements by clicking the element push buttons in the periodic table.

The push buttons for the elements you select turn from gray to red. The isotopes for each element are selected automatically according to the default recommended isotopes. A convenient table with these recommended isotopes is available, (Relative Isotopic Abundance Table).

4 Double-click on the element push button to select additional isotopes that are not in the default method.

A dialog box for selecting isotopes will pop up. To select isotopes, simply click on the buttons with isotope numbers and red check marks will appear next to the number. To delete, click the right mouse button. You can select one or all isotopes for the selected element.



Select Isotopes Dialog Box (e.g. Ge)

NOTE

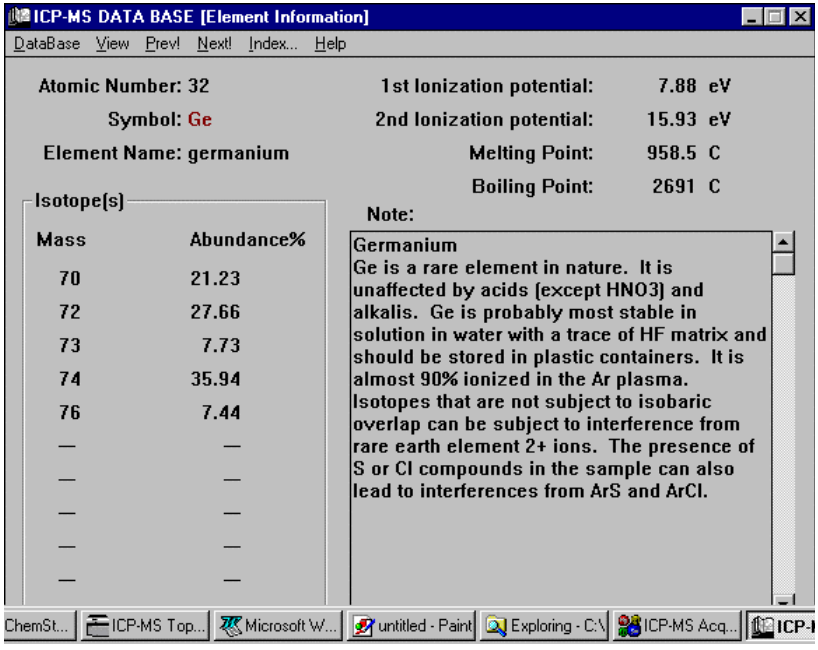
The pre-selected isotopes are contained in default AMU Select File (default.amu). You can create your own AMU file to select different isotopes than default.amu. To do so, refer to "Modifying a Method" later in this chapter.

5 Click *Element Information* for some information about the element.

The ICP-MS DATA BASE (Element Information) window will appear. There is more information available in this database. The name in parentheses in the title bar indicates the type of information currently displayed, of 3 types: Element Information, Interference Information, and AMU Information.

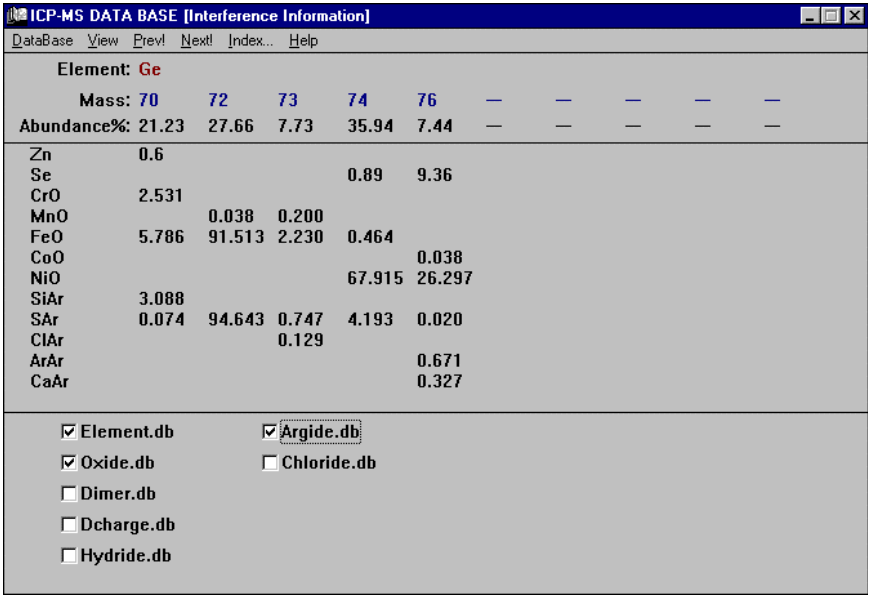
To view other information, select ***View>> Interference Information* or *AMU Information.***

The Element Information contains data on ionization potentials, melting point, boiling point, and isotopic abundance for the selected element. It also contains notes that provide additional information about the selected element, such as the element's toxicity, solubility, and stability.



ICP-MS DATA BASE (Element Information) Dialog Box

Interference Information provides information about isobaric interferences where elements have isotopes that overlap at the same mass. You can also view possible interferences from polyatomic ions by clicking in the check boxes at the bottom of the dialog box. Polyatomic ions of the selected specific type that could interfere with the masses of the element will be displayed. Multiple types of polyatomic ions can be selected.

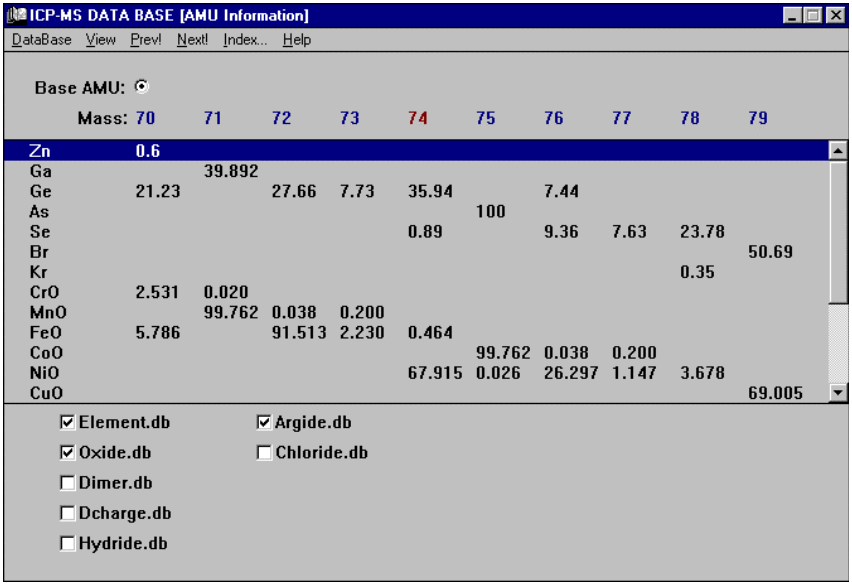


ICP-MS DATA BASE (Interference Information) Dialog Box

NOTE

Click ***Prev!*** to see interference information about the previous element (in this case Ga). Click ***Next!*** to see interference information about the next element (in this case As). Click ***Index*** to see information about any other elements. An element index will pop up. Click on the element then click ***OK***.

The AMU Information contains information about the abundance of an element at a specific mass. You can check interferences from polyatomic ions by clicking in the check boxes at the bottom of the dialog box. The polyatomic ions of the selected specific type that could interfere with the masses displayed will be displayed. Multiple types of polyatomic ions can be selected.



ICP-MS DATA BASE (AMU Information) Dialog Box

NOTE

Click ***Prev!*** to see interference information about the previous AMU. Click ***Next!*** to see interference information about the next AMU. Click ***Index*** to see information about any other masses. An AMU index will pop up. Click on an AMU then click ***OK***.

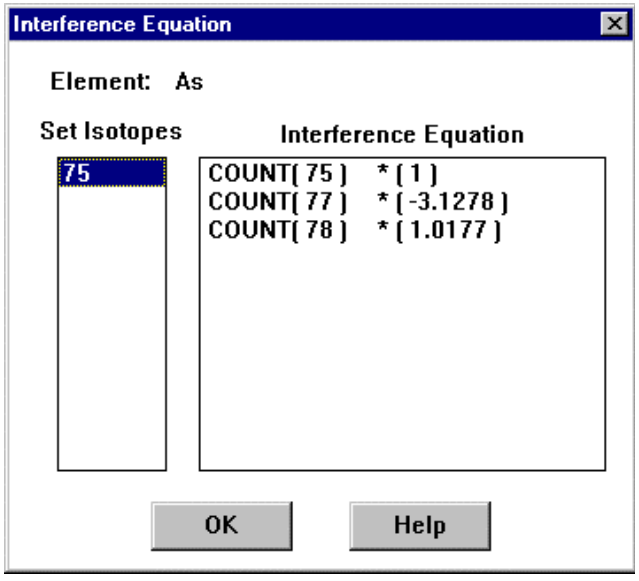
To close the ICP-MS DATABASE dialog box, double-click the Control Box Menu in the upper left corner of the dialog box. ChemStation returns to the ***Select Isotopes*** dialog box.

6 Click *OK* after selecting all isotopes you want to analyze for that element.

ChemStation returns to the periodic table. The numbers of isotopes you selected are indicated in the right bottom corner of each element push button.

You can see equations to correct isobaric and polyatomic interferences.

To view the interference equation being used for an element, click the ***Show Interference Equation*** check box, then the element for which you want to view the equation. The Interference Equation dialog box appears showing interference equations for selected isotopes of the element.



Interference Equation Dialog Box

NOTE

Do not click ***Show Interference Equation*** before selecting the elements and isotopes for your method. If you do, ChemStation prevents you from selecting elements or viewing isotopic information.

Click ***OK*** to return to the Periodic Table dialog box. You can check the interference equations for all isotopes you selected.

7 After you have finished selecting all isotopes and elements you want to analyze, click on the *Mass Table* button.

A table will display which AMU's you selected and for which elements. You can check if there are any mistakes here. When you select the elements which have the

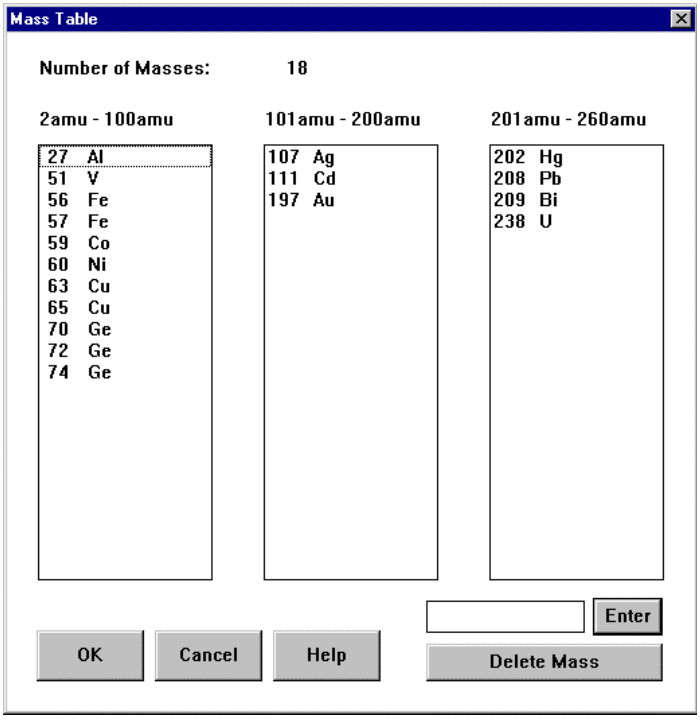
AMU's used in the interference equation at the Periodic Table Masses window, the element names in the Mass Table window will be displayed automatically and be put in parentheses.

You can delete or add elements and AMU's on this screen.

To add: type a mass number and element in the Text box (bottom right) and click ***Enter***. e.g. 205 Tl

To delete: click on the mass you want to delete and then ***Delete Mass***.

NOTE Ensure that the AMU's used in the interference equation are entered.



Mass Table Dialog Box

8 Click *OK*.

ChemStation will return to the periodic table.

9 Click *OK*.

ChemStation will return to the Spectrum Acquisition Parameter screen.

10 Click the *Full Quant (3)* in the Peak Pattern (left bottom) box.

ChemStation will display the 3 peak top points. The width of each bar is 0.05 AMU.

11 Set the integration time (sec).

ChemStation enables you to set the same integration time for all the selected masses in the method or to set a different integration time for each mass. Set the integration time by following one of these procedures:

- Setting the same integration time for all masses

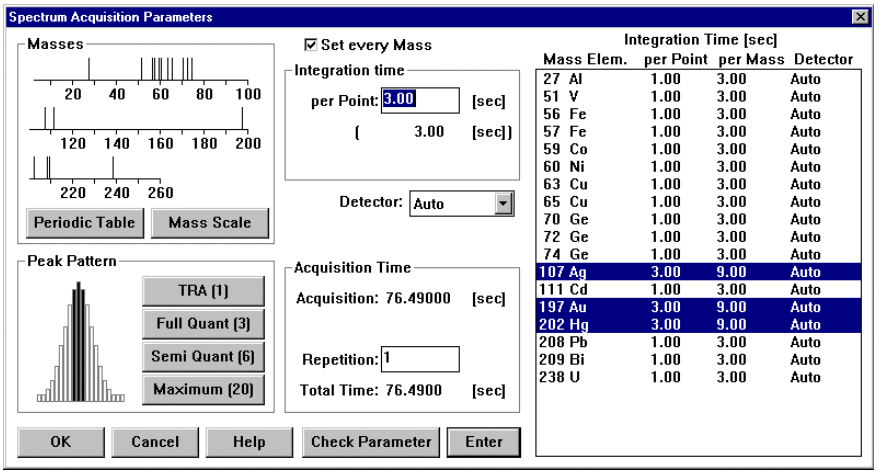
  To set the same integration time for all masses, double-click or drag on the Text box in the Integration Time (right top) box. Input the time in seconds and click ***Enter***.

- Setting a different integration time for each mass.

  a) Click the check box of ***Set Every Mass*** at the top right corner of the Spectrum Acquisition Parameter dialog box. An additional panel will appear showing all selected masses and elements.

  b) Click the mass for which you want to change the integration time. You can select multiple elements by pressing Shift or Control and clicking on several elements. Change its integration time by double-clicking or dragging on the text box in the integration time (right top) box. Input the time in seconds and click ***Enter***.

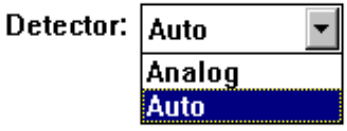
  c) Repeat these processes for each mass for which you want to change the integration time.



Spectrum Acquisition Parameters Dialog With *Set Every Mass* Panel

12 Set the Detector Mode.

Click the masses in the list which you want to analyze using the analog mode. Then select ***Analog*** in the Detector mode box. Click ***Enter***.



Detector Mode Box

13 Set the number of repetitions by double-clicking or dragging on the text box in the Acquisition Time (right bottom) box, type a number, and click *Enter.*

A regular quantitative analysis might require 3-5 repetitions to acquire good data. ChemStation will automatically calculate how much time it will take to acquire data using the method, and it will be displayed in the Acquisition Time box.

14 Click *Check Parameters.*

This is to make sure that you haven't made any major errors, such as forgetting to select some of the masses to be acquired, etc. If there are no errors in the method, a dialog box will pop up to indicate so. If there is an error, then some description

telling you what the mistake is will pop up. Click ***OK*** on the pop up box.

15 Click *OK*.

All data acquisition parameters are set as you edited them. The screen goes on to Peristaltic Pump Program, explained later in this chapter.

Prevention of EM deterioration

When higher signals are analyzed for a long time with too high sensitivity, the EM may deteriorate fast. EM deterioration causes the drift of the signal and the lifetime of the EM may be shorten. The following methods will prevent EM deterioration:

- Set suitable integration time

  Shorten the integration time according to the suitable setting required for the production of reproducible data. The following table shows the recommended value of integration time for the prevention of the EM deterioration. Please refer to it and set the integration time for each mass.

Recommended Value for Integration Time

The Standard of concentration *	Counts	Integration Time per Point	Detector Mode
< a few 10 ppb	0 to 500kcps	> 0.1sec	Pulse
A few 10 ppb to approx.100ppb	500kcps to 1Mcps	0.01sec	Pulse
> approx.100ppb	1Mcps to 4Gcps	0.01sec	Analog

* the sensitivity is 10,000 cps/ppb

  These values are only for reference, so please set the longer integration time according to your application, if necessary. For more information regarding the settings for integration time, please refer to Chapter 5, "Creating a method" and the Application Handbook.

- Decrease the intensity

  When the group of signals that you want to analyze is large, high sensitivity is not required. It is recommended to decrease the sensitivity. If you want to decrease the intensity, adjust the ion lens voltage (not EM voltage). To adjust the ion lens voltage, see "Tuning for Sensitivity" in this chapter.

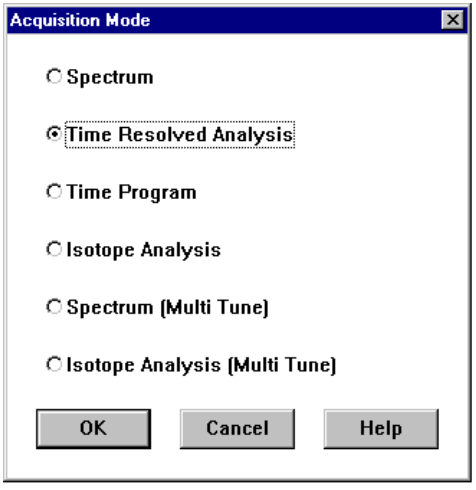
Time Resolved Analysis (TRA)

Time Resolved Analysis acquisition (TRA) produces a transient signal that appears as an intensity versus time plot (time chart). This acquisition mode is usually used when the signal changes with time (transient signal). You can monitor the signals including a time factor for each selected mass. It is also convenient for applications which combine the Agilent 7500 with peripheral equipment, such as the Laser Ablation, the Ion Chromatograph, etc.

To set the Acquisition Mode to Time Resolved Analysis, complete the following steps:

1 **Click the *Time Resolved Analysis* button.**

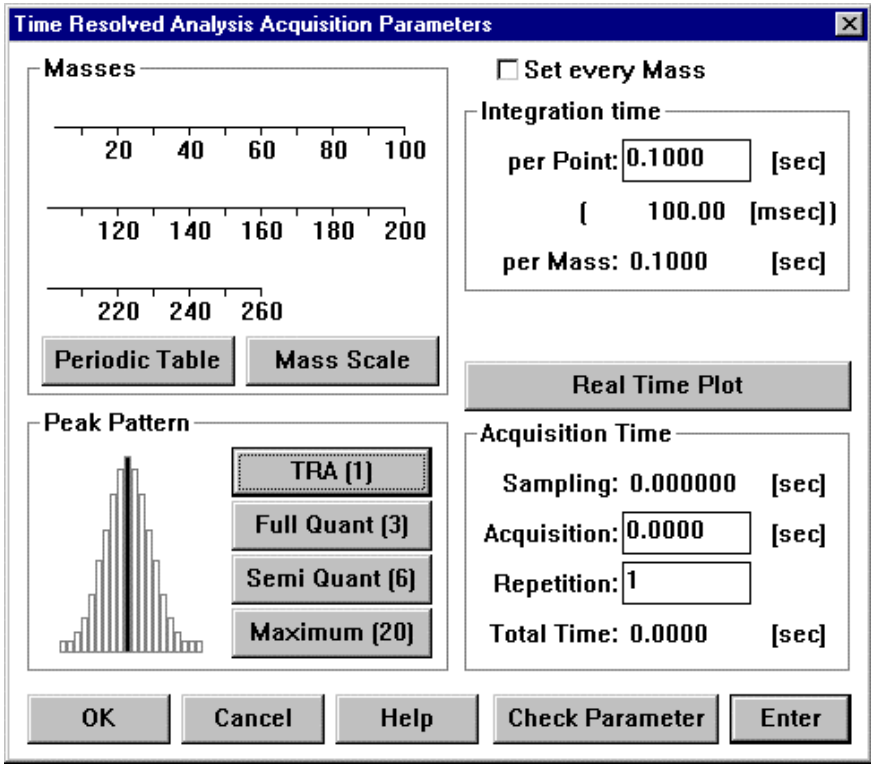
The button is marked and the selected mode is outlined.



Acquisition Mode Dialog Box---Time Resolved Analysis

2 Click *OK*.

ChemStation sets Time Resolved Analysis as the acquisition mode for the method and the ***Time Resolved Analysis*** dialog box appears.



Resolved Analysis Acquisition Parameters Dialog Box

Setting Parameters for Time Resolved Analysis

Time Resolved Analysis provides information about changes in signals of specific masses with time.

Because setting the parameters for TRA is similar to Spectrum Analysis mode, refer to "Setting Parameters for Quantitative Analysis", earlier in this chapter, as well as the following procedure:

1 Select the mass numbers for acquisition by using Periodic Table or Mass Scale.

To select the mass, see "Setting Parameters for Quantitative Analysis" in this chapter.

2 Click the *TRA (1)* in the Peak Pattern (left bottom) box

ChemStation will display the peak top point which it will scan. The width of each bar is 0.05AMU.

3 Set the integration time (sec) and the detector mode.

To set the integration time and the detector mode, see "Setting Parameters for Quantitative Analysis" in this chapter.

NOTE The sampling period, which is displayed in the Acquisition Time area, must be set at 10msec or longer. For more information about the sampling period, see "***Acquisition Parameters*** dialog box" in the Online Help.

NOTE To get reproducible results, at least 10 points per peak is recommended. So, the integration time depends on the number of elements and peak width and integration time affects the sampling time.

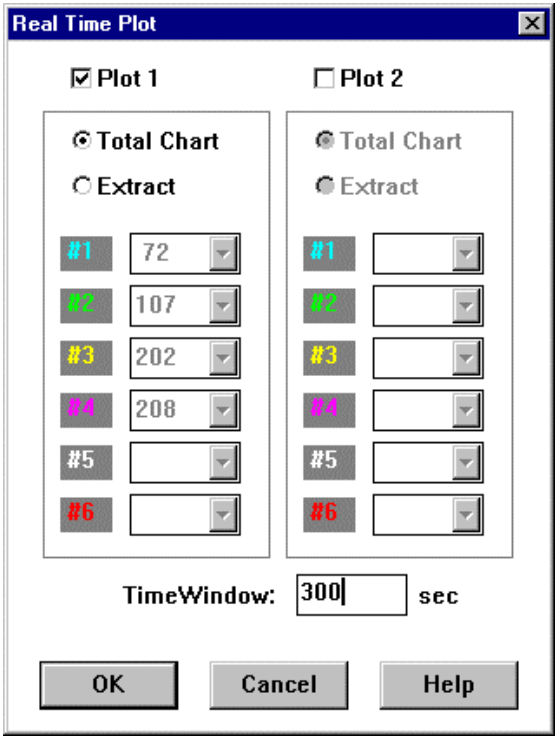
4 Input the acquisition time in seconds in the Acquisition Time (right bottom) box.

The acquisition time for TRA is the total amount of time that data is acquired. You can type the time in seconds by double-clicking or dragging in the text box in the Acquisition Time box. The value entered will be replaced by the nearest integral multiple of the sampling time.

5 Click the *Real Time Plot* button.

The ***Real Time Plot*** dialog box will appear. This dialog box allows the user to control the display of up to two real time plots. Each plot can be configured to display either the Total Ion or the Extract by selecting the appropriate button. If

Extract is selected, then the available masses will turn to black from gray; it will then be possible to change the order of the mass list.



Real Time Plot Dialog Box

6 **Input the X-axis for the real-time display by double-clicking or dragging on the text box for *TimeWindow*.**

You can set the time to display the real time signal.

7 **Click *OK*.**

ChemStation will return to Time Resolved Analysis Acquisition Parameters dialog box.

8 **Set the number of repetitions by double-clicking or dragging on the text box in the Acquisition Time (right bottom) box, type a number, and click *Enter*.**

Usually input 1 for the number of repetition.

9 Click *Check Parameters*.

This is to make sure that you haven't made any major errors, such as forgetting to select some of the masses to be acquired, etc. If there are no errors in the method, a dialog box will pop up to indicate so. If there is an error, then some description telling you what the mistake is will pop up. Click ***OK*** on the pop up box.

10 Click *OK*.

All data acquisition parameters are set as you edited. The screen goes on to Peristaltic Pump Program, explained later in this chapter.

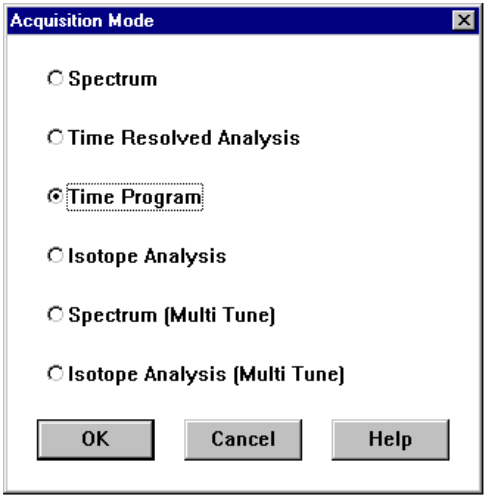
Time Program

ChemStation enables you to set acquisition parameters for up to 10 sections, known as steps, in a time program. This enables you to change any method parameters part of the way through a time resolved analysis. For example, you can change the masses or load a different tuning file at a specific time in the sample run. You can set different parameters for each step, or you can copy parameters from one step to another. You complete this step in creating a method only if you are using Time Program acquisition.

To set the Acquisition Mode to Time Program, complete the following steps:

1 **Click the *Time Program* button.**

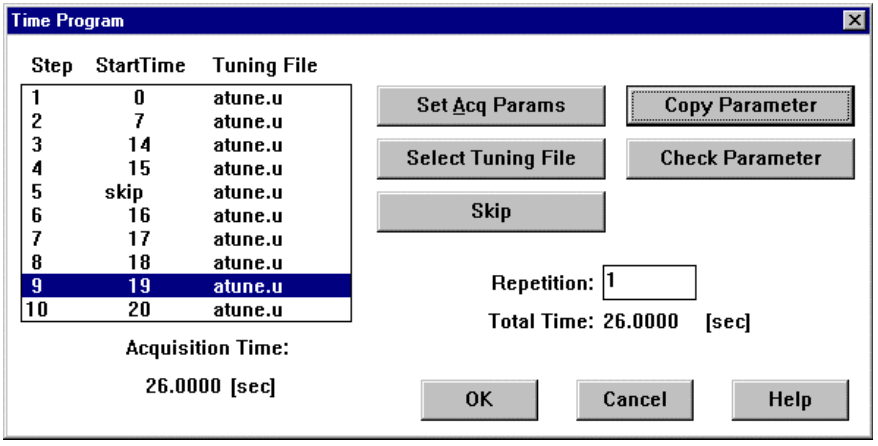
The button is marked and the selected mode is outlined.



Acquisition Mode Dialog Box-Time Program

2 Click *OK*.

ChemStation sets Time Program as the acquisition mode for the method and the ***Time Program*** dialog box appears.



Time Program Dialog Box

Setting Parameters for Time Program

To create and set a time program, complete the following steps. Please refer to "***Time Program*** dialog box" in the Online Help.

1 **To select a step for which to set parameters, click the step in the displayed list.**

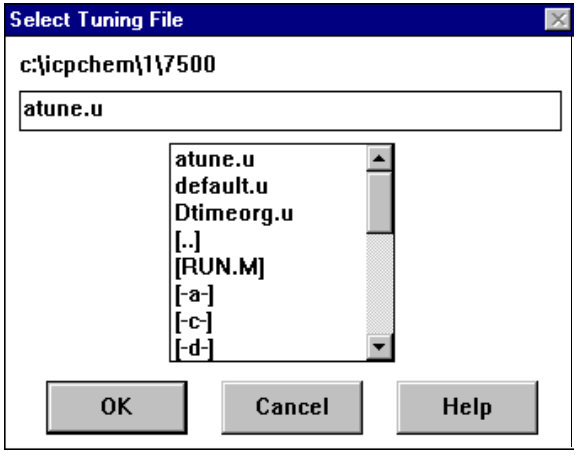
ChemStation highlights the step. All the parameters you set apply to the selected step.

2 **To set acquisition parameters for a step, click the *Acquisition Parameter* push button.**

ChemStation enables you to access the dialog box for setting the acquisition parameters (masses, peak pattern, detector mode, integration time, acquisition time and real time plot) for each step in the time program. To set acquisition parameters, go to "Setting Parameters for Time Resolved Analysis" (in the previous section). Click ***OK*** after you finish setting the parameters, and ChemStation will return to the Time Program dialog box.

3 **To select a tune file for a step, click *Select Tuning File*.**

The ***Select Tuning File*** dialog box appears.



Select Tuning File Dialog Box

Select the tune file in one of the following ways:

- Type the name of the tune file and click ***OK***. If the directory path in the Select File dialog box is incorrect, you must type the directory path as well as the name of

the file.

- Double-click a tune file in the displayed list.

ChemStation will return to the Time Program dialog box.

4 To set the start time for a step, click *Skip*.

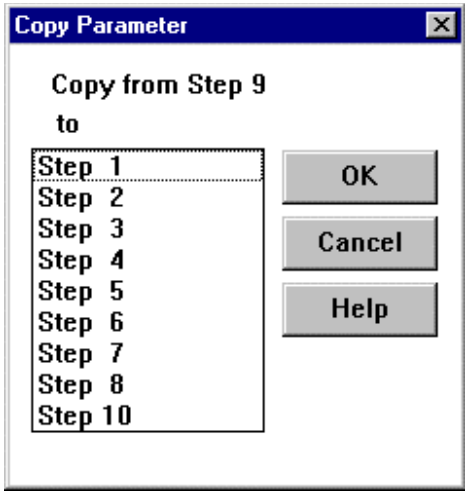
For executing a step of interest, click on the ***Skip*** button to display the start time for that step. The start time displayed for a step includes the acquisition time for the preceding step. Note that the integration time and acquisition time for a step must be set before the start time for that step can be set.

When you want to skip a specific step without executing it, you can also use this button to display "skip" for that step in the list box.

5 To copy parameters from the current step to another one, complete the following steps:

a) Click *Copy Parameter.*

The ***Copy Parameter*** dialog box appears, asking which step you want to copy the current parameters to.



Copy Parameter Dialog Box

b) Click the step in the dialog box that you want to copy parameters to and then click *OK*.

ChemStation copies the parameters you set for the current step to the step you just highlighted and return to the Time Program dialog box.

6 **Repeat Steps 1 through 5 for each step.**

7 **Click *OK* after all parameters for all of the steps in the time program are set.**

All of the Time Program is set as you edited. The screen goes on to Peristaltic Pump Program, explained later in this chapter.

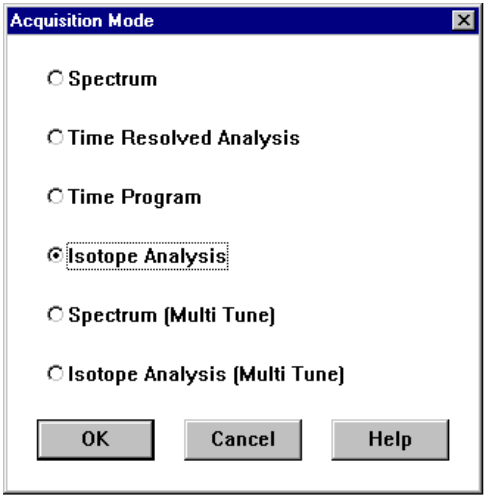
Isotope Analysis

Isotope Analysis is similar to Spectrum Analysis. The difference is that the dwell time per mass is 10 times shorter. This mode should be used when precision for isotopic information (isotopic ratio and isotope dilution) is required (e.g. radioactive isotope analysis for determining age, etc.).

To set the Acquisition Mode to Isotope Analysis, complete the following steps:

1 **Click the *Isotope Analysis* button.**

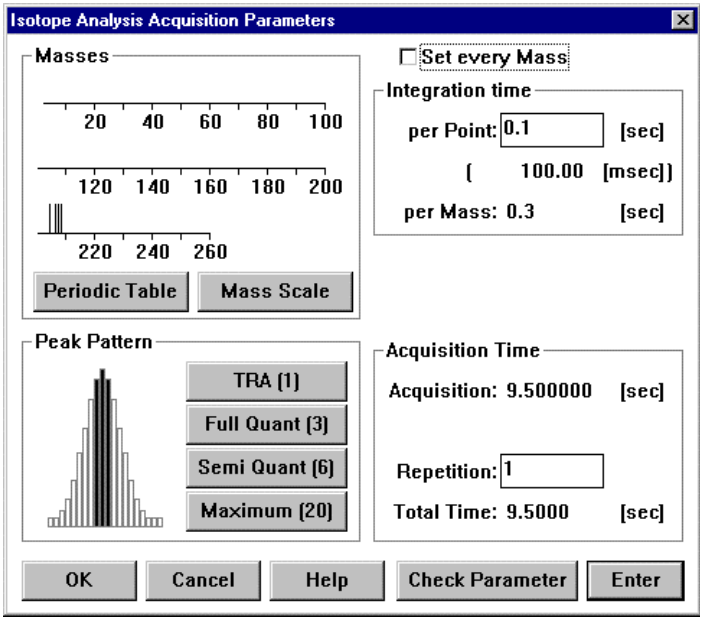
The button is marked and the selected mode is outlined.



Acquisition Mode Dialog Box---Isotope Analysis

2 **Click *OK*.**

ChemStation sets Isotope Analysis as the acquisition mode for the method and the ***Isotope Analysis Acquisition Parameters*** dialog box appears



Isotope Analysis Acquisition Parameters Dialog Box

Setting Parameters for Isotope Analysis

Because Isotope Analysis is similar to quantitative analysis in Spectrum Analysis, setting the acquisition parameters is the same. Refer to "Setting Parameters for Quantitative Analysis" in this chapter to set acquisition parameters for Isotope Analysis.

NOTE

The ***Full Quant (3)*** in the Peak Pattern (left bottom) box is better than 1 point on the Peak Pattern for Isotope Analysis.

Spectrum Multi Tune

For semiconductor applications, Cool plasma allow for K, Ca and Fe to be measured at low level. However Some elements with high ionization potentials can not measured by cool plasma because cool plasma can not ionize then efficiently, so the sensitivity is low. Every sample has to be run twice if the analyte list includes the elements not possible by cool plasma.

Multi Tune allows cool and normal plasma samples to run at one time even when the analyte list includes the elements whose sensitivity is too low to be run by cool plasma. Multi Tune allows running the sample by both hot plasma and cool plasma with a single acquisition.

NOTE

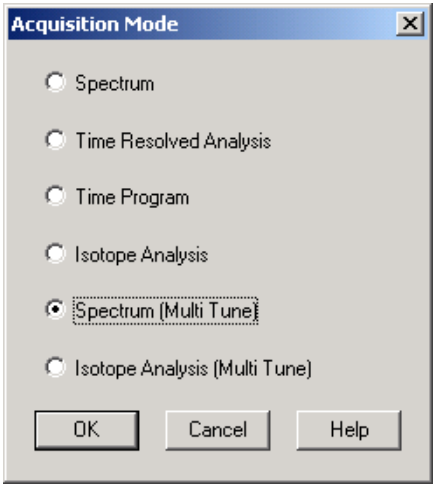
To run cool plasma, the Shield Torch system is necessary. If the system does not have a Shield Torch system, and you have the an application that requires it contact your local Agilent sales office for an upgrade.

Using ***Spectrum [Multi Tune]*** mode, you can perform ***qualitative***, ***semiquantitative***, and ***quantitative*** analysis. This section will explain how to set parameters for all three types of analysis.

To set Acquisition Mode to Spectrum, complete the following steps:

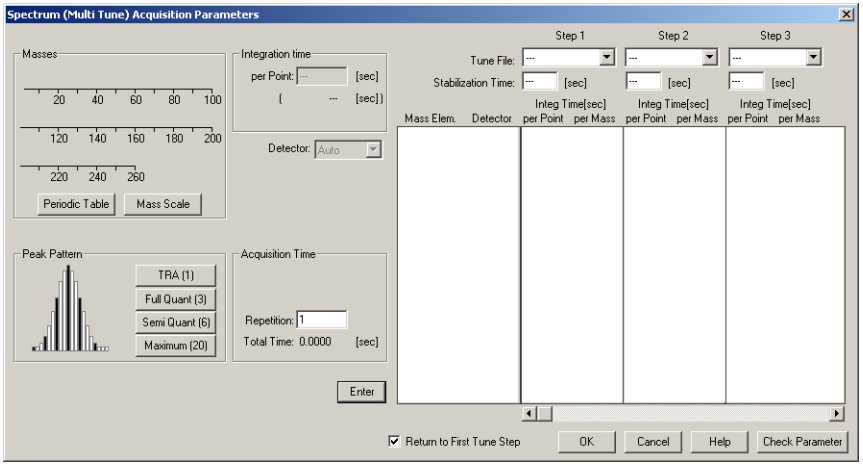
1 **Click *Spectrum [Multi Tune].***

The button is marked and the selected mode is outlined.



2 Click *OK*.

ChemStation sets Spectrum [Multi Tune] as the acquisition mode for the method and the ***Spectrum [Multi Tune] Acquisition Parameters*** dialog box appears.



Spectrum [Multi Tune] Acquisition Parameters Dialog Box

Setting Spectrum [Multi Tune] Acquisition Parameters

Select the masses, Integration time and Peak Pattern for the elements to be acquired. Refer to Setting Parameters section discussed in the Creating a Method section of this manual.

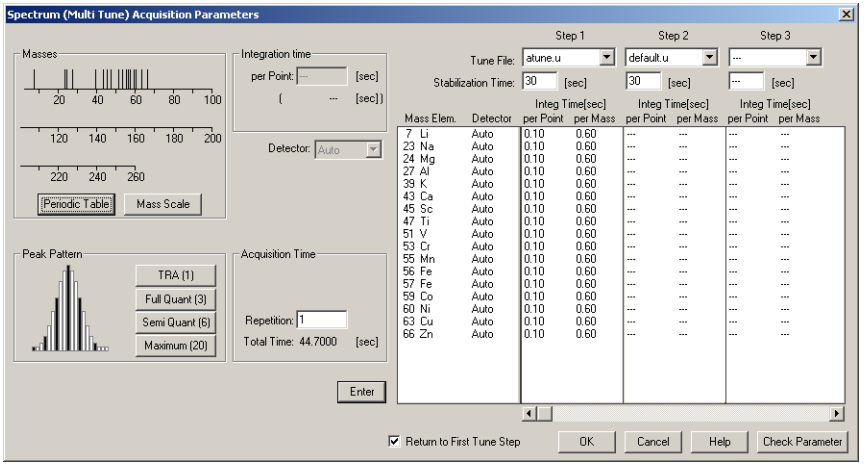
1 Select the Tune File to be used for the first set of elements.

Select a tune file for Step 1 from the drop-down list in the upper right section and enter the stabilization time (standard setting: approx. 30 sec) in the list box. When 30 seconds is entered for ***Stabilization Time***, measurement will start 30 seconds after the file for Tune Step 1 is loaded.

2 Select the second tune file to be used for the second set of elements.

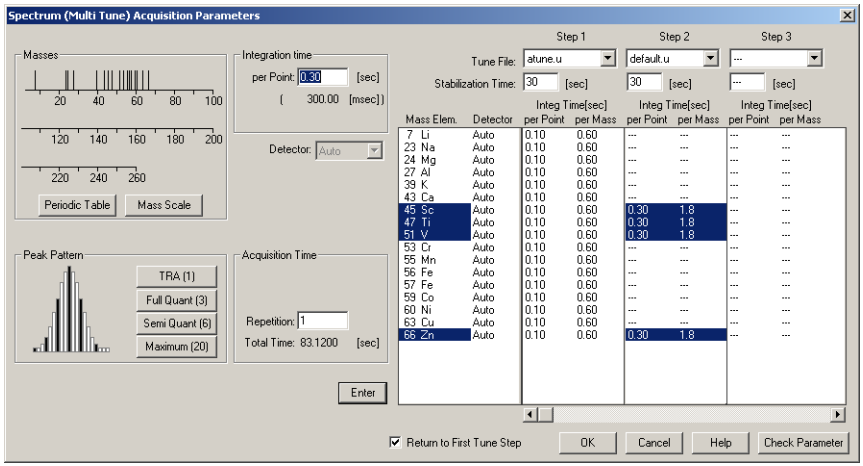
Similarly, select a tune file for Step 2 and enter the stabilization time.

Up to six tune files may be set (for Tune Steps 1 to 6). The integration time set in Tune Step 1 will be the same for each element of all subsequent Tune Steps.

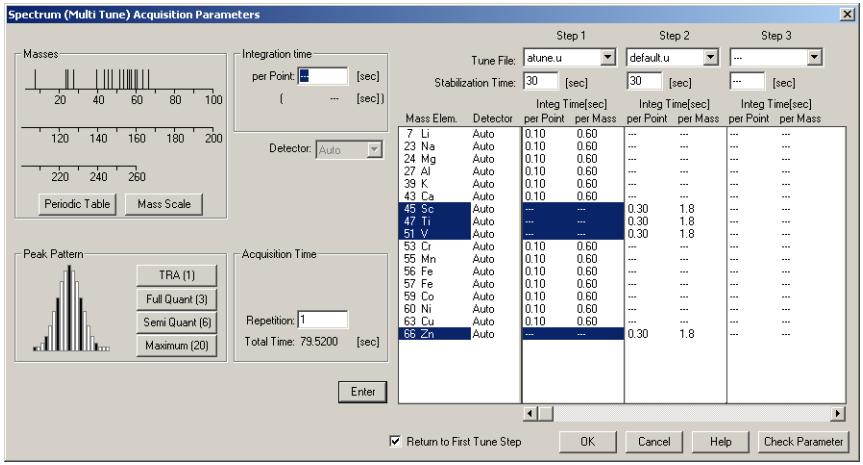


3 Highlight the mass (see below) by clicking on the masses while holding down the Ctrl key.

Set the integration time for each tune step. The integration time can be set for multiple tune steps in each mass number. To set the integration time, click the integration time area which is crossed by the target mass number and the target tune step, then enter the integration time and click **Enter**.



If you do not measure the mass number on particular tune step, enter " - " or " --- " in the integration time cell. But you should enter at least 1 integration time for each mass number.



If more than 2 steps (tune files) are being used, repeat step 3 for the tune files.

If you want to remove tune files from the ***Tune Step***, highlight the tune files and click ***Remove Tune Step***.

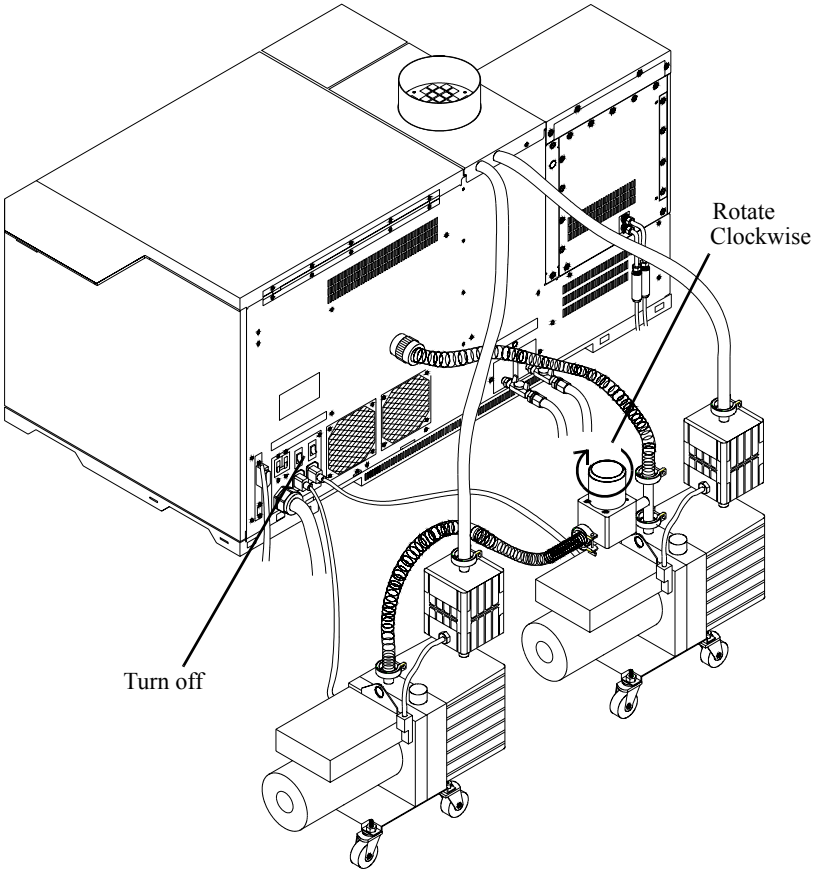
Turn the ***Return to First Tune Step*** check box on when you want to reset the tuning parameters of the ICP-MS after finishing data acquisition. If the check box is off, after finishing the data acquisition, the tuning parameters of the ICP-MS are the tuning parameters in the last tune step file.

Setup

This document will give you an example of the operation procedure (cool and hot) from setting up the system to make a method using Multi Tune.

1 Rotary Pump

Confirm that only one Rotary Pump is running when running cool plasma.
Check the top on the valve between the two Rotary Pumps is rotated clockwise (closed).
Check its breaker located behind the ICP-MS mainframe is turned off.

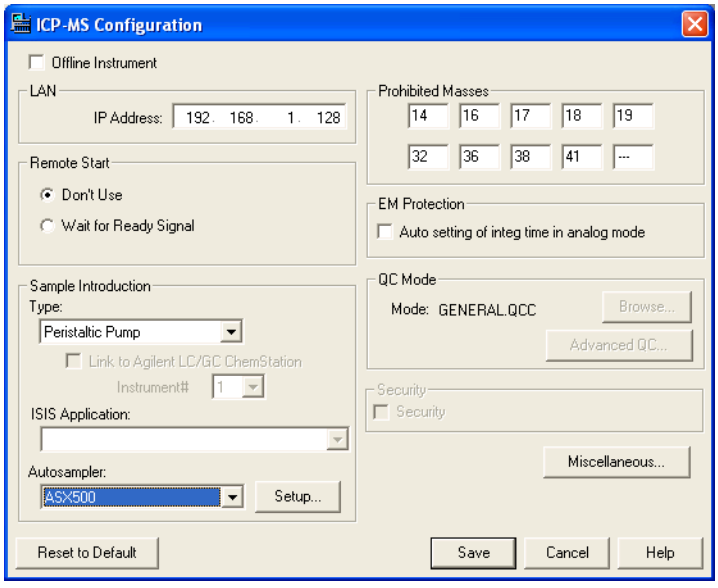


2 Install the shield plate

Refer to pages 1-12 to 1-16 in "*Agilent 7500 ICP-MS Option Instruction Manual"* about shield torch installation.

3 Remove mass 40 AMU as one of the Prohibit masses at Configuration window when you need to measure ^{40}Ca.

Click Configuration Icon
Delete 40 from ***Prohibited Masses***



Click ***Save***

CAUTION When you are no longer using the Shield Torch, reset mass 40 AMU in the Prohibited Masses to prevent potentioal detector damage from the high 40 AMU signal in normal plasma.

4 Ignite plasma

Refer to page from 3-9 to 3-11 in "*Agilent 7500 ICP-MS Hardware Manual"*.

5 Tuning

When changing to Analysis Mode is completed, Click Tune button in ICP-MS Top Window.
Tune for cool plasma and hot plasma conditions, save individual files for each

mode.

Normal plasma tuning procedure

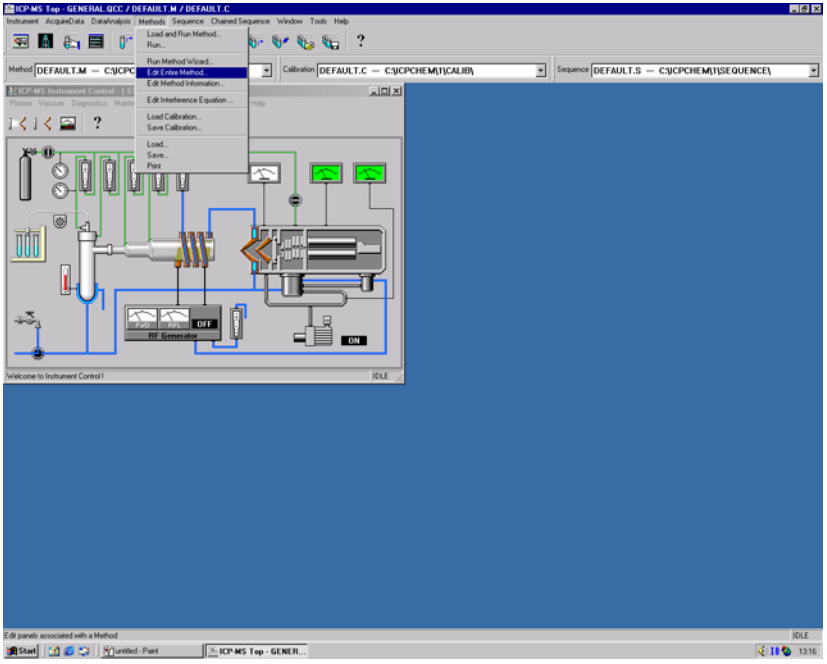
Refer to Chapter 4 in "*Agilent 7500 ICP-MS ChemStation Operator's Manual"*.

Cool plasma tuning procedure

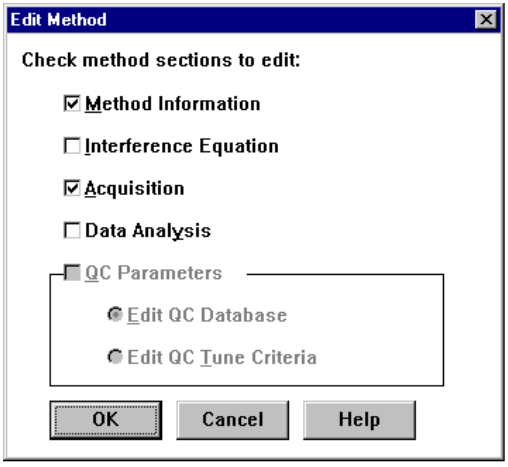
Refer to page from 1-25 to 1-30 in "*Agilent 7500 ICP-MS Option Instruction Manual"*.

Making the Method

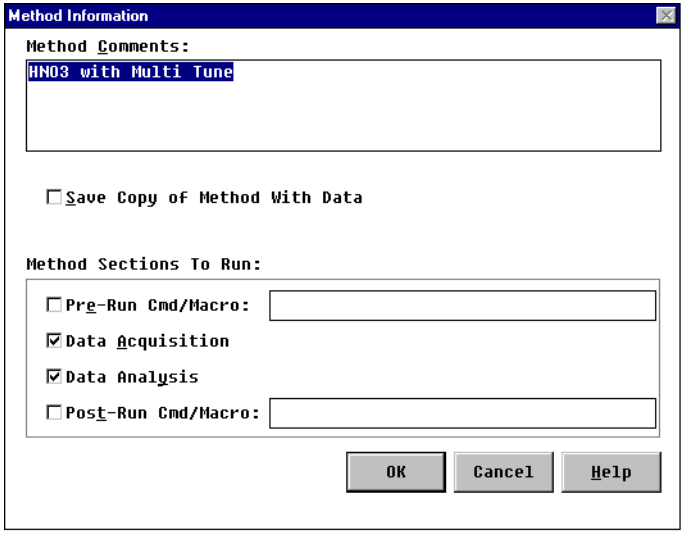
1 Go to *Method >> Edit Entire Method*



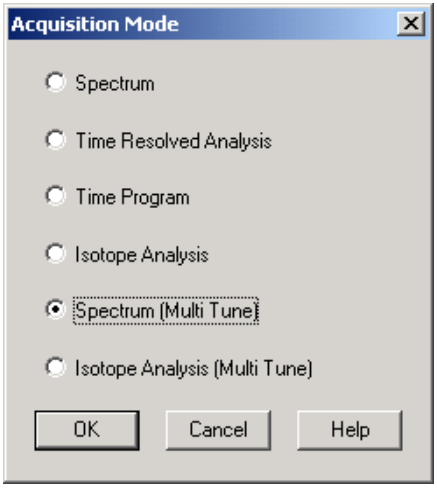
2 Check the Method section to edit.



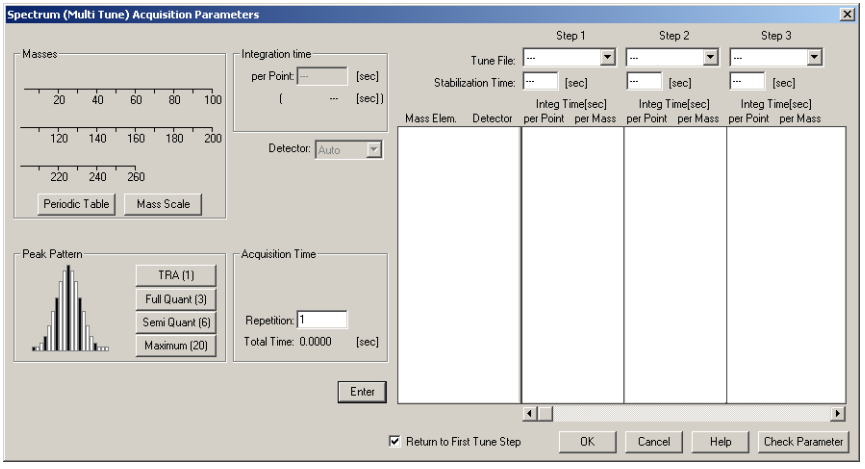
3 Enter Method comment as you like, then Click OK



4 Select *Spectrum [Multi Tune]*, then click *OK*.

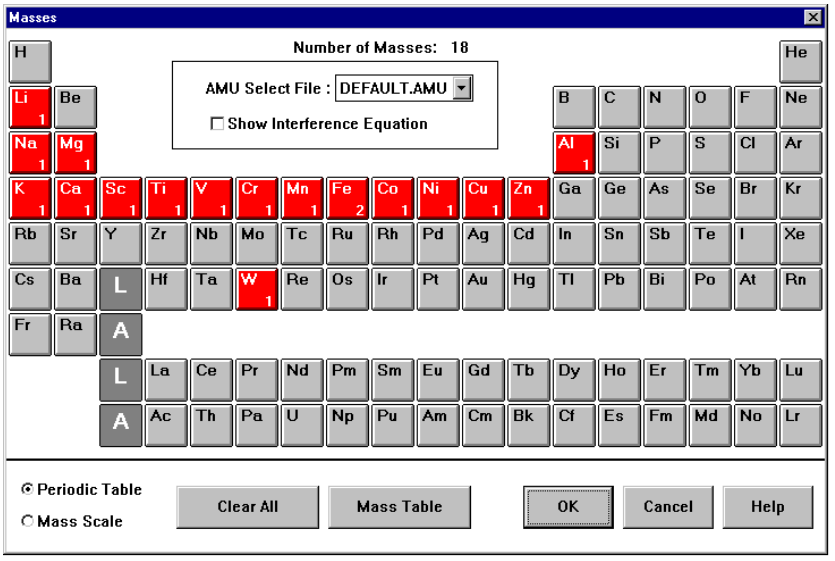


5 Acquisition [Multi Tune] parameters windows appear

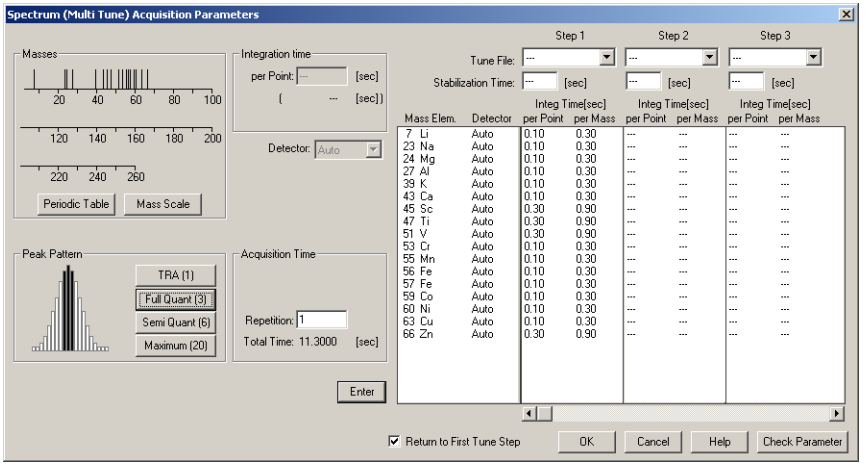


Click ***Periodic Table***

6 Select masses you analyze, then Click OK.

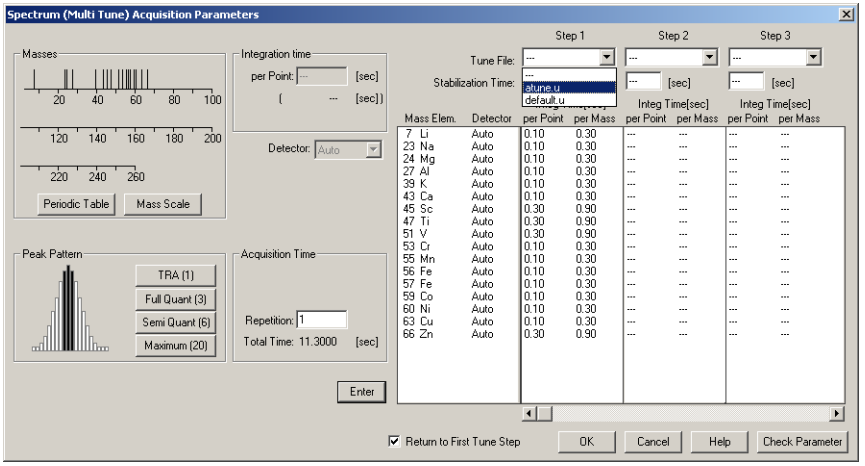


7 Select Peak pattern [3]

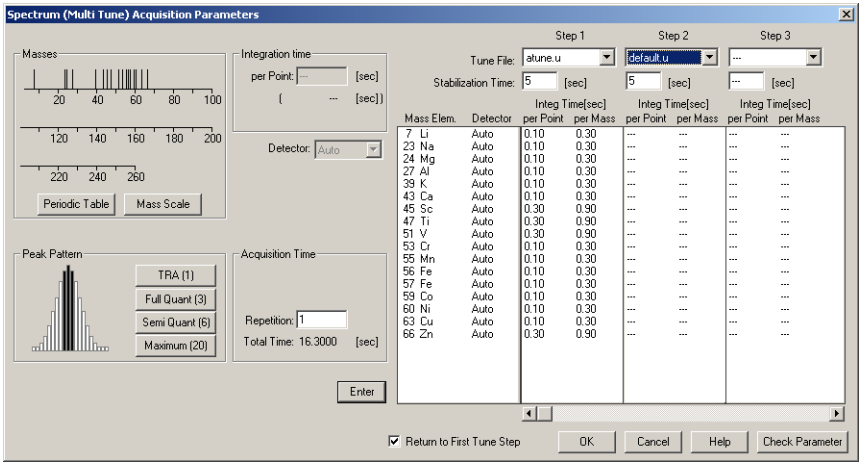


Click ***Tune Step*** 1
Click ***Browse…*** to select the Tune files

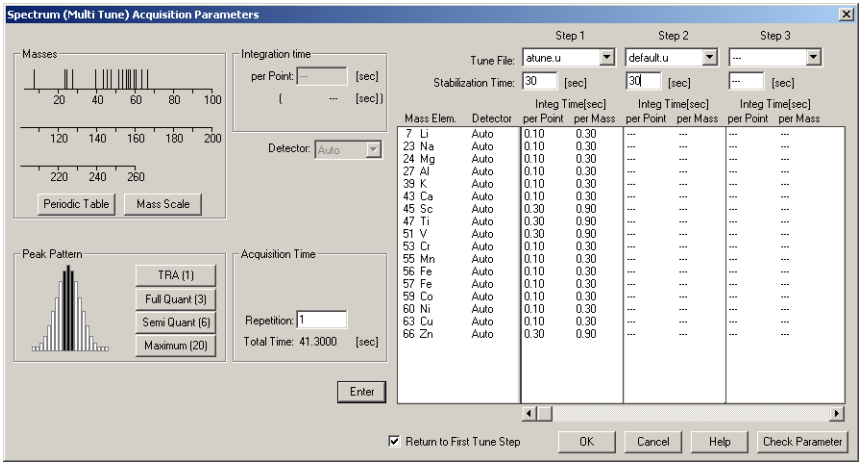
8 Select a tune file for Step 1 from the drop-down list in the upper right section.



9 Click *Tune Step* 2.



10 Enter the stabilization time (e.g., 30 sec) for Step 1 and Step 2 in the text boxes in the upper right section.



NOTE

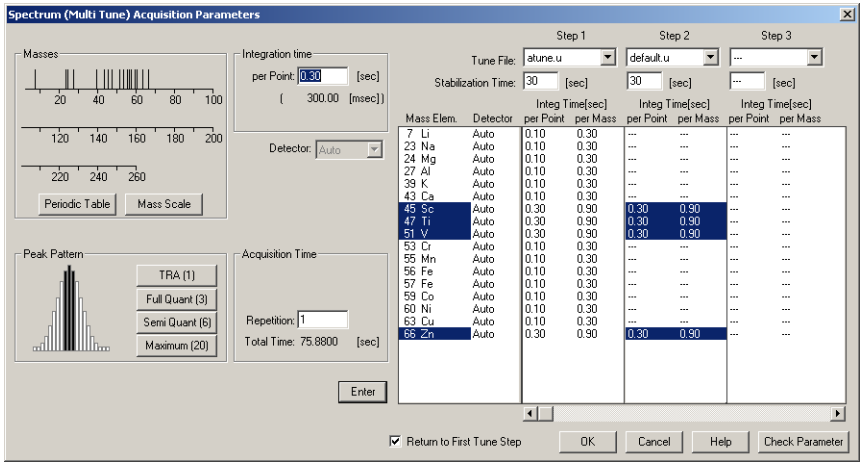
The recommended stabilization time is for most applications:

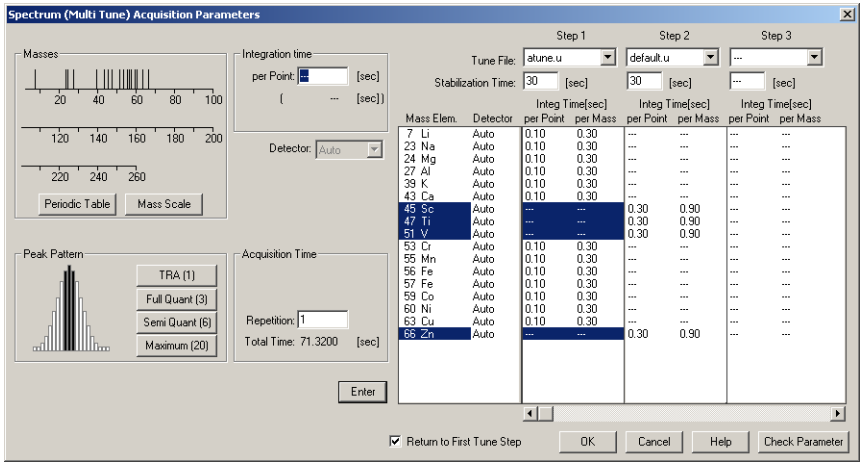
<30 seconds from cool plasma to hot
<30 seconds from hot to cool plasma

Continue 8 and 9 as you select tune files.

11 Select a tune step for each element.

Refer to page 5-50 "Highlight the mass (see below) by clicking on the masses while holding down the Ctrl key".



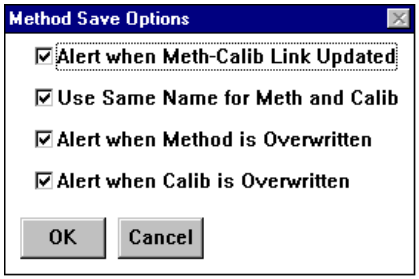


NOTE

Recommended element to be run by cool plasma
: Li, Na, Mg, Al, K, Ca, Cr, Mn, Fe, Co, Ni, Cu
Others elements are recommend to be run by hot plasma

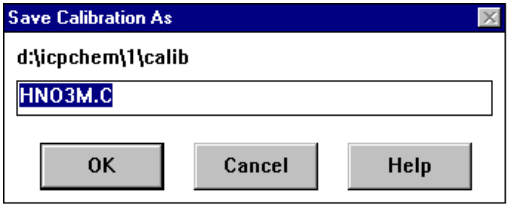
Click ***Check Parameter*** and confirm no errors reported.

12 Click OK.



Refer to Saving a Method and Calibration on page 5-87 in "*Agilent 7500 ICP-MS ChemStation Operator's Manual"*.

13 Enter calibration name, then click OK



Creating this method is now complete.

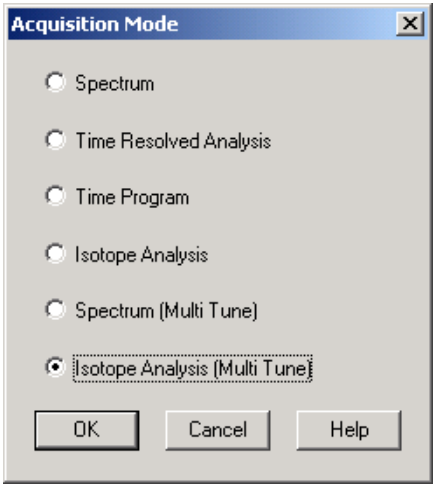
Isotope Analysis [Multi Tune]

Isotope Analysis [Multi Tune] is similar to Spectrum Analysis [Multi Tune]. The difference is that the dwell time per mass is 10 times shorter. This mode should be used when precision of isotopic information (isotopic ratio and isotope dilution) is required (e.g. radioactive isotope analysis for determining age, etc.).

To set the Acquisition Mode to Isotope Analysis [Multi Tune], complete the following steps:

1 **Click the *Isotope Analysis [Multi Tune]* button.**

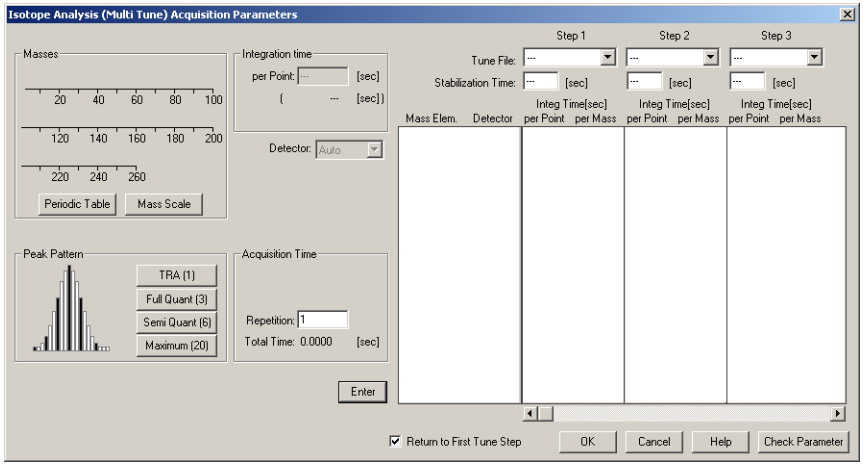
The button is marked and the selected mode is outlined.



Acquisition Mode Dialog Box---Isotope Analysis [Multi Tune]

2 **Click *OK*.**

ChemStation sets Isotope Analysis as the acquisition mode for the method and the ***Isotope Analysis [Multi Tune] Acquisition Parameters*** dialog box appears



Isotope Analysis [Multi Tune] Acquisition Parameters Dialog Box

Setting Parameters for Isotope Analysis [Multi Tune]

Because Isotope Analysis [Multi Tune] is similar to quantitative analysis in Spectrum Analysis [Multi Tune], setting the acquisition parameters is the same. Refer to "Setting Parameters for Quantitative Analysis" on page 5-25 in this chapter to set acquisition parameters for Isotope Analysis. Refer to "Setting Spectrum [Multi Tune] Acquisition Parameters" on page 5-50 in this chapter for Multi Tune Parameter Setting.

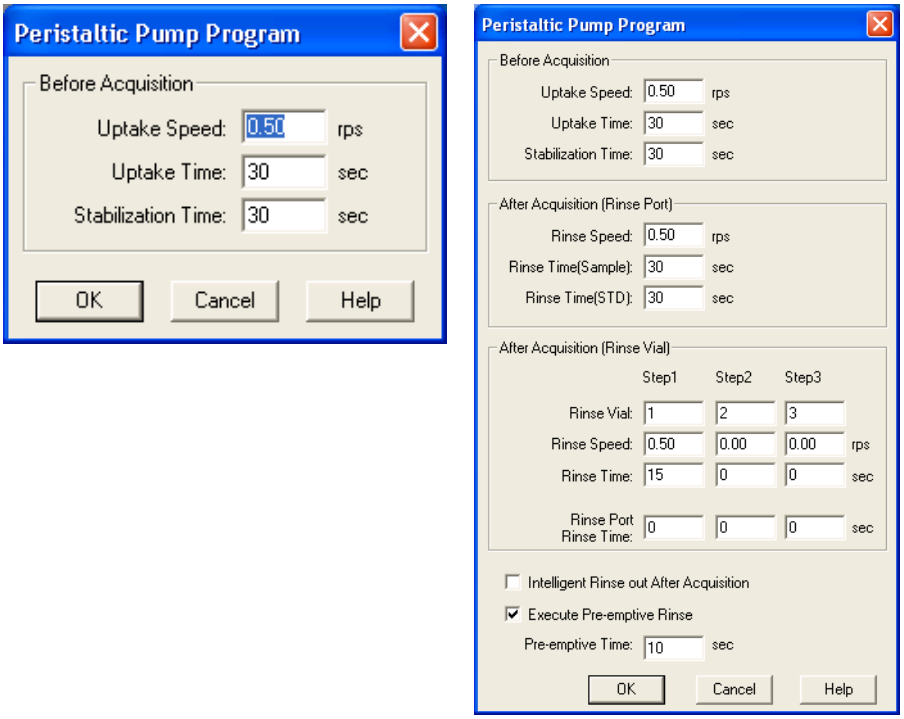
NOTE

The ***Full Quant (3)*** in the Peak Pattern (left bottom) box is better than 1 point on the Peak Pattern for Isotope Analysis.

Setting the Peristaltic Pump Program

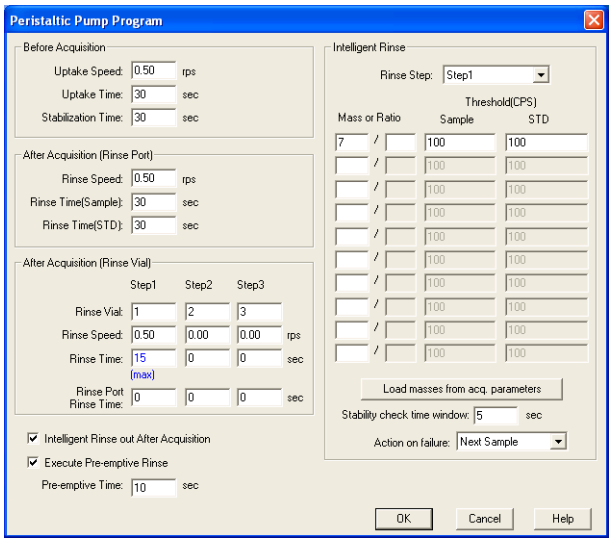
You can determine the speed at which the peristaltic pump runs, and set a delay to allow for the time it takes for the sample to reach the plasma and stabilize. If the ASX-500 or ASX-100 is set as the autosampler in the ICP-MS Configuration, then the window to set up the time and pump speed during the rinse is added.

Before the acquisition of data, the sample uptake runs. During uptake, the pump speed increases to the set value. After Uptake Time has elapsed, the pump speed goes to the value to be set in the Tuning Parameters, and pumps at that value for the duration of the stabilization time. After stabilization, data acquisition begins. If an autosampler is configured, you can set post-acquisition rinse parameters.



Peristaltic Pump Program Dialog Box
(left: without the autosampler, right: with the autosampler)

ChemStation allows you to set Intelligent Rinse for the post-acquisition rinsing. The probe rinse normally begins after the termination of acquisition. However, you can also start the rinse before acquisition ends.



Peristaltic Pump Program Dialog Box (Intelligent Rinse)

To set the peristaltic pump program, complete the following steps:

1 **Set the uptake speed by double-clicking or dragging on the *Uptake Speed* text box and typing a value in revolutions per second. (0.50 maximum)**

The value set determines the speed at which the pump takes the sample into the nebulizer.

NOTE When using the sample pump, the maximum speed is 1.0 rps.

2 **Set the uptake time by double-clicking or dragging on the *Uptake Time* text box and typing a value in seconds.**

The uptake time is the time allowed for the sample to reach the nebulizer.

3 **Set the stabilization time by double-clicking or dragging on the *Stabilization Time* text box and typing a value in seconds.**

The stabilization time is the time allowed for the pump and signal to stabilize before acquisition begins.

4 To use the autosampler, enter the settings for the post-acquisition probe rinse in the *After Acquisition (Rinse Port)* section.

Rinse solution in the rinse port of the autosampler is taken up into the nebulizer.

NOTE When using the Peristaltic Pump2, the maximum speed is 1.0 rps.

5 To use the autosampler, enter the settings for the post-acquisition rinse in the *After Acquisition (Rinse Vial)* section.

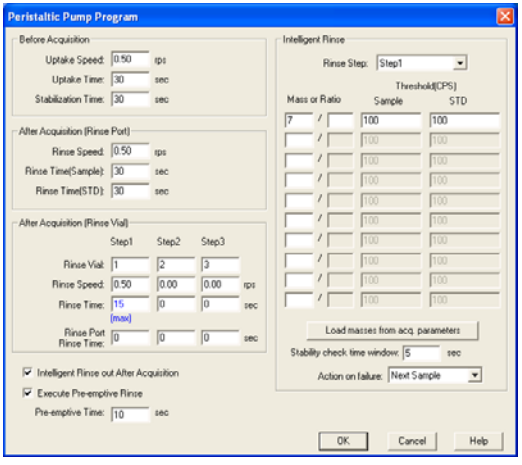
Rinse Speed, Rinse Time and the Rinse Vial that will be used for post-acquisition rinse. Each time the sample is switched, the rinse operation will be conducted using the solution in the specified rinse vial.

6 To initiate Intelligent Rinse after acquisition, enter the following settings.

In Intelligent Rinse, rinsing will continue until masses within the sample decrease to the set threshold (CPS) or lower. Enter the settings in ***Mass***, ***Threshold, Stability check time Window***, and ***Action on Failure***.

1) Select the ***Intelligent Rinse out After Acquisition*** check box.

The dialog box expands and shows the Intelligent Rinse setting items.



Peristaltic Pump Program Dialog Box

2) Select a rinse step from the Rinse Step drop-down list.

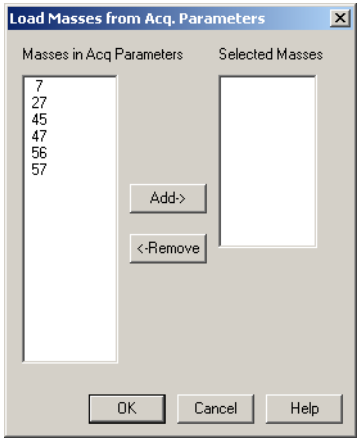
Selecting ***Rinse Port*** will cause the indicators in the Rinse Time (Sample) and Rinse Time (STD) text boxes to change to blue. Selecting Step No. will cause the indicator in the Rinse Time text box to change to blue. < max > will appear next to both text boxes to indicate the rinse time limit.

3) To input mass data directly, enter mass values in the left-side text boxes under "Mass or Ratio."

4) To load masses from acquisition parameters, click ***Load Masses from Acq. Parameters***.

The ***Load Masses from Acq. Parameters*** dialog box will appear. Select mass data. Up to 10 masses can be selected. Choose the desired mass data in the ***Masses in Acq. Parameters*** list box and click ***Add->*** to select them as the masses for Intelligent Rinse. To cancel a selection, select the desired masses in the ***Selected Masses*** list box and click ***<- Remove***. Selections may also be added or removed to or from a list by double-clicking them.

Click ***OK*** to close the ***Load Masses from Acq. Parameters*** dialog box and load the selected masses.



Load Masses from Acq. Parameters Dialog Box

5) To set a strength ratio, enter mass values in the right-side boxes under "Mass or Ratio."

6) For a standard sample, set the threshold (CPS) of the count value of the mass in the ***Sample*** text box in the Threshold (CPS) section.

7) In the case of a calibration standard, set the threshold (CPS) of the count value of the mass in the ***STD*** text box.

8) Set a stability confirmation time setting (1 to 300 sec) in the ***Stability check time Window*** text box. The moving average will be obtained in the set time period.

9) Select the process to be executed when the threshold value is not reached at the set rinse time from the Action on Failure drop-down list.

7 Do the following to start the rinse before the end of acquisition:

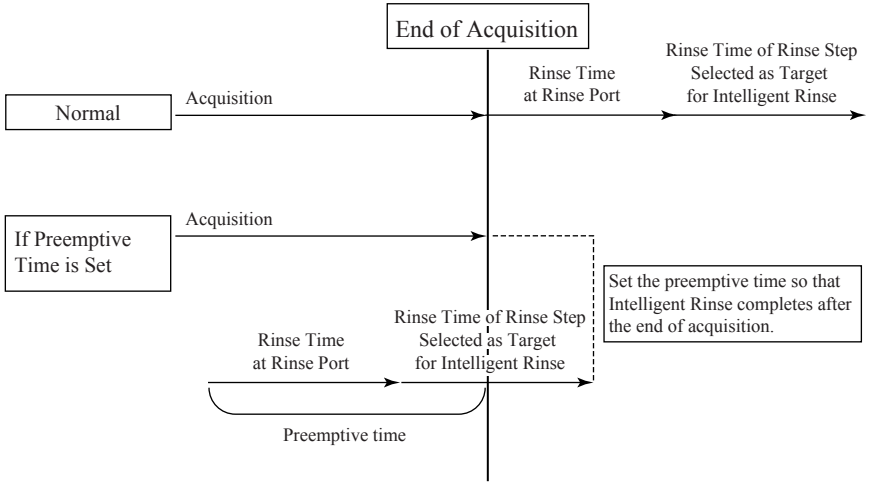
1) Select the ***Execute Pre-emptive Rinse*** checkbox.

2) In the ***Pre-emptive Time*** text box, set the number of seconds before the end of acquisition at which you wish to start the rinse.
If you proceed with preemptive rinse with an improper preemptive time, the rinse solution may be introduced during acquisition. Be very careful when specifying preemptive times. Keep in mind that performing Intelligent Rinse through preemptive rinse will prevent monitoring of threshold values during acquisition. To proceed with Intelligent Rinse, set a preemptive time so that the rinse step selected as the Intelligent Rinse target ends after acquisition ends.

NOTE

- To repeat the acquisition, set a time shorter than the individual repeated data acquisition times.
- For Multi Tune mode, set a time shorter than the last tune step. A tune step with a long acquisition time at the end can lengthen preemptive times.

If Intelligent Rinse is selected for Step 1



- Preemptive Time < Rinse Time in After Acquisition (Rinse Port) + Rinse Time in Step 1 in After Acquisition (Rinse Vial)

NOTE

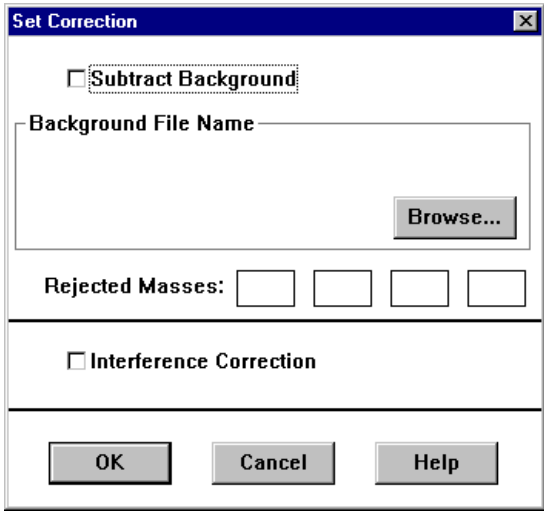
If you start the rinse before the end of acquisition, the Rinse Speed setting will be disabled until acquisition is complete.

8 To accept the values you set for the peristaltic pump program, click *OK*.

The ***Set Correction*** dialog box appears.

Setting Corrections for the Method

ChemStation enables you to set corrections for both background and isobaric interferences. Correcting for background interference subtracts the background signal from the sample signal acquired with the same method. Correction for interference uses equations to adjust for interference between masses.



Set Correction Dialog Box

To use the correction features ChemStation provides, complete the following steps:

1 Click *Subtract Background*.

2 Click *Browse*.

The ***Select Data File*** Dialog box appears, showing the path of the data file directory and a list of data files.

3 Select a file name from the list by clicking it and then clicking *OK*.

ChemStation subtracts the data in the selected file from all other data files acquired with the same method.

4 Select the box, *Rejected Masses* and enter the masses for which background spectrum you do not want subtracted.

The background spectrum for the masses you edited in the Rejected Masses box will not be subtracted. It is used when you do not want to subtract the spectrum for specific masses; for example the spectrum for internal standards.

NOTE

Subtract Background is used to subtract a background spectrum from a sample spectrum. When Subtract Background is selected, the abundance values for each mass in the background spectrum are subtracted from the abundance values of the corresponding mass in the sample spectrum. Subtract Background is different from *Blank Subtraction* available in Data Analysis where the blank quantitative results are subtracted from sample quantitative results.

5 Click *Interference Correction*.

Any interference correction equations stored in a given method will be used to correct all data acquired by that method, if Interference Correction is selected.

6 To set the corrections you have chosen, click *OK*.

The ***Select Reports*** dialog box appears.

Selecting Reports

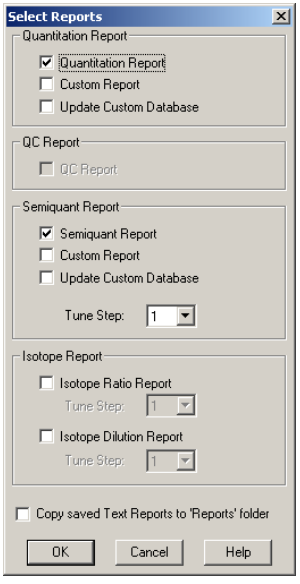
ChemStation gives you several choices for generating reports as part of the method. You can generate reports to the screen, to the printer, or to a file that you specify. ChemStation also enables you to generate custom reports and databases that can be updated automatically when the method runs. Generating reports with the method is optional. If you do not want to generate a report, make sure nothing is selected in the Select Report dialog box and click ***OK***.

The ChemStation software provides default templates for custom reports and databases, or you can design your own templates. For information about creating your own custom report template, see Chapter 11, "Creating Custom Reports."

In the **Tune Step**, select the tune step which is used. If you do not use the Multi Tune, select **1**.

CAUTION Select the same tune step as used in the acquisition parameter. If the selected tune step is different, an error occurs.Select Reports Dialog Box

NOTE If your QC configuration does not include any QC parameters, the QC Report section will be grayed out.



Select Reports Dialog Box

To select the type of report you want to generate when the quantitation method runs, complete the following steps:

1 **Select a report format in the *Quantitation Report* area.**

To generate the default quantitation report, select the box for ***Quantitation Report*** .

To generate a custom report template for quantitative analysis when the method runs, click the ***Custom Report*** box.

To update a custom report database for quantitative analysis when the method runs, click the ***Update Custom Database*** check box.

2 **To produce a batch file of the report, select the check box *Copy saved Text Reports to 'Reports' folder.***

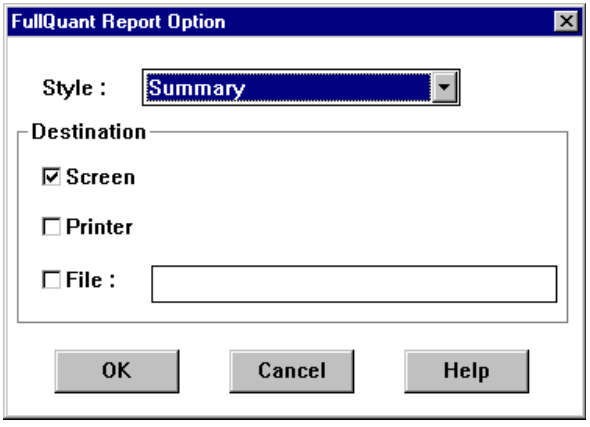
If the setting to save reports as a file is selected as described in step 4, the same information will be saved in a file named "fq_FILE NAME.tex" in the Reports directory.
This setting is used to allow an external applications to obtain reports periodically.

3 **Click *OK*.**

4 **Set the generating style of the quantitation report as follows:**

To generate the default quantitation report, complete the following steps:

a) The *FullQuant Report Option* dialog box appears.



FullQuant Report Option Dialog Box

b) To see the list of default report styles for FullQuant, click on the arrow next to Style.

Three default report styles are available. For more information about report styles, please refer to appropriate sections of Chapters 12.

c) Select an item from the list by clicking it.

d) Select the destination for the report by clicking the appropriate boxes and click *OK*.

You can click one or more of the following boxes:

- Screen

  Generates the report and displays it on the ChemStation screen.

- Printer

  Generates the report and sends it to the ChemStation printer. You must select this destination for a detailed report.

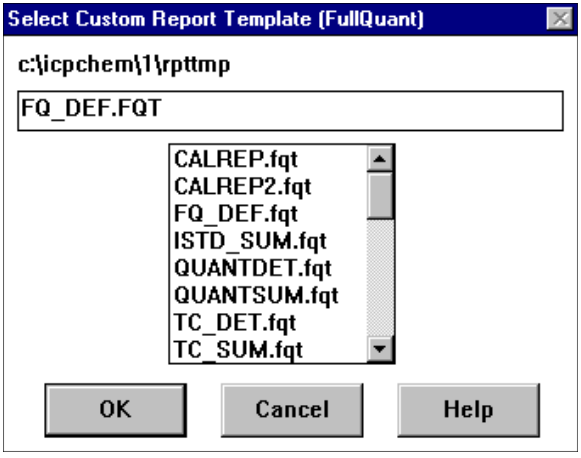
- File

  Generates the report and saves it as a file. Use the dialog box to give this file a name.

For more information about FullQuant report output, refer to Chapter 12, "Generating a Quantitative Report".

To generate a custom report template for quantitative analysis when the method runs, complete the following steps:

a) The *Select Custom Report Template (FullQuant)* dialog box appears.



Select Custom Report Template (FullQuant) Dialog Box

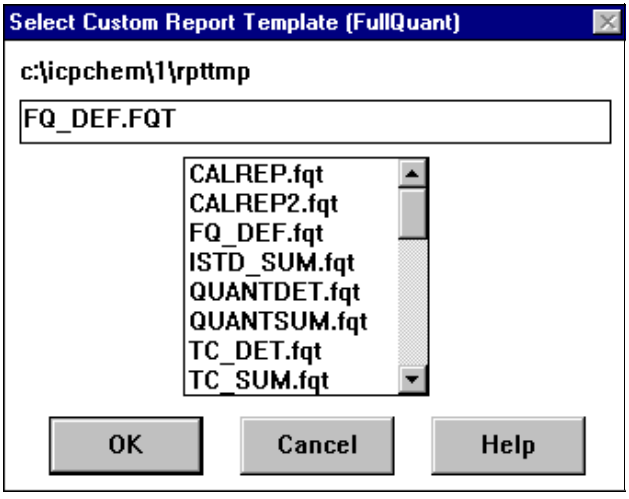
b) Select the custom report template (*.fqt* extension) in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

For more information about custom reports, refer to Chapter 11, "Creating Custom Reports".

To update a custom report database for quantitative analysis when the method runs, complete the following steps:

a) The *Select Custom Report Database (FullQuant)* dialog box appears.



Select Custom Report Database (FullQuant) Dialog Box

b) Select the custom report database (.fqd extension) in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

For more information about the custom database, refer to Chapter 11, "Creating a Custom Report Database".

After setting all selected report options, the screen goes on to Edit Analysis Parameters, explained later in this chapter.

To generate a QC Report, complete the following steps:

NOTE If your QC configuration does not include any QC parameters, the QC Report section will be grayed out.

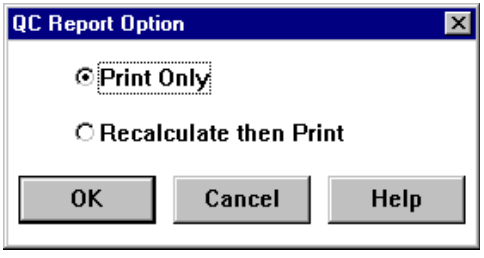
1 **Click the *QC Report* box and click *OK*.**

2 **To produce a batch file of the report, select the check box *Copy saved Text Reports to 'Reports' folder.***

If the setting to save reports as a file is selected as described in step 4, the same information will be saved in a file named "qc_FILE NAME.tex" in the Reports directory.
This setting is used to allow an external applications to obtain reports periodically.

3 **Click *OK*.**

The ***QC Report Options*** dialog box appears.



QC Report Option Dialog Box

4 **Select either *Print Only* or *Recalculate then Print* for QC Report, and click *OK*.**

For more information about the QC report, refer to the appropriate sections of the Intelligent Sequence Manual.

After setting all selected report options, the screen goes on to Edit Analysis Parameters, explained later in this chapter.

To select the type of report you want to generate when the semiquant method runs, complete the following steps:

1 **Select a report format in the *Semiquant Report* area.**

To generate the default semiquant report, click the ***Semiquant Report*** box and click ***OK***.

To generate a custom report template for semiquantitative analysis when the method runs, click the ***Custom Report*** box and click ***OK***.

To update a custom report database for semiquantitative analysis when the method runs, click the ***Update Custom Database*** box and click ***OK.***

2 **To produce a batch file of the report, select the check box *Copy saved Text Reports to 'Reports' folder.***

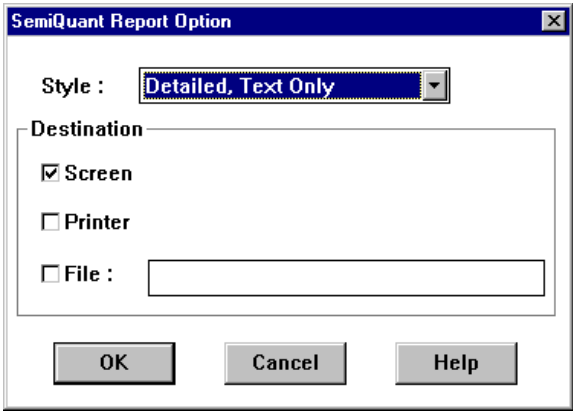
If the setting to save reports as a file is selected as described in step 4, the same information will be saved in a file named "sq_FILE NAME.tex" in the Reports directory.
This setting is used to allow an external applications to obtain reports periodically.

3 **Click *OK*.**

4 **Set the generating style of the semiquant report as follows:**

To generate the default semiquant report, complete the following steps:

a) The *Semiquant Report Option* dialog box appears.



SemiQuant Report Option Dialog Box

b) To see the list of default report styles for SemiQuant, click on the arrow next to Style.

Two default report styles are available. For more information about report styles, please refer to appropriate sections of Chapters 13.

c) Select an item from the list by clicking it.

d) Select the destination for the report by clicking the appropriate boxes and click *OK*.

You can click one or more of the following boxes:

- Screen

Generates the report and displays it on the ChemStation screen.

- Printer

Generates the report and sends it to the ChemStation printer. You must select this destination for a detailed report.

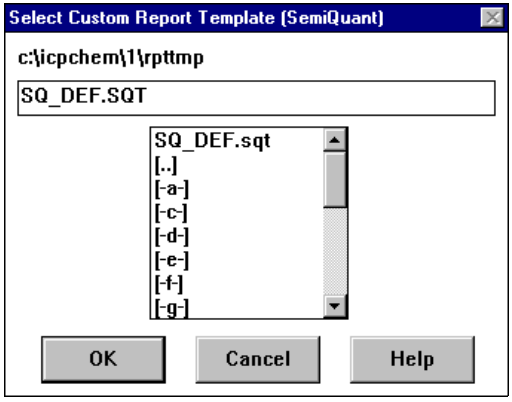
- File

Generates the report and saves it as a file. Use the dialog box to give this file a name.

For more information about SemiQuant report output, refer to Chapter 13, "Generating a Semiquantitation Report".

To generate a custom report template for semiquantitative analysis when the method runs, complete the following steps:

a) The Select Custom Report Template (SemiQuant) dialog box appears.



Select Custom Report Template (SemiQuant) Dialog Box

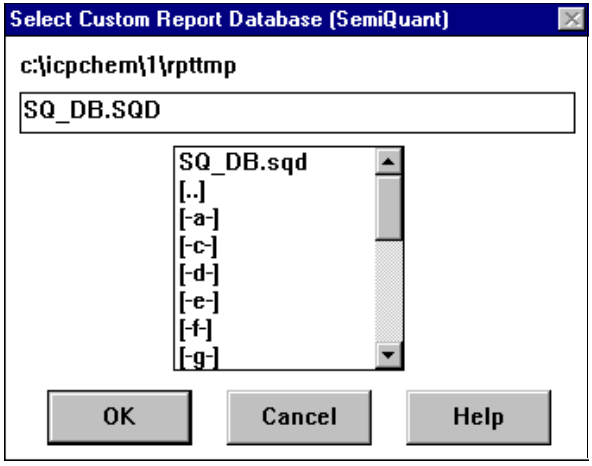
b) Select the custom report template (*.sqt* extension) in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

For more information about custom reports, refer to Chapter 11, "Creating Custom Reports".

To update a custom report database for semiquantitative analysis when the method runs, complete the following steps:

a) The Select Custom Report Database (SemiQuant) dialog box appears.



Select Custom Report Database (SemiQuant) Dialog Box

b) Select the custom report database (*.sqd* extension) in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

For more information about the custom database, refer to Chapter 11, "Creating a Custom Report Database".

After setting all selected report options, the screen goes on to Edit Analysis Parameters, explained later in this chapter.

To generate an Isotope Ratio Report, complete the following steps:

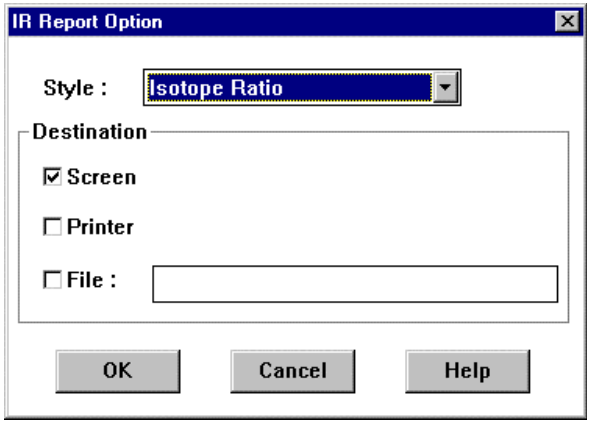
1 **Select the *Isotope Ratio Report* check box in the *Isotope Report* area.**

2 **To produce a batch file of the report, select the check box *Copy saved Text Reports to 'Reports' folder.***

If the setting to save reports as a file is selected as described in step 4, the same information will be saved in a file named "ir_FILE NAME.tex" in the Reports directory.
This setting is used to allow an external applications to obtain reports periodically.

3 **Click *OK*.**

The ***IR Report Options*** dialog box appears.



IR Report Option Dialog Box

4 **To see the list of default report styles for Isotope Ratio, click on the arrow next to Style.**

Two default report styles are available. For more information about report styles, please refer to appropriate sections of Chapters 14.

5 **Select an item from the list by clicking it.**

6 **Select the destination for the report by clicking the appropriate boxes and click *OK*.**

You can click one or more of the following boxes:

- Screen

  Generates the report and displays it on the ChemStation screen.

- Printer

  Generates the report and sends it to the ChemStation printer.

- File

  Generates the report and saves it as a file. Use the dialog box to give this file a name.

For more information about the Isotope Ratio report, refer to Chapter 14, "Generating an Isotope Ratio Analysis Report".

After setting all selected report options, the screen goes on to Edit Analysis Parameters, explained later in this chapter.

To generate an Isotope Dilution Analysis Report, complete the following steps:

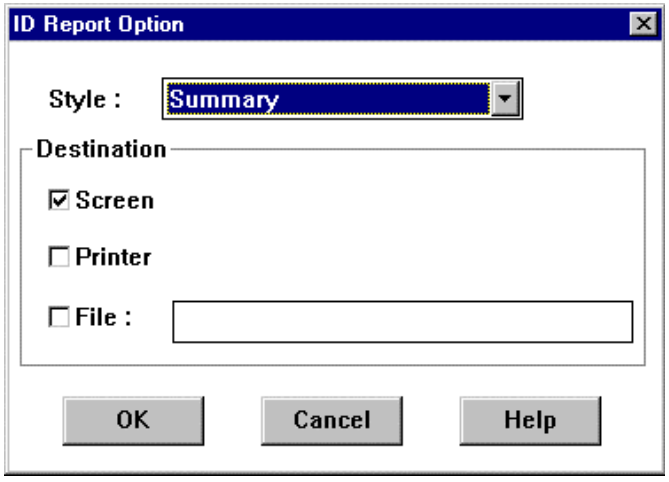
1 **Select the *Isotope Dilution Report* check box in the *Isotope Report* area.**

2 **To produce a batch file of the report, select the check box *Copy saved Text Reports to 'Reports' folder.***

If the setting to save reports as a file is selected as described in step 4, the same information will be saved in a file named "id_FILE NAME.tex" in the Reports directory.
This setting is used to allow an external applications to obtain reports periodically.

3 **Click *OK*.**

The ***ID Report Options*** box appears.



ID Report Option Dialog Box

4 **To see the list of default report styles for Isotope Ratio, click on the arrow next to Style.**

Two default report styles are available. For more information about report styles, please refer to appropriate sections of Chapters 15.

5 **Select an item from the list by clicking it.**

6 **Select the destination for the report by clicking the appropriate boxes and click *OK*.**

You can click one or more of the following boxes:

- Screen

  Generates the report and displays it on the ChemStation screen.

- Printer

  Generates the report and sends it to the ChemStation printer.

- File

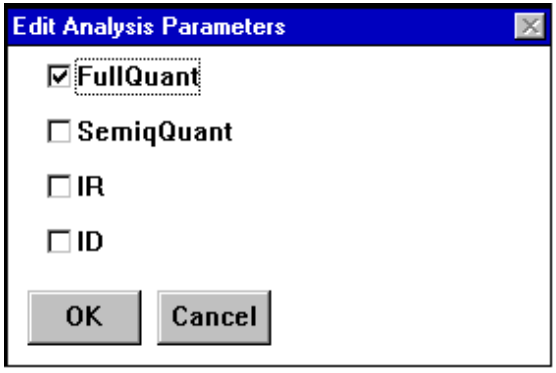
  Generates the report and saves it as a file. Use the dialog box to give this file a name.

For more information about the Isotope Dilution Analysis report, refer to Chapter 15, "Generating an Isotope Dilution Analysis Report"

After setting all selected report options, the screen goes on to Edit Analysis Parameters, explained later in this chapter.

Selecting the Edit Analysis Parameters

After you set the report options, the ChemStation enables you to edit the analysis parameters for FullQuant, SemiQuant, Isotope Ratio (IR) and Isotope Dilution (ID). Please select the items you want to edit and click ***OK***.



Edit Analysis Parameters Dialog Box

If you select FullQuant and click ***OK***, the ***Edit Calibration Parameters*** dialog box appears, followed by the ***Blank Conc. Subtraction (FullQuant)*** dialog box.

If you select SemiQuant and click ***OK***, the ***SemiQuant Parameters*** dialog box appears, followed by the ***Internal Standard Correction (Normal Mode)*** and ***Blank Conc. Subtraction (SemiQuant)*** dialog box.

If you select IR and click ***OK***, the ***IR Parameters*** dialog box appears.

If you select ID and click ***OK***, the ***ID Parameters*** dialog box appears.

For more information about editing these analysis parameters, please refer to the appropriate sections of Chapters 12-15.

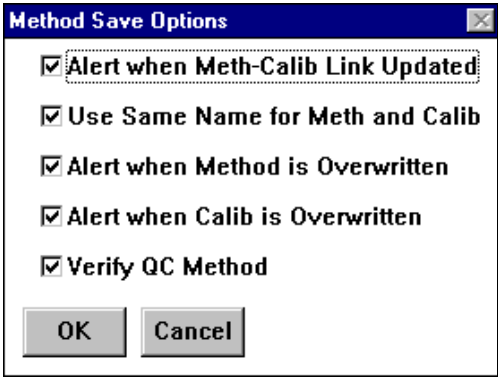
When you have finished editing the analysis parameters, either the ***QC Database*** dialog box or the ***QC Tune Criteria Editor*** dialog box appears.

NOTE If your QC configuration does not include any QC parameters, the QC database or the QC Tune Criteria Editor dialog box will not appear.

Editing QC Parameters

For more information about editing QC Database and QC Tune Criteria parameters, please refer to the appropriate sections of the Intelligent Sequence Manual.

When you have finished editing the QC parameters, the ***Method Save Options*** dialog box appears.



Method Save Options Dialog Box

NOTE If your QC configuration does not include any QC parameters, the Verify QC Method option will not appear.

Saving a Method and Calibration

To save the selected method and calibration just created, complete the following steps:

1 **Select the Method Save Options by clicking on the boxes for each option.**

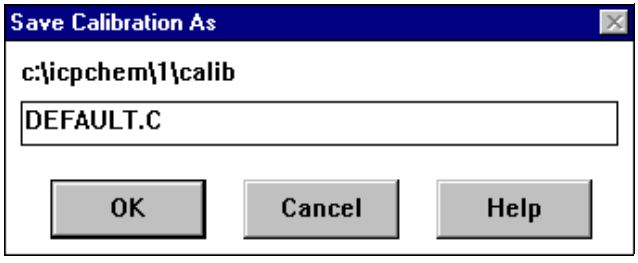
There are 5 options available on the Method Save Options dialog box:

- Click the ***Alert when Meth-Calib Link Updated*** box if you want the ChemStation to alert you with a message when the Method and Calibration link is changed
- Click the ***Use Same Name for Meth and Calib*** box if you want to save the method and calibration with the same name.
- Click the ***Alert when Method is Overwritten*** box if you want the ChemStation to alert you with a message when an existing method will be overwritten.
- Click the ***Alert when Calib is Overwritten*** box if you want the ChemStation to alert you with a message when an existing calibration will be overwritten.
- Click the ***Verify QC Method*** box if you want ChemStation to verify the QC method.

2 **After selecting the options, click *OK*.**

If you have selected the ***Verify QC Method*** option, ChemStation will verify the QC method.

After verifying the QC method, the ***Save Calibration As*** dialog box appears.



Save Calibration As Dialog Box

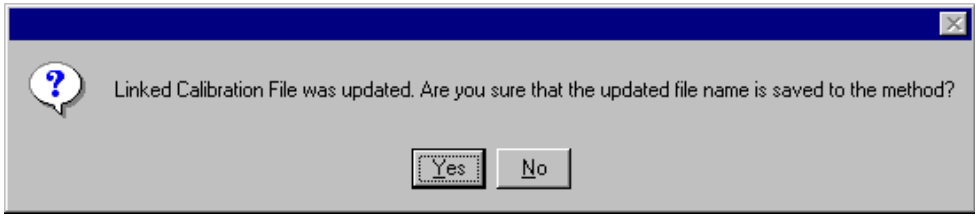
3 Give your new Calibration a name and click *OK*.

The name can be up to eight characters long. Do not use the following characters in the method name:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (=)	Space ( )
Colon (:)	Quotation mark (“)	

ChemStation automatically adds a.c to the name to indicate that it is a calibration file. The calibration is saved in c:\icpchem\1\calib.

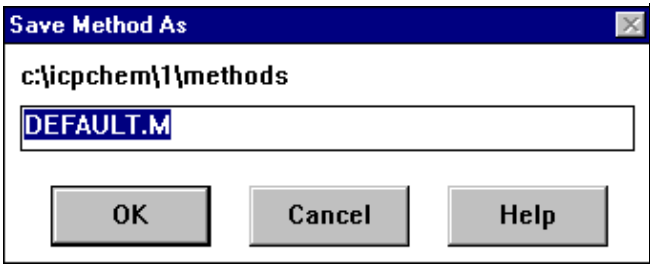
If you have selected the ***Alert when Meth-Calib Link Updated*** option and have changed the method-calibration link, the alert message appears.



Alert Message

Click ***Yes*** to continue, or ***No*** to cancel method save.

If you click ***Yes***, the ***Save Method As*** dialog box appears.



Save Method As Dialog Box

4 Give your new Method a name and click *OK*.

The name can be up to eight characters long. Do not use the following characters in the method name:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (=)	Space ( )
Colon (:)	Quotation mark (“)	

ChemStation automatically adds a ***.m*** to the file name to indicate that it is a method file. The method is saved in c:\icpchem\1\methods.

If you had selected the ***Use Same Name for Meth and Calib*** option, the method will automatically be saved under the same name you specified for the calibration and the ***Save Method As*** dialog box will not appear.

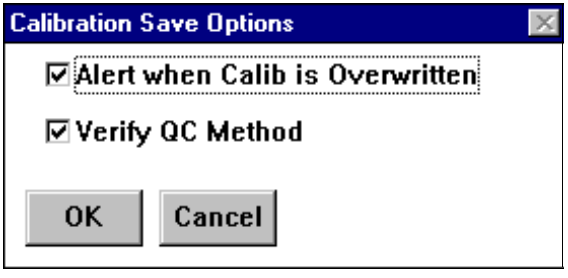
NOTE

You can save your method and calibration after modifying the method and/or calibration. Select ***Methods>>Save*** from the ***ICP-MS Top*** window. The ***Method Save Options*** dialog box will appear. Continue with the steps as described earlier in this section to save your method and calibration.

For changes only made in your calibration, you can save those changes in the calibration file. To save your calibration, complete the following steps:

1 Select *Methods>>Save Calibration* from the *ICP-MS Top* window.

The ***Calibration Save Option*** dialog box appears.



Calibration Save Options Dialog Box

2 Select the Calibration Save Options by clicking on the boxes for each option.

There are 2 options available on the ***Calibration Save Options*** dialog box:

- Click the ***Alert when Calib is Overwritten*** box if you want the ChemStation to alert you with a message when an existing calibration will be overwritten.
- Click the ***Verify QC Method*** box if you want ChemStation to verify the QC method.

3 After selecting the options, click *OK*.

If you have selected the ***Verify QC Method*** option, ChemStation will verify the QC method.

After verifying the QC method, the ***Save Calibration As*** dialog box.

4 Give your new Calibration a name and then click *OK*.

Modifying a Method

ChemStation enables you to easily modify specific parameters in a method without having to edit the entire method. You can edit the method information, data acquisition parameters, data analysis sections, and QC parameters of the method separately. To do so, you must first load the method and calibration you want to modify into memory. When you have modified the method, you must save the changes to the current method-calibration or save the changed method-calibration as a new file.

The following sections explain how to load a method and calibration, and how to modify and save the method information, data acquisition parameters data analysis sections, and QC parameters of a method.

Loading a Method and Calibration

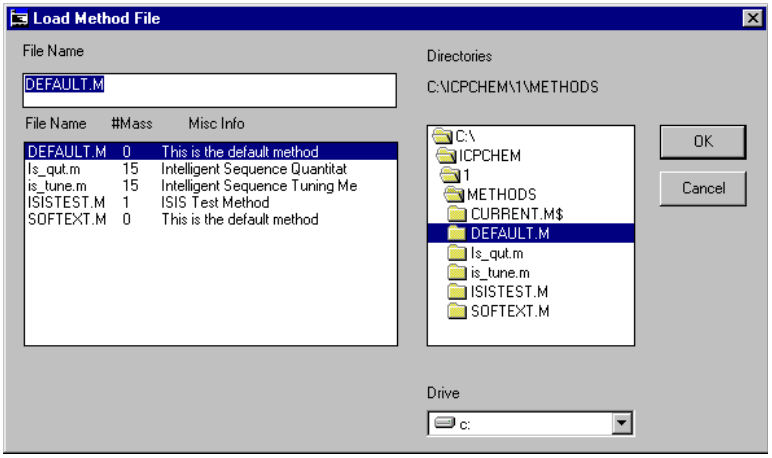
Before you can modify a method, you must first load it into memory. To do so, complete the following steps:

1 ***Select Top>>Methods.***

The Methods menu appears.

2 **Select *Methods>>Load* from the ICP-MS Top window.**

The ***Load Method File*** dialog box appears.



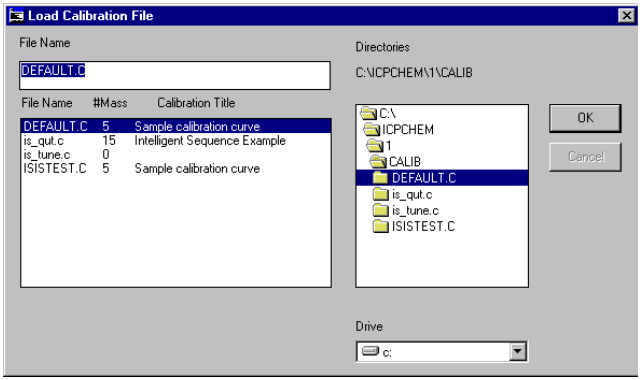
Load Method Dialog Box

3 **To load the method, select it in one of the following ways:**

- Type the name of the method and click ***OK***. If the directory path in the Load Method dialog box is incorrect, you must select the correct directory path.
- Double-click the method in the displayed list.

The three most recently loaded methods are listed as numbered menu items, with the first entry corresponding to the most recently loaded method. Selecting any one of these numbered menu items will cause that method to be loaded.

The ***Load Calibration File*** dialog box appears.



Load Calibration File Dialog Box

4 To load the calibration, select it in one of the following ways:

- Type the name of a calibration and click ***OK***. If the directory path in the Load Calibration dialog box is incorrect, you must type the directory path as well as the name of the calibration.
- Double-click a calibration in the displayed list.

The method and calibration are loaded into memory and ChemStation displays the method name and calibration name in the title bar of the ICP-MS Top window. You are now ready to modify the method.

NOTE You can load the calibration file to link with the currently loaded method by selecting ***Methods>>Load Calibration***.

Modifying the Method Information

If you do not want to change the method parameters, but want to add comments about the method or change the conditions for running and saving the method, you can edit the method information section of the method separately. To do so, complete the following steps:

1 **Select *Top>>Methods*.**

The Methods menu appears.

2 **Select *Methods>>Edit Method Information*.**

The ***Method Information*** dialog box appears. For more information about this dialog box, see "Entering the Method Information" in this chapter.

3 **Make the changes you want and click *OK*.**

The dialog box disappears, but changes are not recorded to the method until you save the method.

4 **To save the method, select *Methods>>Save*.**

The ***Method Save Options*** dialog box appears. Save the method as described in Saving a Method and Calibration earlier in this chapter.

NOTE

You can also modify this section of a method by selecting ***Method>>Edit Entire Method***. Click only the ***Method Information*** box in the Edit Entire Method dialog box, and click ***OK***. When the Method Information dialog box appears, make the changes you want and click ***OK***. Then, complete the saving steps as described in Saving a Method and Calibration earlier in this chapter.

Modifying Data Acquisition Parameters

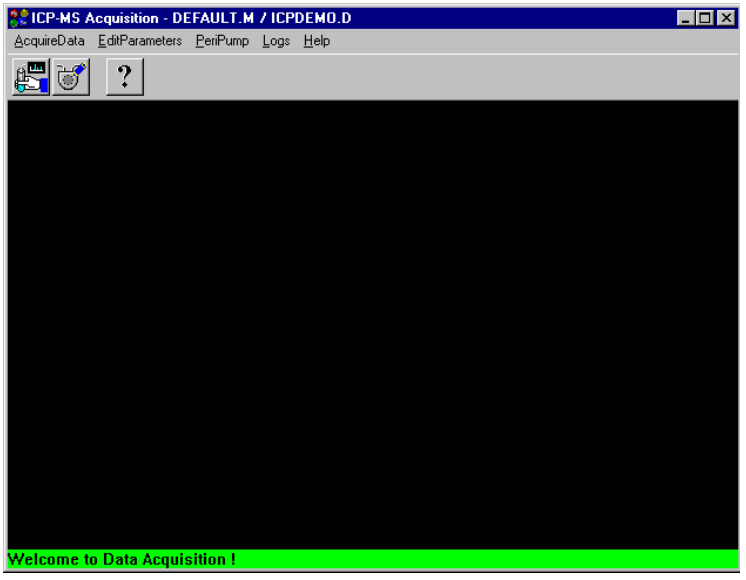
If you want to change only the data acquisition parameters of a method, ChemStation enables you to change the parameters from the ICP-MS Acquisition window. To do so, complete the following steps:

1 Select *Top>>Acquire Data*.

The Acquire Data menu appears.

2 Select *Acquire Data>>Main Panel*.

The ***ICP-MS Acquisition*** window appears.



ICP-MS Acquisition Window

3 To change the acquisition mode, complete the following steps:

a) Select *Edit Parameters>>Set Mode*.

The ***Acquisition Mode*** dialog box appears.

b) Click on the button of an acquisition mode and then click *OK*.

The dialog box closes and ChemStation returns to the ICP-MS Acquisition window. The acquisition mode is set, but it is not permanently recorded to the

method until you save the method.

4 To change acquisition parameters, select *Edit Parameters>>Set Parameters*.

Set Parameters opens ***Acquisition Parameters*** dialog box, where represents the selected acquisition mode. For more information about the parameters that appear in the Acquisition Parameters dialog box, see the appropriate section earlier in this chapter.

NOTE When setting a different integration time for each mass, you must click ***Enter*** after you type a new value in the integration time text box. When setting the detector mode, you also need to click ***Enter*** to input your choice.

5 Click *OK* to set the changes you make to the acquisition parameters.

The changes are set, but they are not permanently recorded to the method until you save the method.

6 To change the peristaltic pump program, complete the following steps:

1) Select *PeriPump>>Set PeriPump Program*.

The ***Peristaltic Pump Program*** dialog box appears.

2) Double-click or drag on the text box that corresponds to the parameter you want to change.

Type in the new value for the parameter.

3) Click *OK*.

The dialog box closes and ChemStation returns to the ICP-MS Acquisition Window. The Peristaltic Pump Program is set, but it is not permanently recorded to the method until you save the method.

7 To save the method, complete the following steps.

You must close the ICP-MS Acquisition window before you can save the method.

1) Double-click the Control Box Menu in the top left corner of the ICP-MS Acquisition window.

The ICP-MS Acquisition window closes, and ChemStation returns to the Top window.

2) Select *Top>>Methods*.

The Methods menu appears.

3) Select *Methods>>Save*.

The ***Method Save Options*** dialog box appears. Save the method as described in Saving a Method and Calibration earlier in this chapter.

NOTE

You can also modify this section of a method by selecting ***Method>>Edit Entire Method***. Click only the ***Acquisition*** box in the Edit Entire Method dialog box, and click ***OK***. When the dialog box appears, make the changes you want and click ***OK***. Then, complete the saving steps as described in Saving a Method and Calibration earlier in this chapter.

Printing a Summary of Method Acquisition Parameters

ChemStation enables you to print a summary of the acquisition parameters for each method you create. The summary also includes error messages that ChemStation generates if appropriate.

To print a summary of the acquisition parameters for a method, complete the following steps:

1 **Select *Top>>Acquire Data*.**

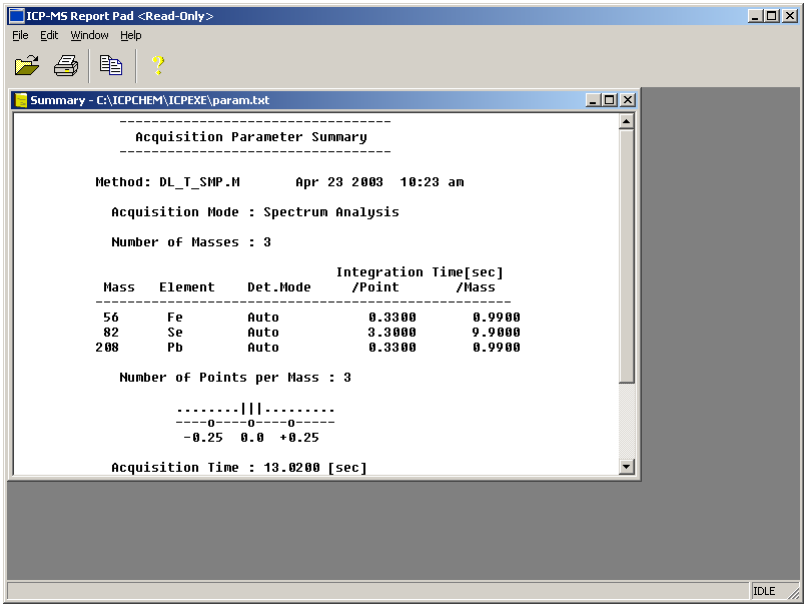
The Acquire Data menu appears.

2 **Select *Acquire Data>>Main Panel*.**

The ***ICP-MS Acquisition*** window appears.

3 **Select *Edit Parameters>>Print Summary*.**

The ***Param.txt - Report pad*** window appears.



Param.txt - Report Pad Window

4 **Select *File>>Print*.**

A summary of the acquisition parameters is printed out.

Modifying AMU Information

ChemStation enables you to modify the AMU Select File. The AMU Select File is the file where you can edit the isotopes to be pre-selected when you click an element push button on the periodic table.

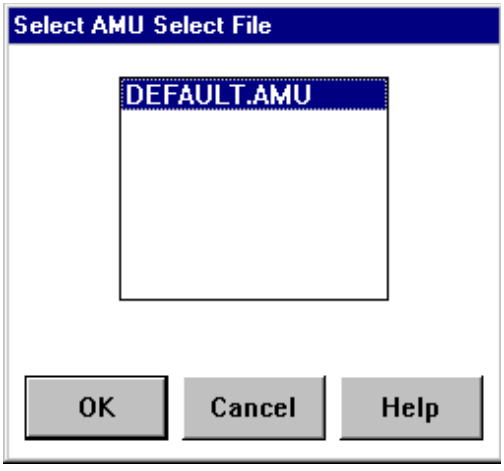
To edit and modify the AMU Select File, complete the following steps:

1 **Select *Top>>Acquire Data*.**

The Acquire Data menu appears.

2 **Select *Acquire Data>>Edit AMU Select File (.amu)*.**

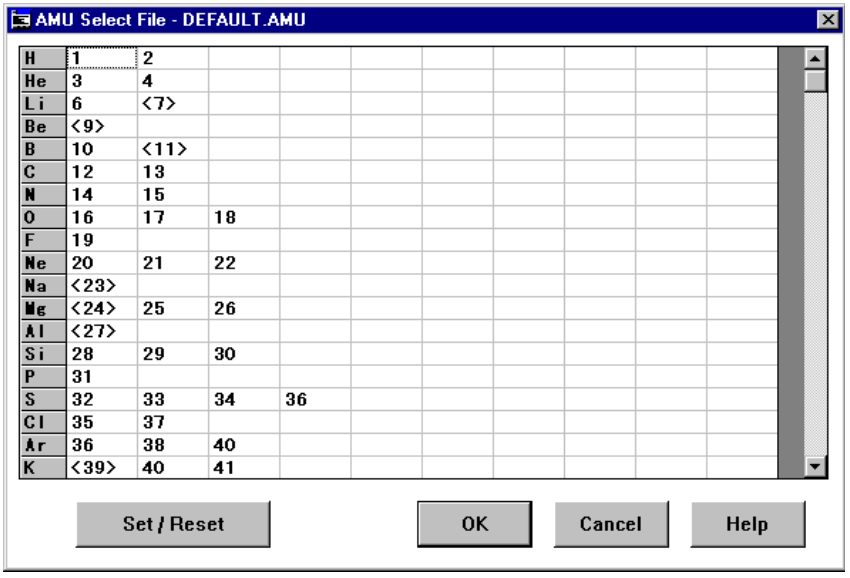
The ***Select AMU Select File*** dialog box appears.



Select AMU Select File Dialog Box

3 **Select a file and click *OK*.**

The AMU Select File that you selected appears.



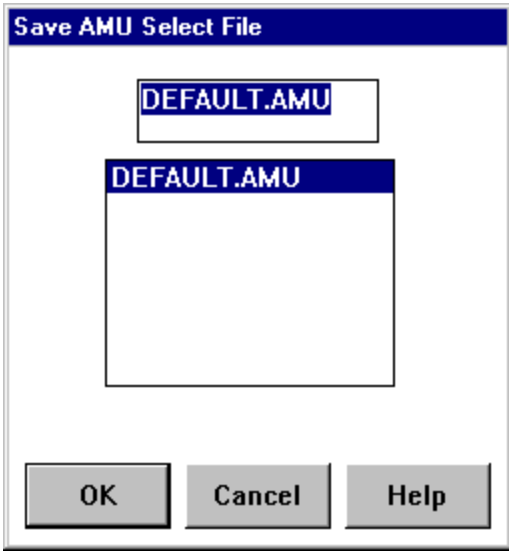
AMU Select File Dialog Box

4 **Click on the isotope (mass number) you want to select or deselect then click the *Set/Reset* button.**

The masses selected will be indicated in < > (brackets). The masses without brackets are not selected. You can select as many isotopes as you like.

5 **Click *OK* when you finish editing the AMU Select File.**

The ***Save AMU Select File*** dialog box will appear.



Save AMU Select File Dialog Box

6 Save the AMU Select File.

If you want to save the changes to the AMU Select File you loaded, click ***OK***. If you want to save the changed AMU Select File as a new file, type in the new file name and click ***OK***.

Modifying Data Analysis Parameters

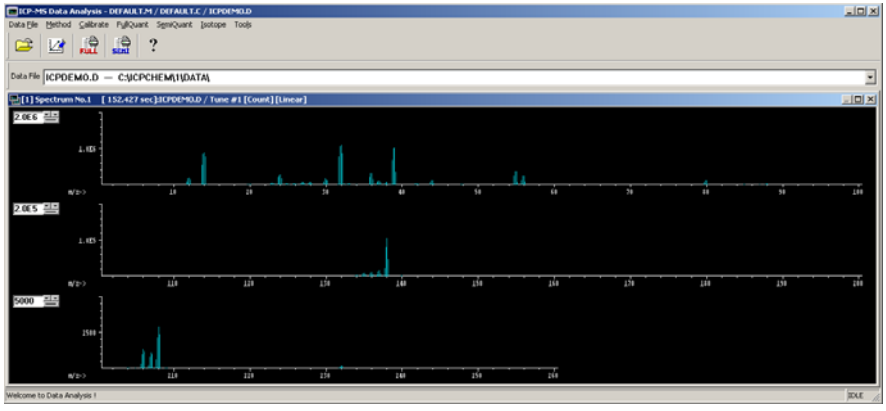
ChemStation enables you to change each parameter separately. To do so, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The Data Analysis menu appears.

2 **Select *Data Analysis>>Main Panel*.**

The ***ICP-MS Data Analysis*** window appears.



ICP-MS Data Analysis Window

3 **Modify all the data analysis parameters that need to be changed.**

Data analysis parameters include:

- Data Correction (Background Subtraction, Interference Correction)
- Select Reports
- Calibration Table for FullQuant
- Blank Subtraction for FullQuant / SemiQuant
- Edit SemiQuant parameters for Semiquantitative Analysis
- Internal Standard Correction for Semiquantitative Analysis
- Edit IR Parameters Isotope Ratio Analysis (IR)
- Edit ID Parameters for Isotope Dilution Analysis (ID)
- Layout custom reports

For more information about editing these data analysis parameters, please refer to the appropriate sections of Chapters 12-15.

The changes you make are not recorded to the method until you save the method.

4 To save the method, complete the following steps:

You must close the Data Analysis window before you can save the method.

1) Double-click the Control Box Menu in the top left corner of the Data Analysis window.

The Data Analysis window closes, and ChemStation returns to the Top window.

2) Select *Top>>Methods*.

The Methods menu appears.

3) Select *Methods>>Save*.

The ***Method Save Options*** dialog box appears. Save the method as described in Saving a Method and Calibration earlier in this chapter.

You can also modify this section of a method by selecting ***Method>>Edit Entire Method***. Click only the ***Data Analysis*** box in the Edit Entire Method dialog box, and click ***OK***. When the dialog box appears, make the changes you want and click ***OK***. Then, complete the saving steps as described in Saving a Method and Calibration earlier in this chapter.

Printing a Summary of Data Analysis Parameters

ChemStation enables you to print a summary of the data analysis parameters for each method you create.

To print a summary of the data analysis parameters for a method, complete the following steps:

1 **Select *Top>>Data Analysis*.**

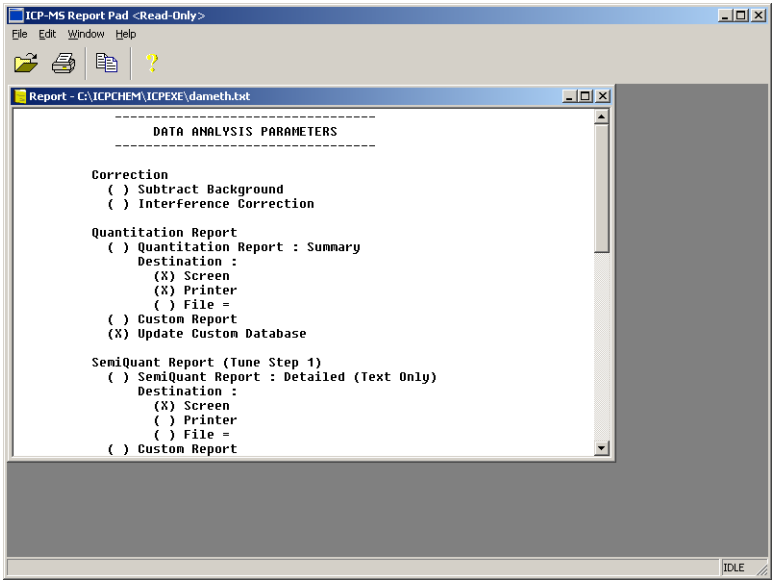
The Data Analysis menu appears.

2 **Select *Data Analysis>>Main Panel*.**

The ***ICP-MS Data Analysis*** window appears.

3 **Select *Method>>View Summary*.**

The ***Dameth.txt - Report Pad*** window appears.



Dameth.txt - Report Pad Window

4 **Select *File>>Print*.**

A summary of the acquisition parameters is printed out.

Modifying QC Parameters

To edit the QC Database and QC Tune Criteria parameters, please refer to the appropriate sections of the Intelligent Sequence Manual.

6

Setting Up a Sequence

Setting Up a Sequence

A sequence is designed to allow you to analyze a number of samples automatically. Multiple methods can be used within a single sequence, if desired. Also, you can run a sequence, either with or without using an autosampler. Create a sequence by entering sample information into the sample log table. This will determine the order in which the Agilent 7500 analyzes the samples, as well as, indicate which method is used for the analysis, and assign the name of the data file generated for each sample.

A sequence can be used to reprocess data files. In this case, ChemStation runs only the data analysis portion of the method.

For information about running samples using a sequence, see Chapter 8, "Running a Sample Analysis."

If the Agilent 7500 is configured with an autosampler, it must be connected to the instrument before a sequence can be run. Arrange the samples in the autosampler racks to correspond to the sequence created with the sample log table.

This chapter explains how to create a sequence, simulate a sequence, and modify an existing sequence.

NOTE

The data acquisition parameters to be used to acquire a given sample are determined by which method is specified for that sample in the sequence. Similarly, data analysis parameters used to process a data file are determined by the method. For instance, if you want to have sample reports automatically printed out for each data file as the sequence runs, any method used must have the appropriate data analysis parameters set prior to running the sequence. Refer to Chapters 12-15 for details on generating specific reports.

Creating a Sequence

To create a sequence, use the Sample Log Table to enter information that the Agilent 7500 uses when analyzing samples. The samples are analyzed in the order in which they appear in the Sample Log Table. For information about how to arrange samples for quantitative, semiquantitative, isotope ratio, and isotope dilution analyses, see Chapter 12 through 15.

To create a Sample Log Table, complete the following steps from the ***ICP-MS Top*** window:

1 **Select Sequence>>Edit Sample Log Table.**

The ***Sample Log Table*** dialog box appears. To create a sequence, edit the Sample Log Table for the sequence currently in memory.

NOTE

To use a new Sample Log Table to set up the sequence, you must first create one. To do so, load the default sequence and edit the Sample Log Table, using the 'Delete' key to delete all unwanted information. Then, close the Sample Log Table and save the sequence as the default. Each time you want to use the blank Sample Log Table you can load the default, edit the Sample Log Table to create the sequence you want, and save the new sequence under a new name. For information about saving a sequence, see "Saving the Sequence" at the end of this chapter.

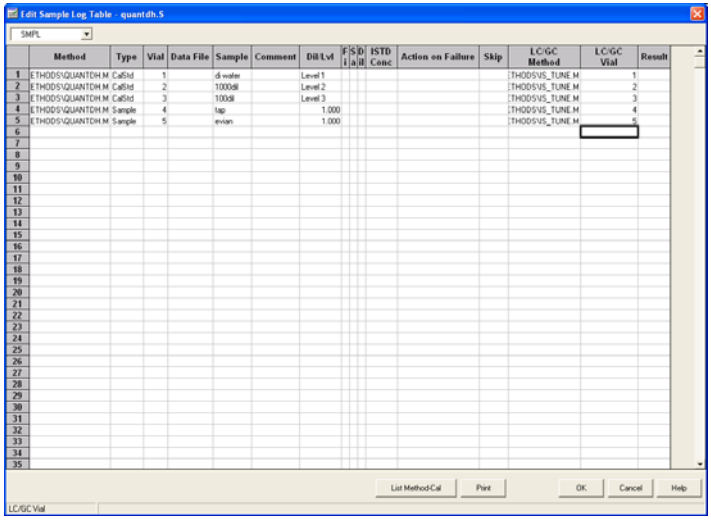
Although you can change the sheet by clicking the arrow beside the Sheet dropdown list box at the upper left, other sheets except the Sample sheet and the Whole List sheet are used when the intelligent functions are applied. The Whole List sheet shows the same list set in the Sample sheet, except it contains several keywords.

NOTE

Intelligent functions are available when the Intelligent Sequence software is installed.

When a QC mode, which has intelligent functions, is selected in Configuration a different sheet appears. Make sure General.qcc is selected as a QC Mode for general use (without any intelligent function).

For more information about intelligent functions, see the Intelligent Sequence Manual.



Sample Log Table Dialog Box

2 Set the method name by double-clicking the cell, and selecting the appropriate method in Select File dialog box.

ChemStation uses the method to analyze the sample. Although a different method can be used for each sample in your sequence, normally you will use the same method for all samples in a sequence.

3 Select a sample type by clicking the cell and clicking an item in the drop-down list.

If general.qcc is selected in ***ICP-MS Configuration***, select one of the following six types.

- Sample
  Select this type when acquiring and processing data on an unknown sample.

- Analysis
  Select this type when generating quantitative results for an unknown being analyzed by the standard addition calibration method. When a standard addition calibration method is used, all spiked samples to be used in updating the standard addition calibration table should be set as type CalStd. The line of the sequence controlling data acquisition of the unknown should also be set as type CalStd with the level set at zero concentration. The sequence line following this entry should be set as type Analysis, using the data file name used as sample. In this way, a standard addition quantitative report can be generated for an unknown.

- CalStd
  Select this type when updating an existing calibration table in the specified method. Usually this type is used for standard solutions. The calibration table should have been created (see Chapter 12) prior to beginning the sequence. When this type is selected, the appropriate calibration level in the calibration table must be selected. The level can be select from 1 to 20.

- QuantBLK
  Select this type when specifying a sample that will be used to provide blank subtraction data for a quantitative method. The concentration of the blank will automatically be substracted from the quantitative results obtained using the method after the line specifying QuantBLK.

- SemiQBLK
  Select this type when specifying a sample that will be used to provide blank subtraction data for a semi-quantitative method. The concentration of the blank will automatically be substracted from the quantitative results obtained using the method after the line specifying QuantBLK.

- Keyword
  Select this type when you wish to invoke certain commands available in sequencing. For example, you can use keywords to turn off the plasma automatically. For more detailed information on available keywords, refer to the Online Help of ChemStation.

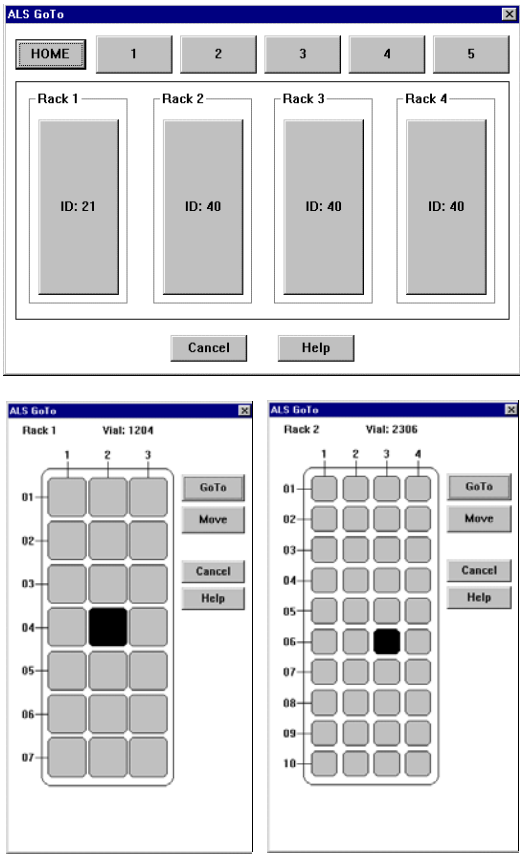
  The sample type you select appears in the Type field of the Sample Log Table.

4 Set a vial number by clicking the cell, and typing in the number.

Refer to the diagram below for information about how the vials are numbered.

e.g. No's 1~5 correspond to the five 250 mL bottles at the rear of the ASX 500 auto sampler.

1204, 2306... Rack number, Row (vertical) number, and Line (horizontal) number



ALS GoTo Dialog Box

5 Set a file name by clicking the cell, and typing.

The data file name can be up to eight characters long. It must not contain the following characters:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (+)	Space ( )
Colon (:)	Quotation mark (“)	

If you do not enter a data file name, ChemStation automatically assigns one. The system-assigned name is a 3-digit number with the sample type specific suffix. A 3 digit number is a run counter beginning at 001 for the first sample analyzed and incremented by 1 for each subsequent sample. When ChemStation writes the files to disk, it adds a.d extension to the file name to indicate that it is a data file.

6 Type a sample name and comment if necessary.

7 Select a level number or dilution factor.

If CalStd is selected as a sample type, select a level number from the drop-down list. For the level number of the standard solution for calibration, select a number corresponding to the level number of the calibration table set in Data Analysis for the method.

For other samples, set a dilution factor by the following method.

1)To directly input a dilution factor, type a dilution factor in the Dilution/Level field.

No further steps are required.

2) When a similar final weight is applied to a multiple number of samples, the dilution factor can be calculated based on the final weight, sample weight, and dilution multiplier. Click the right mouse button on the Dilution/Level field and select ***Show Dil factor calculation*** in the popup menu.

The ***Measured Value, Sample***, and ***Dilution*** will appear.

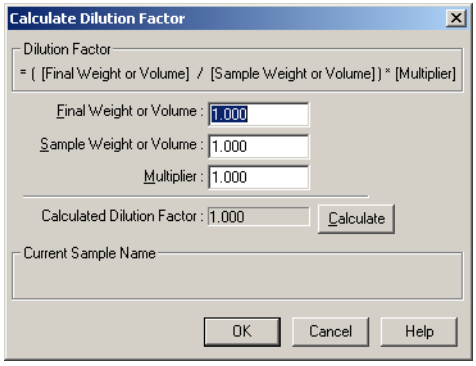
3) Enter a numerical values in the Final Weight or Volume, Sample Weight or Volume, and Multiplier fields.

The dilution factor will be automatically calculated and the result displayed in the Dilution/Level field.

No further steps are required.

4) To calculate the dilution factor for a specific sample, double-click on the Dilution/Level field.

The ***Calculate Dilution Factor*** dialog box appears.



Calculate Dilution Factor Dialog Box

5) Enter a numerical value in each text box.

6) Click ***Calculate***.

The dilution factor will be automatically calculated.

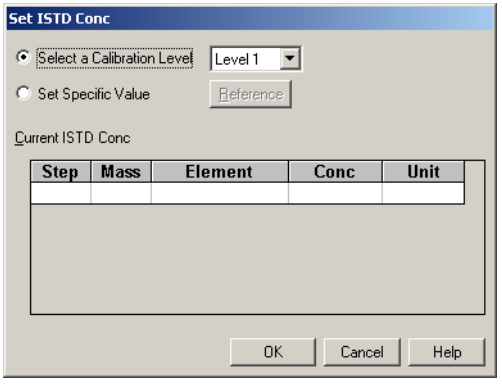
7) Click ***OK***.

The ***Calculate Dilution Factor*** dialog box will close and the dilution factor, final weight/volume, sample weight/volume, and dilution multiplier will be set in the ***Edit Sample Log Table*** dialog box.

8 Set an internal standard concentration level.

1) Double-click on the ISTD Conc field.

The ***Set ISTD Conc*** dialog box appears.



Set ISTD Conc Dialog Box

2) To select a concentration from the calibration levels, choose the ***Select a Calibration Level*** radio button and select a level from the drop-down list.

Proceed to step 5).

3) To set a specific value for the current sample, select the ***Set Specific Value*** radio button.

4) Set a concentration in the ***Conc*** field of the Current ISTD Conc table.

The Current ISTD Conc table shows the internal standard currently set for calibration. Click on the ***Conc*** field and enter a concentration value.

Click the right mouse button to display the popup menu, which allows you to copy, paste, or delete the field.

Click ***Browse*** to set the concentration setting for each calibration level in the Concentration field.

5) Click ***OK***.

The ***Set ISTD Conc*** dialog box will close and the ISTD concentration level will be set in the ***Edit Sample Log Table*** dialog box.

If the ***Select a Calibration Level*** radio button was selected, the level number will be displayed. If the ***Set Specific Value*** radio button was selected, "Manual" will be displayed.

9 To execute an action other than the action set as the method in case of a QC check failure, select a process to be executed from the *Action on Failure* drop-down list.

10 To skip a method in the sequence, double-click on *Skip* and select Skip.

Double-clicking ***Skip*** will alternately display and hide Skip.

11 To use the LC/GC ChemStation, set a method and vial number to use with the LC/GC ChemStation.

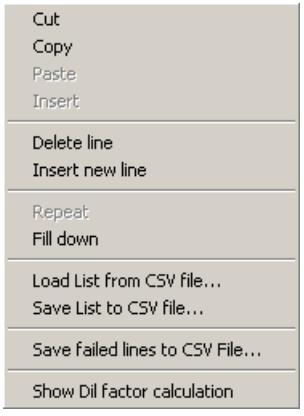
1) Click the right mouse button and select ***Show Columns for LC/GC ChemStation*** in the popup menu.

2) Double-click the ***LC/GC Method*** field, then select a desired LC/GC ChemStation method from the ***Select File*** dialog box displayed.
If you leave the ***LC/GC Method*** field blank, the method already loaded in the LC/GC ChemStation will be used during the sequence.

3) Set an LC/GC vial number in the ***LC/GC Vial*** field.
   If you leave the ***LC/GC Vial*** field blank, the method in the LC/GC ChemStation will not be executed. You can set a vial number from -1 to 200. (The range of numbers you can set varies depending on the instrument connected.)

12 Repeat the above steps for each sample to complete the Sample Log Table.

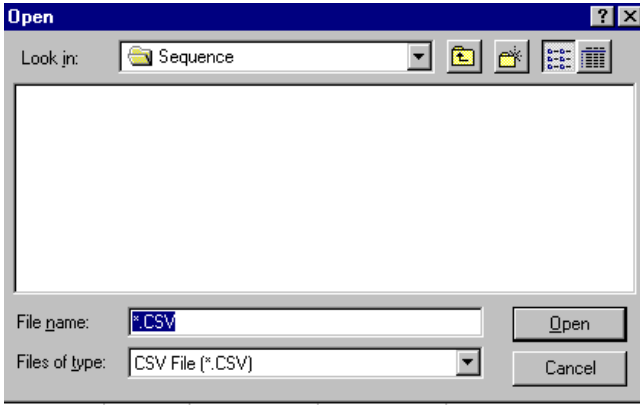
You can use the popup menu by clicking the right mouse to make operation easier. Any numeric portion except the method name can be automatically incremented by using Fill down or Repeat.



Popup Menu for Setting

NOTE The 4-digit vial number for the standard or sample racks is incremented corresponding to the current autosampler configuration. The 1 digit vial number for the water, blank, and tuning solution rack is incremented up to 5.

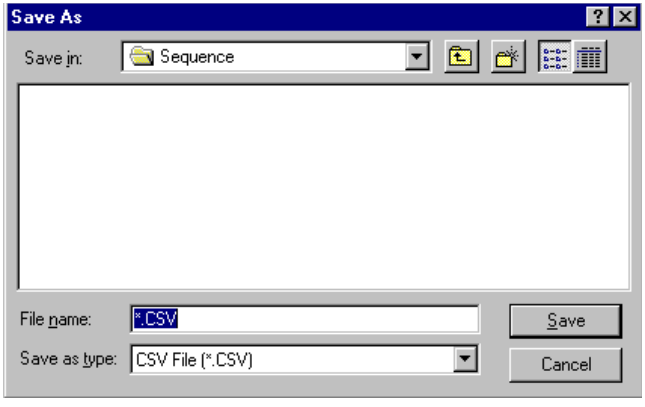
You can set up the sequence by directly loading a CSV file (comma separated value ASCII file). Select ***Load List from CSV file*** from the popup menu, and then the ***Open*** dialog box appears.



Open Dialog Box

Select a CSV file you want to load. CSV file has a.csv extension.

You can also save the sequence as a CSV file. Select Save ***List to CSV file*** from the popup menu and then the ***Save As*** dialog box appear.

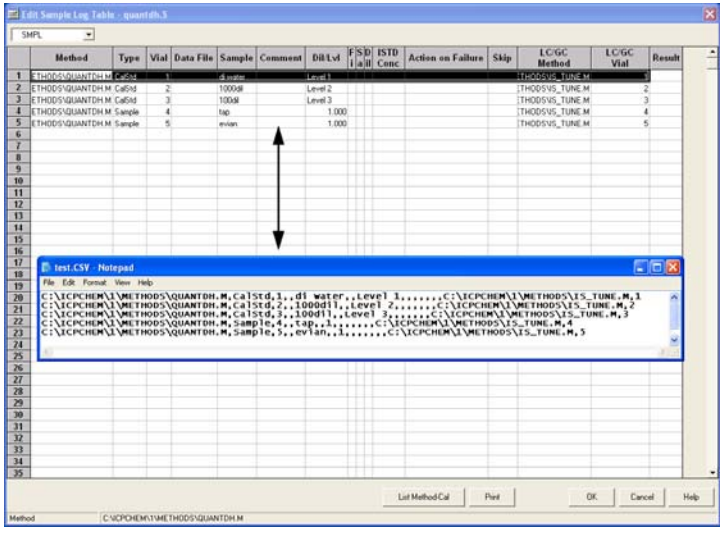


Save As Dialog Box

CAUTION ISTD settings cannot be downloaded from a CSV file.

Enter the name of the CSV file, and then click ***Save***.

The following figure shows the relationship between the Sample Log Table and the CSV file. If there is a entry which you want to leave blank, a comma should be set for that in the CSV file.



Sample Log Table and CSV File

13 Click *OK*.

The sequence is saved into memory. To save it onto the hard drive and assign a name for the sequence, see "Saving a Sequence" later in this chapter. You must save the sequence before exiting the ChemStation, otherwise the Sample Log Table information will be lost.

ChemStation returns to the Top window.

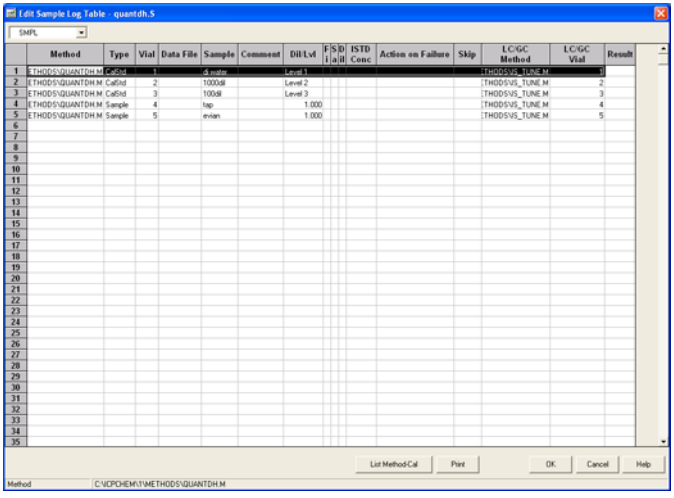
Position and Run

You can start the sequence from the middle of the Sample Log Table. To position and run a sequence, complete the following steps:

1 **Select *Sequence>>Position and Run*.**

The ***Position and Run*** dialog box appears. It contains several keywords in blue which are mainly used with the intelligent functions*. For more detailed information, see the Intelligent Sequence Manual.

*Intelligent functions are available when the Intelligent Sequence software is installed.



Position and Run Dialog Box

2 **Click the line of the Sample Log Table where you want to start.**

3 **Click *OK*.**

The Start Sequence dialog box appears.

Simulating a Sequence

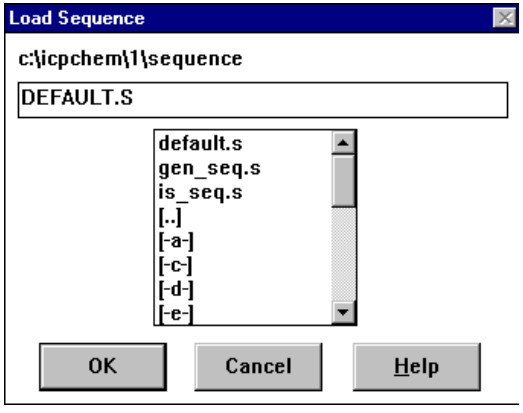
You can check a sequence before running it by "simulating" the sequence. The simulate sequence command runs through the sequence to check for duplications in file names, incorrect directory destinations, and to check whether there is enough disk space available to store your data. The information is contained in a sequence log file. This section explains how to load and simulate a sequence and how to view the sequence log.

Simulating a Sequence

A sequence must be loaded before you can simulate it. To load and simulate a sequence, complete the following steps:

1 **Select *Sequence>>Load*.**

The ***Load Sequence*** dialog box appears.



Load Sequence Dialog Box

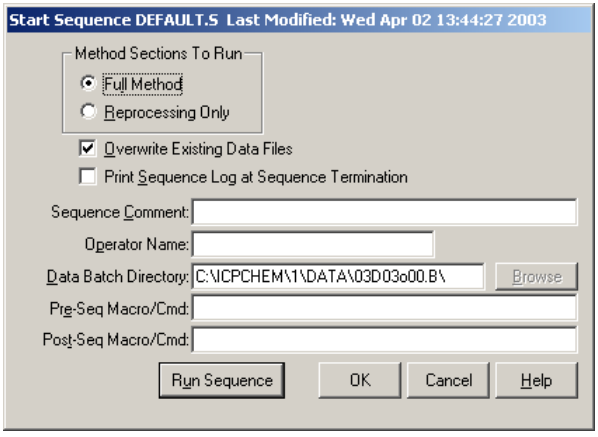
2 Select a file using one of the following methods:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the Name and click ***OK***.

The sequence is loaded.

3 Select Sequence>>*Simulate Sequence.*

The ***Start Sequence*** dialog box appears.



Start Sequence Dialog Box

4 Select *Full Method* as the Method Sections To Run.

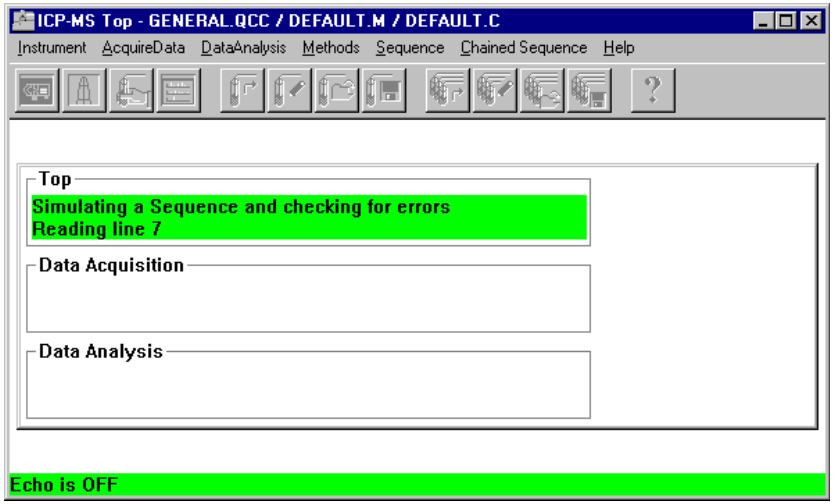
This must be checked so that the simulation checks for free disk space.

5 Complete the dialog box.

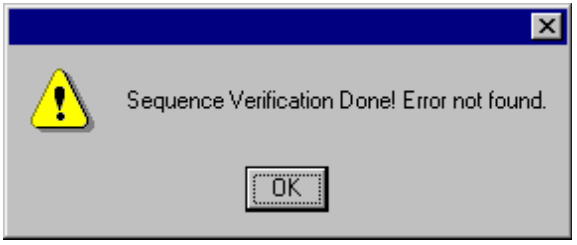
Refer to "Running a Sequence" in Chapter 8.

6 Click *Run Sequence.*

A status box appears. The top section of the status box indicates how much disk space is needed for the sequence and how much is available. When the ***Overwrite Existing Data Files*** check box is not checked, the simulation checks whether the same data file exist or not. When ChemStation completes this simulation, a box appears to indicate that the sequence verification is done.



Run Sequence Status Box



Confirmation Dialog Box

NOTE

Click ***OK*** to accept the edited information without simulating the sequence. Click ***Cancel*** to cancel the edited information.

7 Click *OK*.

ChemStation returns to the Top window.

Viewing the Sequence Log File

The Sequence Log file is generated every time a sequence is simulated or run. The file contains a list of vial numbers, names of data files, and any error messages generated during the sequence.

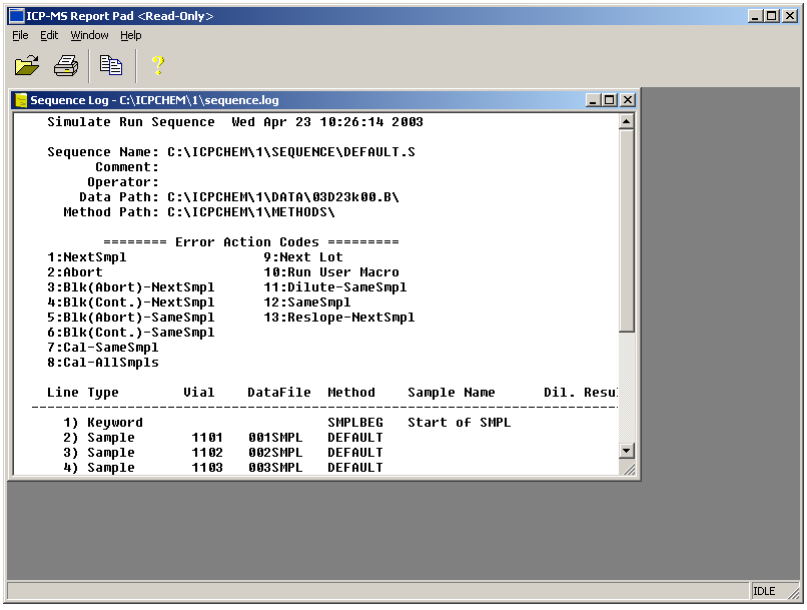
To view the sequence log file, complete the following steps:

1 **Select *Sequence>>View Sequence Log*.**

A Report Pad window appears showing the sequence log file for the sequence you just simulated or ran. ChemStation saves the log file as a Sequence Log each time you simulate or run a sequence.

Error Action Codes at the header part and Error Check and Action sections are effective when the intelligent function* is used. For more detailed information, see the appropriate sections of the Intelligent Sequence Manual.

* Intelligent functions are available when the Intelligent Sequence software is installed.



Sequence.log - Report Pad Window

2 To print the sequence log file, select F*ile*>>*Print* from the Report Pad menu.

ChemStation sends the file to the printer.

You can also print the Sequence Log by selecting ***Print Sequence Log*** from the Sequence menu.

3 To close the notepad file, select *File*>>*Exit* from the Report Pad menu.

Modifying a Sequence

You can modify a sequence when you edit the Sample Log Table, and also by selecting different Sequence Options.

Editing Sample Log Table to Modify a Sequence

Before you can edit a Sample Log Table for the sequence you want to modify, you must load the sequence into memory. See Steps 1 and 2 of "Simulating a Sequence" in this chapter. Once the sequence is loaded, complete the following steps:

1 **Select *Sequence>>Edit Sample Log Table*.**

The Sample Log Table appears.

2 **Edit the fields to make changes to the sequence.**

For information about the fields in the sample log table, see "Creating a Sequence" in this chapter.

3 **Click *OK*.**

The Sample Log Table closes and changes are saved in the memory. If you exit ChemStation without saving the sequence, the changes you made to the Sample Log Table are lost. To save the sequence file to disk and assign a name to the sequence, you must save the sequence. See "Saving the Sequence" in this chapter.

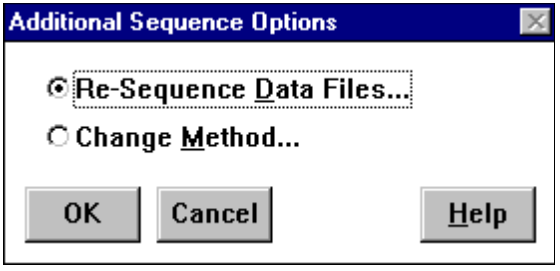
Modifying Sequence

You can also modify a sequence without editing the Sample Log Table. ChemStation provides additional options to change a sequence: re-sequencing data files and changing the method. When you use these sequence options, ChemStation automatically changes information in the Sample Log Table in the manner specified. Check the Sample Log Table to verify that the changes made are what you want.

To use the additional sequence options, complete the following steps:

1 **Select *Sequence>>More*.**

The ***Additional Sequence Options*** dialog box appears.



Additional Sequence Options Dialog Box

2 **Select one of the sequence options and click *OK*.**

The following options are available:

- Re-Sequence Data Files...
  This option changes the data file names. The files will be renamed in ascending numerical order starting with the data file name entered. If you do not indicate a beginning data file name, ChemStation starts with the first file name in the sequence.
- Change Method...
  This replaces a method originally used in the sequence with a method you specify.

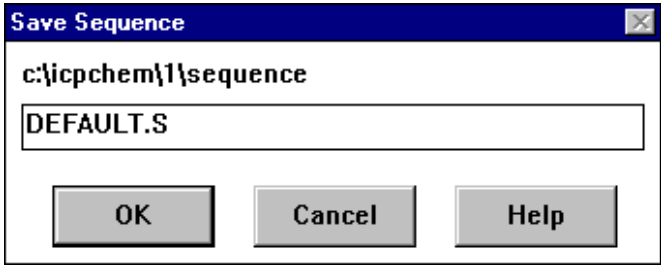
NOTE When the Intelligent functions are applied, do not run "Re-Sequence Data Files…".

Saving the Sequence

After the changes are made to the Sample Log Table, you can save the changes to disk by completing the following steps:

1 **Select *Sequence>>Save*.**

The ***Save Sequence*** dialog box appears.



Save Sequence Dialog Box

2 **Enter the name of the sequence and click *OK*.**

ChemStation saves the sequence to disk and the dialog box disappears.

The name can be up to eight characters long. It must not contain the following characters:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (+)	Space ( )
Colon (:)	Quotation mark (“)	

ChemStation adds a ***.s*** to the end of the name to indicate it is a sequence file.

All sequences are normally stored in the c:\icpchem\1\sequence directory.

7

Chained Sequence

Chained Sequence

A chained sequence is designed to allow you to run a series of sequences. Also, you can assign different tuning parameters to each sequence.

Any sequences used must have the appropriate sample log table set prior to running a chained sequence. For information about setting sequences, see Chapter 6, "Setting Up a Sequence".

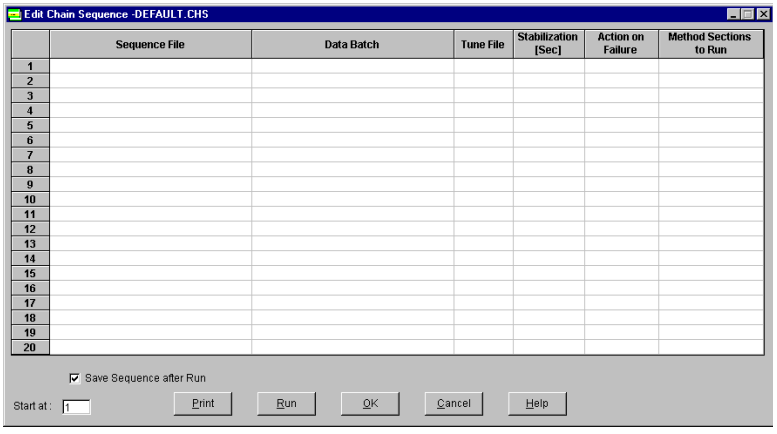
This chapter explains how to create a sequence, simulate a sequence, and modify an existing sequence.

Creating a Chained Sequence

To create a chained sequence, complete the following steps from the ***ICP-MS Top*** window:

1 **Select *Chained Sequence>>Edit and Run*.**

The ***Chained Sequence Table*** dialog box appears. To create a sequence, you edit the sample log table for the chained sequence currently in memory.

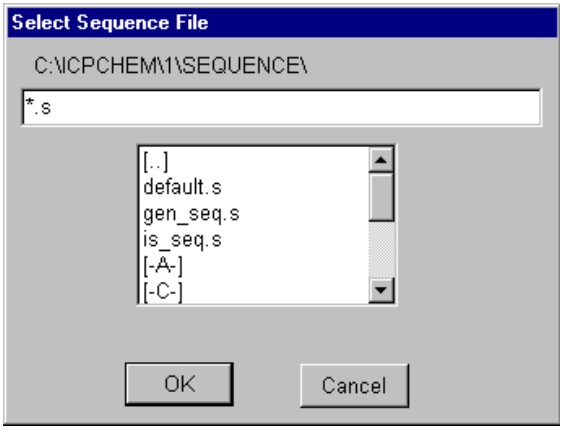


Edit Chain Sequence Dialog Box

2 Set the following items of the chained sequence table.

- Sequence File…

Double-click the Sequence File field you want to set. The ***Select Sequence File*** dialog box appears. Select an existing sequence file you want to run.



Select Sequence File Dialog Box

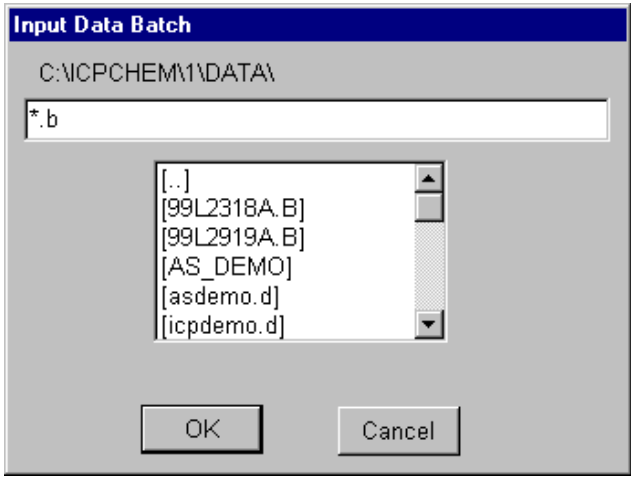
NOTE

When you use ***Reprocessing Only*** as the Method Section to Run, select the "result.s" file in the data batch directory you want to reprocess, which contains the actual sequence log. The original sequence file contains no data file names and does not reflect any additional analysis which the intelligent sequencing may have scheduled as a result of QC failures or a periodical run.

Intelligent functions are available when the Intelligent Sequence software is installed.

- Data Batch…

When the "Sequence File" area is left without any name, a new data batch directory as the destination for the data files is created in the c:\icpchem\1\data at the time you begin your sequence using Full Method. It is named for the current date and time. You can also select or create a data batch directory by double-clicking the Data Batch field. The ***Input Data Batch*** dialog box appears. Select an existing data batch, or type the file name into the Data Batch Directory field.

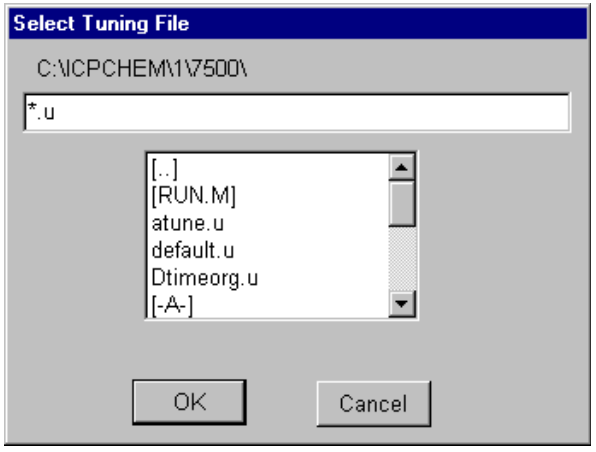


Input Data Batch Dialog Box

NOTE When you use ***Reprocessing Only*** as the Method Section to Run, select the existing data batch directory you want to reprocess.

- Tune File…

You must choose the tune file for each sequence. Double-click a Tune File field you want to set. The ***Select Tuning File*** dialog box appears. Select an existing tuning file you want to use.



Select Tuning File Dialog Box

- Stabilization [Sec]…

  You can set a delay to allow for the stabilization time when changing the tuning conditions. Select a Stabilization Time field, and type a value.

- Action on Failure…

  You can set Action on Failure when a sequence is aborted. Select ***Abort*** or ***Continue*** by double-clicking the field.

- Method Section to Run…

  Select ***Full Method*** or ***Reprocessing Only.***

You can use the edit menu which contains ***Cut***, ***Copy***, ***Paste***, ***Insert***, ***Delete line***, and ***Insert new line*** by clicking the right mouse button.

3 Selecting Other items if needed.

There are two options for running a chained sequence.

- Save Sequence after Run…

  When it is on, the sequence modified during the run can be overwritten after running the sequence.

- Start at…

  You can select the first line to be executed by typing the line number. The upper lines, rather than the line number typed, are not executed.

Simulating a Chained Sequence

You can check a chained sequence before running it by "simulating" the chained sequence. The simulated chained sequence command runs through each sequence line to check for duplications in file names, incorrect directory destinations, and also to check whether there is enough disk space available for your data.

Viewing the Chained Sequence Log File

The chained sequence log file is generated every time a chained sequence is simulated or run.

To view the chained sequence log file, complete the following steps:

1 **Select *Chain Sequence>>View Chain Seq. Log*.**

A Report Pad window appears showing the chained sequence log file for the chained sequence you just simulated or ran. ChemStation saves the log file as a ChnSeq.log each time you simulate or run a chained sequence.

2 **To print the chained sequence log file, select *File>>Print* from the Report Pad menu.**

ChemStation sends the file to the printer.

3 **To close the Report Pad file, select *File>>Exit* from the Report Pad menu.**

Report Pad closes and ChemStation returns to the Top window.

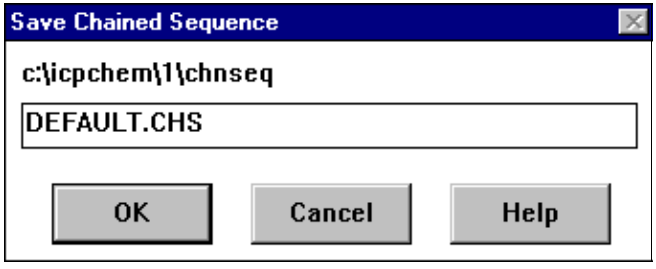
You can also close the file by double-clicking the Control Box Menu in the upper left corner of the Report Pad window.

Saving the Chained Sequence

After the changes are made to the chained sequence table, you can save these changes to disk by completing the following steps:

1 **Select *Chained Sequence>>Save*.**

The ***Save Chained Sequence*** dialog box appears.



Save Chained Sequence Dialog Box

2 **Enter the name of the chained sequence.**

3 **Click *OK*.**

ChemStation saves the chained sequence to disk, and the dialog box disappears.

The name can be up to eight characters long. It must not contain the following characters:

Period (.)	Slash (/)	Brackets ([])
Comma (,)	Backslash (\)	Vertical bar (|)
Semicolon (;)	Equal sign (+)	Space ( )
Colon (:)	Quotation mark (“)	

ChemStation adds a ***.chs*** to the end of the name to indicate it is a chained sequence file. All chained sequences are stored in the c:\icpchem\1\chnseq directory.

8

Running a Sample Analysis

Running a Sample Analysis

You can run samples through the Agilent 7500 in one of two ways:

- Running a sequence enables you to determine the order in which multiple samples are analyzed automatically. Set up the sequence according to your analytical requirements. For example, the types of samples you run and the sequence of those samples can depend on whether you are performing a quantitative, semiquantitative, isotope ratio, and isotope dilution analysis. For information about arranging samples, see Chapter 12 through 15.
- Running samples manually enables you to introduce samples one at a time using the method of your choice to determine the measurement parameters. After running the samples, you can create a sequence to perform calculations for the previously acquired data.

Regardless of how you are running the samples, ChemStation provides a real-time display in the acquisition window. In this window you can change the type and scale of the display, or halt the run.

This chapter explains how to run samples both using a sequence and a method, as well as how to monitor and stop the run.

Running Samples by Using a Sequence

Use a sequence when you want to analyze a large number of samples automatically. The arrangement of samples in the autosampler must match the arrangement you set up in the sample log table. The autosampler automatically aspirates the samples in the order you specify. For more information about creating a sequence, see Chapter 6, "Setting Up a Sequence".

To run samples using a sequence, you must first load the sequence and then run it. You can do these two steps together or separately. To load and run a sequence in one step, select ***Sequence>>Load and Run Sequence*** from the Top window.

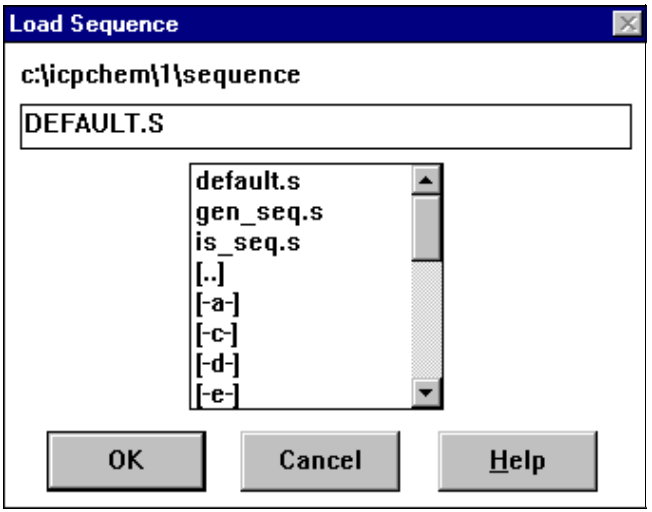
The steps are then similar to the steps in the following sections, which describe how to load and run a sequence separately. Simply omit the first step in each section.

Loading a Sequence

To load an existing sequence, complete the following steps from the Top window:

1 **Select *Sequence>>Load*.**

The ***Load Sequence*** dialog box appears.



Load Sequence Dialog Box

2 **Use one of the following methods to choose the file name:**

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the Name and click ***OK***.

Running a Sequence

Running a sequence starts the sample analysis. To run a sequence that is already loaded, complete the following steps from the Top window:

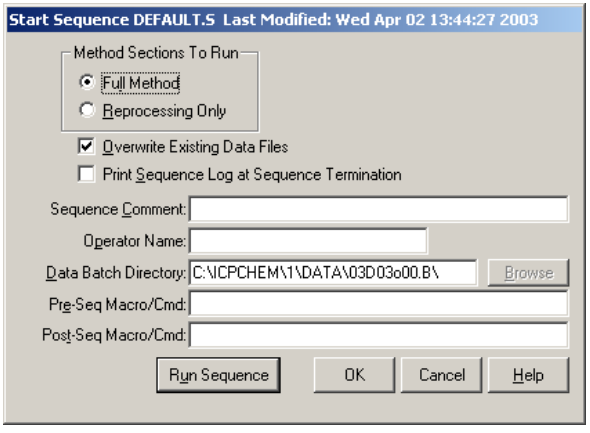
1 **Confirm that the currently loaded method at the Top level is saved in the same directory as the method to be used for reprocessing.**

If not, load the method to be used for reprocessing by using ***Methods >>Load***. Confirm that the edited method and calibrations is saved.

NOTE When a sequence is executed using ***Run Sequence***, methods set in the Sample Log Table are always loaded from the directory where the currently loaded method at the Top level is saved.

2 **Select *Sequence>>Run*.**

The ***Start Sequence*** dialog box appears.



Start Sequence Dialog Box

A data batch directory as the destination for the data files is created based on the current date and time. The format is year, month (A-L corresponds to 1-12), day, hour(A-X corresponds to 0-23), and a letter(00,01,02,..zy,zz). If you wish, you can type your own directory name, instead. The name can be up to eight characters long.

3 Select method sections to run.

Select ***Full Method*** if you are ready to acquire data.

Select ***Reprocessing Only*** if the data has already been acquired (e.g. if the data has been acquired manually, or if you are reprocessing data files acquired previously in a sequence).

4 Complete the dialog box.

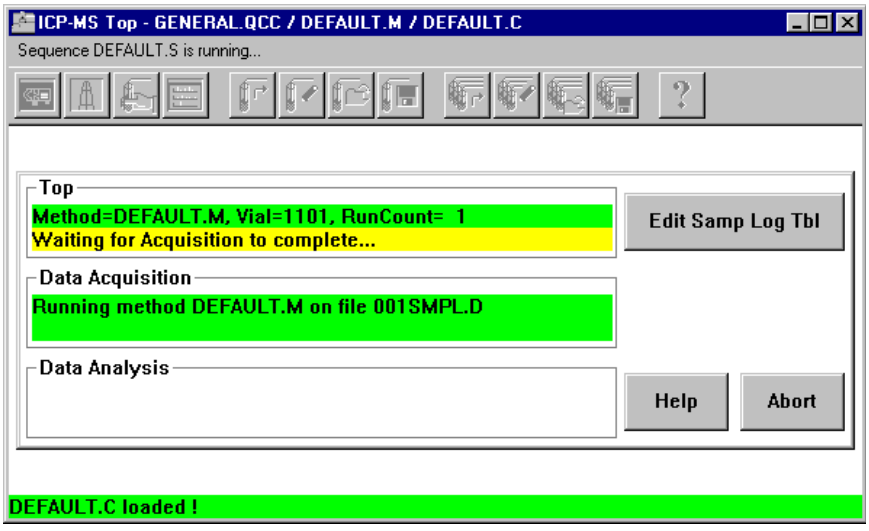
This dialog box enables you to add comments about the sequence, set a pre- or post-sequence macro or command to be executed, and to verify or correct the directory path for the data files the sequence generates. You can create a new directory as the destination for the data files simply by typing the desired path into the Data File Directory field and running the sequence.
When the Print Sequence Log at Sequence Termination box is checked, the sequence log is printed out upon completion of the sequence.

NOTE Any data files in the specified data file directory whose names are the same as those in your Sample Log Table will be lost if the ***Overwrite Existing Data Files*** check box is selected.

5 Click *Run Sequence*.

The analysis starts and the Run Sequence status box appears. This status box remains open during the sample run. You can view a real-time display of the run without closing the status box. See "Monitoring a Run" later in this chapter for instructions.

NOTE Click ***OK*** to accept the edited information without executing the sequence.
Click ***Cancel*** to cancel the edited information.



Run Sequence Status Box

NOTE

Urgent samples can be appended to or inserted into the sample log table, even when the sequence is running. Click on the ***Edit Sample Log Tbl*** push button. The log table will be displayed. All samples already analyzed will appear "grayed out", indicating the current position in the sequence. A new sample line can be inserted in the table, anywhere after the current position. If the auto sampler reaches the position you are editing, it will wait until you have finished, and then resume the sequence.

Running a Sample by Using a Chained Sequence

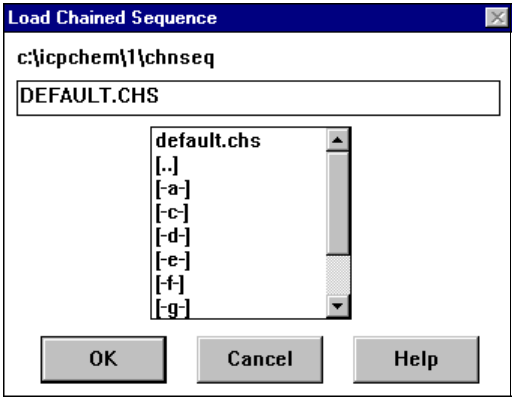
A chained sequence is designed to allow you to run a series of sequences. To run samples using a chained sequence, you must first load the chained sequence and then run it.

Loading a Chained Sequence

To load an existing sequence, complete the following steps from the Top window:

1 **Select *Chained Sequence>>Load*.**

The ***Load Chained Sequence*** dialog box appears.



Load Chained Sequence Dialog Box

2 **Use one of the following methods to choose the file name:**

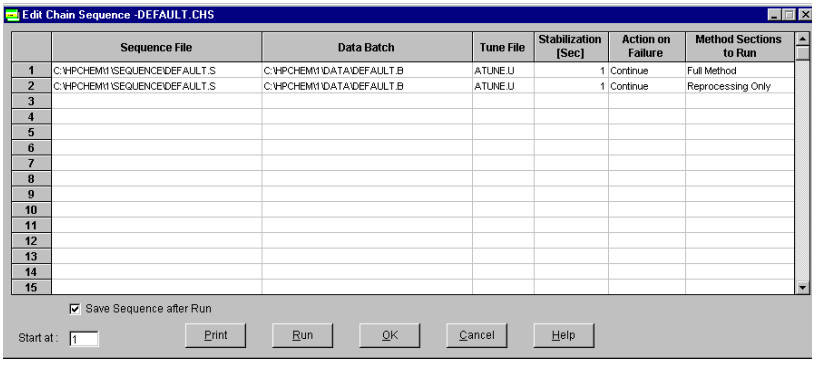
- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the Name and click ***OK***.

Running a Chained Sequence

Running the chained sequence starts the sample analysis. To run a sequence that is already loaded, complete the following steps from the Top window:

1 Select *Chained Sequence>>Edit and Run*.

The ***Edit Chained Sequence*** dialog box appears.



Edit Chained Sequence Dialog Box

2 Click *Run*.

The analysis starts and the sequence currently running is indicated in blue. The table remains open during the sample run. You can view a real-time display of the run without closing the table. For more information about monitoring a run, see "Monitoring a Run" later in this chapter for instructions.

Running Samples Manually

You can run a method to analyze samples if you do not have an autosampler. However, you may have other reasons to run a method rather than a sequence. For example, if you have only one or two samples to analyze, it may be more efficient to run the samples manually rather than set up the autosampler.

When you analyze samples manually, you must make sure the appropriate method is loaded and then run the sample. To load and run a method in one step, select ***Methods>>Load and Run*** from the Top window.

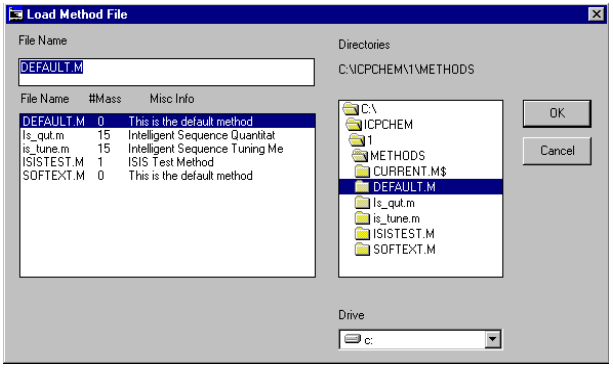
The steps are then similar to the steps in the following sections, which describe how to load and run a method separately. Simply omit the first step in each section.

Loading a Method

Before you run a sample, make sure the appropriate method is loaded. Check the title bar of the Top window to see what method is currently in memory. Then, if necessary, load the method you need. To load a method, complete the following steps from the Top window:

1 **Select *Methods>>Load*.**

The ***Load Method File*** dialog box appears.

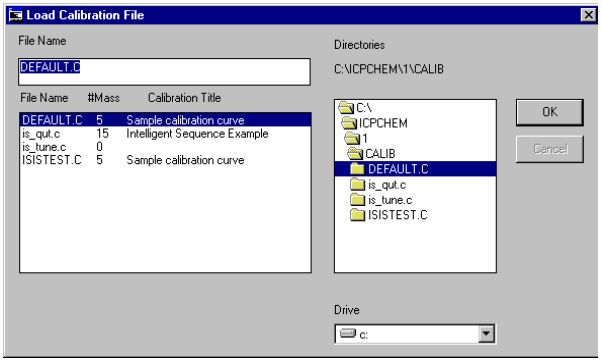


Load Method File Dialog Box

2 To load the method, select it in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

The ***Load Calibration File*** dialog box appears



Load Calibration File Dialog Box

3 To load the method, select it in one of the following ways:

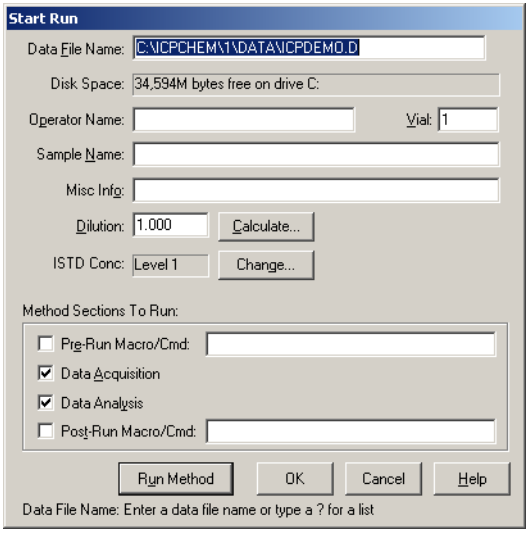
- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

Acquiring Sample Data

After loading the method, there are two ways to run a sample manually, Method Run and Acquired Data. To use Method Run, complete the following steps from the Top window:

1 Select *Methods>> Run*.

The ***Start Run*** dialog box appears.



Start Run Dialog Box

2 Check the data file directory path in the *Data File Name* text box and correct the path if necessary.

3 To use the autosampler, enter a vial number in the *Vial* text box.

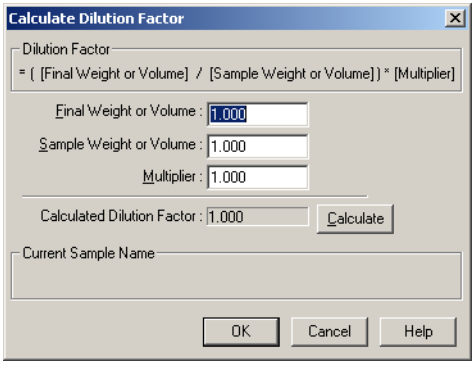
4 Type a sample name and comment.

5 Set a dilution factor if necessary.

Enter a dilution factor directly or click ***Calculate*** and follow the procedure described below.

1) Click ***Calculate***.

The ***Calculate Dilution Factor*** dialog box will appear.



Calculate Dilution Factor Dialog Box

2) Enter a numerical value in each text box.

3) Click ***Calculate***.

The dilution factor will be automatically calculated.

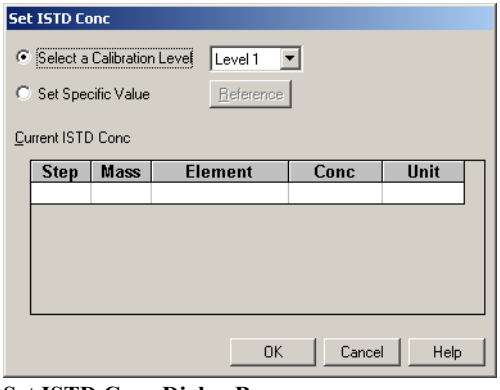
4) Click ***OK***.

The ***Calculate Dilution Factor*** dialog box closes and the dilution factor will be set in the ***Start Run*** dialog box.

6 Set the internal standard concentration level if necessary.

1) Click ***Change***.

The ***Set ISTD Conc*** dialog box appears.



Set ISTD Conc Dialog Box

2) To select a concentration from the calibration levels, choose the ***Select a Calibration Level*** radio button and select a level from the drop-down list.

Proceed to step 5).

3) To set a specific value for the current sample, select the ***Set Specific Value*** radio button.

4) Set the concentration in the ***Conc*** field in the Current ISTD Conc table.

The Current ISTD Conc table shows the internal standard currently set for calibration.

Click on the ***Conc*** field and enter a concentration level. Click the right mouse button to display the popup menu, which allows you to copy, paste, or delete the field.

Click ***Browse*** to set the concentration setting for each calibration level in the Concentration field.

5) Click ***OK***.

The ***Set ISTD Conc*** dialog box will close and the internal standard concentration level will be set in the ***Start Run*** dialog box.

If the ***Select a Calibration Level*** radio button was selected, the level number will be displayed. If the ***Set Specific Value*** radio button was selected, "Manual" will be displayed.

7 Set a section in the *Method Sections To Run*: area.

NOTE

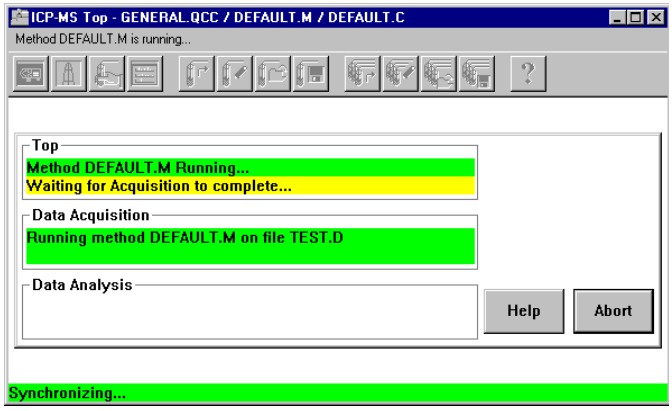
By using the ***Pre-Run Macro/Cmd:*** or ***Post-Run Macro/Cmd:*** check boxes, it is possible to specify whether the action is performed before or after data acquisition. ChemStation is equipped with macros that enable automatic startup and shutdown of the equipment. Contact your local Agilent Technologies for information on additional customized macros.

8 Click *Run Method*.

The analysis starts and the ***Run Method Status*** box appears. This status box remains open during the sample run. You can view a real-time display of the run without closing the status box. See "Monitoring a Run" later in this chapter for instructions.

NOTE

The ***Start Run*** dialog box contains ***OK***, ***Cancel*** and ***Help*** buttons.
Click ***OK*** to accept the edited information without executing the method.
Click ***Cancel*** to cancel the edited information.



Run Method Status Box

After the data acquisition, the data analysis runs when the Data Analysis check box in the Start Run dialog box is checked.

NOTE

After Run Method is executed, the method is automatically saved. Therefore, if you have modified the method before executing Run Method, the modified information will be overwritten under the same method name after the Run Method is finished.

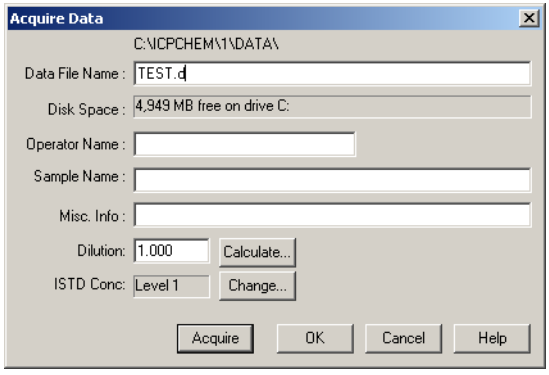
To use Acquired Run, complete the following steps from the Top window:

1 **Select *AcquireData>>Main Panel*.**

The ***ICP-MS Acquisition*** window appears.

2 **Select *AcquireData>>Acquire Data*.**

The ***Acquire Data*** dialog box appears.



Acquire Data Dialog Box

3 **Type the data file name and directory where you want to save the file.**

To change the directory, type a question mark (**?**) in the Data File Name text box.

4 **Type the operator name and sample information.**

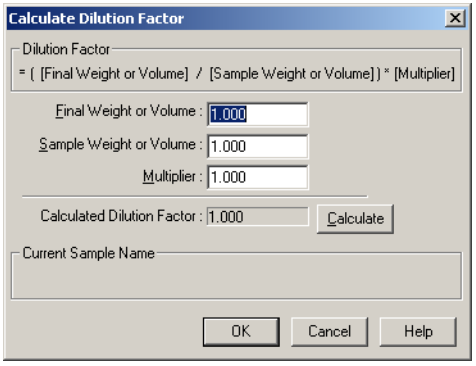
ChemStation records this information as a header for the data file.

5 **Set a dilution factor if necessary.**

Enter a dilution factor directly or click ***Calculate*** and follow the procedure described below.

1) Click ***Calculate***.

The ***Calculate Dilution Factor*** dialog box will appear.



Calculate Dilution Factor Dialog Box

2) Enter a numerical value in each text box.

3) Click ***Calculate***.

The dilution factor will be automatically calculated.

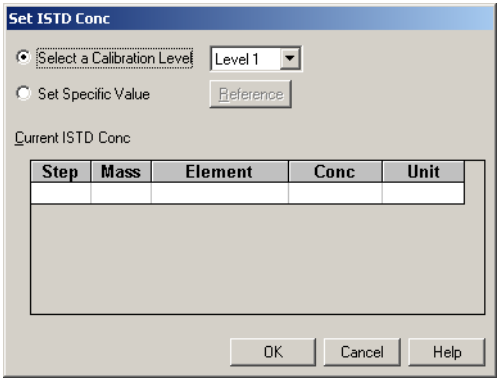
4) Click ***OK***.

The ***Calculate Dilution Factor*** dialog box closes and the dilution factor will be set in the ***Acquire Data*** dialog box.

6 Set the internal standard concentration level if necessary.

1) Click ***Change***.

The ***Set ISTD Conc*** dialog box appears.



Set ISTD Conc Dialog Box

2) To select a concentration from the calibration levels, choose the ***Select a Calibration Level*** radio button and select a level from the drop-down list.

Proceed to step 5).

3) To set a specific value for the current sample, select the ***Set Specific Value*** radio button.

4) Set the concentration in the ***Conc*** field in the Current ISTD Conc table.

The Current ISTD Conc table shows the internal standard currently set for calibration. Click on the ***Conc*** field and enter a concentration level.

Click the right mouse button to display the popup menu, which allows you to copy, paste, or delete the field.

Click ***Browse*** to set the concentration setting for each calibration level in the Concentration field.

5) Click ***OK***.

The ***Set ISTD Conc*** dialog box will close and the internal standard concentration level will be set in the ***Acquire Data*** dialog box.

If the ***Select a Calibration Level*** radio button was selected, the level number will be displayed. If the ***Set Specific Value*** radio button was selected, "Manual" will be displayed.

7 Place the sample uptake tube into the sample.

8 Click *Acquire*.

The analysis starts and the ***Real-Time display*** window appears. For more information about this window, see "Viewing a Real-Time Display" in this chapter.

NOTE

The Acquire Data dialog box contains ***OK***, ***Cancel*** and ***Help*** buttons.
Click ***OK*** to accept the edited information without acquiring the data.
Click ***Cancel*** to cancel the edited information.

After the analysis starts, the Peristaltic Pump program runs. To set this, refer to "Setting the Peristaltic Pump Program" in Chapter 5.

Monitoring a Run

The Real-Time display in the ***ICP-MS Acquisition*** window allows you to view a run in progress.
Samples analyzed using a method for spectrum acquisition will show a display covering the full range of masses. You can alter the type and scale of the spectral display. In addition to providing a graphic display of data acquisition, the real-time display shows the remaining time for each repetition as the run progresses.

This section explains how to view, alter, and clear the real-time display.

When a time-resolved analysis method is run, the acquired data is displayed in real time. On the real-time display screen, data can be saved and displayed during acquisition (snapshot data).

Viewing a Real-Time Display

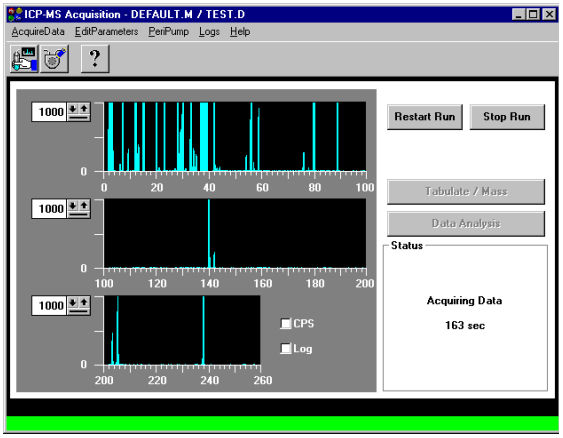
To view the real-time display, complete the following steps:

1 **Run a sequence or method.**

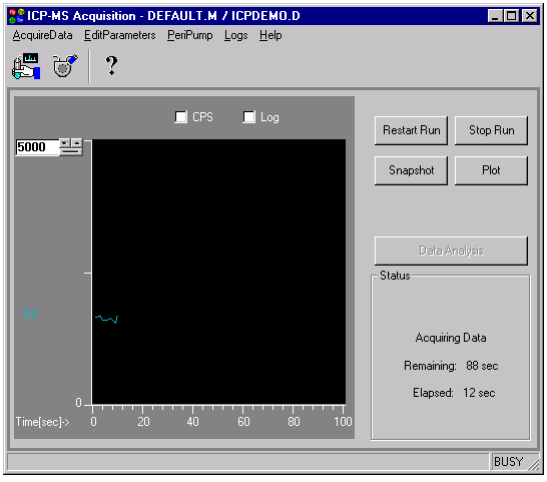
See "Running Samples Using a Sequence" or "Running Samples Manually", earlier in this chapter.

2 **Click the *ICP-MS Acquisition* on the Windows Task Bar.**

The Real-Time display window appears.



Real-Time Display Window for Spectrum Acquisition



Real-Time Display Window for Time-Resolved Analysis

Click on ***Snapshot*** to display snapshot data on the online data analysis screen. Using this function, an interim analysis can be performed during long-term analysis. Snapshot data is saved as a file named "SNAPSHOT.D" in "C: \icpchem\icpexe\." Every time ***Snapshot*** is clicked, the existing file is updated.

Upon completion of acquisition, ***Snapshot*** is grayed out (it cannot be selected).

NOTE The detector mode is reflected in the acquired data when ChemStation finishes data acquisition. Therefore, snapshot data cannot save the correct detector mode. The mass number, for which ***Analog*** is selected in ***Detector Mode***, are handled as data acquired in the analog mode, and the other mass numbers are handled as data acquired in the pulse mode.

As the Agilent 7500 acquires data, ChemStation updates the spectrum in real time until the acquisition finishes. During acquisition, the spectrum appears in blue; when acquisition is complete, the spectrum changes to green. At this point, the data is saved and can be loaded into the Data Analysis window.

Alternatively, you can launch the data analysis application from the Acquisition screen by clicking the ***Data Analysis*** push button. ChemStation automatically loads the last data file acquired into the Data Analysis window. You can also click on the ***Tabulate/Mass*** push button to obtain counts for the masses analyzed. For more information about data analysis, refer to Chapter 9, "Viewing Spectra".

You can change the type and scale of the Real-Time display. See the following section, "Altering the Display".

NOTE If you are analyzing samples using a method for Time Resolved Analysis or Time Program Acquisition, ChemStation displays up to 12 masses in the Real-Time display window. ChemStation displays the masses in two plots, each showing up to six masses. You can click ***Stop, Restart***, and ***Plot***. Clicking Plot opens the Display Parameters dialog box, which enables you to select the mass, order, and color of the mass plot.

Altering the Display

To alter the information displayed in the Real-Time window, choose any of the following:

- To adjust the vertical scale of the display, click the up (↑) or down (↓) arrow. Or, click the bar below the arrows and then click an appropriate scale in the drop-down list that appears.
- To display the information using a logarithmic intensity scale, click the ***Log*** check box.
- To display the information in counts per second, click the ***CPS*** check box.

Stopping a Run

Stopping a Run from the Real-Time Display Window

To stop a run from the Real-Time display before the run time elapses, click the ***Stop Run*** push button. The Agilent 7500 will stop in the middle of the current repetition.

CAUTION

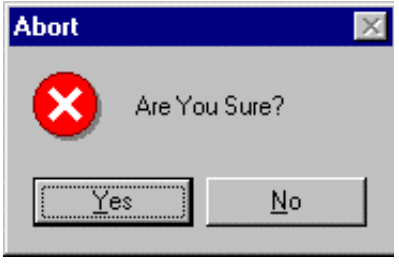
When you manually stop the run, data acquired for the current repetition is not saved.

NOTE

You can start the run again by clicking the ***Restart*** push button. ChemStation starts acquisition on the current sample using the sample information already provided. If you must change the sample information before running the sample, click ***Acquire Data>>Acquire Data*** in the ICP-MS Acquisition window, type the new sample information in the dialog box that appears, then click the ***Acquire*** push button.

Stopping a Run by Stopping a Method

If you want to stop the method you are currently running, click the ***Abort*** push button from the Run Method status window. A dialog box will pop up asking if you are sure you want to abort. Click ***Yes***, and the entire method becomes inactive.



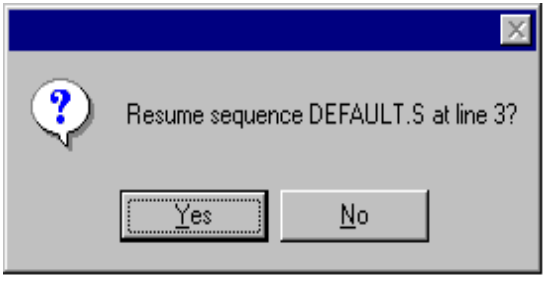
Abort Dialog Box

Stopping a Run by Stopping a Sequence

If you want to stop the sequence you are currently running, click the ***Abort*** push button from the Run Sequence status window. A dialog box will pop up asking if you are sure you want to abort. Click ***Yes***, and the entire sequence becomes inactive. ChemStation will, however, finish acquiring data from any method it has already begun, unless ***Stop Run*** has been pressed in the Real Time Display window.

ChemStation will, however, finish acquiring data from any method it has already begun, unless ***Stop Run*** has been pressed in the Real Time Display window.

If you start the same sequence once again, ChemStation will ask you if you want to start from where you stopped, or from the beginning. Click ***Yes*** if you want to continue, ***No*** if you want to start from the beginning. If you start from the beginning any previously saved files will be overwritten unless otherwise the data directory is no changed.



Restarting Sequence Dialog Box

NOTE

You can also stop just the current acquisition by double-clicking the ***ICP-MS Data Acquisition*** icon, then clicking the ***Stop Run*** push button. This procedure will not stop the sequence itself; it will stop data acquisition and data analysis for the current sample, and go on to the next line in the Sequence Log Table.

Stopping a Run by Stopping a Chained Sequence

To stop the chained sequence during the run, click the Abort button from the Edit Chained Sequence table. ChemStation will finish the chained sequence after running a sequence it has already begun.

To stop the sequence you are currently running, click the ***Abort*** push button from the Run Sequence status window. A dialog box will pop up asking if you are sure you want to abort. Click ***Yes***, and the entire sequence becomes inactive. ChemStation will, however, finish acquiring data from any method it has already begun, unless ***Stop Run*** has been pressed in the Real Time Display window.

9

Viewing Spectra

Viewing Spectra

The Spectrum acquisition mode displays data acquired from sample analysis as intensity versus mass (mass spectrum). ChemStation generates spectra for data acquired with a method using Spectrum or Isotope Analysis as the acquisition mode. For information about creating a method using these acquisition modes, see Chapter 5, "Creating a Method."

ChemStation offers three options for changing the spectral display type and enables you to change both the vertical and horizontal scales of the display. ChemStation also provides tools for identifying unknown peaks in a spectrum, enabling you to access information from the element databases in the process. Finally, ChemStation gives you the option of viewing data from sample analysis in tabulated as well as spectral form.

This chapter explains how to display and analyze spectra and view data in tabulated form.

Displaying Spectra

When you acquire data with a method for Spectrum acquisition and then access data analysis directly from data acquisition, ChemStation displays that data file. If you access the Data Analysis window from the Top menu, the Data Analysis window displays the ICPDEMO.D data file which is indicated in the title bar. If you access from the Offline Data Analysis, the last data file from your previous ChemStation session, will be loaded automatically. If the file name shown in the title bar is not the one you want to view, you must load the appropriate data file.

The spectrum that appears is divided into three rows and has defaults selected for the display type and the vertical and horizontal scales. The default display type is total counts, bar graphic mode, and linear Y-axis. The Y scale automatically adjusts, depending on the intensity of the highest peak in each row.

The following sections explain how to load a data file, change the display type, use a count box, and change the vertical and horizontal scales of the display.

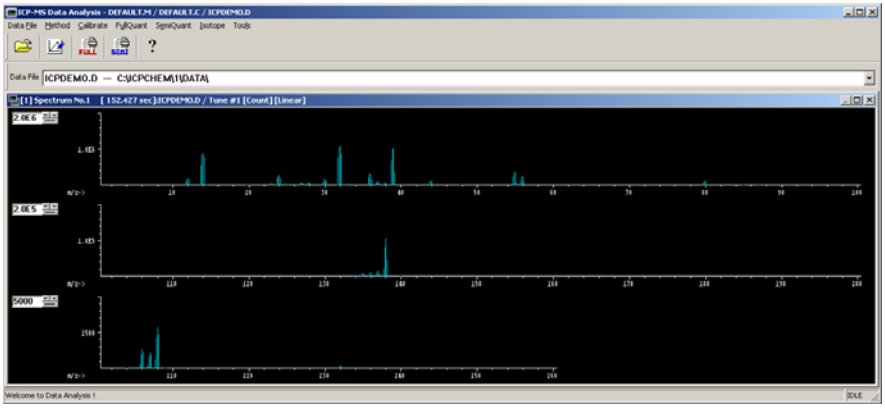
Loading a Data File

To load a different data file, complete the following steps:

1 **Select *Top>>Data Analysis>>Main Panel.***

The ***ICP-MS Data Analysis*** window appears, showing a display of the ICPDEMO.D data file.

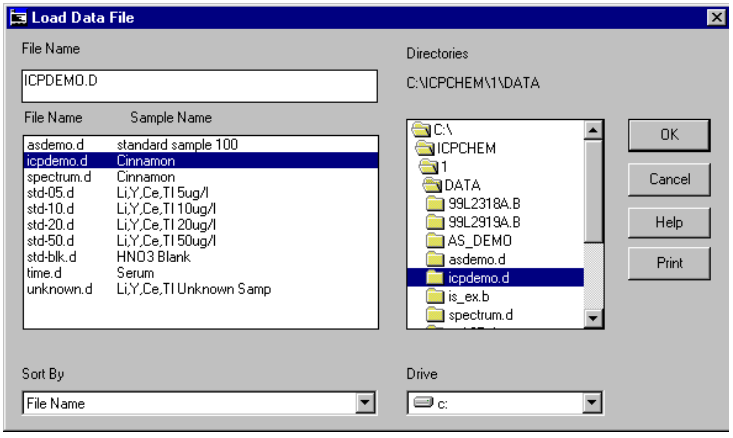
When data is acquired with Multi Tune, multiple spectrum windows appear which correspond with each Tune Step. The tune step number, for example **Tune#1**, **Tune#2**..., is shown in the title bar. When the Multi Tune is not used, only one spectrum is shown with **Tune#1** on the title bar.



ICP-MS Data Analysis Dialog Box

2 Select *Data File>>Load*.

The ***Load Data File*** dialog box appears, showing the path for the data file directory. Allows the user to select the data file to be loaded into data analysis for processing.



Load Data File Dialog Box

- Sort By

  Allows the user to sort the list of data files in the current directory by the selected type of information. Options include File Name, Sample Name, Misc Info, and Acquired Date.

NOTE

Recently opened data files are also selected from the ***Data File*** list box in the ***ICP-MS Data Analysis*** window.

3 Select the data file in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

If the directory path in the Load Data File dialog box is incorrect, you must select the correct directory path.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

The spectrum for the loaded file appears in the ICP-MS Data Analysis window, showing a mass spectrum for all elements analyzed. This file contains the mean values for data acquired from all repetitions set in the method.

There are four colors used in the spectrum.

- Pulse counting — Blue
- Analog modes — Green
- Mix — Purple
- Skipped due to excessive signal — Yellow

Changing the Display Type

ChemStation provides options for changing three aspects of the spectral display type. You can choose whether the Y-axis display is logarithmic or linear, whether the display reflects total counts or counts per second (CPS), and whether the display uses a bar or line graphic mode. The default display type is a linear Y-axis showing total counts using a bar graphic mode.

To change the spectral display type, complete the following steps:

1 **Click the horizontal Control Box Menu at the top left of the mass spectrum display window.**

The window menu appears.

2 **Select a display type by clicking *Log, CPS*, or *Line Display.***

- Log

  The Y-axis will change to a logarithmic scale.

- CPS

  The spectrum will be displayed in counts per second (cps).

- Line Display

  The spectrum will be displayed as a line graph.

NOTE You can only select one display type at a time. If you want to select more, repeat the above steps.

3 **To deselect display types, click the Control Box Menu and then click the display type you want to deselect.**

The display types are selected if they have check marks to the left of the type. Clicking the selection again removes the check mark and deselects the display type.

NOTE The display type remains selected until you deselect it or until you load a new data file.

Using the Display Count Box

Additionally, you can view the counts acquired for each point by using the Count Box. This ChemStation feature gives you an easy method to check the total counts or CPS at any point in each peak. You can use this feature to estimate the concentration of any element relative to the internal standard.

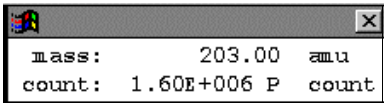
To display the count box, complete the following steps:

1 **Click the horizontal Control Box Menu at the top left corner of the mass spectrum display window.**

The window menu appears.

2 **Click *Display Count Box* at the bottom of the menu.**

The Count Box appears, showing the AMU value at that point and the intensity in either total counts or CPS. The letter following the value indicates the detector mode used analog (A) or pulse (P).



Count Box

3 **To view counts at any point, place the cursor at the specific point on the display and look at the count box.**

If the spectral display appears empty at the cursor but the count box shows counts for that mass, you may want to change the vertical scale of the display. For information, see "Changing the Vertical Scale of a Spectral Display" later in this chapter.

NOTE

The count box displays until you close it by double-clicking the count box Control Box Menu or by exiting the spectral display.

Changing the Vertical Scale of a Spectral Display

The scale for each row of the spectrum appears to the left of the row, and the number in the scale box indicates the scale of the current display. Increase or decrease the vertical scale as required.

To change the vertical scale for each row of the spectral display, complete one of the following steps:

- Click the up or down arrow to the right of the scale box.

  The full scale height decreases if you click the down arrow and increases if you click the up arrow.

- Click the horizontal bar to the right of the scale box (under the two arrows). Select a number from the displayed list by clicking it.

  The full scale height decreases if you click a smaller number than the number that appears in the scale box and increases if you click a larger number than the number that appears in the scale box.

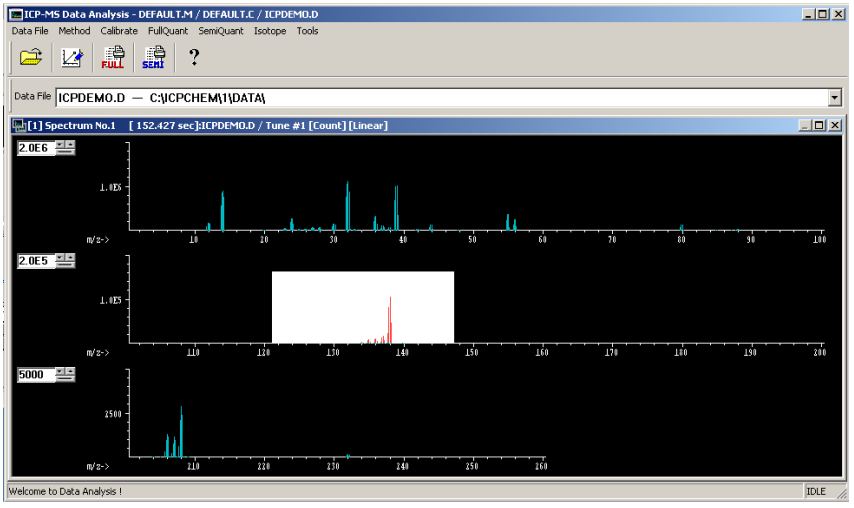
NOTE Regardless of which method you use, you must repeat the process for each row in the display window.

Changing the Horizontal Scale of a Spectral Display

Just as you can change the vertical scale of a spectral display, you can also change the horizontal scale. You can change the horizontal scale of the entire display or you can zoom in on one part of the display.

To change the horizontal scale of the entire display, position the cursor within the spectral display and double-click the left mouse button.

The spectral display changes from three rows to one row that shows masses from 0 to 260.



One-Row Spectral Display

NOTE To return to the default horizontal scale of three rows, double-click the left mouse button again.

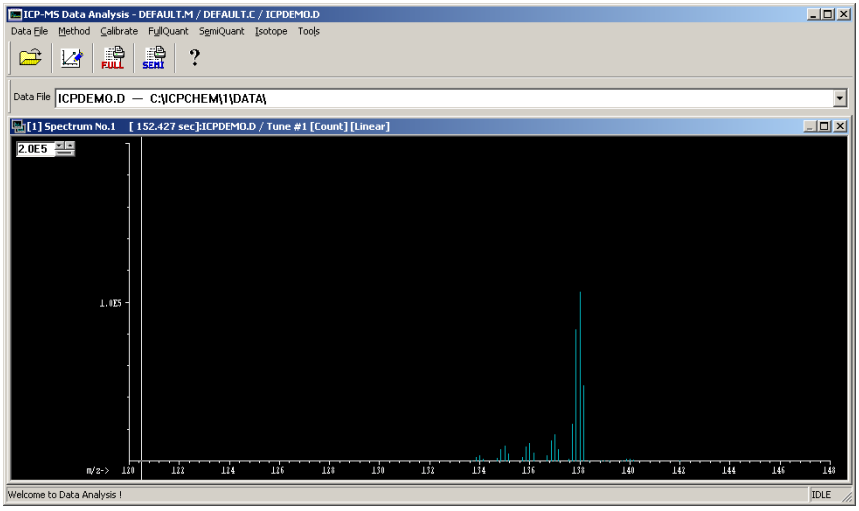
To zoom in on one section of the display, complete the following steps:

1 **Drag across the section of the display you want to enlarge.**

The section is highlighted.

2 **Release the mouse button.**

Only the section you highlighted fills the display window.



Spectral Display after Zooming In

Return to the default horizontal scale by completing one of the following steps:

- To return to the default from the one-row spectral display, double-click the left mouse button.

  The spectrum returns to the default display of three rows.

- To return to the default after zooming in on a section of the display, double-click the left mouse button twice.

  ChemStation returns you first to the one-row display and then to the three-row display.

Analyzing Spectra

To analyze spectra in ChemStation, you can access information from the element databases to identify unknown peaks. You can then match this information with a spectrum by using a template that overlays a selected peak on the spectrum.

Using the Template

ChemStation enables you to match information from the element databases with the data displayed in a spectrum. To analyze the spectrum, however, you may want to focus on a particular section of the mass scale. For information about enlarging a portion of the mass scale, see "Changing the Horizontal Scale of a Spectral Display" in this chapter.

NOTE You cannot use the template if the adjacent masses were not measured originally.

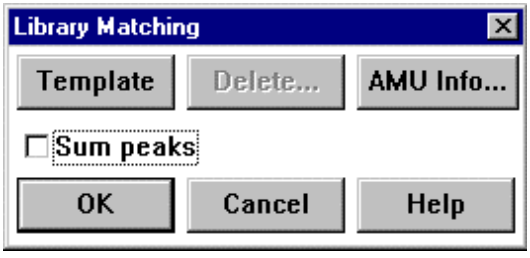
1 **Place the cursor on a peak that you want to analyze.**

A vertical line extends through the entire height of the display, dissecting the peak where you place the cursor.

To view a peak more clearly, change to a line display. See "Changing the Display Type" earlier in this chapter for more information.

2 **Double-click the right mouse button.**

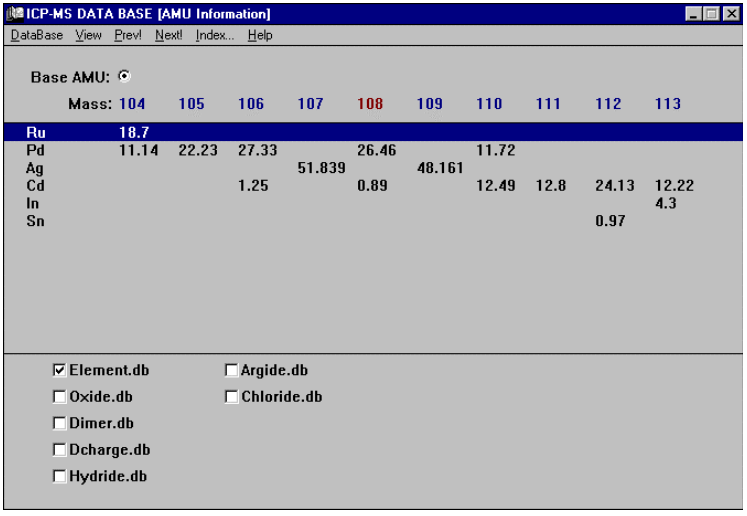
The ***Library Matching*** dialog box appears.



Library Matching Dialog Box

3 Click *AMU Info*.

The ***ICP-MS DATABASE [AMU Information]*** dialog box appears, listing information about the abundance of elements at specific masses.



ICP-MS DATABASE (AMU Information) Dialog Box

ChemStation displays the mass you selected in red.

As a default, only the element database (element.db) is loaded. You can activate other databases containing various polyatomic species such as oxides and hydrides by clicking the appropriate database check box at the bottom of the dialog box. You can select multiple databases.

This dialog box also enables you access to other information. Refer to Chapter 17, "Editing The Database" for more information about the ICP-MS DATABASE.

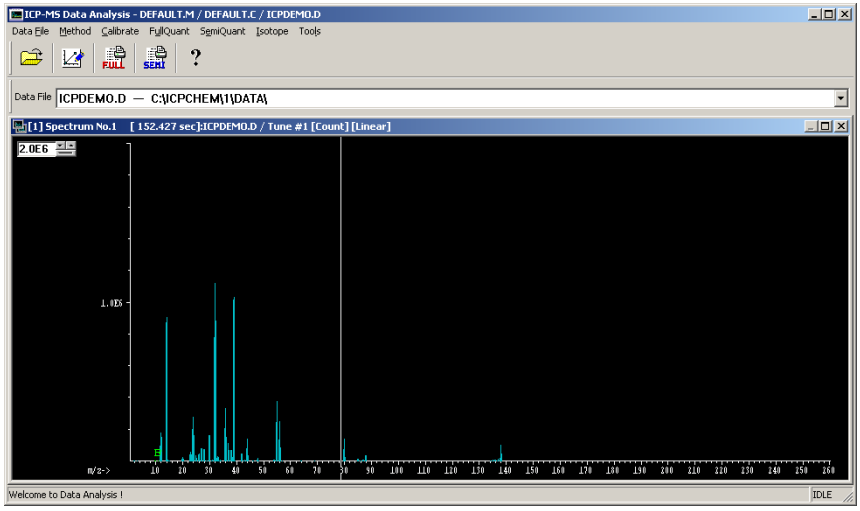
4 Click the element in the list for which you want to use a template overlay.

The element in the list is highlighted. A radio button is placed indicating which mass intensity the template will be based on, called the Base AMU.

Select the element which has an isotope at the mass you selected.

5 Click *Template* in the *Library Matching* dialog box.

The template for the selected element now overlays the spectral display. The template is displayed based on the Base AMU - a signal at the Base AMU is counted as 100% for this element.



Library Matching Template (e.g. Ba)

6 To place more templates, repeat the above steps.

The templates for additional elements appear. The template for each element has a unique color and shows the element name at the top of Base AMU. The templates can be displayed up to ten elements.

To add two templates together, click the ***Sum Peaks*** check box and then ***OK*** in the Library Matching dialog box. ChemStation enables you to see how the combined template matches the peak you are analyzing. ChemStation shows combined masses in red.

When the mass used as the template of another element is applied as the Base AMU, a rest peak that the total signal minus template is counted as 100% of the element, and the second template will be displayed based on a height of the rest peak. At the mass you selected, the template matches the peak height for the mass. If the element template fits - i.e. if the other template bars all match up with peaks in the spectrum, at their corresponding heights, the element ID is probably correct. If not, try other elements at the same mass to see if there is a better fit. If no element fits, then poly-atomic interference is probable.

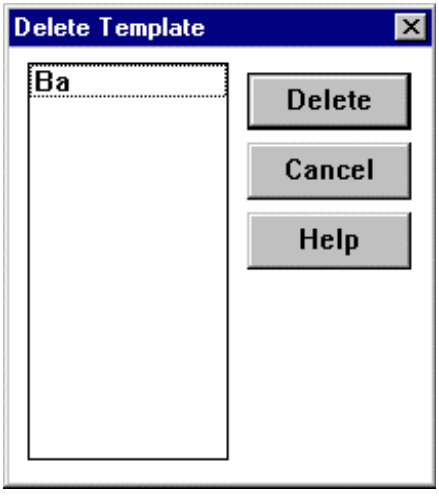
To delete a template from the spectral display, complete the following steps:

1 **Double-click with the right button on a peak with a template overlay.**

The ***Library Matching*** dialog box appears.

2 **Click *Delete* in the dialog box.**

The ***Delete Template*** dialog box appears.



Delete Template Dialog Box

3 **Click the element you want to delete the template for and then click *Delete*.**

The dialog boxes close, and the template disappears from the spectral display.

Labeling Spectra

You may want to label a spectrum after you have added templates to it or before you print it. ChemStation enables you to name or add identifying information to a spectrum, a feature which is useful if you want to save a hard copy of a spectrum for future reference.

To label a spectrum, complete the following steps:

1 **Load the appropriate file in the *ICP-MS Data Analysis* window.**

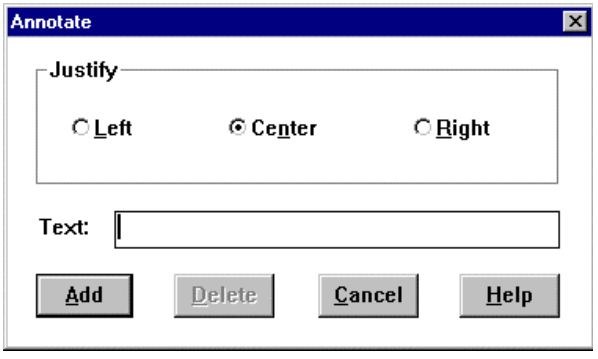
For more information, see "Loading a Data File" in this chapter.

2 **Place the mouse pointer exactly where you want the label to appear.**

Note both the horizontal and vertical placement of the mouse pointer.

3 **Click both mouse buttons at the same time.**

The ***Annotate*** dialog box appears.



Annotate Dialog Box

4 **Click the *Left, Center*, or *Right* Justify button.**

The label will appear to the right, center, or left of the location you specified in Step2.

5 **Type a name or other identifying information in the text box.**

6 **Click *Add*.**

ChemStation returns to the spectrum, and the information you typed appears at the location of the cursor. You can make as many labels as you like.

NOTE You can change and delete the text by clicking exactly on top of the previously labeled point, rewriting the text, then clicking ***Update*** in the Annotate dialog box.

NOTE Exiting the Data Analysis window or loading the another file erases the template and annotation.

Printing Spectra

You can print a displayed spectrum at any time during the analytical process. For example, if you have changed the scale or the spectrum type, the newly displayed spectrum will be printed.

The following header information will be printed on the spectrum.

- File name
- Operator name (ACQ, DA)*

  * ACQ and DA are differentiated only when the access control pack is used.
- Date/time of measurement
- Measurement mode
- Sample type
- Sample name
- Comment
- Pre-dilution factor
- Automatic dilution factor
- Overall dilution factor
- Method
- ISTD concentration

To print the displayed spectrum, select ***Data File >> Print***.

Subtracting Background Spectra

It is sometimes useful to be able to subtract background polyatomic species to visually inspect a spectrum. ChemStation can subtract any data file from another. This background correction will subtract the abundance of each mass in the background spectrum from the abundance of the corresponding mass in the spectrum of the current data file. It is normally used for graphical purposes. It is not to be confused with blank subtraction, which subtracts concentration data in quantitative analysis.

To subtract the background spectrum from another spectrum, complete the following steps:

1 **Select *Method>>Data Correction*.**

The ***Set Correction*** dialog box appears.

2 **Click *Subtract Background.***

3 **Click *Browse*.**

The ***Select Data File*** Dialog box appears, showing the path of the data file directory and a list of data files.

4 **Select a file name from the list by clicking it and then clicking *OK*.**

ChemStation subtracts the data in the selected file from all other data files acquired with the same method.

5 **Select the box, *Rejected Masses* and enter the masses for which background spectrum you do not want subtracted.**

The background spectrum for the masses you edited in the Rejected Masses box will not be subtracted. It is used when you do not want to subtract the spectrum for specific masses; for example the spectrum for internal standards.

6 **Click *OK*.**

The net spectrum will be displayed.

Viewing a Tabulated Spectrum

ChemStation enables you to view results from sample analysis in tabulated as well as graphic form. Two modes of tabulation are available for each data file you load into the Mass Spectrum display window. The first mode provides information about counts reported for each point per peak in the sample. The second mode provides information about the cumulative counts of each mass measured in the sample. The following sections explain how to view these two modes.

Performing a Points per Peak Tabulation

ChemStation provides detailed information about each point per peak in a sample. You can view this detailed information by performing a points per peak tabulation.

Before you can perform any tabulation, you must load a data file into the Data Analysis window if one is not already showing. For information about loading the data file, see "Loading a Data File" in this chapter.

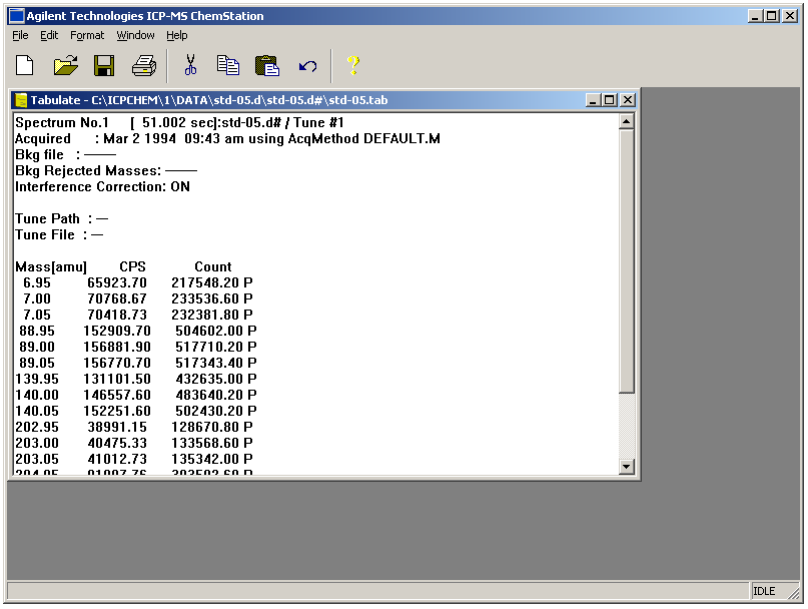
To perform a points per peak tabulation on a data file, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

The ICP-MS Data Analysis window appears.

2 **Select *Data File>>Tabulate Spectrum/Point*.**

The Report Pad - ***DATA FILE NAME.TAB*** window appears. This window shows a tabulated file giving information about the points per peak for the current data file. The spectrum number, the acquisition time in seconds, and the data file name appear at the top of the file.



Points per Peak Tabulation

The tabulation provides information about the mass, counts per second (CPS), count, and detector mode for each point per peak of each element analyzed. The amount of information contained in the tabulation depends on the peak pattern set and number of elements selected in the method. For example, if you use a method with a peak pattern of three points per peak, the tabulation contains information about three masses for each selected mass. Thus tabulation might contain information for Li (mass 7) at the following three masses: 6.95, 7.00, and 7.05. The sum of the abundance for these three masses is equal to the counts detected for the nominal mass, as displayed on the mass tabulation (see below) or on the quantitative report.

To print out the tabulation, select ***File>>Print.***

When you are finished working with the file, select ***File>>Exit.***

Performing a Mass Tabulation

ChemStation also provides detailed information about each mass in a sample. You can view this detailed information by performing a mass tabulation.

Before you can perform any tabulation, you must load a data file into the Data Analysis window if one is not already showing. For information about loading the data file, see "Loading a Data File" in this chapter.

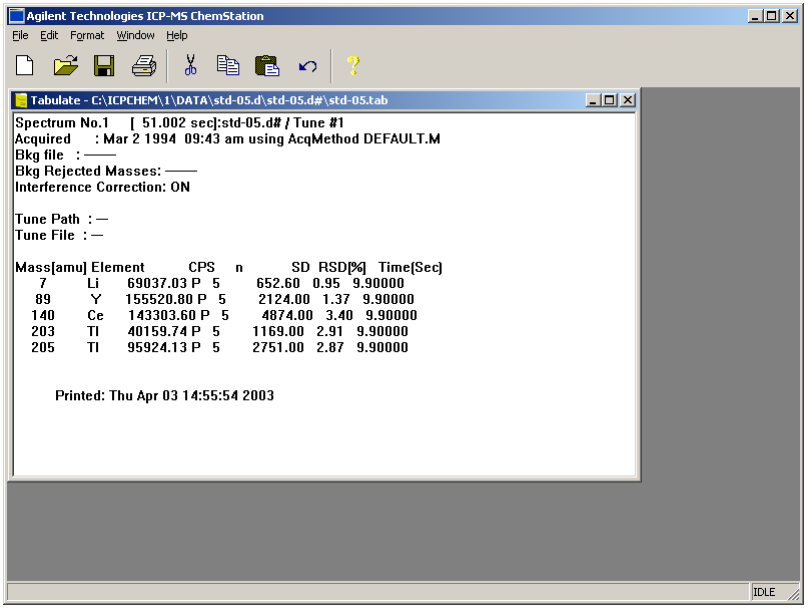
To view a tabulation giving information about the elements in a sample, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

The ICP-MS Data Analysis window appears.

2 **Select *Data File>>Tabulate Spectrum/Mass*.**

The Report Pad - ***DATA FILE NAME.TAB*** window appears. This window shows a tabulation containing information about the elements for the current data file. The spectrum number, the total acquisition time in seconds, and the data file name appear at the top of the file.



Mass Tabulation

The tabulation provides information about the mass, element name, counts per second (CPS), detector mode, repetition, SD, RSD and integration time for each element analyzed in the sample.

You may occasionally want to save a tabulated file to work with at a later time. To do so select ***File>>Save As*** and give the file with a new name. Otherwise, ChemStation will overwrite the DATA FILE NAME.TAB file the next time you view a tabulation.

To print out the tabulation, select ***File>>Print***.

When you are finished working with the file, select ***File>>Exit.***

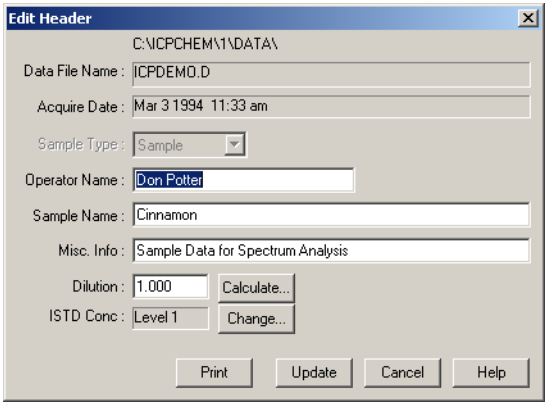
Editing and Printing Out the Header Information

ChemStation enables you to edit the header information (the same information you edited when you acquired data) in the ICP-MS Data Analysis window.

To edit the header, complete the following steps:

1 **Select *Data File>>Edit Header.***

The ***Edit Header*** dialog box appears.



Edit Header Dialog Box

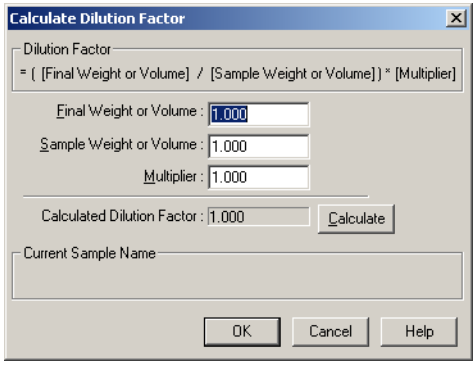
2 **Type a sample name and comment if necessary.**

3 **Set a dilution factor if necessary.**

Enter a dilution factor directly or click ***Calculate*** and follow the procedure described below.

1) Click ***Calculate***.

The ***Calculate Dilution Factor*** dialog box will appear.



Calculate Dilution Factor Dialog Box

2) Enter a numerical value in each text box.

3) Click ***Calculate***.

The dilution factor will be automatically calculated.

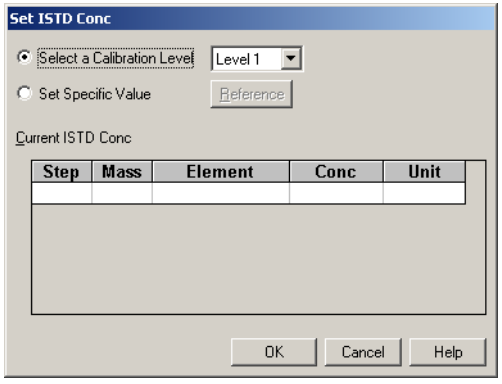
4) Click ***OK***.

The ***Calculate Dilution Factor*** dialog box closes and the dilution factor will be set in the ***Edit Header*** dialog box.

4 Set the internal standard concentration level if necessary.

1) Click ***Change***.

The ***Set ISTD Conc*** dialog box appears.



Set ISTD Conc Dialog Box

2) To select a concentration from the calibration levels, choose the ***Select a Calibration Level*** radio button and select a level from the drop-down list.

Proceed to step 5).

3) To set a specific value for the current sample, select the ***Set Specific Value*** radio button.

4) Set the concentration in the ***Conc*** field in the Current ISTD Conc table.

The Current ISTD Conc table shows the internal standard currently set for calibration. Click on the ***Conc*** field and enter a concentration level.

Click the right mouse button to display the popup menu, which allows you to copy, paste, or delete the field.

Click ***Browse*** to set the concentration setting for each calibration level in the Concentration field.

5) Click ***OK***.

The ***Set ISTD*** Conc dialog box will close and the internal standard concentration level will be set in the ***Edit Header*** dialog box.

If the ***Select a Calibration Level*** radio button was selected, the level number will be displayed. If the ***Set Specific Value*** radio button was selected, "Manual" will be displayed.

NOTE If your QC configuration does not include any QC parameters, the Sample Type section will be grayed out.

5 Click the *Update* button.

The dialog box disappears and the new information will be updated (e.g. if you change the dilution factor here, it will be reflected in the reports you generate).

NOTE You can display the Header information on the screen by simply clicking the ***Print*** push button in this dialog box. If you want to print it on the printer, select ***Data File>>Print***.

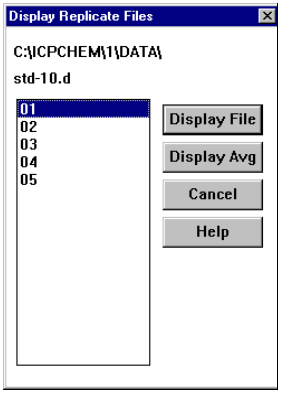
Displaying Replicate Files

When more than one repetition is set in a method for spectrum acquisition, ChemStation creates a data file, or replicate, for each repetition.

To view each individual replicate, complete the following steps:

1 **Select *Data File>>Display Replicate Files*.**

The ***Display Replicate Files*** dialog box appears with the list of repetitions.



Display Replicate Files Dialog Box

2 **Select the replicate file that you want to view, then click the *Display File* push button.**

The selected replicate file appears on the ICP-MS Data Analysis window. The selected file will be indicated on the title bar with the number as the extension of the file (e.g. UNKNOWN.02). You can perform data analysis on that replicate file.

3 **To return to the Average file, select *Data File>>Display Replicate Files* once again and click on the *Display Avg* push button.**

The ICP-MS Data Analysis will display the Average file. It will be indicated on the title bar with the.d extension. If the method is set to correct data (using either background subtraction or interference correction), the file extension will be.***d#.***

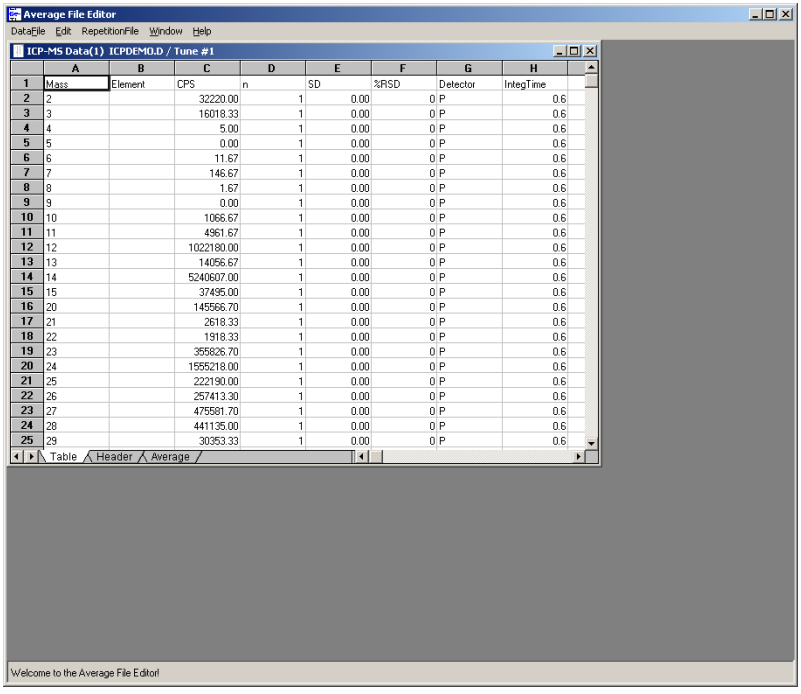
Editing Spectral Information

ChemStation enables you to transfer data to a database to view individual replicates, edit data files, and other data from the ICP-MS Data Analysis window. This section explains how to transfer data files to the database, and how to work on the workbook.

Performing Edit Average Files

ChemStation enables you to view information about all replicate, as well as, average the data in a workbook. The information includes the header information, masses and elements analyzed, counts, number of repetitions, standard deviation (SD), detector mode used for acquisition, and integration time.

To view this database spreadsheet containing the above information, Select ***Data File>>Edit Average File***.



	A	B	C	D	E	F	G	H
1	Mass	Element	CPS	n	SD	%RSD	Detector	IntegTime
2	2		32220.00	1	0.00	0	P	0.6
3	3		16018.33	1	0.00	0	P	0.6
4	4		5.00	1	0.00	0	P	0.6
5	5		0.00	1	0.00	0	P	0.6
6	6		11.67	1	0.00	0	P	0.6
7	7		146.67	1	0.00	0	P	0.6
8	8		1.67	1	0.00	0	P	0.6
9	9		0.00	1	0.00	0	P	0.6
10	10		1066.67	1	0.00	0	P	0.6
11	11		4961.67	1	0.00	0	P	0.6
12	12		1022180.00	1	0.00	0	P	0.6
13	13		14056.67	1	0.00	0	P	0.6
14	14		5240607.00	1	0.00	0	P	0.6
15	15		37495.00	1	0.00	0	P	0.6
16	20		145566.70	1	0.00	0	P	0.6
17	21		2618.33	1	0.00	0	P	0.6
18	22		1918.33	1	0.00	0	P	0.6
19	23		355826.70	1	0.00	0	P	0.6
20	24		1555218.00	1	0.00	0	P	0.6
21	25		222190.00	1	0.00	0	P	0.6
22	26		257413.30	1	0.00	0	P	0.6
23	27		475581.70	1	0.00	0	P	0.6
24	28		441135.00	1	0.00	0	P	0.6
25	29		30353.33	1	0.00	0	P	0.6

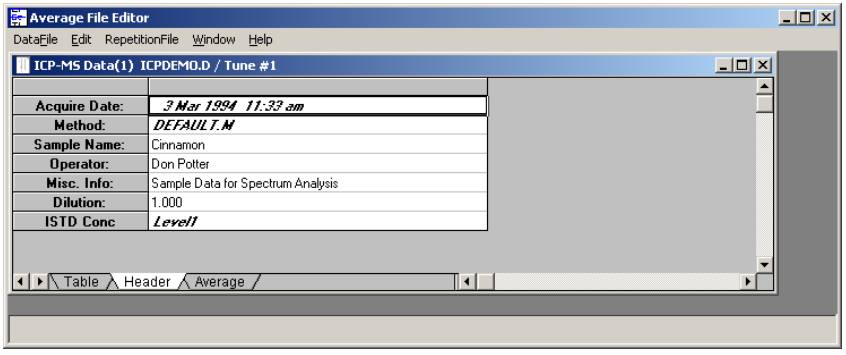
Workbook

Editing Header Information

You are able to edit the header information on the database spreadsheet.

Edit the header information on the ***Header*** sheet (indicated at the bottom of the work sheet).

The sample name, operator, miscellaneous information and dilution factor can be edited.



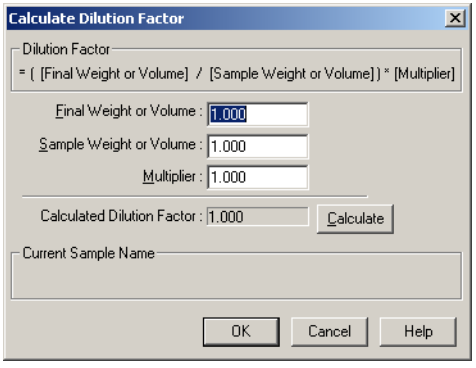
Header Information Work Sheet

The sample name and comments can be entered directly.

The dilution factor can be set using the following procedure.

1 **Double-click on the Dilution: field.**

The ***Calculate Dilution Factor*** dialog box will appear.



Calculate Dilution Factor Dialog Box

2 **Enter a numerical value in each text box.**

3 **Click *Calculate*.**

The dilution factor will be automatically calculated.

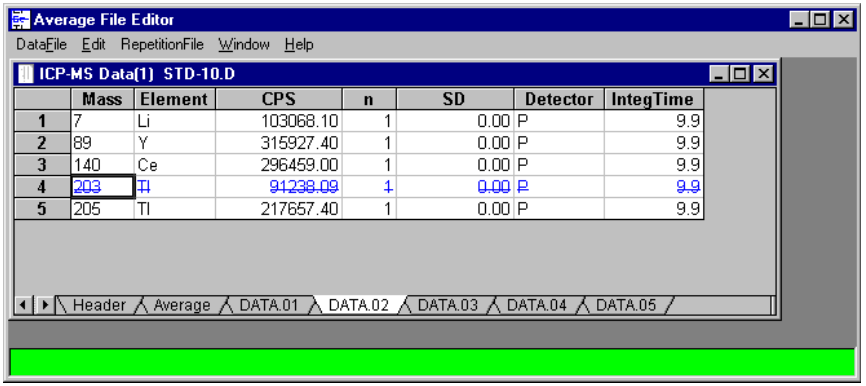
4 Click *OK*.

The ***Calculate Dilution Factor*** dialog box will close and the dilution factor will be set.

Viewing and Editing Replicate Files

You are able to view and edit replicate files individually on the workbook. Editing the replicate files includes excluding certain data from the average file in case the data was not acceptable. An example would be in the event there was insufficient sample volume in the sample vial, with the result that the sample ran out during acquisition. For auditing purposes, only the spread sheet is altered, not the original data files, which are binary files and not editable.

Select the sheet with the replicate data file name indicated at the bottom of the work sheet, then the replicate data file appears.



	Mass	Element	CPS	n	SD	Detector	IntegTime
1	7	Li	103068.10	1	0.00	P	9.9
2	89	Y	315927.40	1	0.00	P	9.9
3	140	Ce	296459.00	1	0.00	P	9.9
4	~~203~~	~~Tl~~	~~91238.09~~	~~1~~	~~0.00~~	~~P~~	~~9.9~~
5	205	Tl	217657.40	1	0.00	P	9.9

Data of a Replicate File (One Element Excluded)

To exclude a certain element on a certain replicate file, or to exclude a whole replicate file from the average file, complete the following steps:

1 **Place the cursor on the element or any other cell in the same row you want to exclude and click.**

The selected cell will be outlined.

2 **Select *RepetitionFile>>Exclude Element(s).***

The selected element will be crossed out in blue.

NOTE To exclude the whole replicate file, ***RepetitionFile>>Exclude All Elements***.

3 **Select *RepetitionFile>>Recalculate Average.***

The database will recalculate the average file not including the data just excluded and display the average data sheet.

To include the element you excluded on a certain replicate file, or to include a whole replicate file that you excluded from the average file, complete the following steps:

1 **Place the cursor on the element or any other cell in the same row you want to include and click.**

The selected cell will be outlined.

2 **Select *RepetitionFile>>Include Element(s).***

The crossed out element will be restored.

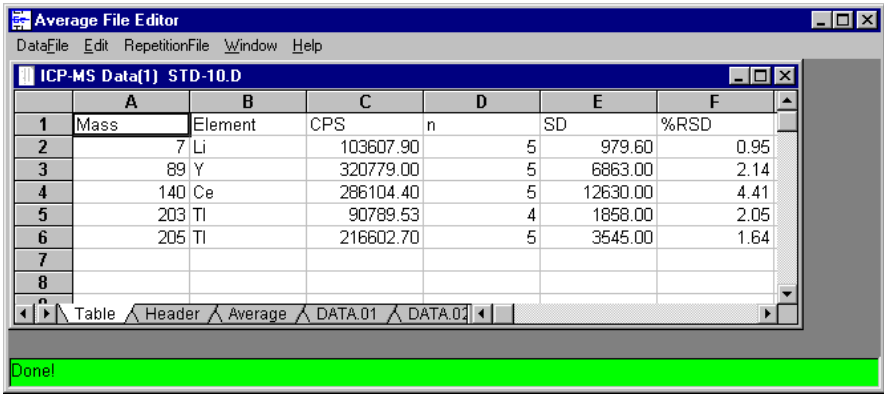
NOTE To include the whole replicate file, select ***RepetitionFile>>Include All Elements***.

3 **Select *RepetitionFile>>Recalculate Average*.**

The data base will recalculate the average file, including the data you just restored and display the average data sheet.

Tabulating the Spectral Information

You can tabulate the spectral information on one sheet. The information to be tabulated are: mass, element, average counts per second (CPS), number of repetitions, standard deviation (SD), relative standard deviation (RSD), detector mode, integration time, and the counts per second data for each replicate file. If some elements or replicate files are excluded, this will be reflected on the table by leaving a blank cell, where data was excluded.



	A	B	C	D	E	F
1	Mass	Element	CPS	n	SD	%RSD
2	7	Li	103607.90	5	979.60	0.95
3	89	Y	320779.00	5	6863.00	2.14
4	140	Ce	286104.40	5	12630.00	4.41
5	203	Tl	90789.53	4	1858.00	2.05
6	205	Tl	216602.70	5	3545.00	1.64
7						
8						

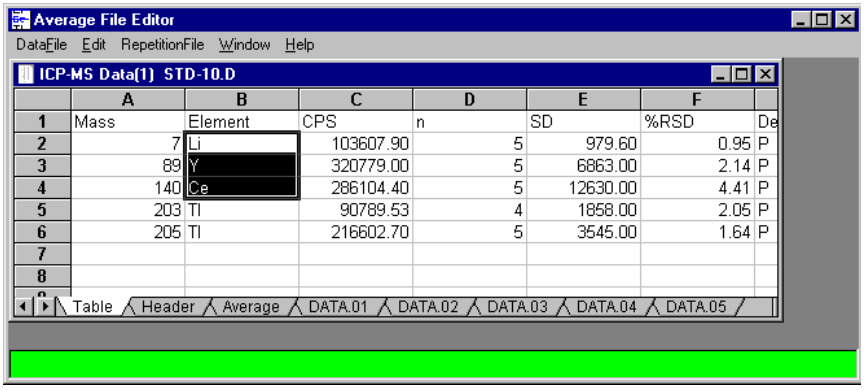
Table Work Sheet

Drawing a Graph from the Tabulated Information

Once you have tabulated the spectral information, you can draw a graph for the elements of interest.

To draw a graph for the elements of interest, complete the following steps:

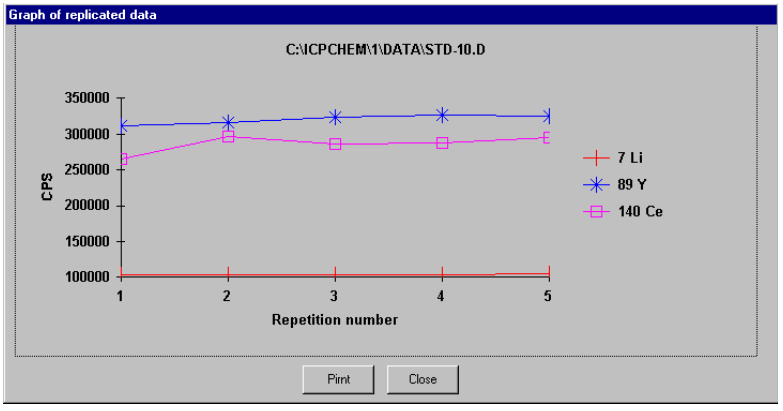
1 **Select the cells for elements of interest in the Table work sheet.**



	A	B	C	D	E	F	
1	Mass	Element	CPS	n	SD	%RSD	De
2	7	Li	103607.90	5	979.60	0.95	P
3	89	Y	320779.00	5	6863.00	2.14	P
4	140	Ce	286104.40	5	12630.00	4.41	P
5	203	Tl	90789.53	4	1858.00	2.05	P
6	205	Tl	216602.70	5	3545.00	1.64	P
7							
8							

2 **Select *Edit>>Graph*.**

The graph is automatically drawn.



Graph of replicated data

You can print the graph by clicking on ***Print***.

NOTE You can save what you edited on the workbook by selecting ***DataFile>>Save***. Data can be saved as text by selecting ***DataFile>>Save as Text***.

NOTE **You can load a different data file by selecting *DataFile>>Open***, then selecting a file.

10

Viewing a Time Chart

Viewing a Time Chart

A time chart displays data acquired from sample analysis as intensity versus time. ChemStation generates a time chart for data acquired with a method using Time Resolved Analysis or Time Program as the acquisition mode. For information about creating methods using these two acquisition modes, see Chapter 5, "Creating a Method".

You can view a time chart showing the combined results for all elements analyzed or view a separate chart for each element.

This chapter explains how to view a time chart for a data file you load, change the type and scale of the display, and analyze the time chart.

NOTE ChemStation provides Time Resolved Analysis and Time Program acquisition modes for transient signal monitoring for applications such as Laser Ablation, ETV, IC and LC. You must use peripheral equipment in combination with the Agilent 7500 for these applications. For more information, refer to the manual provided with the peripheral equipment you are using.

Displaying a Time Chart

When you acquire data with a method for Time Resolved Analysis or Time Program acquisition and then access Data Analysis directly from Data Acquisition, ChemStation displays the time chart you just acquired. If you access the Data Analysis window from the Top menu, the Data Analysis window displays the ICPDEMO.D data file which is indicated in the title bar. If you access from the Offline Data Analysis icon, the last data file from your previous ChemStation session, will be loaded automatically. That display may be a spectral display rather than a time chart, so you must load the appropriate method and data file to view a time chart.

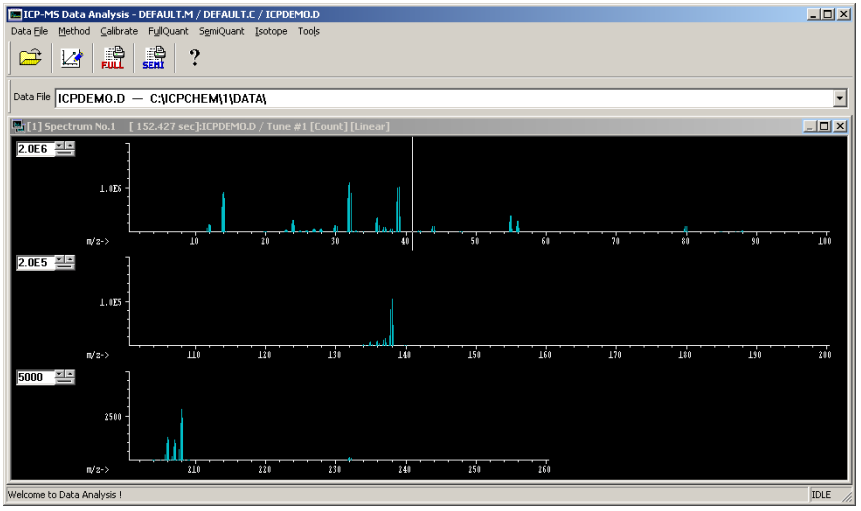
The following sections explain how to load a data file, change the type of chart being displayed in the time chart window, and change the vertical and horizontal scales of the display.

Loading a Data File

To load a different data file, complete the following steps:

1 **Select *Top>>Data Analysis>>Main Panel.***

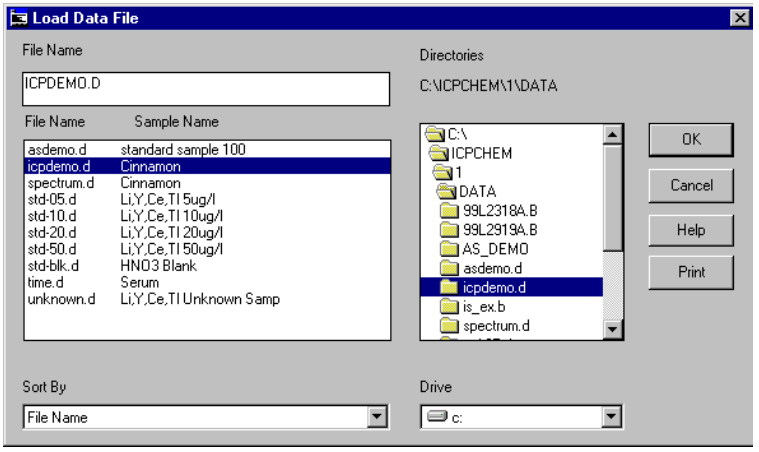
The ***ICP-MS Data Analysis*** window appears, showing a display of the ICP-DEMO.D data file.



ICP-MS Data Analysis Window

2 Select *Data File>>Load*.

The ***Load Data File*** dialog box appears, showing the path for the data file directory. Allows the user to select the data file to be loaded into data analysis for processing.



Load Data File Dialog Box

- Sort By

  Allows the user to sort the list of data files in the current directory by the selected type of information. Options include File Name, Sample Name, Misc Info, and Acquired Date.

NOTE

Recently opened data files are also selected from the ***Data File*** list box in the ***ICP-MS Data Analysis*** window.

3 Select the data file in one of the following ways:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

If the directory path in the Load Data File dialog box is incorrect, you must select the correct directory path.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

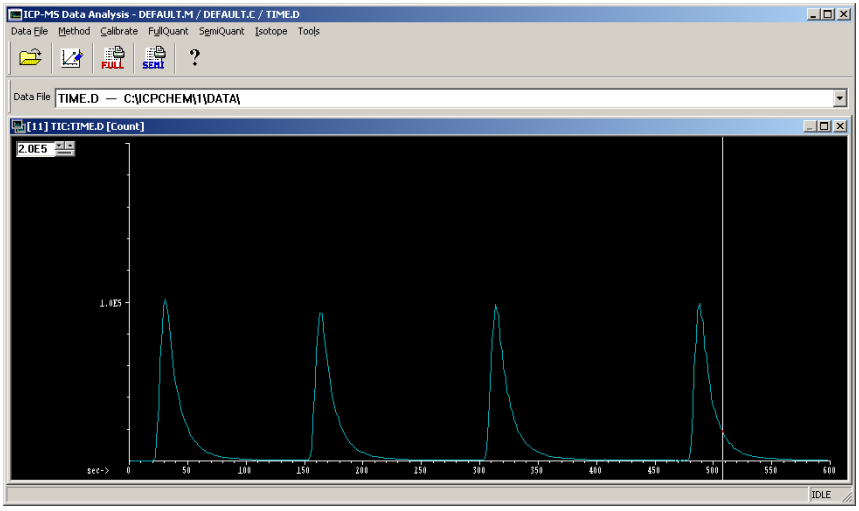
and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

The spectrum for the loaded file appears in the ICP-MS Data Analysis window, showing a mass spectrum for all elements analyzed. This file contains the mean values for data acquired from all repetitions set in the method.

The Total Ion Chart for the loaded file appears in the Data Analysis display window. This chart shows total intensity versus time for all elements analyzed. The time is shown in seconds and represents the total data acquisition time.



Total Ion Chart Display

Changing the Chart Type

The Total Ion Chart is the default time chart shown in the ICP-MS Data Analysis display window when an appropriate data file is loaded. It provides a single intensity versus time plot for all elements analyzed in a sample. However, ChemStation also enables you to view an extracted ion chart, which provides an intensity versus time plot for each mass.

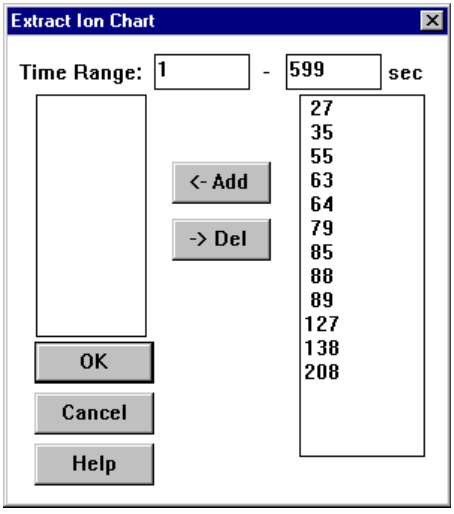
To view an extracted ion chart, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

The ICP-MS Data Analysis window appears.

2 **Select *Data File>>Extract Ion Chart*.**

The ***Extract Ion Chart*** dialog box appears.



Extract Ion Chart Dialog Box

3 **To select the part of the acquisition period for which you want the extracted ion chart to appear, type the new values, in seconds, in the Time Range text boxes at the top of the dialog box.**

The default time range is the total acquisition period.

4 **To select the element(s) for which you want to display an extracted ion chart, click the element in the displayed list and then click the *Add* push button.**

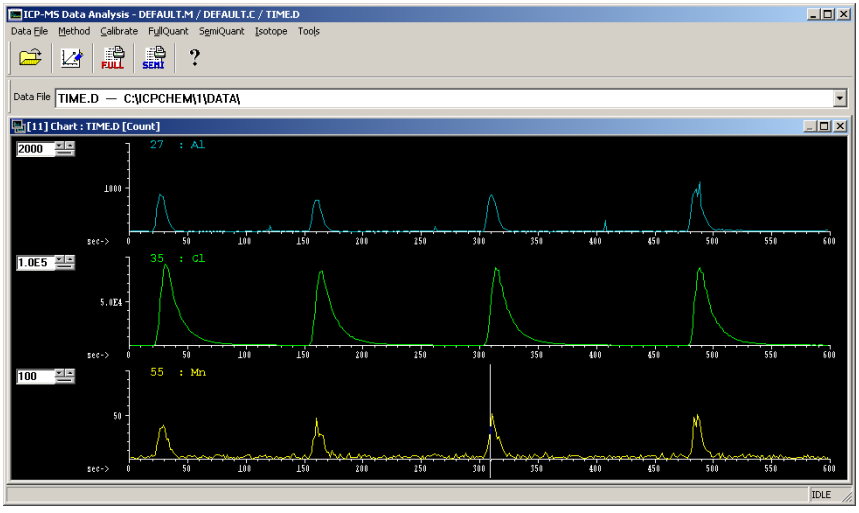
The selected element appears in the box on the left side of the dialog box. To select more than one element, hold down the Control key and click the elements in the displayed list or drag in the list, then click ***Add***. You can select up to 10 elements.

NOTE

You can delete elements by clicking on the elements you want to delete on the left side list, then clicking the ***Del*** push button.

5 Click *OK*.

ChemStation returns to the Data Analysis window and an extracted ion chart appears for each selected element for the time range you indicated.

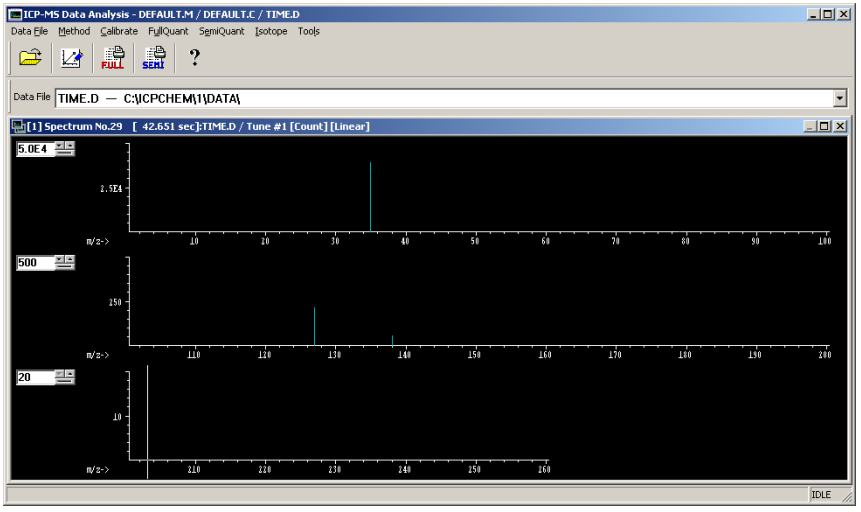


Extracted Ion Chart

Each extracted ion chart shows the element mass and name. Each chart appears in a different color.

NOTE

You can also view mass spectrum information for any time slice in a time chart. To do so, position the cursor at the time of interest in the Time Chart display and double-click the right mouse button. The data displayed at that time slice in the time chart appears in a Mass Spectrum display. To return to the Time Chart display, double-click the Control Box Menu in the Mass Spectrum display window.



Mass Spectrum for a Time Slice

Using the Display Count Box

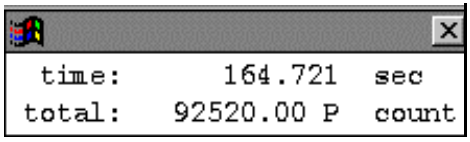
Regardless of which display type you have selected, you can view the counts recorded for each time on the Time Chart display by using a count box. This ChemStation feature enables you to quickly view the intensity at any given time slice. To display the Count Box, complete the following steps:

1 **Click the horizontal Control Box Menu at the top left corner of the time chart display window.**

The window menu appears.

2 **Click *Display Count Box* at the bottom of the menu.**

The count box appears, showing the time slice and the total counts. The letter following the value indicates the detector mode used analog (A) or pulse (P).



Count Box

3 **To see counts for a time slice on the time chart, place the cursor at one point on the Time Chart display and view the Count Box.**

NOTE The Count Box displays until you close it by double-clicking the Control Box Menu or by exiting the time chart display.

Changing the Vertical Scale of a Time Chart

Whether you are viewing a Total Ion Chart or an Extracted Ion Chart, ChemStation enables you to change the vertical scale of the display. The scale for each chart appears to the left of the chart, and the number in the scale box indicates the scale of the current display. Increase or decrease the vertical scale as required.

To change the vertical scale of the displayed time chart, complete one of the following steps:

- Click the up or down arrow to the right of the scale box.

  The full scale height decreases if you click the down arrow and increases if you click the up arrow.

- Click the horizontal bar to the right of the scale box. Select a number from the displayed list by clicking it.

  The full scale height decreases if you click a smaller number than the number that appears in the scale box and increases if you click a larger number than the number that appears in the scale box.

NOTE Regardless of which method you use, you must repeat the process for each chart shown in the Time Chart window.

Changing the Horizontal Scale of a Time Chart

Just as you can change the vertical scale of a Time Chart display, you can also change the horizontal scale. However, you change the horizontal scale of a Time Chart by zooming in on one part of the display. This means that you will no longer be viewing data for the total acquisition period, but only for that part of the acquisition period you selected.

To change the horizontal scale of a Time Chart display, complete the following steps:

1 **Drag across the section of the Time Chart you want to enlarge.**

The section is highlighted.

2 **Release the mouse button.**

The section you highlighted now fills the display window.

NOTE When you change the horizontal scale of one Extracted Ion Time Chart, ChemStation changes the scale of all others showing in the window.

To return the chart to the original horizontal scale, double-click the left mouse button.

Using the Overlay Feature

ChemStation provides an overlay feature that enables you to view signals for several Extracted Ion Charts in one chart. For example, if you have four Extracted Ion Charts showing in the display window, you can use the overlay feature to change the display to one chart showing a signal for each of the four masses.

NOTE You cannot view integration windows when you are in the Overlay mode. See the "Analyzing Results Using a Time Chart" section later in this chapter.

To use the Overlay feature, ensure that Extracted Ion Charts show in the display window and complete the following steps:

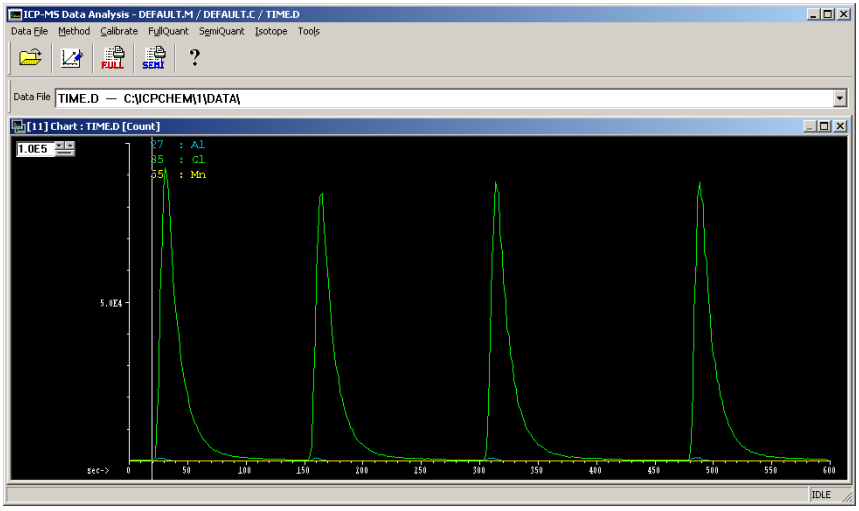
1 **Click the Control Box Menu in the display window.**

The window menu appears.

2 **Click *Overlay*.**

One display appears showing a signal for each of the masses previously shown in the Extracted Ion Charts. A label for each signal appears in the upper left corner of the display, and each signal appears in the same color as its label.

If you want to view the number of counts for each of the selected masses, display the Count Box. See "Using the Display Count Box" in this chapter for information.



Extracted Ion Chart in Overlay Display

NOTE

You can change the scale of the display to view individual signals. To adjust the vertical scale of the Overlay display, follow the instructions given in "Changing the Vertical Scale of a Time Chart" in this chapter. To adjust the horizontal scale of the display, follow the instructions given in "Changing the Horizontal Scale of a Time Chart" in this chapter.

Labeling a Time Chart

You may want to label the Total Ion Chart created by integrating results for elements in a sample. You can also label an Extracted Ion Chart. ChemStation enables you to name or add identifying information to a time chart prior to printing.

To label a time chart, complete the following steps:

1 **Ensure that a time chart displays in the ICP-MS Data Analysis window.**

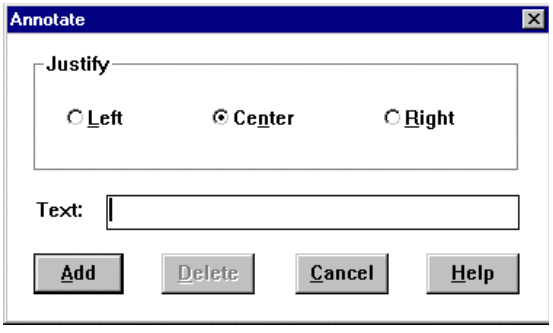
If the ICP-MS Data Analysis window displays a spectrum, you must load the appropriate data file. For more information, see "Loading a Data File" in this chapter

2 **Place the cursor exactly where you want the label to appear.**

Note both the horizontal and vertical placement of the cursor.

3 **Click both mouse buttons at the same time.**

The ***Annotate*** dialog box appears.



Annotate Dialog Box

4 **Click the *Left, Center,* or *Right* Justify button.**

The label will appear to the left, center, or right of the location you specified in Step2.

5 **Type a name or other identifying information in the text box.**

6 **Click *Add*.**

ChemStation returns to the time chart, and the information you typed appears at the location of the cursor.

NOTE

You can change and delete the text by clicking exactly on top of the previously labeled point, rewriting the text, then clicking ***Update*** in the Annotate dialog box.

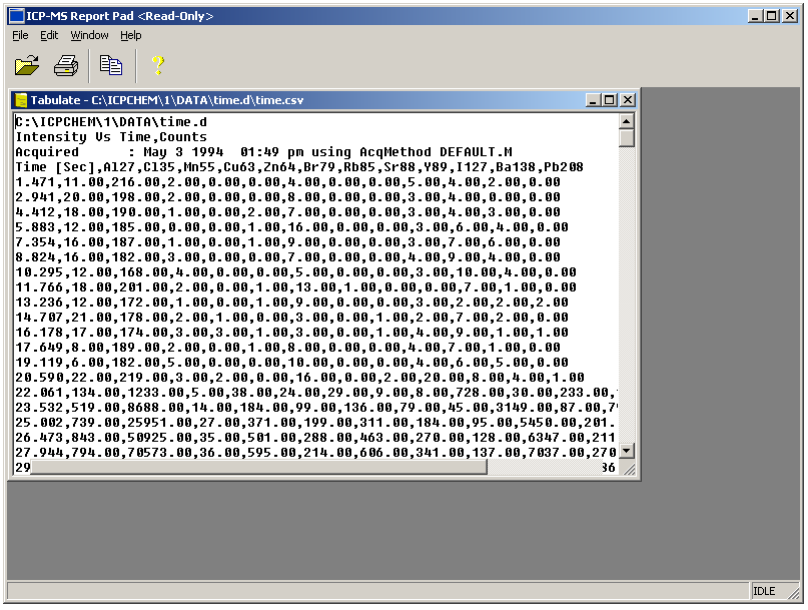
NOTE Exiting the Data Analysis window or loading the another file erases the annotation.

Display of Data at Points of Time

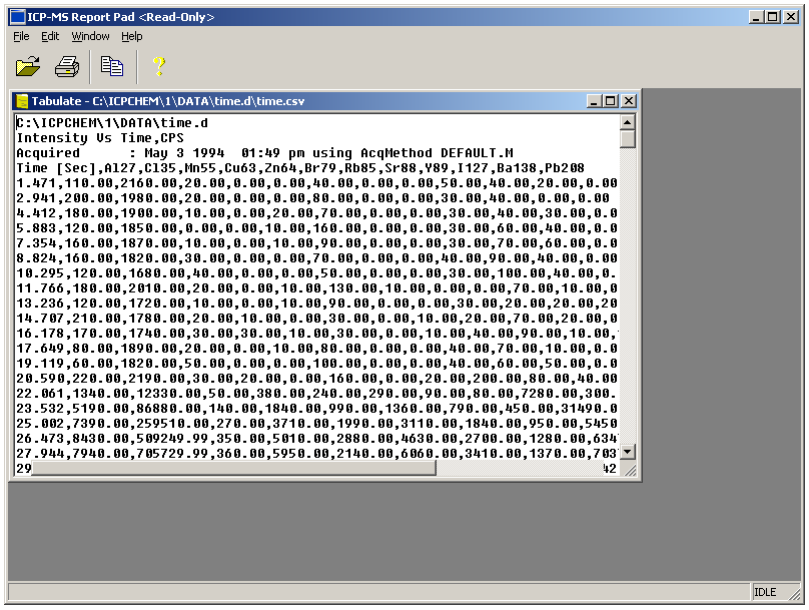
During data analysis, data acquired at specific points of time can be displayed. Follow the procedure described below.

1 Choose *Tabulate Chart Raw Data to CSV* or *Tabulate Chart CPS Data to CSV* in the *Data File* menu.

- ***Tabulate Chart Raw Data to CSV***: Displays the time-resolved analysis raw data (counts).
- ***Tabulate Chart CPS Data to CSV***: Displays the time-resolved analysis CPS data.



Raw Data



CPS Data

Exporting Data

You can save the data file currently displayed in the window in AIA format or as LC/MSD raw data.

1 **Select *Export AIA format for Agilent LC/GC* or *Export Agilent LC/MSD raw data* from the *Data File* menu.**

- Export AIA format for Agilent LC/GC:
  The data file is saved to the data file folder in AIA format. This file is saved to the data file folder. Use the Agilent LC/GC ChemStation to import this file for display or analysis.
- Export Agilent LC/MSD raw data:
  The data file is saved to the data file folder as LC/MSD raw data. This file is saved to the data file folder. Use the Agilent LC/MSD ChemStation to load, display, or analyze this file.

Printing a Time Chart

You can print a time chart display at any time during the analytical process. For example, if you have changed the horizontal scale to show only the acquisition period from 50 to 100 seconds, you can print the resulting time chart display. If you have changed the display type, you can print the time chart display showing the new type.

The following header information is printed on the time chart.

- File name
- Operator name (ACQ, DA)*

  * ACQ and DA are differentiated only when the access control pack is used.
- Date/time of measurement
- Measurement mode
- Sample type
- Sample name
- Comment
- Pre-dilution factor
- Automatic dilution factor
- Overall dilution factor
- Method
- ISTD concentration

To print the displayed time chart, select ***Data File >> Print***.

Analyzing Results Using a Time Chart

ChemStation enables you to define integration parameters for the peaks in a sample and to integrate them to obtain concentration values. You can use these two features to examine the data acquired with a method for Time Resolved Analysis or Time Program acquisition.

The following sections explain how to select integration parameters and integrate peaks in a sample.

Defining Integration Windows

Before you can integrate the peaks in a sample, you must define an integration window. Integrating peaks in a plot gives the intensity of that peak and therefore an indication of the concentration of that component.

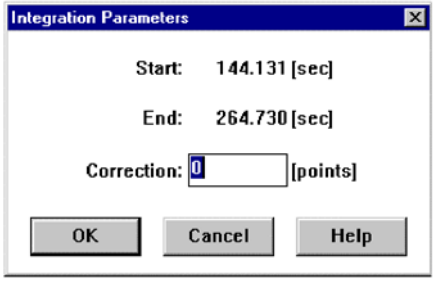
Ensure that you have extracted ion charts showing in the display window. To set the integration window, complete the following steps:

1 **Drag across a section of the Extracted Ion Chart using the right mouse button.**

The section is highlighted.

2 **Release the mouse button.**

The ***Integration Parameters*** dialog box appears.



Integration Parameters Dialog Box 1

The start and end times shown in the dialog box are the beginning and end points of the section you highlighted in Step 1.

3 **To select a correction, enter a value in points in the Correction box.**

To compensate for a sloping baseline, you can adjust the integration window by selecting different points. The points relate to the number of time slices either side of the window that the baseline is measured. The mean point between the sides of the original window and the outer window is fixed as the baseline correction. For more information, please refer to Online Help.

NOTE The integration window is set for the selected mass, though you can set the window for all masses. To do so, ensure that a Total Ion Chart shows in the display window and complete Steps 1 through 4. You can also save the selected integration window by saving the method. ChemStation will apply the selected integration window to all samples in the method.

4 **To set the integration parameters and the correction, click *OK*.**

ChemStation returns to the time chart. Vertical bars now indicate the parameters you set for the selected mass**.**

5 **To set the parameters for each mass you want to integrate, repeat Step 1 through Step 4.**

You can set different integration parameters for each mass.

NOTE Before setting your own integration parameters, ChemStation enables you to view the default parameters using the integration window from the time chart display. To use the integration window, click the Control Box Menu in the display window and then click ***Display Integration Window***. You can use this feature only if you are viewing Extracted Ion Charts.

Integrating Acquisition Parameters

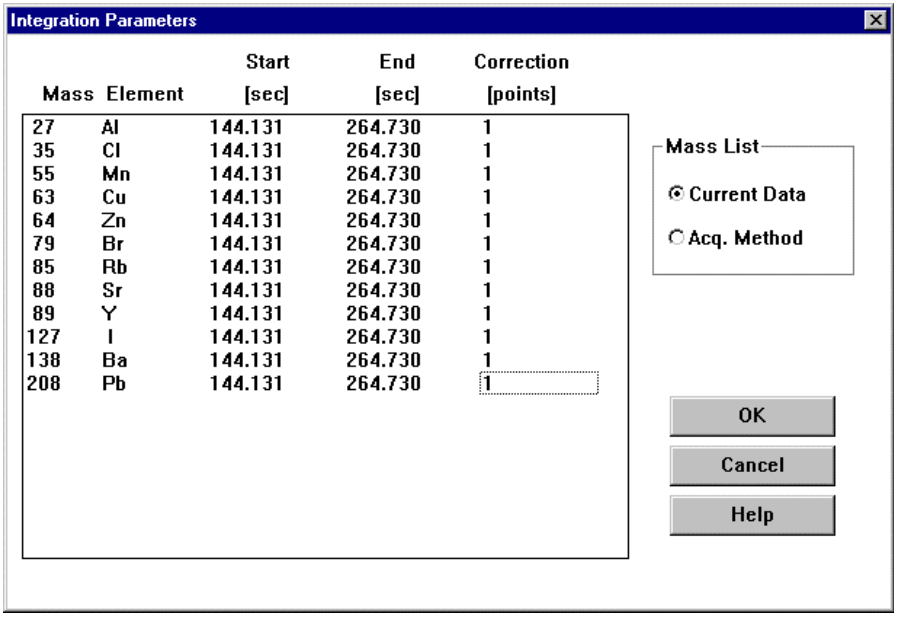
When you have set the integration parameters you want, you can display and edit those parameters for all masses. To do so, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

The ICP-MS Data Analysis window appears.

2 **Select *FullQuant>>Integration Parameters*.**

The ***Integration Parameters*** dialog box appears, showing the parameters and correction set for each mass listed.



Integration Parameters Dialog Box 2

3 Click the *Current Data* or *Acq. Method*.

Current Data shows in the dialog box the masses acquired in the current file. ***Acq. Method*** shows the masses set by the current acquisition parameters. Select Acq. Method when setting up Data Analysis integration parameters prior to data acquisition via running a method or a sequence.

4 To save the integration parameters listed in the dialog box, click *OK*.

NOTE After you set the integration parameters, you must save the method. To do so, select ***Methods>>Save*** from the ICP-MS Top window menu.

Integrating Results

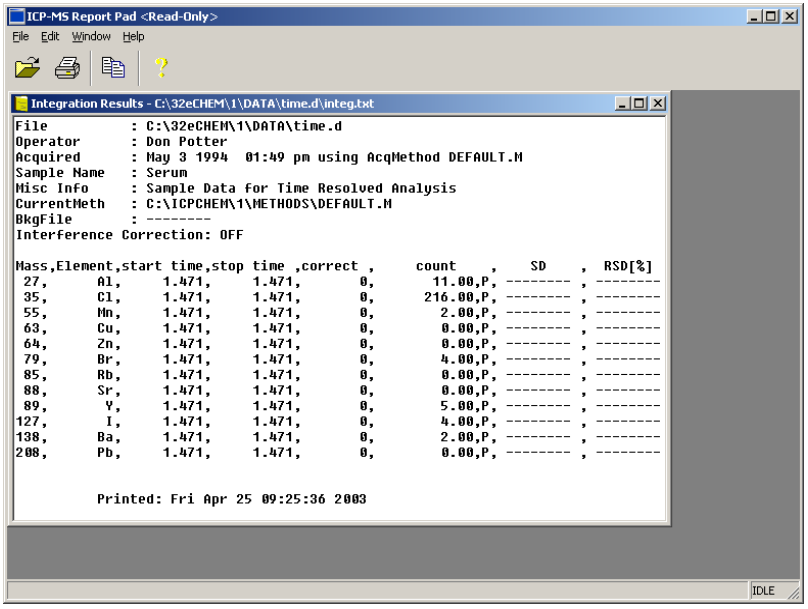
Integrating the total ion or extracted ion chart gives total area counts for all the integration windows selected. To integrate results for elements in a sample, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

The ICP-MS Data Analysis window appears.

2 **Select *FullQuant>>Integration Results*.**

The Report Pad - INTEG.TXT file appears, showing the results for all elements for a selected part of the acquisition period.



Integration Results File

When the method or sequence runs and the Quantitation Report is selected in the Data Analysis, the quantitative results will be automatically reported.

For more information about creating the calibration curve and the Quantitation Report, see chapter 12, "Performing a Quantiative Analysis".

11

Creating Custom Reports/Database

Creating Custom Reports/Database

Custom Reports/Database allows you to:

- Create a report with a customized layout.
- Create a database for recording the statistical and graphical results of multiple analyses.

You design the Custom reports and Custom databases by creating templates. After the template for the report or database is set up and linked to a method, you can generate a report and update a database either manually in Data Analysis or automatically each time the method runs.

For information about loading a method and editing method parameters, see Chapter 5, "Creating a Method". If the appropriate report types are not selected, you will not be able to generate a custom report when the method runs.

Creating a Custom Report

You can use ChemStation's Custom Report feature to create reports that includes only the information needed. Create a report template for each method you use. This section explains how to create a Custom Report and how to save the report as a template.

If you are creating a quantitative report template, you must check the method to ensure that the calibration curves are created for that method. For information about creating calibration curves, see Chapter 12, "Performing a Quantitative Analysis".

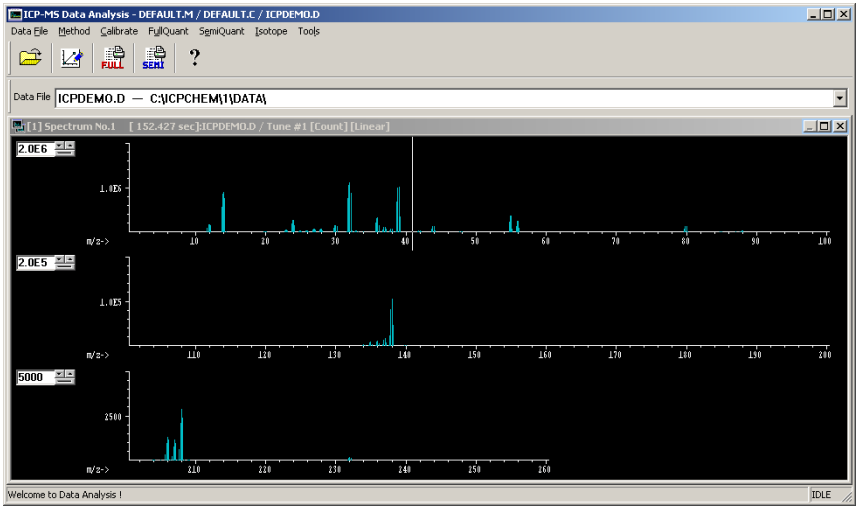
NOTE The procedures in this section assume that you are creating a quantitative custom report. If you are creating a semiquantitative custom report, select the appropriate item from the ***SemiQuant*** menu in Data Analysis.

Creating a Custom Report Template

To create a template, complete the following steps:

1 **Select *Data Analysis>>Main Panel*.**

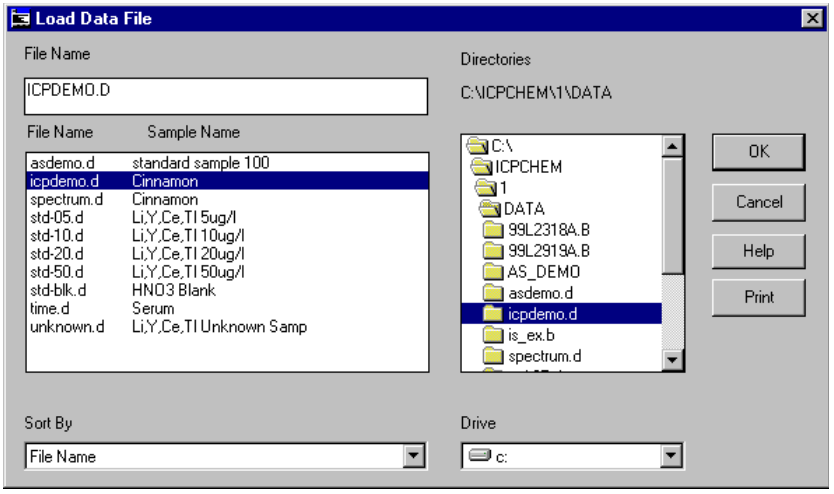
The ***Data Analysis*** window appears.



ICP-MS Data Analysis Window

2 Select *Data File>>Load.*

The ***Load Data File*** dialog box appears. It allows the user to select the data file to be loaded into data analysis for processing.



Load Data File Dialog box

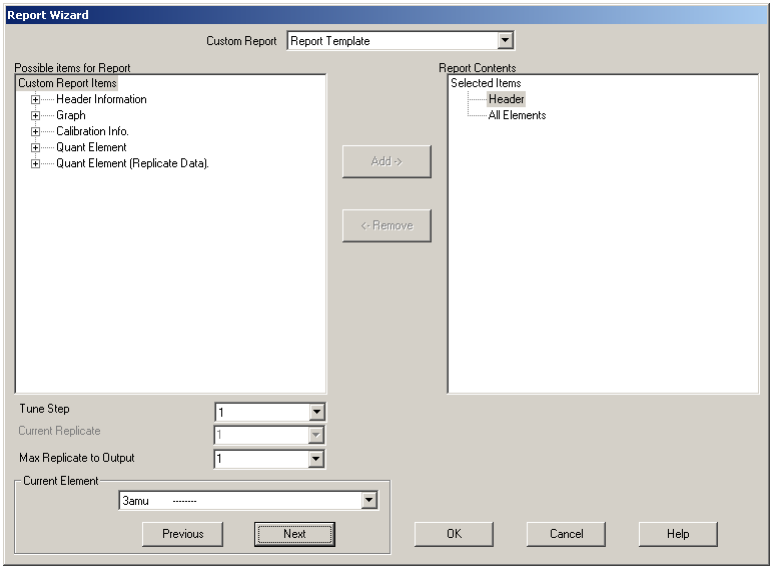
NOTE Recently opened data files are also selected from the ***Data File*** list box in the ***ICP-MS Data Analysis*** window.

3 Select a file using one of the following methods:

- Click a file name in the displayed list and click *OK*.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK.***

4 Select *FullQuant>>Layout Custom Report.*

If the Custom report template is not linked to the method, the Report Wizard dialog box appears. If the Custom report template is already linked to the method, the ***Report Wizard*** dialog box does not appear. In this case, select ***Templates>>New*** to show the Report Wizard.



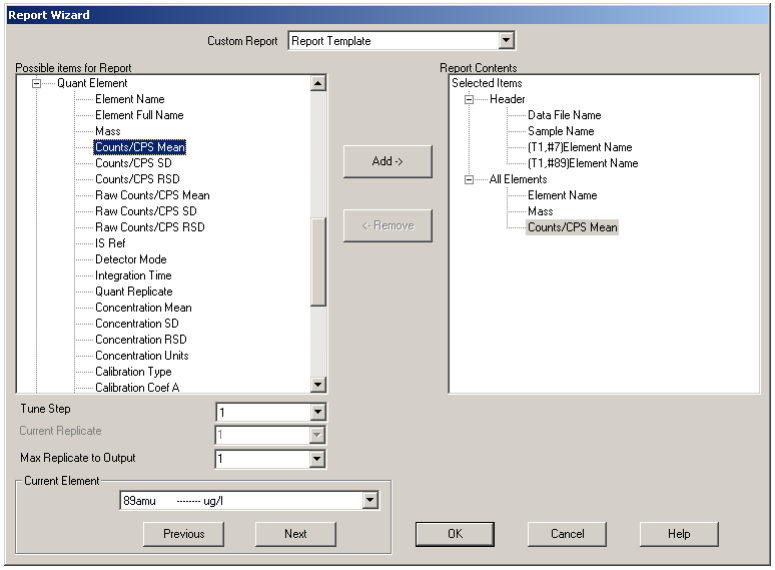
Report Wizard

Confirm the ***Report Template*** is set in the ***Custom Report*** box.

5 Select the items in the left box and double click it or click *Add-> button*.

To add ***Raw Counts/CPS*** of ***Quant Element (Replicate Data)***, click on ***Raw Counts/CPS*** and select a numerical value in the each list box for ***Tune Step***, ***Current Replicate***, and ***Max Replicate Output***. Select ***Current Element*** and then click on ***Add***. A number can be entered for ***Current Replicate*** and ***Max Replicate Output*** instead of selecting from the list boxes.

The selected items appear in the right box.



- ***Header*** tree in the right box

  After clicking ***Header*** in the right box, you can select the items under ***Header Information, Graph, Calibration Info., Quant Element*** and ***Quant Element (Replicate Data)*** tree in the left box, and add to the ***Header*** tree in the right box.

  When you select the items under the ***Quant Element*** tree in the left box, the selected items are displayed with each mass number like (#89) that is currently selected in the ***Current Element*** box.

  When ***Raw Counts/CPS*** is selected under the ***Quant Element (Replicate Data)*** tree in ***Possible Items for Report***, "(Tt, #n) Raw Counts/CPS[r]" is shown (t: number of tune steps; n: mass number; r: number of current replicate-data sets). The total number of data sets displayed depends on the setting of ***Max Replicate Output***.

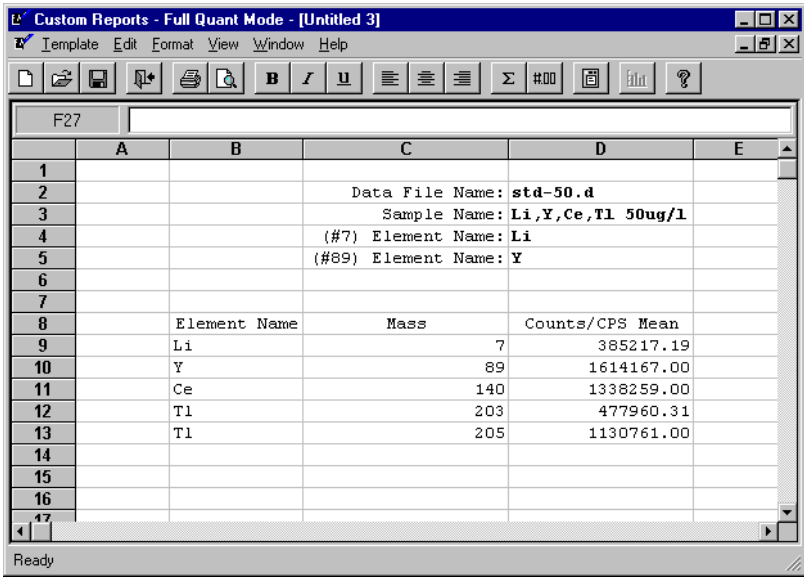
- ***All Elements*** tree in the right box

  After clicking ***All Elements*** in the right box, you can select the items under the ***Quant Element*** and ***Quant Element (Replicate Data)*** tree in the left box and add to the ***All Elements*** tree in the right box. It enables you to layout data for all elements by selecting one item.

  When ***Raw Counts/CPS*** is selected under the ***Quant Element (Replicate Data)*** tree in ***Possible Items for Report***, "Raw Counts/CPS" appears under the All Elements tree. When a report is created, the data is displayed according to the setting of ***Max Replicate Output***.

6 When you have selected all the necessary items from the left box, Click *OK*.

The Custom report template appears.



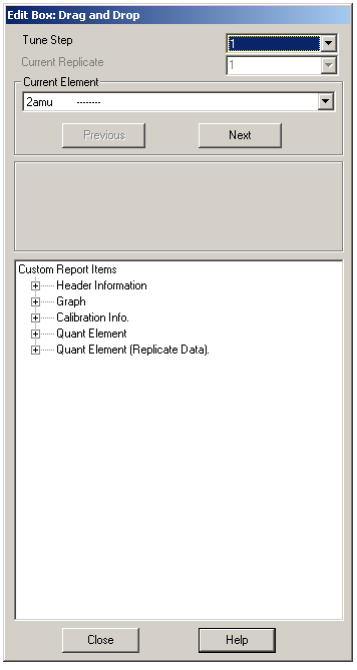
Custom Report Template - Full Quant Mode

When ***Template>>Load new data file*** is selected, ChemStation updates these links to show the new information.

NOTE

The ***Script*** box in the Font dialog box (***Format>>Font***), is not applicable on the Custom report.

When ***View>>Edit Box*** (or click ***Displays Edit box*** button on the tool bar) is selected, the ***Edit Box*** dialog box appears. You can drag and drop the desired items from the Edit box to the desired position on the Custom Report Template.
To add ***Raw Counts/CPS*** of ***Quant Element (Replicate Data)***, click on ***Raw Counts/CPS*** and select a numerical value in the each list box for ***Tune Step*** and ***Current Replicate*** (a number for ***Current Replicate*** can be entered directly). Select ***Current Element*** and then drag and drop ***Raw Counts/CPS*** onto the template. Of the raw data on the element selected in ***Current Element***, only the replicate data set in ***Current Replicate*** will be displayed.



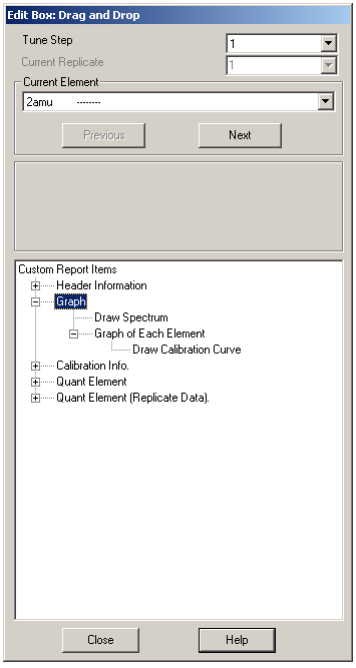
Edit Box Dialog Box

Adding a Chart to a Custom Report

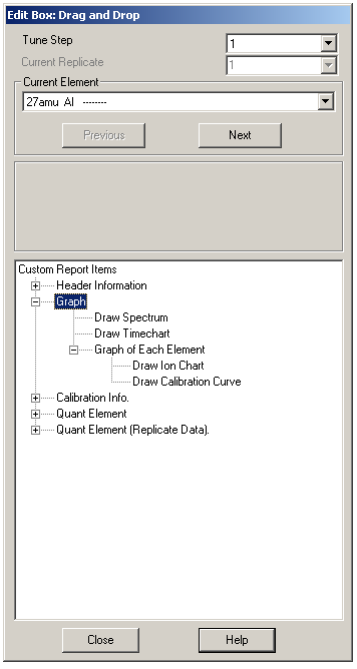
You can add a chart of the current data file to a custom report. To add a spectrum chart, Time chart, Ion Chart, or Calibration curve, complete the following steps:

1 Select *View>>Edit Box.*

The ***Edit Box*** dialog box appears. The displayed information in the Edit box depends on the data type (Spectrum or Time chart).



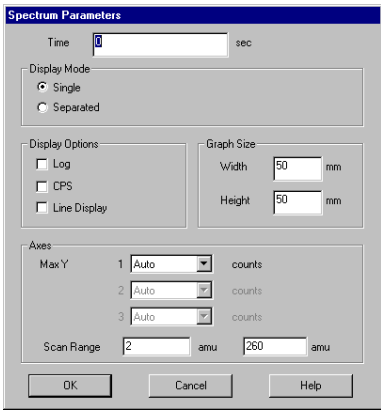
Spectrum
Edit Box Dialog Box



Time chart

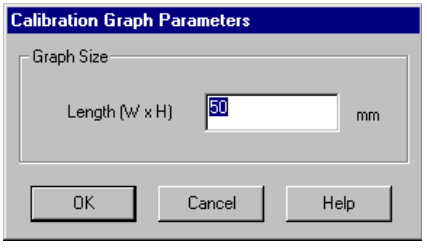
2 Select *Draw Spectrum* (or *Draw Timechart*) under the *Graph* tree, and drag and drop it to the desired position on the custom report. Or select *Draw Ion Chart* (or *Draw Calibration Curve*) under the *Graph* tree, and drag and drop it to the desired position on the custom report.

The following dialog box appears.

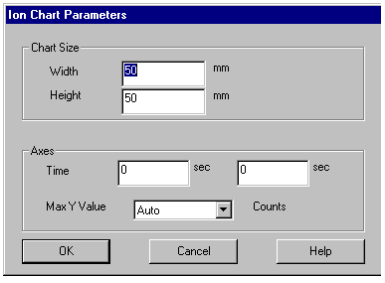


Spectrum Parameters Dialog Box

Time Plot Parameters Dialog Box



Calibration Graph Parameters Dialog Box



Ion Chart Parameters Dialog Box

3 Select the desired options.

This dialog box allows you to manipulate how the chart will look.

(The ***Time*** box in the Spectrum Parameters dialog box is for Time Resolved Analysis data).

4 Click *OK*.

The chart is drawn. To move the chart, press the Ctrl key, click the chart, and click and drag the chart with the mouse. To size the chart, press the Ctrl key, and click one of the dots and drag it to the desired size.

Preview a report

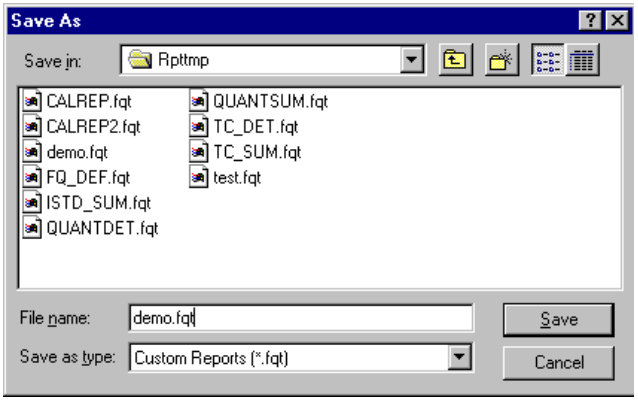
Before you set the file as a template for custom reports, you can view the report and make any formatting changes you want. To view and format the template, select ***Template***>>***Print Preview***.

Saving the Template

To set the report file as the custom report template, complete the following steps:

1 Select *Template>>Save As*.

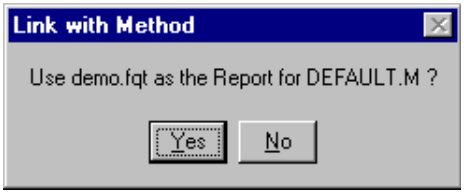
The ***Save As*** dialog box appears.



Save As Dialog Box

2 Type a name for the file and click *Save*.

The ***Link with Method*** dialog box appears. Click ***Yes*** to link the template to the current method file.



Link with Method Dialog Box

3 Select *Template>>Exit*.

ChemStation returns to the Data Analysis window.

NOTE After you link the file as the custom report template to the method, you must also save the method. To do so, select ***Methods>>Save*** from the Top menu.

Generating a Custom Report

You can generate a custom report using the custom report template you created.

Once you set a custom report template for a method, ChemStation automatically generates a custom report if the proper report parameters are set in the method. For information about setting report parameters, see Chapter 5, "Creating a Method".

ChemStation also enables you to generate a custom report manually. To generate a custom report manually, Select ***FullQuant >> Print Custom Report***.

If you want to generate SemiQuant custom report, select ***SemiQuant >> Print Custom Report***.

Creating a Custom Report Database

You create a custom report database in much the same way as you create a custom report. However, the database has a more fixed format and can accommodate data from a series of analyses. ChemStation adds new data to the end of the database. To create a custom report database you need to define the database and format the database. This section explains how to create a custom report database and how to save the database as a template.

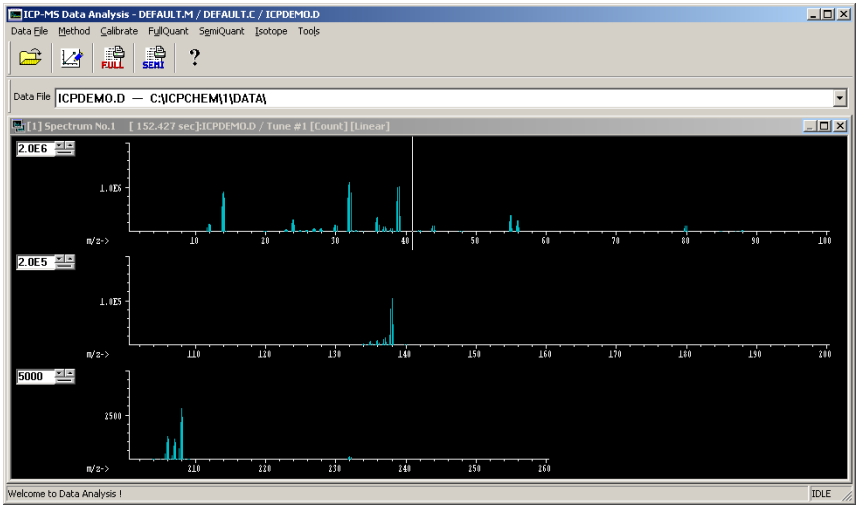
The procedures in this section assume that you are creating a quantitative custom database template. If you are creating a semiquantitative custom database template, select the appropriate item from the SemiQuant menu in Data.

Creating a Template

To create a template, complete the following steps:

1 **Select *Data Analysis>>Main Panel.***

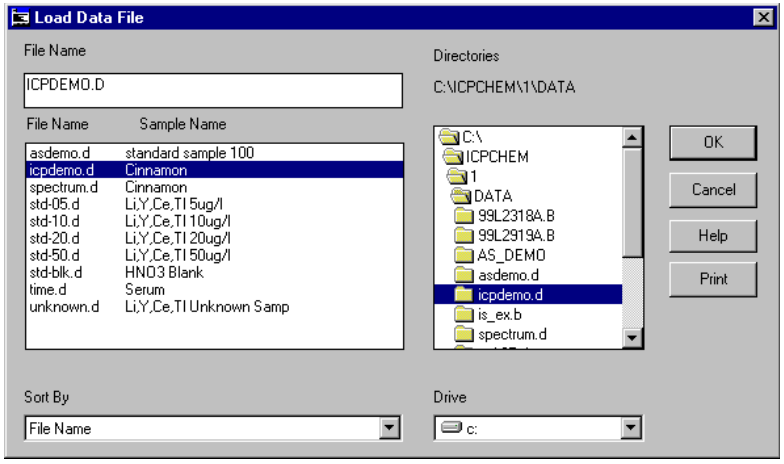
The ***Data Analysis*** window appears.



ICP-MS Data Analysis Window

2 Select *Data File>>Load.*

The ***Load Data File*** dialog box appears. It allows the user to select the data file to be loaded into data analysis for processing.



Load Data File Dialog box

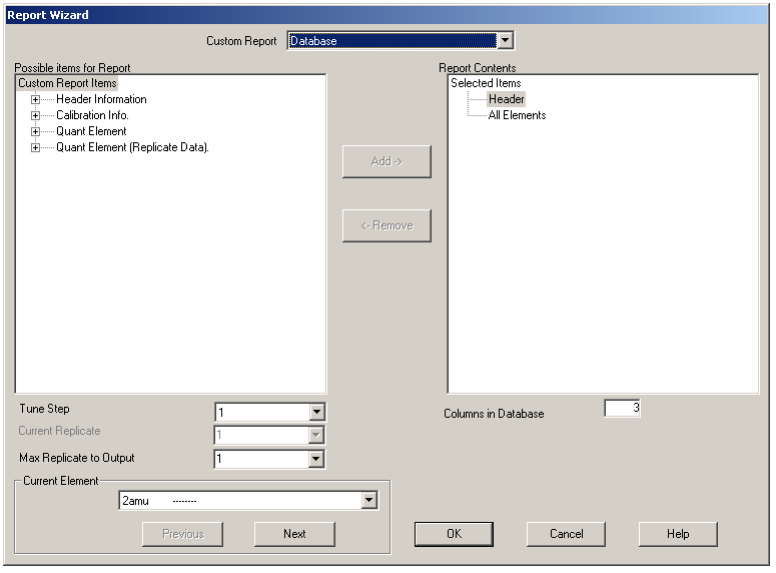
NOTE Recently opened data files are also selected from the ***Data File*** list box in the ***ICP-MS Data Analysis*** window.

3 Select a file using one of the following methods:

- Click a file name in the displayed list and click ***OK.***
- Double-click a file name in the displayed list.
- Type the file name and click ***OK.***

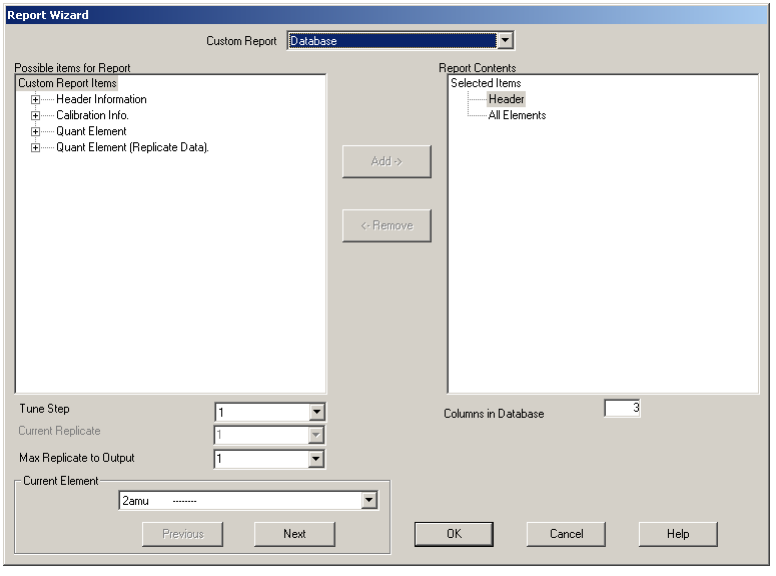
4 Select *FullQuant>>Layout Custom Report.*

If the Custom report template is not linked to the method, the Report Wizard dialog box appears. If the Custom report template is already linked to the method, the ***Report Wizard*** dialog box does not appear. In this case, select ***Templates>>New*** to show the Report Wizard.



Report Wizard

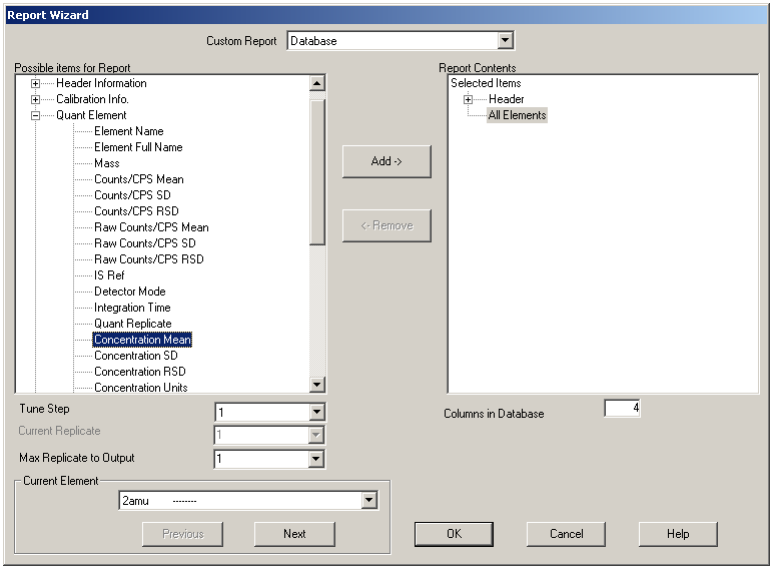
5 Select the *Database* in the Custom Report box.



Report Wizard (Database is selected)

6 Select the items in the left box, and double click it or click *Add->* button. The selected items appear in the right box.

To add ***Raw Counts/CPS*** of ***Quant Element (Replicate Data)***, click on ***Raw Counts/CPS*** and select a numerical value in the each list box for ***Tune Step***, ***Current Replicate***, and ***Max Replicate Output***. Select ***Current Element*** and then click on ***Add***. A number can be entered for ***Current Replicate*** and ***Max Replicate Output*** instead of selecting from the list boxes.



- ***Header*** tree in the right box

  After clicking ***Header*** in the right box, you can select the items under ***Header Information, Graph, Calibration Info., Quant Element*** and ***Quant Element (Replicate Data)*** tree in the left box, and add to the ***Header*** tree in the right box.

  When you select the items under the ***Quant Element*** tree in the left box, the selected items are displayed with each mass number like (#89) that is currently selected in the ***Current Element*** box.

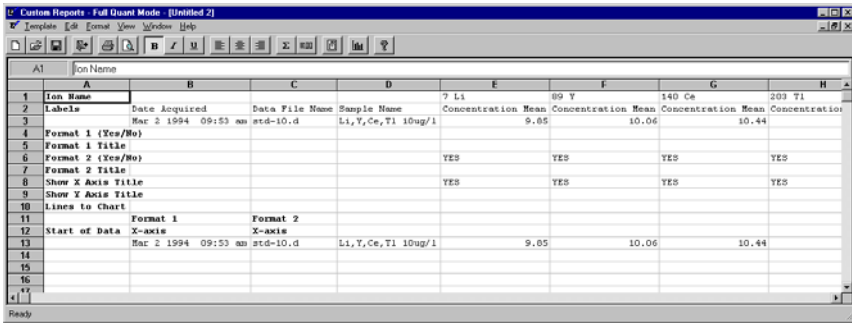
  When ***Raw Counts/CPS*** is selected under the ***Quant Element (Replicate Data)*** tree in ***Possible Items for Report***, "(Tt, #n) Raw Counts/CPS[r]" is shown (t: number of tune steps; n: mass number; r: number of current replicate-data sets). The total number of data sets displayed depends on the setting of ***Max Replicate Output***.

- ***All Elements*** tree in the right box

  After clicking ***All Elements*** in the right box, you can select the items under the ***Quant Element*** and ***Quant Element (Replicate Data)*** tree in the left box and add to the ***All Elements*** tree in the right box. It enables you to layout data for all elements by selecting one item.

7 When you have selected all the necessary items from the left box, Click *OK*.

The Custom report database template appears.



	A	B	C	D	E	F	G	H
1	Ion Name				7 Li	89 Y	140 Ce	203 Tl
2	Labels	Date Acquired	Data File Name	Sample Name	Concentration Mean	Concentration Mean	Concentration Mean	Concentration
3		Mar 2 1994 09:53 am	std-10.d	Li,Y,Ce,Tl 10ug/l	9.85	10.06	10.44	
4	Format 1 (Yes/No)							
5	Format 1 Title							
6	Format 2 (Yes/No)				YES	YES	YES	YES
7	Format 2 Title							
8	Show X Axis Title				YES	YES	YES	YES
9	Show Y Axis Title							
10	Lines to Chart							
11		Format 1	Format 2					
12	Start of Data	X-axis	X-axis					
13		Mar 2 1994 09:53 am	std-10.d	Li,Y,Ce,Tl 10ug/l	9.85	10.06	10.44	
14								
15								
16								

Custom Report Database Template ñ Full Quant Mode

Line 1: Ion name

Line 2: Label

Line 3: The current data

Line 4 ~12: The parameters set in the ***Global Chart options*** dialog box and the ***Individual Chart options*** dialog box are displayed.

NOTE Do not edit the information between the Line 4 and Line 12.

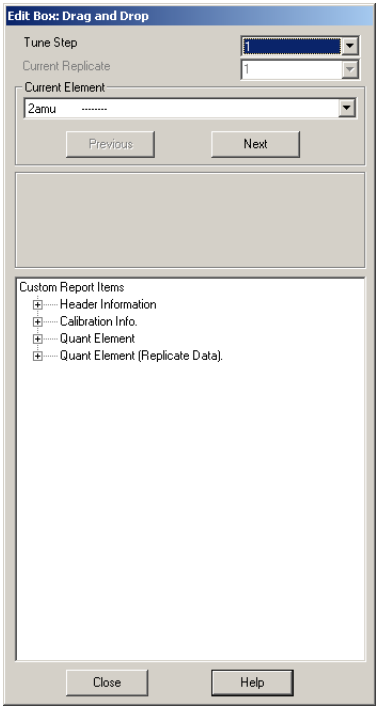
When ***Template>>Load new data file*** is selected, ChemStation adds the new data to the bottom line of the database.

NOTE The ***Script*** box in the Font dialog box (***Format>>Font***), is not applicable on the Custom report.

To add ***Raw Counts/CPS*** of ***Quant Element (Replicate Data)***, click on ***Raw Counts/CPS*** and select a numerical value in the each list box for ***Tune Step*** and

Current Replicate (a number for ***Current Replicate*** can be entered directly). Select ***Current Element*** and then drag and drop ***Raw Counts/CPS*** onto the template. Of the raw data on the element selected in ***Current Element***, only the replicate data set in ***Current Replicate*** will be displayed.

When ***View>>Edit Box*** (or click ***Displays Edit box*** button on the tool bar) is selected, the ***Edit Box*** dialog box appears. You can drag and drop the desired items from the Edit box to the raw 3 on the Custom Report Template.



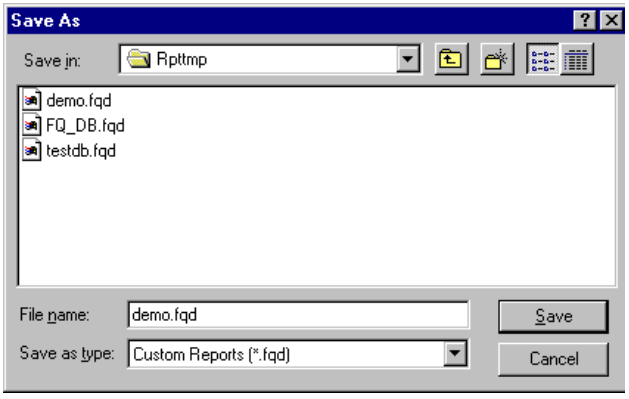
Edit Box Dialog Box

Saving the Database as a Template

Before ChemStation can automatically update the database you created, you must first set the database as a template. To do so, complete the following steps:

1 **Select *Template>>Save As*.**

The ***Save As*** dialog box appears.



Save As Dialog Box

2 **Type a name for the file and click *Save*.**

The ***Link with the Method*** dialog box appears. Click ***Yes*** to link the template to the current method file.

Link with Method Dialog Box

3 **Select *Template>>Exit*.**

ChemStation returns to the Data Analysis window.

NOTE After you link the file as the custom database template to the method, you must also save the method. To do so, select ***Methods>>Save*** from the Top menu.

Updating a Custom Report Database

ChemStation enables you to update the custom report database once the database template is created. Once you link a custom database template to a method and set the method to update the database, your database can be updated automatically when running a method or a sequence. For information about setting report parameters, see Chapter 5, "Creating a Method". ChemStation also allows you to update a database manually for any data file.

To generate a results file and update the database manually, complete the following steps:

1 **Load a data file**

2 **Select *FullQuant>>Update DataBase*.**

ChemStation updates the database. You can view the database now that you have updated it. For information, see the following section, "Viewing the Database".

3 **Repeat Steps 1 and 2 for every file you want to include in the custom report database.**

As you update the database, the database should grow longitudinally. For each data file that you update into the database, there should be an additional row of data containing information specific to that data file appended to the bottom of the database.

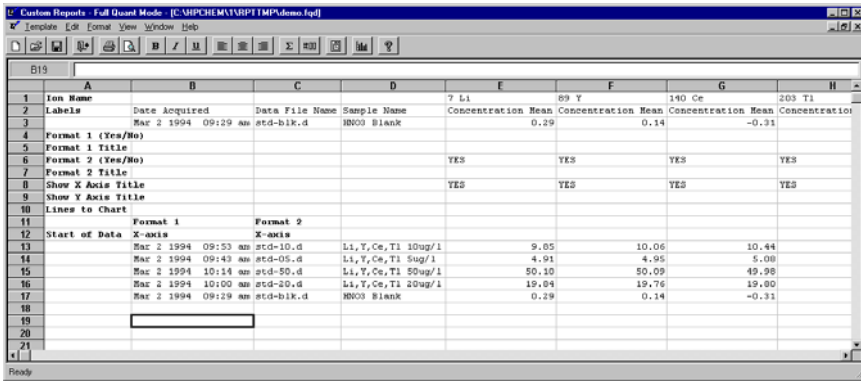
Viewing the Database

ChemStation enables you to open a database after you have updated it. You can then use features available in the database menu to create graphs and charts from the database.

To open and view a database, complete the following steps from the Data Analysis window:

1 **Select *FullQuant>>Layout Custom Report.***

The database appears.



	A	B	C	D	E	F	G	H
1	Ion Name				7 Li	89 Y	140 Ce	203 Tl
2	Labels	Date Acquired	Data File Name	Sample Name	Concentration Mean	Concentration Mean	Concentration Mean	Concentratio
3		Mar 2 1994 09:29 am	std-blk.d	HNO3 Blank	0.29	0.14	-0.31	
4	Format 1 (Yes/No)							
5	Format 1 Title							
6	Format 2 (Yes/No)				YES	YES	YES	YES
7	Format 2 Title							
8	Show X Axis Title				YES	YES	YES	YES
9	Show Y Axis Title							
10	Lines to Chart							
11		Format 1	Format 2					
12	Start of Data	X-axis	X-axis					
13		Mar 2 1994 09:53 am	std-10.d	Li,Y,Ce,Tl 10ug/l	9.85	10.06	10.44	
14		Mar 2 1994 09:43 am	std-05.d	Li,Y,Ce,Tl 5ug/l	4.91	4.95	5.08	
15		Mar 2 1994 10:14 am	std-50.d	Li,Y,Ce,Tl 50ug/l	50.10	50.09	49.98	
16		Mar 2 1994 10:00 am	std-20.d	Li,Y,Ce,Tl 20ug/l	19.84	19.76	19.80	
17		Mar 2 1994 09:29 am	std-blk.d	HNO3 Blank	0.29	0.14	-0.31	
18								
19								
20								
21								

Custom Database

Charts and graphs can be created from the saved database. For information, see the following section, "Creating Charts from a Custom Report Database".

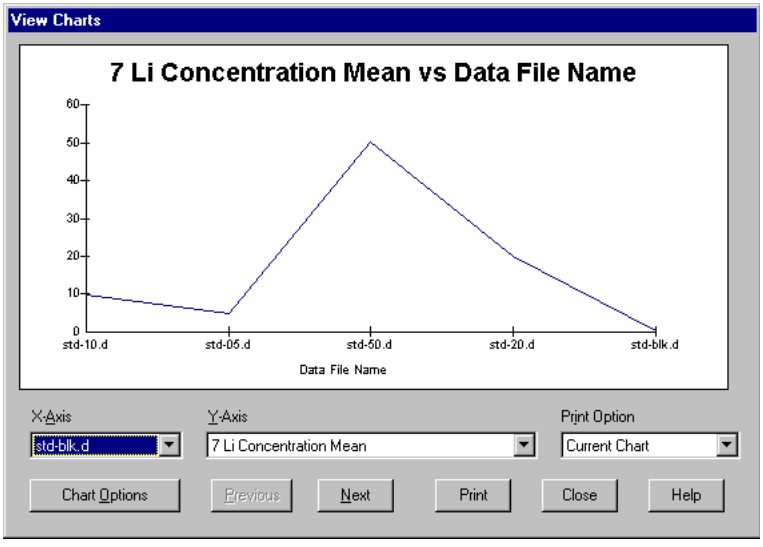
Creating Charts from a Custom Database

ChemStation enables you to create charts from data contained in the custom database. This section describes how to create a control chart.

To create a chart, complete the steps outlined in "Viewing the Database" in this chapter, then complete the following steps:

1 **Select .FQD file and select *Template>>Create Database Chart.***

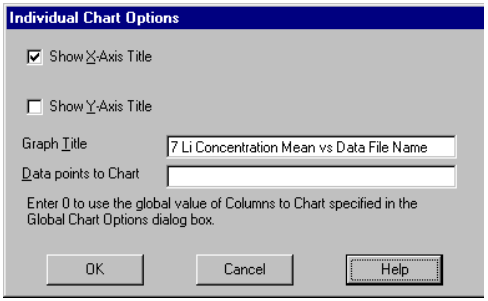
The Chart Configuration dialog box appears.



View Charts Dialog Box

2 **Click the chart in the View Charts dialog box.**

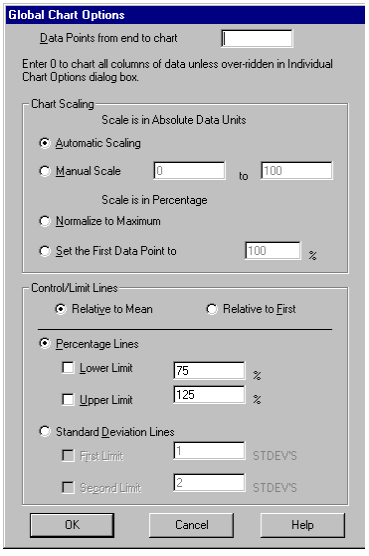
The Individual Chart Options dialog box appears. The ***Individual Chart Options*** dialog box allows you to manipulate how the individual chart will look.



Individual Chart Options Dialog Box

3 Click *Chart Options* in the View Chart dialog box.

The ***Global Chart Options*** dialog box appears. The ***Global Chart Options*** dialog box allows you to manipulate how the chart will look. You can select the scaling for the chart, select control lines, and select standard deviation lines for the chart.



Global Chart Options Dialog Box

4 To print the graph you created, click *Print*.

The graph is sent to the printer.

5 To exit, click *Close*.

12

Performing a Quantitative Analysis

Performing a Quantitative Analysis

Quantitative analysis enables you to determine the concentrations of specific elements in unknown samples. You run the sample solutions and then calculate concentrations for the elements of interest by comparing them to calibration plots for those elements (external calibration). To perform a quantitative analysis you must first analyze calibration standards and set up a calibration plot for each element in the calibration solution. ChemStation can then automatically calculate concentrations in the unknown samples based on the calibration plots.

Normally, internal standards are added to both calibration standards and samples to normalize instrument response. Typically 1-3 internal standard elements are used. Internal standards compensate for changes in instrument sensitivity that occur if the sample matrix is different from the calibration standard matrix (normally HNO_3), and for any drift in sensitivity.

ChemStation can also quantitate using standard addition, where several aliquots of the unknown sample are spiked with calibration standards. The calibration plot is determined in the sample matrix itself. Standard addition can lead to improved accuracy in unknown matrices, but is more time consuming.

This chapter explains how to run calibration standards, set up and use calibration plots, alter calibration plots by removing individual points, subtract blank data files from sample data files, and generate quantitative reports.

Running Calibration Standards

To perform a quantitative analysis you must first prepare and analyze calibration standards. Usually you will run a blank solution and then a series of standard solutions containing known concentrations of the analytes. Each calibration standard contains a different concentration of the analyte, and it is typical to run four calibration standards, including the blank or 0 standard. Thus the number of points on a calibration curve corresponds to the number of calibration standards you run.

Although you can run the calibration standards alternately with the unknown samples, it is advisable to run the calibration standards first. You can then create and view the calibration curves to determine if the calibration is acceptable. If you need to re-run the calibration, you can do so before you run the unknown samples.

If you are analyzing samples using a sequence, enter the calibration standards before the unknown samples in the sample log table. The sequence automatically create the calibration standards and analyze the unknown samples. You can stop the sequence after analysis of the calibration standards and check the calibration curve, and then re-start the sequence to analyze the unknown samples. For information about running a sequence, see Chapter 8, "Running a Sample Analysis".

Calibration standards need not be comprised of elements all at the same concentration; they can be made up with elements present at different concentrations within the same solution. In addition, sets of calibration standards containing different groups of elements can be run sequentially in the same sequence, and quantitative analysis performed on the samples for all elements at the same time. For example, one set of standards containing only transition elements is prepared, along with a set of standards containing only rare earth elements. The two sets are run sequentially, followed by the samples, which are quantitated for both sets of elements at the same time. This is useful when measuring large suites of elements which may be chemically unstable when combined all together in one set of solutions.

Generating a Calibration Curve

ChemStation uses calibration curves to calculate analyte concentrations in unknown samples. You set up calibration curves using the data files ChemStation generated after analyzing calibration standards.

The calibration curve created in an External Calibration method is based on calibration standards which have been prepared by spiking one or more elements of known concentrations into a clean matrix, such as acidified water. In contrast, the calibration curve created in a Standard Addition method is based on calibration standards prepared by spiking aliquots of the unknown sample with known concentrations of the elements of interest.

The descriptions provided in this section are for setting up an External Calibration curve. Any differences between setting up an External Calibration curve and a Standard Addition curve will be noted where appropriate.

This section explains how to create a calibration curve by performing the following functions: setting up calibration files, calculating calibration concentration levels and selecting calibration equations. Finally, this section explains how to view calibration curves and determine if they are acceptable.

If the calibration curves are not acceptable, you can exclude points from the curves; this might be necessary if, for example, one of the standards was found to be incorrectly prepared. You can also add points to the curve by running additional calibration standards and rechecking the calibration curves before you continue.

Setting a background file allows the user to subtract background counts from any data file. This subtracts the raw counts before calculation; the abundance for each mass acquired in the background file are subtracted from the abundance of the corresponding mass in non-background file. Background subtraction (or background correction) should not be confused with blank concentration subtraction which is performed after concentrations have been calculated and is used to subtract the reagent blank from an unknown sample. Background correction should NOT be used in a quantitative analysis using an internal standard because the internal standard element counts will be subtracted from the non-background file.

(The background file and interference correction can be set at ***Method>>Data Correction*** in the ICP-MS Data Analysis window).

Creating a New Calibration File

The following procedure will help in creating a new calibration file.

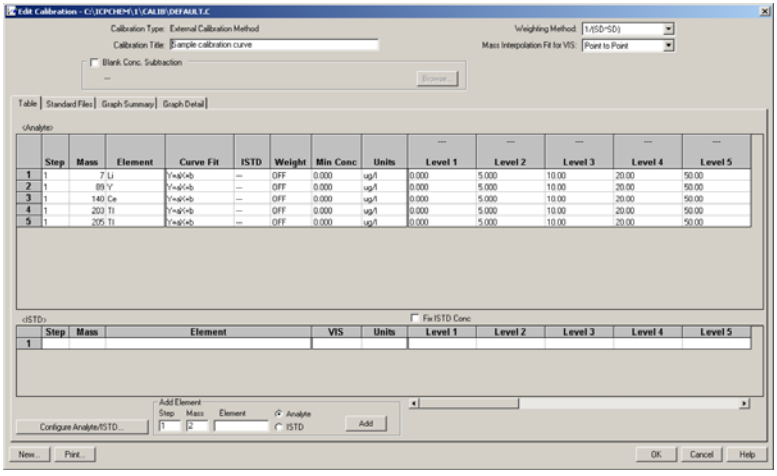
A calibration file can be created in either Online or Offline Data Analysis. The following example is based on Online Data Analysis.

1 **Select T*op >> Data Analysis >> Main Panel*.**

The ***ICP-MS Data Analysis*** window will appear.

2 **Select *Calibrate >> Edit Calibration*.**

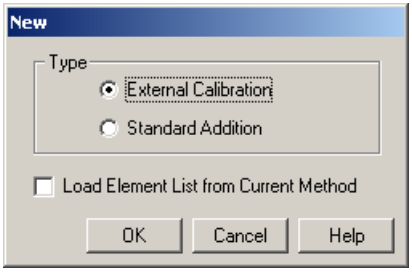
The ***Edit Calibration*** dialog box will appear.



Edit Calibration Dialog Box

3 **Click *New*.**

The ***New*** dialog box will appear.



New Dialog Box

4 **Select a calibration type in the Type area.**

External Calibration is the most common method of performing quantitative analysis.

To load an element list from the current method, select the ***Load Masses from Current Acq. Params*** check box.

5 Click *OK*.

The ***New*** dialog box will close and a new calibration file will be created.

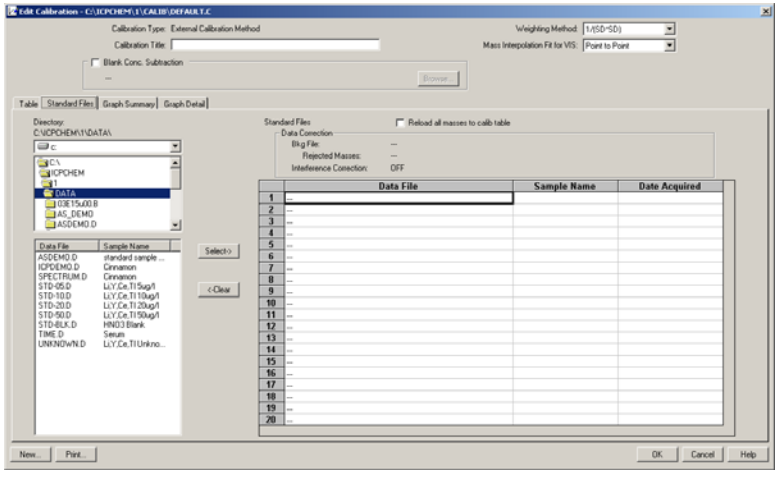
6 Enter a file name in *Calibration Title*.

Selecting a Data File

To create a new calibration file, select the data file of a sample standard.

1 Click the *Standard Files* tab.

The ***Standard Files*** tab screen will appear. Select the data file of a sample standard.



Standard Files Tab

2 Select the *Reload all masses to calib table* check box.

When a data file is chosen with this check box selected, the masses contained in the data file selected on this tab screen will be loaded to the ***Table*** tab screen.

3 In the table on the right side, click on the *Data File* field of the level to which the datafile is set.

The data file to be set must correspond to the selected level. For example, when Level 1 is selected, the data file to be set must represent the calibration solution for the first level of concentration. The first level is normally a blank solution.

NOTE Up to 20 levels can be set in a data file.

4 **Select the location of the data file using *Directory:* in the upper left section.**

Select a drive in the upper list box and choose a folder in the lower list box. A list of data files contained in the folder will be shown in the lower left section.

5 **Select a data file from the list box in the lower left section and click *Add->*.**

The selected data file will be added to the Data File field in the right-side table.

Double-clicking on a data file in the lower left section will also add that data file to the Data File field. The selection can be cancelled by double-clicking on the Data File field.

6 **Repeat Steps 3 and 4 to select more data files.**

NOTE To save a data file together with a method, it is necessary to save the method. For data analyses, close the ***Data Analysis*** window and select ***Top*** window **>> *Method* >> *Save***. For offline data analysis, select ***Offline Data Analysis*** window **>> *Method* >> *Save***.

Setting Concentration Levels

After selecting data files corresponding to standard samples, set the concentration information for each sample standard.

1 Select the *Table* tab on the *Edit Calibration* dialog box.

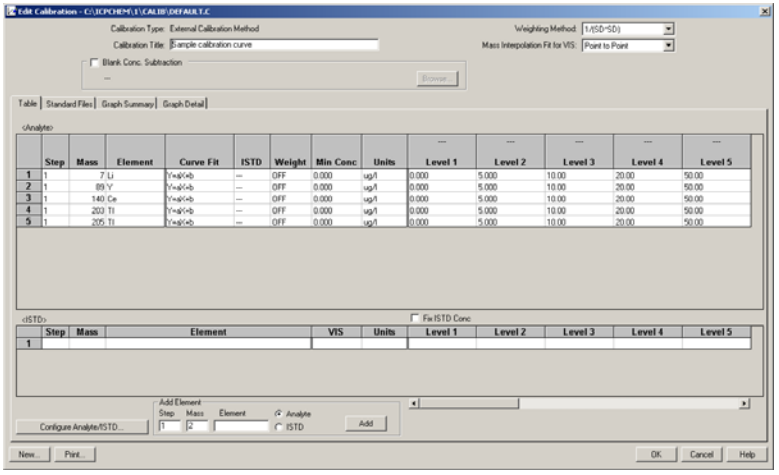


Table Tab

The Analyte table shows the tune step, mass, and element symbol of each element for which a calibration file has been created. This table allows the setting of the curve fit, ISTD, weight, minimum concentration, units, and concentration values of levels. Each level is indicated with the name of the data file set on the ***Standard Files*** tab screen.

The ISTD table shows the tune step, mass, and element symbol of the element subject to ISTD correction and VIS correction. This table allows the setting of the VIS, units, and concentration values of levels.

When you click the right mouse button with the mouse pointer positioned on a table, a popup menu appears to let you rearrange the table order by step, mass, or element; copy or paste a field; delete an element; set a concentration factor; or display the ***Graph (Detail)*** tab screen.

2 Click and select the field of the desired level to input a concentration value.

Click on a field and drag the cursor to select two or more continuous fields in a vertical direction. Click on a column title to select all the fields in that column.

The scroll bar in the lower right section enables scrolling of the level display.

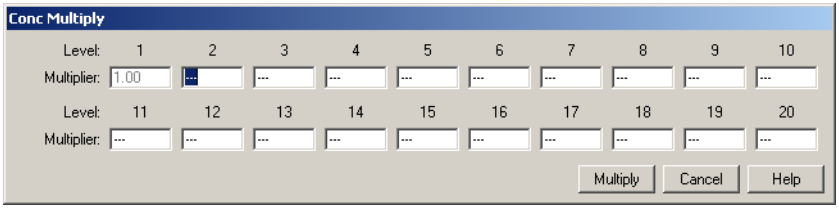
3 Enter a concentration value.

To set a standard addition calibration, the concentration of the background (bkg) file must be set to bkg or -1 for all elements. The concentration of an unspiked standard sample should be set to 0 for all elements.

4 If the concentration value entered for a level is used to calculate the concentration values for other levels using multipliers, set multipliers using the following method.

1) Select the field of the level to which a concentration value has been entered and click the right mouse button and select ***Conc Multiply***.

The ***Conc Multiply*** dialog box will appear.



Conc Multiply Dialog Box

The multiplier for the level input with a concentration value will show 1.00, and the indication will be grayed out (the value cannot be changed).

When a multiplier is entered in the text box for each level, the value calculated by multiplying the concentration value by the entered multiplier will be set for that level.

2) Enter a multiplier in the text box for each level and click ***Multiply***.

The ***Edit Calibration*** dialog box will appear again and indicate the calculated concentration values for the levels set with multipliers.

5 Click the *Curve Fit* field and select a calibration equation to be used for drawing the calibration curve.

For External Calibration:

- Y=aX
- Y=aX+[blank]
- Y=aX+b . Normally, select this equation.
- Y=aXX+bX
- Y=aXX+bX+c
- logY=a(logX)+b
- Excluded

For standard addition:

- Y=aX+[blank]+bkg
- Y=aX+b+bkg. Normally, select this equation.
- Excluded

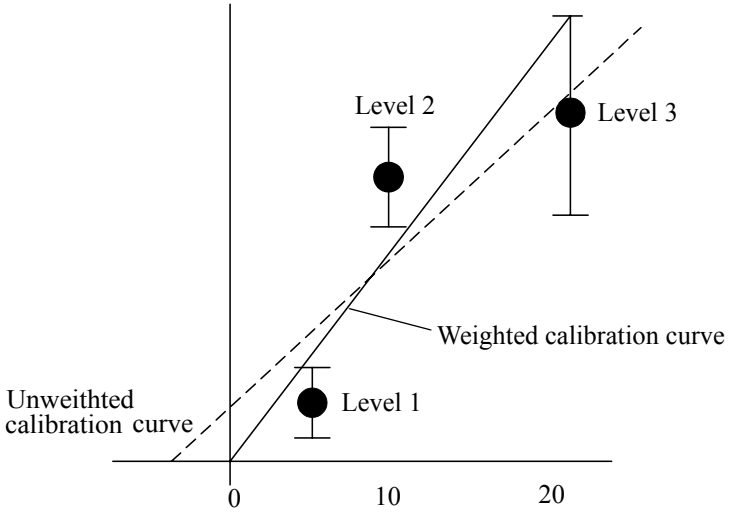
6 To weight the equation, make the following settings.

1) Select an equation from the ***Weighting Method*** drop-down list in the upper right section of the ***Edit Calibration*** dialog box.
2) Double-click on the ***Weight field*** to turn on weighting.

Double-clicking the field will alternately toggle the setting between on and off.

Generally, weighting is turned on when a low concentration is quantitated using a calibration curve with a wide dynamic range. When weighting is turned on, the calibration curve is drawn with weighting applied to a lower standard deviation (SD) level. The calibration curve is calculated based on the standard deviation SD at each concentration level. In other words, the calibration curve is affected more by a high-SD level. Generally, the SD of a high-concentration sample tends to be higher than that of a low-concentration sample. Consequently, calibration without weighting results in a smaller error on the high concentration side and a larger error on the low concentration side.

Therefore, when a low concentration is quantitated using a calibration curve with a wide dynamic range, set weighting to on and draw the calibration curve with a heavier weighting applied to the low concentration side (low SD) than the high concentration side (high SD). When the data used for individual levels contains a different number of replicates, apply weighting to the level with a lower standard deviation (SD) for the drawing of the calibration curve.



7 Click on the Units field and select the unit to be used for concentration.

8 Click on the Min Conc field and enter a minimum concentration value.

If the quantitative result is lower than the value set in Min Conc (for example, if ug/L is selected for units and 1.00 is entered in Min Conc), the quantitative report will show "< lug/l." If "---" is entered in Min Conc, the actual concentration will be indicated on the quantitative report.

9 Add elements to the table and set the concentration, if necessary.

To add an element to the table, enter the tune step, mass, and element symbol in the ***Add Element*** area, select the ***Analyte*** radio button, and click ***Add***.

NOTE The element used for interference correction is shown in ( ). This element is not included in the quantitative analysis.

10 Delete elements from the table, if necessary.

To delete an element from the table, click the right mouse button with the cursor positioned at the setting value of the element to be deleted and select ***Delete Element***.

11 Click *OK*.

The set concentration information will be saved.

NOTE To save edited calibration information, the calibration must be saved. For online data analyses, select ***Top*** window ***>> Method >> Save Calibration***. For offline data analyses, select ***Offline Data Analysis*** window ***>> Method >> Save Calibration***.

Setting the Internal Standard Correction

ChemStation allows you to set an internal standard for each element.

Select the internal standard closet to the mass of the analyte. The element set as an internal standard will not be quantitatively analyzed.

The CPS of an analyte can be corrected based on the value (VIS) calculated using an interpolation formula without changing the existing internal standard correction.

The internal standard correction can be set using the following procedure.

NOTE If an internal standard is not added to the standard sample and sample, the following step is not necessary.

1 Select an interpolation formula in the *Mass Interpolation Fit*: drop-down list in the upper right section of the *Edit Calibration* dialog box.

Refer to "Interpolation Formulas for Virtual-Internal-Standard Correction" in "Quantitation" in Appendix B "Formulas." for details on interpolation formulas.

NOTE If VIS is not used, it is not necessary to select an interpolation formula.

2 Select the Table tab.

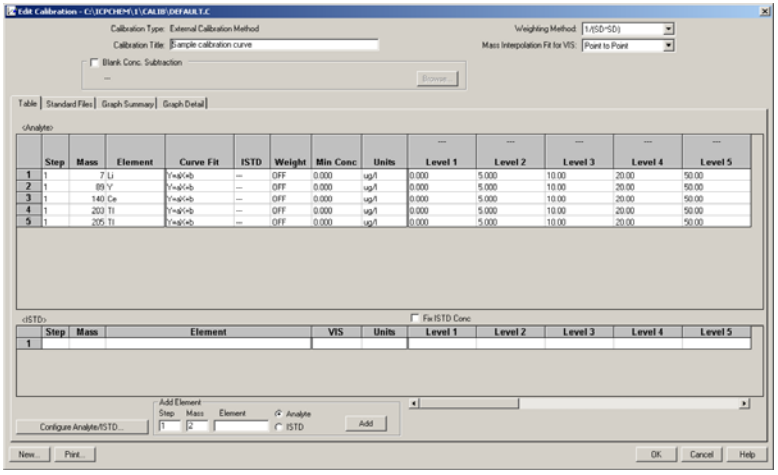


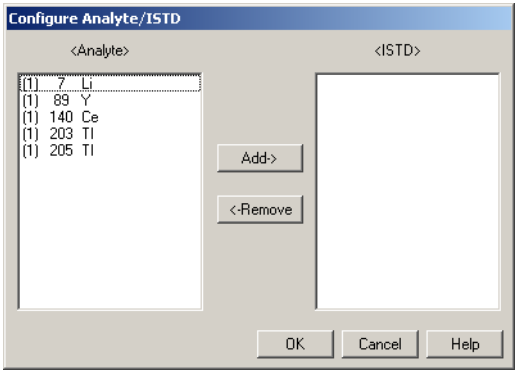
Table Tab

3 Enter the tune step, mass, and element symbol to be used as an internal standard in the *Add Element* area, select the *ISTD* radio button, and click *Add*.

An internal standard will be added to the ISTD table.

4 To change an element set as an analyte to an internal standard, click *Configure Analyte/ISTD* and select the element to be used as an internal standard.

When the ***Configure Analyte/ISTD*** dialog box appears, select an element. Choose an element in the Analyte list box and click ***Add->*** to select it. To cancel the selection, select an element in the ISTD list box and click ***<- Remove***. Selections may also be added to or removed from a list by double-clicking them. Click ***OK*** to close the ***Configure Analyte/ISTD*** dialog box and the selected element will be shown in the ISTD table on the ***Table*** tab screen.



Configure Analyte/ISTD Dialog Box

5 Click and select the field of the level in which a concentration level is to be entered.

Two or more consecutive fields may be selected by clicking on a field and dragging the cursor in a vertical direction.

6 Enter a concentration value.

To set a standard addition calibration value, the concentration of the background (bkg) file must be set to bkg or -1 for all elements. The concentration of a spiked standard sample should be set to 0 for all elements.

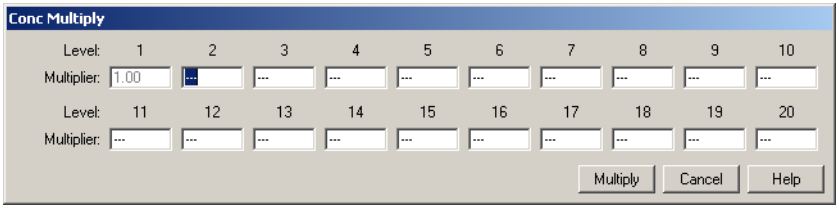
7 If the concentration value entered for a level is used to calculate the concentration values for other levels using multipliers, set multipliers using the following method.

NOTE

If the same concentration value will be used for all levels, select the ***Fix ISTD Conc*** check box. If the ***Fix ISTD Conc*** check box is selected, the following procedure is not necessary.

1) Select the field of the level to which a concentration value has been entered and click the right mouse button and select ***Conc Multiply***.

The ***Conc Multiply*** dialog box will appear.



Conc Multiply Dialog Box

The multiplier for the level input with a concentration value will show 1.00 and the indication will be grayed out (the value cannot be changed).

When a multiplier is entered in the text box for each level, the value calculated by multiplying the concentration value by the entered multiplier will be set for that level.

2) Enter a multiplier in the text box for each level and click ***Multiply***.

The ***Edit Calibration*** dialog box will appear again and indicate the calculated concentration values for the levels set with multipliers.

8 Click the *Units* field and select the unit used for concentration from the drop-down list.

9 To use VIS correction, double-click on the *VIS* field and add an "X" mark.

This will set a VIS element.

It is necessary to set one or more VIS elements for a point-to-point mass interpolation formula, two or more VIS elements for a linear formula, and three or more VIS elements for a quadratic formula.

Double-clicking the VIS field will alternately display and hide the X mark.

10 Click the *ISTD* field in the Analyte list and select an internal standard from the drop-down list.

For internal standard correction, select a mass. For VIS correction, select VIS.

Virtual Internal Standard correction

1 Definition of Virtual Internal Standard

The latest revision of ICP-MS ChemStation includes a function that allows the internal standard correction factor that is applied to a given analyte to be calculated from the actual signal change of one or more of the internal standards, based on the relative mass of the internal standards and the analyte. This can improve the accuracy of the internal standard correction factor applied, particularly when available internal standards are widely separated in mass and when long-term drift occurs which has a component that is mass dependent.

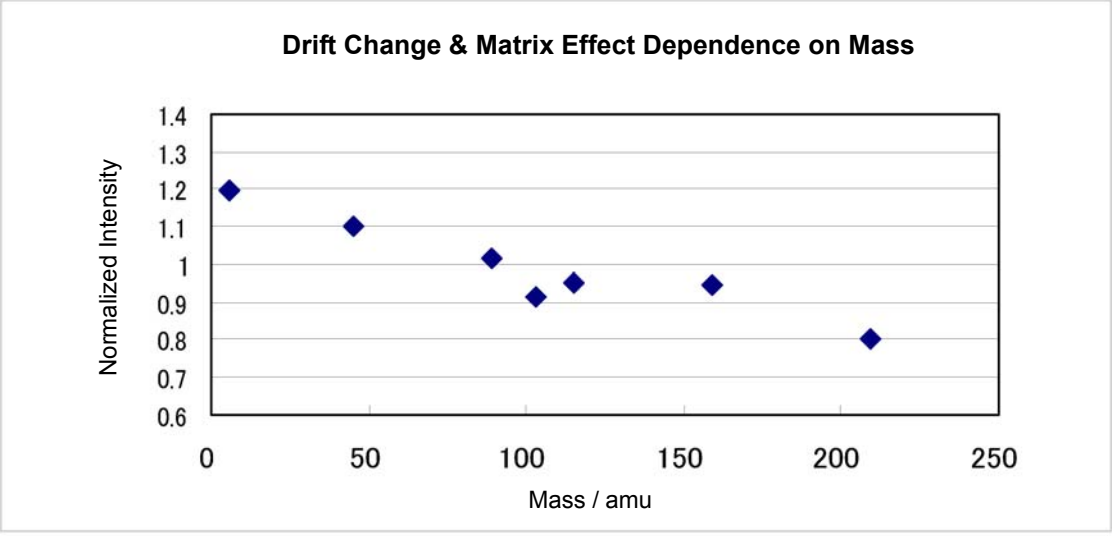
2 How to use

2-1. Background

The main reason for the use of internal standards in ICP-MS is to correct for long-term drift, which results from changes to the characteristics of the interface cones, especially small changes to the profile of the sample cone orifice, which can occur after a prolonged period of analysing high dissolved solids samples. The fact that this signal drift is sometimes different for different analyte masses means that several internal standards may be useful to accurately correct for the signal drift. In cases where the analytes are many mass units away from the nearest internal standard, or a limited number of internal standards are available, the accuracy of the correction can be improved by using interpolation of the drift correction factor between the measured internal standard elements.

In addition to the accurate correction of mass dependent drift, interpolation of internal standards can be used to correct for some aspects of matrix effects, since it is said that the space charge is one of the biggest contributions to the matrix effect. In this case we might expect to observe some mass dependency of the matrix effect, similar to that observed due to drift caused by some heavy matrix introduction. Virtual Internal Standard correction may also be used to correct for matrix effect, where the main component of the matrix effect is mass dependent, in particularly when available ISTDs are limited. Because this is just an optional function, you should confirm that this option works well for your analysis.

2-2. Effective Example



In this scatter plot Li6, Sc45, Y89, Rh103, In115, Tb159 and Bi209 show a clear mass dependency. In such a case this virtual ISTD correction option is useful, in particular some ISTDs are not available.

2-3. Limitation

It is important to note that space charge effects are not the only matrix effect. In particular, a high matrix component in the sample is likely to lead to ionisation suppression in the plasma, in which case the main component of the signal change will be the analyte ionisation potential, not the analyte mass. If this is the case, then mass-based interpolation of the internal standard signal will not necessarily improve the data and a better approach would be to select internal standards that are close to the analytes in terms of ionisation potential. If the relationship between the mass of analytes and the sensitivity change is not consistent, the Virtual Internal Standard correction option should be used with caution. The ISTDs behavior on mass axis can be checked easily and, if necessary, you can make sure a further detailed check by a recovery test.

VIS correction formula

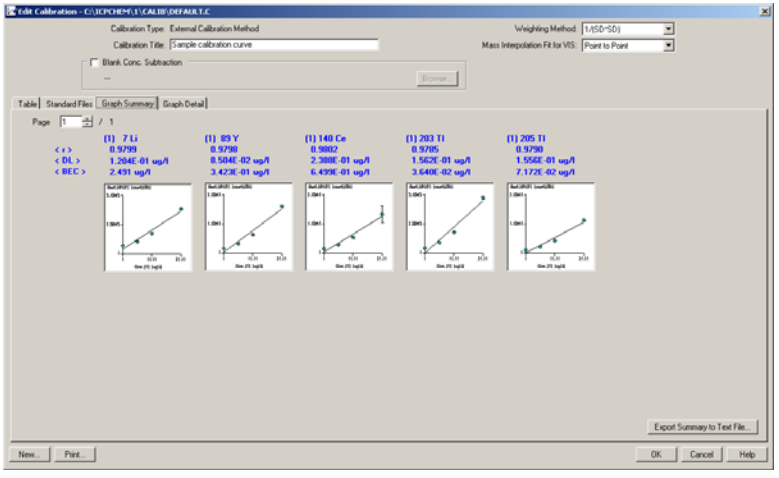
Point-to-Point	Linear interpolation (no extrapolation) applied to data of two VIS elements	One or more VIS elements required
Linear	Linear formula obtained based on data of all VIS elements	Two or more VIS elements required
Quadratic	Quadratic formula obtained based on data of all VIS elements	Three or more VIS elements required

Confirming the Graph Summary

The ***Graph Summary*** tab screen permits checking of the correlation coefficient factor (r), DL, and BEC (background-equivalent concentration) of all elements for the calibration set on the ***Table*** tab screen.

1 Select the *Graph Summary* tab in the *Edit Calibration* dialog box.

The ***Graph Summary*** dialog box will appear.



Graph Summary Dialog Box

If there are too many elements to fit on one page, they will be displayed on two or more pages. Up to 12 elements can be shown on one page. You can move to the next or previous page by entering a numeric value directly into the Page list box or clicking ▲ or ▼ .

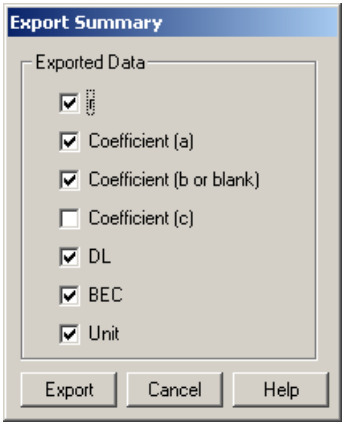
The title of each element shows the (tune step number), mass, and element symbol, as well as the correlation coefficient factor (r), DL, BEC (background-equivalent concentration), and calibration curve.

Clicking on a calibration curve will switch to the ***Graph Detail*** tab and detailed information on the selected calibration curve will be displayed.

2 The following procedure enables production of text file output of the tune step, mass, correlation coefficient factor (r), DL, BEC (background-equivalent concentration), and unit for each element.

1) Click ***Export Summary to Text File***.

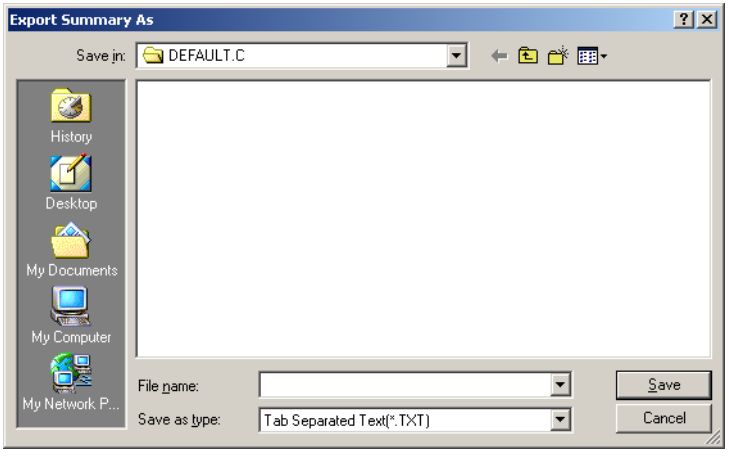
The ***Export Summary*** dialog box will appear.



Export Summary Dialog Box

2) Select the data to output and click ***Export***.

The ***Export Summary As*** dialog box will appear.

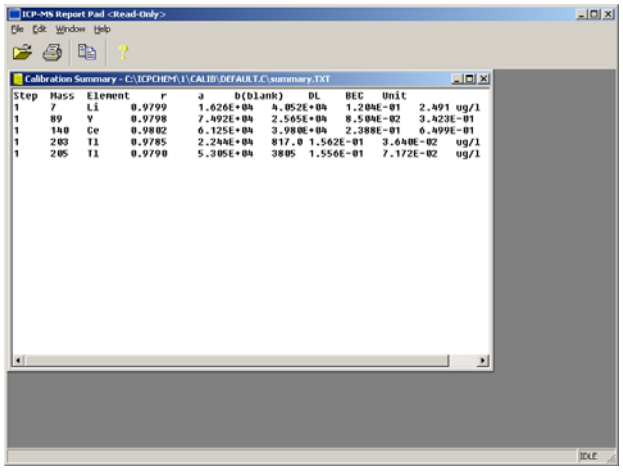


Export Summary As Dialog Box

3) Select the location to save the data, enter a name for the text file, and click ***Save***.

The data will be saved in a text file.

The ***ICP MS Report Pad*** window will display the saved text file.



ICP-MS Report Pad Window

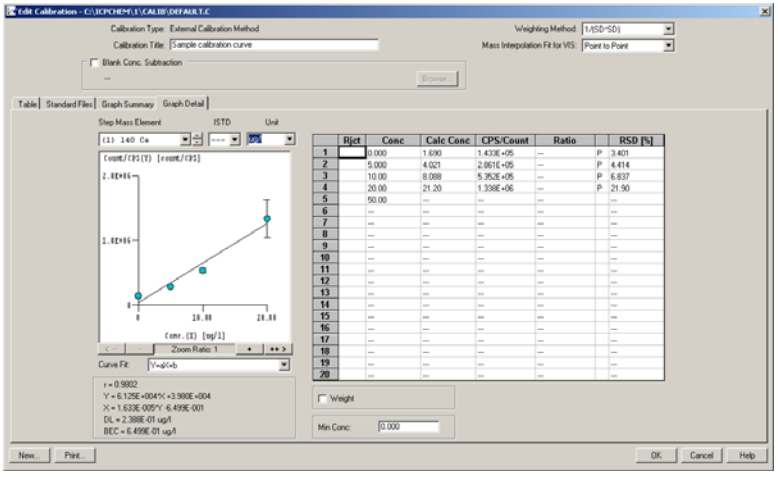
Confirming the Graph Detail

The details of the calibration set on the ***Table*** tab screen can be checked by examining the calibration curve displayed on the ***Graph Detail*** tab screen.

It is also possible to make the same settings as can be made on the ***Table*** tab screen while observing the changes on the graph. The set values will also be reflected in the ***Table*** tab screen.

1 Select the *Graph Detail* tab in the *Edit Calibration* dialog box.

The ***Graph Detail*** dialog box will appear.



Graph Detail Dialog Box

The left side of the dialog box shows the calibration information and a calibration curve. Clicking [-] will zoom out and clicking [+] will zoom in. Clicking [<--] will reset the zoom and clicking [++>] results in maximum zoom.

The table on the right side shows the concentration, calculated concentration value, CPS or count, strength ratio (if internal standard correction is used), and detector mode (P or A) for each level, as well as the RSD table for the element at each calibration level.

2 To check a point of a level on the calibration curve, perform the following procedure.

1) Select a line in the table on the right side.

The selected line will be highlighted and a circle will appear around a point on the graph.

The color of the point on the calibration curve provides an indicator as follows: light blue for pulse mode and green for analog mode.

2) To reject a point on the calibration curve, double-click on the ***Rjct*** field. The ***Reject*** field will show "*."

3 Change the calibration settings as necessary.

The set values are also reflected in the ***Table*** tab screen.

- Changing the element displayed on the graph

  Select from the Step Mass Element drop-down list. If an internal standard is selected, the ***Weight*** check box will change to the ***VIS Conc*** check box. For VIS, select the check box.

- Setting an internal standard

  Select from the ***ISTD*** drop-down list (selection can be made only when the Internal Standard has been set on the ***Table*** tab screen). When a selection is made, the Ratio field will display a value.

- Selecting an equation

  Select from the ***Curve Fit*** drop-down list.

- Setting concentration

  Enter a value in the ***Conc*** field. Click the right mouse button to open the popup menu, which allows you to copy, paste, or fill down the content of the field, or set a concentration multiplier.

- Setting weight for analyte

  Select the ***Weight*** check box.

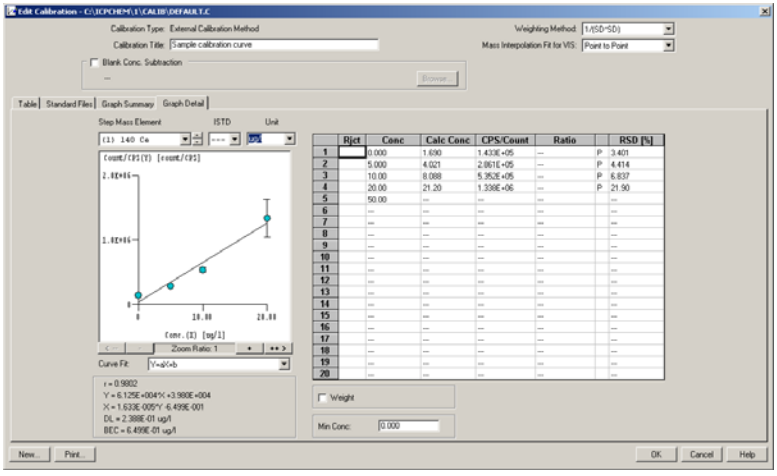
- Setting the minimum concentration.

  Enter a value in the ***Min Conc*** field.

Printing Calibration Data

Calibration data can be printed out using the following method.

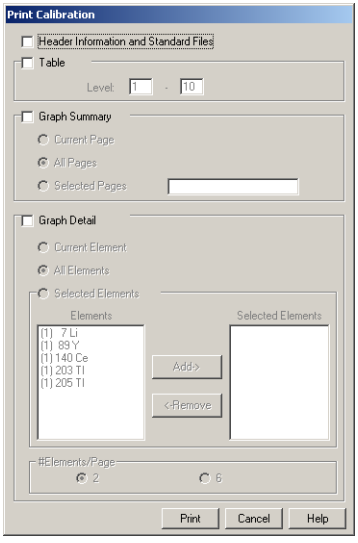
1 **Click *Print* in the lower left section of the *Edit Calibration* dialog box.**



Edit Calibration Dialog Box

The ***Calibration - Print*** dialog box appears.

2 To output the header information (title, standard data file name, etc.) and the data file information, select the *Header Information and Standard Files* check box.



Calibration - Print Dialog Box

3 To print the content of the *Table* tab screen, select the *Table check* box.

4 To print the content of the *Graph Summary* tab screen, select the *Graph Summary* check box.

5 To print the content of the *Graph Detail* tab screen, select the *Graph Detail* check box, and enter the following settings.

1) Select the element or elements for which you want to print out the calibration data.

- ***Current Element***: element whose calibration curve is displayed on the ***Graph Detail*** tab screen
- ***All Elements***: all elements in the table on the ***Graph Detail*** tab screen
- Selected Elements: The elements to be printed can be selected by highlighting the selected elements in the Elements list box and clicking ***Add->***. To cancel a selection, select an element in the ***Selected Elements*** list box and click ***<-Remove***. Selections can also be added to or removed from a list by double-clicking them. Multiple consecutive elements can be selected by pressing the Shift key and clicking on consecutive elements. Any multiple elements can be selected by pressing the Ctrl key and clicking on the desired elements.

2) Select the number of elements to be printed on one page using the ***#Elements/Page*** radio button.

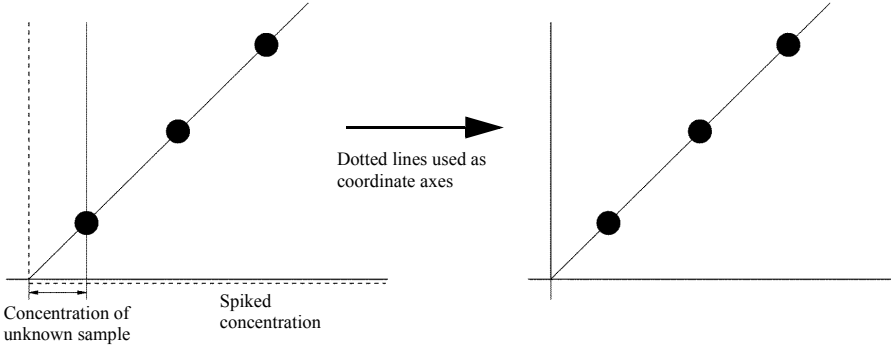
When "6" is selected, the calibration concentration levels, counts, and RSD will not be printed.

6 Click Print.

The calibration data will be printed out.

Converting to Calibration Method

For standard addition, selecting ***Calibrate >> Convert to Calibration Method*** will shift the concentration of each level in the calibration curve produced via standard addition by the quantitative calibration result and convert it to an absolute calibration curve.



Blank Subtraction

ChemStation enables you to subtract a blank data file from the sample data file, for example, where the sample reagent blank is different from the standard reagent blank. This blank subtraction is performed after both the blank data file and the sample data file are quantitated (unlike the background subtraction where the background counts are subtracted from the sample counts). Hence, the calculated concentration of a given element in the blank data file is subtracted from the calculated concentration of that same element in the unknown sample, resulting in a blank-subtracted concentration for the unknown.

Background correction is not ordinarily used in quantitation. Its main purpose is to subtract a background spectrum from a sample spectrum so the user can view the net spectrum for qualitative purposes. Since background correction subtracts raw counts before normalizing for internal standard response, it could give incorrect results with internal standards.

To subtract the blank data file from your sample data files, complete the following steps:

1 **Select the *Blank Conc Subtraction* check box in the *Edit Calibration* dialog box.**

2 **Click *Browse*.**

The ***Select Data File*** dialog box will appear.

3 **Select a blank data file.**

4 **Click *OK*.**

The ***Select Data File*** dialog box will close and the blank concentration subtraction value will be set.

NOTE Blank data can be printed by selecting ***Calibration >> Print Blank Conc***.

Generating a Quantitation Report

ChemStation generates a report showing the results of a quantitative analysis in either of two ways:

- You can tell ChemStation to generate the report automatically each time a method runs. When you do so, you specify whether to generate a summary or detailed report, and whether to send the report to the screen, the printer or a file. For information about how to generate a quantitation report automatically (using a method), see Chapter 5, "Creating a Method".
- You can create a custom report with the data acquired each time the method runs. Before you can create a custom report in this way, you must set up a custom report template. For more information about custom reports and databases, see Chapter 11, "Creating Custom Reports".
- If you did not tell ChemStation to generate a report automatically, you can also generate a report manually by completing the following steps:

1 Load a data file by selecting *Data File>>Load*.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d

Soil02.d

Soil03.d

Water01.d

Water02.d

Water03.d

and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***Data File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

2 Select *FullQuant>>Generate Report*.

The ***FullQuant Report Option*** dialog will appear.

3 Select the report style.

The following report styles are available:

- Summary

  Gives the counts per second (CPS) or counts (for Time Resolved Analysis), concentration, and relative standard deviation (RSD) for each mass in the sample.

- Detailed (Text Only)

  Gives the same information as the summary report, as well as, the standard deviation (SD). The format is slightly different but the SD and RSD for the counts per second (CPS) or counts (for Time Resolved Analysis) and the concentration for each mass are still displayed.

- Detailed

  Includes the same information as the Detailed (Text Only) report, but which also includes a spectrum of the sample or a time chart of each element at the bottom of the report. This report can only be sent to the printer.

4 Select the destination for the report.

You can click one or more of the following check boxes:

- Screen

  Generates the report to the ChemStation screen.

- Printer

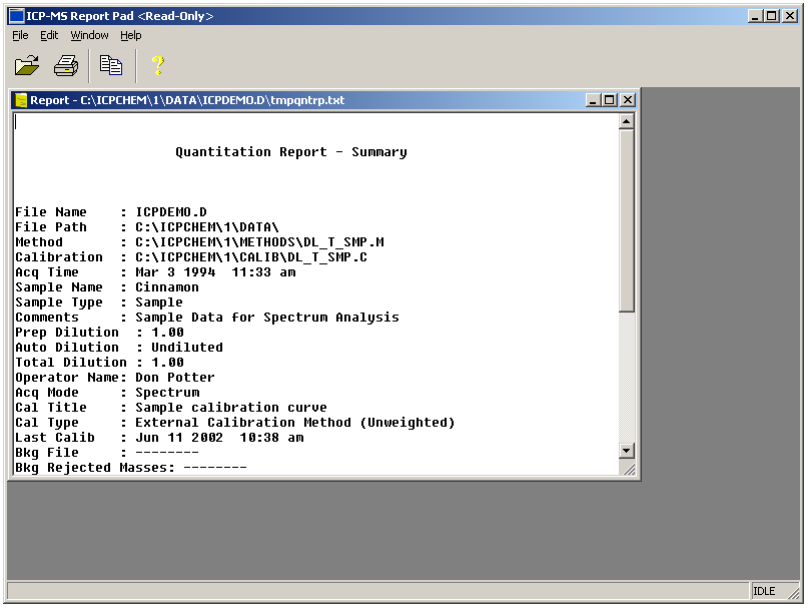
  Generates the report to the ChemStation printer. You must select this destination for a detailed report.

- File

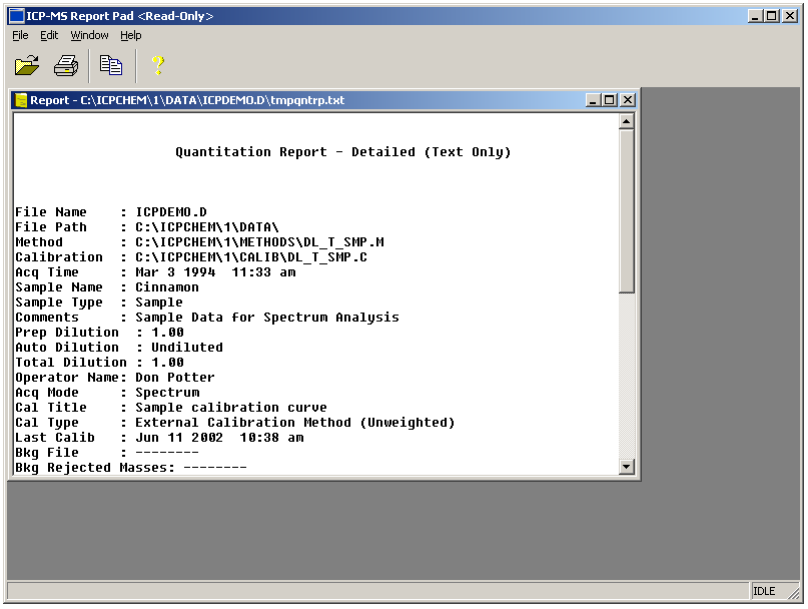
  Generates the report to the file you indicated in the text box.

5 Click *OK*.

The report will be generated to the selected destination in the selected style.



Quantitation Report - Summary



Quantitation Report - Detailed (Text Only)

NOTE

In the report, the concentration of the internal standard is placed in < > in the specification of the concentration of the element.
This value is the value entered for calibration and does not represent a quantitative result.

13

Performing a Semiquantitative Analysis

Performing a Semiquantitative Analysis

You perform a semiquantitative analysis to obtain information about the concentrations of all elements that are present in an unknown sample. It is a very powerful tool to obtain concentration information for a large number of elements (>70) without the need for external calibration. It is very useful as a screen prior to quantitative analysis on target elements. Typically, semiquantitative analysis is accurate to +/- 30% or better on completely unknown samples. ChemStation can subtract a sample reagent blank and compensate for changes in sample matrix using internal standardization

This chapter explains how to set element response factors, view the responses from a semiquantitative analysis, and generate a semiquantitative report.

NOTE

You can not use Semiquantitative analysis with ShieldTorch (cool plasma) conditions or Agilent 7500ce reaction mode. The reason for this is that all element response factors in the ChemStation data base, are calculated using normal plasma conditions and ion energies.

Running a Calibration Sample

Before performing a semiquantitative analysis, it is recommended that the user first prepares and analyzes a calibration standard to set the element response factors for semiquantitative calculations. Use a sample containing known concentrations of three or four elements as a calibration standard for semiquantitative analysis. If the unknown sample matrix is known, the calibration standard can be matrix matched to improve the accuracy of the analysis to that approaching full quantitative analysis.

Run the calibration standard prior to running the unknown samples. If you are analyzing the samples manually, run the calibration standard and then the unknown samples. ChemStation calculates responses for all elements in the mass spectrum based on the response to the known concentration of the elements in the calibration solution. These concentrations of elements should cover the low, medium, and high ranges of the mass spectrum. A multi-element solution including Li, Y, Ce, and Tl can be used for calibration of Semiquantitative Analysis. For information about running the samples, see Chapter 8, "Running a Sample Analysis".

Viewing the Results of Semiquantitative Analysis

ChemStation contains original response values for each element in the periodic table. The higher the response value, the more efficiently that element is ionized in the plasma, which gives higher sensitivity for a given concentration. These original response factors have been empirically determined by Agilent Technologies. After you run the calibration standard and edit the existing semiquantitative parameters, ChemStation adjusts the original response factors to give "corrected factors", which take into account the mass response of the individual instrument. The corrections can also be reviewed graphically in the response graph.

This section explains how to load a data file, enter concentration values for the calibration elements, correct responses for all elements based on the calibration standard, and view the response graph.

Loading a Data File

Before you can view responses from an unknown sample, you must first load the data file ChemStation generates during data acquisition. To do so, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The Data Analysis menu appears.

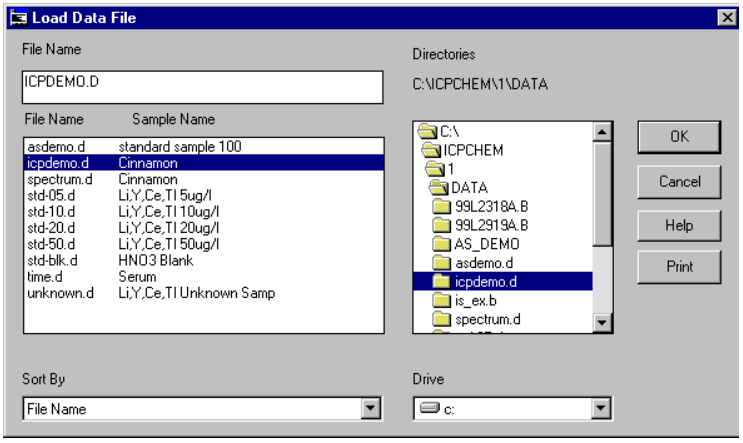
2 **Select *Data Analysis>>Main Panel*.**

The Data Analysis window appears.

NOTE You can also double-click on the ***Off-line Data Analysis*** icon.

3 **Select *Data File>>Load*.**

The ***Load Data File*** dialog box appears. Allows the user to select the data file to be loaded into data analysis for processing.



Load Data File Dialog Box

- Sort By

  Allows the user to sort the list of data files in the current directory by the selected type of information. Options include File Name, Sample Name, Misc Info, and Acquired Date. The File Name Only option allows the user to display a listing of file names only, without sample name or any other additional information.

4 Select a data file using one of the following methods:

- Click a file in the displayed list and click ***OK.***
- Double-click a file in the displayed list.
- Type the file name and click ***OK***.

ChemStation loads the data file and returns to the Data Analysis window.

Selecting ***File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d

Soil02.d

Soil03.d

Water01.d

Water02.d

Water03.d

and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***Data File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

NOTE

Before you start sample data analysis, you should correct the original ChemStation data with the standard solution response. First load your standard file, correct the sensitivity, then analyze your sample data.

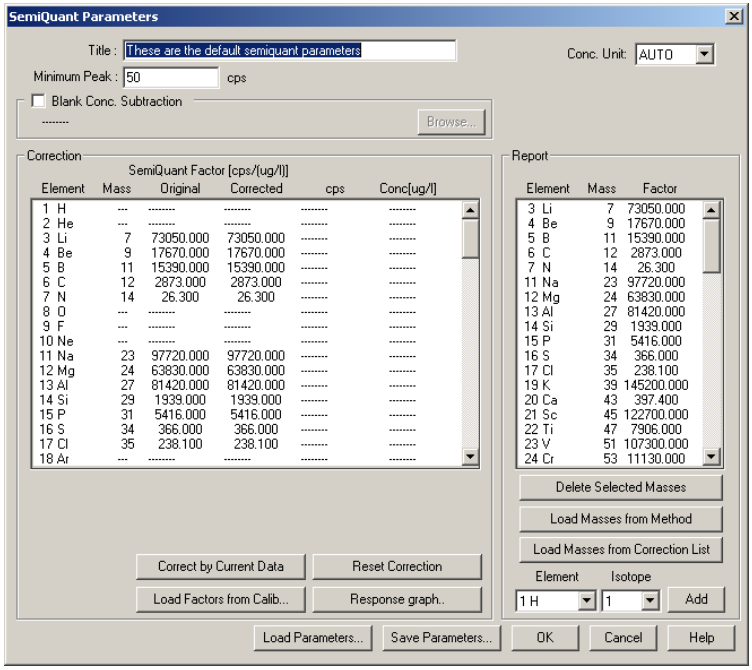
Entering Concentration Values

After you load the data file, you must enter the concentrations for the calibration elements before you can view responses for the other elements.

To enter concentrations for the calibration elements, complete the following steps:

1 Select *SemiQuant>>Edit SemiQuant Parameters.*

The *SemiQuant Parameters* dialog box appears.



SemiQuant Parameters Dialog Box

2 Place the cursor on the Conc column for the element for which you want to enter the concentration.

ChemStation highlights the Conc field. A Concentration text box and an Enter push button appear at the bottom of the dialog box.

Concentration Text Box

3 **Type the concentration for the selected element and click *Enter.***

The concentration value appears in the Conc field for the selected element.

4 **Repeat these steps for each element in the calibration solution.**

NOTE If you want to enter the same concentration value for multiple calibration elements, you can press ***Control*** and then click the ***Conc*** field for each element in the calibration solution. Then, enter the concentration value and click ***Enter***. ChemStation inserts the same concentration value for all the elements you select.

Remain in the Edit SemiQuant Parameters dialog box to view the responses for the other elements; that is, those elements that are not in the calibration solution. As mentioned earlier, 3-4 elements covering the mass range are usually sufficient to produce corrected responses for all other elements. See "Correcting Responses for All Elements" in this chapter for instructions.

NOTE SemiQuant response factors for all elements that are not present in the standard are calibrated in the following way:
The initial SemiQuant response stored in the software for a given element is adjusted using a factor derived from the corrected responses obtained for elements used in the SemiQuant calibration.

The nearest two elements on the high mass and low mass sides of the non-calibrated mass are used. A line is drawn between these two masses, and a factor calculated at the mass of interest. If the mass of interest is lower than the lowest mass used in the SemiQuant calibration, then the factor obtained for the lowest mass is applied. The same method is followed if the mass of interest is higher than the highest mass used in the SemiQuant calibration.

Entering Minimum Peak Value

Enter the minimum cps that must be present for a given mass in order that the element corresponding to that mass is recognized as being detected in the sample. If the cps for a mass is greater than or equal to the Minimum Peak value, semiquant analysis will calculate and report a concentration for that element. If the cps for a mass is less than the Minimum Peak value, the corresponding element will be reported as "< xxx", where "xxx" is equal to concentration that would be derived using the Minimum Peak value and the semiquant factor for the element in question.

The default value is set to 50 cps.

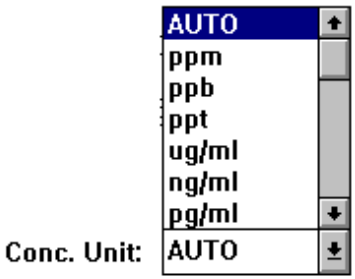
Selecting Concentration Units for the Report

ChemStation will select a suitable concentration unit for each element being reported, or you can choose the concentration unit for all elements being reported.

To select the concentration unit, complete the following steps:

1 **Click on the arrow of the Conc. Unit box.**

The Concentration Unit box appears.



Concentration Unit Box

2 **Select a unit.**

The selected unit will be used for all elements when the report is generated. You can also select ***AUTO***, and then ChemStation will select a suitable unit, dependent on the concentration value found.

Entering and Deleting Counts Per Second (cps) Values

Although you would normally use the units obtained from the calibration standard to set the corrected responses, there may be times when you want to enter the counts per second (cps) values manually. An example would be when performing laser ablation, where a suitable standard may not be available and the approximate cps value for certain elements is already known from tuning.

To enter cps values, complete the following steps:

1 **Place the cursor on the cps column of the element for which you want to enter a cps value.**

ChemStation highlights the cps field. A cps text box, an Enter push button and a Delete push button appear at the bottom of the dialog box.

CPS Text Box

2 **Type the cps value for the selected element and click *Enter*.**

The cps value appears in the cps field for the selected element.

3 **Repeat these steps for any other elements for which you want to enter cps values.**

You can also delete cps values. To do so, complete the following steps:

4 **Place the cursor on the cps column of the element for which you want to delete the cps value.**

ChemStation highlights the cps field. A cps text box, an Enter push button and a Delete push button appear at the bottom of the dialog box.

5 **Click *Delete*.**

The cps value for the selected element will be eliminated.

6 **Repeat these steps for all of the elements for which you want to delete cps values.**

Changing the Original Response Values

In normal circumstances, original response values should *not* be changed. However, there may be situations when you want to change the original values; for example, for research purposes or following significant hardware changes.

CAUTION 

Before you change the original response values that are loaded with ChemStation, make a copy of the semiq.prm file if required. The semiq.prm file is created under the method. For information about copying files, refer to the documentation for Microsoft Windows and for DOS.

To change the original response values, complete the following steps:

1 **Click the original value you want to change.**

A text box and an Enter push button appear at the bottom of the dialog box.

Original Response Text Box

2 **Type the new value and click *Enter.***

The changed value appears for the selected element.

NOTE

When you exit ChemStation, the response value you entered will change back to the original response value. To permanently change an original response value, you must save the method. To do so, select ***Methods>>Save*** from the Top window, and complete the saving steps as described in Saving a Method and Calibration in Chapter 3. You should not overwrite the original SemiQuant Factors in **default.m**. If you lose the original SemiQuant Factors by overwriting **default.m,** and want to refer back to them, you can load the original values by copying over the original **default.m** method from **\icpchem\icpsetup\methods\default.m**.

Changing the Mass to be Analyzed

In normal circumstances, the default masses displayed in the SemiQuant Parameters window are the best choices, so there is no need to change them. However, there may be cases when you want to change the default mass; for example, to see the difference in using alternate mass, or in the event that the sample matrix gives rise to a polyatomic ion that would interfere with the default analyte mass.

To change the default mass, complete the following steps:

1 **Click the mass you want to change.**

A text box and an Enter button appear at the bottom of the dialog box.

M/Z Text Box

NOTE

The original response factor listed in the SemiQuant Parameters panel for a given element was determined based on the default mass for that element. If the mass is changed to a different isotope, the response value is not automatically changed to reflect the different relative abundance of the alternate isotope. Therefore, if it is necessary to change the mass, a new response value must be entered. The new response value can be calculated by correcting the original response value for the relative isotopic abundance difference between the default mass and the alternate mass. For example, the default mass for Cu is 63; the default response value for Cu is 8123. If it was necessary to measure Cu using mass 65 rather than 63, the new response value would be calculated as follows:

x = [(rel. abn. Cu65)/(rel. abn. Cu63)] * orig response value for Cu
= [(30.8)/(69.2)] * 8123
= 3615
Relative isotopic abundances can be taken from the Relative Isotopic Abundance Table laminated card provided with your Agilent 7500.

2 **Type in the new mass and response value and click *Enter.***

The changed value appears for the selected element.

NOTE

Default masses are chosen so as to avoid isobaric and common polyatomic overlaps. Where there are several interference free isotopes, then the most abundant is selected.

Correcting Responses for All Elements

After you have loaded the data file and entered concentration values for the calibration elements, you can view the responses for all other elements.

NOTE The procedures in the following 8 sections assume that you are in the Edit SemiQuant Parameters dialog box. For information about accessing this dialog box, see "Entering Concentration Values" in this chapter.

To view responses for all elements, complete the following step:

Click *Correct by Current Data*.

The corrected response values change and counts per second (cps) values appear for all elements normally analyzed by ICP-MS. The original response values remain unchanged.

NOTE The number of counts detected for the most abundant POINT of all points measured for the semiquant mass is used in calculating the SemiQuant Factor. This value can be examined by selecting ***Spectrum>>Tabulate / Point...*** within Data Analysis, if desired.

Resetting the Response Values

ChemStation enables you to reset the original response values. To do so, complete the following step:

Click Reset Correction.

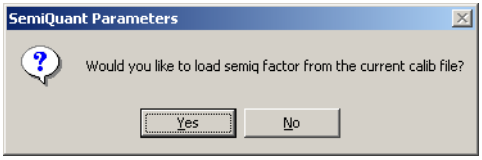
ChemStation changes the corrected values to the original response values.

Loading Factors from a Calibration File

ChemStation enables you to load semiquantitative factors from a calibration file.

1 Click *Load Factors from Calib*.

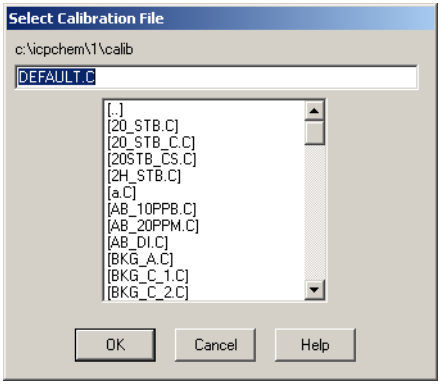
The ***SemiQuant Parameters*** dialog box will appear.



SemiQuant Parameters Dialog Box

2 To load data from a calibration file that is being edited and has not been saved, click *Yes*. To load data from an existing calibration file, click *No*.

Clicking ***No*** will bring up the ***Select Calibration File*** dialog box.



Select Calibration File dialog box.

3 Select a calibration file using one of the following methods.

- Select a file name from the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Enter a file name and click ***OK***.

NOTE

If multiple tune steps exist in the selected calibration file, the ***Select Tune Step*** dialog box will appear. Select a tune step using this dialog box.
If multiple isotopes exist in the selected calibration file, the ***Select Mass*** dialog box will appear. Select an isotope using this dialog box.

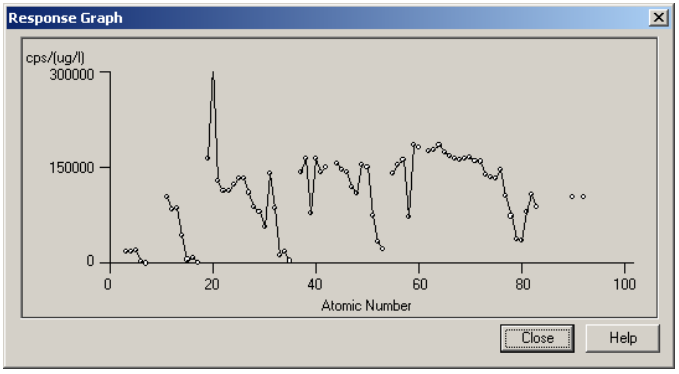
The ***Select Calibration File*** dialog box will close and the selected calibration file will be loaded.

Viewing the Response Graph

After you correct responses for all elements, you can view a graph of those responses. To do so, complete the following steps:

1 Click *Response Graph*.

The ***Response Graph*** dialog box appears.



Response Graph Dialog Box

The scale for the X-axis of the graph is 0 to 103 and shows responses for each element by atomic number. The Y-axis shows the counts per second (cps).

2 To close the dialog box, click *OK*.

ChemStation returns to the SemiQuant Parameters dialog box

Specifying an Element for Report Output

In semiquantitative analysis, it is possible to control the inclusion of individual elements in the report. For instance, elements receiving interference from oxides, doubly charged ions, etc can be excluded from the report.

Output settings can be entered using the following procedure.

1 **In the list displayed in the Report area, select the elements that you do not want to include in the report.**

In the default setting, all elements are shown in the list.

Select multiple consecutive elements by holding down the Shift key.
Select any multiple elements by holding down the Ctrl key.

2 **Click *Delete Selected Masses*.**

The selected elements will be deleted from the list.

3 **Select the elements/isotopes to add to the report from the drop-down list and click *Add*.**

The selected elements will be added to the list.

4 **To load elements from a method, click *Load Masses from Method*.**

5 **To load masses from the correction list on the left side, click *Load Masses from Correction List*. All masses selected in the correction list will be loaded to the report list.**

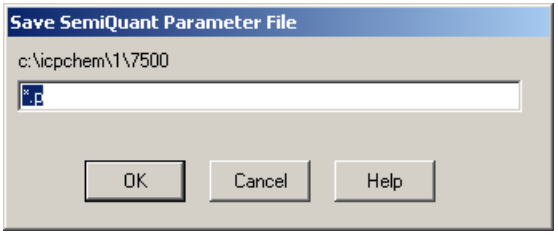
Elements will be loaded from the method.

Saving Parameters

Set semiquantitative parameters can be saved using the following method.

1 **Click *Save Parameters*.**

The ***Save SemiQuant Parameter File*** dialog box will appear.



Save SemiQuant Parameter File Dialog Box

2 Enter a file name and click *OK*.

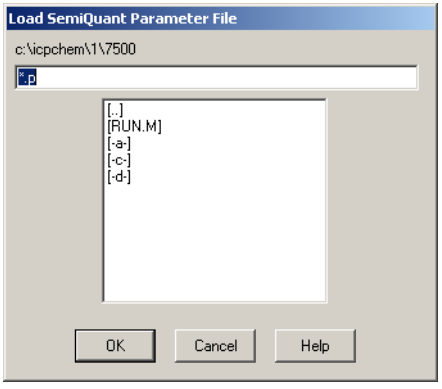
The ***Save SemiQuant Parameter File*** dialog box will close and the semiquantitative parameters will be saved.

Loading Parameters

The saved semiquantitative parameters can be loaded into ChemStation using the following method.

1 Click *Load Parameters*.

The ***Load SemiQuant Parameter File*** dialog box will appear.



Load SemiQuant Parameter File Dialog Box

2 Select a calibration file using one of the following methods.

- Select a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Enter a file name and click ***OK***.

The Load SemiQuant Parameter File dialog box will close and the selected semiquantitative parameters will be loaded.

Using Internal Standard Correction

ChemStation enables you to make internal standard corrections if you add internal standards to your sample in known concentrations. In semiquantitative analysis, internal standard correction is strongly recommended, since changes in sample matrix can effect instrument sensitivity, which will directly affect the reported concentration values.

There are two modes of internal standard addition: Auto Add Mode and Normal Mode. The settings are not saved when you change between modes.

Auto Add Mode

The Auto Add Mode is used when the internal standard is added automatically with a peristaltic pump (on-line ISTD addition). In this case, the precise internal standard (ISTD) concentration may not be known, but the ISTD concentration in every type of sample - calibration standard, a blank, an unknown sample, etc. - will be identical.

The internal standard factor is calculated using the following equation:

ISTD factor = (cps of ISTD in the Internal Standard Data File)
/ (cps of ISTD in the sample)

The concentration for each element is calculated using the following equation:

Conc. = (cps in sample) x (ISTD factor) x (dilution factor)
/ (SemiQuant factor)

where the dilution factor is obtained from the Start Run panel if running a method or in the Sample Log Table if running a sequence (default = 1.0); and the SemiQuant factor is obtained from the SemiQuant parameters panel.

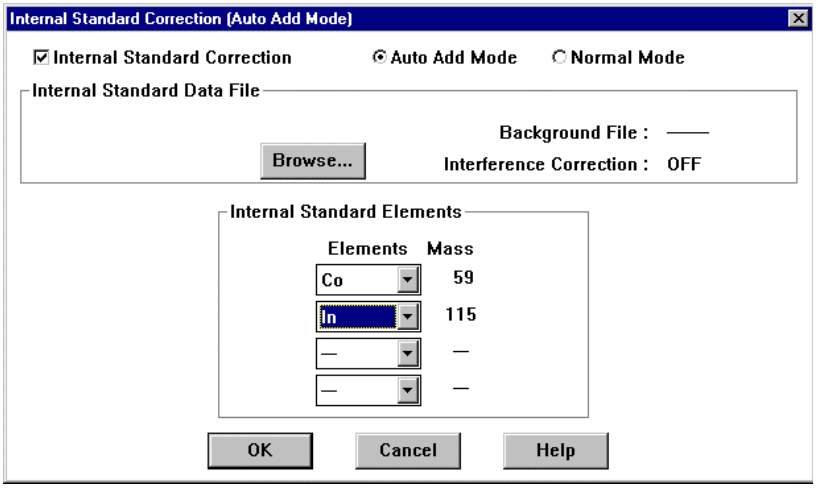
To set up the Internal Standard Correction, perform the following steps in Data Analysis:

NOTE

To use the Internal Standard Correction manually, a data file to be used as the "Internal Standard Data File" should be acquired prior to setting up the Internal Standard Correction. When a sequence is used, the Internal Standard Correction can be set in the **Type**. The solution should be either a blank or a calibration standard acquired using on-line ISTD addition.

1 **Select *SemiQuant>>Internal Standard Correction*.**

The ***Internal Standard Correction*** dialog box appears.



Internal Standard Correction Dialog Box

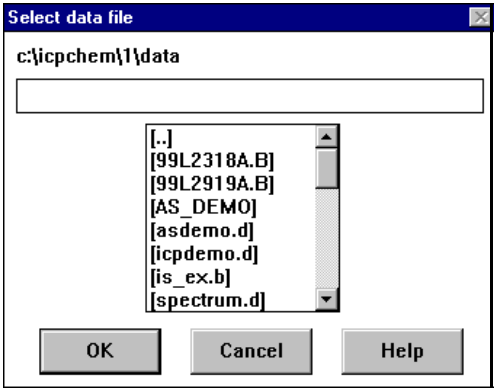
2 **Select Auto Add Mode button on the top of the dialog box if it is not already selected.**

3 **Click the Internal Standard Correction check box on the top of the dialog box if it is not already checked.**

The Internal Standard Correction check box will be checked.

4 **Click the *Browse* push button.**

The ***Select Data File*** dialog box appears.



Select Data File Dialog Box

5 **Select the Internal Standard Data File previously acquired using one of the following methods:**

- Click the file in the displayed list and click ***OK***.
- Double-click a file in the displayed list.
- Type the file name and click ***OK***.

This may be the same data file used to correct the semiquant parameters. Alternatively, a separate data file containing only the internal standard can be used for correction.

The dialog box will disappear and the selected internal standard data file will be displayed to the left of the Browse push button. When a background file or an interference correction in the current method is set, the background file name will be displayed to the right of the Browse button or the Interference Correction will be ON.

NOTE

To set the Background File and Interference Correction, select ***Method >> Data Correction*** in the ICP-MS Data Analysis window.

The background data file and interference correction, are parameters in the method. However, they are not updated without loading the Internal Standard Data File here even if these parameters are changed in the method.

6 Click the arrow under Elements in the Internal Standard Elements box.

All elements are in this list.

7 Select the internal standard element from the list.

The mass to be used as the internal standard will automatically be selected from the SemiQuant Parameters.

NOTE The mass defined in the Internal Standard Correction and the SemiQuant parameters must be the same.

8 Select up to 4 elements for internal standards, repeating steps 5 and 6 as needed.

You can specify up to four elements to be used as internal standards. If only one element is selected, all reported elements will be corrected using the single internal standard element specified. If more than one internal standard element is selected, the internal standard correction factor used for each element between any two adjacent internal standard elements is derived by interpolation of the linear response between those two internal standard elements.

Mass displays the mass of the specified element as set in the semiquant parameters panel.

9 Click *OK*.

The internal standard correction will now be set and ChemStation returns to the ICP-MS Data Analysis window.

Normal Mode

The Normal Mode is used when the ISTD is added to the unknown sample only. This mode can also be used with Laser Ablation, where the matrix element is used as the ISTD. The ISTD factor is calculated from the SemiQuant factor, so no Internal Standard Data File is required for this mode.

The internal standard (ISTD) factor is calculated from the following equation:

ISTD factor = (SemiQuant Factor of ISTD) x (ISTD Concentration) / (cps of ISTD in the sample)

The concentration for each element is calculated using the following equation:

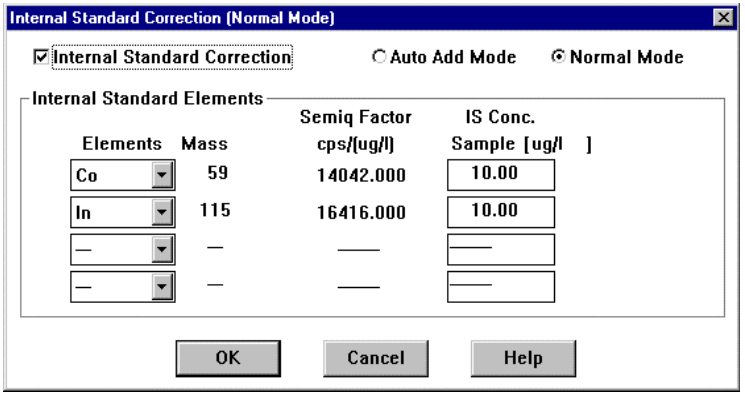
Conc. = (cps in sample) x (ISTD factor) x (dilution factor) / (SemiQuant factor)

where the dilution factor is obtained from the Start Run panel if running a method or in the Sample Log Table if running a sequence (default = 1.0); and the SemiQuant factor is obtained from the SemiQuant parameters panel.

To set up internal standard correction, perform the following steps:

1 **Select *SemiQuant>>Internal Standard Correction*.**

The ***Internal Standard Correction*** dialog box appears.



Internal Standard Correction Dialog Box

2 **Select the Normal Mode button on the top of the dialog box if it is not already selected.**

3 **Click the Internal Standard Correction check box on the top of the dialog box if it is not already checked.**

The Internal Standard Correction check box will be checked.

NOTE To set the Background File and Interference Correction, select ***Method >> Data Correction*** in the ICP-MS Data Analysis window.

4 Click the arrow under Element in the Internal Standard Elements box.

All elements are in the list.

5 Select the internal standard element from the list.

The mass to be analyzed will automatically be selected according to the element, and will be displayed under Mass.

The SemiQuant Factor of the each ISTD will be displayed under SemiQuant Factor.

NOTE The mass defined in the Internal Standard Correction and the SemiQuant parameters must be the same.

6 Select up to 4 elements for internal standards repeating steps 4 and 5.

You can specify up to four elements to be used as internal standards. If only one element is selected, all reported elements will be corrected using the single internal standard element specified. If more than one internal standard element is selected, the internal standard correction factor used for each element between any two adjacent internal standard elements is derived by interpolation of the linear response between those two internal standard elements.

Mass displays the mass of the specified element as set in the semiquant parameters panel.

7 Fill in the text boxes under "IS Conc" with the concentration of ISTD in the sample.

Enter the concentration of the internal standard element(s) in the sample. The units of concentration must be the same both for the sample and for the standard.

8 Click *OK*.

The internal standard correction will now be set and ChemStation returns to the ICP-MS Data Analysis window.

Subtracting the Blank Data File

ChemStation enables you to subtract a blank data file from the sample data file. This blank subtraction is performed after semi-quantitation is performed on both the blank data file and the sample data file (unlike background subtraction where the background counts are subtracted from the sample counts). Hence, the calculated concentration of a given element in the blank data file is subtracted from the calculated concentration of that same element in the unknown sample, resulting in a blank-subtracted concentration for the unknown.

Background correction is not ordinarily used in semi-quantitation. Its main purpose is to subtract a background spectrum from a sample spectrum so the user can view the net spectrum for qualitative purposes. Since background correction subtracts raw counts before normalizing for internal standard response, it could give incorrect results with internal standards.

To subtract the blank data file from your sample data files, complete the following steps:

1 **Select the *Blank Conc Subtraction* check box in the *SemiQuant Parameters* dialog box.**

2 **Click *Browse*.**

The ***Select Data File*** dialog box will appear.

3 **Select a blank data file.**

NOTE To set a background file and interference correction, select ***ICP-MS Data Analysis window >> Method >> Data Correction***.

4 **Click *OK*.**

The ***Select Data File*** dialog box will close and the blank concentration subtraction will be set. The selected blank file will be corrected in accordance with the settings of the background file and interference correction of the current method.

NOTE You can print out the blank data by selecting S***emiQuant >> Print Blank Conc***.

Generating a Semiquantitation Report

ChemStation generates a report showing the results of a semiquantitative analysis in either of two ways.

- You can tell ChemStation to generate the report automatically each time a method runs. When you do so, you specify whether to generate a summary or detailed report, and whether to send the report to the screen, the printer or a file. For information about how to generate a semiquantitation report automatically (using a method), see Chapter 5, "Creating a Method".
- You can create a custom report with the data acquired each time the method runs. Before you can create a custom report in this way, you must set up a custom report template. For more information about custom reports and databases, see Chapter 11, "Creating Custom Reports".

If you did not tell ChemStation to generate a report automatically, you can also generate a report manually by completing the following steps:

1 **Load a data file.**

2 **Select *SemiQuant>>Generate Report*.**

The ***SemiQuant Report Option*** dialog box will appear.

3 **Select the report style.**

The following report styles are available:

- Detailed, Text Only

  Gives the concentration, counts (cps), background counts, integration time and possible interference flag for each mass in the sample.

- Detailed

  Includes the same information as the Detailed, Text Only report, but which also includes the spectra generated for the sample at the bottom of the report. This report can only be sent to the printer.

4 Select the destination for the report.

You can click one or more of the following check boxes:

- Screen

  Generates the report to the ChemStation screen.

- Printer

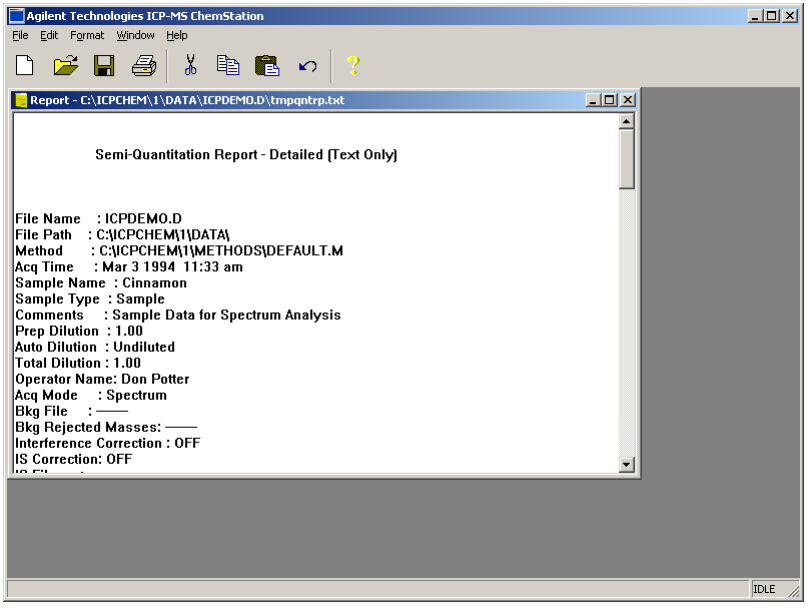
  Generates the report to the ChemStation printer. You must select this destination for a detailed report.

- File

  Generates the report to the file you indicate in the text box.

5 Click *OK*.

The report will be generated to the selected destination in the selected style.



Semi-Quantitation Report - Detailed (Text Only)

14

Performing an Isotope Ratio Analysis

Performing an Isotope Ratio Analysis

The Isotope Ratio Analysis function enables the operator to measure and quantitate isotope ratios using the Agilent 7500. It is mainly used for geological and nuclear applications such as determining the age and origin of geological samples, and measuring uranium enrichment.

This chapter explains how to set isotope ratio parameters and generate an isotope ratio report.

Setting Isotope Ratio Parameters

To generate a report for isotope ratio analysis, you must first set the parameters from the ICP-MS Data Analysis window. Setting isotope ratio parameters includes setting mass bias correction, selecting the element, and specifying the isotope ratio.

To set the isotope ratio parameters, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The ***ICP-MS Data Analysis*** window appears.

Load a data file by selecting ***Data File >> Load*** in the ICP-MS Data Analysis window.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

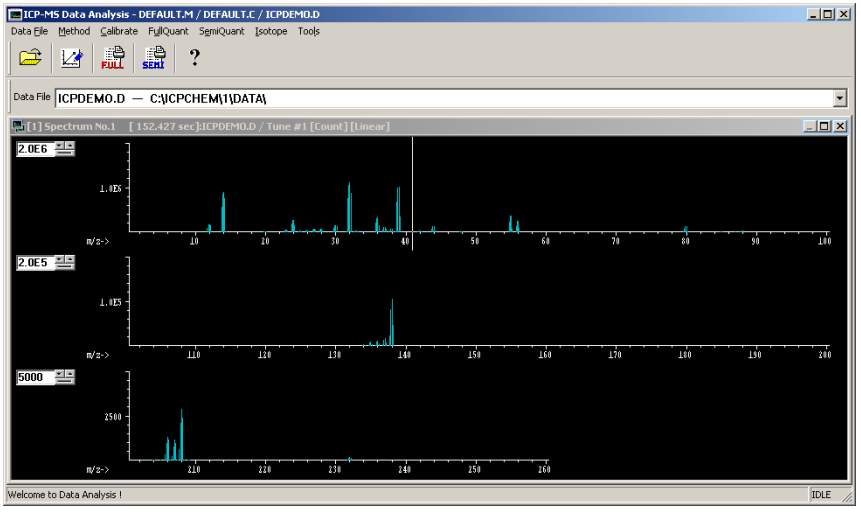
and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***Data File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

NOTE

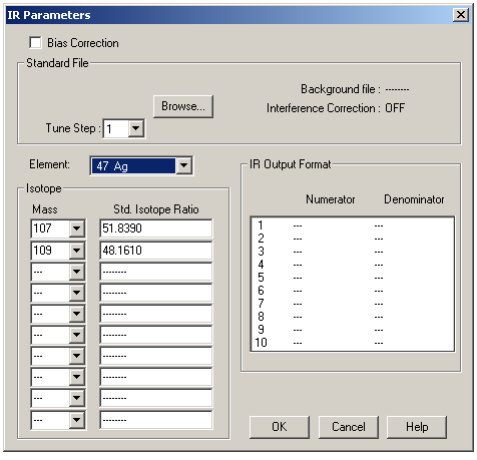
You can also work on the Offline Data Analysis. To do so, Double-click the ***Offline Data Analysis*** icon.



ICP-MS Data Analysis Window

2 Select *Isotope>>Edit IR Parameters*.

The ***IR Parameters*** dialog box appears.



IR Parameters Dialog Box

3 Click the *Bias Correction* check box to make mass bias correction.

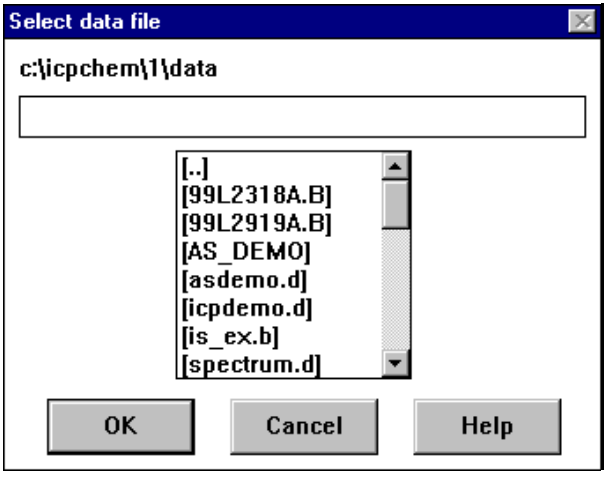
The Bias Correction check box will be checked.

NOTE

ICP-MS does not have the exact same sensitivity for all masses. Variations in mass response (mass bias) occur in the interface, ion lenses, analyzer and detection systems. Mass bias is more severe at low mass, where the percentage mass difference between adjacent masses is higher. In general, however, mass bias is relatively constant and easily corrected for by calculating the mass bias factor obtained from a certified isotopic standard. It is recommended that the mass bias factor be calculated using the same element that is to be measured.

4 Click the *Browse* push button.

The ***Select Data File*** dialog box appears.



Select Data File Dialog Box

5 Select the standard data file to be used for calculating the mass bias correction, using one of the following methods:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK.***

The dialog box will disappear and the selected standard data file will be indicated next to the ***Browse*** push button.

When data is acquired in the Multi Tune mode, select the tune step in ***Tune Step*** to use for the data analysis. When the Multi Tune mode is not used, select "1" for the tune step.

CAUTION 

Make sure the tune step used for data acquisition is the same as that selected here. Otherwise, an error may occur.

- Background File

  The name of the background file specified for count correction appears. The display is updated when you specify the standard file by clicking ***Browse***.

  Setting a background file allows the user to subtract background counts from any data file. This subtracts the raw counts before calculation; the abundance for each mass acquired in the background file is subtracted from the abundance of the corresponding mass in non-background file.

- Interference Correction

  Displays whether the interference correction specified for count correction is set to ON or OFF. The display is updated when you specify the standard file by clicking ***Browse***.

NOTE To set the Background File and Interference Correction, select ***Method >> Data Correction*** in the ICP-MS Data Analysis window.

6 Click on the arrow of the Element text box and select the element to analyze.

The element will be high lighted in the Element text box, and the relative isotopic abundance will be displayed in the left panel of the dialog box.

NOTE You can change the order in which the masses will be displayed if you wish. To do so, click on the arrow next to the Mass text box and select the required mass.

7 Enter the exact isotopic ratio for the standard.

ChemStation calculates the mass bias factor by comparing the obtained isotope ratio for the standard with the exact certified value. To enter the certified values, double-click or drag on the text box, and type in the value of each isotope ratio (in percent). To reset the ratio back to the natural isotopic abundance, re-select the element.

8 Fill in the IR Output Format panel.

To do so, complete the following steps:

a) Click on a cell under the Numerator.

The cell will be high-lighted and a Numerator text box with a drop down list and an ***Enter*** button appears. The drop down list contains all isotope masses of the selected element.

b) Select a mass to be the numerator of the isotope ratio, then click *Enter.*

The selected cell remains high-lighted with the selected mass number.

c) Click on a cell under the Denominator.

The cell will be high-lighted and a Denominator text box with a drop down list and an ***Enter*** button appears. The drop down list contains all isotope masses of the selected element and Total, which is the sum of all isotopes of that element.

d) Select a mass to be the denominator of the isotope ratio, then click *Enter.*

The selected cell remains high-lighted with the selected mass number.

NOTE

You can select up to 10 isotope ratios. Repeat the above steps, or press and hold the Control key to select multiple cells and input the same selection.

9 To accept all the settings, click *OK.*

The IR Parameters dialog box disappears and ChemStation returns to the ICP-MS Data Analysis window.

Generating an Isotope Ratio Analysis Report

You can tell ChemStation to generate the report automatically each time a method runs. When you do so, you specify whether to generate an Isotope Ratio Report or a Count Report (see below), and whether to send the report to the screen, the printer or a file. For information about how to generate an Isotope Ratio Analysis report automatically (using a method), see Chapter 5, "Creating a Method".

If you did not tell ChemStation to generate a report automatically, you can also generate a report manually.

To do so, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The Data Analysis menu appears.

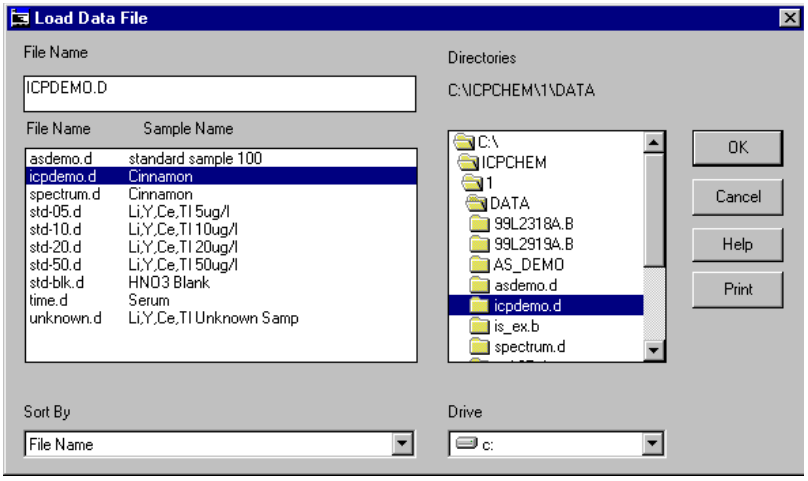
2 **Select *Data Analysis>>Main Panel*.**

The Data Analysis window appears.

NOTE You can also work on the Offline Data Analysis by double-clicking on the ***Offline Data Analysis*** icon.

3 **Select *Data File>>Load*.**

The ***Load Data File*** dialog box appears.



Load Data File Dialog Box

- Sort By

  Allows the user to sort the list of data files in the current directory by the selected type of information. Options include File Name, Sample Name, Misc Info, and Acquired Date. The File Name Only option allows the user to display a listing of file names only, without sample name or any other additional information.

4 Select a data file using one of the following methods:

- Click a file in the displayed list and click ***OK***.
- Double-click a file in the displayed list.
- Type the file name and click ***OK***.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***Data File*** menu in ***Data Analysis***).

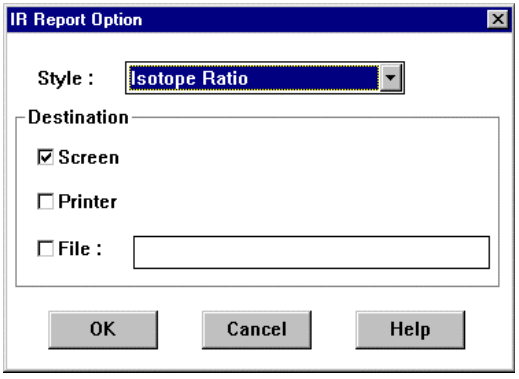
This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

ChemStation loads the data file and returns to the Data Analysis window.

5 Select *Isotope>>Generate IR Report*.

The ***IR Report Option*** dialog box will appear.



IR Report Option Dialog Box

6 Select the report style.

The following report styles are available:

- **Isotope Ratio Report** includes header information of the analysis, specified isotope ratios for each repetition, average ratios, standard deviation (SD), relative standard deviation (RSD), and mass bias coefficient.
- **Count Report** includes header information of the analysis, counts for each selected mass (numerator) for each repetition, average counts of the repetitions, standard deviation (SD), and relative standard deviation (RSD).

7 Select the destination for the report.

You can click one or more of the following check boxes:

- Screen

  Generates the report and displays it on the ChemStation screen.

- Printer

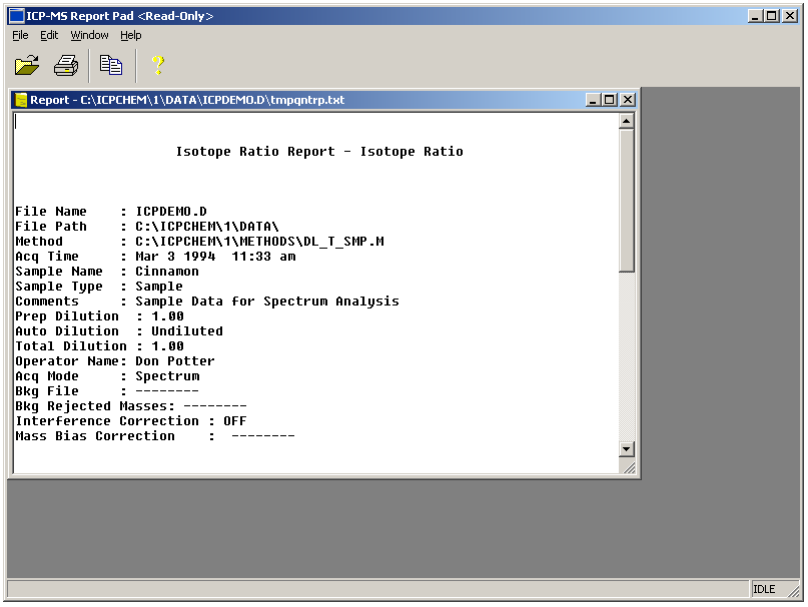
  Sends the report to the ChemStation printer.

- File

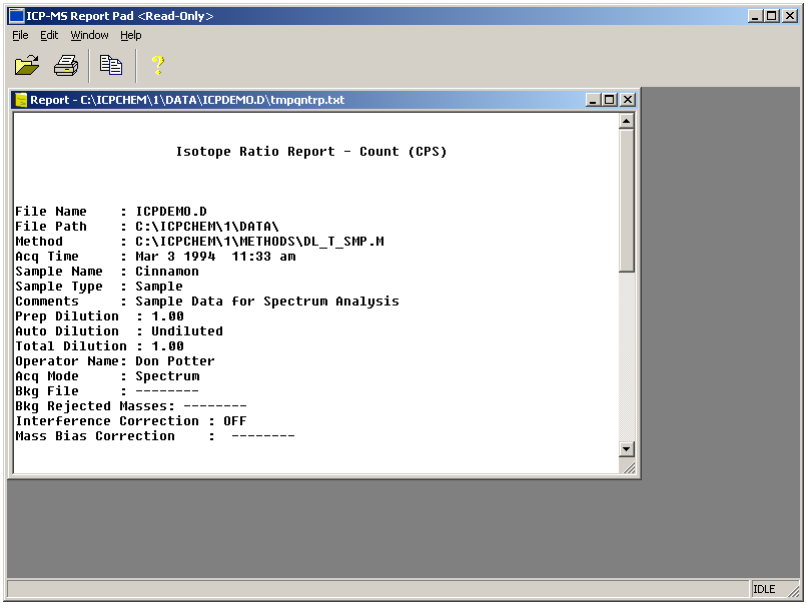
  Generates the report and saves it as a file. Use the dialog box to give this file a name.

8 Click *OK*.

The report will be sent to the selected destination in the selected style.



Isotope Ratio Report - Isotope Ratio



Isotope Ratio Report - Count (CPS)

15

Performing an Isotope Dilution Analysis

Performing an Isotope Dilution Analysis

Isotope dilution (ID) is an analytical technique used when the highest accuracy is demanded. Typical applications include the certification of standards and reference materials. ID is highly accurate since it uses known isotopic ratios to quantify analyte concentration. Since isotope ratios can be measured with much greater precision than elemental concentrations (~0.2% vs. 5%), this translates to greater accuracy in ID determinations.

To be able to use ID to measure a given element, the element must possess 2 interference free isotopes, and a non-natural elemental spike must be readily available. A known amount of an elemental spike is added to a known amount of the sample. The chosen isotope ratio is measured. Since the spike isotope ratio, spike amount, and sample amount are known, the original analyte concentration can be calculated. The ChemStation ID software automatically calculates the analyte concentration after the required data is entered.

Data files to be used for isotope dilution analysis should be acquired in the isotope ratio acquisition mode.

This chapter explains how to set isotope dilution analysis parameters and generate the isotope dilution analysis report.

Setting Isotopic Dilution Analysis Parameters

To perform isotope dilution analysis, you must first enter the ID parameters via the ICP-MS Data Analysis window. Isotope dilution analysis parameters to be set include mass bias correction and spike information.

To enter the isotope dilution analysis parameters, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The ***ICP-MS Data Analysis*** window appears.

Load a data file by selecting ***Data File>>Load*** in the ICP-MS Data Analysis window.

Selecting ***Data File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

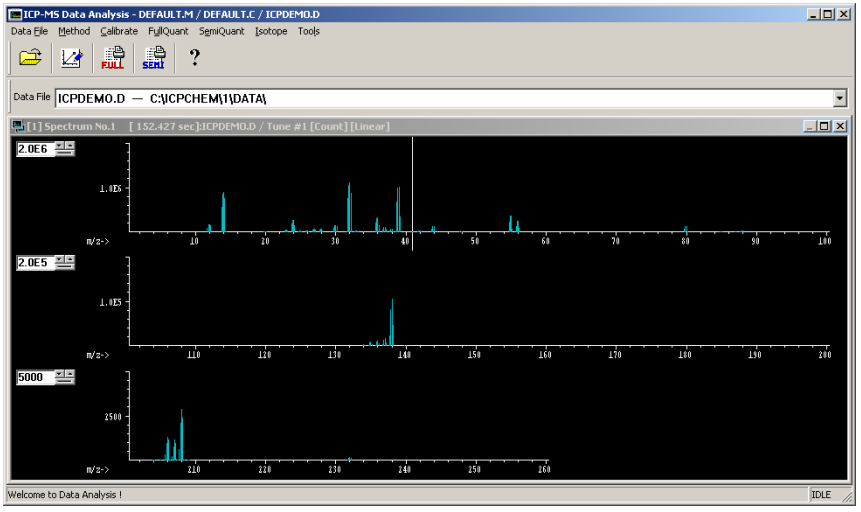
and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***Data File*** menu in ***Data Analysis***).

This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

NOTE

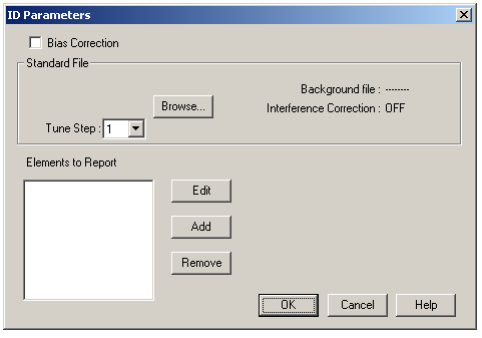
You can also work on the Offline Data Analysis. To do so, Double-click the ***Offline Data Analysis*** icon.



ICP-MS Data Analysis Window

2 Select *Isotope>>Edit ID Parameters*.

The ***ID Parameters*** dialog box appears.



ID Parameters Dialog Box

3 Click the *Bias Correction* check box to make mass bias correction.

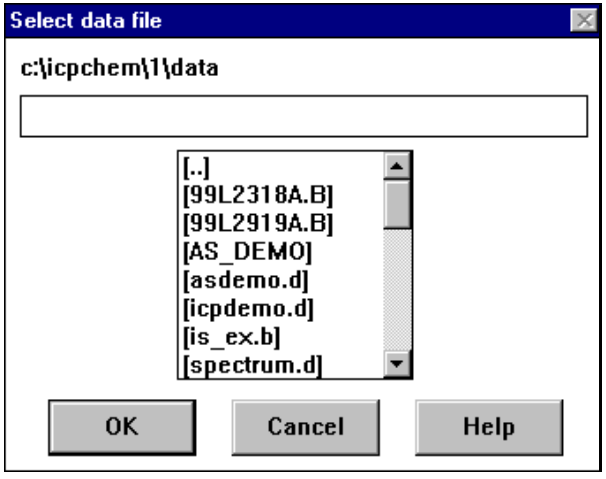
The Bias Correction check box will be checked.

NOTE

ICP-MS does not have the exact same sensitivity for all masses. Variations in mass response (mass bias) occur in the interface, ion lenses, analyzer and detection systems. Mass bias is more severe at low mass, where the percentage mass difference between adjacent masses is higher. In general, however, mass bias is relatively constant and easily corrected for by calculating the mass bias factor obtained from a certified isotopic standard.

4 Click the *Browse* push button.

The ***Select Data File*** dialog box appears.



Select Data File Dialog Box

5 Select the standard data file to be used for calculating the mass bias factor, using one of the following methods:

- Click a file name in the displayed list and click ***OK***.
- Double-click a file name in the displayed list.
- Type the file name and click ***OK***.

The dialog box will disappear and the selected standard data file will be indicated next to the Browse push button.

When data is acquired in the Multi Tune mode, select the tune step in ***Tune Step*** to use for the data analysis. When the Multi Tune mode is not used, select "1" for the tune step.

CAUTION 

Make sure the tune step used for data acquisition is the same as that selected here. Otherwise, an error may occur.

- Background File

  The name of the background file specified for count correction appears. The display is updated when you specify the standard file by clicking ***Browse***.

  Setting a background file allows the user to subtract background counts from any data file. This subtracts the raw counts before calculation; the abundance for each mass acquired in the background file is subtracted from the abundance of the corresponding mass in non-background file.

- Interference Correction

  Displays whether the interference correction specified for count correction is set to ON or OFF. The display is updated when you specify the standard file by clicking ***Browse***.

NOTE To set the Background File and Interference Correction, select ***Method >> Data Correction*** in the ICP-MS Data Analysis window.

6 Fill in the ID Parameters panel.

- Edit

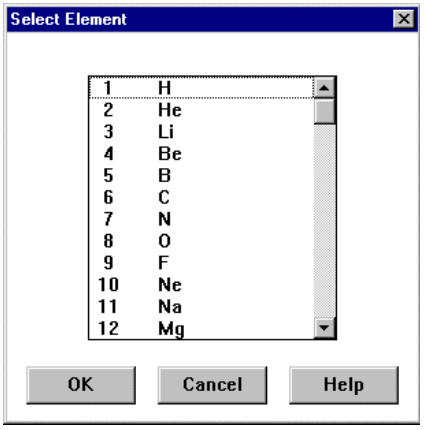
  Press this button to change the parameters for the elements already selected for output. When the ***Edit*** button is pressed, the ***Edit Element*** dialog box appears.

- Add

  Press this button to specify a new element to be analyzed with the Isotope Dilution method. When the ***Add*** button is pressed, the ***Select Element*** dialog box appears.

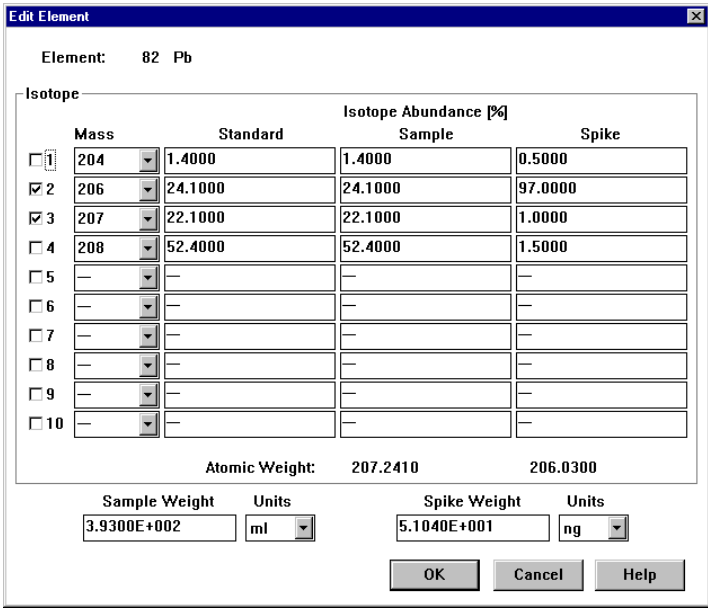
- Remove

  Press this button to cancel the elements already selected for output. Select an element from the display and press ***Remove***.



Select Element Dialog Box

When an element is selected, the ***Edit Element*** dialog box appears.



Edit Element Dialog Box

The ***Edit Element*** dialog box displays the masses and isotope abundances of the element selected in the Select Element dialog box. To output the results of the Isotope Dilution analysis, enter the following items.

- Check Box

  Select two isotopes.

- Spike

  Enter the certified isotope ratio of the added spike sample in each cell of the mass column. The atomic weight of the spike is automatically calculated and displayed at the bottom. Ensure that the total of the spike isotope abundance values adds up to exactly 100.

- Sample Weight

  Enter the mass or volume of the sample.

- Units

  Select the sample concentration units (mass or volume).

- Spike Weight

  Enter the exact mass of the spike.

- Units

  Select the spike concentration units.

- Standard

  The isotopic abundance of the standard should be edited to display the certified abundances of the isotopes contained in the standard used for mass bias correction.

7 To accept all the settings, click *OK*.

The ID Parameters dialog box disappears and ChemStation returns to the ICP-MS Data Analysis window.

Generating an Isotope Dilution Analysis Report

You can tell ChemStation to generate the report automatically each time a method runs. When you do so, you specify whether to generate an isotope dilution analysis report, and whether to send the report to the screen, the printer or a file. For information about how to generate an Isotope Dilution Analysis report automatically (using a method), see Chapter 5, "Creating a Method".

If you did not tell ChemStation to generate a report automatically, you can also generate a report manually.

To do so, complete the following steps:

1 **Select *Top>>Data Analysis*.**

The Data Analysis menu appears.

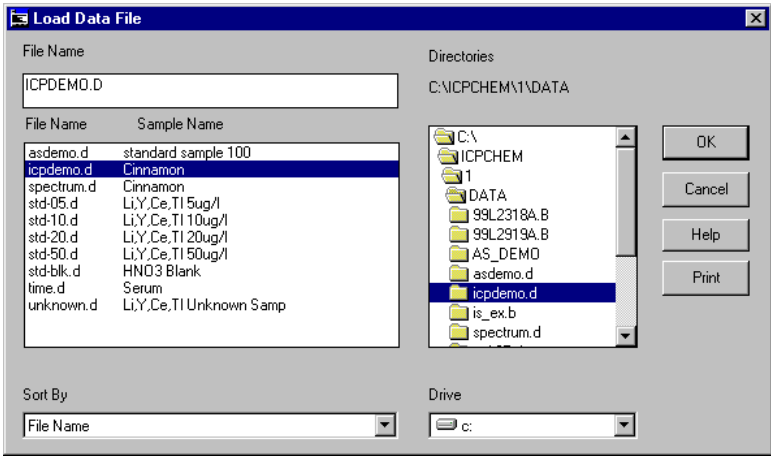
2 **Select *Data Analysis>>Main Panel*.**

The Data Analysis window appears.

NOTE You can also work on the ***Offline Data Analysis*** by double-clicking on the ***Offline Data Analysis*** icon.

3 **Select *Data File>>Load.***

The ***Load Data File*** dialog box appears.



Load Data File Dialog Box

- Sort By

  Allows the user to sort the list of data files in the current directory by the selected type of information. Options include File Name, Sample Name, Misc Info, and Acquired Date. The File Name Only option allows the user to display a listing of file names only, without sample name or any other additional information.

4 Select a data file using one of the following methods:

- Click a file in the displayed list and click ***OK***.
- Double-click a file in the displayed list.
- Type the file name and click ***OK***.

Selecting ***File>>Next Data File*** will load the data file that alphabetically follows the current data file in the same directory. For example, if the directory contains the following files:

Soil01.d
Soil02.d
Soil03.d
Water01.d
Water02.d
Water03.d

and soil03.d is currently displayed, selecting ***Next Data File*** will load water01.d. If the current data file is the last one in the directory (in this example water03.d) the message ***No Next File*** is displayed. To initiate this feature, the user must have first selected a data file from the ***Load Data File*** dialog box (from the ***File*** menu in ***Data Analysis***).

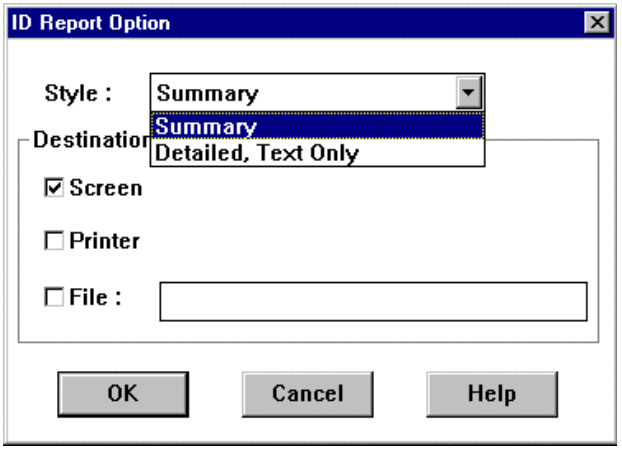
This menu item is not related to the numbered items near the bottom of the menu, which display the most recently loaded data files.

The three most recently loaded data files are listed as numbered menu items, with the first entry corresponding to the most recently loaded data file. Selecting any one of these numbered menu items will cause that file to be loaded into data analysis.

ChemStation loads the data file and returns to the Data Analysis window.

5 Select *Isotope>>Generate ID Report.*

The ***ID Report Option*** dialog box will appear.



ID Report Option Dialog Box

6 Select the report style.

The following report styles are available:

- Summary

  Outputs the analyzed value and count generated with the Isotope Dilution method.

- Detailed, Text Only

  Reports parameters used in the Isotope Dilution method in addition to the analyte value and counts.

7 Select the destination for the report.

You can click one or more of the following check boxes:

- Screen

  Generates the report and displays it on the ChemStation screen.

- Printer

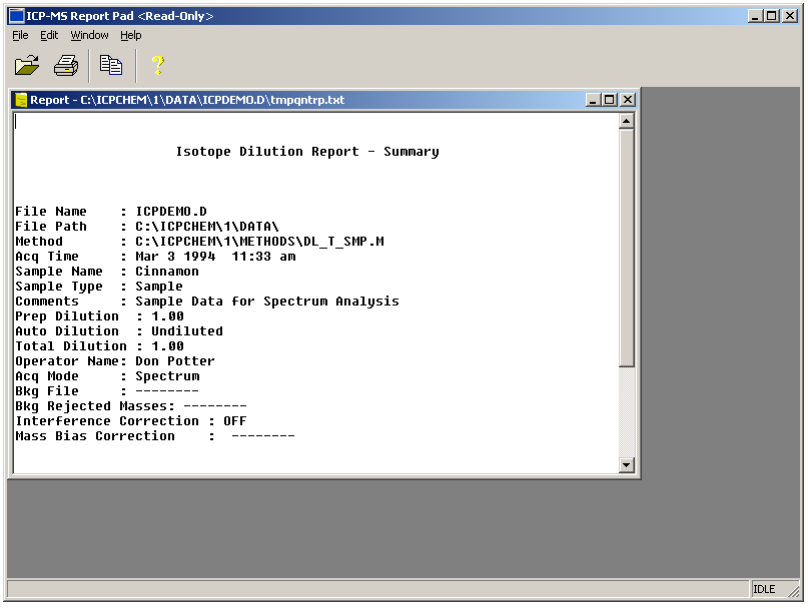
  Sends the report to the ChemStation printer. You must select this destination for a detailed report.

- File

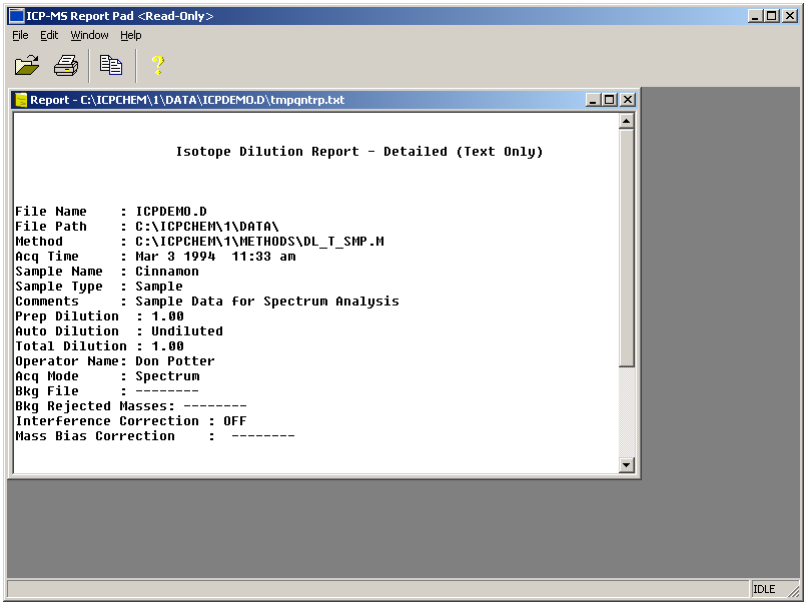
  Generates the report and saves it as a file. Use the dialog box to give this file a name.

8 Click *OK*.

The report will be sent to the selected destination in the selected style.



Isotope Dilution Report - Summary



Isotope Dilution Report - Detailed (Text Only)

16

Tools Menu

Tools Menu

ChemStation provides the tool to generate multiple types of reports for multiple data files continuously. ChemStation also enables you to select report types and destinations for the data files you want to report. It is convenient to generate reports for many data files at once, after you have finished acquiring all data.

DoList

DoList allows you to generate the reports you want to the destination of your choice for multiple data files. There are many available report options; these include report type and destination, printing custom reports and updating custom databases, and running user-specified macros. Report options can be selected in ***Configure DoList*** dialog box. Calibration information used in generating the reports will come from the current method, but the report settings in DoList selection take precedence over the report settings in the method.

How to Use DoList?

To select report options that are already available in the DoList, complete the following steps:

1 **Select *Top>>Data Analysis*.**

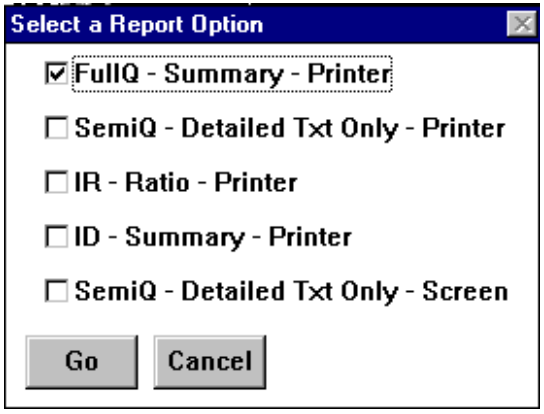
The Data Analysis menu appears.

2 **Select *Data Analysis>>Main Panel*.**

The ***ICP-MS Data Analysis*** window appears, displaying the last data file.

3 **Select *Tools>>DoList***

The ***Select a Report Option*** dialog box appears.



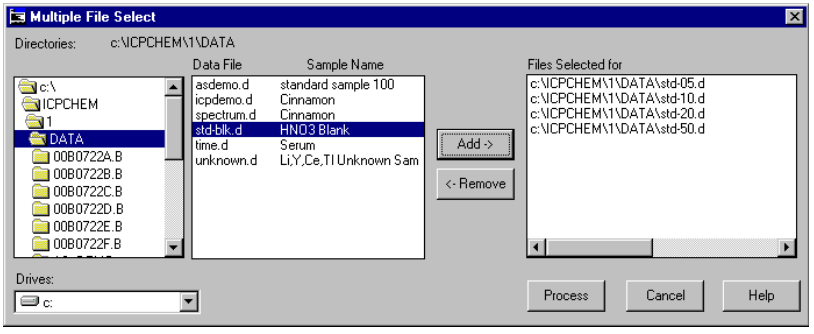
Select a Report Option Dialog Box

4 **Select the report options you want to generate.**

Select at least one report option. If you do not select a report option, this dialog box closes as you click ***Go***, and no report is generated.

5 **Click *Go* after you have made your selection(s).**

The ***Multiple File Select*** dialog box will then appear, allowing selection of one or more data files for processing.



Multiple File Select Dialog Box

6 Move the data files to the right list box by selecting the data files and then clicking *Add*.

- If you wish to delete data files from the right list box, click *Remove* after you select the files in the right list box. You can also add and delete by double-clicking.List Box (Middle)

  Select the data files which are in the current directory appearing in the list box (left). Selected files appear in the right list box (right).

- List Box (Right)

  The files selected from the list box (middle) are displayed. To remove the files, select them in this list box.

- Add

  Select the file from the list box (middle) and click this button to add it to the list box (right).

- Remove

  Select the file you want to delete from the list box (right) and click this button to remove it from the display.

NOTE A series of files can be selected by clicking and dragging to highlight a list of data file names. Alternatively, the shift key can be used to allow selecting a consecutive series of data files, whereas the control key can be used to allow selecting a non-consecutive series of data files.

- Process

  Click this button to generate reports. ChemStation begins to generate reports according to your selection.

- Cancel

  Cancels displaying the files selected for the multiple report and generating the reports.

7 Click *Process* to generate reports.

ChemStation begins to generate reports according to your selection.

Configure DoList

Configure DoList enables you to select the report options for DoList. There are many available report options including report type and destination, printing custom reports and updating custom databases, and running user-specified macros.

NOTE The DoList configuration is not saved to the method. Rather, it is saved in the file \icpchem\icpexe\qlistout.ini. This file is overwritten every time the DoList configuration is changed.

How to Configure DoList?

To configure DoList, complete the following steps:

1 **Select *Top>>Data Analysis*.**

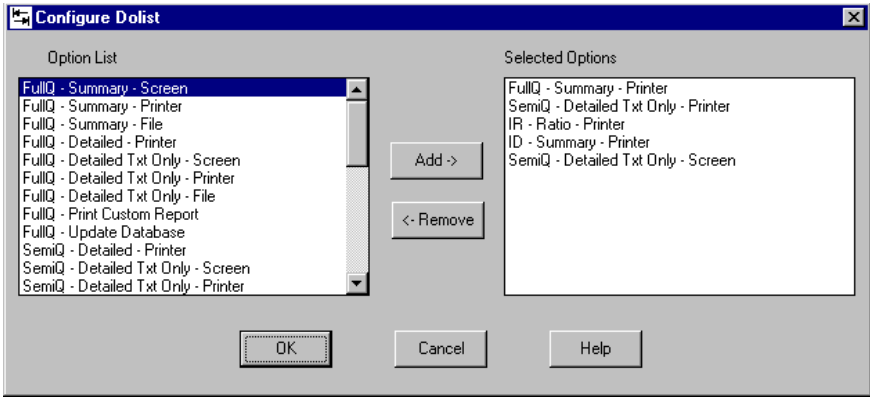
The Data Analysis menu appears.

2 **Select *Data Analysis>>Main Panel*.**

The ***ICP-MS Data Analysis*** window appears, displaying the last data file.

3 **Select *Tools >> Configure DoList.***

The ***Configure DoList*** dialog box appears.



Configure DoList Dialog Box

The available report options are displayed in the Options List. These options include report type and destination, as well as the ability to generate custom reports, update custom databases, and run user-specified macros.

4 Select the report options to include in the Selected Options list by clicking on an option in the Options List, then clicking *Add.*

Up to six options can be selected for inclusion in the Selected Options list. If you want to remove any of the options from the Selected Options list, select the option to be removed then click ***Remove***. You can also double-click the appropriate option to add or delete. Options can be selected only one at a time.

- Option List Box

  Select the report options appearing in this list box. Selected options appear in the Selected Options list box.

- Selected Options List Box

  The report options selected from the Option List box are displayed. To remove the options, select them in this list box. You can select 1- 6 types of option.

- Add

  Select the report options from the Option List box and click this button to add them to the Selected Options list box.

- Remove

  Select the options you want to delete from the Selected Options list box and click this button to remove them from the display.

- OK

  End setting and close the dialog box.

- Cancel

  Cancels setting the configuration DoList.

5 After you finish selecting the report options you desire, click *OK*.

The options selected will be the options that are subsequently displayed in the Select a Report Option panel that appears after selecting DoList from the Tools menu.

17

DataBase Editor

DataBase Editor

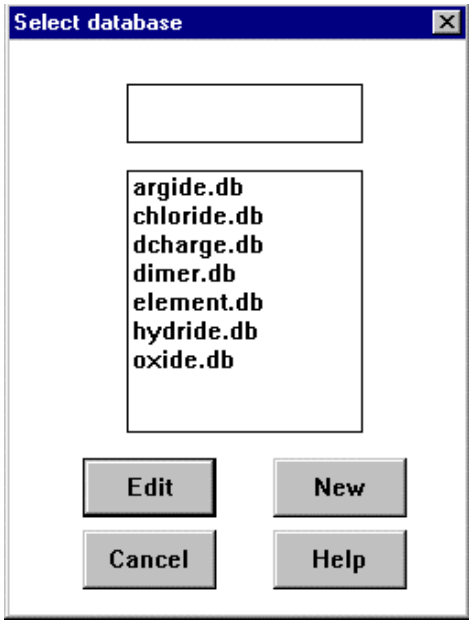
This chapter explains how to edit the ICP-MS databases which are supplied with the ChemStation.

Starting the DataBase

Click ***Start*** button on the Windows Task bar and select ***Program>>ICP-MS ChemStation>>DataBase Editor***.

Select Database

When the database editor is started, the ***Select database*** dialog box appears. Several databases are displayed.



Select database Dialog Box

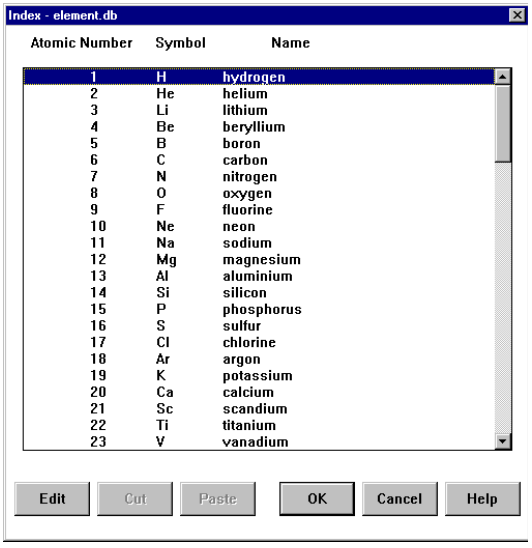
- Edit

  Press this button to edit an existing database. Select a database and press ***Edit.*** The ***Index*** dialog box appears.

- New

  Press this button to create a new database.

Editing the Database



Index Dialog Box

- Edit

  Pressing ***Edit*** after selecting an element or polyatomic molecule brings up the Molecular Database Editor. The atomic numbers of items that have been edited, cut, or pasted are preceded by an asterisk (*).

- Cut

  Press this button to cut information on the selected element or polyatomic molecule and temporarily store it in the internal buffer. When element.db is selected, "Cut" is disabled to prevent accidental damage to the elemental database.

- Paste

  Press this button to paste the information cut with ***Cut*** to the selected atomic number. When element.db is selected, "Paste" is disabled.

- OK

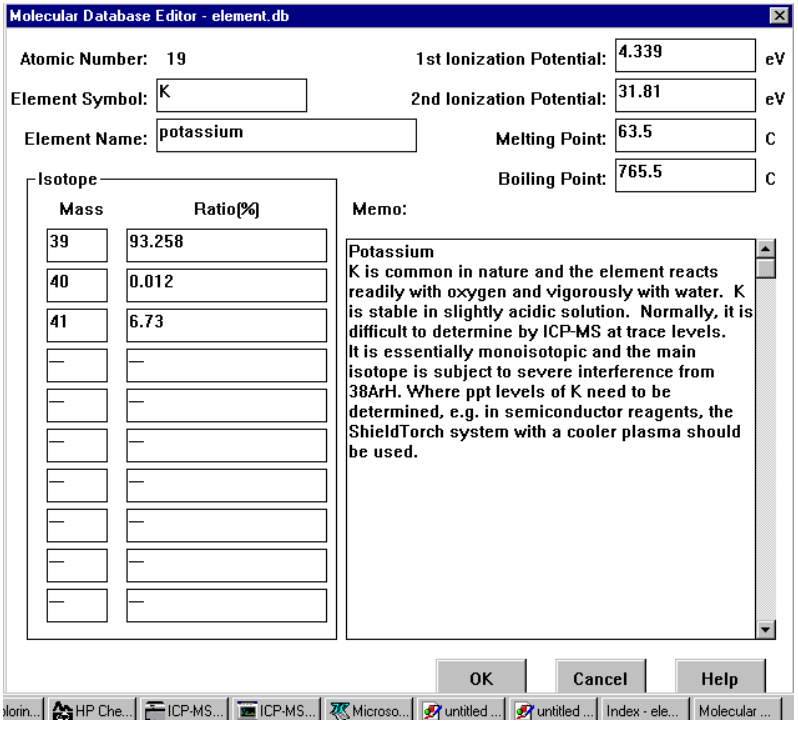
  Displays the ***Save Database*** dialog box.

- Cancel

  Cancels editing the database.

Using the Molecular Database Editor

You can enter or change information such as description of elements or polyatomic molecules in various databases, using the ***Molecular Database Editor***.



Molecular Database Editor

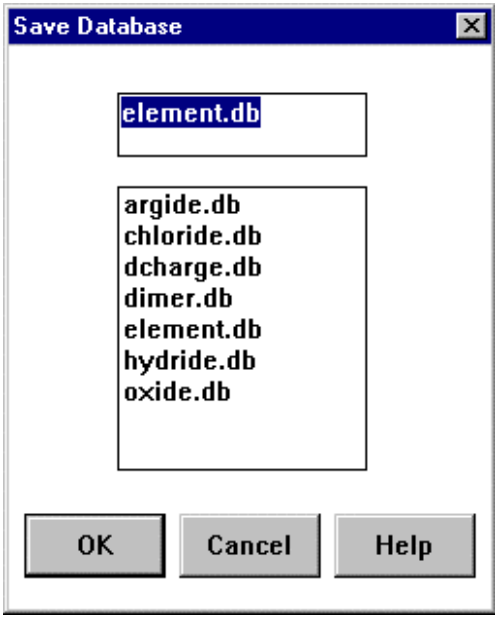
- OK

  Sets the contents displayed on the screen. After clicking ***OK***, the ***Save Database*** dialog box appears.

- Cancel

  Cancels editing the element/polyatomic molecule database.

Save Database



Save Database Dialog Box

- OK

  Select an existing database from the list or enter a new database name and click ***OK*** to update the database.

- Cancel

  Cancels saving the database.

18

Installing the Agilent 7500 ChemStation Software (Windows XP)

Installing the Agilent 7500 ChemStation Software (Windows XP)

This section explains how to set up the computer and install the ChemStation software G1834B (Rev. B.02.00 or later) for Windows XP.

WARNING  **Many computers have voltage selection switches, check for the correct setting according to your local supply before inserting the power cord.**

NOTE Confirm the instrument is in shutdown mode before installing ChemStation. If instrument is in standby mode, Put the instrument into Shutdown mode via the ChemStation before installing ChemStation because it is necessary to turn the instrument power on to download the firmware after installing the ChemStation.

CAUTION  The setup procedure in this section is a typical example. The procedure depends on the computer and printer.

Windows XP Configuration

NOTE If the O/S is Windows 2000 / XP selectable install Windows XP.

NOTE The partition for the ChemStation should be NTFS format.

Windows XP Configuration

1 Turn on the computer and check the following BIOS settings.

- Power setting: Disabled (for example: Auto Suspend Timeout, Hard Drive Timeout, Modem Ring)
- Plug and Play: Enable

NOTE All items will not always appear.

NOTE Refer to the computer's manual for the BIOS setting.

Following is a typical example.

Select F2 (F8 for VL420, F10 for Evo D510) during computer initialization, check that the following settings are true. BIOS settings need not be changed for the D530.

Table 18-1 BIOS Settings for Vectra VL400

Items	Setting
Power >> Auto Suspend Timeout	Disabled
Power >> Modem Ring	Disabled
Advanced > Plug & Play O/S	Yes

Table 18-2 BIOS Settings for Vectra VL420

Items	Setting
Main >> PnP OS	YES

Table 18-3 BIOS Settings for Evo D510

Items	Setting
Power >> Hard Drive Timeout	Never
Power >> System Timeout	Never

Exit and save the settings.

2 Start Windows XP.

The first time Windows XP is started, the setup wizard appears. Follow the instructions.

When you have completed the wizard steps, the computer will automatically restart.

3 Logon as Administrator.

User name: Administrator
Password: 3000hanover or blank
(Depending on the computer)

4 Setting display

Double-click ***Display*** in the Control Panel.

Select the ***Theme*** tab and confirm the following settings:

Theme	Windows XP

Select the ***Screen Saver*** tab and confirm the following settings:

Screen Saver	None

Select the ***Power*** button and confirm the following settings.

Turn off monitor	Never
Turn off hard disks	Never
System standby	Never

Select the ***Settings*** tab in Display Properties and confirm the following settings:

Screen resolution	1280 by 1024 pixels
Color quality	Medium (16 bit)

Select the ***Advanced*** button in the Setting tab, and confirm the following settings.

Screen refresh rate	75 Hertz

* If 75 Hertz does not work correctly, select OS default setting.

5 Setting taskbar

Double-click ***Taskbar and Start Menu*** in the Control Panel.

Select the ***Start Menu*** tab, and select the Start Menu.

Click OK.

6 Folder Options

Right-click **Start** and open **Explore**.

Select ***Tools>>Folder Options***.

Select the ***View*** tab and confirm the following settings:

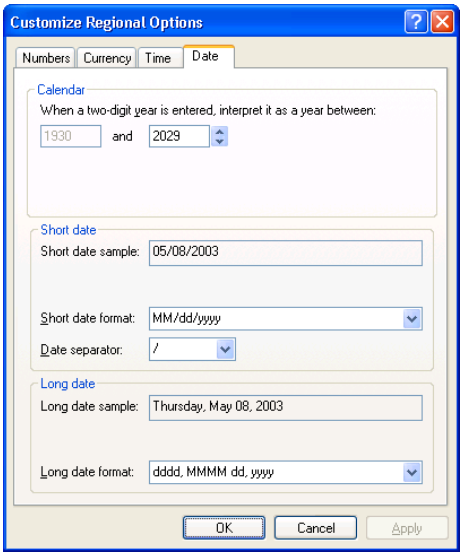
Select	Do not show hidden files and folders
Set ON	Hide file extensions for known file types
Set ON	Hyde protected operating system files
Set OFF	Use simple file sharing

7 Format Day/Time

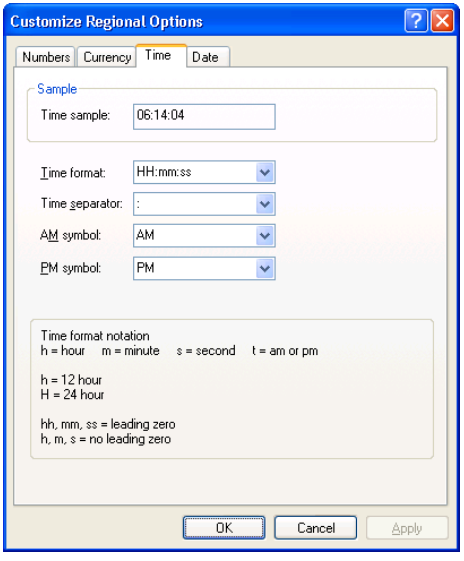
Double click the ***Regional and Language Options*** icon in the Control Panel.

Click the ***Customize*** button in the Regional and Language Options.

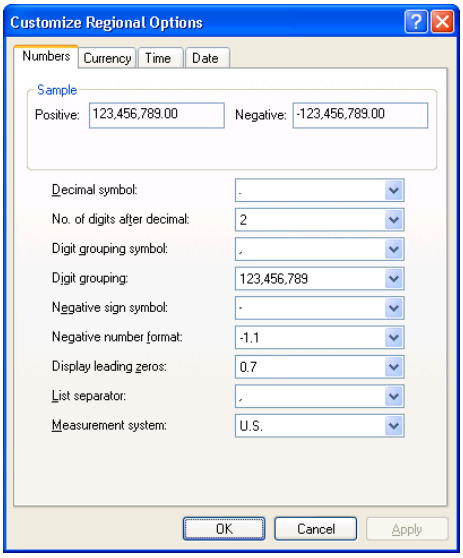
Select the ***Date*** tab and select ***MM/dd/yyyy*** in the Short Date Style box.



Select the ***Time*** tab and select ***HH:mm:ss***.



Click on the ***Number*** tab and Select "***.*** ***(Dot)*** in the Decimal Symbol box.



Click ***OK***.

8 Logon to Support, Chemist, and User. And set the same setting of step 4 through step 7.

9 Internet Protocol (TCP / IP) Properties

1 Logon as Administrator.

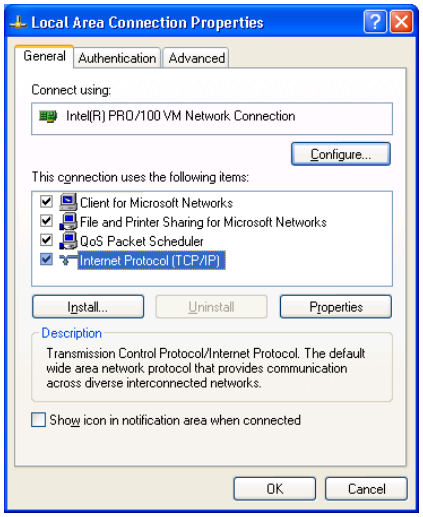
2 Double click ***Network Connections*** icon in the Control Panel.

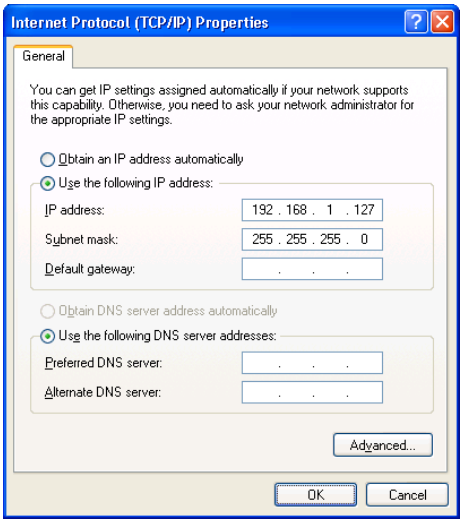
3 Right click ***Local Area Connection,*** select ***Properties***.

4 Select ***Internet Protocol (TCP / IP),*** select ***Properties.***

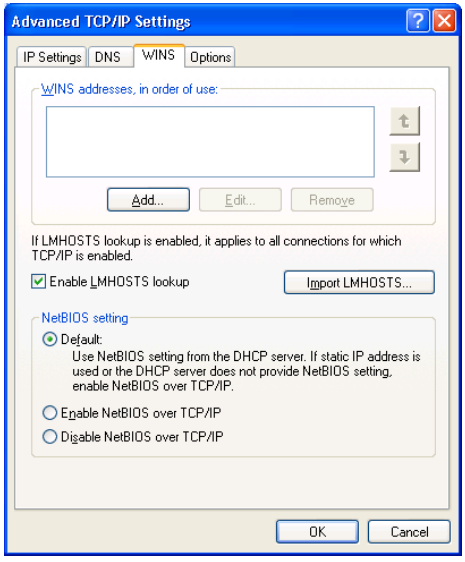
5 Input the following information for a simple direct connection.

IP address : 192.168.1.127
Subnet mask : 255.255.255.0





6 Click ***Advanced*** button, select ***WINS*** tab, then select ON the ***Enable LMHOSTS lookup***.



7 Click ***OK***.

NOTE

If you connect the instrument to the local network, you will need to talk with the network administrator. You may want to call your local ICP-MS tech support team for additional assistance.
To connect to site LAN, you are expected to understand the Windows XP operating system and TCP/IP networking, such as host name, IP address, subnet mask settings. You should also be familiar with the particular hardware devices and configurations of your organization's local area network.
Agilent Technologies is not responsible for any software problems, LAN configuration conflicts or performance problems that may result when a system is connected to a non-isolated LAN.

Creating User Account

Creating User Account as follows:

User Name	Description	Password	Password property	Group
Support	Product Support	HPCE	Select ***Password never expires*** check box only. Do not select other check boxes.	Administrators
Chemist	ChemStation Chemist	hp	Select ***Password never expires*** check box only. Do not select other check boxes.	Power Users
User	ChemStation User		Select ***Password never expires*** check box only. Do not select other check boxes.	Users
Administrator	(keep the displayed description.)	3000hanover	Select ***Password never expires*** check box only. Do not select other check boxes.	Administrators

To create or modify the user account, complete the following steps;

1 **Logon as Administrator.**

2 **Select *Administrative Tools>>Computer Management* in the Control Panel.**

3 **Select *Local Users and Groups>>Users* in the left area of the Computer Management.**

4 **Right-click the user name, then select *Set Password* and set the password for each user name.**

NOTE To create a new user account, select ***Action>>New User*** menu in the Computer Management.

5 **Right-click the user name, then select *Set Properties.***

6 **Enter the Description and set the password properties.**

7 **Select *Member Of* tab, then click *Add* button.**

8 **Click *Advanced* button in Select Groups.**

9 **Click *Find Now* button, then select the group name and click *OK*.**

10 **Close the *Computer Management* after setting the users.**

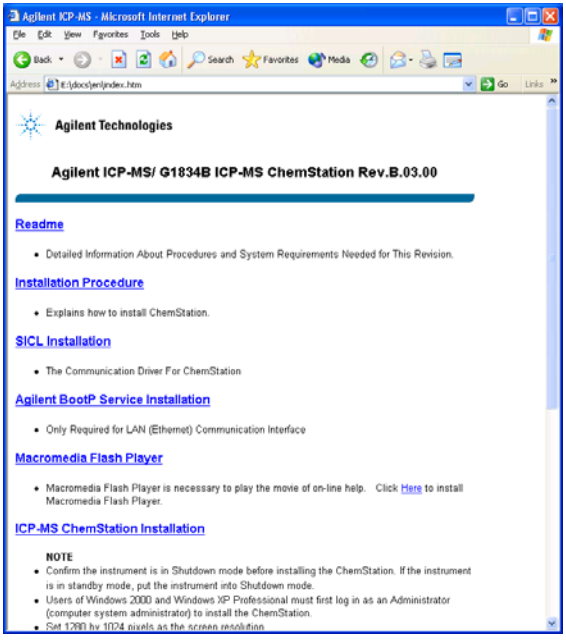
NOTE When create a new user, set the same setting of section 4 through section 7.

CAUTION  The local user account in this section is a typical example. The information contained in this section is subject to change without notice.

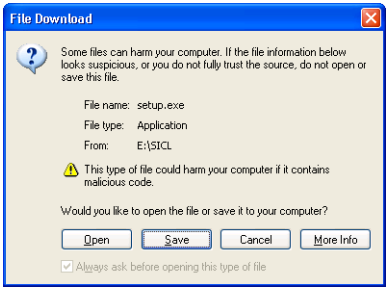
I/O Library Setup

1 Insert Agilent ICP-MS ChemStation System disk into CD-ROM Drive.

Internet Explorer automatically appears. Click ***SICL Installation***.

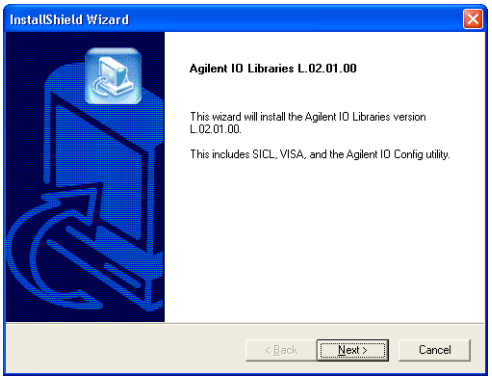


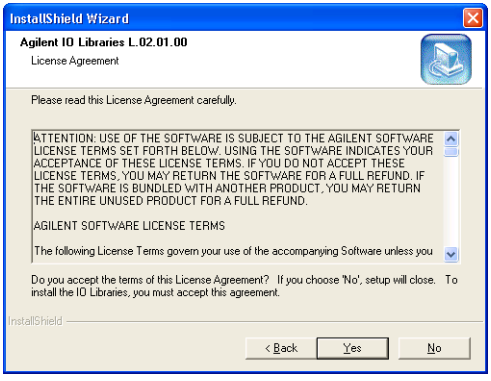
2 Click *Open*.

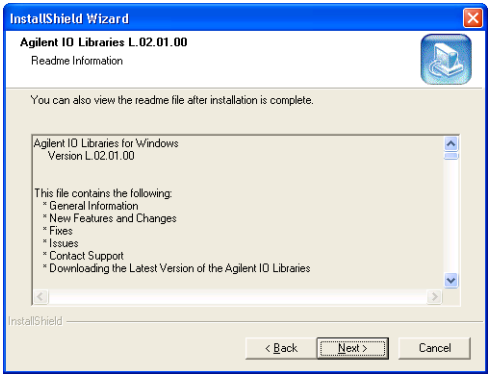


3 InstallShield Wizard

Click ***Next*** or ***Yes***

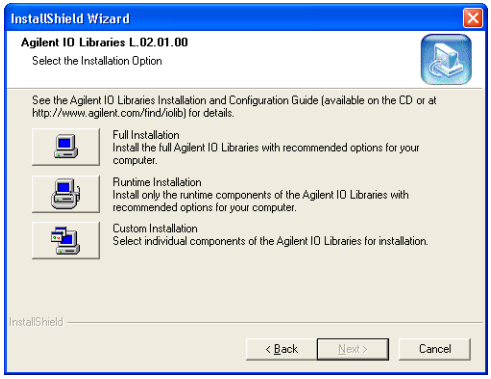






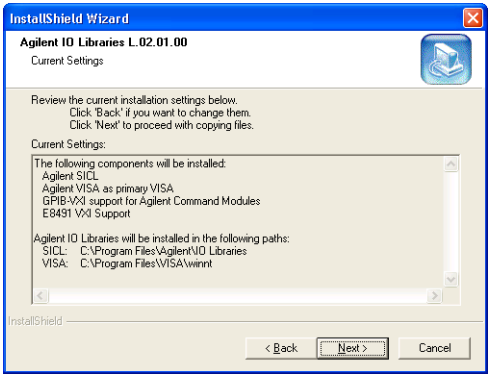
4 Select Installation Option

Select ***Full Installation***. Then Click ***Next***



5 Current Setting

Click ***Next***

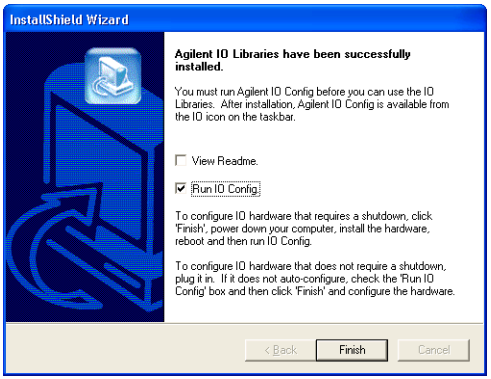


Installation status is shown. Wait until installation has completed.

6 Installation Completion

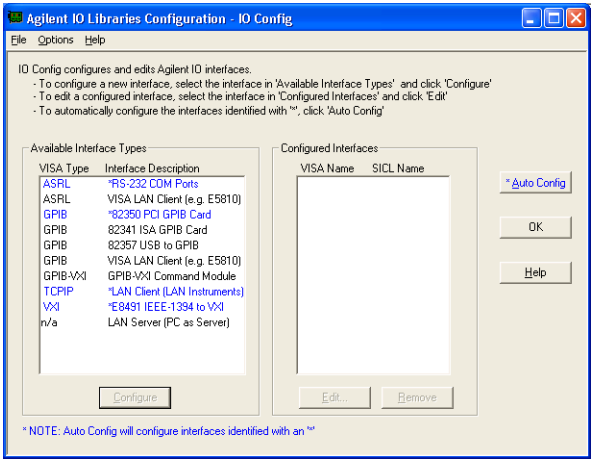
When the installation is complete, the following dialog box will appear.

Select [**RUN IO Config**]. And Click ***Finish***.

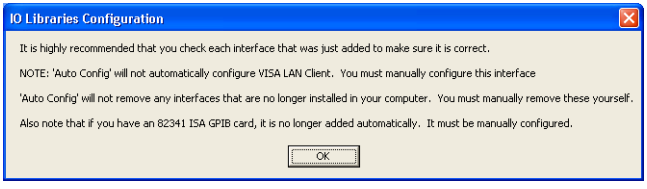


7 IO Config

Following dialog box will appear. Click [**Auto Config**].



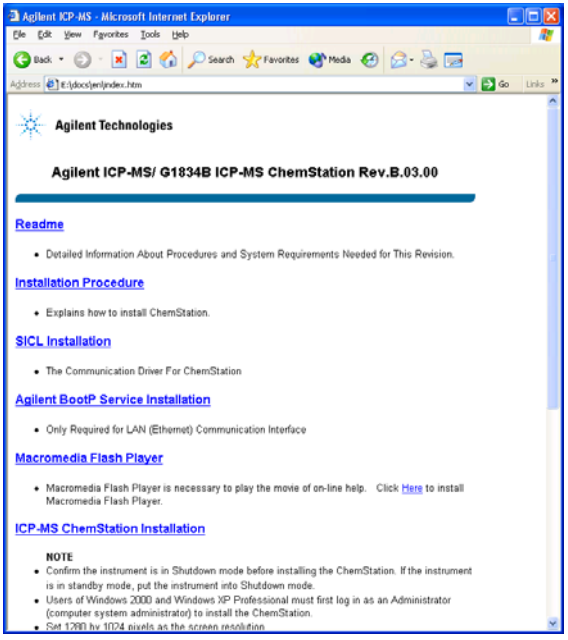
Following message will appear. Click ***OK***.



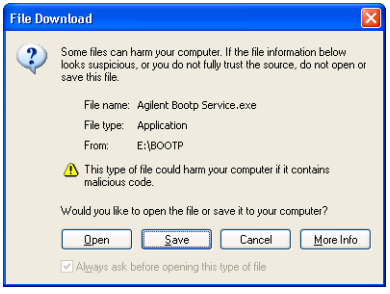
BootP Service Setup

When GPIB is used skip this BootP Service Setup. Configure GPIB see page 18-36.

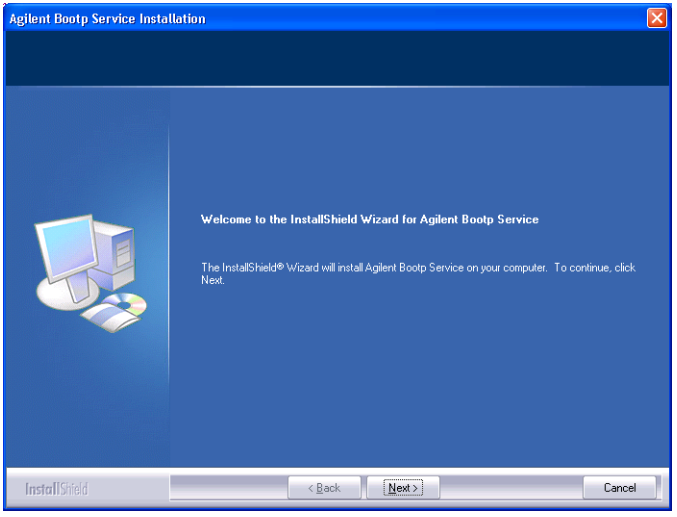
1 **Click *Agilent BootP Service Installation*.**

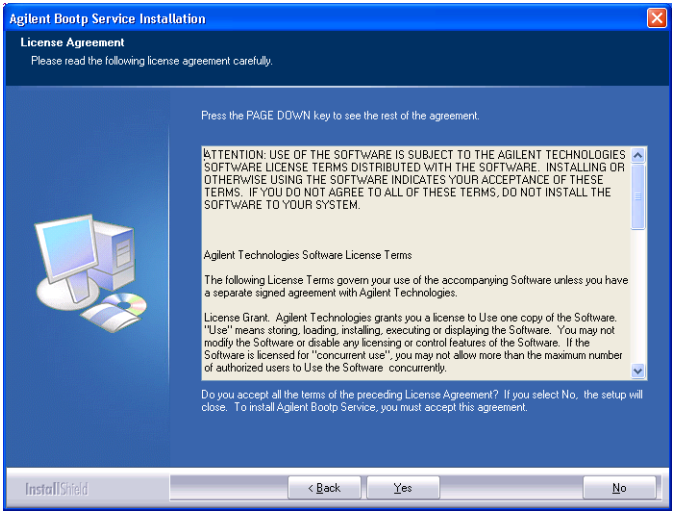


2 **Click *Open*.**



3 Installation Wizard will appears.
Click *Next* or *Yes.*





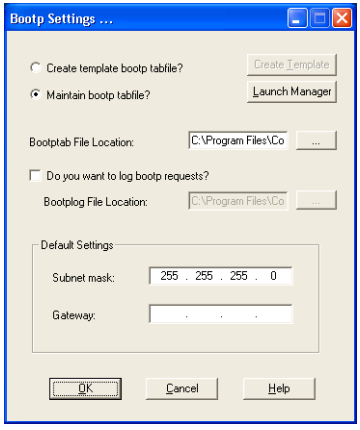
Installation status is shown. Wait until installation has completed.

4 The Bootp Settings dialog box appears.

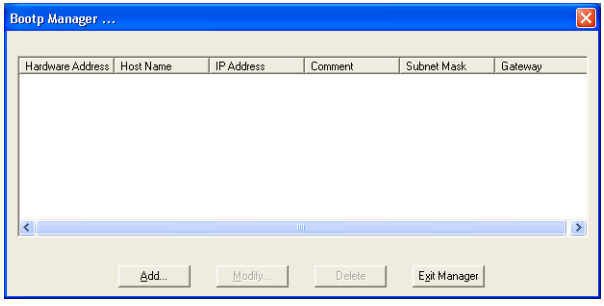
5 Select *Maintain bootp tabfile?*, enter the value of the Subnet mask, then click *Launch Manager.*

Subnet mask:255.255.255.0

Keep the displayed location of the Bootptab File Location.



The BootP Manager appears.



6 Click *Add* button.

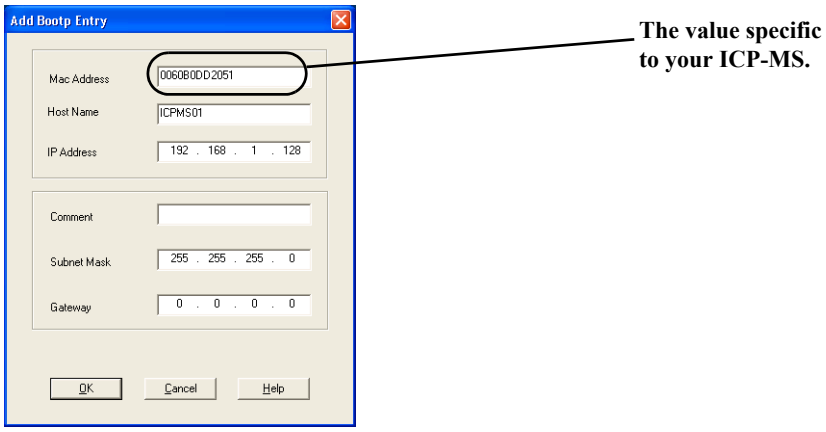
7 Enter the parameters.

MAC Address: Enter the MAC Address of the Agilent 7500. Refer to the next step to know the MAC Address.

Host Name: any name

IP Address: 192.168.1.128 (default)

Subnet Mask: 255.255.255.0 (default)



NOTE After installing the ChemStation software, you must set the same IP address in the ICP-MS Configuration Window as set in the Bootp configuration window.

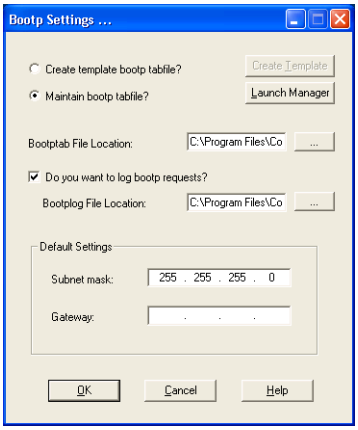
NOTE The Host Name must be entered.
Host name is restricted to the following characters only:
alphabet (A-Z), digits (0-9), and hyphen (-).
No distinction is made between upper and lower case. The first character must be an alpha character. The last character must not be a hyphen.

8 If a paper notification of the MAC Address is not attached, complete the following steps to get the MAC Address.

1 Select ***Maintain bootp tabfile?***.

2 Select ***Do you want to log bootp requests?***. Keep the displayed location of the **Bootplog File Location**.

3 Enter the ***Subnet mask*** (255.255.255.0).



4 Click **OK**.

5 Reboot the computer.

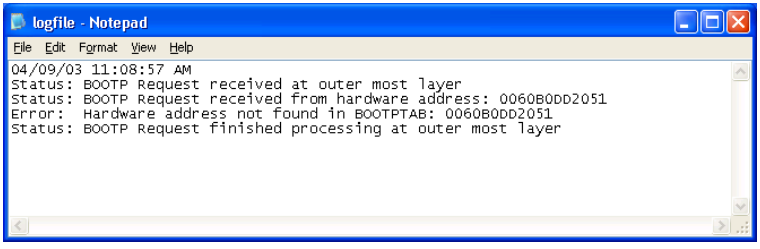
NOTE If the PC and the ICP-MS is connected to site LAN, disconnect the cabel from site LAN. This will avoid to have incorrect MAC Address from another hardware.

6 Logon the computer as *Support*.

7 Turn off the power of the Agilent 7500, then turn on the power.

8 Right-click **Start** button, then select **Explore**.

9 Select *C:\ProgramFiles\CommonFiles\AgilentShared\BootP\bin\logfile*. Right-click **logfile**, select **Open**. Select Notepad in **Open With** screen, then click **OK**. MAC Address is shown after "Hardware Address" in the Notepad. MAC Address of the Agilent 7500 starts with "0060B0....".



10 Select **Start>>All Programs>>Agilent BootP Service>>Edit BootP Settings**, then click **Launch Manager** button. Click **Add** in the Bootp Manager. Copy and paste the MAC Address from the logfile. Enter the Host Name, the IP Address,

and the Subnet mask. Refer to the earlier steps.

11 After setting, return to the **Bootp Settings**. Remove check at ***Do you want to log bootp requests?***.

9 Close the *Bootp Settings*.

10 Reboot the computer.

NOTE Reboot the computer after changing the Bootp Settings.

NOTE If you connect the instrument to the local network, you will need to talk with the network administrator. You may want to call your local ICP-MS tech support team for additional assistance.
To connect to site LAN, you are expected to understand the Windows XP operating system and TCP/IP networking, such as host name, IP address, subnet mask settings. You should also be familiar with the particular hardware devices and configurations of your organization's local area network.
Agilent Technologies is not responsible for any software problems, LAN configuration conflicts or performance problems that may result when a system is connected to a non-isolated LAN.

Printer Settings

This section describes the printer setting.

The printer setting will execute automatically, when you connect the printer and the computer with the cable and turn on the power. If the printer setting does not execute automatically, complete the following steps to setup the printer.

NOTE Use the printer driver included in the Windows XP.

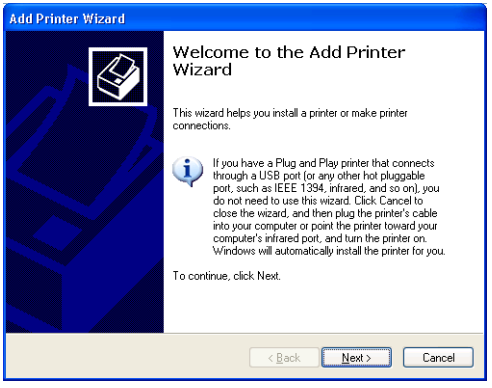
1 **Go to *Start>>Printers and Faxes.***

The ***Printers and Faxes*** screen appears.

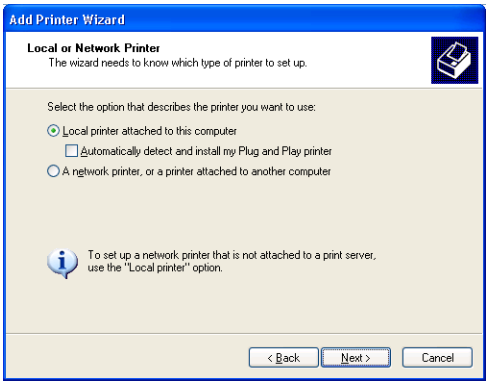
2 **Delete the *HP Laser Jet 4000 PCL5e* and the *HP Laser Jet;* if these printer drivers are pre installed.**

3 **Double-click *Add Printer.***

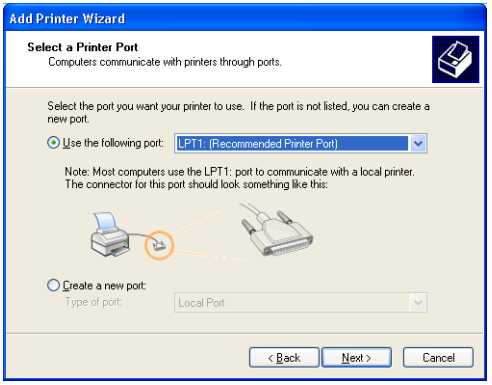
The ***Add Printer Wizard*** appears. Click ***Next***.



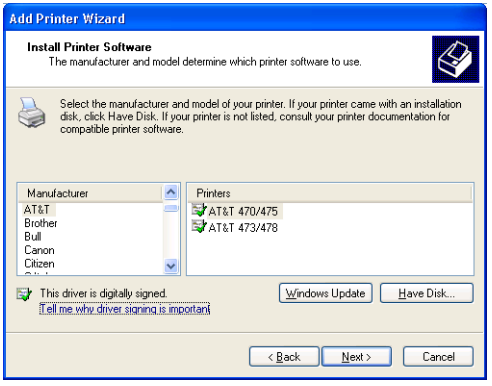
4 Select *Local printer* and remove check at *Automatically detect and install my Plug and Play printer.* Then click *Next.*



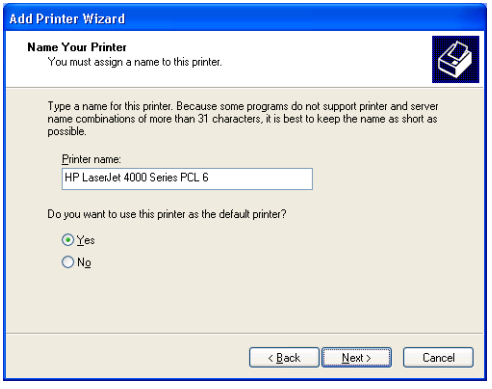
5 Depending on the cable attached to the printer, the selection of Printer Port is variable. If the attached cable is a Printer Cable, select LPT1 and click *Next.*



6 Select the printer name and click *Next.*

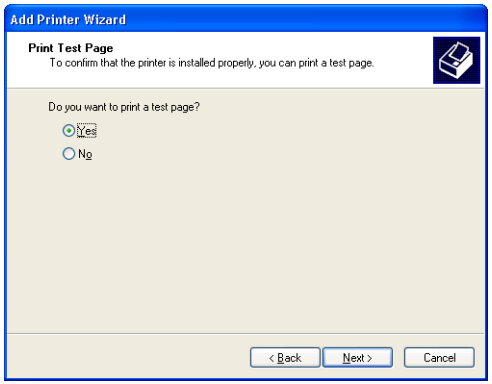


7 Select *Yes*.



8 Select *Do not share this printer.*

9 Select *Yes* or *No* and click *Next.*
If you select *Yes*, confirm Test Page.

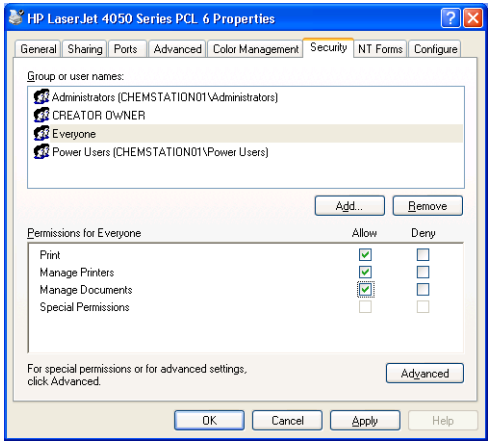


10 Click *Finish* to complete driver installation.

Then printer driver is now installed.

11 Right-click the appropriate printer in the printer window, then select *properties*.

12 Select *Security* tab. Select *Everyone* in the Group or user name. Select *Allow check box* of Print, Manage Printers, and Manage Documents.



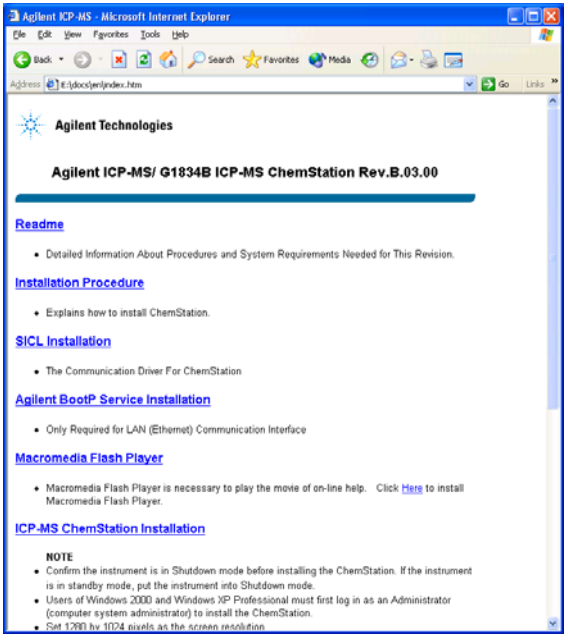
13 Click *OK*.

Installing the FlashPlayer

Macromedia Flash Player is necessary to play the movies in the on-line help.

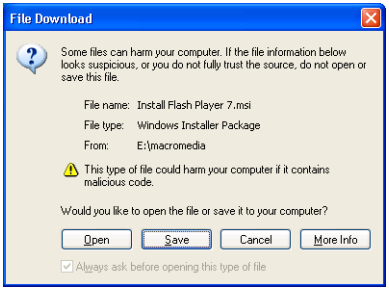
1 **Insert Agilent ICP-MS ChemStation System disk into CD-ROM Drive.**

Internet Explorer automatically appears. Click ***Macromedia Flash Player***.



2 **Click *Open*.**

FlashPlayer is now installed.



Installing the Agilent 7500 ChemStation Software

NOTE Before installing the ICP-MS ChemStation software, the followings must be installed.
- Microsoft Windows XP Service Pack 2
- SICL driver (I/O Libraries ver. L.02.01.00 or later)
- Agilent BootP Service
- Internet Explorer 6 Service Pack 1

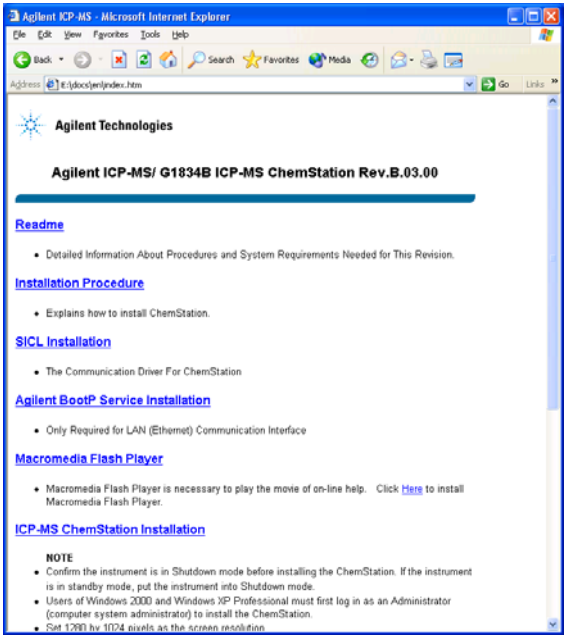
NOTE To use the LC/GC ChemStation, install the LC/GC ChemStation before installing the ICP-MS ChemStation, or without installing the chromatographic software.

NOTE 200 MB or more (hard disk space) is required to install the ChemStation Software.

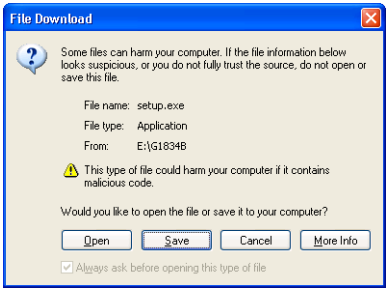
NOTE Confirm the instrument is in shutdown mode before installing ChemStation. If instrument is in standby mode, Put the instrument into Shutdown mode via the ChemStation before installing ChemStation because it is necessary to turn the instrument power on to download the firmware after installing the ChemStation.

1 Insert the ICP-MS ChemStation System disk into the CD-ROM drive.

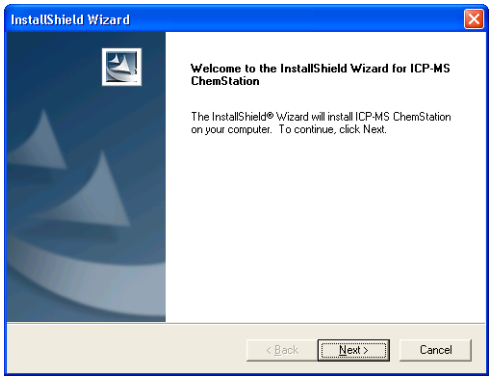
Internet Explorer automatically appears. Click ***ICP-MS ChemStation Installation***.



2 Click *Open*

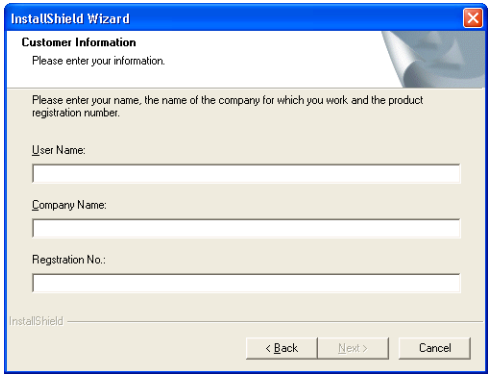


3 Click *Next*

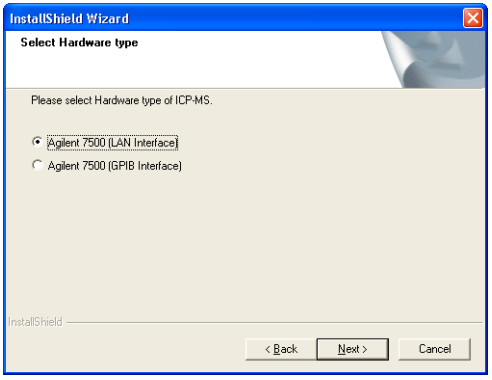


4 Registration dialog box appears.

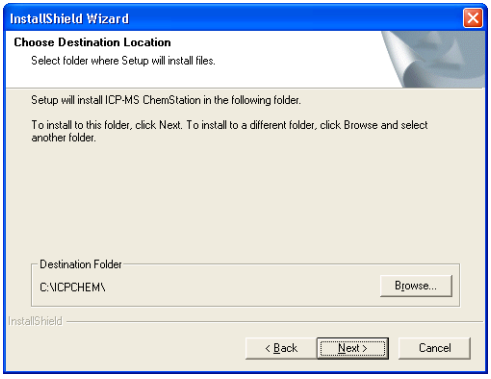
Enter the User Name and the Company Name.
Input *Reg#* that is in the registration pack, then click ***Next***.



5 Select *Hardware type*, then click *Next*.

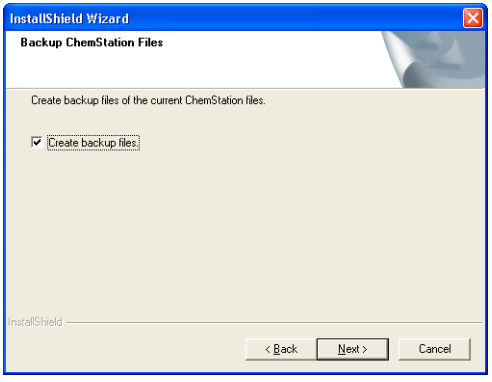


6 Select the destination folder, then click *Next*.



NOTE If the ChemStaiton is already installed, this screen does not appear.

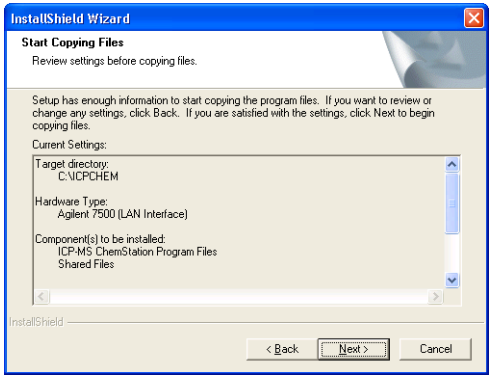
7 *Backup ChemStation Files* dialog box appears.



NOTE If the ChemStaiton is not installed yet, this screen does not appear.

Please make a backup of the existing ChemStation. To backup the existing ChemStation, check Create backup files and click *Next*. (When Create backup files is checked, specify the directory to be backed up in the next step.) If the PC hard disk does not have enough free space, remove check from Create backup files and click Next. In this case, files in the ICPCHEM folder will be deleted or overwritten.

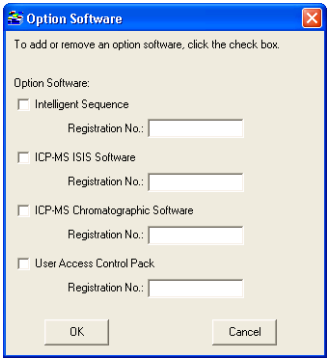
8 ***Start Copying Files*** **dialog box appears.**



Click ***Next***.

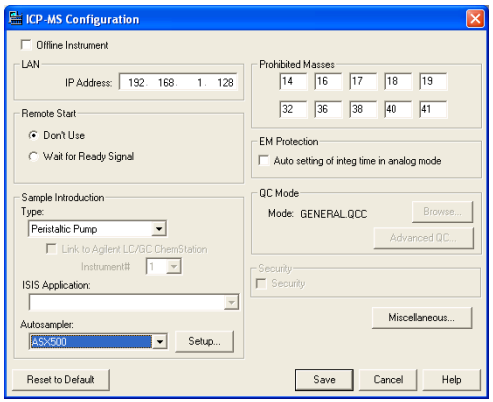
9 Follow the displayed message, install the ChemStation software and Help files.

After the installation, the ***Option Software*** dialog box appears.



Select the optional software, which is to be added and enter the registration numbers they are in the registration pack if purchased.

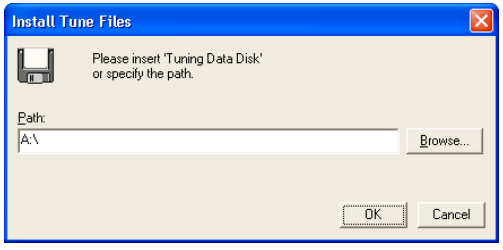
10 Click *OK*, then *ICP-MS configuration dialog box* appears.



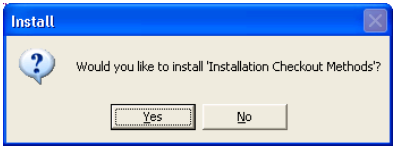
Check the ICP-MS Configuration and then click the *Save* button to save the configuration.

NOTE The IP address must be the same as set in the Bootp configuration window.

11 Insert G1833-60840 Parameter Disk Assy into Floppy disk driver and click *OK*.

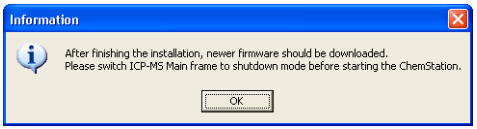


12 Click *Yes* if you need the "Installation Checkout Method".



13 The *Readme.txt* appear. Read the *Readme.txt* and Exit the *Readme.txt*.

14 **The message asking for the Agilent 7500 to be placed in Shutdown mode is displayed. Click *OK*.**



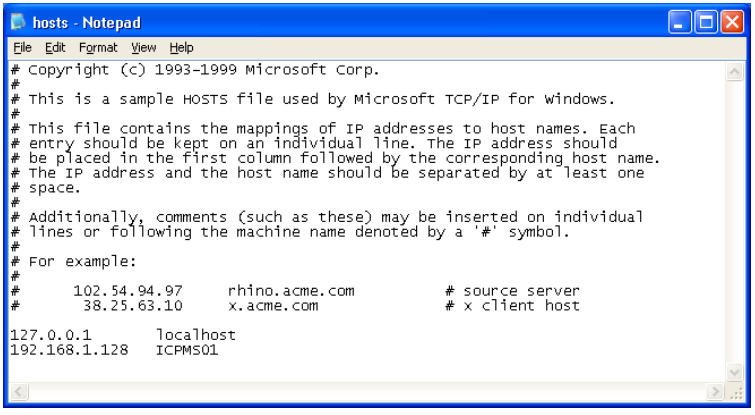
NOTE

If the instrument is not in shutdown mode, press the shutdown switch on the underside of the right instrument cover. The instrument transfers to shutdown mode (it takes approximately 5 minutes). When it is in shutdown mode, the status LED indicator on the cover turns off.

15 **Reboot the computer.**

16 **Install the Patch File if necessary. Refer to page 18-35.**

17 **Select *Start>>All Programs>>ICP-MS ChemStation>>Edit hosts*. Enter the IP Address and the Host Name of Agilent 7500 which is setup in the Bootp Service. Save the hosts file.**



18 **Reboot the computer.**

19 **Turn off the power of the Agilent 7500. Confirm the cable connection between the Agilent 7500 and the computer. Then turn on the Agilent 7500.**

CAUTION

After restarting the computer, turn off and turn on the Agilent 7500.

20 Start the ICP-MS ChemStation and download the firmware.

Double click ICP-MS Top.

When the ***ICP-MS Top*** window appears, download of the firmware starts automatically. (it takes approximately 10minutes)

21 Go to standby mode.

Select ***Vacuum>>Vacuum ON*** from the ICP-MS Instrument Control window to go to standby mode.

CAUTION 

Do not specify *.B, *.D, *.M, *.C, or long file names as ChemStation installation destinations. If these are specified, there is a possibility that the various methods, data, data batch, and calibration can not be loaded.

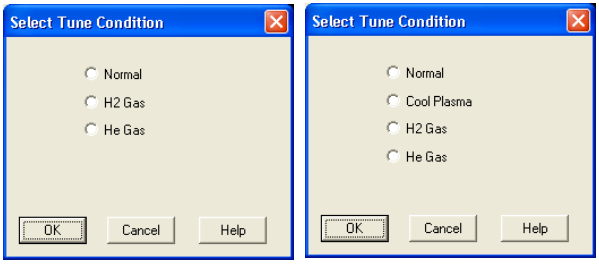
Initialization of a Tune File to the Factory Default Condition

The following procedure allows initialization of a tune file to the factory default condition.

1 Select *Top* window >> *Instrument* >> *Tuning*.

2 Select *File* >> *Load Factory Defaults*.

The ***Select Tune Condition*** dialog box will appear.



7500ce Display **7500cs Display**

3 Select the tune file to return to the factory default condition.

The tune files displayed will vary depending upon the model.

- 7500a: No tune file is displayed.
- 7500ce: Standard, H2 gas, He gas
- 7500cs: Standard, cool plasma, H2 gas, He gas

4 Click *OK*.

The selected tune file will be initialized to the factory default condition.

Installation the Patch Files

Before reinstalling ChemStation, the patch file had been installed, it is necessary to install the Patch file after reinstalling ChemStation.

You can update the ChemStation Revision by installing the patch file. You can download the patch file from following URL:

http://www.chem.agilent.com/scripts/cag_checkreg.asp

1. Verify the instrument is in Shutdown mode. If the instrument is in Standby mode, put the instrument in Shutdown mode from the ChemStation, then close ChemStation.
2. Click **Configuration** and Note the current Configuration settings (e.g. Auto Sampler, Tray setting, QC Mode). Configuration will be set to default settings after installing the Patch File
3. Close ChemStation and reboot your PC.
4. Copy "arpatch.exe" to \icpchem\icpexe\.
5. Copy the patch file "g1834b_0X_0X_00X.ptf" in any temp directory e.g. \temp.
6. Select ***Start*** >> ***Programs*** >> ***ICP-MS ChemStation*** >> ***Install Patch File***
7. Patch file installer for ChemStation window will open. Click ***Patch files...*** and select the patch file, which has been copied in "Step 5". Click ***Install***.
8. ICP-MS Configuration dialog box will open, then click ***Save***. The Readme.txt will open. Read Readme.txt then close.
9. "Patch File were installed completely" dialog box will appear. Then Click OK.
10. Turn the instrument power OFF and ON.
11. Click Configuration and input the information recorded in step 2. Then Click ***Save***.
12. Click ***ICP-MS Top*** and put the instrument in Standby mode.

Installation of the patch file is complete.

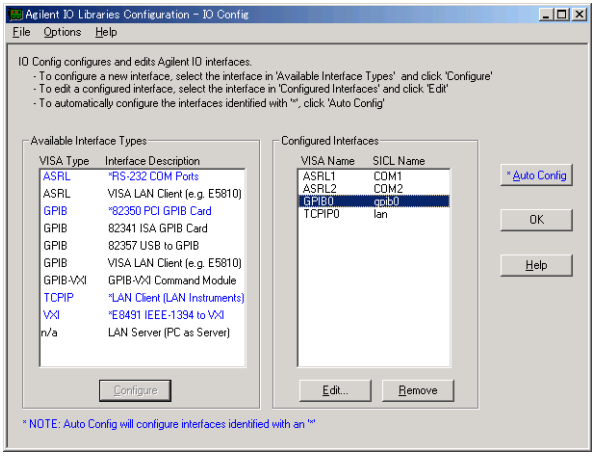
NOTE The patch installer scans the suitability of the ChemStation Software version and patch file, and only installs the necessary patch files.

GPIB Configuration

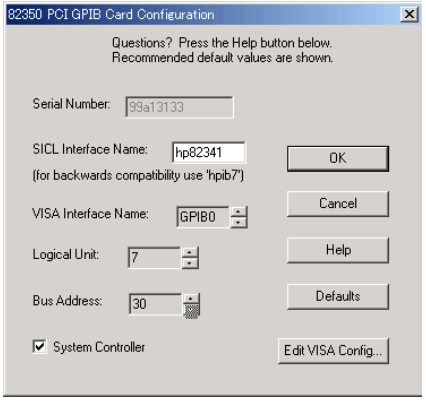
When GPIB is used for the communication, set the following.

1 GPIB Setting

Highlight (Click) [GPIB0] and Click ***Edit***

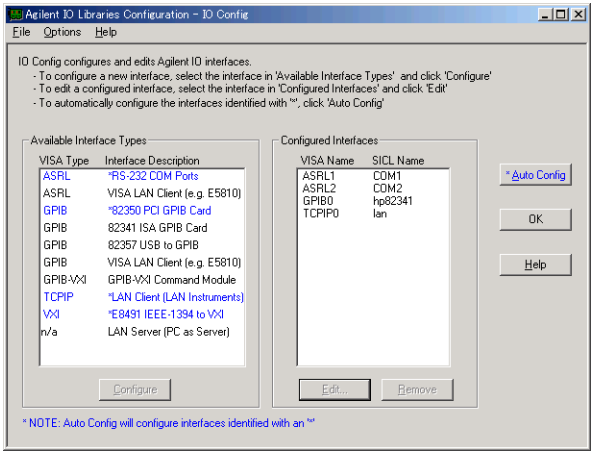


Set as follow. Then Click ***OK***.



2 IO Config Setting

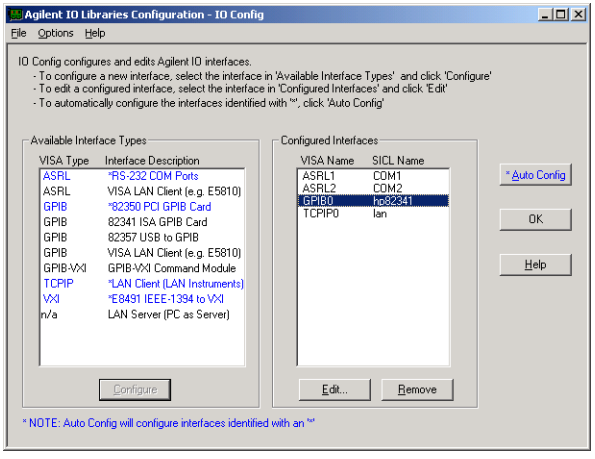
Click ***OK***



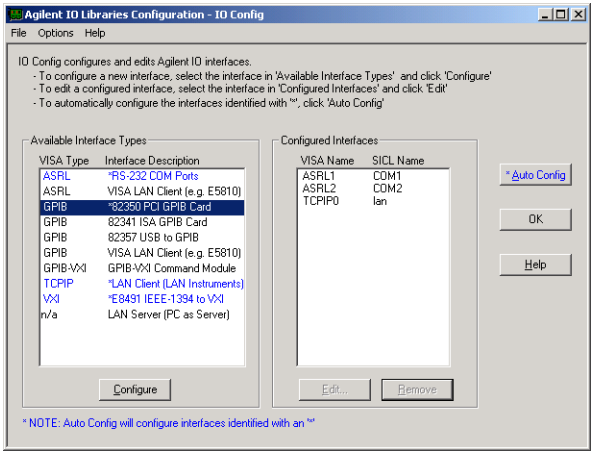
If No communication with 7500 when GPIB is 82350A or 82350B

If ChemStation can not communicate with the 7500 when the GPIB is 82350A or 82350B, Remove the Setting and reconfigure the GPIB setting manually as follows.

1 Select [GPIB hp 82341] in the Configured Interface and Click ***Remove***.

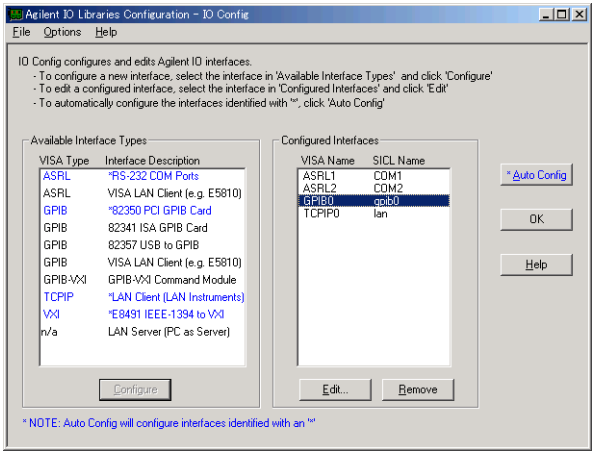


2 Select [GPIB 82350 PCI GPIB Card] in the Available Interface Type and Click ***Configure***.

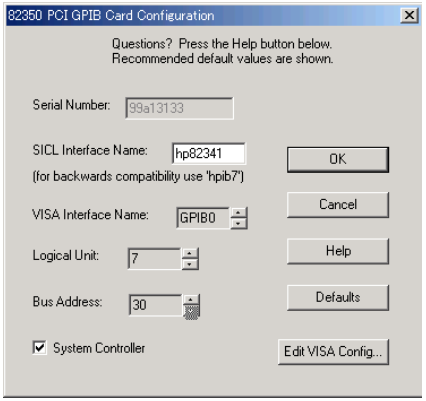


3 GPIB Setting

Highlight (Click) [GPIB0] and Click ***Edit***

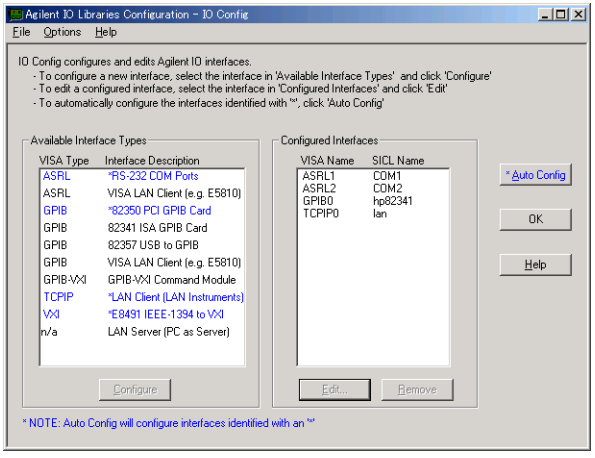


Set the following. Then Click ***OK***.



4 IO Config Setting is now Complete

Click ***OK***



Changing Windows Firewall Settings to Run ICP-MS ChemStation Under Windows XP SP2

Introduction

This section describes the changes in settings that need to be made to run the ICP-MS ChemStation on PCs running Windows XP SP2.

Follow the description in this section to change your Windows Firewall settings and enable the ICP-MS program and Bootp Server/Client port communications.

Target Programs: icpacq.exe and icptune.exe

Target Ports: 67 TCP/UDP and 68 TCP/UDP

- Note: It is NOT necessary to change Windows Firewall settings in the following cases:

  1. If the operating system (OS) is not Windows XP.
  2. If the OS is Windows XP, but SP2 is not installed.
  3. If you are using a GP-IB interface for the Agilent 7500 (Windows Firewall settings must be changed with LANs).

CAUTION 

If the Agilent 7500 is connected to the ICP-MS ChemStation through the site LAN, any change in settings may affect the entire network. Consult with your network administrator before changing the settings.

CAUTION 

Change your Windows Firewall settings after installing the ICP-MS ChemStation. (Install the ICP-MS ChemStation software if you have not yet done so.)

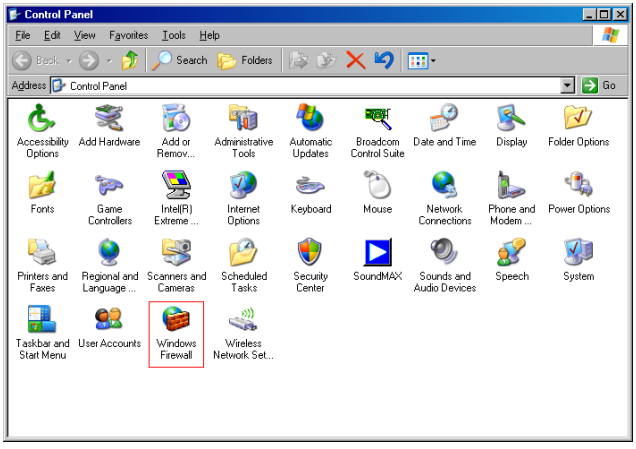
Changing the Windows Firewall Configuration

As described in the following steps, enable communications for programs icp-acq.exe and icptune.exe and ports 67 and 68. The detailed port settings to be made are as follows:

Name	Port No.	TCP/UDP
Bootp Client 68 TCP	68	TCP
Bootp Client 68 UDP	68	UDP
Bootp Server 67 TCP	67	TCP
Bootp Server 67 UDP	67	UDP

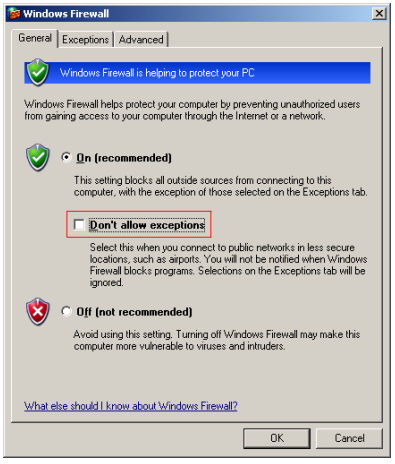
1 **Logon to Windows as a user with Administrator privileges.**

2 **Open the Control Panel from the Windows *Start* button and double-click the *Windows Firewall* icon.**

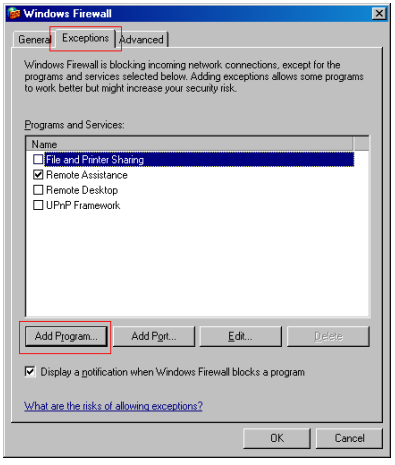


The ***Windows Firewall*** dialog box appears.

3 Click the *General* tab, then select *On (recommended)* and uncheck the *Don't allow exceptions* checkbox.

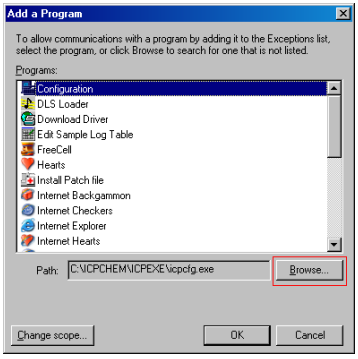


4 Click the *Exceptions* tab first, then the *Add Program*….



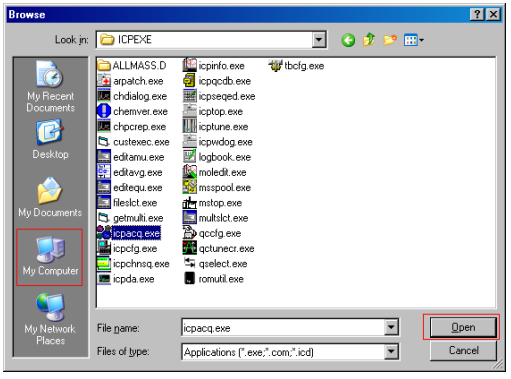
The ***Add a Program*** dialog box appears.

5 Click *Browse….*



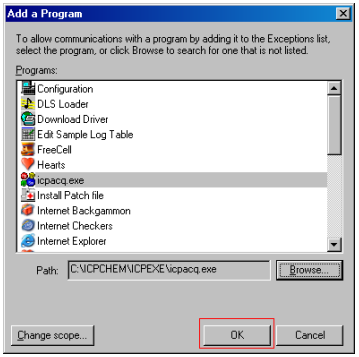
The ***Browse*** dialog box appears.

6 Click *My Computer* from the list on the left hand side of the Browse dialog box. Select *(C:) --> ICPCHEM --> ICPEXE --> icpacq.exe* from the list on the right. Confirm selection of "icpacq.exe" as shown in the screen below, then click *Open*. If the ChemStation has been installed into a drive other than C:, specify the correct drive.



The ***Add a Program*** dialog box appears.

7 **Confirm "icpacq.exe" is listed in the *Add a Program* dialog box, then select "icpacq.exe" and click *OK*.**



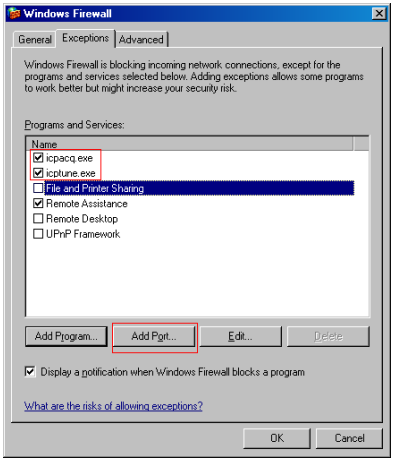
The ***Windows Firewall*** dialog box appears.

8 **Confirm "icpacq.exe" is added to *Program and Services* in the *Windows Firewall* dialog box.**

9 **Add "icptune.exe" in the same way as described in steps 4 to 8.**

10 **Confirm that "icpacq.exe" and "icptune.exe" have been added to the *Windows Firewall* dialog box.**

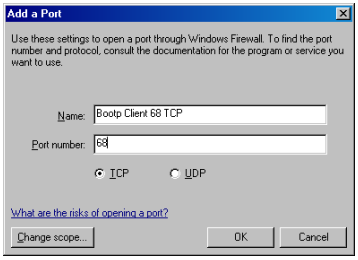
11 **Click the *Add Port*….**



The ***Add a Port*** dialog box appears.

12 Set the following items and click *OK*.

- Name: Type in "Bootp Client 68 TCP."
- Port Number: Type in "68."
- Click to select TCP or UDP. Here, select TCP.

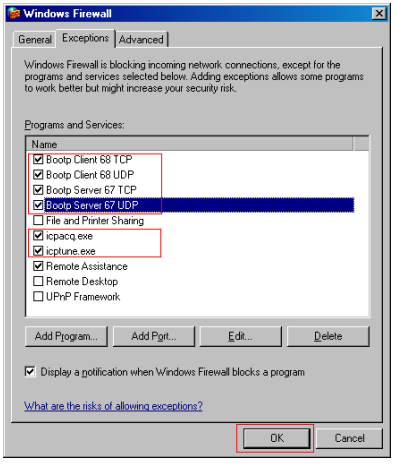


13 Follow Steps 11 and 12 to add three more ports.

Name	Port No.	TCP/UDP	
Bootp Client 68 TCP	68	TCP	Done
Bootp Client 68 UDP	68	UDP	Add
Bootp Server 67 TCP	67	TCP	Add
Bootp Server 67 UDP	67	UDP	Add

14 Confirm that all of the following items have been added in the *Windows Firewall* dialog box. Click *OK*.

- Bootp Client 68 TCP
- Bootp Client 68 UDP
- Bootp Server 67 TCP
- Bootp Server 67 UDP
- icpacq.exe
- icptune.exe



Changes to the firewall settings are now complete.

15 Open the ICP-MS ChemStation and confirm that communication with the Agilent 7500 is possible.

19

Installing the Agilent 7500 ChemStation Software (Windows 2000)

Installing the Agilent 7500 ChemStation Software (Windows 2000)

This section explains how to set up the computer and install the ChemStation software G1834B(Rev. B.01.04 or later) for Windows 2000.

WARNING  **Many computers have voltage selection switches, check for the correct setting according to your local supply before inserting the power cord.**

NOTE Confirm the instrument is in shutdown mode before installing ChemStation. If instrument is in standby mode, Put the instrument into Shutdown mode via the ChemStation before installing ChemStation because it is necessary to turn the instrument power on to download the firmware after installing the ChemStation.

CAUTION  The setup procedure in this section is a typical example. The procedure depends on the computer and printer.

Windows 2000 Configuration

NOTE If the O/S is Windows NT / 2000 selectable install Windows 2000.

NOTE The partition for the ChemStation should be NTFS format.

Windows 2000 Configuration

1 Turn on the computer and check the following BIOS settings.

- Power setting: Disabled (for example: Auto Suspend Timeout, Hard Drive Timeout, Modem Ring)
- Plug and Play: Enable

NOTE All items will not always appear.

NOTE Refer to the computer's manual for the BIOS setting.

Following is a typical example.

Select F2 (F8 for VL420, F10 for Evo D510) during computer initialization, check that the following settings are true. BIOS settings need not be changed for the D530.

Table 19-1 BIOS Settings for Vectra VL400

Items	Setting
Power >> Auto Suspend Timeout	Disabled
Power >> Modem Ring	Disabled
Advanced > Plug & Play O/S	Yes

Table 19-2 BIOS Settings for Vectra VL420

Items	Setting
Main >> PnP OS	YES

Table 19-3 BIOS Settings for Evo D510

Items	Setting
Power >> Hard Drive Timeout	Never
Power >> System Timeout	Never

Exit and save the settings.

2 Start Windows 2000.

If the logon dialog box is automatically displayed, go to step 3. If it's not displayed, it's necessary to setup the Administrator's User name. When finished, go to step 3.

3 Logon as Administrator.

User name: Administrator
Password: 3000hanover or blank
(Depending on the computer)

4 Setting screen saver

Double-click ***Display*** in the Control Panel.
Select the ***Screen Saver*** tab and confirm the following settings:

Screen Saver	None

Select the ***Power*** button and confirm the following settings.

Turn off monitor	Never
Turn off hard disks	Never
System standby	Never

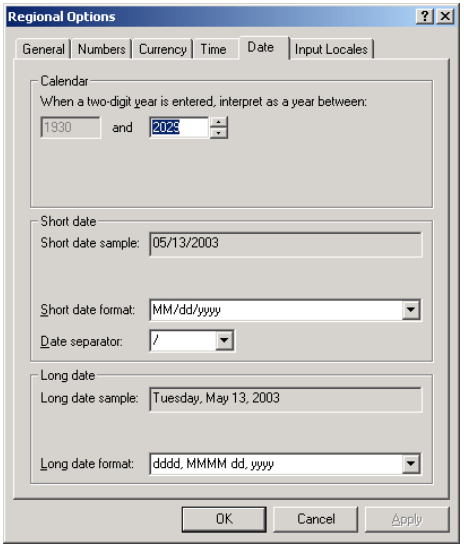
Select the ***Settings*** tab in Display Properties and confirm the following settings:

Colors	High color (16 bit)
Screen Area	1280 by 1024 pixel

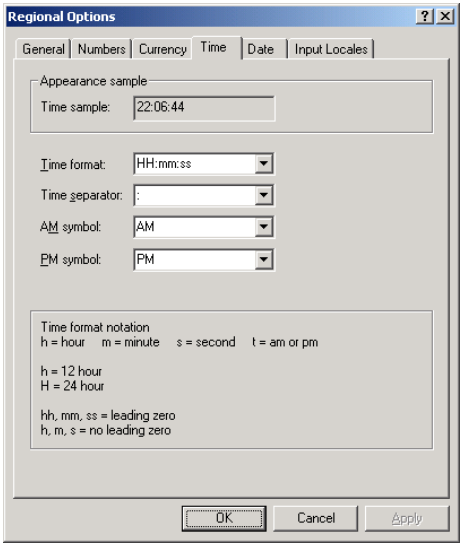
5 Format Day/Time

Double click the ***Regional Options*** icon in the Control Panel.

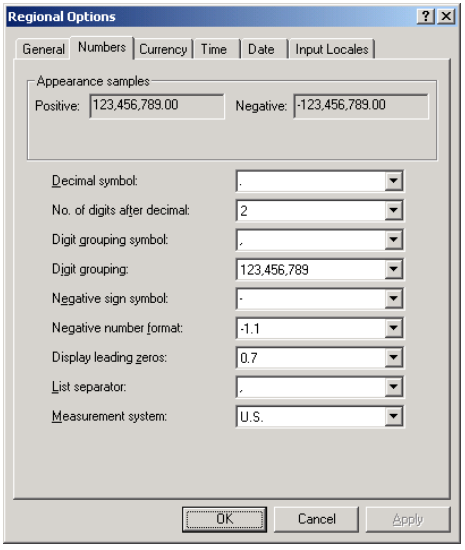
Select the ***Date*** tab and select ***MM/dd/yyyy*** in the Short Date Style box.



Select the ***Time*** tab and select ***HH:mm:ss***.



Click on the ***Number*** tab and Select "***.*** ***(Dot)*** in the Decimal Symbol box.



Click ***OK***.

6 Internet Protocol (TCP / IP) Properties

1 Double click ***Network and Dial-up Connections*** icon in the Control Panel.

2 Right click ***Local Area Connection,*** select ***Properties***.

3 Select ***Internet Protocol (TCP / IP),*** select ***Properties.***

4 Input the following information for a simple direct connection.

IP address : 192.168.1.127
Subnet mask : 255.255.255.0

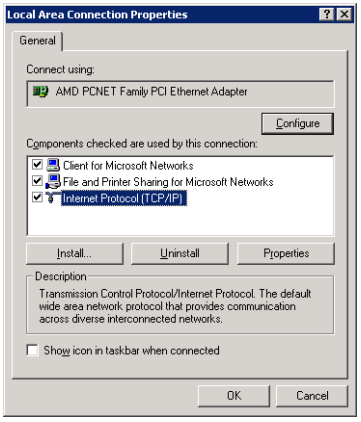
5 Click ***Advanced*** button, select ***WINS*** tab, then select ON the ***Enable LMHOSTS lookup***.

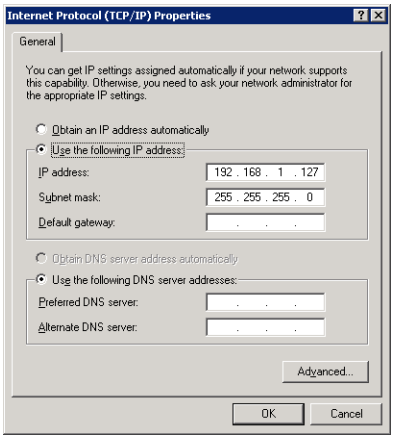
6 Click ***OK***.

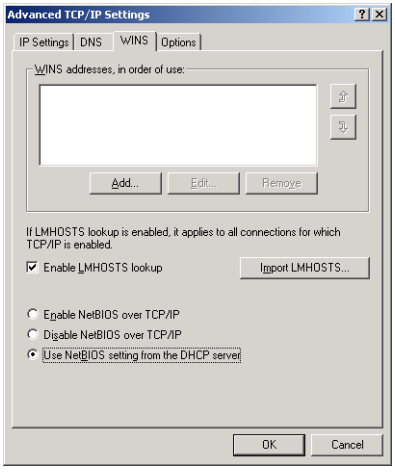
NOTE

If you connect the instrument to the local network, you will need to talk with the network administrator. You may want to call your local ICP-MS tech support team for additional assistance.
To connect to site LAN, you are expected to understand the Windows XP operating system and TCP/IP networking, such as host name, IP address, subnet mask settings. You should also be familiar with the particular hardware devices and configurations of your organization's local area network.

Agilent Technologies is not responsible for any software problems, LAN configuration conflicts or performance problems that may result when a system is connected to a non-isolated LAN.







Creating User Account

Creating User Account as follows:

User Name	Description	Password	Password property	Group
Support	Product Support	HPCE	Select ***Password never expires*** check box only. Do not select other check boxes.	Administrators
Chemist	ChemStation Chemist	hp	Select ***Password never expires*** check box only. Do not select other check boxes.	Power Users
User	ChemStation User		Select ***Password never expires*** check box only. Do not select other check boxes.	Users

To create or modify the user account, complete the following steps;

1. **Select *Administrative Tools>>Computer Management* in the Control Panel.**
2. **Select *Local Users and Groups>>Users* in the left area of the Computer Management.**
3. **Select *Action>>New User* menu in the Computer Management.**
4. **Enter the User name, Description, Password, and Password property. Then click *Create*.**
5. **Right-click the user name in the Computer Management, then select *Set Properties.***
6. **Select *Member Of* tab, then click *Add* button.**
7. **Click *Advanced* button in Select Groups.**
8. **Click *Find Now* button, then select the group name and click *OK*.**
9. **Close the *Computer Management* after setting the users.**

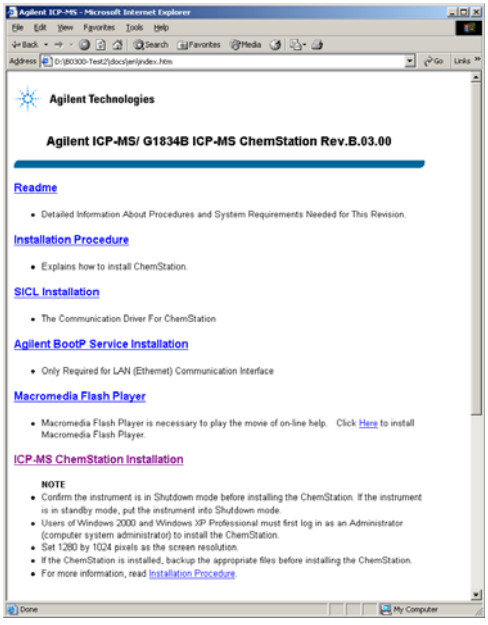
CAUTION 

The local user account in this section is a typical example. The information contained in this section is subject to change without notice.

I/O Library Setup

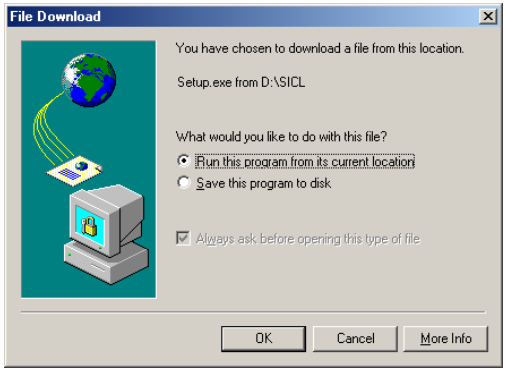
1 Insert Agilent ICP-MS ChemStation System disk into CD-ROM Drive.

Internet Explorer automatically appears. Click **SICL Installation**.



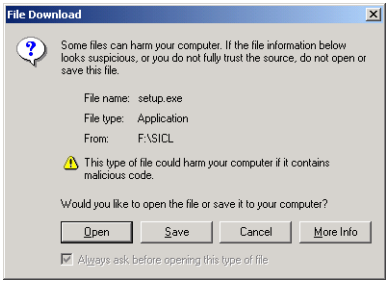
2 Select [Run this program from its current location], click *OK*.

When Internet Explorer is Version 6, another dialog Box will appear. Skip to 2-1.



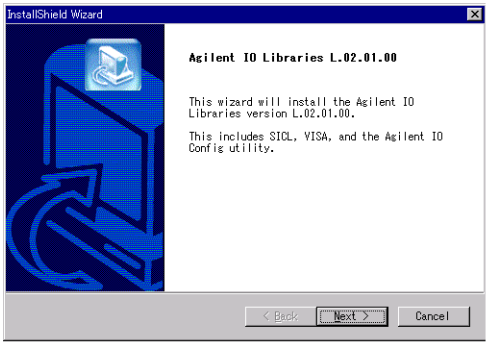
Click ***Yes***.

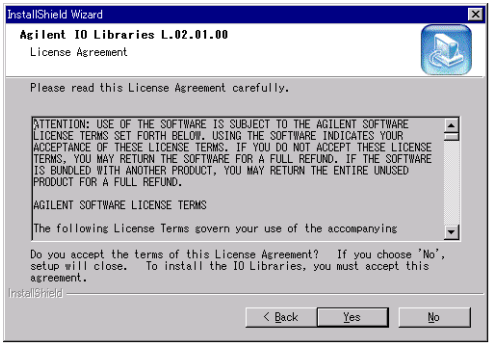
2-1 When Internet Explorer is Version 6, the following dialog Box will appear.
Click *Open*

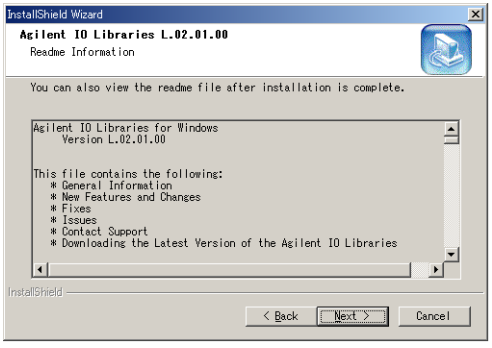


3 InstallShield Wizard

Click ***Next*** or ***Yes***

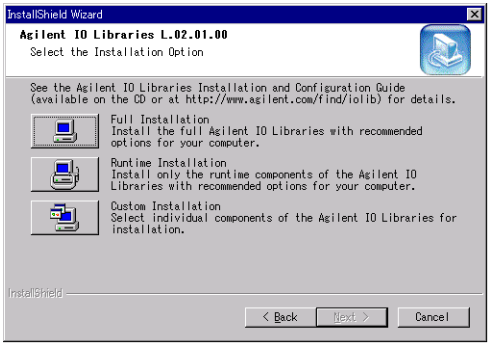






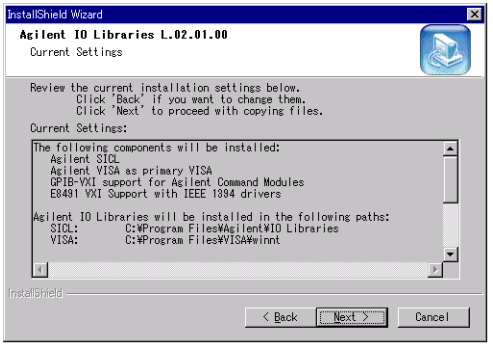
4 Select Installation Option

Select Full Installation. Then Click ***Next***



5 Current Setting

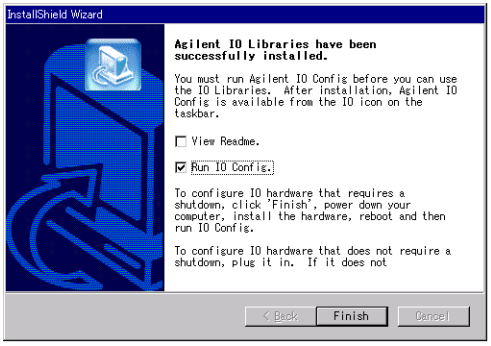
Click ***Next***



Installation status is shown. Wait until installation has completed.

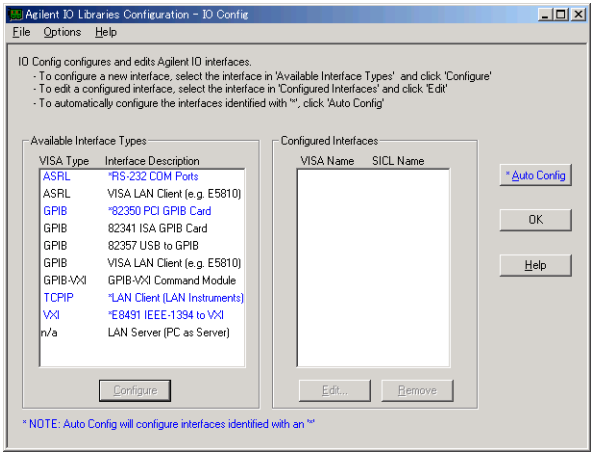
6 Installation Completion

When the installation is complete, the following dialog box will appear. Select [**RUN IO Config**]. And Click ***Finish***.

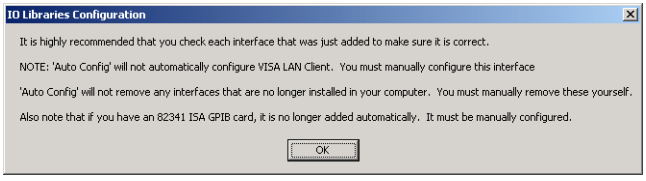


7 IO Config

Following dialog box will appear. Click [**Auto Config**].



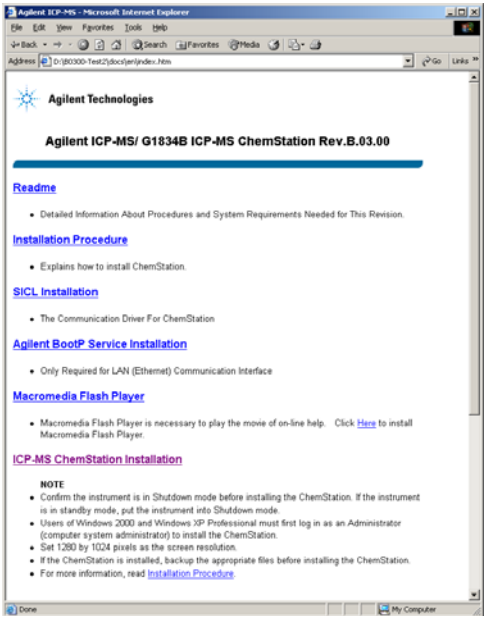
Following message will appear. Click ***OK***.



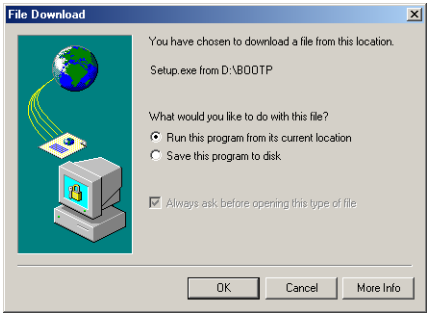
BootP Service Setup

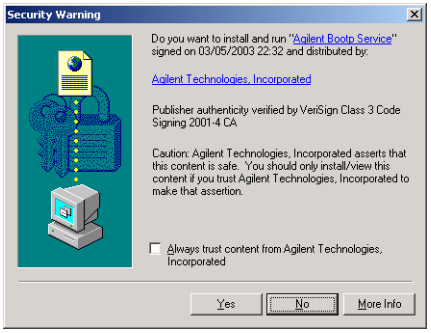
When GPIB is used skip this BootP Service Setup. Configure GPIB see page 18-36.

1 Click **Agilent BootP Service Installation**.



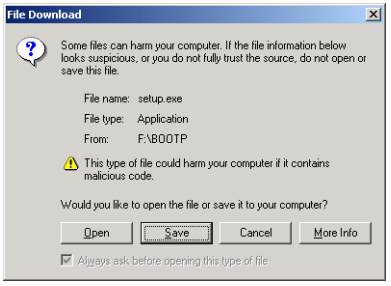
2 Following dialog Box will appear.
When Internet Explorer is Version 6, another dialog Box will appear. Skip to 2-1
Click ***OK.*** .



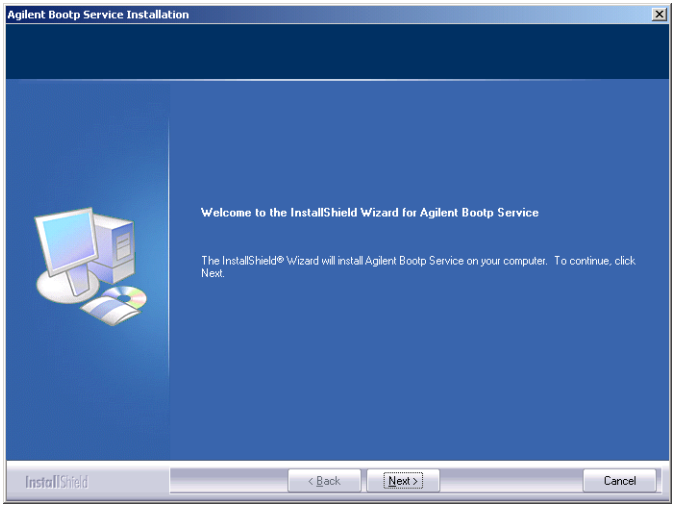


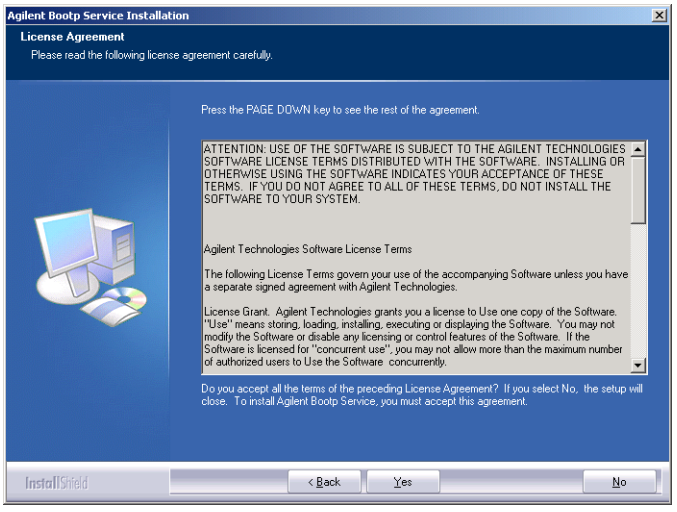
Click ***Yes***.

2-1 When Internet Explorer is Version 6, the following dialog Box will appear. Click ***Open***



3 Installation Wizard will appears. Click ***Next*** or ***Yes.***





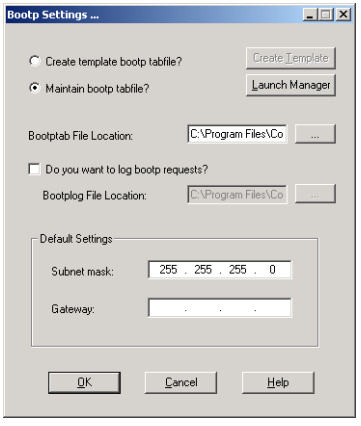
Installation status is shown. Wait until installation has completed.

4 The Bootp Settings dialog box appears.

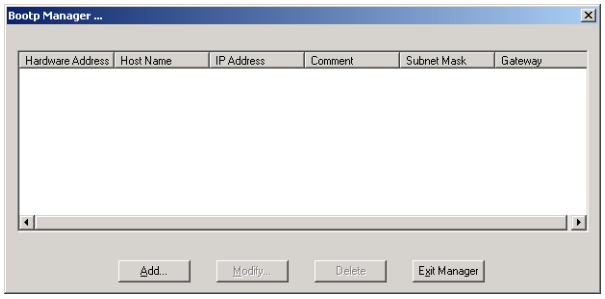
5 Select *Maintain bootp tabfile?*, enter the value of the Subnet mask, then click *Launch Manager.*

Subnet mask: 255.255.255.0

Keep the displayed location of the Bootptab File Location.



The BootP Manager appears.



6 Click *Add* button.

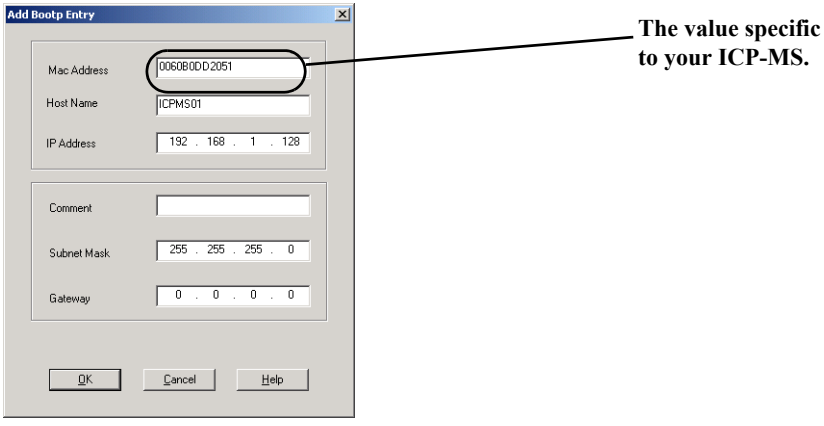
7 Enter the parameters.

MAC Address: Enter the MAC Address of the Agilent 7500. Refer to the next step to know the MAC Address.

Host Name: any name

IP Address: 192.168.1.128 (default)

Subnet Mask: 255.255.255.0 (default)



NOTE After installing the ChemStation software, you must set the same IP address in the ICP-MS Configuration Window as set in the Bootp configuration window.

NOTE

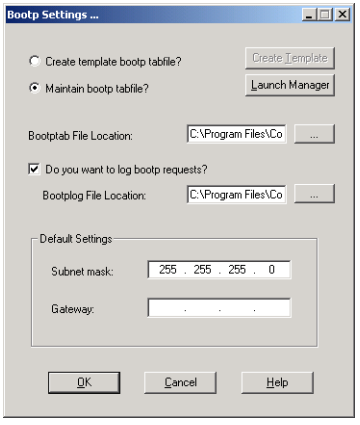
The Host Name must be entered.
Host name is restricted to the following characters only:
alphabet (A-Z), digits (0-9), and hyphen (-).
No distinction is made between upper and lower case. The first character must be an alpha character. The last character must not be a hyphen.

8 If the paper of MAC Address is not attached, complete the following steps to get the MAC Address.

1 Select ***Maintain bootp tabfile?***.

2 Select ***Do you want to log bootp requests?***. Keep the displayed location of the **Bootplog File Location**.

3 Enter the ***Subnet mask*** (255.255.255.0).



4 Click **OK**.

5 Reboot the computer.

NOTE

If the PC and the ICP-MS is connected to site LAN, disconnect the cabel from site LAN. This will avoid to have incorrect MAC Address from another hardware.

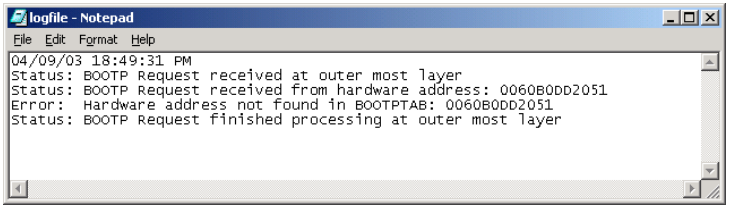
6 Logon the computer as *Support*.

7 Turn off the power of the Agilent 7500, then turn on the power.

8 Right-click **Start** button, then select **Explore**.

9 Select *C:\ProgramFiles\CommonFiles\AgilentShared\BootP\bin\logfile*. Right-click **logfile**, select **Open**. Select Notepad in **Open With** screen, then click

OK. MAC Address is shown after "Hardware Address" in the Notepad. MAC Address of the Agilent 7500 starts with "0060B0....".



10 Select **Start>>Programs>>Agilent BootP Service>>Edit BootP Settings**, then click **Launch Manager** button. Click **Add** in the Bootp Manager. Copy and paste the MAC Address from the logfile. Enter the Host Name, the IP Address, and the Subnet mask. Refer to the earlier steps.

11 After setting, return to the **Bootp Settings**. Remove check at ***Do you want to log bootp requests?***.

9 Close the *Bootp Settings*.

10 Reboot the computer.

NOTE Reboot the computer after changing the Bootp Settings.

NOTE If you connect the instrument to the local network, you will need to talk with the network administrator. You may want to call your local ICP-MS tech support team for additional assistance.
To connect to site LAN, you are expected to understand the Windows XP operating system and TCP/IP networking, such as host name, IP address, subnet mask settings. You should also be familiar with the particular hardware devices and configurations of your organization's local area network.
Agilent Technologies is not responsible for any software problems, LAN configuration conflicts or performance problems that may result when a system is connected to a non-isolated LAN.

Printer Settings

NOTE LJ 4000 or 4050 - Use the printer driver included in the ChemStation CD-ROM.

NOTE LJ 2200 - Use the printer driver shipped with the printer.

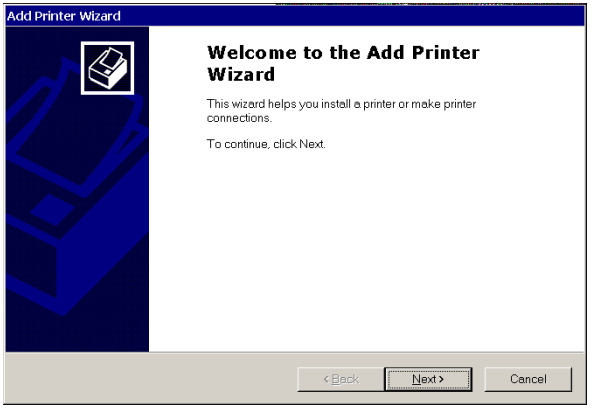
1 **Go to *Start>>Setting>>Printer.***

The ***Printer*** dialog box appears.

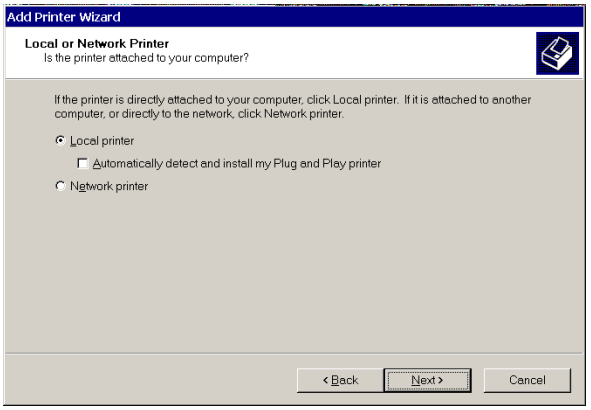
2 **Delete the *HP Laser Jet 4000 PCL5e* and the *HP Laser Jet;* if these printer drivers are pre installed.**

3 **Double-click *Add Printer.***

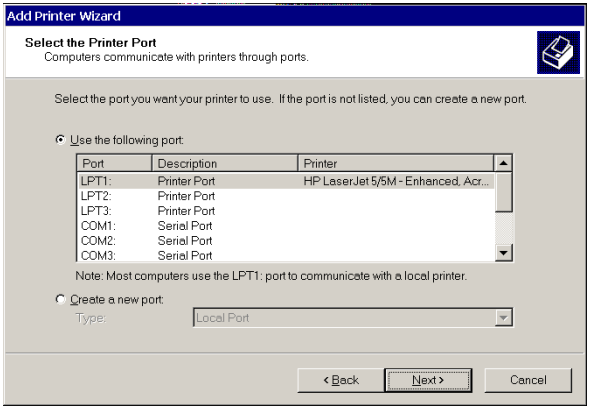
The ***Add Printer Wizard*** appears. Click ***Next***.



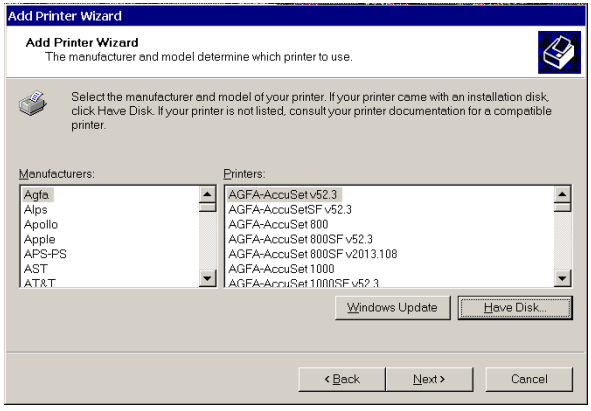
4 Select *Local printer* and remove check at *Automatically detect and install my Plug and Play printer.* Then click *Next.*



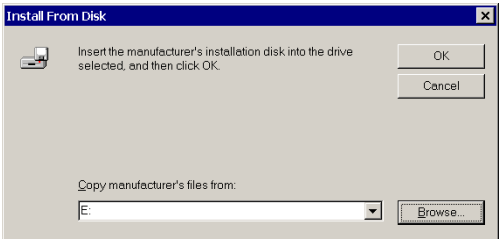
5 Depending on the cable attached to the printer, the selection of Printer Port is variable. If the attached cable is a Printer Cable, select LPT1 and click *Next.* If the attached cable is a USB cable, select USB printer port.



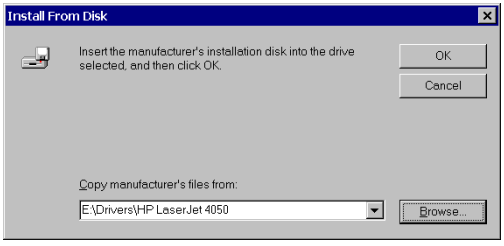
6 Click *Have disk*.



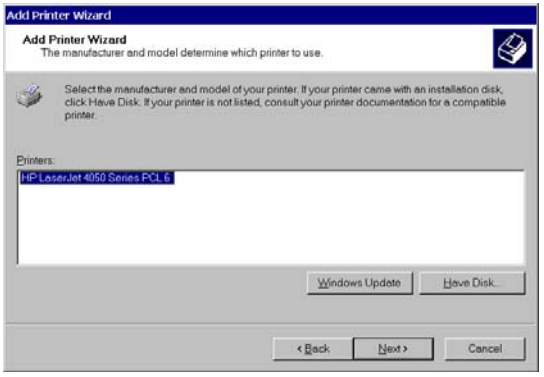
The ***Install From Disk*** dialog box appears.



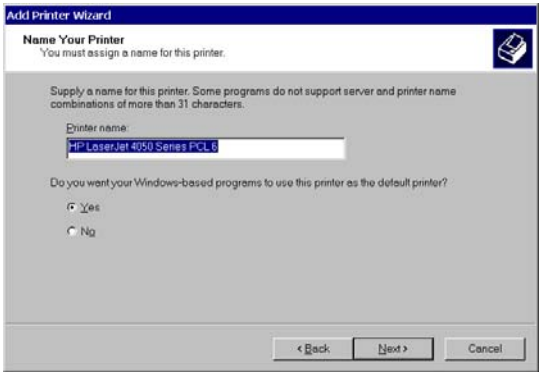
7 Insert the ICP-MS ChemStation System CD-ROM and select the CD-ROM drive. Click *Browse* and select the folder (\Drivers\HPLaserJet4050) in which the appropriate printer driver exists.Then Click *OK*.



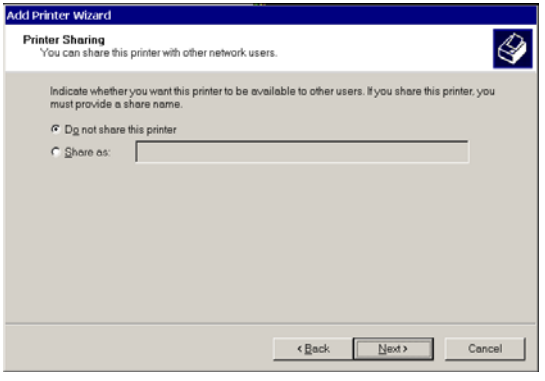
8 Select the printer name and click *Next*.



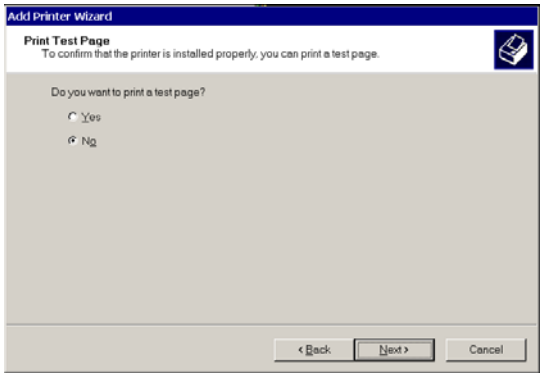
9 Select *Yes*.



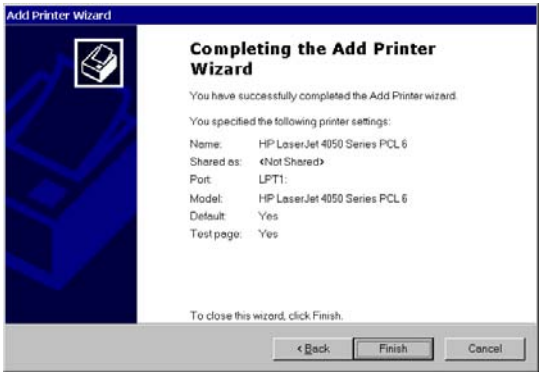
10 Select *Do not share this printer.*



11 Select *Yes* or *No* and click *Next.*
If you select *Yes*, confirm Test Page.



12 Click *Finish* to complete driver installation.



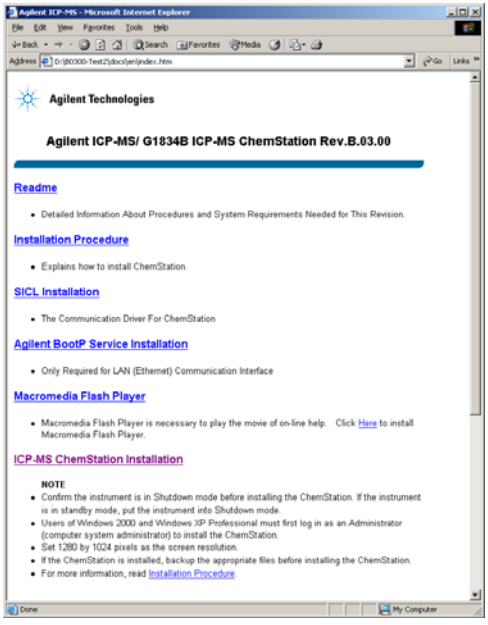
Then printer driver is now installed.

Installing the FlashPlayer

Macromedia Flash Player is necessary to play the movies in the on-line help.

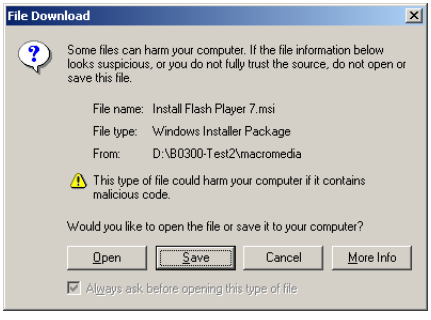
1 **Insert Agilent ICP-MS ChemStation System disk into CD-ROM Drive.**

Internet Explorer automatically appears. Click ***Macromedia Flash Player***.



2 **Click Open.**

FlashPlayer is now installed.



Installing the Agilent 7500 ChemStation Software

NOTE Before installing the ICP-MS ChemStation software, the followings must be installed.
- Microsoft Windows 2000 Service Pack 4
- SICL driver (I/O Libraries ver. L.02.01.00 or later)
- Agilent BootP Service
- Internet Explorer 6 Service Pack 1

NOTE To use the LC/GC ChemStation, install the LC/GC ChemStation before installing the ICP-MS ChemStation, or without installing the chromatographic software.

NOTE 200 MB or more (hard disk space) is required to install the ChemStation Software.

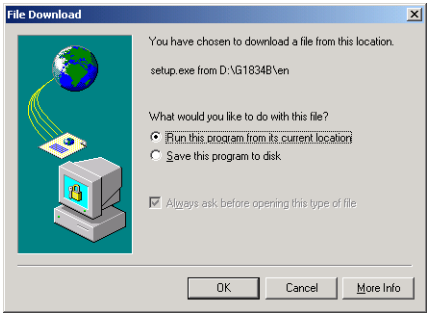
NOTE Confirm the instrument is in shutdown mode before installing ChemStation. If instrument is in standby mode, Put the instrument into Shutdown mode via the ChemStation before installing ChemStation because it is necessary to turn the instrument power on to download the firmware after installing the ChemStation.

1 Insert the ICP-MS ChemStation System disk into the CD-ROM drive.

Internet Explorer automatically appears. Click **ICP-MS ChemStation Installation**.

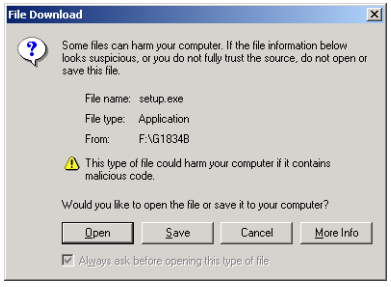
2 Select [Run this program from its current location] click *OK*.

When Internet Explorer is Version 6, another dialog Box will appear. Skip to 2-1.

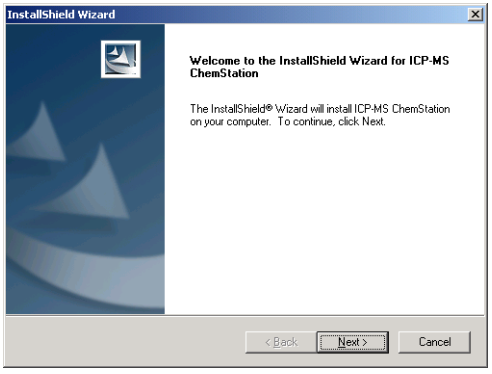


Click ***Yes***.

2-1 When Internet Explorer is Version 6, the following dialog Box will appear. Click ***Open***

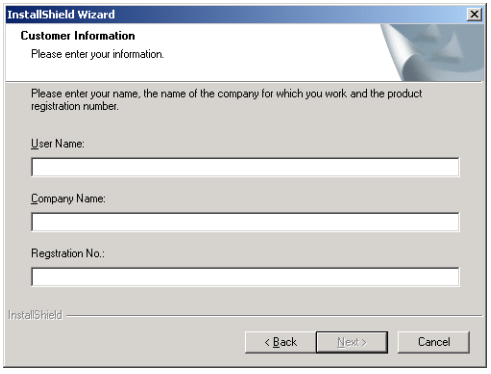


3 Click *Next*

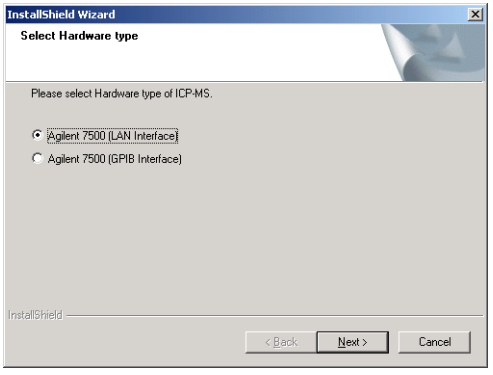


4 Registration dialog box appears.

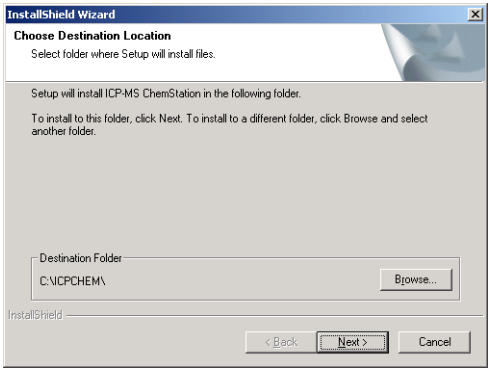
Enter the User Name and the Company Name.
Input *Reg#* that is in the registration pack, then click ***Next***.



5 Select *Hardware type*, then click *Next*.

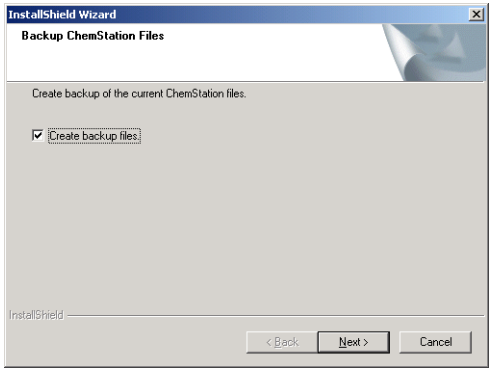


6 Select the destination folder, then click *Next*.



NOTE If the ChemStaiton is already installed, this screen does not appear.

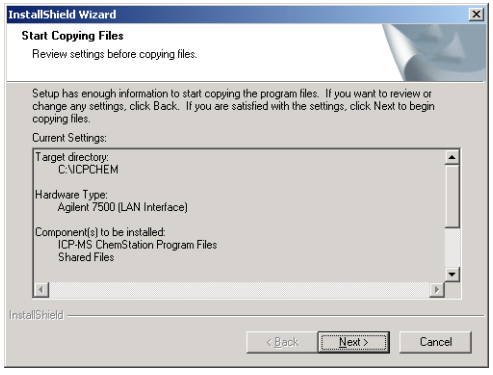
7 ***Backup ChemStation Files*** **dialog box appears.**



NOTE If the ChemStaiton is not installed yet, this screen does not appear.

Please make a backup of the existing ChemStation. To backup the existing ChemStation, check Create backup files and click *Next*. (When Create backup files is checked, specify the directory to be backed up in the next step.) If the PC hard disk does not have enough free space, remove check from Create backup files and click Next. In this case, files in the ICPCHEM folder will be deleted or overwritten.

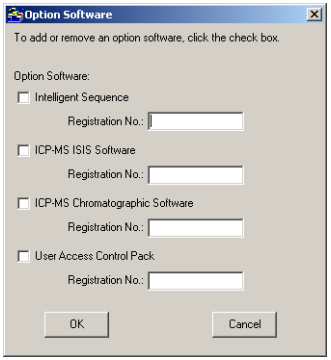
8 ***Start Copying Files*** **dialog box appears.**



Click ***Next***.

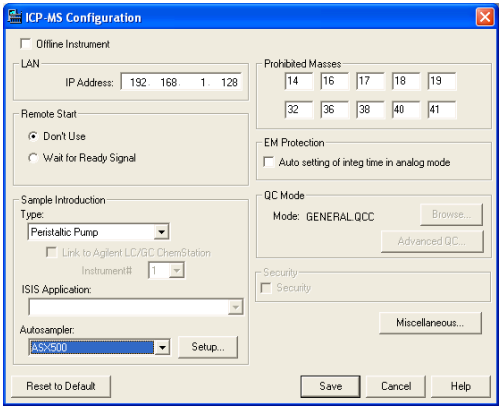
9 Follow the displayed message, install the ChemStation software and Help files.

After the installation, the ***Option Software*** dialog box appears.



Select the optional software, which is to be added and enter the registration numbers they are in the registration pack if purchased.

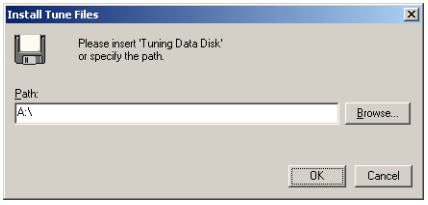
10 Click *OK*, then *ICP-MS configuration dialog box* appears.



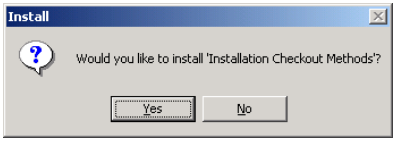
Check the ICP-MS Configuration and then click the *Save* button to save the configuration.

NOTE The IP address must be the same as set in the Bootp configuration window.

11 Insert G1833-60840 Parameter Disk Assy into Floppy disk driver and click *OK*.

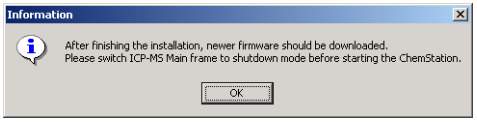


12 Click *Yes* if you need the "Installation Checkout Method".



13 The *Readme.txt* appear. Read the *Readme.txt* and Exit the *Readme.txt*.

14 The message asking for the Agilent 7500 to be placed in Shutdown mode is displayed. Click *OK*.



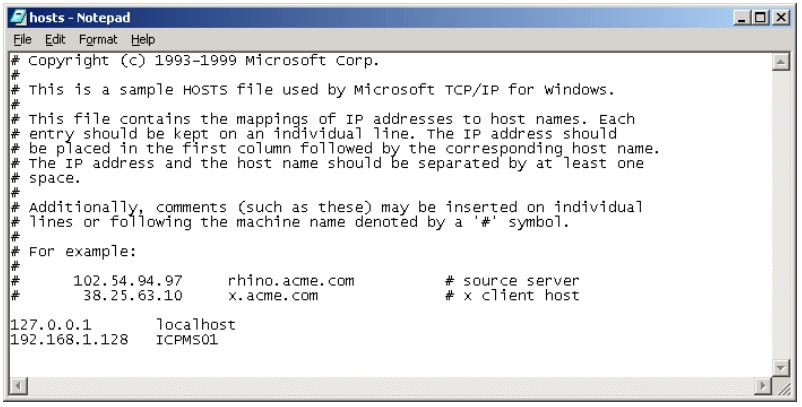
NOTE

If the instrument is not in shutdown mode, press the shutdown switch on the underside of the right instrument cover. The instrument transfers to shutdown mode (it takes approximately 5 minutes). When it is in shutdown mode, the status LED indicator on the cover turns off.

15 Reboot the computer.

16 Install the Patch File if necessary. Refer to page 19-35.

17 Select *Start>>Programs>>ICP-MS ChemStation>>Edit hosts*. Enter the IP Address and the Host Name of Agilent 7500 which is setup in the Bootp Service. Save the hosts file.



18 Reboot the computer.

19 Turn off the power of the Agilent 7500. Confirm the cable connection between the Agilent 7500 and the computer. Then turn on the Agilent 7500.

CAUTION After restarting the computer, turn off and turn on the Agilent 7500.

20 Start the ICP-MS ChemStation and download the firmware.

Double click ICP-MS Top.

When the ***ICP-MS Top*** window appears, download of the firmware starts automatically. (it takes approximately 10minutes)

21 Go to standby mode.

Select ***Vacuum>>Vacuum ON*** from the ICP-MS Instrument Control window to go to standby mode.

CAUTION Do not specify *.B, *.D, *.M, *.C, or long file names as ChemStation installation destinations. If these are specified, there is a possibility that the various methods, data, data batch, and calibration can not be loaded.

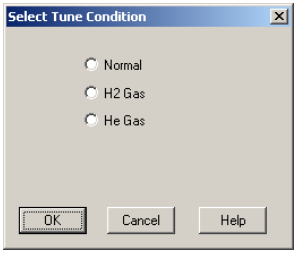
Initialization of a Tune File to the Factory Default Condition

The following procedure allows initialization of a tune file to the factory default condition.

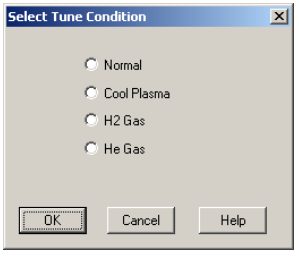
1 **Select *Top* window >> *Instrument* >> *Tuning*.**

2 **Select *File* >> *Load Factory Defaults*.**

The ***Select Tune Condition*** dialog box will appear.



7500ce Display



7500cs Display

3 **Select the tune file to return to the factory default condition.**

The tune files displayed will vary depending on the specific model.

- 7500a: No tune file is displayed.
- 7500ce: Standard, H2 gas, He gas
- 7500cs: Standard, cool plasma, H2 gas, He gas

4 **Click *OK*.**

The selected tune file will be initialized to the factory default condition.

Installation the Patch Files

Before reinstalling ChemStation, the patch file had been installed, it is necessary to install the Patch file after reinstalling ChemStation.

You can update the ChemStation Revision by installing the patch file. You can download the patch file from following URL:

http://www.chem.agilent.com/scripts/cag_checkreg.asp

1. Verify the instrument is in Shutdown mode. If the instrument is in Standby mode, put the instrument in Shutdown mode from the ChemStation, then close ChemStation.
2. Click **Configuration** and Note the current Configuration settings (e.g. Auto Sampler, Tray setting, QC Mode). Configuration will be set to default settings after installing the Patch File
3. Close ChemStation and reboot your PC.
4. Copy "arpatch.exe" to \icpchem\icpexe\.
5. Copy the patch file "g1834b_0X_0X_00X.ptf" in any temp directory e.g. \temp.
6. Select ***Start*** >> ***Programs*** >> ***ICP-MS ChemStation*** >> ***Install Patch File***
7. Patch file installer for ChemStation window will open. Click ***Patch files...*** and select the patch file, which has been copied in "Step 5". Click ***Install***.
8. ICP-MS Configuration dialog box will open, then click ***Save***. The Readme.txt will open. Read Readme.txt then close.
9. "Patch File were installed completely" dialog box will appear. Then Click OK.
10. Turn the instrument power OFF and ON.
11. Click Configuration and input the information recorded in step 2. Then Click ***Save***.
12. Click ***ICP-MS Top*** and put the instrument in Standby mode.

Installation of the patch file is complete.

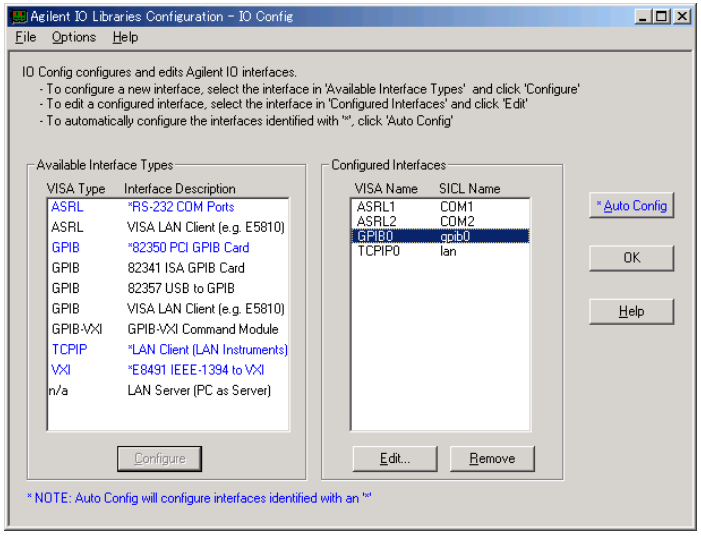
NOTE The patch installer scans the suitability of the ChemStation Software version and patch file, and only installs the necessary patch files.

GPIB Configuration

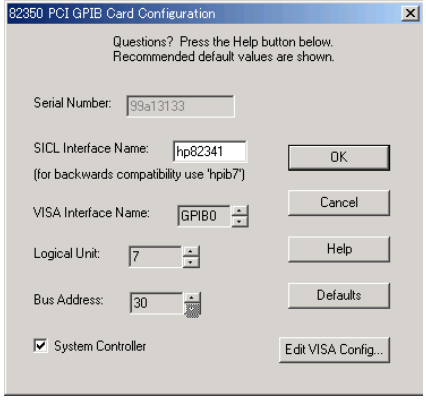
When GPIB is used for the communication, set the following.

1 GPIB Setting

Highlight (Click) [GPIB0] and Click ***Edit***

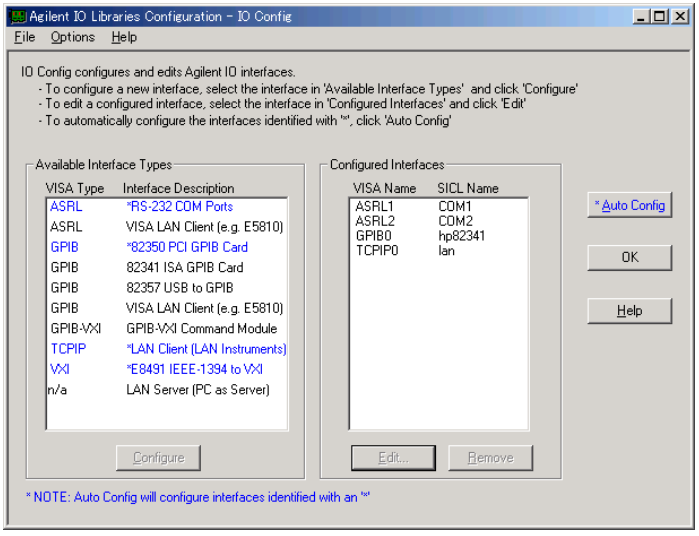


Set as follow. Then Click ***OK***.



2 IO Config Setting

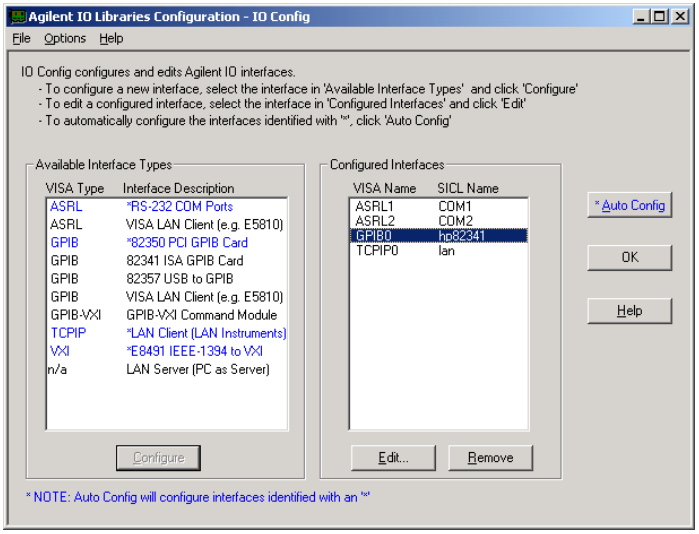
Click ***OK***



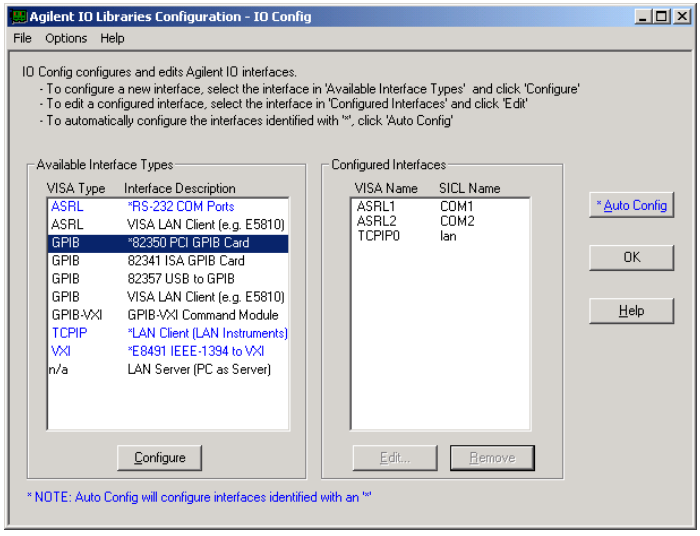
If No communication with 7500 when GPIB is 82350A or 82350B

If ChemStation can not communicate with the 7500 when the GPIB is 82350A or 82350B, Remove the Setting and reconfigure the GPIB setting manually as follows.

1 Select [GPIB hp 82341] in the Configured Interface and Click ***Remove***.

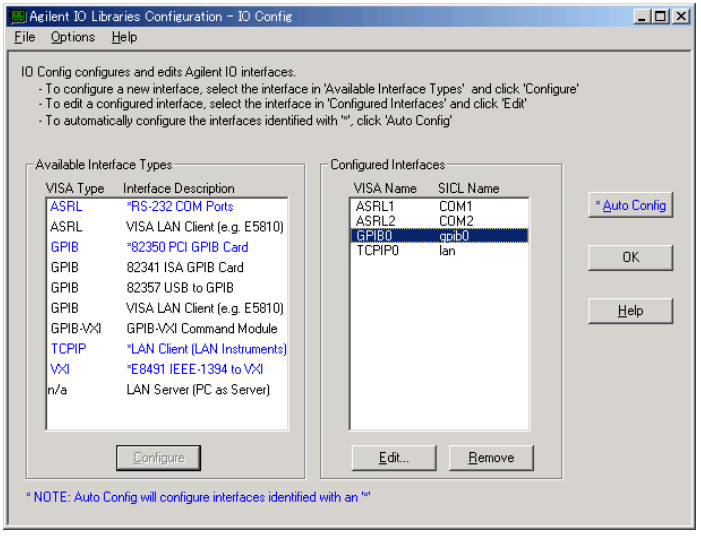


2 Select [GPIB 82350 PCI GPIB Card] in the Available Interface Type and Click ***Configure***.

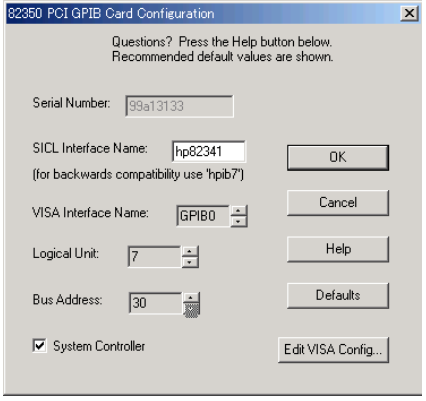


3 GPIB Setting

Highlight (Click) [GPIB0] and Click ***Edit***

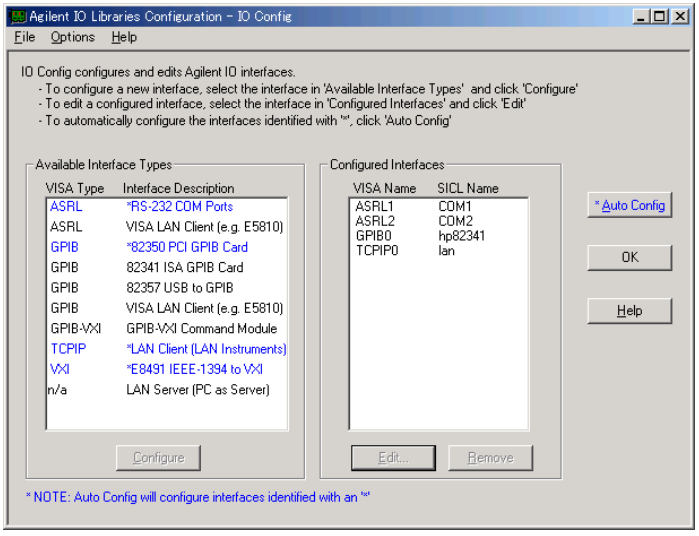


Set the following. Then Click ***OK***.



4 IO Config Setting is now Complete

Click ***OK***



Appendix

Appendix A

This appendix includes the ChemStation menus. It enables you to quickly determine which menu to access for a specific purpose.

Top Task

[Instrument]

- [Instrument Control...]
- [Tune...]
- [Exit]

[AcquireData]

- [Main Panel...]
- [Edit AMU Select File (.amu)...]

[DataAnalysis]

- [Main Panel...]

[Methods]

- [Load and Run Method...]
- [Run...]
- [Run Method Wizard...]
- [Edit Entire Method...]
- [Edit Method Information...]
- [Edit Interference Equation...]
- [Load Calibration...]
- [Save Calibration...]
- [Load...]
- [Save...]
- [Print]

[Sequence]

- [Load and Run Sequence...]
- [Run...]
- [Reprocess Data Batch…]
- [Edit Sample Log Table...]
- [Position and Run...]
- [Simulate Sequence...]
- [View Sequence Log...]
- [Print Sequence Log]
- [Load...]
- [Save...]
- [Print]
- [More...]

[Chain Sequence]

- [Edit and Run...]
- [Simulate Chain Seq]
- [View Chained Seq Log…]
- [Load...]
- [Save...]

[Window]

- [Reset Window Position]

[Tools]

- [Offline Data Analysis]
- [Offline Sample Log Table]
- [Archive Files...]
- [Extract Files...]

[Help]

- [Help topics]
- [About...]

Instrument/Tuning Task

Instrument Control

[Plasma]

- [Plasma ON]
- [Plasma OFF]
- [Exit]

[Vacuum]

- [Vacuum ON]
- [Vacuum OFF]

[Diagnostics]

- [View Error Log...]
- [Clear Error Log]
- [Running Time...]
- [Ion Lenses Test]
- [Nebulizer Test]

[Maintenance]

- [Sample Introduction...]
- [Restart S/C Control]
- [Reaction Gas] ---For models 7500ce and 7500cs
- [Octopole] ---For models 7500ce and 7500cs
- [Hardware Settings...]

[ALS]

- [Go To...]
- [Home]
- [Rinse]
- [ALS Initialize]

[Meters]

- [Meter Control Panel...]

[Maintenance Log]

[Log Book…]

[Help]

[Help topics]
[About...]

Tuning

[File]

[Load Tune Values...]
[Save Tune Values...]
[Load Factory Defaults]
[Generate Report]
[Print]
[Copy Tune Parameters...]
[Exit]

[Tune]

[Sensitivity]
[Oxide Ion]
[Doubly Charged Ion]
[Resolution/Axis]
[P/A Factor]
[Generate P/A Factor Report]
[Reaction Gas] ---For models 7500ce and 7500cs
[Full Spectrum]
[Autotune…]
[Peri Pump Program for Autotune]
[RF Matching]
[Dead time calibration...] ---When dead-time calibration is used

[Acq. Params]

[Acquisition Parameters...]

[ALS]

[Goto...]
[Home]
[Rinse]
[ALS Initialize]

[Meters]

[Meter Control Panel...]

[Maintenance Log]

[Log Book…]

[Help]

[Help topics]
[About...]

Data Acquisition Task

Main Panel

[AcquireData]

[Acquire Data...]
[Exit]

[Edit Parameters]

[Set Mode...]
[Set Parameters...] ---For an operation other than Time Program acquisition
[Set Time Program...]
[Print Summary...]

[PeriPump]

[Set Peripump Program...]

[ALS]

[Goto...]
[Home]
[Rinse]
[ALS Initialize]

[Logs]

[View Acq Log...]
[Clear Acq Log]

[Help]

[Help topics]
[About...]

Data Analysis Task

[Data File]

[Load...]
[Edit Header...]
[Display Replicate Files...]
[Edit Average File...]
[Print...]
[Next Data file]
[Tabulate Spectrum/Point...]
[Tabulate Spectrum/Mass...]
[Draw Total Ion Chart]
[Extract Ion Chart...]
[Tabulate Chart Raw Data to CSV...]
[Tabulate Chart CPS Data to CSV...]
[Export AIA format for Agilent LC/GC]
[Export Agilent LC/MSD raw data]
[Exit]

[Method]

[Data Correction...]
[Select Reports...]
[Load...] ---Offline Data Analysis only
[Save...] ---Offline Data Analysis only
[Save to Online] ---Offline Data Analysis only
[Check Dilution...] ---When ISIS is used
[View Summary...]
[Run Analysis Method] ---Offline Data Analysis only

[Calibrate]

- [Edit Calibration]
- [Print Blank Conc.]
- [Load Calibration...]
- [Save Calibration...]
- [Convert to Calibration Method] ---in Standard Addition only

[FullQuant]

- [Generate Report...]
- [Layout Custom Report...]
- [Print Custom Report...]
- [Update DataBase]
- [Integration Parameters...]
- [Integration Results]

[SemiQuant]

- [Generate Reports...]
- [Layout Custom Report...]
- [Print Custom Report...]
- [Update DataBase]
- [Edit SemiQuant Parameters...]
- [Internal Standard Correction...]
- [Blank Subtraction...]
- [Print Blank Conc]

[Isotope]

- [Generate IR Report...]
- [Generate ID Report...]
- [Edit IR Parameters...]
- [Edit ID Parameters...]

[Tools]

[DoList...]
[Configure DoList...]
[Copy Window...]
[Reset Window]
[Help topics]
[About…]

[Help]

[Help topics]
[About...]

Data Base Task

[DataBase]

- [Element.db]
- [Oxide.db]
- [Dimer.db]
- [Dcharge.db]
- [Hydride.db]
- [Argide.db]
- [Chloride.db]
- [Exit]

[View]

- [Element Information]
- [Interference Information]
- [AMU Information]

[Prev!]

[Next!]

[Index...]

[Help]

- [About ICP-MS DataBase...]

Report Pad

[File]

[Open]
[Print]
[Exit]

[Edit]

[Copy]
[Select All]

[Window]

[Cascade]
[Tile]
[Arrange Icon]

[Help]

[Help Topics]

Appendix B

This appendix includes descriptions and equations for Calibration, Quantitation, SemiQuantitation and Averaging Repetition Files.

Calibration

Weighted Regression

When data with more than one repetition is used to create a calibration curve, weighted regression can be selected. The count error for data collected in this manner is represented as the standard deviation of the counts. Usually the count error for higher concentrations is larger than that for lower concentrations. When weighted regression is selected, lower concentrations are given more weight because it is more desirable that the curve pass through points having lower error than points having higher error.

The weight of each point is calculated as follows.

$$w_i = S_i^{-2} / (\sum_i S_i^{-2} / n)$$

where

S_i: standard deviation of each point

n: number of point

In case of 1/count, s_i changes to 1/count.
However, if a count0 level exists, weighting is not possible.
In case of 1/conc, s_i changes to 1/conc.
However, if 0 concentration exists in calibration levels, weighting is not possible.

Only linear regressions can be weighted.

Internal Standard

When an internal standard is selected, the count of each data point in the calibration curve is divided by the ratio of the count per concentration of the internal standard of the same level.

$$y = y_\sigma / \left(y_i / x_i\right) \left(y = y_\sigma \text{ if } x_i = 0\right)$$

where

x_i: concentration of internal standard

y_i: count of internal standard

y_σ: count of sample data

This value is used as the measured value of y in the following sections.

Correlation Coefficient

This value is calculated using the following formula.

$$\tau = \frac{\sum_i \{(x_i - \bar{x})(y_i - \bar{y})\}}{\left\{\left[\sum_i (x_i - \bar{x})^2\right]\left[\sum_i (y_i - \bar{y})^2\right]\right\}^{1/2}}$$

where

$\bar{x}$: average of x_i

$\bar{y}$: average of y_i

x_i : measured value of x

y_i : measured value of y

This is available only for linear regressions.

Coefficients of Calibration Curves

Coefficients are calculated as follows.

where

n: number of points

x_i: numbered value of x

y_i: measured value of y

w_i: weight of each point (w_i=1.0 in the case of unweighted regressions)

1 y = ax

$$a = \frac{\sum_i x_i y_i w_i}{\sum_i x_i^2 w_i}$$

2 y = ax + b

$$a = \frac{n\left(\sum_i x_i y_i w_i\right) - \left(\sum_i x_i w_i\right)\left(\sum_i y_i w_i\right)}{n\left(\sum_i x_i^2 w_i\right) - \left(\sum_i x_i w_i\right)^2}$$

$$b = \frac{\sum_i y_i w_i}{n} - a\frac{\sum_i x_i w_i}{n}$$

3 $\mathbf{y = ax^2 + bx}$

$$a = \frac{\left(\sum_i x_i^3 w_i\right)\left(\sum_i x_i y_i w_i\right) - \left(\sum_i x_i^2 w_i\right)\left(\sum_i x_i^2 y_i w_i\right)}{\left(\sum_i x_i^3 w_i\right)^2 - \left(\sum_i x_i^2 w_i\right)\left(\sum_i x_i^4 w_i\right)}$$

$$b = \frac{\sum_i x_i y_i w_i - a\sum_i x_i^3 w_i}{\sum_i x_i^2 w_i}$$

4 $\mathbf{y = ax^2 + bx + c}$

$$a = \frac{S_{(x^2y)}S_{(xx)} - S_{(xy)}S_{(xx^2)}}{S_{(xx)}S_{(x^2x^2)} - \{S_{(xx^2)}\}^2}$$

$$b = \frac{S_{(xy)}S_{(x^2x^2)} - S_{(x^2y)}S_{(xx^2)}}{S_{(xx)}S_{(x^2x^2)} - \{S_{(xx^2)}\}^2}$$

$$c = \frac{\sum_i y_i w_i}{n} - b\frac{\sum_i x_i w_i}{n} - a\frac{\sum_i x_i^2 w_i}{n}$$

where

$$s_{(xx)} = \left(\sum_i x_i^2 w_i\right) - \frac{\left(\sum_i x_i w_i\right)^2}{n}$$

$$s_{(xy)} = \left(\sum_i x_i y_i w_i\right) - \frac{\sum_i x_i w_i \sum_i y_i w_i}{n}$$

$$s_{(xx^2)} = \left(\sum_i x_i^3 w_i\right) - \frac{\left(\sum_i x_i w_i\right)\left(\sum_i x_i^2 w_i\right)}{n}$$

$$s_{(x^2y)} = \left(\sum_i x_i^2 y_i w_i\right) - \frac{\left(\sum_i x_i^2 w_i\right)\left(\sum_i y_i w_i\right)}{n}$$

$$s_{(x^2x^2)} = \left(\sum_i x_i^4 w_i\right) - \frac{\left(\sum_i x_i^2 w_i\right)^2}{n}$$

5 log (y) = a (log x) + b

$$a = \frac{n\left\{\sum_i (\log x_i \log y_i) w_i\right\} - \left(\sum_i \log x_i w_i\right)\left(\sum_i \log y_i w_i\right)}{n\left\{\sum_i (\log x_i)^2 w_i\right\} - \left(\sum_i \log x_i w_i\right)^2}$$

$$b = \frac{\sum_i \log y_i w_i}{n} - a\frac{\sum_i \log x_i w_i}{n}$$

6 y = ax + b + bkg (Standard Addition)

$$a = \frac{n\left(\sum_i x_i y_i w_i\right) - \left(\sum_i x_i w_i\right)\left(\sum_i y_i w_i\right)}{n\left(\sum_i x_i^2 w_i\right) - \left(\sum_i x_i w_i\right)^2}$$

$$b = \frac{\sum_i y_i w_i}{n} - a\frac{\sum_i x_i w_i}{n} y_{bkg}$$

where

y_{bkg} : count of the background

7 y = ax +[blank]

$$a = \frac{\sum_i x_i (y_i - Blk) w_i}{\sum_i x_i^2 w_i}$$

where

Blk : number of counts in the calibration blank

Quantitation

Internal Standard

When an internal standard is selected in the current calibration curve, the count of the target ion of the sample data is divided by the ratio of the count per concentration of the internal standard in the sample data. In this calculation, the concentration of the internal standard in the first level of the calibration curve is used as the concentration of the internal standard in the sample data. The count value for each element reported on a quantitative report is therefore a count ratio.

$$y = y_\sigma / \left(y_i / x_i\right) \left(y = y_\sigma \text{ if } x_i = 0 \quad\right)$$

where

x_i: concentration of the internal standard of the first level in the calibration curve

y_i: count of internal standard

y_σ: count of the target ion

This value is used as the measured value of y in the following sections.

Calculating Concentration

Concentration of the target ion of the sample data is calculated as follows.

where

x: concentration of the target ion

y: measured count of the target ion

a: coefficient "a" in the calibration curve

b: coefficient "b" in the calibration curve

c: coefficient "c" in the calibration curve

1 y = ax

$$x = \frac{y}{a}$$

2 y = ax + b

$$x = \frac{y - b}{a}$$

3 $y = ax^2 + bx$

$$x = \frac{-b + \sqrt{b^2 + 4ay}}{2a}$$

4 $y = ax^2 + bx + c$

$$x = \frac{-b + \sqrt{b^2 - 4a(c - y)}}{2a}$$

5 log y = a (log x) + b

$$x = \left(\frac{y}{10^b}\right)^{\frac{1}{a}}$$

6 y = ax + b + bkg (Standard Addition)

$$x = \frac{b}{a}$$

7 y = ax + [blank]

$$x = \frac{y - Blk}{a}$$

where

Blk : number of counts in the calibration blank

DL

DL refers to a concentration equivalent to 3σB.

The equation for DL varies depending on the calibration formula.

If a linear equation such as y = ax + b is used:

DL = 3σB/a

Where,

3σB: value three times the standard deviation of the count at the 0 concentration level.

a: a in y = ax + b

The unit set in the calibration should be used and must not be changed.

Standard Deviation of the Concentration

When a linear regression is used, the standard deviation is calculated as follows.

$$SD = \sqrt{\frac{\sum_i x_i^2 - \frac{1}{n}\left(\sum_i x_i\right)^2}{n-1}}$$

where

n : number of sample repeats

x_i : concentration

Interpolation Formulas for Virtual-Internal-Standard Correction

These formulas are used to calculate the modification rates for all internal standards used for the latest calibration curve and the current sample data. The concentration level of the current data internal standards uses the value of level 1 of the calibration curve, as with the existing internal-standard correction. In the case of "Linear" and "Quadratic," one formula is created from all internal-standard modification rates. In the case of "Point to Point," a formula is created for every two internal standards.

$$Ri = \frac{Cps\_ci / Conc\_ci}{Cps\_si / Conc\_si}$$

where

Cps_ci, Conc_ci: CPS and concentration of the internal standard of the data last used to update the calibration curve

Cps_si, Conc_si: CPS and concentration of the internal standard of the current sample data (For the concentration, use the value set for level 1 of the calibration curve.)

Point to Point (Y = aX + b; Two adjacent internal standards are used; Default)

Linear (Y = aX + b; All internal standards are used.)

$$a = \frac{n\sum_i MiRi - \sum_i Mi \sum_i Ri}{n\sum_i Mi^2 - (\sum_i Mi)^2}$$

$$b = \frac{\sum_i Ri}{n} - a\frac{\sum_i Mi}{n}$$

$$Ra = aMa + b$$

where

Mi: Mass number of internal standards

n: Number of internal standards (two in the case of "Point to Point")

Ri: Modification rate of the internal-standard CPS between the standard data and sample data

Ma: Mass number of the element to be quantitatively analyzed

Ra: Virtual-internal-standard correction coefficient for correcting the CPS of the element to be quantitatively analyzed

Quadratic ($Y = aX^2 + bX + c$; All internal standards are used.)

$$a = \frac{S_{(M^2R)}S_{(MM)} - S_{(MR)}S_{(MM^2)}}{S_{(MM)}S_{(M^2M^2)} - \{S_{(MM^2)}\}^2}$$

$$b = \frac{S_{(MR)}S_{(M^2M^2)} - S_{(M^2R)}S_{(MM^2)}}{S_{(MM)}S_{(M^2M^2)} - \{S_{(MM^2)}\}^2}$$

$$c = \frac{\sum_i Ri}{n} - b\frac{\sum_i Mi}{n} - a\frac{\sum Mi^2}{n}$$

$$Ra = aMaMa + bMa + c$$

where

Mi: Mass number of internal standards

n: Number of internal standards

Ri: Modification rate of the internal-standard CPS between the standard data and sample data

Ma: Mass number of the element to be quantitatively analyzed

Ra: Virtual-internal-standard correction coefficient for correcting the CPS of the element to be quantitatively analyzed

$$S_{(MM)} = \sum_i Mi^2 - \frac{(\sum_i Mi)^2}{n}$$

$$S_{(MR)} = (\sum_i MiRi) - \frac{\sum_i Mi \sum_i Ri}{n}$$

$$S_{(MM^2)} = \sum_i Mi^3 - \frac{\sum_i Mi \sum_i Mi^2}{n}$$

$$S_{(M^2R)} = \sum_i Mi^2 Ri - \frac{\sum_i Mi^2 \sum_i Ri}{n}$$

$$S_{(M^2M^2)} = \sum_i Mi^4 - \frac{(\sum_i Mi^2)^2}{n}$$

Correct the CPS of the element to be quantitatively analyzed using the virtual-internal-standard correction coefficient.

$$Cps\_na = Cps\_sa * Ra$$

where

Cps_na: Corrected CPS of the element to be quantitatively analyzed

Cps_sa: CPS of the element to be quantitatively analyzed

Ra: Virtual-internal-standard correction coefficient for the CPS of the element to be quantitatively analyzed

SemiQuantitation

The counts used in semiquantitation were the counts of the most abundant point of the mass specified in the semiquant parameters panel for a given element.

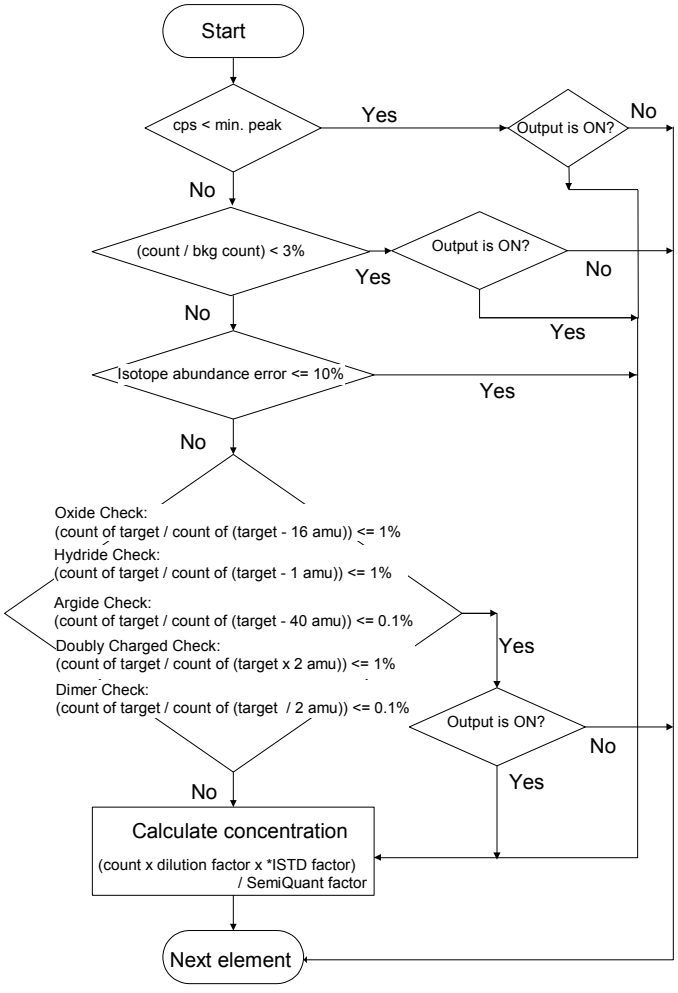
There are some exceptions to the execution of interference check protocol as follows:

Oxide check is not executed for ^{72}Ge.

Doubly charged ion check is not executed for ^{7}Li.

Dimer check is not executed for ^{7}Li, ^{24}Mg, ^{60}Ni, ^{72}Ge.

Argide check is not executed for ^{60}Ni, ^{72}Ge.



where

Auto Add Mode

*ISTD factor = (cps of ISTD in the STD) / (cps of ISTD in the sample)

Normal Mode

*ISTD factor = (concentration of ISTD in the sample) x (SemiQuant factor)
/ (cps of ISTD in the sample)

Averaging Repetition Files

When the repetition data files are averaged to create an average results file, the following formulas are used:

$$x_{avg} = \frac{\sum_i n_i x_i}{\sum_i n_i}$$

$$s_{avg} = \sqrt{\frac{\sum_i n_i \sum_i \bar{x}^2 - \left(\sum_i \bar{x}\right)^2}{\sum_i n_i \left(\sum_i n_i - 1\right)}}$$

where

$$\sum_i \bar{x} = \sum_i n_i x_i$$

$$\sum_i \bar{x}^2 = \sum_i \frac{n_i (n_i - 1) s_i^2 + (n_i x_i)^2}{n_i}$$

$\boldsymbol{x_{avg}}$: average count of the averaged results file

$\boldsymbol{s_{avg}}$: standard deviation of the averaged results file

$\boldsymbol{n_i}$: repetition number of the individual repetition file

$\boldsymbol{x_i}$: average count of the individual repetition file

$\boldsymbol{s_i}$: standard deviation of the individual repetition file

Appendix C

This appendix includes descriptions for the dead time calibration.

Dead Time Calibration

Dead time is the interval following the arrival of an ion at the detector, during which the counting of a pulse takes place. It is determined by the width of the multiple output pulse, electronic pulse widths and circuit recovery times. Another ion arriving during this time will not be recorded.

A typical value of dead time is 40 nsec

Dead time correction is performed in the following calculation.

$$N = n / (1-nT)$$

N: Count after correction (cps)

n: Count before correction (cps)

T: Correction parameter (= Dead Time)

NOTE Dead time calibration needs to be performed after replacement of the electron multiplier.

Execution of Dead Time Calibration

This section describes how to execute the Dead Time Calibration by using erbium (Er) solution. Indium (In) is also available for dead time calibration.

For executing the Dead Time calibration, follow the next procedure.

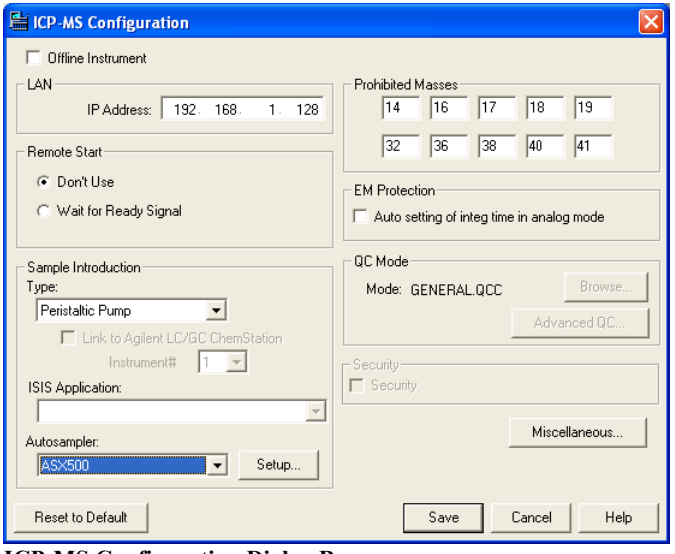
NOTE Dead time calibration needs to be performed after replacement of the electron multiplier.

1 **Perform a new EM and Adjust Discriminator tune.**

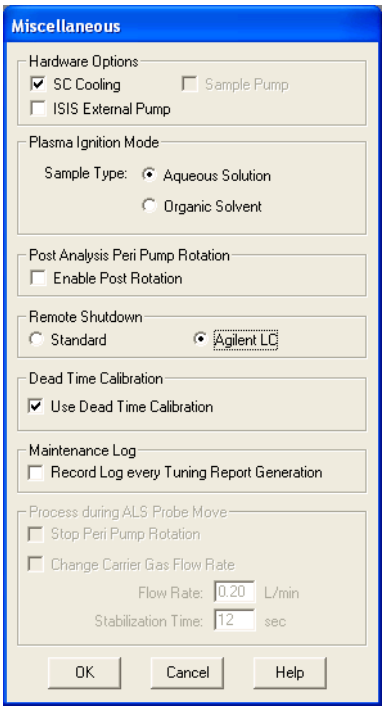
2 **Prepare the calibration standard solution for Dead Time Calibration.**

For example, 50ppb Er and 1ppm Er.

3 **Select the *Use Dead Time Calibration* in the *ICP-MS Configuration* dialog box.**
The *ICP-MS configuration* dialog box is displayed by selecting *Start >> Programs >> ICP-MS Chemstation >> Configuration >> Miscellaneous*.



ICP-MS Configuration Dialog Box



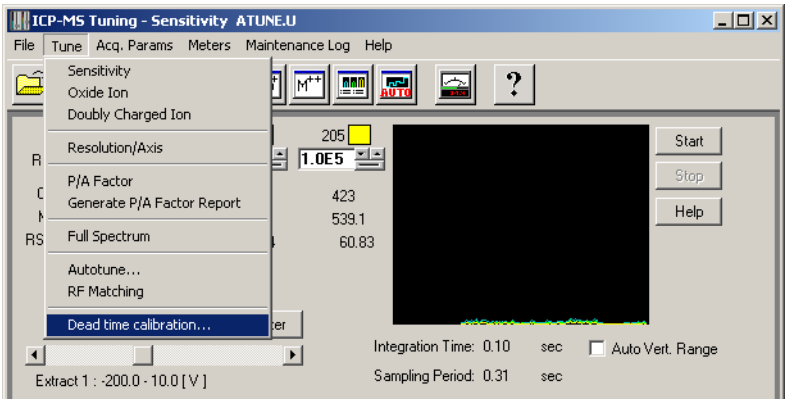
Select Use Dead Time Calibration.

4 Introduce the 50ppb Er solution.

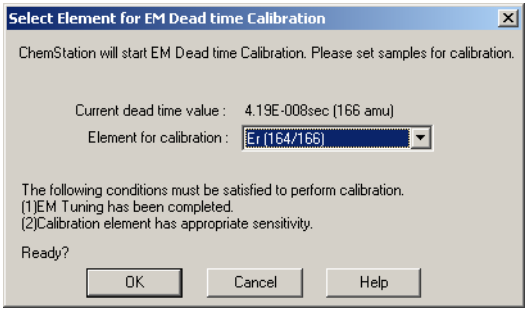
Tune the sensitivity to make the count of the major isotope ^{166}Er in the range of 50,000 - 1,000,000 count / 0.1 sec.

5 Select *Tune >> Dead time calibration..*

Select Element for EM Dead time Calibration dialog box is displayed.



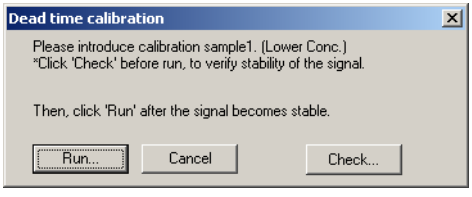
6 Select Er[164/166] then click OK..

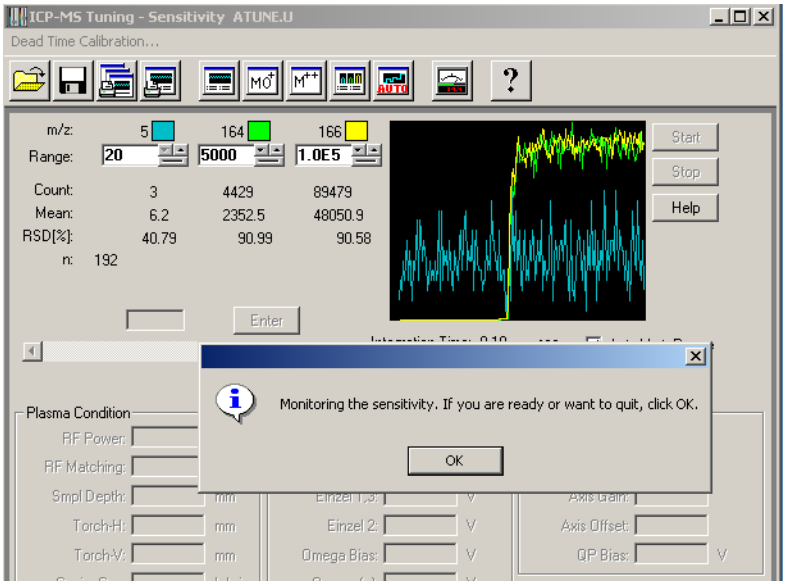


Select Element for EM Dead time Calibration dialog box

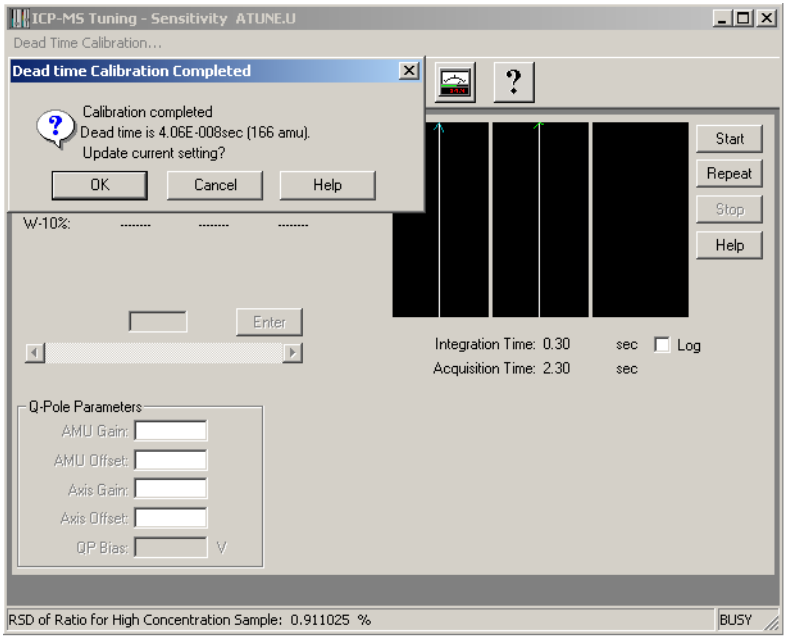
7 Check sensitivity for 50ppb Er

Select **Check**.. the sensitivity must be above 50,000 counts. Select **OK** then **Run**..





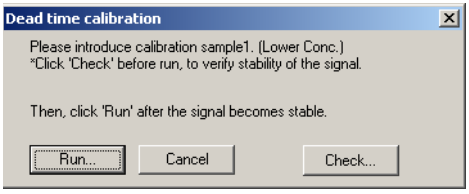
50ppb check.

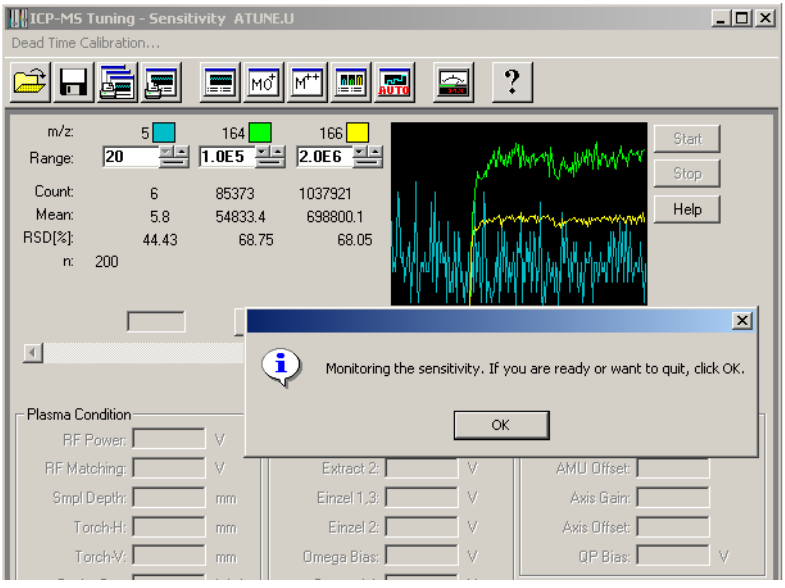


50ppb finished.

8 Introduce the 1ppm Er solution. Check sensitivity for 1ppm Er

Select **Check**.. note the increase in sensitivity with this higher concentration. Select **OK** then **Run**..

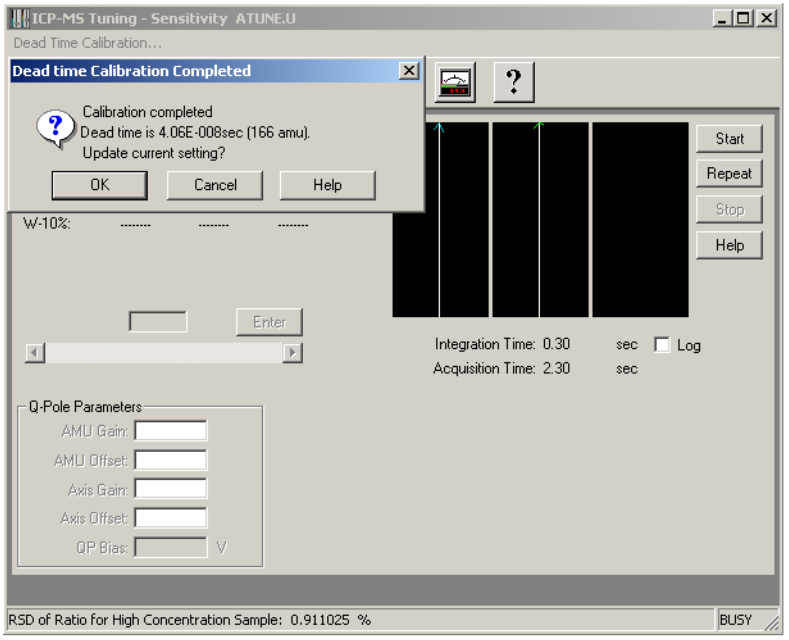




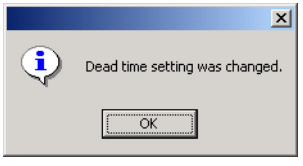
1ppm Check.

9 Select OK to save the new dead time.

Typical Dead Time 30 - 60 nano seconds



Calibration complete



Appendix D Compression and Decompression of Files

For troubleshooting purposes, files such as equipment maintenance log, data, method, calibration, and sequence files can be compressed into zip files.

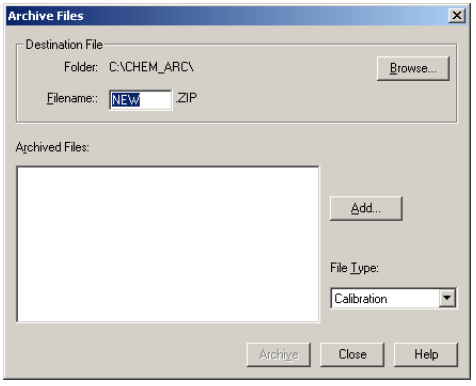
Compressed files can be decompressed.

Compressing Files

Files such as data, method, calibration, and sequence files can be compressed using the following method.

1 Select *Top* window >> *Tool* >> *Archive Files*.

The ***Archive Files*** dialog box will appear.

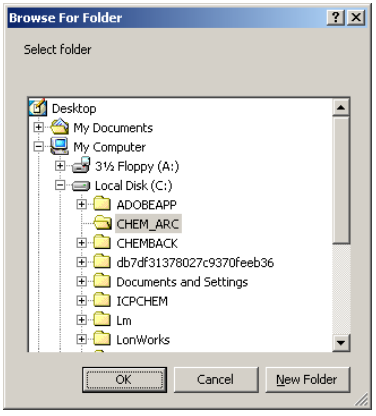


Archive Files Dialog Box

2 Enter a file name in the *File Name*: text box.

3 Click *Browse* to change the destination folder, if necessary.

When the ***Browse For Folder*** dialog box appears, select a destination folder and click ***OK***.



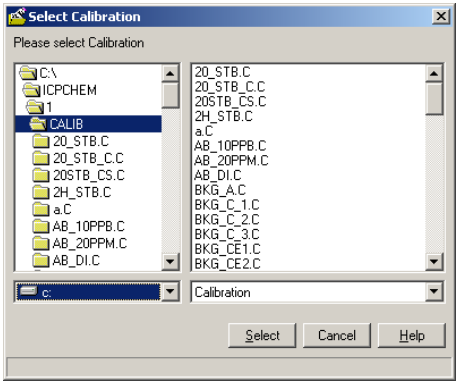
Browse For Folder Dialog Box

4 Select the type of file to be compressed in the *File Type* list box.

5 Click *Add* and select the file to be compressed.

The list section on the upper right displays files of the type selected in step 4. Select a file and click ***Select***.

If you cannot find the file that you are looking for, change the search location using the folder list in the upper left section or the drive list in the lower left section.



Selection of File

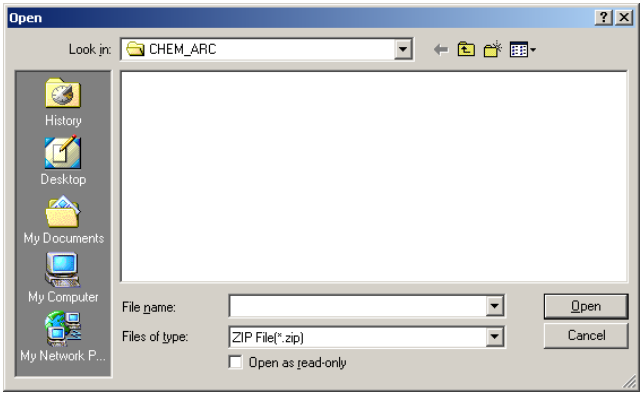
6 Click *Archive.*

The selected file will be compressed.

Decompressing a File

1 **Select *Top* window >> *Tool* >> *Extract Files*.**

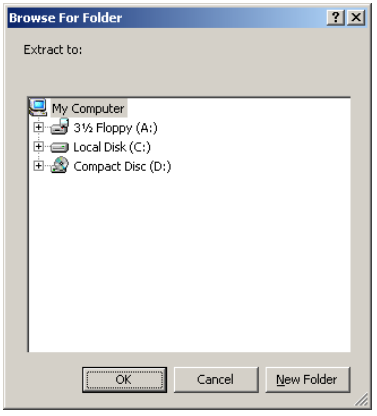
The ***Open*** dialog box will appear.



Open Dialog Box

2 **Select the location of the compressed file and click *Open*.**

The ***Browse For Folder*** dialog box will appear.



Browse For Folder Dialog Box

3 **Select the folder in which to place the decompressed file and click *OK*.**

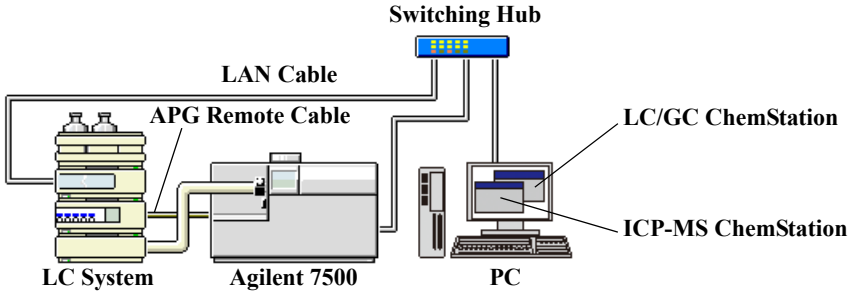
The selected file will be decompressed.

Appendix E Precautions on Using the ICP-MS ChemStation with the LC/GC ChemStation

Installing the ICP-MS ChemStation and the LC/GC ChemStation to a single PC makes it possible to execute an LC/GC ChemStation method in an ICP-MS ChemStation sequence.

Connecting the Agilent 7500 and the LC/GC

Connect the Agilent 7500 and the LC/GC as shown below:



- Use an APG remote cable to connect the Agilent 7500 and LC/GC.
- Connect the PC and the ICP-MS or LC/GC with a LAN cable via a switching Hub. In general, use straight cables only. If the Hub is AUTO-MDIX-compatible, either straight or crossover cables may be used.

Refer to the instruction manual for each instrument for information on making connections. Note that Agilent 7500 ICP-MS or LC/GC Instruments with the GPIB interface cannot be synchronized with the LC/GC ChemStation.

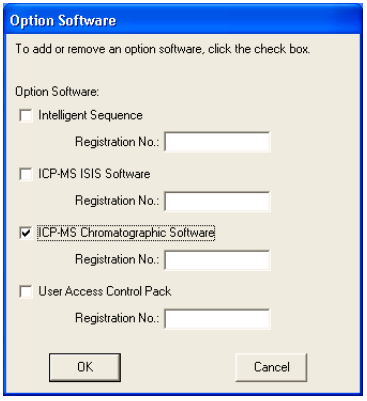
Precautions on Installing the LC/GC ChemStation on a PC on which the ICP-MS ChemStation is already installed

If the optional ICP-MS chromatographic software is installed on the PC, take the following steps to uninstall the chromatographic software and install the LC/GC ChemStation.

1 **Select *Start --> All programs --> ICP-MS ChemStation --> Add (Delete) Optional Software* from the Windows taskbar.**

This displays the ***Optional Software*** dialog box.

2 **Uncheck the *ICP-MS Chromatographic Software* checkbox and click *OK*. This will uninstall the ICP-MS chromatographic software.**



3 **Install the LC/GC ChemStation. Refer to the LC/GC ChemStation Operation Manual for how to install this software.**

When the LC/GC ChemStation has been installed, reinstall the ICP-MS chromatographic software, if necessary.

4 **Open the *Optional Software* dialog box in the same way as described in Step 1.**

5 **Check the *ICP-MS Chromatographic Software* checkbox, enter a Registration No., and click *OK*.**

Precautions during LC/GC Startup

Open the LC/GC after the ICP-MS ChemStation enters Analysis mode. If you open the LC/GC first, initialize the instrument from the LC/GC ChemStation. Refer to the LC/GC ChemStation Operation Manual for initialization directions.

Setting Configuration

You must change the configuration in the ***ICP-MS Configuration*** dialog box before using the LC/GC ChemStation with the ICP-MS ChemStation. Refer to "Configuration" in Chapter 2 for more information.

Precautions During Execution of a Sequence

Never perform LC/GC ChemStation operations while executing a sequence. Any such attempt will halt the sequence.