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Agilent Technologies

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4000 GC/MS **Version 6**

Software Operation Manual



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Contents

Getting Started.....	5
Additional Manuals/Help	5
 Workstation Toolbar.....	 7
Overview	7
Elements of the Workstation Toolbar	8
Launching Applications from the Workstation Toolbar	11
Workstation Toolbar Options	11
 System Control	 17
Overview	17
Navigating the 4000 MS Instrument Window	17
Manual Control Tab	36
Auto Tune Tab	45
Temperatures Tab	50
Diagnostics Tab	53
Startup/Shutdown Tab	58
Acquisition Tab	63
Parameters That Affect MS Operation	66
Using 4000 MS Methods in System Control.....	67
Effects of Hardware Configuration Changes	70
3800 GC System Control Window	70
Configuring the GC/MS System	76
 Building GC/MS Methods.....	 97
Using Star Assistant to Create a New Method	97
Method Builder Window	99
4000 MS Method Section	104
3800 GC Method Section	145
 Acquiring GC/MS Data	 159
Injecting a Single Sample	159
Injecting Multiple Samples	167
Automation File Editor	175
Automated MS Report Generation	185
 Security Administration	 187
Overview	187
Passwords	187
Application Locking.....	188
File Revision Settings	189

4000 MS Ionization Techniques.....	191
Choices of Ionization Mode	191
Internal Electron Ionization	191
Internal Positive Chemical Ionization.....	194
External Electron Ionization.....	199
External Positive or Negative Chemical Ionization.....	202
Hybrid Positive or Negative Chemical Ionization.....	206
 Ion Preparation Techniques	 209
MS/MS - Tandem Mass Spectrometry	209
Selected Ion Storage	214
 System Control Command Reference	 217
System Control Menus	217
 4000 MS System Control Command Reference	 227
Display	227
System Control Tabs	228
Manual Control	229
Auto Tune	234
Temperature(s).....	239
Diagnostics	241
Startup/Shutdown	244
Acquisition	246
 3800 GC System Control Command Reference	 249
Status and Control Window	249
8400/8410 SampleList Window Extensions	266
 4000 MS Method Builder Command Reference	 271
Internal EI and CI.....	272
 3800 GC Method Command Reference.....	 283
3800 GC AutoSampler	283
3800 GC Sample Delivery	294
3800 GC Injector.....	294
3800 GC Flow/Pressure	297
3800 GC Column Oven	301
3800 GC Detector.....	302
3800 GC Output.....	309
3800 GC Data Acquisition	310
 Automation File Editor Command Reference	 311
Menus	311
Main Toolbar.....	312
Open Automation File Dialog Box	313
Save Automation File As Dialog Box.....	314
Print Setup Dialog Box	315
Edit Notes Dialog Box.....	315
RecalcList Window	316

SampleList Window	322
SequenceList Window	328

Getting Started

This manual shows you how to acquire data, build methods, and operate the Varian 4000 GC/MS using the Varian MS Workstation. The manual also describes the configuration of the 3800 GC for standard Ethernet communication with the 4000 MS and MS Workstation's System Control application. Use this manual with the other manuals supplied with your MS Workstation and your 3800 GC. All of the information included in this manual is also available in the On-Line Help section of the software.

Additional Manuals/Help

Other sources of information are available to help you get the most from this product.

4000 GC/MS Users Guides

These Users Guides are tailored to help the user get started with the 4000 GC/MS system in any of its three ionization modes. There is advice on configuring and starting up the GC/MS. Then there is advice on tuning, building methods, and acquiring data. There are also discussions of the ionization and ion preparation (MS/MS) methods that can be used in the configuration.

- 4000 MS Internal Ionization Users Guide
- 4000 MS External Ionization Users Guide
- 4000 MS Hybrid Ionization Users Guide

4000 GC/MS Hardware Operation Manual

The Hardware Operation Manual provides the necessary information for installing, maintaining, using and repairing your 4000 GC/MS System. All of the information included in this manual is also available in the On-Line Help section of the software.

MS Workstation Software Reference Manual

This Manual provides a practical way to quickly learn how to perform basic tasks using the MS Workstation Software. These operations include displaying and reviewing chromatographic and mass spectral data, library searching, and quantitative analysis. Although these tutorials use Saturn 2000 ion trap files, they illustrate the principles of the Varian WS software.

3800 GC Operator's Manual

This manual is included on the Varian MS Workstation CD-ROM and describes in full detail the GC method, instrument operation, and the process of connecting your GC to a PC or to an existing network.

Context-Sensitive Help

The MS Data Review, Method Builder and System Control sections of the software contain Context-Sensitive Help. Help can be obtained simply by positioning the mouse pointer on the item of interest, right-click and select

What's This?

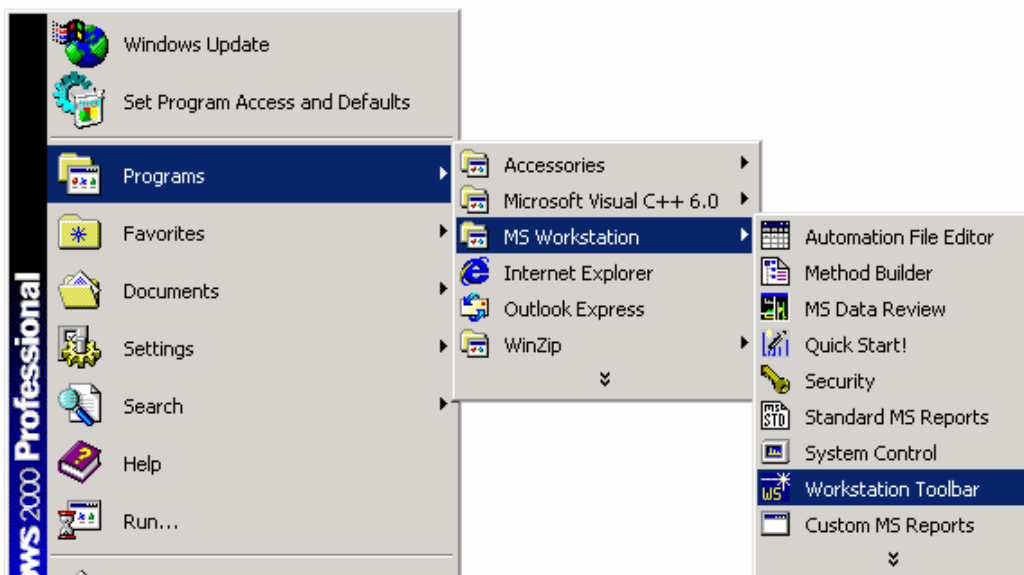
Workstation Toolbar

Overview



The 4000 GC/MS Workstation is a suite of applications for controlling chromatographs, collecting data from chromatograph detectors, and analyzing those data. The Workstation Toolbar provides quick and easy access to the 4000 GC/MS Workstation applications. When activated, the Workstation Toolbar behaves like the Windows Taskbar. It can be docked on any of the four sides of the display screen and other Windows programs will not cover or go behind it when they are opened in full screen mode.

If the Workstation Toolbar is not already opened on your 4000 GC/MS Workstation, open it from the Windows Start Menu.

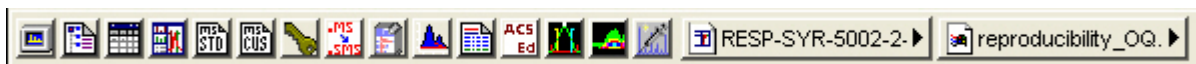


Elements of the Workstation Toolbar

Application Buttons give immediate access to the selected application. You can display them as large buttons



or as small buttons



Tooltips show the application name when the cursor rests on the Application Button.

Quick Link Buttons provide menu selections of operations to be performed on the listed file.

Application Descriptions give brief descriptions of the application that will be opened when the cursor rests on the Application Button.

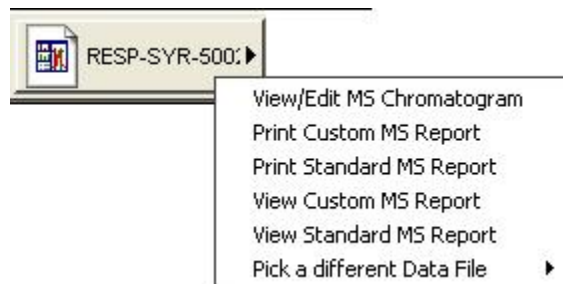
Application Buttons

System Control/ Automation		Monitor instrument status, and perform automated injections and batch recalculations.
View/Edit Methods		View and edit instrument operation, data acquisition, and data handling methods.
Edit Automation Files		Do off-line editing of SampleLists, RecalcLists and SequenceLists.
Review/Process MS Data		Review chromatograms and spectra, perform library searches, and review and process quantitation results.
Standard MS Reports		Create, edit, and view standard MS reports.
Custom MS Reports		Create, edit, and view customized MS reports.
Security Administration		Set 4000 GC/MS Workstation security options and passwords.
SMS/MS File Conversion		Convert data files between DOS and Windows formats.
Batch Reporting		Generate standard reports for a group of Data Files by dragging and dropping them on the Batch Report Window.
View/Edit Chromatograms		Review standard GC chromatograms, interactively edit data handling parameters, and recalculate results.
Standard Chrom Reports		Preview standard chromatogram and results reports.
Compound Set Editor		
Quick Start		Run a sample without a Sample List.

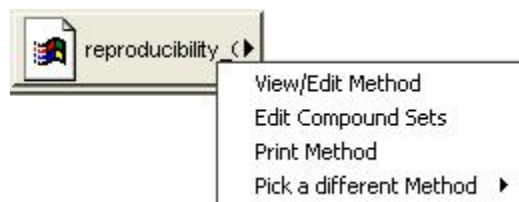
When additional 4000 GC/MS Workstation options are installed, such as StarFinder and Star Custom Report Writer, other application buttons may be added to the Workstation Toolbar.

Quick Link Buttons

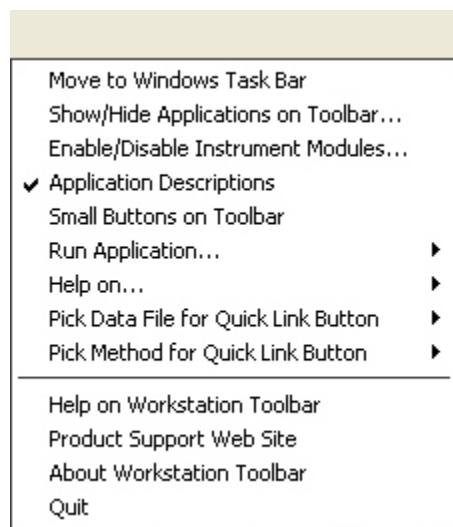
Most Recently Used Data File. Click this to display operations that can be performed on the Most Recently Used Data File. A different data file can be selected from a list of most recently used files.



Most Recently Used Method. Click this displays a menu of operations that can be performed on the Most Recently Used Method. A different method can be selected from a list of most recently used methods.



Workstation Toolbar Options



Right-click on the empty area of the bar to display the Toolbar Options. A menu displays Workstation Toolbar options and operations.

Launching Applications from the Workstation Toolbar

Using the Application Icon Buttons



4000 GC/MS Workstation applications appear as icon buttons on the Workstation Toolbar. As you move the mouse over these buttons, the name of the application is shown in a tooltip window below the cursor.

Click once on any icon button in the Workstation Toolbar to launch the corresponding application.

Using the Data File and Method Quick Link buttons



The two buttons to the right of the application buttons are Quick Link buttons. These two Quick Link buttons correspond to recently used Data Files and Methods. On each Quick Link button is the name of the file associated with that button. When you click on the **Quick Link** button, a menu is displayed showing operations that can be performed on the corresponding file.

The Quick Link buttons in the Workstation Toolbar are always updated with the most recently used Data File and Method. If the Data File or Method you wish to use is not displayed in the **Quick Link** button, you can choose from the eight most recently used files by selecting "Pick a different Data File (or Method)" from the Quick Link button menu.

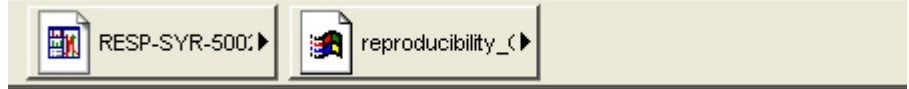
Workstation Toolbar Options

Moving the Workstation Toolbar

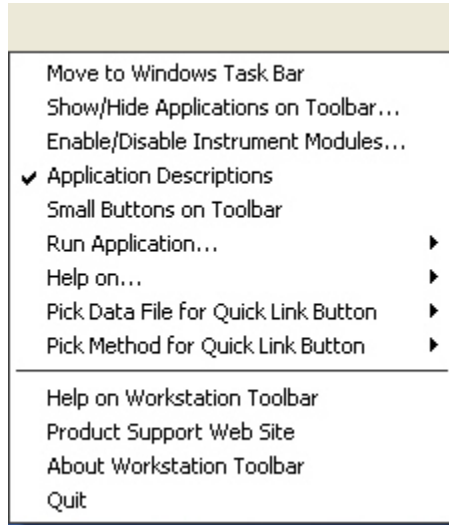
The Workstation Toolbar can be moved to any edge of the Windows screen. Click on a portion of the Workstation Toolbar that does not contain application or Quick Link buttons and drag the toolbar to the edge of the screen that you desire. When you release the mouse, the toolbar will remain on that edge. The Workstation Toolbar remembers its location the next time it is started.

Additionally, you can display the Workstation Toolbar as a Windows Taskbar icon. To do so, select **Move to Windows Taskbar** from the Workstation Toolbar options menu. Taskbar icons appear in the lower right (or bottom) of the Windows Taskbar (the bar on which the "Start" button appears). When displayed as a Taskbar icon, the toolbar no longer takes up space on the screen. When you click on the **Workstation Toolbar** icon, the options menu is displayed.

The Workstation Toolbar Options Menu



When you move the mouse over an area of the Workstation Toolbar not containing application or QuickLink buttons, right-click to display the Toolbar option menu.

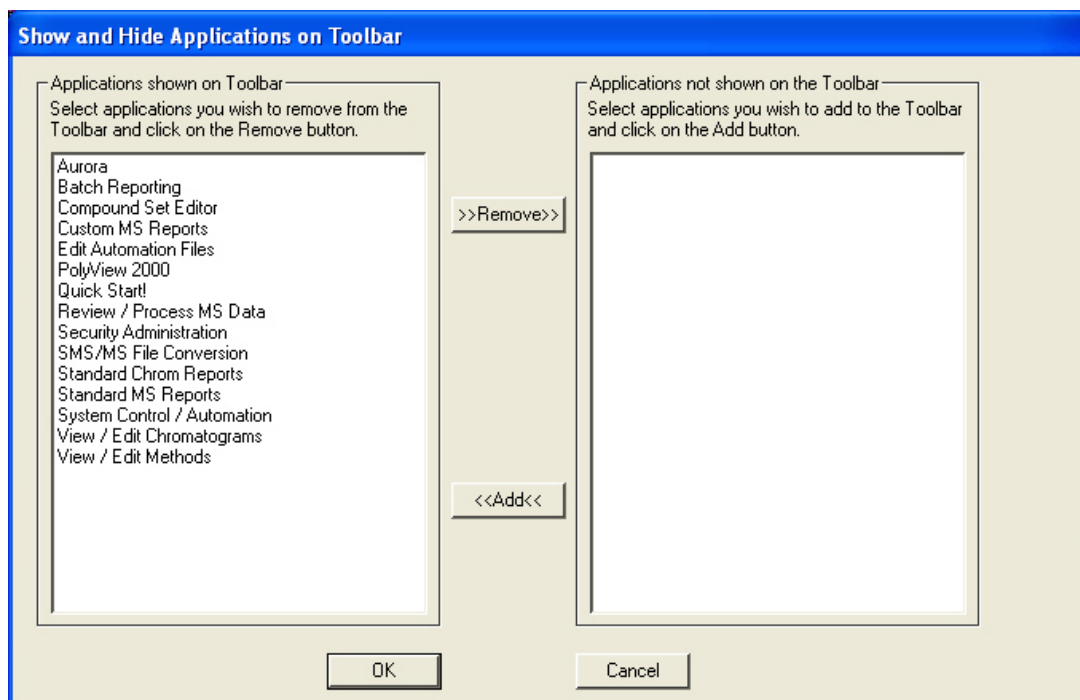


Move to Windows Taskbar

You can display the Workstation Toolbar as a Windows Taskbar icon. Taskbar icons appear in the lower right (or bottom) of the Windows Taskbar (the bar on which the “Start” button appears). When displayed as a Taskbar icon, the toolbar no longer takes up space on the screen. When you click on the **Workstation Toolbar** icon, the options menu is displayed.

Show/Hide Applications on Toolbar

Selecting this option displays the Show and Hide Applications on the Toolbar dialog box.



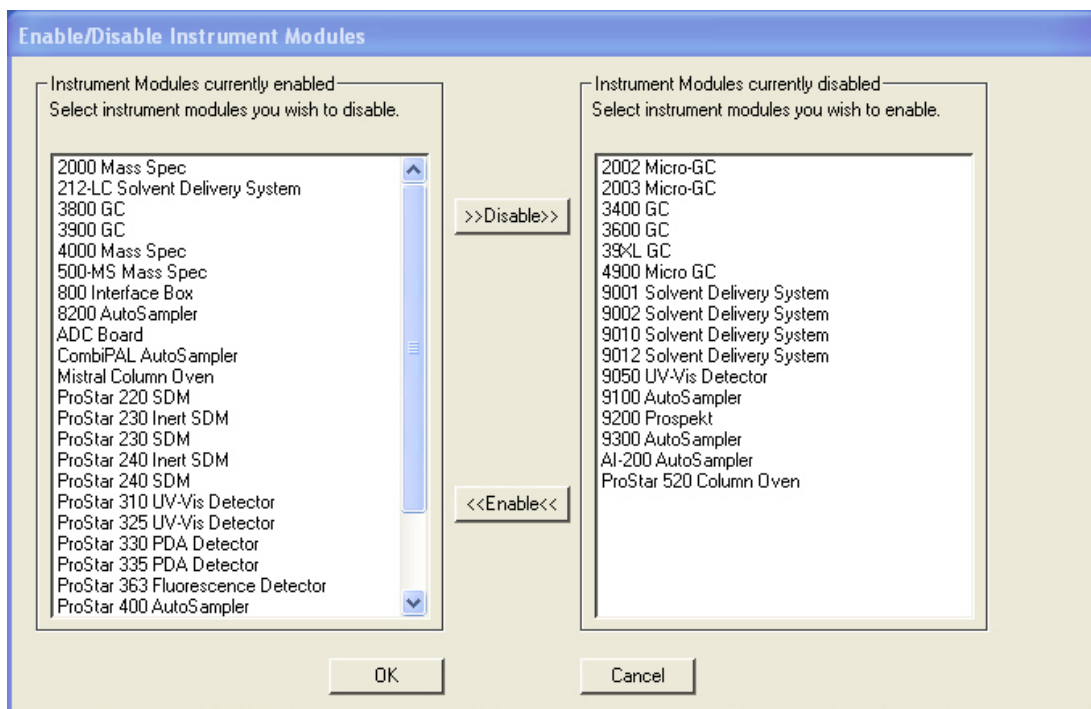
Use this dialog box to select which 4000 GC/MS Workstation applications are represented by icons on the Workstation Toolbar. The box on the right shows all applications that are currently displayed in the toolbar. The box on the left shows all applications that are installed but not displayed in the toolbar.

To remove an icon from the toolbar, select it from the top list and click on the **Remove** button.

To add an icon to the toolbar, select it from the bottom list and click on the **Add** button.

Enable/Disable Instrument Modules

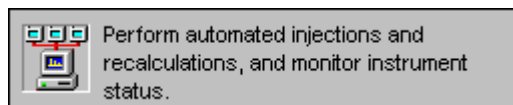
Select this option to display the Enable/Disable Instrument Modules dialog box.



Use this to select which 4000 GC/MS Workstation instrument modules are available. When an instrument module is available, it appears in System Control if the corresponding instrument is connected and turned on, you can create a Method section for it, and format reports.

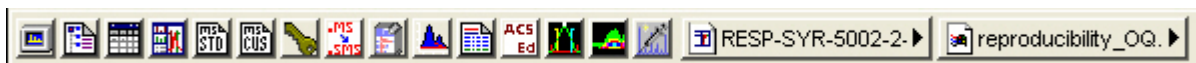
The list on the left shows all instrument modules that are currently installed and enabled in the 4000 GC/MS Workstation. The list on the right shows all instrument modules that are installed but not enabled. To disable an instrument module, select it from the top list and click **Disable**. To enable an instrument module, select it from the bottom list and click **Enable**.

Application Descriptions



When this menu item is checked, application descriptions are displayed when you move the mouse over application buttons in the Workstation Toolbar.

Small Buttons on Toolbar



Click this to display small buttons on the Workstation Toolbar.

Run Application

This hierarchical menu item lists all applications showing on the Workstation Toolbar. When you select an item from this list, the corresponding application is launched.

Help on

This hierarchical menu item lists all applications showing on the Workstation Toolbar. When you select an item from this list, the online help corresponding to the application is displayed.

Pick Data File for Quick Link Button

This hierarchical menu item lists the eight most recently used Data Files in order of use. When you select a Data File from this list, the Data File currently displayed in the Quick Link button is changed to the selected file.

Pick Method for Quick Link Button

This hierarchical menu item lists the eight most recently used Methods in order of use. When you select a Method from this list, the Method currently displayed in the Quick Link button is changed to the selected file.

Help on Workstation Toolbar

Displays the help for the open application.

Product Support Web Site

If you have Internet access and a web browser installed on your computer, this option will automatically open the 4000 GC/MS Workstation Product Support Web Site. This has the latest software and documentation updates for the 4000 GC/MS Workstation products, along with additional notes, tips, and answers to frequently asked questions. Visit this site periodically to see if new information is available that may be pertinent to you.

About Workstation Toolbar

The About box for the Workstation Toolbar contains information about the software version, installation information, and a list of the instrument control modules that you have installed.

Quit

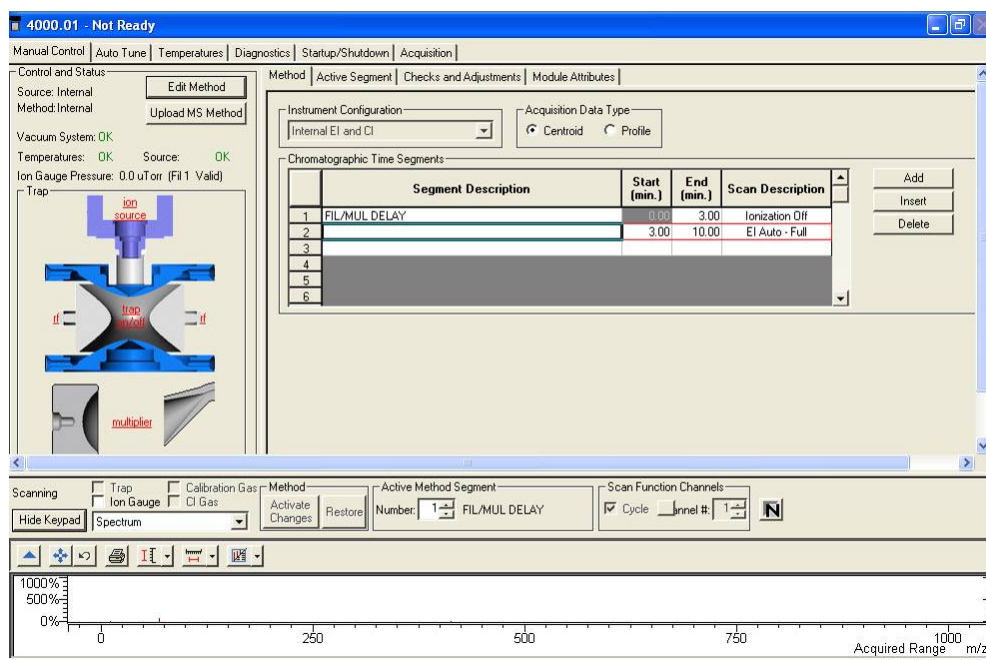
Closes the Workstation Toolbar application. If you elected to run the Workstation Toolbar automatically when Windows starts, the Workstation Toolbar will reappear the next time you start Windows.

System Control

Overview

This section introduces the features of System Control. Later sections explain how to use these features

Navigating the 4000 MS Instrument Window



System Control features include System Control Menu bar, System Control Toolbar, 4000 MS Keypad Dialog, Chromatogram/ Spectrum Toolbar, and Chromatogram/Spectrum Displays.

File Menu

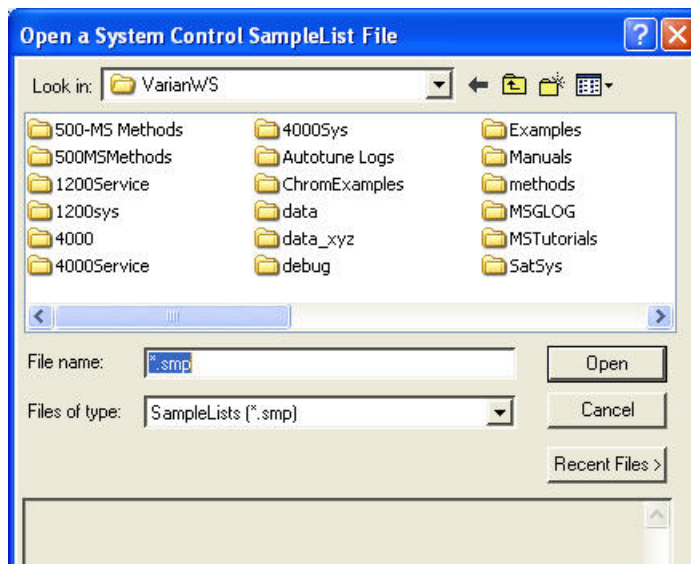
Activate Method...
Upload Active Method from Modules...
New SampleList...
Open SampleList...
New RecalcList...
Open RecalcList...
New SequenceList...
Open SequenceList...
Print
Printer Setup...
☒ Remember Last Open Files
Exit

Activate Method - Choose a method from those already prepared. This will set the conditions for the GC, MS, AutoSampler and data handling. Other sections will also be activated if they are part of the method.

Upload Active Method from Modules - Get the conditions from the existing modules to be saved in a new or existing method. This method becomes the active method with the conditions of the active modules.

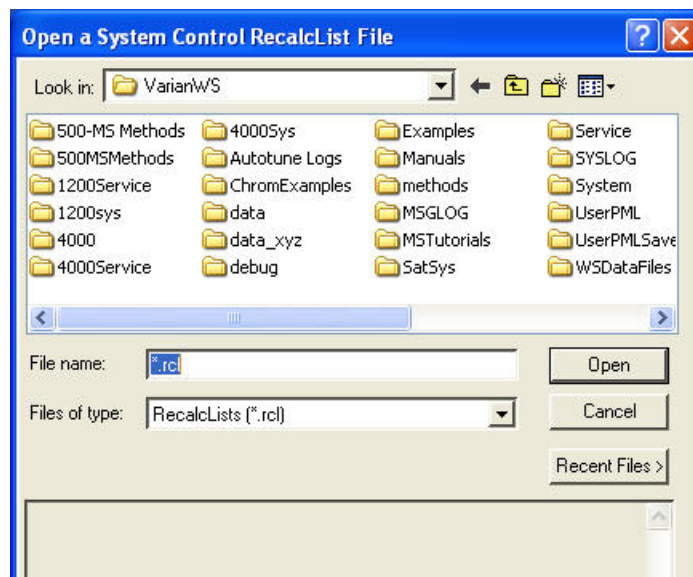
New SampleList - Open the dialog box to create a sample list.

Open SampleList - Select and activate an existing sample list. The sample list dialog box is shown below.



New RecalcList - Open the dialog box to create a Recalculation list.

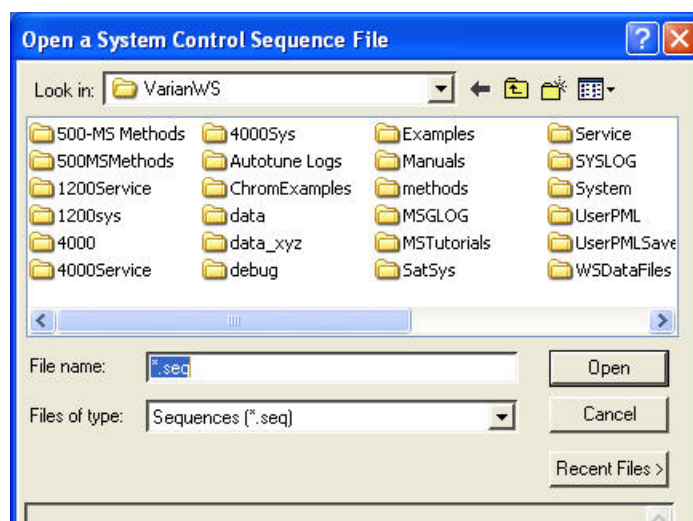
Open RecalcList - Select and activate an existing Recalculation list.



NOTE: You may process a RecalcList either in System Control or in MS Data Review. In MS Data Review use the menu command **Quantitation... Process/Review RecalcList** to access this feature. Also note that processing of a RecalcList takes more time when done from System Control but has the advantage of allowing AutoLink functions including automated reporting with templates such as EnviroPro, ToxProPlus, and Multicompound Reports.

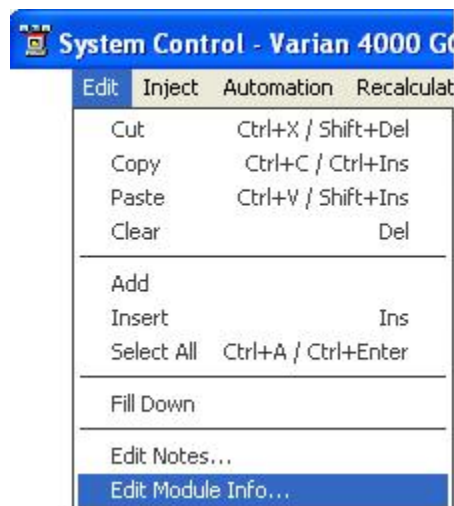
New SequenceList - Open the dialog box to create a Sequence list.

Open SequenceList - Select and activate an existing Sequence list. The SequenceList dialog box is shown below.

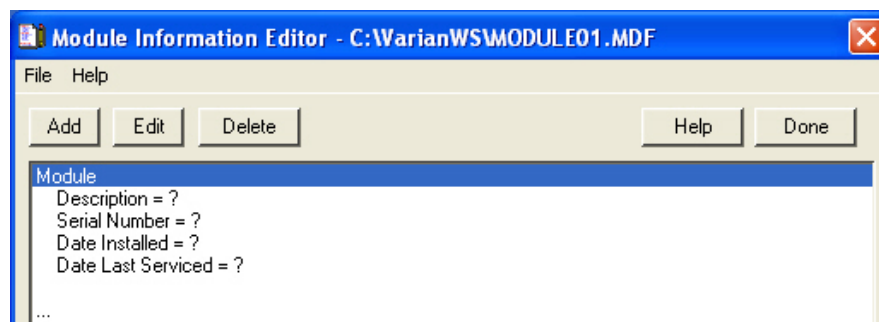


Edit Menu

Click **Edit** and select Edit Module Info.

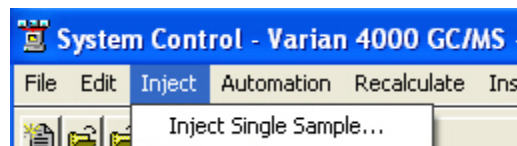


Each module (3800 GC, 4000 MS, or Combi PAL AutoSampler) has a message log and documentation screen. Use Module Information Editor to keep a record of performance, maintenance, hours used, or other comments.



Use the **Add**, **Edit**, and **Delete** buttons to record your comments.

Inject Single Sample Menu



Use Inject Single Sample o to enter sample information before making an injection. The 4000 Module window must be in acquisition mode for the system Not Ready to convert to Ready before making an injection. To process the sample by either a manual injection or an autosampler, click **Inject** in the bottom left corner of the dialog box.

Sample Name	Sample Type	Cal level	Inj.	Injection Notes	AutoLink	Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Sample	Analysis		1	none	none	1	1.0	Pos 1	1	0	1	1	none

Inject the Sample using the Method:

C:\VarianWS\startup1.mth

☐ Clear Coefficients before Calibrating

From the menu select the Sample Type. The following shows the choices.

Sample Name	Sample Type
Default Sample	Analysis
	Analysis
	Calibration
	Verification
	Baseline

Inject the Sample using

Do the same for injection notes. Any notes that you enter are saved with the data file. The **AutoLink** button works in the same way.

AutoLink Parameters	
Command	Other parameters
Browse...	OK Cancel

Enter a command to execute a program after the data file acquisition is completed. For example, you can activate a custom MS Report to print the sample reports. To run a Custom MS Report in automation, enter the directory path and the name of the Custom MS Report template (for example C:\VarianWS\EPA525.swt). Note that Custom MS Reports templates should always be stored in the VarianWS directory. You can use the browse command can to select the command executable file. Select **OK** after you make your selection.

Automation Menu

The following shows the Automation menu and describes the functions.

System Control - Varian 4000 GC/MS - Not Ready	
File	Edit
Inject	Automation
Recalculate	Instrument
W	
- Begin SampleList - Begin Sequence - Begin At Selected SampleList Line... - Begin At Selected Sequence Line... - Suspend Automation - Resume Automation - Stop Automation - Reset Modules ☒ Enable Automated Printing	

Begin SampleList - Automation starts at line 1 and ends at the last line. A SampleList must be open from the File menu to use this feature.

Begin Sequence - Change methods and sample lists during automation. An example of this might be to run a set of samples using Internal EI and then the same set of samples using a different MS method for Internal PCI. A Sequence must be open from the File menu to use this feature.

Begin At - Starts automation at a particular sequence or SampleList line and runs until the last line is completed. A SampleList or SequenceList must be currently open to use this feature.

Suspend Automation - Stops automation at the last completed sample.

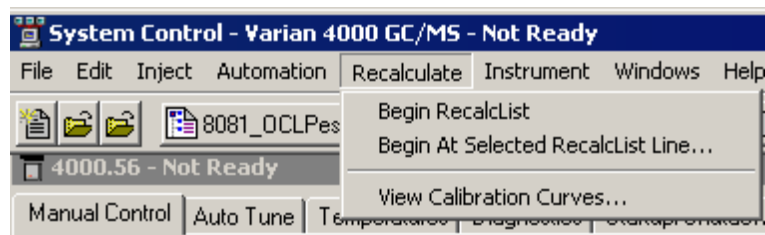
Resume Automation - Starts automation after it has been suspended. Automation starts from where it was suspended.

Stop Automation - Ends automation immediately and resets all modules.

Reset Modules - Brings the various modules to their ready states.

Enable Automated Printing - Allows the printer functions to work during automation.

Recalculate Menu

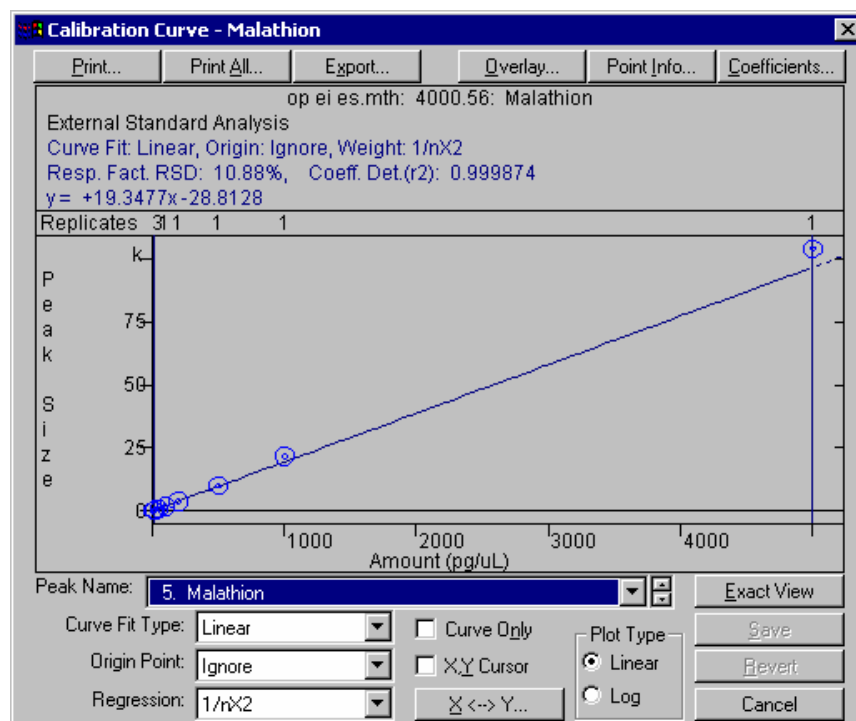


Begin RecalcList - Start an automated Recalculation. A Recalculation list must be prepared and opened under the File menu to do this.

Begin at Selected RecalcList Line - Process a Recalculation list starting with a chosen line and proceeding with the rest of the list.

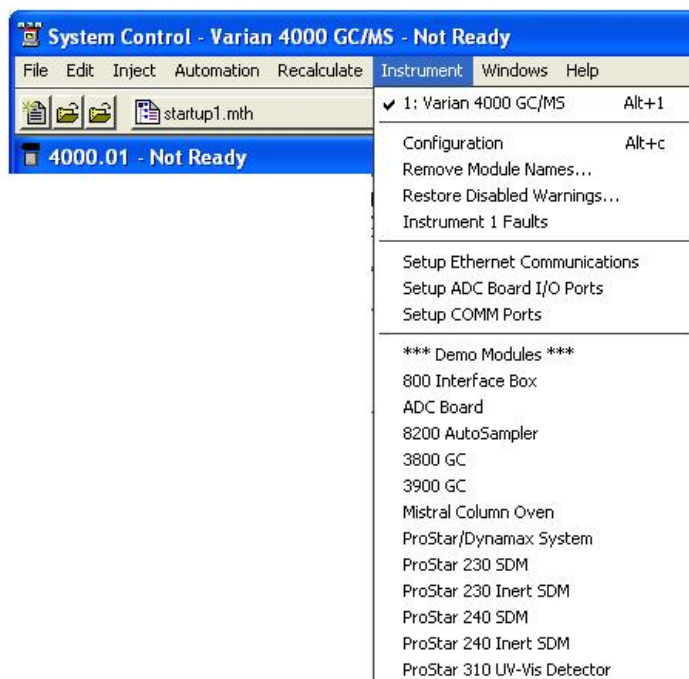
View Calibration Curves - View any of the curves for the calibrated compounds in the active method. An example of the calibration curve screen is given below. For a complete explanation of this screen, go to the Quantitation section.

NOTE: Recalculation may be performed in System Control or in MS Data Review under the menu command **Quantitation Process/Review Recalc List**. Processing a Recalc List takes more time when done from System Control than in MS Data Review but has the advantage of allowing AutoLink functions such as automated reporting with Custom MS Reports templates such as ToxProPlus or EnviroPro.



Instrument Menu

The Instrument menu:

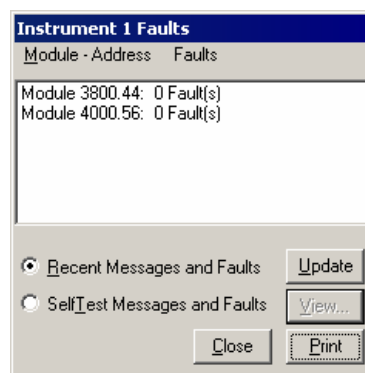


Varian 4000 GC/MS - The configured instrument.

Configuration - Add new modules to the existing instrument. This is necessary if additional hardware is added such as a Combi PAL AutoSampler.

Remove Module Names - Remove any associations between Module Names and Module Addresses. This allows you to connect a Module with a different Module Name at that address. The next time the associated Module connects at that address, you will be prompted to select the correct name for the Module.

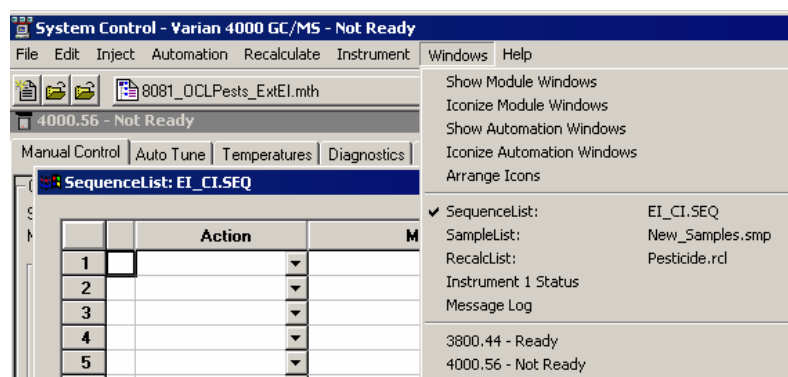
Instrument 1 Faults - See if there are any faults in any of the modules contained in Instrument 1. An example of this screen is shown. Use the update key to periodically check for new faults without opening and closing the dialog box. 4000 MS faults are not reported in this window.



Setup Ethernet Communication - Set up the communication between the 3800 GC and System Control. Depending on your network status go to one of the following sections for more details on this operation at the end of the System Control section: “*Configuring the 3800 GC Communication (No Company Network)*” on page 83 or “*Configuring the GC for a Company Network*” on page 85.

Windows Menu

Use the Windows menu to view other System Control windows.



Show Module Windows - See all module windows configured in the instrument. In this example the modules available are the 3800 GC and the 4000 MS.

Iconize Module Windows - The individual module windows become icons at the bottom of the screen.

Show Automation Windows - Click to view the automation screens that are open. These are the SampleList, RecalcList, and SequenceList.

Iconize Automation Windows - Transform open automation screen windows into icons so you can access windows that were behind the automation windows.

Arrange Icons - Arranges the icons, if any, in a row at the bottom of the System Control window.

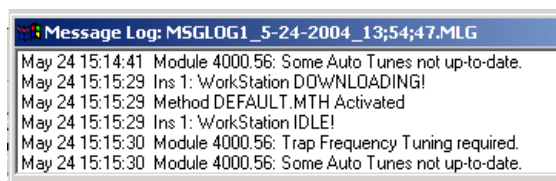
SequenceList - Click to display the active SequenceList.

SampleList - Click to display the active SampleList.

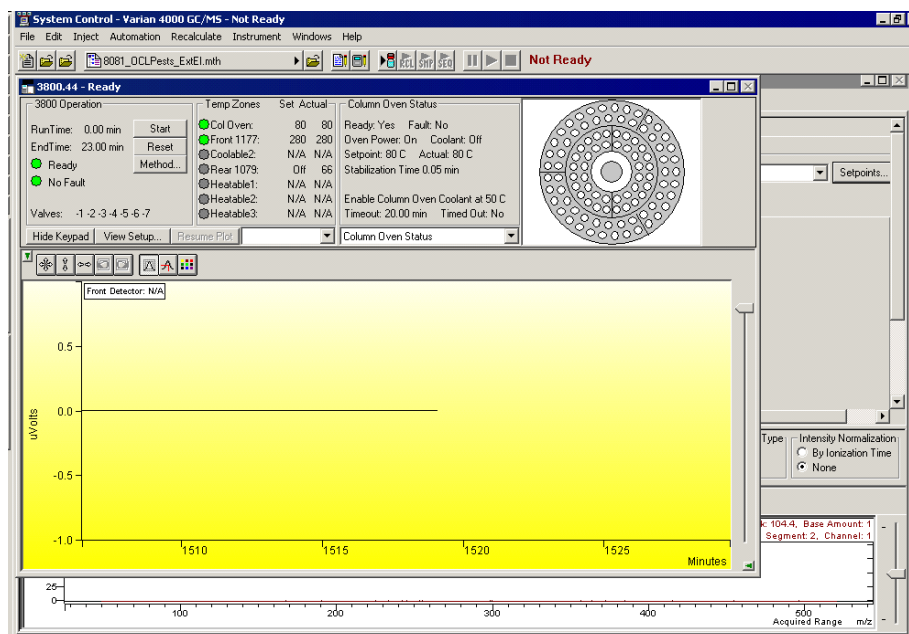
RecalcList - Click to display the active Recalculation list.

Instrument 1 Status - Click to view the status of the modules connected to the system.

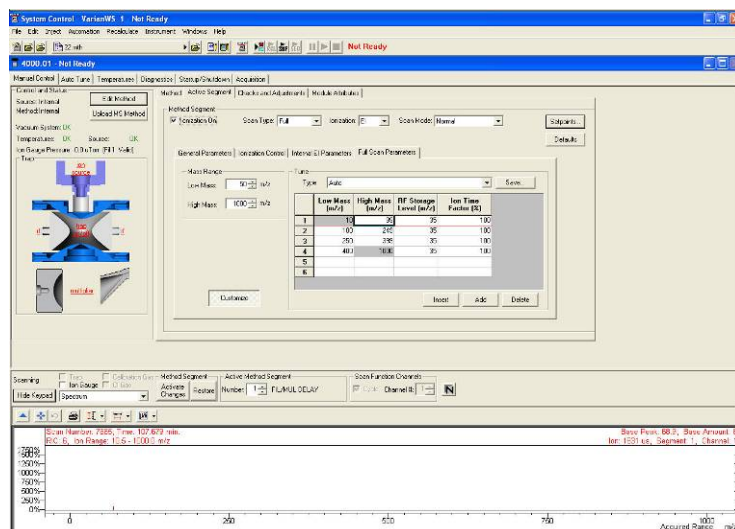
Message Log - Click to view the event log for the MS module. The log display is shown below. The error messages for the 4000 MS Module are displayed.



3800.44 - Click to display the 3800 GC control screen.



4000.56 - Click to display the 4000 MS control screen.



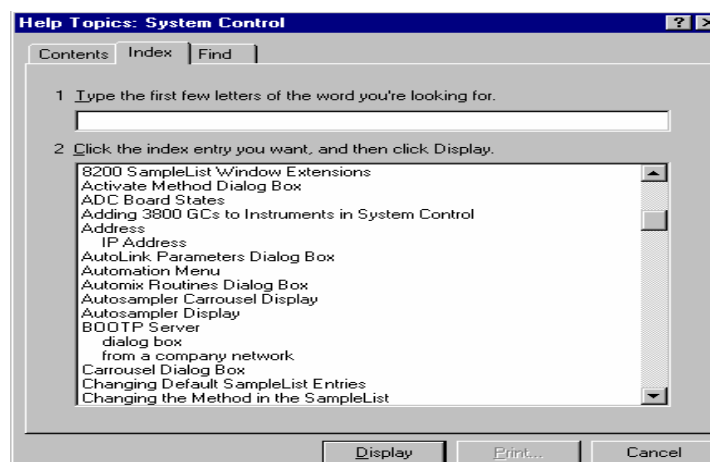
Help Menu

The Help menu is as follows.

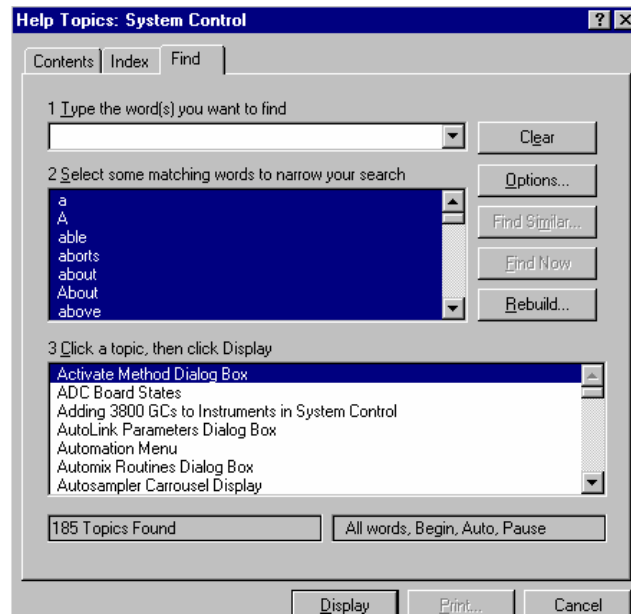


Select **Help Topics** from the Help menu to display the following. Click an item in the Contents box to go to the online help.

In the Help dialog box, from the Index tab, you can select a particular area in which you need help. Click on the selection you want and go to the bottom of the screen and click on **Display**.

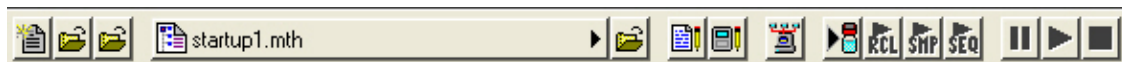


To use the Find dialog, type in a key word or phrase to get help topics.



System Control Toolbar

The system control toolbar is near the top of the screen.



	Create a new automation file.
	The first icon opens an existing automation file.
	The second icon opens the Message Log file.
 startup1.mth 	View/edit or print a Method.
	Open a Method.
	Edit notes for an automation file.
	Edit Module information for any online Module .
	Open Instrument 1 Status
	Inject a single sample.
	Start an open RecalcList.
	Start an open SampleList.
	Start an open SequenceList
	Suspend a running list.
	Begin a list.
	Stop a running list.

Module Keypad Dialog



Click **Hide Keypad**, to display a larger chromatogram and spectrum windows during data acquisition. Click **Show Keypad** to restore the 4000 MS Keypad.

Chromatogram Toolbar

The Chromatogram display can be viewed only when the 4000 MS module is in Acquisition mode. The Chromatogram Toolbar appears in the upper left corner of the display. The functions of buttons on this toolbar are as follows.



Click the **Arrow** to hide the toolbar. Click in the upper left of the display to restore the toolbar.



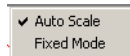
Click **Full-Scale** to normalize the chromatogram vertically and horizontally. Double-clicking in the lower left corner of the display has the same effect.



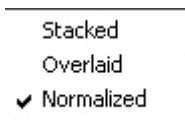
Click **Previous-Scale** to return the chromatogram display to the last scaling before the current one. If you have zoomed the chromatogram twice to view smaller time/intensity ranges, this button steps you back through the previous scales.



Click **Scale** to choose between Auto Scale and Fixed Mode Intensity.



Click **Chro Display** to display this menu



Click **Spectrum Select** to display the spectra. The chromatogram display stops updating.

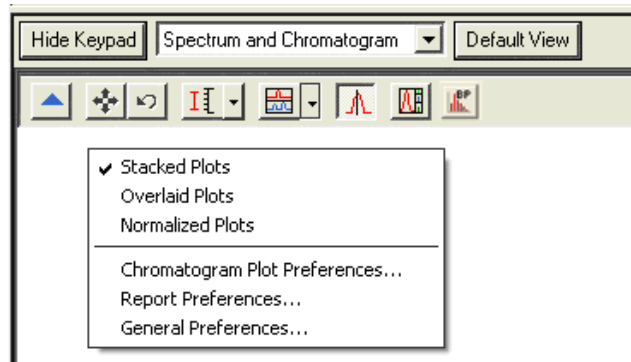


Click **Chromatogram Viewer** to load the acquiring data file into MS Data Review where you can compare this data with other data and do analysis.



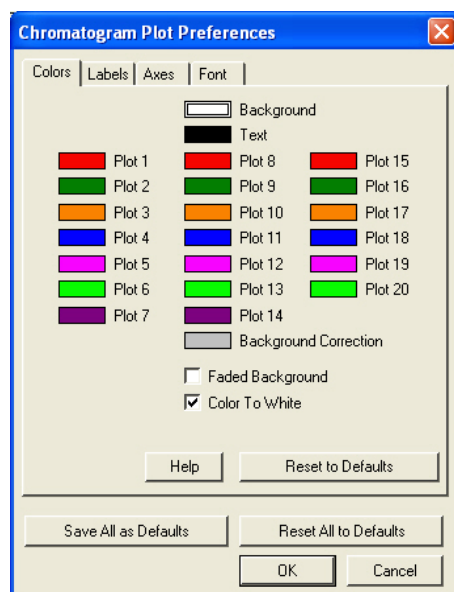
Click to display a plot of the Base Peak ion. To display the trace and the Base Peak plot, click the icon and enter **BPI** in the **Ions** field. Clicking the Base Peak icon does not affect the display of the extracted ion chromatogram.

Chromatogram Preferences

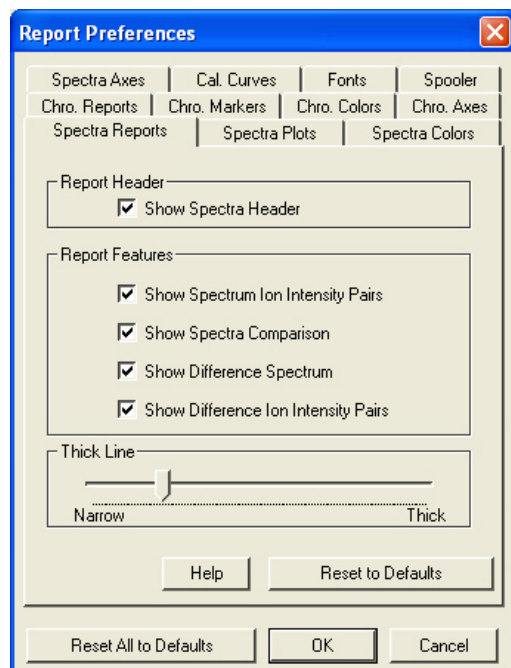


Right-click the chromatogram window to display a menu that offers these selections Chromatogram Plot Preferences, Report Preferences, or General Preferences.

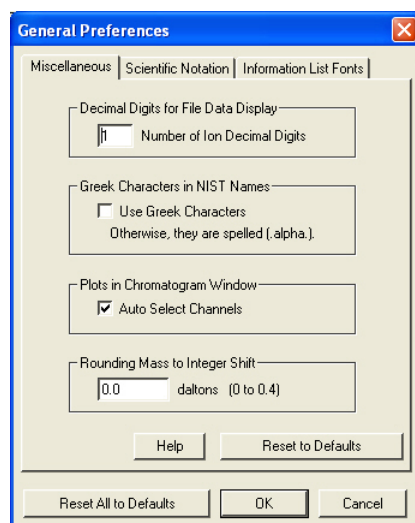
Use Chromatogram Plot Preferences to modify the appearance of the plot. This is used by chromatogram plot displays and reports in all MS applications, including Standard MS Reports, and Custom MS Reports.



Use **Report Preferences** to select chromatogram and spectrum report options.



Use **General Preferences** to change the appearance of numbers and fonts.



Spectrum Toolbar

The Spectrum display can be viewed when the 4000 MS module is in Manual Control, Auto Tune, or Acquisition modes. The Spectrum Toolbar appears in the upper left. The toolbar functions are as follows:



Click the **Arrow** to hide the toolbar. Click in the upper left of the display to restore the toolbar.



Click **Full-Scale** to normalize the chromatogram vertically and horizontally. Double-clicking in the lower left corner of the display has the same effect.



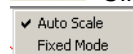
Click **Previous-Scale** to return the chromatogram display to the last scaling before the current one. If you have zoomed the chromatogram twice to view smaller time/intensity ranges, this button steps you back through the previous scales.



Click **Spectrum Report** to preview the spectrum report, which can then be printed.



Click **Scale** to choose between Auto Scale and Fixed Mode Intensity.



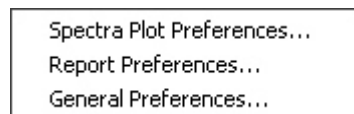
Click **Display Options** to choose Plot, Ion/Intensity List, or Summary Information for the display.



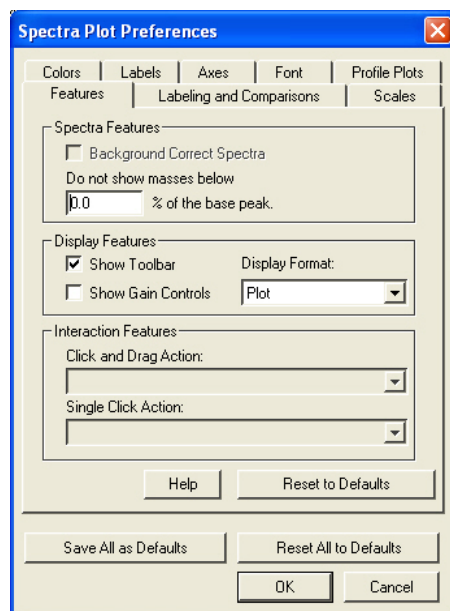
Click the **Display Type** to choose between graphical and text displays of mass spectra or to display additional information about the spectrum.

Spectrum Preferences

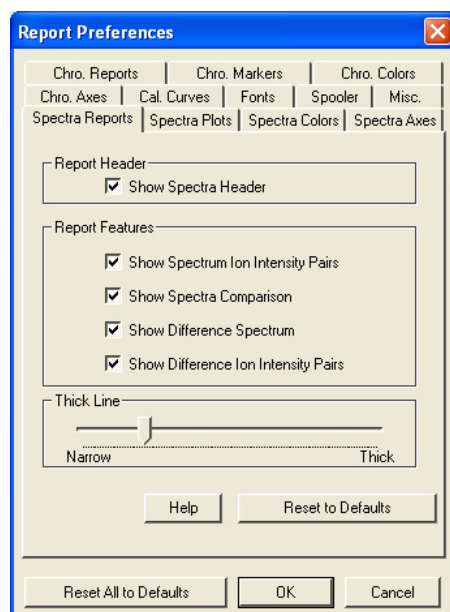
Right click the spectrum window to open a menu. From this menu you can select Spectra Plot Preferences, Report Preferences, or General Preferences.



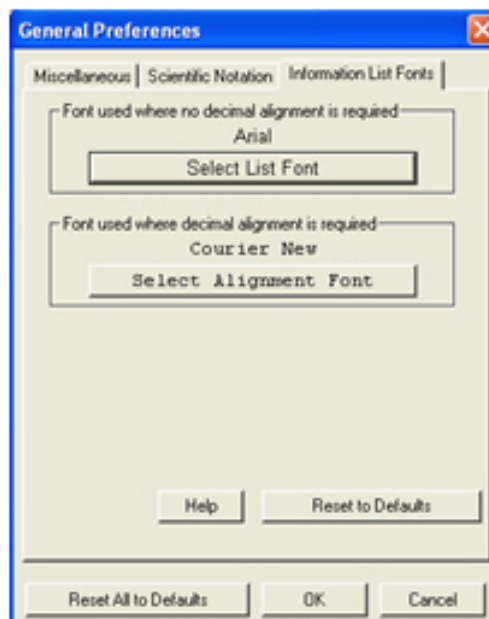
Use **Spectra Plot Preferences** to modify the appearance of the plot.



Use **Report Preferences** to select chromatogram and spectrum report options.

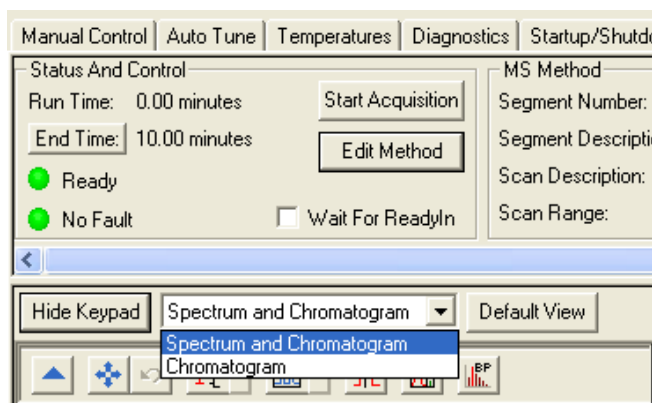


Use **General Preferences** to change the way numbers and fonts appear.

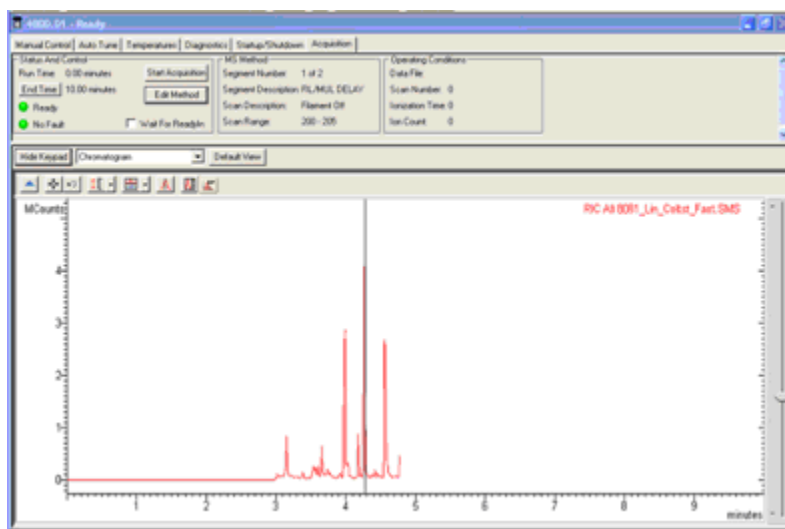


Configuring the Real-Time Chromatogram Display

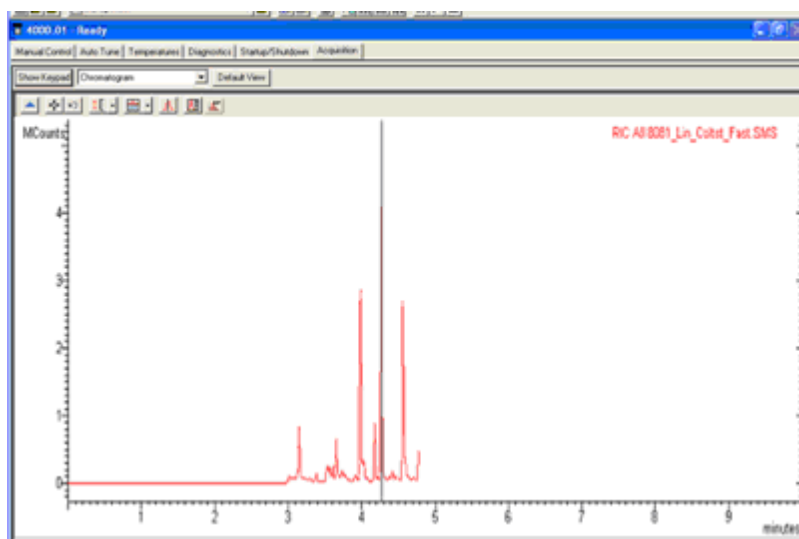
Before doing an analysis, place the MS module window in acquisition mode. If you are in a different mode and start a SampleList or Inject a Single Sample, the mode automatically changes to Acquisition. During an analysis you can see the chromatogram. Select the information you wish to view. Click Default View to display the Spectrum and Chromatogram side by side.



Select **Chromatogram** to see the chromatogram as it is acquired.

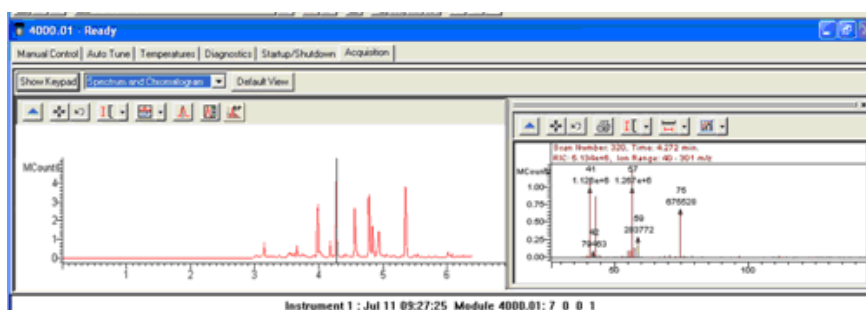


Click **Hide Keypad** to expand the display. In the following picture that the text information at the top is hidden and the chromatogram is expanded.



To display the Keypad, click **Show Keypad**.

Select **Spectrum and Chromatogram** to display them side by side. You can dock the Spectrum window, for example above the Chromatogram.

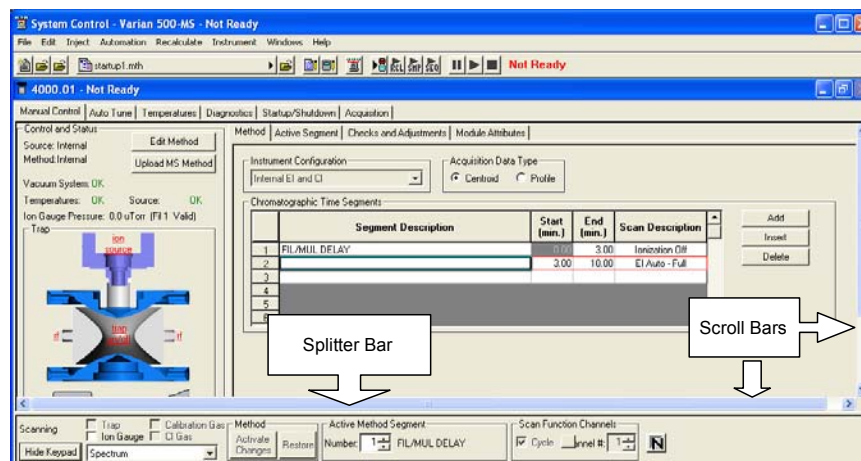


Manual Control Tab

In Manual Control the status of the MS module is displayed in real time. This window is divided into two panes, the 4000 MS Keypad above and the Spectrum/Module Attributes display below. There are many features in this mode and depending upon the screen resolution set in the Display section of Microsoft Windows Control Panel, all of the 4000 MS Keypad features may not be displayed.

Configuring the Display

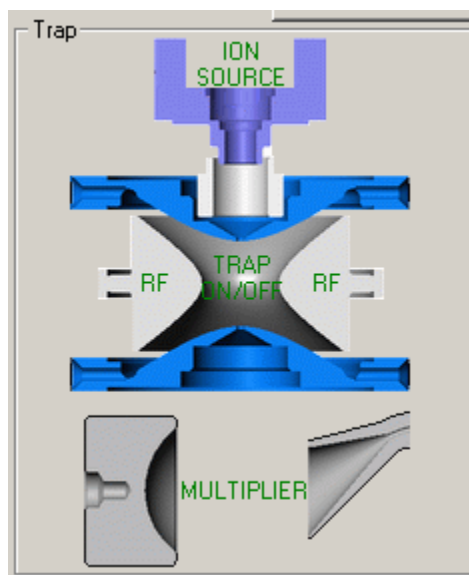
Slide the Scroll Bar on the right to view the lower section of the keypad. Also, you can click the Splitter Bar to expand the keypad viewing area and the Scroll Bars to scroll the view.



The 4000 MS Icon

The ion trap icon is displayed on the left side of the 4000 MS Keypad. If the icon text is red the ion trap is disabled. This is because the current Active Method segment is a filament/multiplier delay segment, one with an ionization mode of None. The ion trap is also disabled if there is an instrument Fault.

Changing the Active Method Segment to #2 changes the trap status to enabled and the color of the icon text changes to black. If the icon is not currently disabled, clicking in the center of the ion trap turns on the trap. The icon text will turn green to denote that the ion trap is on.



You can independently turn On or Off the RF, Multiplier, and Ion Source (Filament) by clicking the appropriate icon.

Quick Control Toolbar

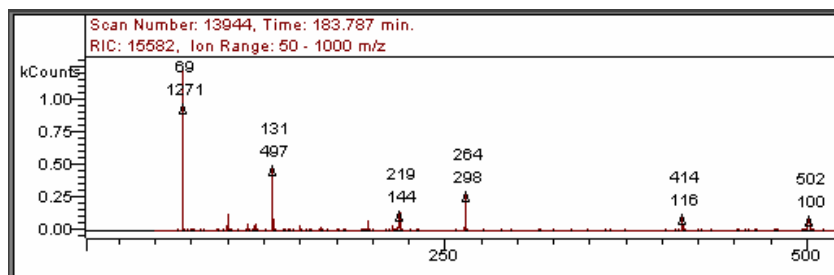
Below the trap icon are selections for turning on the Trap, the Ion Gauge, and the flow of either Calibration Gas or CI Gas.



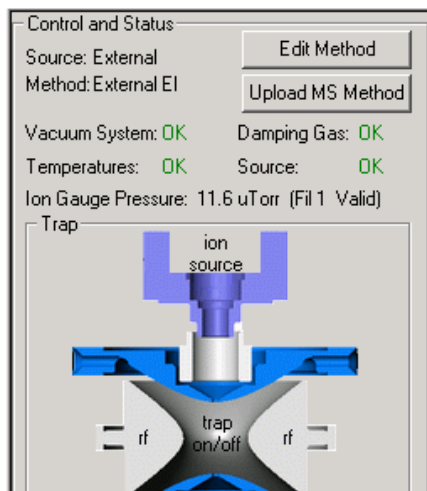
When the trap is turned on (the active method segment must have an ionization method other than None), a mass spectrum appears in the spectrum display.



If the Calibration Gas is turned on in EI mode, you can see the characteristic spectrum with prominent ions at m/z 69, 131, 264, 414, and 502. The mass assignments of these ions and of m/z 464 and 614 are checked during the Mass Calibration procedure in the Auto Tune mode.



When the ion gauge is turned on a pressure reading is displayed in the area above the trap icon in the 4000 MS Keypad. In this image the ion trap is in External EI mode so the Damping Gas check is displayed.

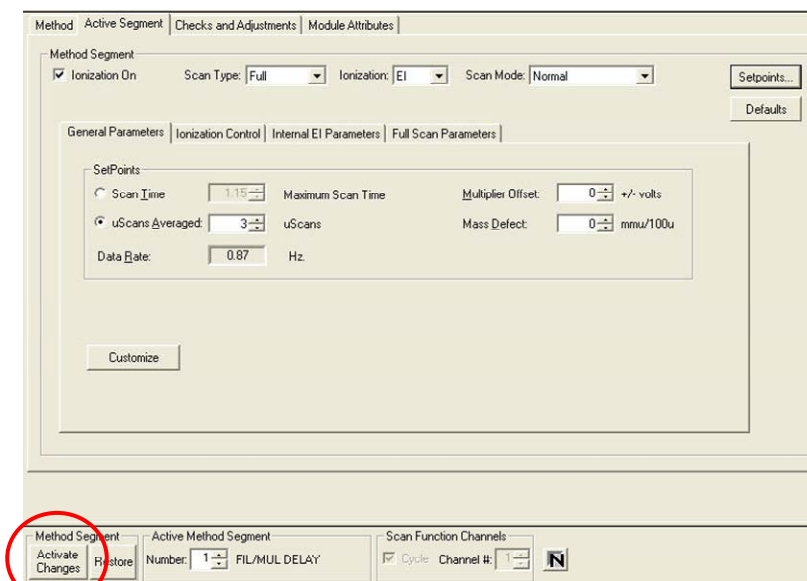


Manual Control Tabs

The 4000 MS Keypad has the following tabs.

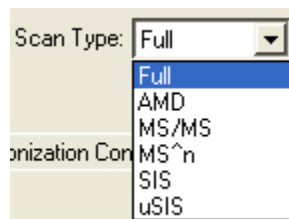


Method Segment

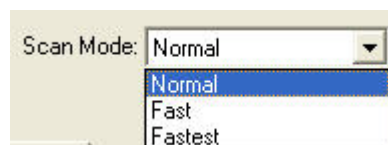


In this image, the Active Segment tab dialog is shown with some method-related controls in the lower pane. The information in the top row shows that Ionization is on, the Scan Type, Ionization mode and the Scan Mode.

The Scan Type choices are as follows:

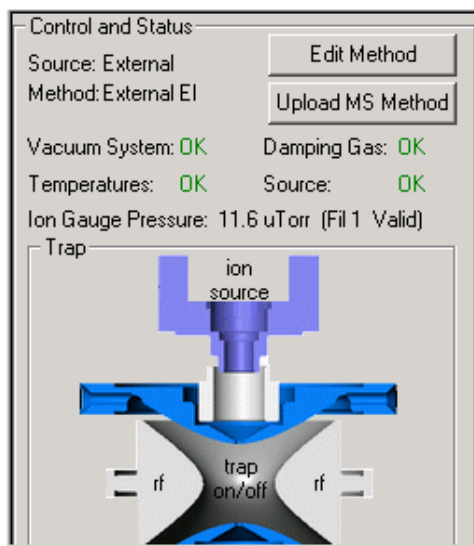


The Scan Mode choices are as follows:



You can examine and edit the parameters in the active MS method and observe the effects of these changes on the mass spectra being acquired. The tab dialogs available depend on the ionization and ion preparation modes in the current method segment. After you edit a parameter implement the change by clicking **Activate Changes**.

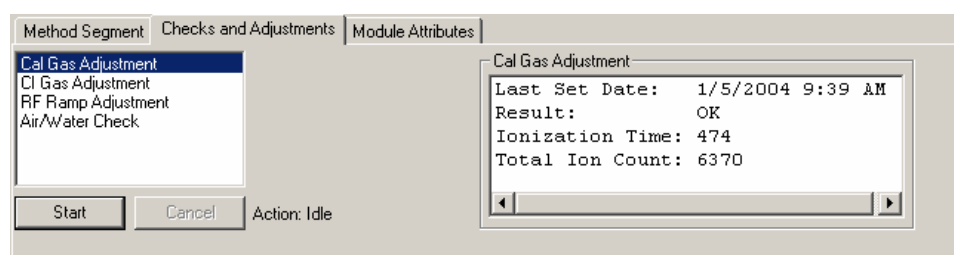
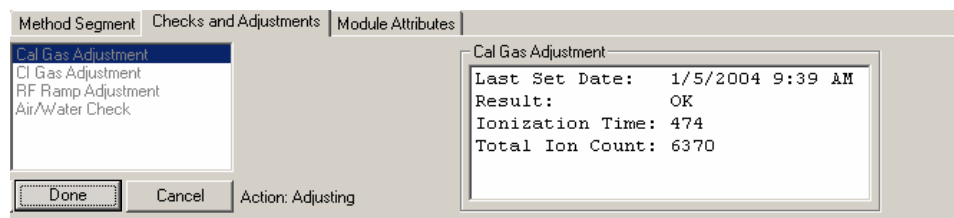
To save changes to the method, click the **Upload MS Method** button above the Ion Trap icon. You can also edit the method in Method Builder by clicking the **Edit Method**.



NOTE: Details about editing the MS method are in the relevant section of Building GC/MS Methods.

Checks and Adjustments

Four adjustments may be made in the Checks and Adjustments tab dialog. In each case, one clicks the **Start** button to begin the adjustment and the **Done** button when the adjustment is completed.

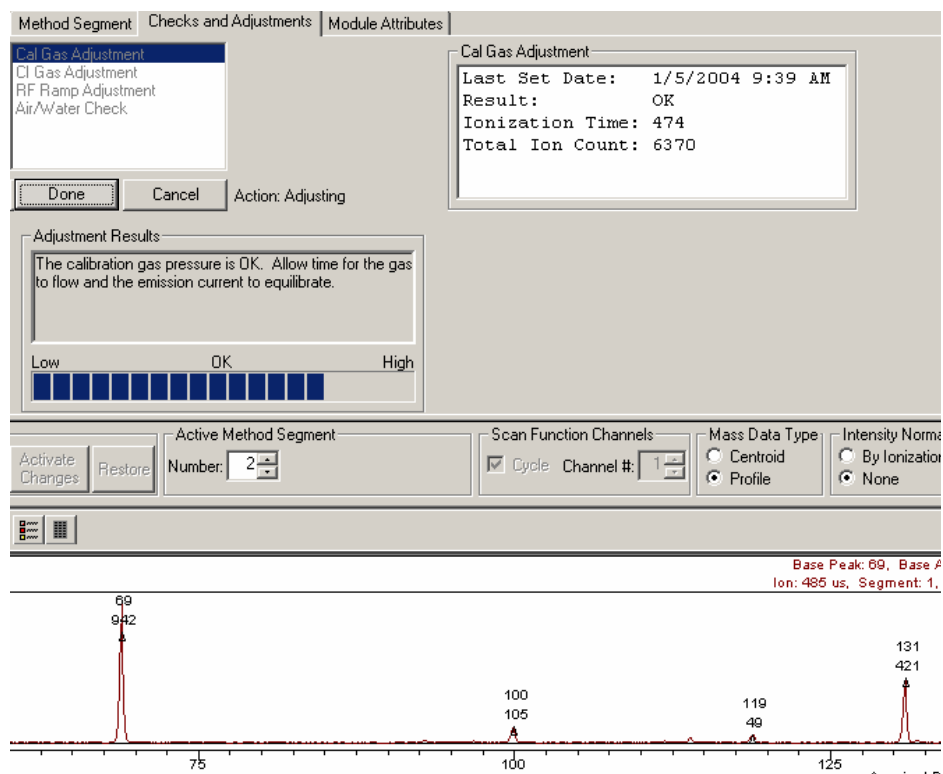


Calibration Gas Adjustment

Make this adjustment using the Calibration Gas valve inside the front door of the 4000 MS. This valve has a black knob with the number 0 labeled on it. Turn the valve counterclockwise to increase Cal Gas flow and clockwise to decrease the flow. Adjust the flow so that the readback in the Adjustment Results field indicates that the flow is OK as seen in the following picture.

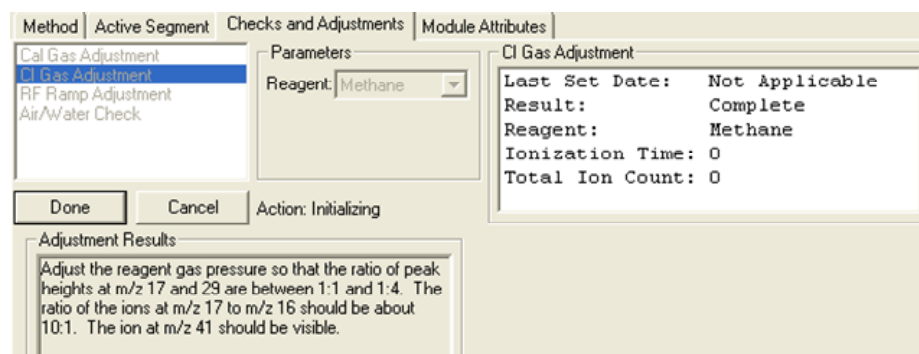
NOTE: If you are doing an initial tune (for example, When first starting up a system), the integrator zero may be far enough off that even with the cal gas closed all the way down, the cal gas adjustment will indicate success. There are interrelationships between the Cal Gas, the Integrator Zero, and the Electron Multiplier. Therefore, stabilize the settings by performing a Cal Gas adjustment more than once.

Initially, set the Cal Gas to the midpoint (8 turns) and then perform the Integrator Zero tune. Then perform the Electron Multiplier tune. Now, go back to the Cal Gas adjustment to set the pressure. Repeat this until the Cal Gas and Electron Multiplier settings stabilize.



CI Gas Adjustment

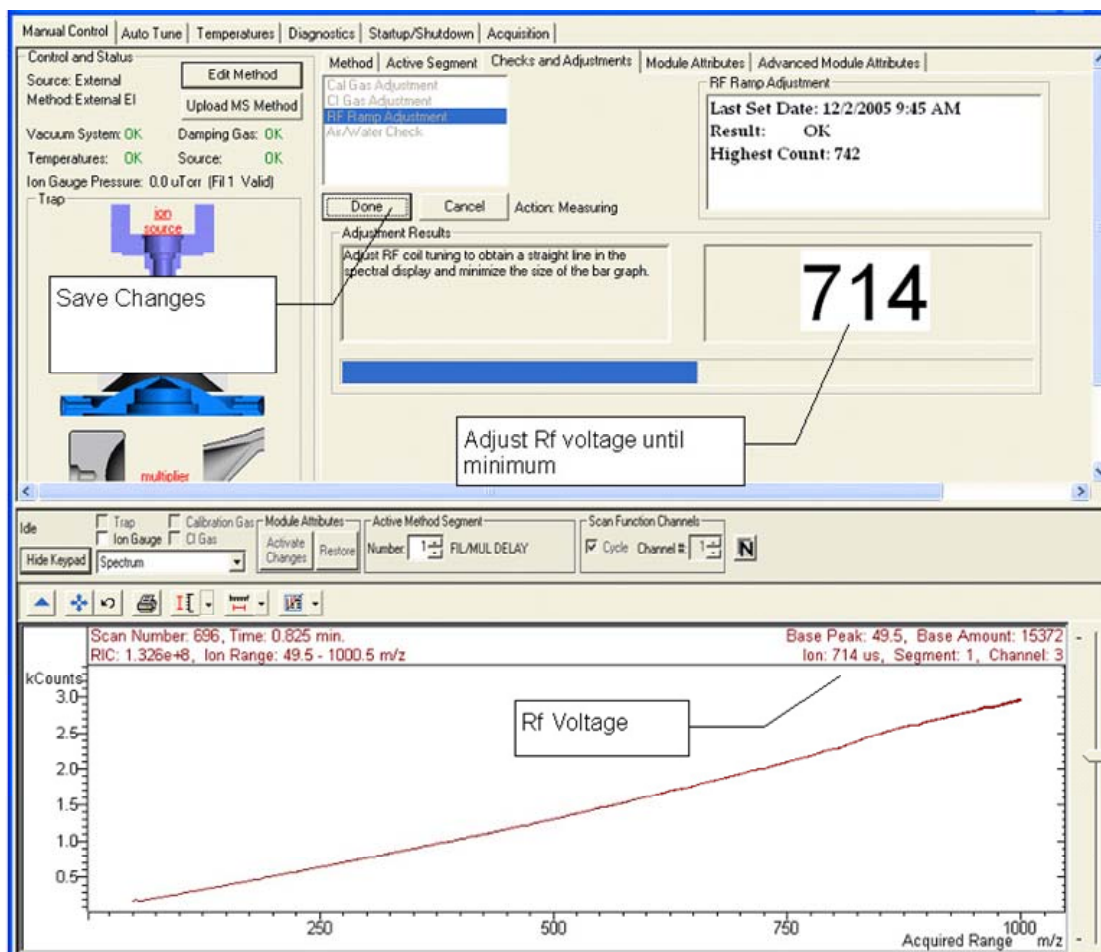
Assure that the appropriate CI reagent is plumbed into the CI gas inlet in the rear of the 4000 MS or contained in the Liquid CI reagent vial. Choose the CI Reagent to adjust from the Parameters Reagent menu. Then adjust the flow using the CI Gas valve inside the front door (labeled with numeral 3). Turn the valve clockwise to increase the flow or counterclockwise to decrease the flow. The readback in the Adjustment Results field identify when the flow is OK.



RF Ramp Adjustment

When you select **RF Ramp Adjustment**, a screen opens that displays if the RF voltage is in tune over the entire mass range of the 4000 MS.

After opening the screen, use a screwdriver to adjust the RF coil screw inside the front door of the 4000 MS. Follow the instructions in the Adjustment Results field until the value of the highest part of the graph is at a minimum.



Air/Water Check

Run the Set Electron Multiplier Auto Tune before doing the Air/Water Check.

The Air/Water Check routine determines the relative amount of air and water vapor in the vacuum manifold. When you display the Air/Water screen, the following happens automatically: The filament, electron multiplier, and RF voltage are turned on; AGC is turned off; and the scan range is set to display the air/water spectrum (m/z 14 to 45). The air background is proportional to the width of the peak for m/z 28 at 10% peak height. The water background is proportional to the ratio of m/z 19 to m/z 18.

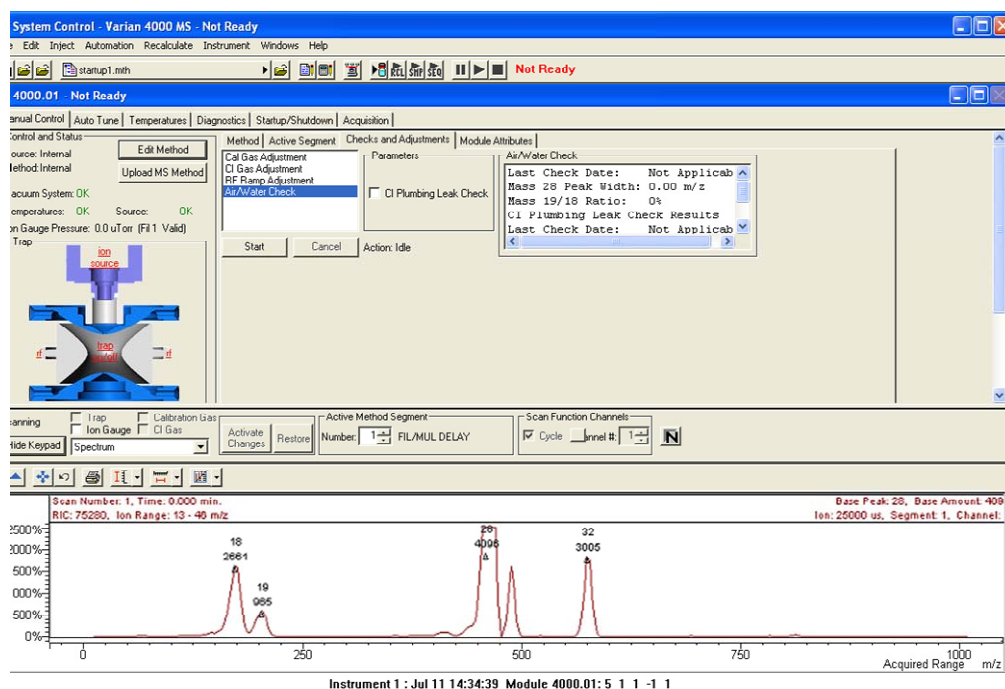
For Internal Mode, the ionization time is fixed at 300 μ sec. For External Mode, the intensity of the highest peak (m/z 18, 19, and 28) is adjusted to a suitable level.

Width of m/z 28 at 10% Peak Height	Ratio 9/18	Acceptance Criteria
< 1.0u	< 20%	OK
< 1.8u	< 50%	Small Leak
< 3.1u	< 91%	Significant Leak
> 3.1u	> 91%	Massive

For External (and Hybrid) Mode, the acceptance criteria (Ratio of m/z 19/18) for presence of water is the same as the Internal mode. However, a number of External (and Hybrid) parameters may affect the intensity of the air peak at m/z 28 (for example, damping gas flow rate, DC offset voltage, lens voltages, etc.). If a massive air peak is present, turn off the Damping Gas flow and run Air/Water Check again. If no peaks are present with the damping gas off, then the most likely cause of a massive nitrogen peak (m/z 28) is inadequate flushing of filters and/or helium line, rather than a leak in the mass spectrometer. If the nitrogen peak (m/z 28) is observed with the damping gas off, please see the Troubleshooting section for more information on finding air leaks.

The CI Plumbing Leak Test is run automatically after the Air/Water Test. The CI solenoid valve is opened and air/water levels are checked. It is not unusual for these values to be initially high. Generally, the CI valve should be opened and flushed (or left under vacuum) several minutes before this test is done.

The Air/Water Check will give advice on the levels of these background components. High water level is indicated both by the intensity of the ion at m/z 18 but also by the percentage of m/z 19 (H_3O^+) relative to m/z 18. The water level is high after the instrument is first pumped down after maintenance. Baking the instrument overnight should bring the level to an acceptable level. A high air level as shown in the example suggests that there may be a leak in the vacuum system or GC. Consult the troubleshooting section in the 4000 GC/MS Hardware Operation Manual for advice on locating sources of air leaks.



Module Attributes

Two filaments are provided in External, Internal, or Hybrid configurations to reduce down time for maintenance. Select the desired filament

Override the Electron Multiplier voltage set from Auto Tune by entering the desired voltage.

Adjust the High Mass Calibration to improve the accuracy of mass assignments of samples that range from 650 to 1000 m/z .

Increase the sensitivity of the detection of low mass ions (<100 m/z), especially for EPA method 521.

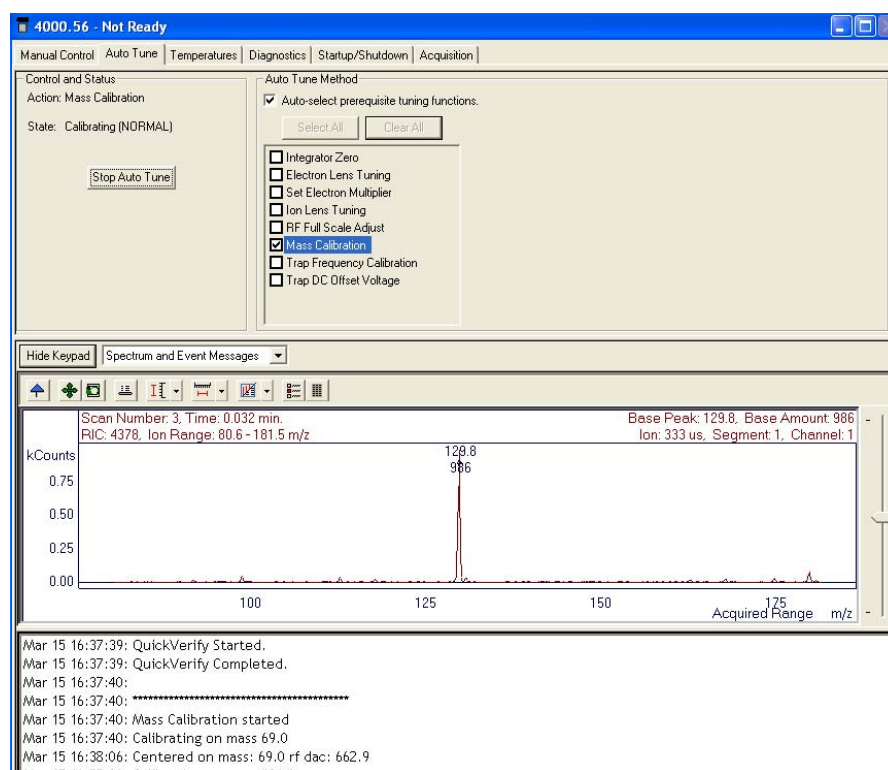
Auto Tune Tab

Auto Tune is an automated process for optimizing the MS 4000 performance over the entire mass range. It is necessary to perform Auto Tune when the instrument is first set up.

NOTE: Each time the system is shut down, do Auto Tune when you restart. Also AutoTune after doing a bakeout.

Before doing Auto Tune it is advisable to check RF ramp, air/water, and cal gas settings under the Adjustments tab in Manual Control mode. Many tuning features involve using Perfluorotributylamine (FC-43) ions to adjust MS4000 parameters (voltages, gain values, etc.) to optimize the signal. Most Auto Tune functions are performed in EI mode, even if the active method is Internal CI, External CI, or one of the Hybrid CI modes. For the last two cases, the CI volume is automatically retracted from the external ionization source during most Auto Tune routines.

When major maintenance is performed, such as cleaning the ion trap or source or switching between internal and external ionization modes, all Auto Tune routines should be run. Some routines should be run after changing operating conditions, such as trap temperature or helium buffer gas flow rate. Specific routines may be run periodically as part of a system check.



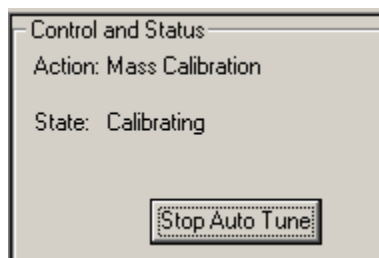
The Auto Tune window has three sections, Control and Status, Spectrum and Event Messages, and Auto Tune Method.

Control and Status

Clicking Start Auto Tune to start the selected routines. Click Stop Auto Tune to stop them.

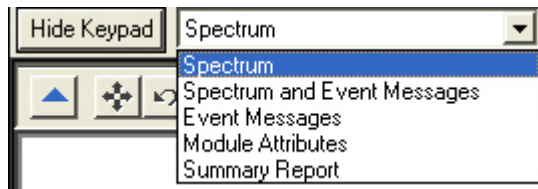
The Action field shows the tuning routine that is being performed.

The State field shows if the 4000 MS is executing an auto tune routine or idle.

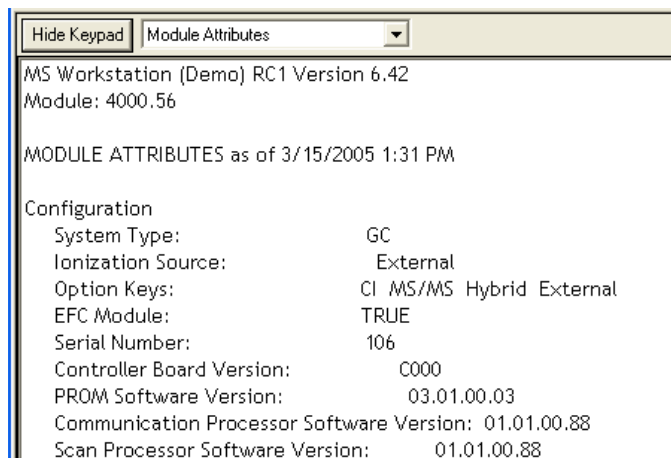


Spectrum and Event Message Window

You can select the information displayed during the auto tune tests by selecting from the list. Click **Hide Keypad** button to hide the upper portion of the screen and expand the lower portion.

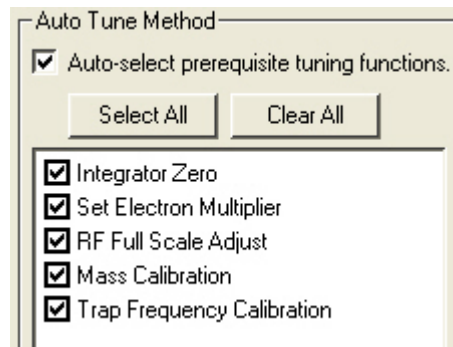


Module Attributes is the information stored from the last auto tune. After you do an AutoTune you can view this log.



Auto Tune Methods

The auto tune checks that you can automatically set up, tune and calibrate the mass spectrometer with depend on the instrument configuration. The following show the checks available for an internal source.

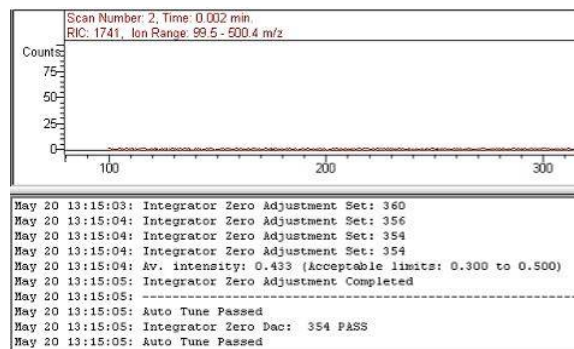


Other functions are only needed when the instrument is in Hybrid or External ionization configuration.

Auto Tune is generally performed in the order listed. All of the auto tune methods are selected if you click Select All or cleared if you click Clear All.

To perform Auto Tune click the desired check boxes and click Start Auto Tune. You can stop the auto tune at any time by clicking Stop Auto Tune.

Integrator Zero



The Integrator Zero Adjustment routine sets the signal level from the Integrator Circuitry with the filament off. When the filament is off, the major source of signal coming from this circuitry is electronic noise. The Integrator zero is adjusted such that the average value of electronic noise is slightly greater than zero. The acceptance range is between 0.3 and 0.5 counts. The integrator then records signals only when ions actually strike the dynode and electrons emitted from the dynode strike the electron multiplier.

Set Electron Multiplier

```
May 21 16:07:25: EM voltage: 1400 V ion time: 65000 usec
May 21 16:07:25: EM voltage: 1500 V ion time: 10000 usec
May 21 16:07:27: EM voltage: 1500 V ion time: 65000 usec
May 21 16:07:28: EM voltage: 1525 V ion time: 65000 usec
May 21 16:07:29: EM voltage: 1550 V ion time: 65000 usec
May 21 16:07:30: EM voltage: 1575 V ion time: 65000 usec
May 21 16:07:31: EM voltage: 1600 V ion time: 65000 usec
May 21 16:07:32: EM voltage: 1625 V ion time: 65000 usec
May 21 16:07:33: EM voltage: 1650 V ion time: 65000 usec
May 21 16:07:34: EM voltage: 1675 V ion time: 65000 usec
May 21 16:07:34: Electron Multiplier gain is 3.71
May 21 16:07:34: Electron Multiplier voltage setting is 1675
May 21 16:07:34: Electron Multiplier Gain Adj completed
May 21 16:07:34: -----
May 21 16:07:34: Auto Tune Passed
May 21 16:07:34: Electron Multiplier Voltage:1675 PASS
May 21 16:07:34: Auto Tune Passed
```

Before starting the EM gain algorithm, the RF Full Scale is adjusted to calibrate the cal gas ion at m/z 502. Set Electron Multiplier adjusts the multiplier voltage and ion time to look at low-mass fragments from the unstable m/z 502 ion. The multiplier voltage is set so that each electron emitted by the dynode creates a known signal from the multiplier. The gain in this example is 3.71×10^5 .

Occasionally, systems with an external or hybrid source fail this adjustment. Increasing the flow of FC-43 usually allows the multiplier gain to be set. An Event Window message is displayed.

When the 10^5 gain voltage of your electron multiplier reaches 2000V order a new multiplier to have on hand. When the 10^5 gain voltage exceeds 2500V should install a new multiplier.

Electron Lens Tuning

In typical external ion sources, electrons are continuously directed into an ion volume containing the sample to be ionized. Eventually the interior surface of the ion volume becomes contaminated by material that can charge up and disturb the extraction of ions from the ion source. To minimize ion volume contamination, the 4000 MS pulses electrons into the ion volume only when needed for ionization. Two plates bracket the filament to control electron pulsing. The plate closest to the ion volume is the electron lens, and the plate away from the ion volume is the Repeller lens. The electron lens is set positive and the Repeller lens negative to admit electrons to the ion volume only during the ionization period. Otherwise the voltages are reversed to exclude electrons from the ion volume. Tuning the Electron/Repeller lens voltages is important for linear signal response and filament lifetime.

The four variables are electron lens ON and OFF voltages, and Repeller lens ON and OFF voltages. ON and OFF refer to ionization ON and OFF. The electron lens ON voltage is set to optimize electron transmission to the ion volume, electron lens OFF voltage is set to thoroughly exclude electrons from the ion volume. The Repeller Lens ON and OFF voltages balance the emission current.

Electron/Repeller Lens Adjustment: Electron lens tuning involves measuring the transient behavior of the emission current immediately after the lenses have been switched on or off. If the lenses are unbalanced, the emission current will change in time and be proportional to the imbalance. If the balance is outside the range of 200 to 300 μA , the algorithm will search the optimal values by changing values of four variables one at a time. If it fails to find the best voltage setting for lens tuning, auto tune will generate an error message, and restore the last values in the instrument.

When the Electron Lens Tuning Box is clicked, an additional “Turn on CI gas flow during tune” option appears. For CI methods in External or Hybrid mode, the Electron/Repeller lens must be tuned with the CI plunger (CI volume) in place and the CI gas turned on. The user should adjust the CI gas flow in Manual Control before this tune function is done.

Ion Lens Tuning

The Ion Lens system consists of three lenses (Lens 1, 2 & 3). These lenses are tuned using Cal Gas ions at m/z 131 and 414. Optimum voltages are determined based on weighted intensities of the two ions.

RF Full Scale Adjust

This routine should always be run before mass calibration and trap function calibration. The RF Full Scale Adjust routine coarsely adjusts the mass calibration of the ion trap. Successive adjustments are performed on PFTBA calibration gas ions at m/z 69, 414 and 614. For this algorithm to work properly, it is important that the specified ions are stable and present in sufficient intensity (>20 counts) under normal scanning conditions.

Mass Calibration

When you initiate this routine, the calibration gas valve is opened and the electron multiplier, filament, and RF voltage are turned on. The algorithm then uses the calibration compound ions at m/z 69, 131, 264, 414, 464, and 614 to create a calibration curve. The nitrogen peak (m/z 28) is not used for calibration. A fixed offset at m/z 0 has been empirically determined to give proper mass assignments between m/z 10 and 69. The calibration is extrapolated from the highest observed calibration ion to the maximum mass (m/z 1000). Mass calibration is performed in both normal scanning (5,000 u/sec) and fast scanning (10,000 u/sec) modes.

Factors Affecting Mass Calibration

For this routine to work properly, the calibration ions must be present in sufficient intensity (> 20 counts) and stable under normal scanning conditions. Temperature changes can shift the mass calibration and this procedure should be initiated after the trap has reached its equilibrium point (approximately 2 hours after the set-point temperature has been established). For External and Hybrid modes, changing the trap damping gas flow may require both Mass Calibration Trap Frequency Calibration. There could also be subtle effects on mass assignments after ion source temperature changes.

Trap Frequency Calibration

When the mass calibration has been completed, the trap frequency calibration must be performed. This routine determines resonant frequencies required for the generation of notched waveforms used in most modes of operation. In particular, the resonant frequencies for FC-43 ions at m/z 69 and 414 are determined. The method takes several minutes to complete.

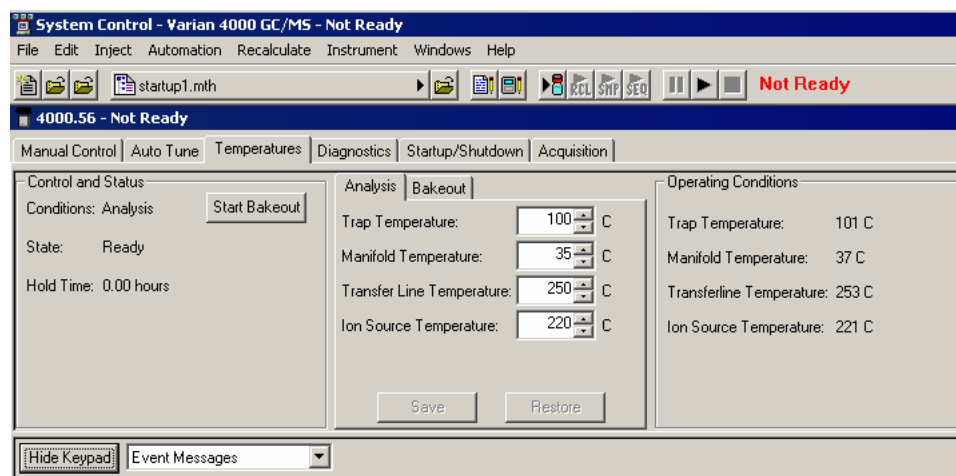
NOTE: Do Trap Frequency Calibration each time you do a mass calibration.

Upon completion of Trap Frequency Calibration, the Event Message Window displays the secular frequency values for the two trap function calibration points. Nominal values are 249 kHz and 248 kHz, respectively. These values can vary up to ± 4 kHz between instruments.

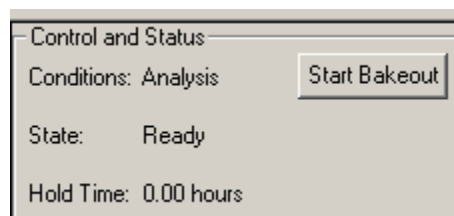
Trap DC Offset Voltage

The trap offset is a DC voltage applied to the ion trap electrodes (end caps and ring electrode) to facilitate the injection of the externally generated ions into the trap. The intensity of the m/z 414 ion from FC-43 is used to maximize the signal, thus optimizing for the midpoint of the mass range. The trap offset has a range from 3 to 15 volts. Lower voltages are useful if an increase in the relative abundance at the low end of the mass range is desired. Furthermore, if your application involves more fragile compounds, then a lower setting can produce a situation where less fragmentation of the molecular ion is observed.

Temperatures Tab



Control and Status



The Control and Status area has Temperature information and identifies if Analysis or Bakeout temperatures are active. State tells if the system is Ready or Equilibrating. If a bakeout has been started, the Hold Time field shows the remaining time before the bakeout is complete.

NOTE: Hold Time does not start counting down until bakeout temperatures have been reached or until the system has warmed up for 20 minutes.

Operating Conditions

Readbacks of the current temperatures in each zone.

Operating Conditions	
Trap Temperature:	100 C
Manifold Temperature:	53 C
Transferline Temperature:	253 C
Ion Source Temperature:	221 C

Event Messages and Module Attributes Display

After a change, the event log at the bottom of the screen updates with the time and identity of the event. You can also look at the Module Attributes.

Hide Keypad	Event Messages	
Jan 07 10:39:33: Bakeout: Cancelled by User (Returning to Analysis Temperatures)	Event Messages	orted (Trap: 220, Source: 200, Manifold: 120, Transferline: 250)
	Module Attributes	

Setting Analysis and Bakeout Temperatures

Clicking in the fields and changing the values will change temperature settings. After changing a setting, click save. The **Restore** button will return the settings prior to your changes. The allowed temperature ranges in degrees and the default temperatures for either Internal or External/Hybrid configurations or bakeout are entered in the following table. All temperature units are °C. The ion source temperature zone will not appear when the instrument is in Internal configuration.

Temp Zone	Min	Max	Default External	Default Internal or Hybrid	Default Bakeout
Ion Trap	50	270	100	150	220
Manifold	0	120	50	50	110
Transfer Line	0	350	170	170	250
Ion Source	0	300	180	N/A	250

NOTE: If the manifold temperature is 70 °C or higher, the source temperature is limited to 250 °C.

Click the **Bakeout** tab to set Bakeout temperatures. The default bakeout temperatures are in the table above. The bakeout t can be between 0 and 100 hours. The default time is 8 hours.

Generally, do a Bakeout after the system has been restarted following shutdown and venting to the atmosphere.

The manifold is heated during bakeout to remove water and other volatile contaminants such as residual light hydrocarbons. The 50 °C default manifold temperature is usually used when acquiring MS data. The transfer line temperature should be no lower than 30 °C below the highest temperature of the GC column oven program in the active method.

NOTE: The transfer line should NOT be heated above the maximum isothermal temperature specified for the capillary column.

Adjustments After Temperature Changes

Whenever the ion trap temperature is changed, **2 hours** should be allowed for equilibration after the trap temperature has reached its set point. Once equilibrium is reached, the RF tuning should be checked and adjusted if necessary under Adjustments in Manual Control and Mass Calibration and Trap Frequency Calibration should be run in Auto Tune.

Bakeout

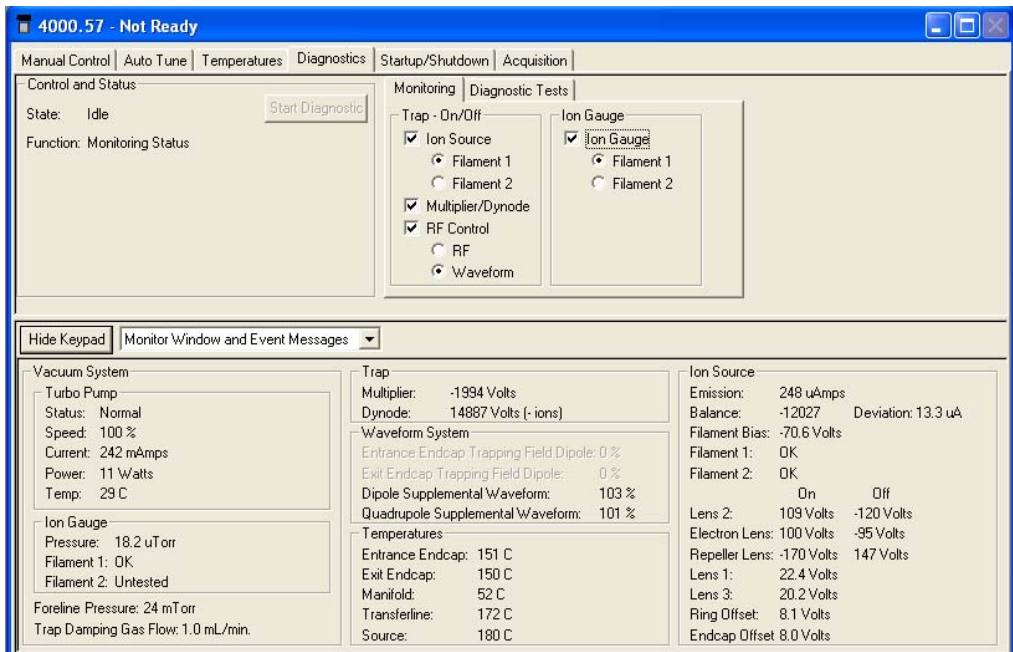
Click **Start Bakeout**. The State becomes Equilibrating until bakeout temperatures are reached, then Holding until the Hold Time counts down to zero.

NOTE: Hold Time does not start counting down until bakeout temperatures have been reached or until 20 minutes have elapsed.

At the end of the bakeout, the trap, manifold, transfer line, and source temperatures will return to the Analysis set points. If you want to change bakeout duration or temperatures during a bakeout, click Stop Bakeout, make changes and Save them, and then restart the bakeout.

Diagnostics Tab

Diagnostics mode is used to monitor the status of the vacuum system, applied voltages, and temperatures in real time. It is also where diagnostic tests are performed. The Manual Control Window is divided into two panes, the Keypad on top and the Module Window and Event Messages display below.

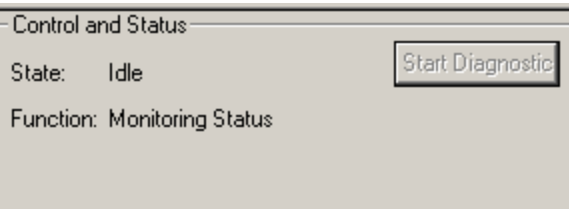


The Keypad Field

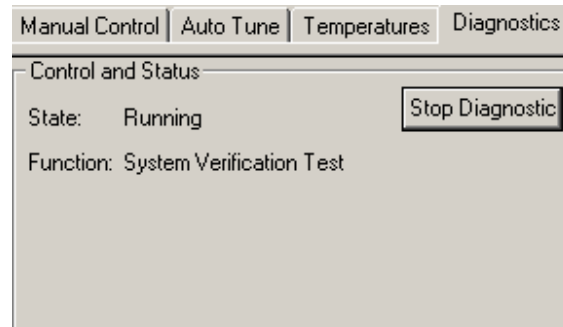
The upper portion of the screen is divided into three sections: a Control and Status field and the Monitoring and Diagnostics Tests tab dialogs.

Control and Status

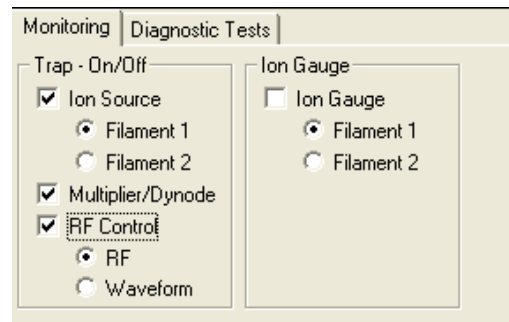
The State field will indicate Idle when diagnostic tests are not being performed or Running if tests are in progress. The Function field will indicate Monitoring Status unless a diagnostic test is being run, in which case the name of the current test will be displayed. The Start Diagnostic button is unavailable unless a test has been selected in the Diagnostic Test tab dialog.



After a diagnostic test is selected and Start Diagnostic clicked, the test starts, and the results are displayed in Event Messages at the bottom of the screen.



Monitoring Tab Dialog

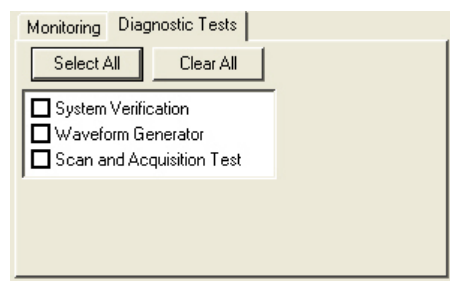


Select the desired ion trap features to monitor in the Trap On/Off area.

- Select Ion Source and the filament emission current is displayed in the Ion Source.
- Select Multiplier/Dynode and the voltages of the electron multiplier and conversion dynode are displayed in the Trap.
- Select RF Control with the RF radio button the Entrance and Exit Trapping Field Dipole values are displayed.
- Select Waveform with RF Control and the Dipole and Quadrupole Supplemental Waveform Voltages are monitored.
- Select Ion Gauge option and the ion gauge pressure in μtorr is displayed in the Ion Gauge area.

NOTE: If System Control is started in Diagnostics Mode, no method was activated and the Dynode readback is a default value of about positive 9,000V. Once a method has been activated in Manual Control or Acquisition, the correct Dynode voltage associated with the method is displayed in Diagnostics.

Diagnostic Tests



Click Select All or Clear All to either check or uncheck all the boxes for diagnostic testing. Test results are displayed in the event messages window. If a test fails, go to the relevant section of the Troubleshooting Guide in the 4000 GC/MS Hardware Operation Manual.

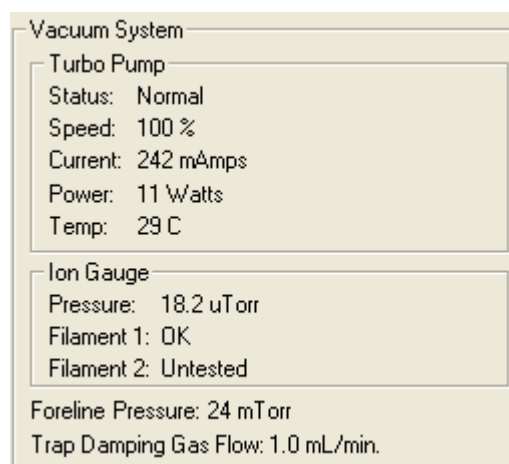
Configuring the Monitor and Event Messages Display

Create a larger area for displaying the Monitor window and Event Messages by clicking Hide Keypad or recover the Keypad by clicking Show Keypad.

You can display the Monitor Window or Event Messages displays alone or in combination by selecting from the drop-down menu.



Vacuum System



The turbomolecular pump current and power readings displayed are typical during routine operation. In this example the instrument is in External configuration with an auxiliary Helium flow of 1 mL/min. The values for current and power are lower in Internal configuration.

The ion gauge reading depends on if there is auxiliary Helium flow; in this case the system is in External configuration and the reading of ~29 μ Torr is reasonable for an auxiliary flow of 4 mL/min. The reading drops to 6 μ Torr when the auxiliary flow is turned off in Startup/Shutdown mode. In Internal configuration the pressure should be <10 μ Torr.

The foreline pressure is determined with a thermocouple gauge detector; its reading is strongly influenced by the auxiliary Helium flow as well.

Trap

Trap	
Multiplier:	-1342 Volts
Dynode:	-10360 Volts (+ ions)

Readings for multiplier and conversion dynode voltages are displayed only when Multiplier/Dynode is checked in the Monitoring tab dialog in the keypad above. The multiplier reading is always negative and reflects the voltage drop between ground and the entrance of the multiplier.

The dynode voltage should be close to 10,000V; the polarity is opposite that of the ions being detected.

Waveform System

Waveform System	
Entrance Endcap Trapping Field Dipole:	100 %
Exit Endcap Trapping Field Dipole:	100 %
Dipole Supplemental Waveform:	444 %
Quadrupole Supplemental Waveform:	444 %

The readings for Entrance/Exit End Cap Trapping Field Dipole are activated when RF control is checked and the **RF radio** button is checked in the Monitoring tab dialog. The values should be (100 $\pm$ 1)%.

Waveform System	
Entrance Endcap Trapping Field Dipole:	0 %
Exit Endcap Trapping Field Dipole:	0 %
Dipole Supplemental Waveform:	101 %
Quadrupole Supplemental Waveform:	100 %

Dipole and Quadrupole Supplemental Waveform readings are activated when RF control is checked and the **Waveform radio** button is checked in the Monitoring tab dialog. The values should be near 100%.

Temperatures

Temperatures	
Entrance Endcap:	99 C
Exit Endcap:	99 C
Manifold:	53 C
Transferline:	252 C
Source:	219 C

The current temperatures of all heated zones are shown here. Note that the temperatures of both Entrance and Exit End Caps are reported. When the trap temperature has been changed, this information helps to monitor the equilibration process.

Ion Source (Internal)

Ion Source		
Emission:	12 μ Amps	
Balance:	0 μ A/sec	Deviation: 0.0 μ A
Filament Bias:	-11.7 Volts	
Filament 1:	OK	
Filament 2:	Untested	
	On	Off
Gate:	121 Volts	-121 Volts

When the system is in Internal configuration, you can set the emission current between 10 and 100 μ Amps. The emission reading may vary by 1 μ Amps from the value set in the MS method. Balance is a measure of how well balanced the emission current is between ionization on and off. Deviation is a measure of the total deviation of emission current as ionization turns on and off. Deviation is an indication of the lens' ability to provide a symmetrical pulse of electrons into the source. If Deviation >5 μ Amps, the electron lens needs to be tuned. The filament bias is its voltage relative to ground. The bias is -11.9V for EI mode and -9.8V for CI mode. The status of both filaments is indicated as OK, Open, or Untested. If the Filament is untested, you can determine its status by activating that filament in Manual Control mode and then returning to Diagnostics mode. The gate is pulsed to +120V during ionization and -120V otherwise so that electrons enter the trap only when they are needed for ionization.

Ion Source (External or Hybrid)

Ion Source		
Emission:	25 μ Amps	
Balance:	195 μ A/sec	Deviation: 0.3 μ A
Filament Bias:	-70.0 Volts	
Filament 1:	OK	
Filament 2:	Untested	
	On	Off
Lens 2:	-80 Volts	120 Volts
Electron Lens:	100 Volts	-110 Volts
Repeller Lens:	-99 Volts	99 Volts
Lens 1:	-25.6 Volts	
Lens 3:	-19.7 Volts	
Ring Offset:	-7.4 Volts	
Endcap Offset:	-7.4 Volts	

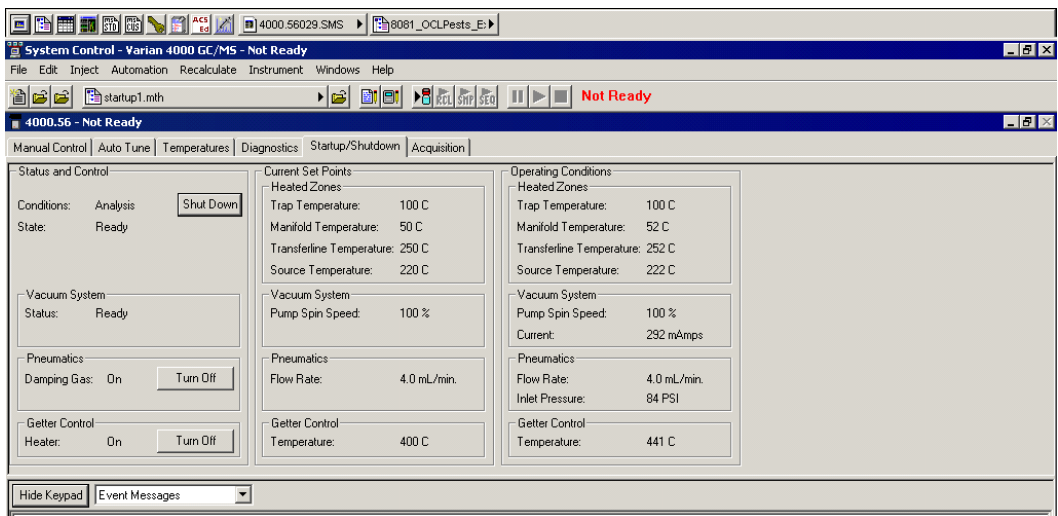
When the system is in External or Hybrid configuration the emission current is set to a default value of 25 μ Amps. In negative CI External or Hybrid, the emission current is set to a default of 250 μ Amps. The emission reading may vary by several μ Amps from this value. Balance is a measure of how well balanced the

emission current is between ionization on and off. Deviation is a measure of the total deviation of emission current as ionization turns on and off. Deviation is an indication of the lens' ability to provide a symmetrical pulse of electrons into the source. If Deviation > 5 μ Amps, the electron lens needs to be tuned. The filament bias is its voltage relative to ground. The bias is always 70V in External and Hybrid modes. The status of both filaments is indicated as OK, Open, or Untested. If the Filament is untested, you can determine its status by activating that filament in Manual Control mode and then returning to Diagnostics mode. For analysis of positive ions, Lens 2 is pulsed negative during ionization and positive otherwise so that ions enter the trap only when they are needed. For negative CI, Lens 2 is pulsed positive during ionization. All lens voltages are set by the Lens Tuning method in Auto Tune. The ring and end cap offsets are set by the Trap DC Offset Voltage method in Auto Tune.

Event Messages Display

This area displays messages when diagnostic tests are performed.

Startup/Shutdown Tab



Status and Control

The screenshot shows the 'Status and Control' panel. At the top, 'Conditions' is set to 'Analysis' and 'State' is 'Ready'. A 'Shut Down' button is visible. Below this is a 'Vacuum System' section with 'Status' set to 'Ready'. The 'Pneumatics' section shows 'Damping Gas' is 'On' with a 'Turn Off' button. The 'Getter Control' section shows 'Heater' is 'On' with a 'Turn Off' button.

Conditions displays either Analysis or Shut Down.

State field is either Ready, Shutting Down, or Starting Up.

This partial screenshot shows 'Conditions' set to 'Shut Down' and 'State' set to 'Shutting Down'. A 'Start Up' button is visible.

This partial screenshot shows 'Conditions' set to 'Start Up' and 'State' set to 'Starting Up'. A 'Shut Down' button is visible.

Vacuum System

The vacuum system is either Not Ready or Ready. The turbomolecular pump speed must be 100% for the Ready state to be reached.

Pneumatics

If the instrument is in External or Hybrid configuration, this field shows if auxiliary Helium damping gas is flowing to the ion trap. Damping gas flow is required for adequate mass spectral resolution and sensitivity in External mode. Although sensitivity is compound-dependent, a flow of 3 mL/min is a suggested starting point and the flow rate is listed in the Current Set Points area. This field does not appear when the instrument is in Internal configuration.

Getter Control

When the instrument is in External or Hybrid configuration, the getter removes water and other contaminants from the helium damping gas flow. Turn on the getter heater after turning on the damping gas flow. This does not appear when the instrument is in Internal configuration.

Current Set Points

Current Set Points	
Heated Zones	
Trap Temperature:	100 C
Manifold Temperature:	50 C
Transferline Temperature:	250 C
Source Temperature:	220 C
Vacuum System	
Pump Spin Speed:	100 %
Pneumatics	
Flow Rate:	4.0 mL/min.
Getter Control	
Temperature:	400 C

Heated Zones

The current set points of all heated zones are displayed. The source temperature is not shown when the instrument is in Internal configuration.

Vacuum System

The pump speed is indicated in the vacuum system field. It should be $100 \pm 2\%$ during normal operation.

Pneumatics

The auxiliary Helium flow rate is shown for instruments in External or Hybrid configuration. The flow is set in the Module Attributes tab dialog of Manual Control. Turn the flow on by clicking Turn On under Damping Gas in the Control and Status field. This does not appear when the instrument is in Internal configuration.

Getter Control

The getter removes water, oxygen, and other contaminants from the Helium auxiliary damping gas flow. This field is shown only when the instrument is in External or Hybrid configuration. After starting the system and turning on the Damping Gas flow, click Turn On button in the Getter Control field to heat the getter to its operating temperature of 400 °C. This does not appear when the instrument is in Internal configuration.

Operating Conditions

Operating Conditions	
Heated Zones	
Trap Temperature:	100 C
Manifold Temperature:	54 C
Transferline Temperature:	251 C
Source Temperature:	223 C
Vacuum System	
Pump Spin Speed:	100 %
Current:	308 mAmps
Pneumatics	
Flow Rate:	4.0 mL/min.
Inlet Pressure:	84 PSI
Getter Control	
Temperature:	402 C

Heated Zones

The current temperatures of all heated zones are displayed. You can compare these readings with the set points to the left in the Current Set Points field.

Vacuum System

The speed of the turbomolecular pump is $100\% \pm 2\%$ during normal operation. Monitor the speed during Startup/Shutdown here. The rate at which the turbomolecular pump is started up is proportional to how long it has been shut down. After short shutdowns for instrument maintenance, the time to reach 100% speed should be less than 2 minutes. After extended shutdowns, the pump speed is increased in stages related to power consumption. This slower startup improves pump lifetime.

NOTE: Failure to reach 100% pump speed in a reasonable time indicates a vacuum leak and corrective action should be taken. Go to the Troubleshooting section in the **4000 GC/MS Hardware Operation Manual**.

Pneumatics

If the instrument is in External or Hybrid configuration, the damping gas flow rate and Inlet Pressure are displayed. Compare the flow rate to the set point in the Current Set Points field. This does not appear when the instrument is in Internal configuration.

Getter Control

If the instrument is in External or Hybrid configuration and the Damping Gas and Getter Heater are turned on, the getter temperature is displayed. Compare this temperature to the set point of 400 °C in the Current Set Points field. This does not appear when the instrument is in Internal configuration.

Shutting Down the System

To shut down the 4000 MS, click Shut Down in the upper left corner of the screen. The heaters are turned off and the speed of the turbo pump is gradually reduced to 35% of full speed. In the following the shutdown command was activated. Notice that the turbo pump speed is being reduced as the heated zones are being cooled.

After the temperature zones are cooled, turn OFF the main power using the switch at rear of system. Manually vent the system for at least 5 minutes by opening the vent valve on the front panel one turn counterclockwise.

NOTE: Retract the transfer line before lifting the analyzer assembly from the vacuum manifold. Failing to retract the transfer line can cause damage to the transfer line tip and to the trap assembly.

The screenshot displays the '4000.56 - Not Ready' control interface. The top navigation bar includes tabs for Manual Control, Auto Tune, Temperatures, Diagnostics, Startup/Shutdown, and Acquisition. The main panel is divided into three columns: Status and Control, Current Set Points, and Operating Conditions.

Status and Control: Conditions: Shut Down, State: Shutting Down, Vacuum System Status: Not Ready, Pneumatics Damping Gas: Off, Getter Control Heater: Off. A 'Start Up' button is visible.

Current Set Points: Heated Zones (Trap, Manifold, Transferline, Source Temperatures: OFF), Vacuum System Pump Spin Speed: OFF, Pneumatics Flow Rate: 4.0 mL/min., Getter Control Temperature: OFF.

Operating Conditions: Heated Zones (Trap, Manifold, Transferline, Source Temperatures: 100 C, 49 C, 234 C, 212 C), Vacuum System Pump Spin Speed: 78 %, Current: 140 mAmps, Pneumatics Flow Rate: 0.0 mL/min., Inlet Pressure: 84 PSI, Getter Control Temperature: 379 C.

At the bottom, there is a 'Hide Keypad' button and an 'Event Messages' dropdown menu. The event log shows the following messages:

- Jan 08 14:57:37: Turning Getter OFF.
- Jan 08 14:57:37: Turning Damping Gas OFF.
- Jan 08 14:57:37: Shutdown: Pump/Heated Zones are shutting down.
- Jan 08 14:57:37: DO NOT PERFORM MAINTENANCE UNTIL SHUTDOWN IS COMPLETE.

To restart the system after activating the shutdown, click Start Up on the left side of the screen. This restarts the pumps and turn on the heaters.

Starting Up the System

Status and Control Conditions: Start Up Shut Down State: Starting Up Vacuum System Status: Not Ready Pneumatics Damping Gas: Off On Getter Control Heater: Off On	Current Set Points Heated Zones Trap Temperature: 100 C Manifold Temperature: 50 C Transferline Temperature: 250 C Source Temperature: 220 C Vacuum System Pump Spin Speed: 100 % Pneumatics Flow Rate: 4.0 mL/min. Getter Control Temperature: OFF	Operating Conditions Heated Zones Trap Temperature: 59 C Manifold Temperature: 51 C Transferline Temperature: 71 C Source Temperature: 63 C Vacuum System Pump Spin Speed: 74 % Current: 849 mAmps Pneumatics Flow Rate: 0.0 mL/min. Inlet Pressure: 85 PSI Getter Control Temperature: 57 C
---	---	--

Hide Keypad | Event Messages

Jan 09 16:08:27: Startup: Pump/Heated Zones are starting up.
Jan 09 16:08:27: Startup: Damping Gas or Getter not turned ON.

Observe the increase in Pump Spin Speed during system startup in Operating Conditions. The software is locked in the Startup/Shutdown mode until the speed reaches 100%. You will also see the temperature readings for heated zones begin to increase in the Operating Conditions field. If the system is in External or Hybrid configuration, turn on the Damping Gas flow and the Getter Heater in the Status Control field after the Pump Spin Speed reaches 100%.

NOTE: Failure of the system to reach 100% pump speed in about five minutes indicates a vacuum leak. See 'Troubleshooting' section in the *4000 GC/MS Hardware Operation Manual*.

Acquisition Tab

About Running Analyses

Selecting the **Acquisition** button activates the 4000 module for analysis. If you start an analysis while the instrument is in another mode, the software will automatically shift the MS module into Acquisition mode.

System Control - Varian 4000 GC/MS - Not Ready

File Edit Inject Automation Recalculate Instrument Windows Help

Manual Control | Auto Tune | Temperatures | Diagnostics | Startup/Shutdown | Acquisition

Status and Control
Run Time: 0:00 minutes
End Time: 1E:00 minutes
Ready
No Fault

MS Method
Segment Number: 1 of 2
Segment Description: Filament Off
Scan Range: 200 - 205

Operating Conditions
Data File
Scan Number: 0
Ionization Time: 0
Ion Count: 0

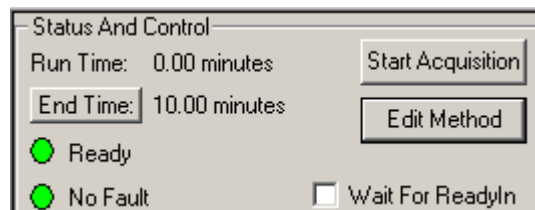
Not Ready

After starting Acquisition, the yellow (Not Ready) light turns green (Ready). The 4000 module is ready for analysis. If the GC is not ready, a Not Ready message is displayed. After the GC and AutoSampler become ready, the Not Ready message changes to Ready. To determine the individual ready states of the components, go to the menu under Windows and see the states for the 4000,

3800, and Combi PAL modules. After all components are ready, an analysis can be started.

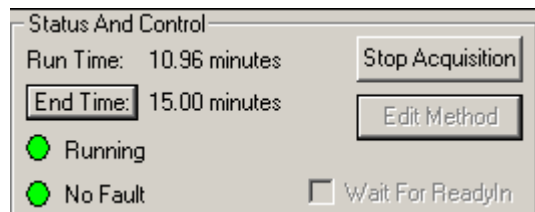
Run an analysis as a single sample or using an automated sequence. To run a single sample, go to the topic “Injecting a Single Sample” on page 159. To run an automation, go to the topic “Injecting Multiple Samples” on page 167.

Status and Control



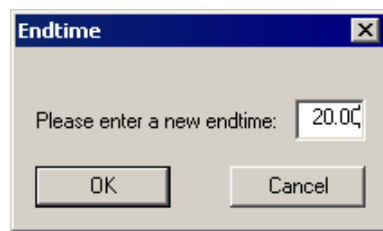
Before an acquisition starts, the Status and Control field looks like the preceding. The Run Time is 0.00 minutes. The End Time is the run length specified for the 4000 MS module in the active method. The Ready and No Fault lights are green.

Use Acquisition to override automation and start a run automatically before the system is Ready. However the file name of the run is 4000.x.sms, not the file name specified for automation runs. Click Edit Method to open the Method Builder and modify the method. You are prompted to re-activate the method after you save changes and return to System Control. **Wait For ReadyIn:** This feature is used with external devices that are not networked in to the system such as Purge and Trap systems. If **Wait For ReadyIn** is selected, the system waits for a signal before starting an acquisition.



After an acquisition starts, the run time increases in 0.01 minute increments. Stop Acquisition terminates the MS run but does not terminate the GC run. Terminate the GC from the GC module window accessed from the Windows menu. To increase the end time, click End Time and select the desired time.

NOTE: Changing the End Time for the MS module does not change the GC End Time. Extend the GC run time from the GC keypad.



MS Method

MS Method	
Segment Number:	1 of 2
Segment Description:	
Scan Description:	Filament Off
Scan Range:	200 - 205

Before an acquisition starts, the active method is in Segment 1, which is normally a Filament/Multiplier Delay segment. The length of the segment keeps the MS off until after the GC solvent elutes.

MS Method	
Segment Number:	2 of 2
Segment Description:	
Scan Description:	EI Auto FULL
Scan Range:	40 - 300

After a run starts, the Segment Number increments as each segment of the method runs. The Segment Description is the user-entered description in the Segment Description field of the MS Acquisition Method in the active method. The Scan Description indicates the ionization and ion preparation methods used for acquiring data; these methods are chosen in the Scan Type/Group and Ionization fields of the Method Segment field of the method. Scan Range is the acquisition mass range.

Operating Conditions

Data File shows the name of the file being acquired. The Scan Number, Ionization Time and Ion Count for the current scan are displayed in their respective fields. This display is updated on a scan-by-scan basis throughout the GC/MS run.

Operating Conditions	
Data File:	8081_Lin_Column_Test
Scan Number:	994
Ionization Time:	2683
Ion Count:	115117

Parameters That Affect MS Operation

Electron Multiplier Gain

Normal 4000 GC/MS operation assumes that the electron multiplier gain is set up to produce approximately 10^5 electrons per ion. The multiplier voltage needed for this gain is determined automatically by the Set Electron Multiplier method in Auto Tune. Changing the multiplier voltage will change the gain of the MS; **calibration and analysis runs should always be run at constant gain to achieve valid results.**

Under certain circumstances (low background, small signal) increasing the electron multiplier gain by +100 or +200 volts above the 10^5 gain can improve the detection limit. As noted above, the same multiplier gain must be used for determining the calibration curve and analyzing unknown samples.

Emission Current

The emission current is an adjustable parameter in Internal configuration only. It is set at a fixed 25 μ Amps for Hybrid and External configurations.

As a starting point for Internal configuration full-scan analyses, the emission current should be set so that the ionization time is slightly less than the Maximum Ionization Time for the method. Then the instrument will have its best dynamic range when data are automatically scaled to account for adjustable ionization time. If the ionization time is already lower than the max ion time when it is set for 10 μ Amps, no sensitivity enhancement (as reflected by signal-to-noise enhancement) will result from increasing the emission current.

In certain cases, a high emission current can be used to improve sensitivity. One example is EI/MS/MS in Internal configuration where the MS/MS isolation step eliminates the higher background that would normally arise from a high emission current.

Ion Trap and Ion Source Temperatures

Internal or Hybrid Configuration

It is important that the ion trap temperature be high enough so that analytes eluting from the column do not condense on trap surfaces. If condensation happens the chromatographic peaks tail. Trap temperatures can be 50-75 °C below the highest temperature for the GC column temperature ramp without compromising chromatographic performance.

Certain compounds (such as. straight chain hydrocarbons) fragment more at higher ion trap temperatures. Using a low column flow rate in combination with a trap temperature below 150 °C will improve the mass spectra when fragile ions are present.

After the system is shut down and the ion trap vented to the atmosphere, do an initial bakeout period at elevated temperatures to rapidly removal water vapor. After this, reduce the trap temperature to 150-175 °C. Use the manifold bakeout functions to do this overnight and reset the temperatures to operating conditions

automatically. Some applications may require higher temperatures and this is acceptable. In general, use the lowest temperature that *provides good chromatography*.

External Configuration

A low ion trap temperature does not cause tailing of chromatographic peaks when the instrument is in External configuration, although a Source temperature set too low may cause similar effects.

As with Internal and Hybrid modes, it is advisable to bake the system out after maintenance.

AGC Target TIC Value

In EI mode with AGC on, the target TIC value determines how many ions AGC allows into the ion trap during the calculated ionization time. While increasing the target TIC value will increase peak heights, increasing it too far will result in a loss of mass resolution. This would be observed as mass miss-assignments and/or incorrect isotope abundance ratios. For full-scan EI operation, a target TIC of 20,000 is a good starting point. Under special circumstances, e.g., MS/MS, a much lower target TIC, such as 5,000, is required. As the m/z distribution of the stored ions becomes smaller, fewer ions can be trapped without a loss of resolution.

Using 4000 MS Methods in System Control

The Startup Methods

When you open System Control and display the 4000 MS System Control Window, the Startup1.mth (first time) or the last active Method for the 4000 GC/MS is activated.

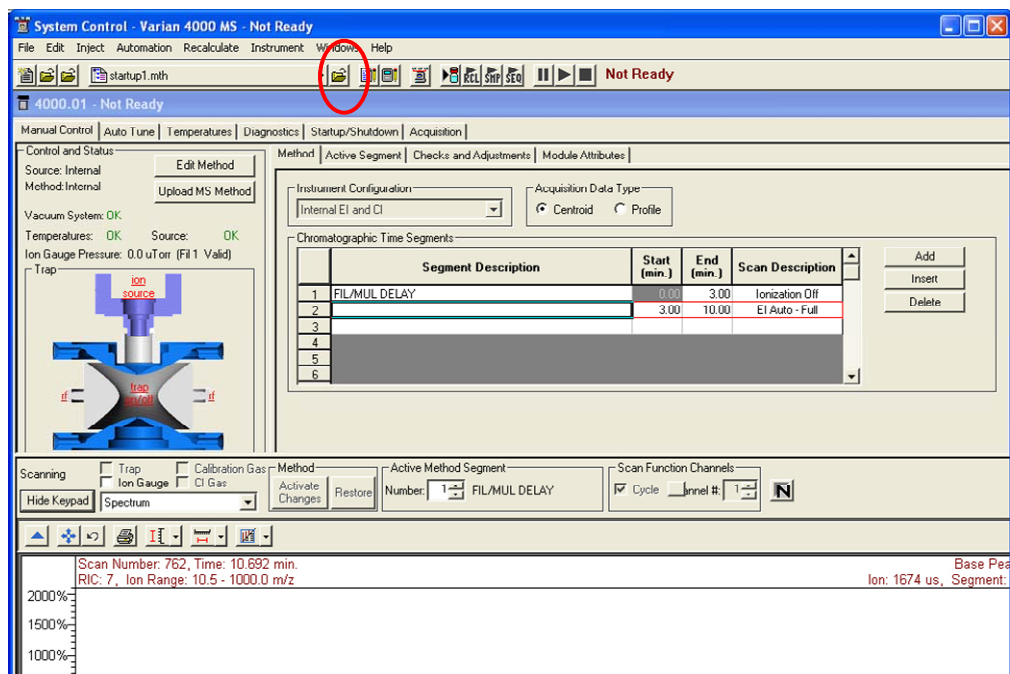
The Startup method is a copy of the Default method. You can use it to build new 4000 MS control methods.

The Default method is placed in the file directory to ensure the existence of a valid method for the operator to build on. Do not alter this method when developing new methods

Change the Startup method if you are going to use a customized set of base parameters to build methods.

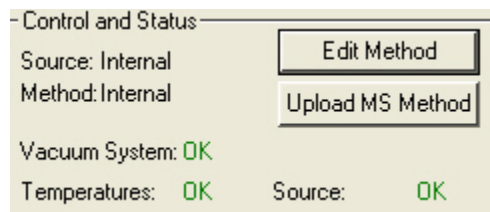
NOTE: In building new methods, clicking Defaults places instrument-valid default values in the section being built, regardless of the values placed in the Default.mth.

To change Methods, click **Activate a Method** on the System Control Toolbar or choose **Activate Method** from the File menu.



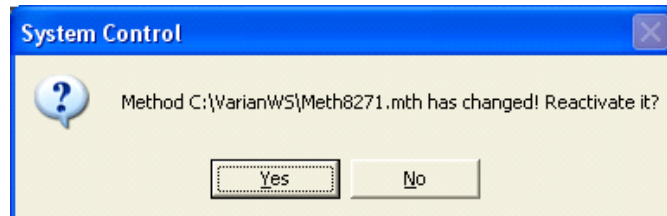
Editing Methods from the 4000 MS Status and Control Window

You can edit the method in Manual Control and save the changes immediately by clicking **Upload MS Method**. However if you wish to also edit other sections such as data handling, reports, or 3800 GC sections, click Edit Method in either the Manual Control or Acquisition Window to open the method in Method Builder.

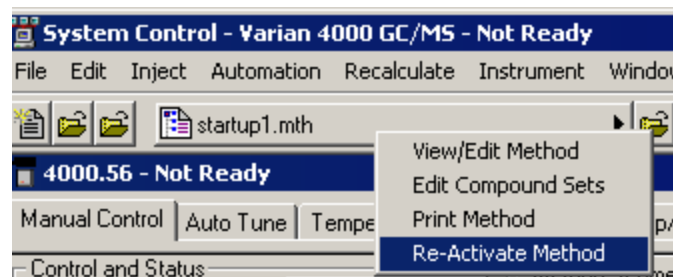


Method Builder opens with the active Method loaded. You can edit that section or other sections of the Method.

After editing and closing the Method Builder window, you are prompted to reactivate the Method. Reactivating the Method downloads the changes.

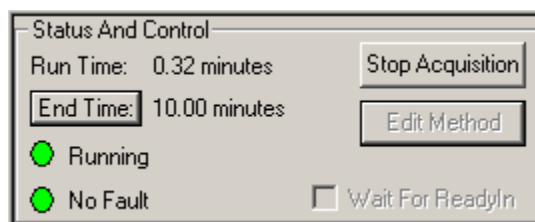


yNOTE: If you access the active method from Method Builder via the Workstation Toolbar, and then close the method editor or save a file, the method must be reactivated in System Control. To reactivate the current method, click the button for the method (in this example startup1.mth) in the System Control toolbar and then select Re-Activate Method.

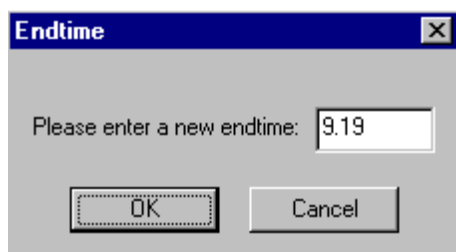


Changing Method End Time from the 4000 MS Status and Control Window

You can change the end time of the acquisition from the Status and Control window.



Click **End Time**.



Enter the new end time and click **OK**.

Effects of Hardware Configuration Changes

After changing the configuration, for example from External to Internal configuration, System Control restarts. When the 4000 MS module connects, it compares the current configuration stored in the current Module Attributes with the configuration reported by the hardware. If these do not match, the Module Attributes are updated (preset) to the appropriate configuration. A similar process occurs for the default method (Default.mth).

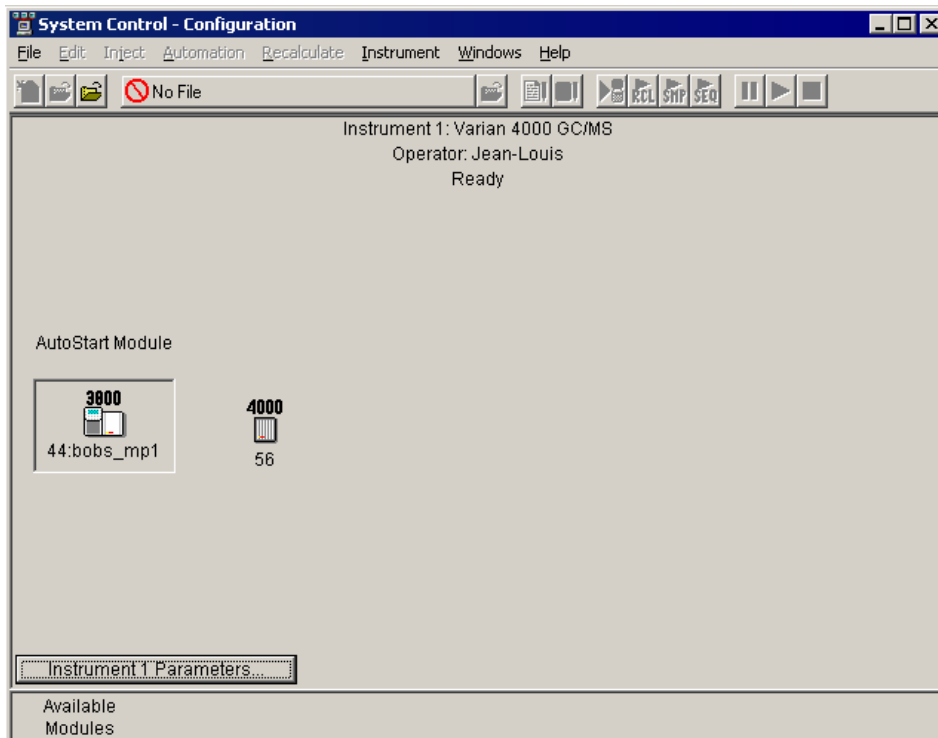
After making the hardware configuration change, any new method has the appropriate instrument configuration by default.

NOTE: The presetting of Module Attributes requires that the user run all Auto Tune routines, as the prior Auto Tune results will be invalid.

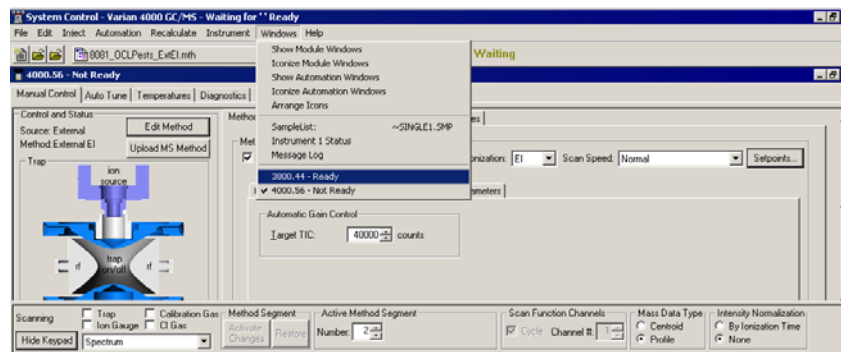
3800 GC System Control Window

The Instrument Window

After you have configured your 3800 GC in an instrument, you may view the Instrument Window. The instrument window allows you to monitor the status of all modules assigned to the instrument, perform injections of one or more samples, and perform batch Recalculations.



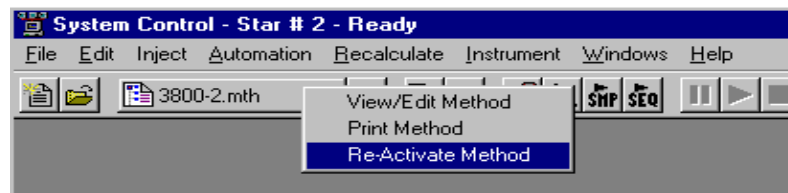
Double-click in the instrument area or select the instrument from the Instrument menu to view the 3800 GC Status and Control window. If the 4000 MS window is displayed, use the Windows menu to select the 3800 GC module.



If the Status and Control Windows for the modules configured in the instrument are not currently displayed, you can select Show Module Windows from the Windows menu and select a module for display at the bottom of the menu.

Elements of the System Control Toolbar

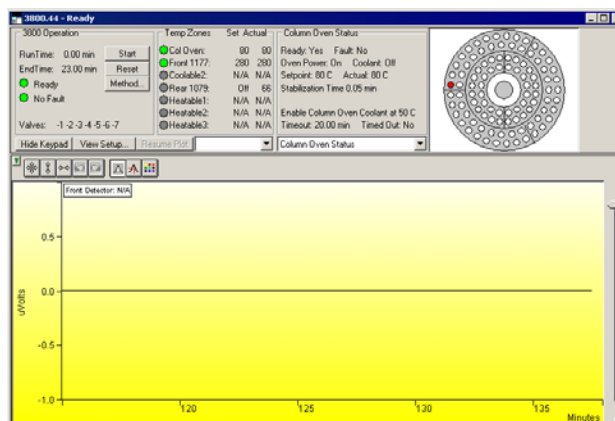
The System Control Toolbar provides you with buttons to do the most common tasks performed in System Control. Most Toolbar buttons correspond to a menu command. You can learn what each button does by resting the pointer on the button. Note that the buttons controlling the SampleList, Sequence and RecalcList become available after you activate a corresponding automation file.



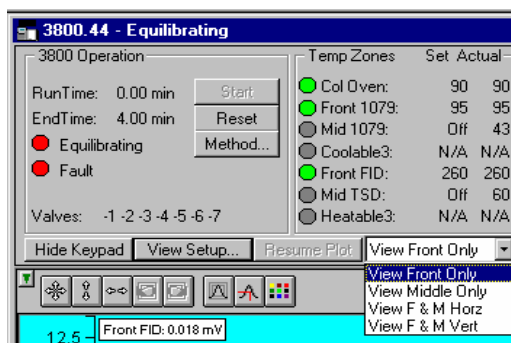
Click **Re-Active Method** for additional options.

The 3800 GC Status and Control Window

The 3800 Module Window is like the following. The left side of the window shows the 3800 Operation parameters: RunTime, End Time, Method State, and Fault State. The **Start**, **Reset**, and **Method** buttons control the 3800 GC.

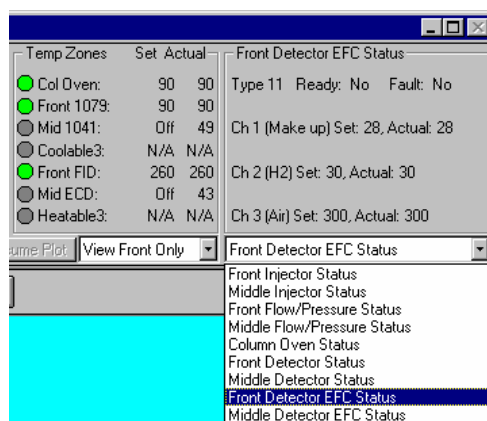


If a detector other than the MS is used for data acquisition, the detector signal can be viewed in the real time chromatogram display..



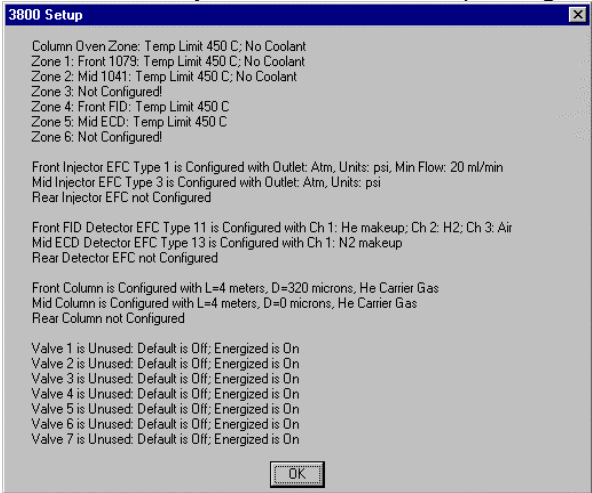
Output from multiple detectors can be viewed horizontally or vertically.

The component status display can be changed to view detailed status of each component in the GC.



Click a temperature entry to select the corresponding component status or select the component status from the list. The component status is displayed in detail.

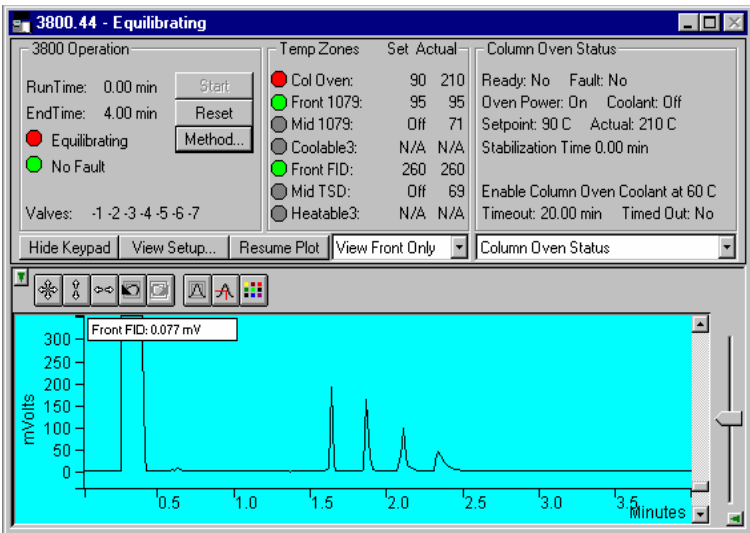
Click **View Setup** to see the 3800 Setup dialog.



The Installed hardware is listed. The setup is changed from the 3800 GC front panel.

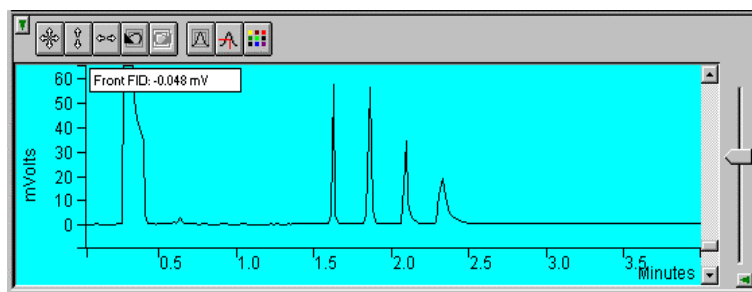
The Real-Time Chromatogram Display

All detectors installed on the 3800 GC display their chromatographic signals in real time. Configuration of the chromatogram display for all detectors is the same.



Hide Keypad

Show only the chromatogram display. Continue to view the signal after the run. Select the detector(s) to view.



Hide/unhide the Toolbar.



Set both the amplitude and time axes to full scale.



Set the amplitude axis to full scale.



Set the time axis to full scale.



Return to previous stored scaling.



Go to next stored scaling.



Enable/disable the Auto Scale feature.

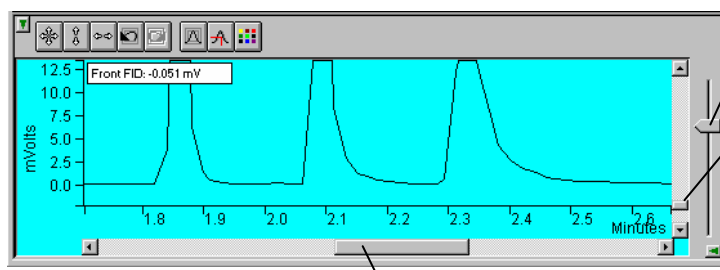
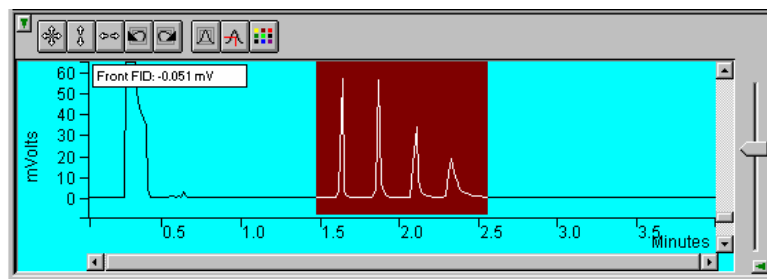


Enable/disable the cursor display.



Select the background color for the display.

Drag an area of the display to view a more detailed section of the plot, or zoom in both axes by pressing and holding the left mouse button on the spot you wish to enlarge. (Holding the Shift key down will allow you to zoom out.)



Adjust amplitude scale.

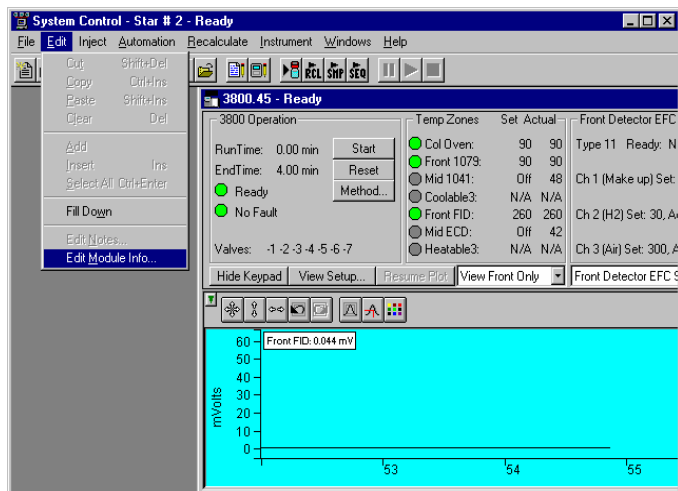
Adjust zero offset.

Hide/unhide vertical amplitude scale slider.

Adjust time scale.

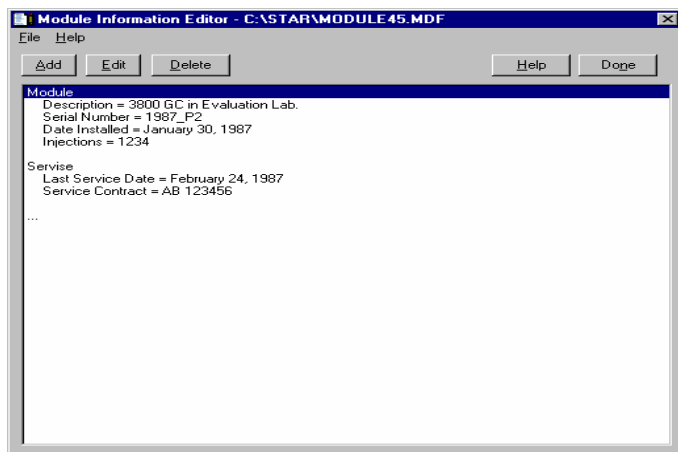
Documenting Module Information

You may wish to document the configuration of your modules, their installation and most recent service dates, and other information pertinent to your instrument. To do so, use the Module Information Editor accessed from the Edit menu in the instrument window.



Click on the module window to give it the focus.

Select **Edit Module Info** to display the Module Information Editor dialog.



Add or edit sections and items in sections.

The injections value is updated for each injection.

Select **Help** for details on creating and editing module information.

Click **Done** to close the window.

Module information is copied into Data Files generated after injections and can be included in the Run Log portion of the results report.

Configuring the GC/MS System

Starting System Control the First Time

Before you enter System Control for the first time, confirm that the proper instrument modules have been enabled and that any instrument modules that are not part of the system have been disabled. To enable or disable instrument modules see the Workstation Toolbar section, "Enable/Disable Instrument Modules" on page 13.

Configuring the Instrument

Before beginning the configuration process, you should know whether you will be attaching your Workstation and 3800 GC to a company network or to an isolated network only used for instrument control. If you have a Network Administrator on site, you may wish to ask which configuration is recommended.

Use this table to determine the order in which you should read the following sections.

1. If an Ethernet card has not been installed and configured on your PC, read "Installing and Configuring the Ethernet Card" on page 76 and one of the following:
 - No Company Network: See "Configuring the 3800 GC Communication (No Company Network)" on page 83.
 - Company Network: See "Configuring the GC for a Company Network" on page 85.
2. Read "Connecting the 3800 GC to a PC or Network" on page 82.
3. If you have not already installed the Varian MS Workstation, do so before proceeding.
 - No Company Network: See "Configuring the 3800 GC Communication (No Company Network)" on page 83.
 - Company Network: Read "Configuring the GC for a Company Network" on page 85.
4. Continue reading the rest of the sections, starting with "Adding a GC and MS to the Instrument" on page 92.

Installing and Configuring the Ethernet Card

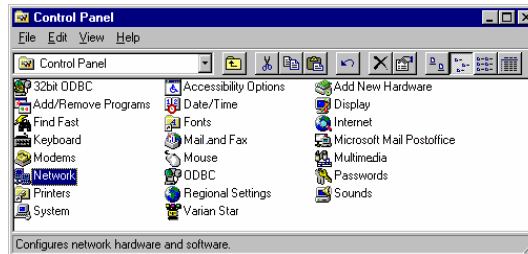
NOTE: The following section describes a procedure that is relatively generic; consult your Windows documentation and that of the Ethernet card manufacturer for more detailed explanations.

See the installation instructions packaged with your Ethernet card for information on installing the Ethernet card in your computer. Before proceeding, your Ethernet card should be recognized by your Windows version. When done, the *Network Neighborhood* icon should appear on your Windows desktop.

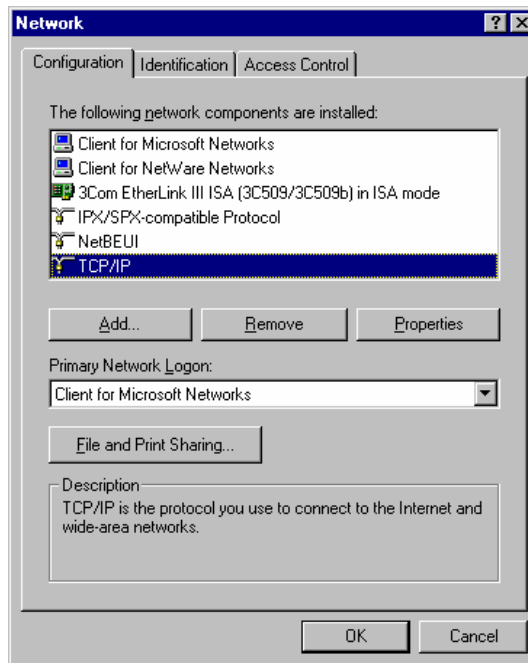
NOTE: For the following procedure, the use of disks other than the ones that were used for the original Windows installation may result in an Ethernet driver version mismatch that keeps Windows from starting. Should this occur, it may be necessary to remove the Ethernet Board from the computer to remove the incorrect Ethernet drivers.

To Configure your computer's network settings to communicate with the 3800 GC do the following:

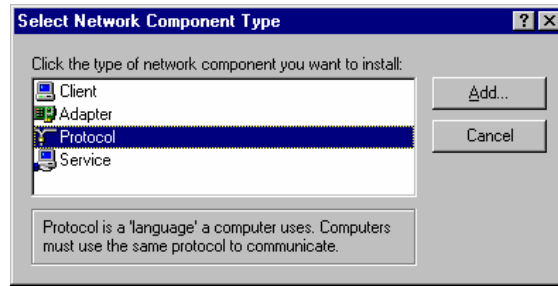
1. Run the Control Panel by selecting it from the Start menu (under Control Panel or Settings).
2. Double-click the **Network** icon.



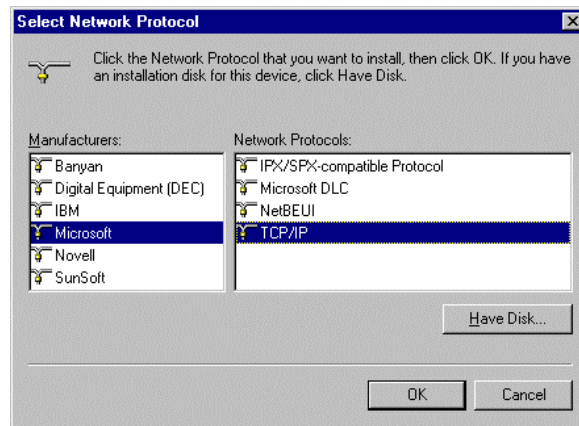
3. If TCP/IP is already listed in the list of network components, skip to the Note after step 6, otherwise, click **Add**.



4. Select Protocol and click **Add**.



5. Select **Microsoft** as the manufacturer.



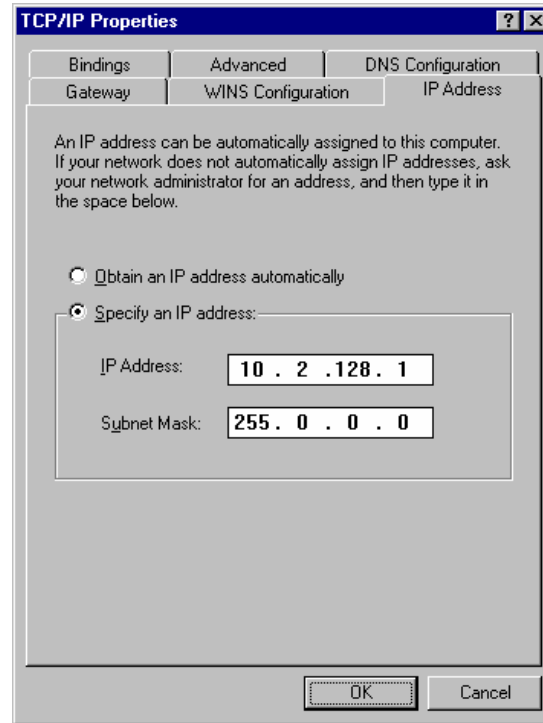
6. Select **TCP/IP** as the protocol. Click **OK**. The protocol is added to your installed network Component list.

NOTE: If you *are not* connecting your Workstation to a company network (that is, you *are not* assigned an IP address by a Network Administrator), follow the next three steps. If you *are* connecting your Workstation to a company network, skip to “Configuring the GC for a Company Network” on page 85.

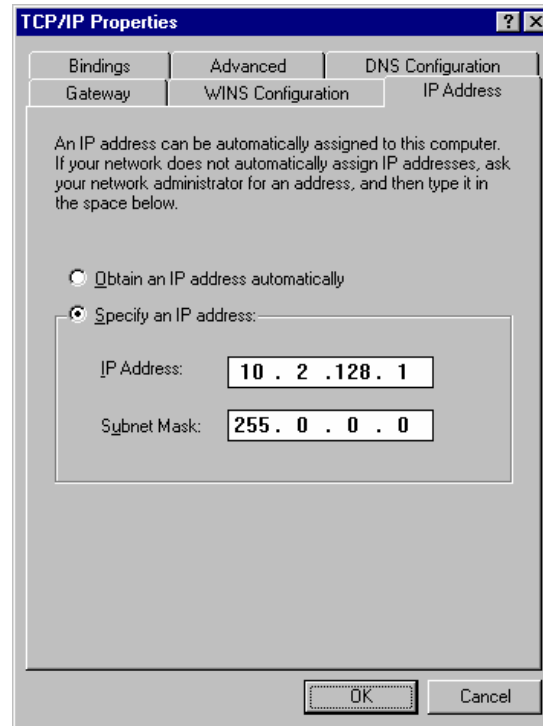
Configuring TCP/IP Parameters with no Company Network

The following three steps assume you have completed steps 1 through 3 from the previous procedure.

1. Select **TCP/IP** from the network components list and click **Properties**. The TCP/IP properties dialog box is displayed.



2. Enter an IP address in the range **10.2.128.1** through **10.255.255.254**. Note that if you are adding more than one Workstation to this network, **each Workstation PC must have a unique IP address within this range**. The Subnet Mask is automatically set to 255.0.0.0. **Do not change this value.**



Ensure that no entries have been made in your Gateway, and that Domain Name Server (DNS) and WINS Configuration have been disabled. **These entries are not used in an isolated network. They may cause problems if they are present.** Delete any settings that may have been entered from a previous configuration.

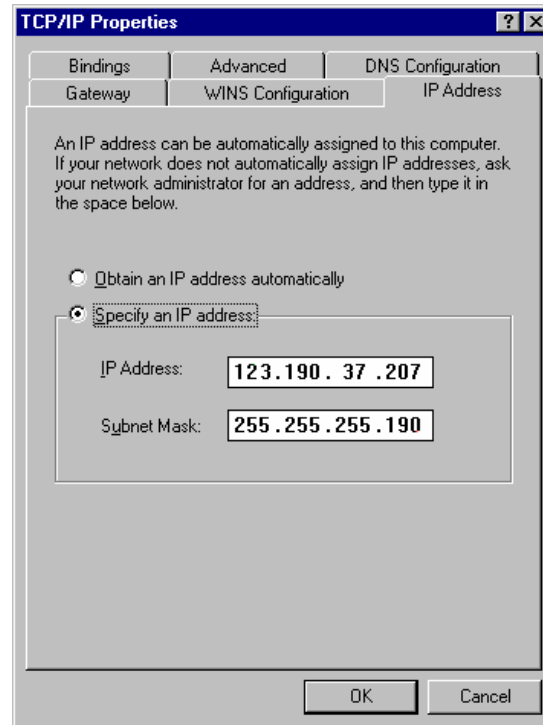
3. Click **OK** in the Network dialog box. You will need to reboot Windows for the changes to take effect.

NOTE: See *Communication Problems* in the *Diagnostic and Troubleshooting* section for information about diagnostic tools to verify that your network installation is correct.

Configuring TCP/IP Parameters for a Company Network

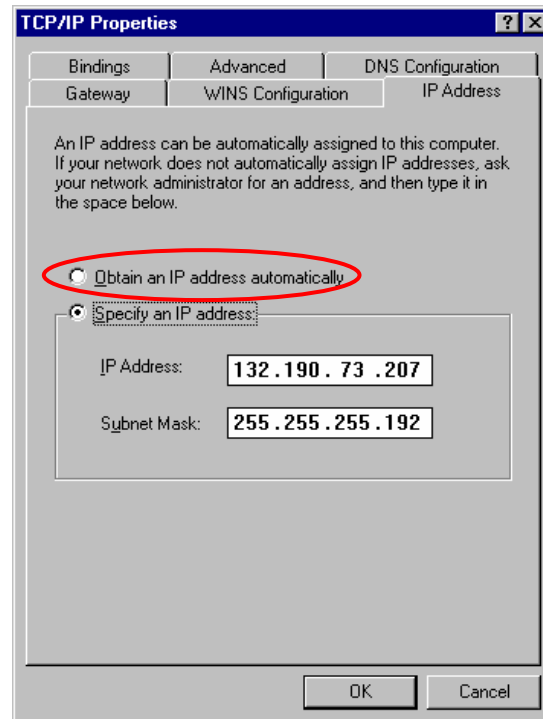
The following three steps assume you have completed steps 1 through 5 from the previous table.

1. Select **TCP/IP** from the network components list and click on the **Properties** button. The TCP/IP properties dialog box is displayed.



Enter the IP address to be used by this Workstation. Contact your Network Administrator (or whoever assigns IP addresses in your network) to get the appropriate address. **Note that each Workstation PC must have a unique IP address.** Enter the appropriate Subnet Mask to be used with this IP address.

Your Network Administrator may instruct you to obtain an IP address automatically by selecting the appropriate radio button.



Contact your Network Administrator (or whoever assigns IP addresses for your network) to see what the appropriate settings are for your Gateway, and whether Domain Name Server (DNS) and WINS Configuration are needed.

1. Your Network Administrator may also instruct you to specify parameters in the Bindings and Advanced tabs.
2. Click on **OK** in the Network dialog box. You will need to reboot Windows for the changes to take effect.

NOTE: See *Communication Problems* in the *Diagnostic and Troubleshooting* section for information about diagnostic tools to verify that your network installation is correct.

Connecting the 3800 GC to a PC or Network

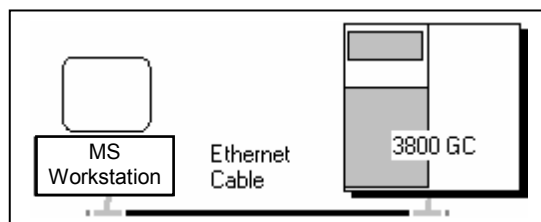
See the *Installation Instructions for the 3800 GC Communications Board* (packaged with the 3800 GC Communications Kit) for details on adding the communications board to your 3800 GC (if it has not been previously installed), and the "Communications" section of the *3800 GC Operator's Manual* for instructions on connecting your GC to the PC or a company network. Before proceeding, your GC should be connected to your PC or network.

Configuring the 3800 GC Communication (No Company Network)

Use the following instructions if both a 3800 GC and Varian MS Workstation PC are being connected to an *isolated network*, that is, a network dedicated to Varian MS Workstations and the instruments they control. In this case, a Network Administrator will not be involved in assigning IP addresses.

NOTE: If you are connecting your 3800 GC and Varian MS Workstation PC to a multi-purpose company network managed by a Network Administrator, see Configuring the 3800 GC Communication for a Company Network.

You can connect one 3800 GC directly to one Workstation...



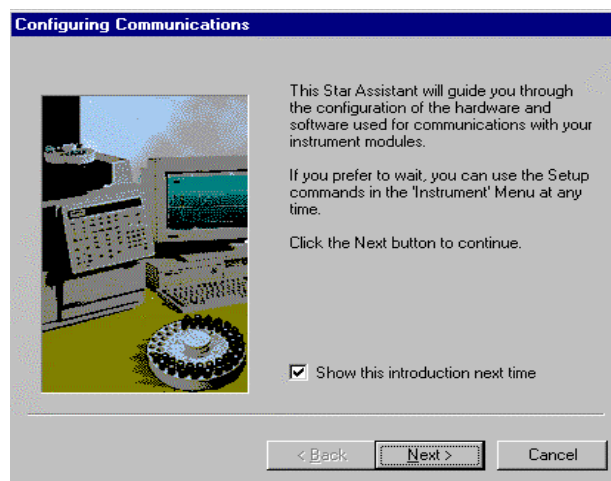
After you have configured your computer's network settings start System Control.



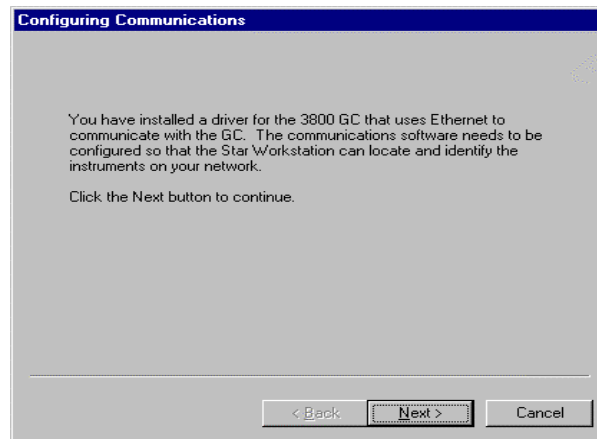
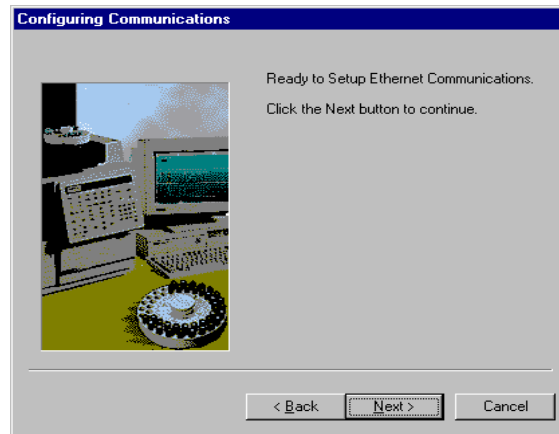
Click on the **System Control/ Automation** button.

When System Control has started for the first time, the Star Assistant Communication Configuration Wizard will appear to guide you through the setup and network connections of your 3800 GC. Use the following procedure for configuring the IP address for your GC.

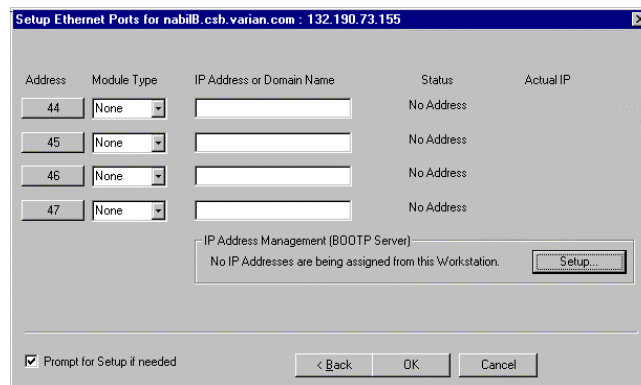
The first time the Star Assistant guides you through the setup of your 3800 you will get the following messages:



Read the description presented in each window and click on the **Next** button to advance to the next step.



From the Setup Ethernet Ports dialog box, click on **Setup** for IP Address Management [BOOTP Server].



Click on the **Setup** button.

When the Setup BOOTP Server dialog box is displayed, turn on the 3800 GC that has been connected to the network.

NOTE: If more than one Workstation is being connected on the same network, only one Workstation should manage the IP addresses for all GCs connected (all others should have the *Manage IP addresses from this Workstation* checkbox **unchecked**). If more than one Workstation is managing IP addresses, naming conflicts may arise.

As the GC is powered on, an entry is added to the table. For each entry, an IP address is automatically assigned. For each GC, you provide a host name that will be used to identify it in the Varian MS Workstation.

	Ethernet Address	IP Address	Host
1	00:60:93:00:84:0e	10.0.132.14	Pik
2			
3			
4			
5			
6			
7			

This Workstation will assign IP Addresses as 3800 GCs are powered on. Use this feature when only Workstations and 3800 GCs are connected to the network. You must enter a Host Name for each 3800 GC, after its IP Address is assigned, before it can connect to any Workstation.

This Workstation will assign these settings to each 3800 GC

Subnet Mask: 255.0.0.0
Gateway: 0.0.0.0
Domain: <unnamed>

Click on the **Manage IP addresses from this Workstation** checkbox to manage your GC's IP address from this Workstation. As the GC is turned on, its Ethernet address is added to the list. The IP address is automatically generated. Enter a host name for each GC. This name is used to identify the GC in the Workstation. **You cannot connect to a GC until you provide it a name.** Click on **OK** when host names for each GC have been entered.

NOTE: Proceed to "Using a Password to Protect BOOTP Settings" on page 90.

Configuring the GC for a Company Network

Use the following instructions if both a 3800 GC and Varian MS Workstation PC are being connected to a *company network*, that is, a multi-purpose network used for services in addition to instrument control. In this case, a Network Administrator will be involved in assigning IP addresses. If the GC and the Varian MS Workstation computer are the only devices being connected, see "Configuring the 3800 GC Communication (No Company Network)" on page 83.

You may connect virtually any number of 3800 and 3900 GCs and any number of Varian MS Workstation computers to a company Ethernet network.

Depending upon your network configuration, all Varian MS Workstations will be able to control all 3800 and 3900 GCs on the network.

After you have configured your computer's network settings start System Control.



Click on the **System Control/ Automation** button on the Workstation Toolbar to start System Control.

When System Control has started for the first time, the Star Assistant Communication Configuration Wizard will appear to guide you through the setup and network connections of your GC.

NOTE: Before proceeding with this section, consult your Network Administrator about whether a central BOOTP Server is available on the network.

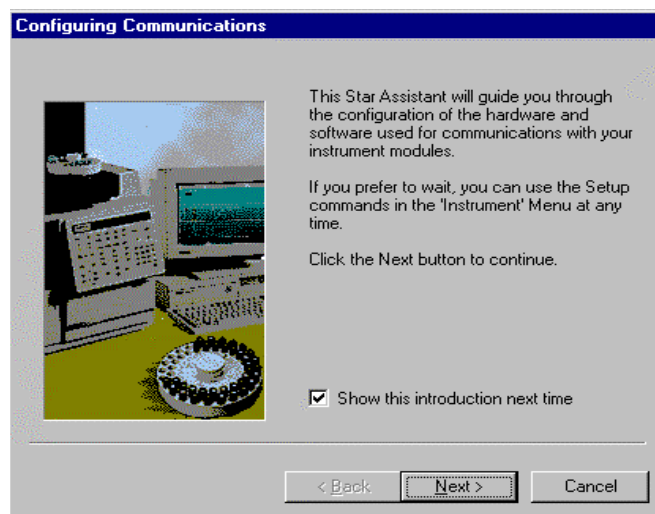
1. If IP addresses are managed by a central BOOTP Server, then proceed to *Specifying IP Addresses from a Central BOOTP Server*.
2. If IP addresses are specified by a Network Administrator but not centrally managed, then continue reading *Specifying IP Addresses from System Control*.

Specifying IP Addresses from System Control

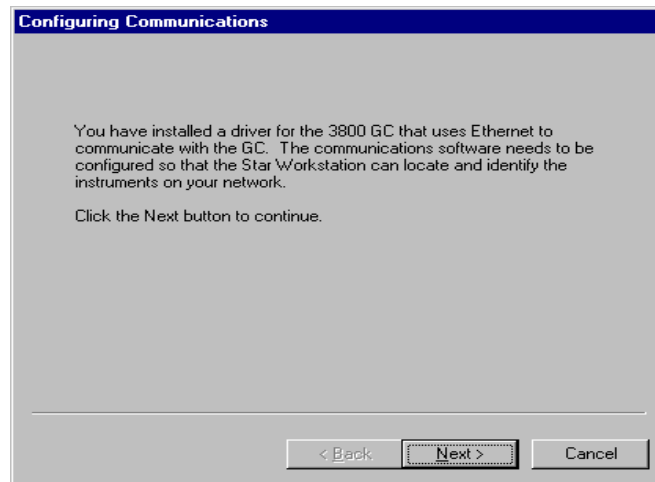
The Varian MS Workstation can be used to specify IP addresses to 3800 and 3900 GCs attached to the network.

Use the following procedure to manage IP addresses from System Control.

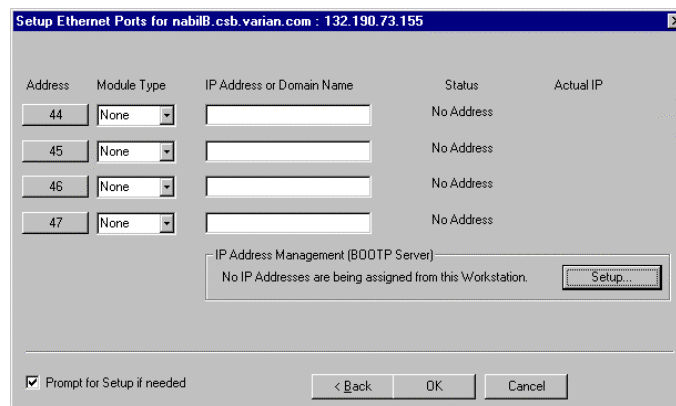
The first time the Star Assistant guides you through the setup of your GC the following opens:



Read the description presented in each window and click **Next**.



From the Setup Ethernet Ports dialog box, select **Setup** to display the *BOOTP Server* dialog box.



Select **Setup** to display the *BOOTP Server* Dialog Box.

The BOOTP Server dialog contains a table listing all 3800 and 3900 GCs to which IP addresses may be assigned.

NOTE: If more than one Workstation is being connected on the same network, only one Workstation should manage the IP addresses for all GCs connected (all others should have the *Manage IP addresses from this Workstation* checkbox **unchecked**). The Workstation acting as BOOTP Server must remain running all the time to allow other Workstations to connect to GCs. If more than one Workstation is managing IP addresses, naming conflicts may arise.

Setup BOOTP Server at 132.190.73.155

☒ Manage IP addresses from this Workstation
☐ Require password entry for this dialog box

	Ethernet Address	IP Address	Host
1	00.60.93.00.84.0e	0.0.0.0	Pil
2			
3			
4			
5			
6			
7			

Manually enter an IP Address and Host Name corresponding to each Ethernet Address in the table. Use this feature when individual IP Addresses and Host Names have been reserved for use by each 3800 GC, but IP Address and Host Name management is not performed by a Network Administrator.

☒ Assign IP addresses manually
☐ Assign: 0 IP addresses starting from: 0.0.0.0

This Workstation will assign these settings to each 3800 GC:

Subnet Mask: 255.255.255.192
Gateway: 132.190.73.129
Domain: csb.varian.com

Ok Cancel

Check Manage IP addresses from this Workstation box to enable the BOOTP Server.

If 3800 or 3900 GCs are already connected to the network and powered on, their Ethernet addresses will appear in the table.

You must enter a host name for each GC. The IP address will not be assigned to the GC until a name is entered.

You may manually assign any IP address to any GC. When this item is selected, type the IP addresses directly into the table. Select OK when all addresses and names have been entered.

When the *Assign # IP addresses starting from: #. #. #. #* radio button is selected, the bottom portion of the dialog changes.

This Workstation will assign IP Addresses from a reserved block of addresses as 3800 GCs are powered on. Use this feature when IP Addresses have been reserved for use by a pool of 3800 GCs, but IP Address management is not performed by a Network Administrator. You must enter a Host Name for each 3800 GC, after its IP Address is assigned, before it can connect to any Workstation.

☐ Assign IP addresses manually

☒ Assign: IP addresses starting from:

This Workstation will assign these settings to each 3800 GC

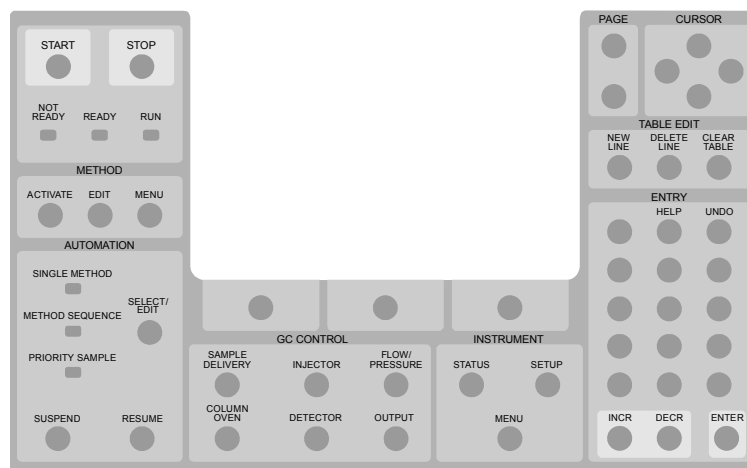
Subnet Mask:	255.255.255.192
Gateway:	132.190.73.129
Domain:	csb.varian.com

Consecutively assign IP addresses beginning from a particular address. Enter the number of IP addresses you want to assign automatically. Enter the starting address here. As 3800s are powered on, IP addresses are automatically assigned starting with this address.

NOTE: Proceed to “Using a Password to Protect BOOTP Settings” on page 90.

Specifying IP Addresses from a Central BOOTP Server

If IP addresses are managed by a Network Administrator from a central source, the 3800 and/or 3900 GCs must be added to the list of devices requiring IP addresses. IP addresses must be assigned to GCs using a BOOTP Server. A BOOTP Server lists Ethernet addresses (which are unique to each communication card installed in each 3800 and 3900 GC) along with the IP addresses that are to be assigned to the corresponding device. You may obtain the Ethernet address for each GC from the GC’s front panel or the label on the Ethernet card within the 3800 GC. Turn on the 3800 GC and press any key to allow it to start in local mode. The Ethernet address will be automatically loaded into the BOOTP Server list for 3800 and 3900 GCs.



When the GC has completed its initialization process, press the **menu** button.

The Instrument menu is displayed. Press **1** to select the Remote/ Local Control screen. The Ethernet address, IP address and Alias (which may not yet be assigned) are displayed.

ACTIVE METHOD	CTRL METHOD	CTRL TIME	CTRL TIME
Method 1	Method 1	0.00	20.00
REMOTE / LOCAL CONTROL			
Enable			
Ethernet Address: XX:XX:XX:XX:XX:XX			
IP Address: XXX.XXX.XX.XXX			
Alias: 3800A			

Use the Ethernet address shown here when setting up the BOOTP Server. The IP Address and Alias (host name) are assigned by the BOOTP Server.

If the BOOTP Server that you are using automatically updates its table with Ethernet addresses broadcast by devices on the network, then the Ethernet addresses from 3800 and 3900 GCs connected to your network and powered on will automatically appear. Note that the frequency with which these addresses are broadcast by the GC decreases over time to a minimum rate of once per minute. This means that the BOOTP Server may not receive an Ethernet address for up to a minute from any given GC.

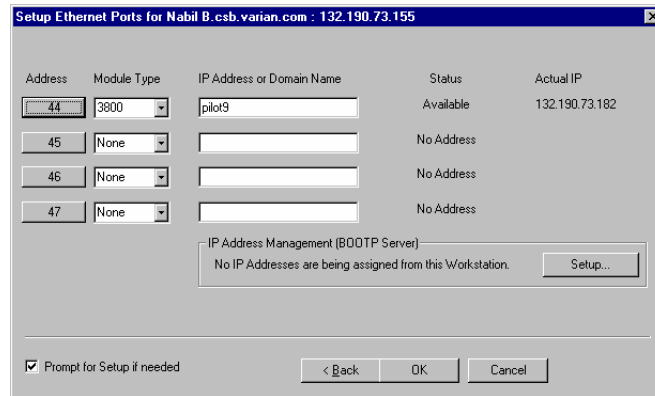
Once you have entered the Ethernet address for each networked GC into the BOOTP Server, power each GC off then on again. Check that the IP address shown on the front panel of each GC (in the Remote/Local Control screen) matches the intended address entered into the BOOTP Server. If they do not match, verify the Ethernet address, updating the BOOTP Server if necessary. Powering the GC off and on will update the IP address.

NOTE: When IP addresses are managed from central location by a Network Administrator, the BOOTP Server on your workstation must be disabled. Verify that the *Manage IP addresses from this Workstation* is unchecked in the Setup Ethernet Ports dialog box.

Setup BOOTP Server at 132.190.73.155			
☐ Manage IP addresses from this Workstation			
☐ Require password entry for this dialog box			
	Ethernet Address	IP Address	Host
1			
2			
3			

Using a Password to Protect BOOTP Settings

You may wish to restrict access to the BOOTP Server dialog box to avoid inadvertent or unauthorized changes to IP address assignments. To do so, enable password protection in the BOOTP Server dialog box. If the Setup Ethernet Ports dialog box is not displayed, select the *Setup Ethernet Communications* menu item from the *Instrument* menu to display the Star Assistant Communication Wizard. Click on the **Next** button to advance to the Setup Ethernet Ports dialog box.



Setup Ethernet Ports for Nabil B. csb.varian.com - 132.190.73.155

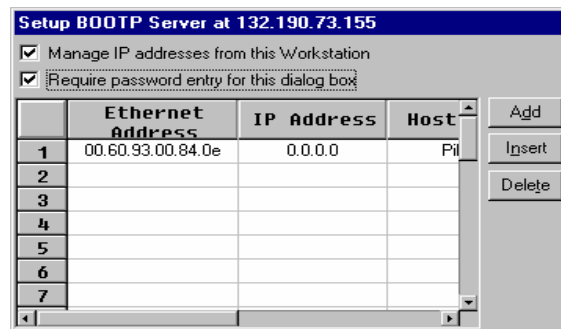
Address	Module Type	IP Address or Domain Name	Status	Actual IP
44	3800	pilot9	Available	132.190.73.182
45	None		No Address	
46	None		No Address	
47	None		No Address	

IP Address Management (BOOTP Server)
No IP Addresses are being assigned from this Workstation. Setup...

☒ Prompt for Setup if needed < Back OK Cancel

Select Setup to display the BOOTP Server dialog box.

The BOOTP Server dialog is displayed.



Setup BOOTP Server at 132.190.73.155

☒ Manage IP addresses from this Workstation
☒ Require password entry for this dialog box

	Ethernet Address	IP Address	Host
1	00.60.93.00.84.0e	0.0.0.0	Pil
2			
3			
4			
5			
6			
7			

Add Insert Delete

Check the box to enable password protection.

The next time you enter the BOOTP Server dialog box from the Setup Ethernet window, you will be prompted for a password.



Password Entry

IP Address management requires password entry. Enter the password below.

OK Cancel

Change Password

Enter new password:

Re-enter new password:

Enter a password. After it is successfully entered, you may change it. Enter a new password twice.

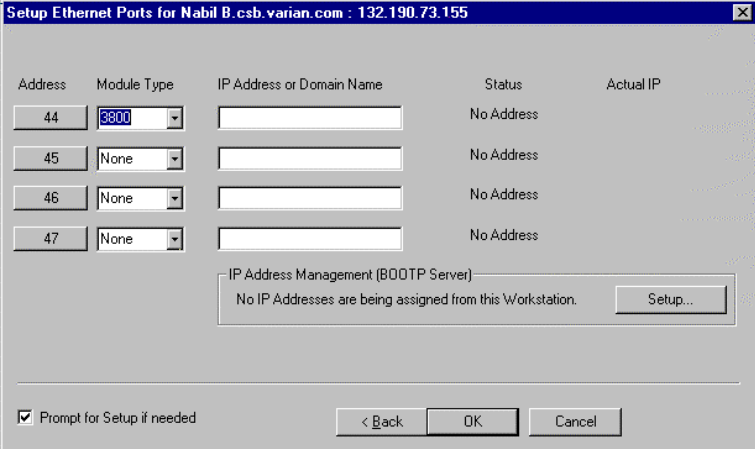
The initial password is blank (no password). To set your password initially, enter the desired password in the *Enter new password* and *Re-enter new password* fields. Subsequent entry into the BOOTP Server will require this password.

NOTE: See *Recovering a Lost Password for BOOTP Server Access* in the *Diagnostics/Troubleshooting* section for instructions on resetting the password.

Adding a GC and MS to the Instrument

Once you have configured your Workstation computer and 3800 GC for network communication, you may select one GC to be controlled from each Varian MS Workstation.

If it is not already running, start System Control. The Star Assistant Communication Configuration Wizard will be displayed if you have not yet configured your GC Ethernet connections. Click on the **Next** button to advance to the Setup Ethernet Ports dialog box.



The dialog box is titled "Setup Ethernet Ports for Nabil B. csb. varian.com : 132.190.73.155". It contains a table with five columns: Address, Module Type, IP Address or Domain Name, Status, and Actual IP. There are four rows for addresses 44, 45, 46, and 47. Address 44 has a dropdown menu showing "3800". All other rows show "None" in the Module Type column. The Status column for all rows is "No Address". Below the table is a section for "IP Address Management (BOOTP Server)" with a message "No IP Addresses are being assigned from this Workstation." and a "Setup..." button. At the bottom, there is a checkbox "Prompt for Setup if needed" which is checked, and three buttons: "< Back", "OK", and "Cancel".

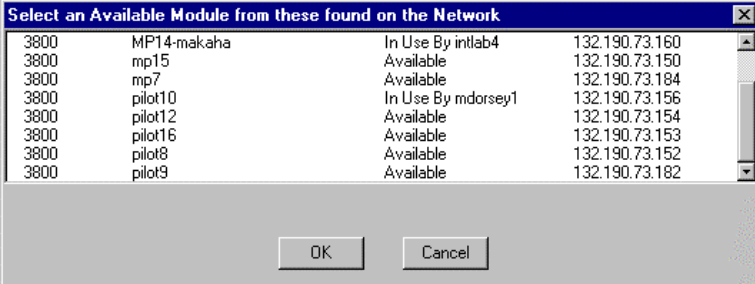
Address	Module Type	IP Address or Domain Name	Status	Actual IP
44	3800		No Address	
45	None		No Address	
46	None		No Address	
47	None		No Address	

IP Address Management (BOOTP Server)
No IP Addresses are being assigned from this Workstation. Setup...

☒ Prompt for Setup if needed < Back OK Cancel

Click on the **Address** button to attach to a 3800 GC on the Ethernet network.

When the **Address** button is selected, the Module Selection dialog box is displayed.



The dialog box is titled "Select an Available Module from these found on the Network". It contains a table with four columns: Module ID, Module Name, Status, and IP Address. The table lists several modules, including MP14-makaha, mp15, mp7, pilot10, pilot12, pilot16, pilot8, and pilot9. The Status column indicates whether the module is "In Use By" a specific user or "Available". The IP Address column shows the corresponding IP for each module. At the bottom, there are "OK" and "Cancel" buttons.

Module ID	Module Name	Status	IP Address
3800	MP14-makaha	In Use By intlab4	132.190.73.160
3800	mp15	Available	132.190.73.150
3800	mp7	Available	132.190.73.184
3800	pilot10	In Use By mdorsey1	132.190.73.156
3800	pilot12	Available	132.190.73.154
3800	pilot16	Available	132.190.73.153
3800	pilot8	Available	132.190.73.152
3800	pilot9	Available	132.190.73.182

OK Cancel

Select the GC that you wish to connect to the address (44). Click on **OK** to make the selection.

NOTE: If you are running on a company network, only GCs on the same local subnet appear in the Select Available Modules dialog box. To connect to a GC in a different subnet, type its IP Address directly into the IP address field in the Setup Ethernet Ports dialog box. Consult your Network Administrator about subnets on your network.

After you have completed your selection, the Setup Ethernet Ports Dialog Box shows the 3800 GC connected to your Workstation.

Address	Module Type	IP Address or Domain Name	Status	Actual IP
44	3800	pilot9	Available	132.190.73.182
45	None		No Address	
46	None		No Address	
47	None		No Address	

IP Address Management (BOOTP Server)
No IP Addresses are being assigned from this Workstation. Setup...

☒ Prompt for Setup if needed < Back OK Cancel

This 3800 GC is connected to Port 44. The status indicates the availability of the GC. The IP address of the GC is shown here. Click on the **OK** button to close the dialog box, and wait for the 3800 to connect.

When the GC connects to System Control, it will be indicated by an icon at the bottom of the Configuration Window. The icon will have a label reading 3800.44, and will include the Host Name of the GC appended to the label. The number 44 is a System Control communication addresses that correspond to the Ethernet port connected to the device.

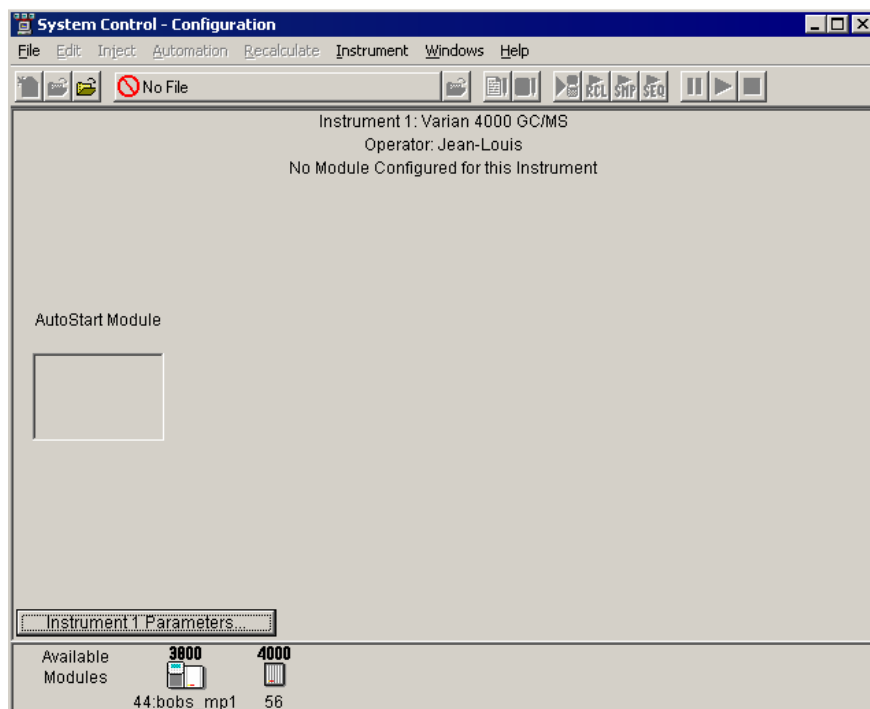
Modules and Instruments Defined

The Varian 4000 GC/MS system consists of two modules, the 3800 GC and 4000 MS. A third module is present if you have the Combi PAL AutoSampler. (Note that the CP-8400 and CP-8410 AutoSamplers will not have a separate icon but will be included on the 3800 Status and Control Window). These modules also have addresses and the message logs frequently refer to the module and address. The modules and *allowed* addresses are listed below.

Module	Name	Address
Mass Spectrometer	4000	56
Gas Chromatograph	3800	44
AutoSampler	Combi PAL	24

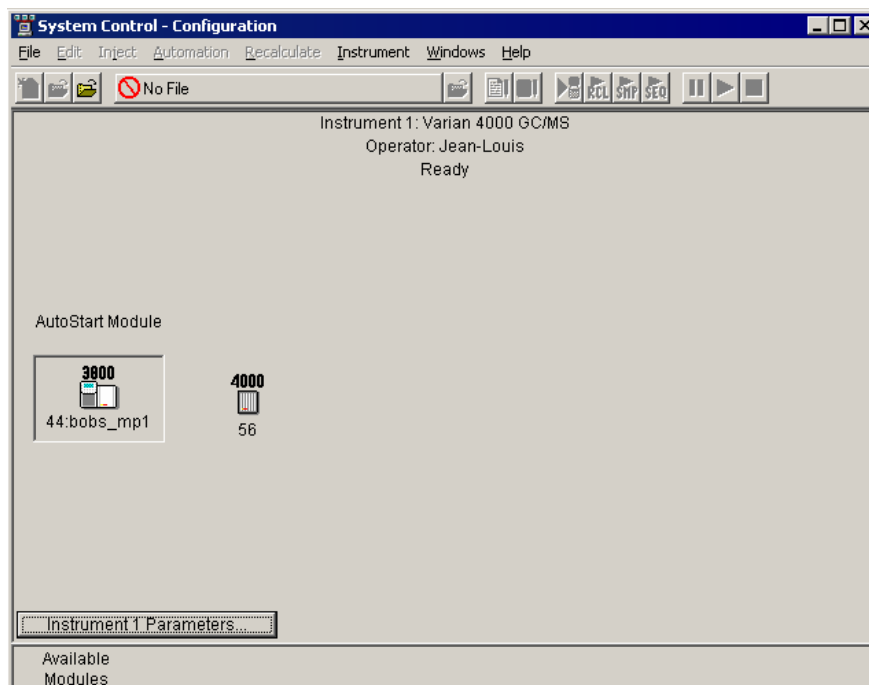
Elements of the Configuration Screen

After the 3800 connects to System Control, configure it in an Instrument by moving its icon from the Available Modules of the Configuration Window into the Instrument Area. System Control will remember where to put the icon the next time that the 3800 connects to System Control.



The 4000 GC/MS is usually configured into 2 modules, the 4000 MS and the 3800 GC. A Combi PAL AutoSampler can also be part of the system. After each module connects to System Control, configure it in an Instrument by moving its icon from the bottom of the Configuration Window into the Instrument Areas. System Control will remember where to put the icon the next time that the system connects.

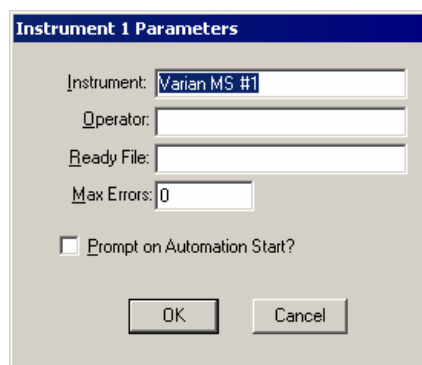
Place the 3800 GC icon in the AutoStart Module window if either a CP-8400 or CP-8410 AutoSampler will be used with the GC. If a Combi PAL will be used for automation, place the Combi PAL icon inside the AutoStart Module window. If the 3800 will be operated *without* an AutoSampler controlled from the Varian MS Workstation, leave the AutoStart Module box empty. If you are using a sampling device with the 3800, the device must be connected to the synchronization port of the GC.



Once the modules are in place you are ready to proceed.

Setting Instrument Parameters

Some parameters used by the Instrument are set in the Instrument Parameters dialog box accessed from the Configuration window.



The instrument name appears in the instrument window. Also, note that the instrument and operator names entered here will appear on any automated reports generated by the MS Workstation. The Ready File is not used with 4000 GC/MS systems. When the maximum number of non-fatal errors (Max Errors) is exceeded during automation, the automation sequence halts. Setting this value to zero disables this feature. You are optionally prompted for this information when you start an automated sequence of injections or Recalculations. If the Prompt on Automation Start box is checked, this dialog will appear when an Automation is started. This feature can be particularly useful if different operators use the GC/MS instrument.

NOTE: Do not enter any characters in the Ready File field or the MS will not come to Ready for injections. For example, if the characters **aa** were entered in the Ready File field, the System Control title bar would contain the “Waiting for ‘aa’ Ready” warning as shown here. Even an invisible space character in this field can cause the problem.



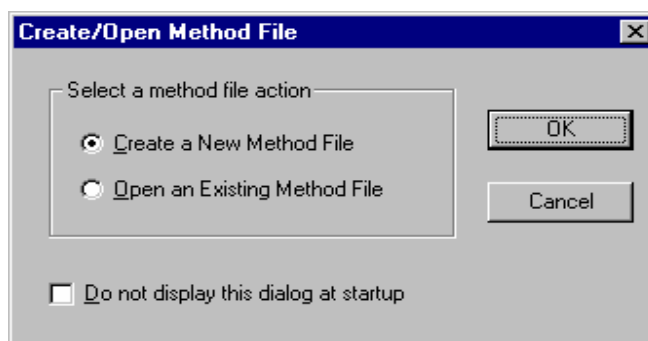
Running an MS Method without a GC Method

To run an MS method without the GC module or GC method, open System Control. Under the menu Instrument, select Configuration. Using the mouse, click and drag the GC icon, and AutoSampler icon, if present, from the middle field to below the bar labeled Available Modules. When an MS method is activated, the GC method will not be downloaded. To activate the GC and AutoSampler modules, click-drag-drop the icons into the active field.

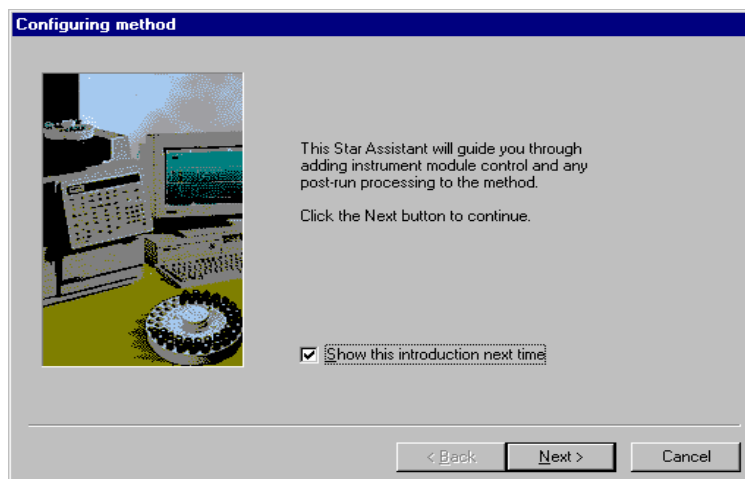
Building GC/MS Methods

Using Star Assistant to Create a New Method

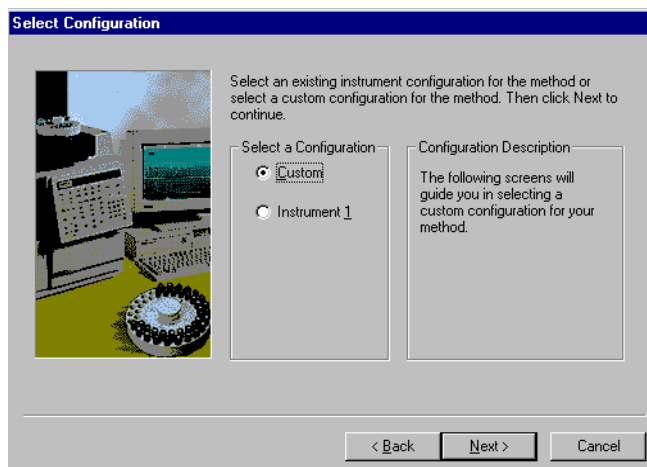
Click the **Method Builder** button  on the Workstation Toolbar, to open the **Create/Open Method File** dialog box.



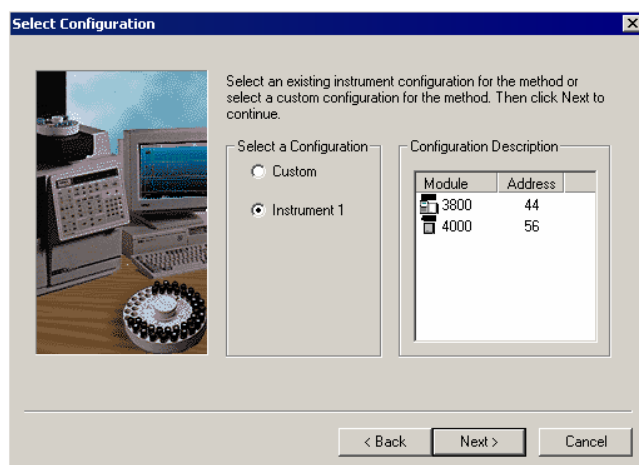
If you choose *Create a New Method File*, the Star Assistant Wizard will guide you in building this new method. To avoid seeing this message again, uncheck the box "Show this introduction next time."



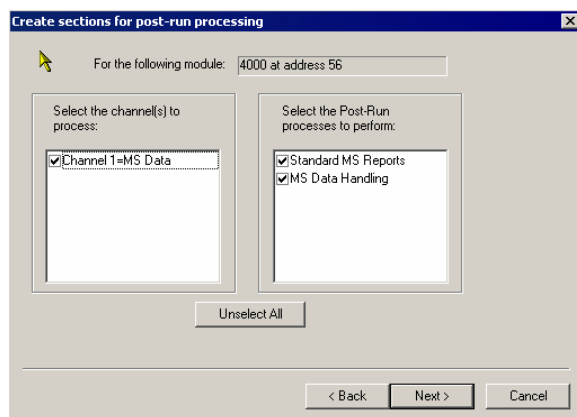
1. Select the instrument configuration for the method, either Custom or Instrument 1. Use Custom configuration to create methods on a PC remote from the instrument.



2. Select Instrument 1 for the 4000 MS and 3800 GC and click **Next**.

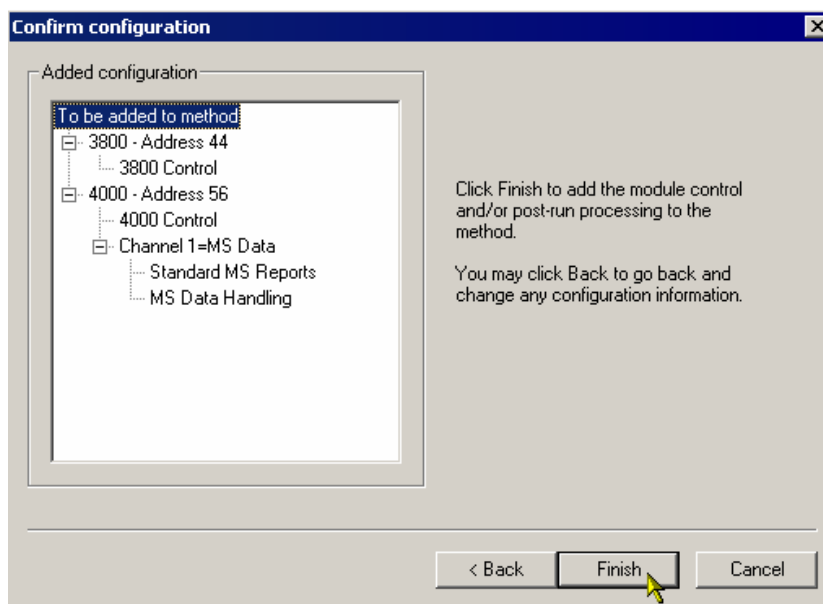


3. Select the channels for post-run processing. If there is a Combi PAL, it is present as a module in the Configuration Description.



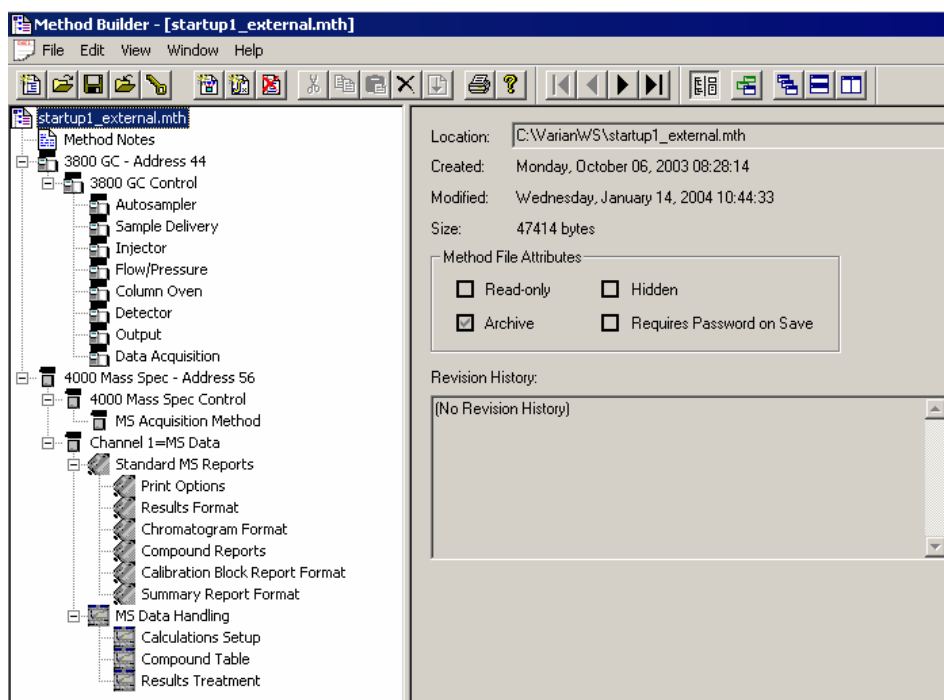
4. After you select the data channels and type(s) of post-run processing for each detector, the Star Assistant creates a Method containing all the sections needed to control the hardware, collect data, and do the post-run processing

specified. These Method sections contain default values for all parameters, which you can edit as needed for your analysis. This manual has data acquisition information for GC/MS methods. Data handling and reports are covered in the MS Workstation Software Reference Manual.



Method Builder Window

Overview



The Method is displayed in two panes. The Directory pane on the left is an outline of the Method. Clicking on an item in the Directory changes the content of the Parameters pane on the right. Click on the splitter bar separating the panes to resize them. To hide the Directory, click the **Show/Hide Method directory**

button  on the Window toolbar.

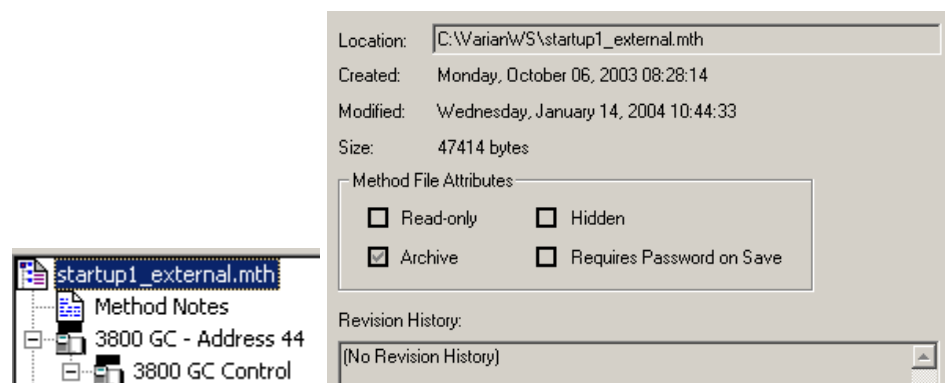
These menus are at the top of the screen: File, Edit, View, Window, and Help. Click on each to see its contents. Below this are the three toolbars: the Main toolbar to open, save, and print Methods; the Directory toolbar to traverse the Directory outline; and the Window toolbar to position Method windows. Hold the mouse cursor over each button to see a Tooltip describing its function. A status bar is at the bottom of the Method Builder window. The toolbars and the status bar may be hidden or shown using the View menu.

The Method Builder can be used to display and edit multiple Method files. If the Method Builder is already displaying one method, you can open another using the **File Open** command. From the Window menu or using the Window toolbar, the Methods can be selected, cascaded, or tiled to compare the same sections in different Method files.

Method Parameters

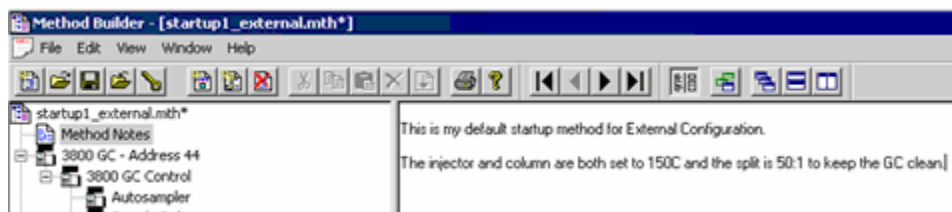
The Parameters pane displays the content of the method section selected in the Directory. Four types of windows can be displayed in the Parameters pane: method file information, method notes, hardware module descriptions, and editable parameters for the instrument modules, data handling, and reports.

File Information



Selecting the file name at the top of the Directory displays method-related information, including name and directory path, creation and modification dates, file size, file attributes, and the revision history.

Method Notes



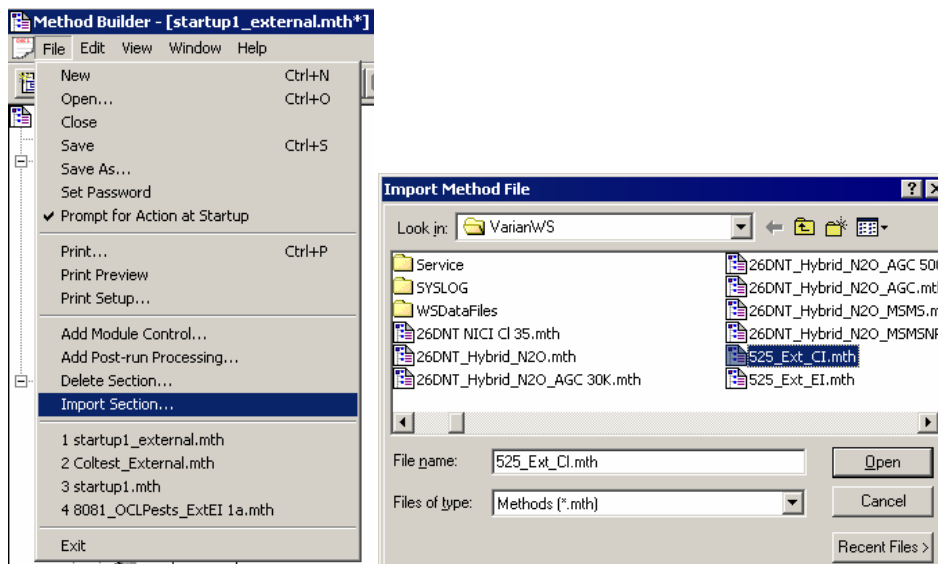
Selecting Method Notes displays a blank Parameters pane. Enter notes about the method as text. You can enter any information you like about the method, sample preparation, laboratory practices, etc. It is possible to copy text from a Microsoft Word or Excel document and paste into the Method Notes; however, graphics/images cannot be copied and pasted in this way. Method Notes are printed when the Method is printed.

Instrument Modules

Highlighting low-level outline branches for the 3800 GC, 4000 MS, or Combi PAL (those that cannot be expanded) displays Parameter panes with editable parameters for controlling the instrument, for data acquisition, and for post-run data processing and reporting.

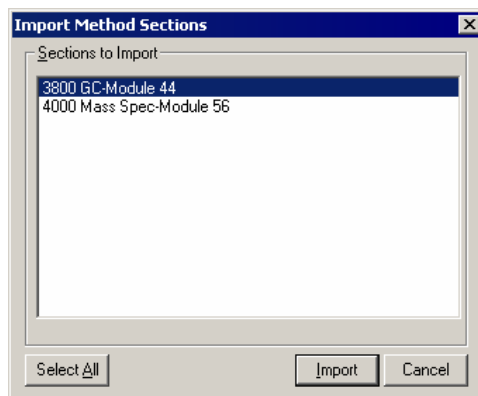
Importing Method Sections

You may wish to copy sections from one Method to another. Open the target Method file. This is the Method to which you want to Import one or more sections. Use **File Import Section** to open the Import Method File dialog.



Select the source Method from which you will import. Select the sections that you wish to import by clicking on them. If you click on one section and the hold down the shift key while clicking on another section, all of the sections in between will be selected. Holding down the control key while clicking on a section adds that selection to those files already selected. Clicking on a highlighted section while

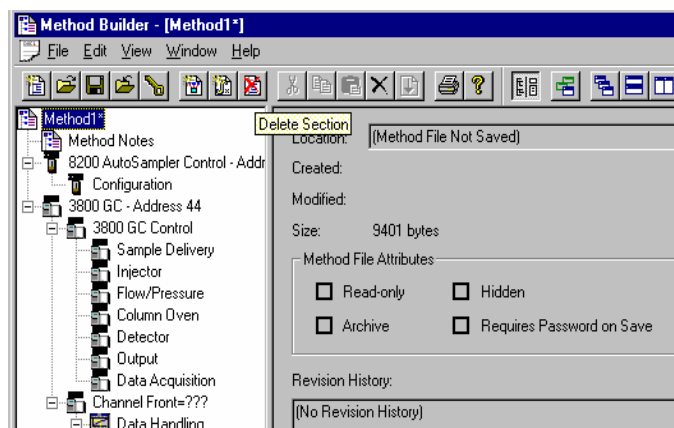
holding down the control key will remove that section from the list of selected files.



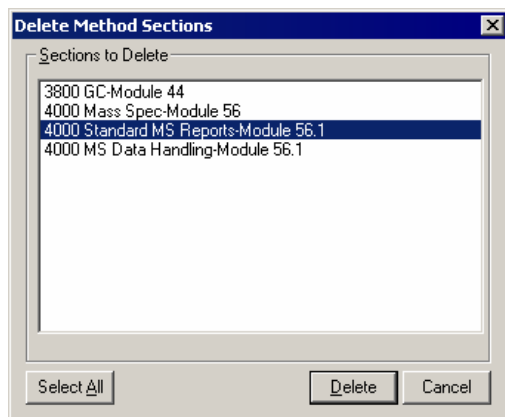
Click **Import** to import selected sections to the target Method. If the Method already has sections with the same module address and channel ID, you will be prompted to assign a new module address and channel number to the imported section or overwrite the existing section in the current method.

Deleting Method Sections

Open the Method containing the sections to remove. Click the **Delete Section** button on the Method Builder Toolbar or use the **File Delete Section**.



A dialog box of the sections in the Method opens. Highlight the sections to delete by clicking on them. Click one section and hold down the shift key while clicking on another section to select a range. Hold down the control key while clicking on a section to add that selection to the list. Click a selected section while holding down the control key to remove that it from the list.

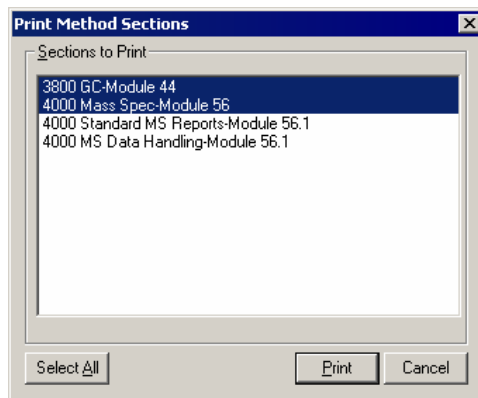


When the desired sections have been highlighted, click the **Delete** button to delete them from the Method. You will be prompted for confirmation before each section is deleted.

Printing the Method

Click the **Print** icon on the Toolbar. 

Select the desired Method sections and click **Print**.



You can print the active Method from System Control (File> Print) and the Workstation Toolbar.

Password Protecting a Method

Click **Set Password**  on the Method Builder Toolbar to open the Add Password dialog.

Add Password

Enter new password:

Re-enter new password:

OK Cancel

Enter the password and then re-enter it to confirm.

Once a Method is password protected, it can be used in System Control for instrument control, data acquisition and data handling. It can be viewed in Method Builder as well.

However, the selected password must be entered to save Method changes. It is now “locked” and can be edited only when the password is known.

4000 MS Method Section

Setting Up the MS Method Editor Screen

Under the Directory item 4000 Mass Spec Control, click **MS Method Editor**. The Parameters pane will now show the MS acquisition method.

Method Builder - [4000 MS Ext Coltest.mth]

File Edit View Window Help

4000 MS Ext Coltest.mth

- Method Notes
- Version Info
- 3800 GC - Address 44
 - 3800 GC Control
 - Autosampler
 - Sample Delivery
 - Injector
 - Flow/Pressure
 - Column Oven
 - Detector
 - Output
 - Data Acquisition
- 4000 Mass Spec - Address 56
 - 4000 Mass Spec Control
 - MS Acquisition Method**

Instrument Configuration: External EI

Acquisition Data Type: ☒ Centroid ☐ Profile

Chromatographic Time Segments

	Segment Description	Start (min.)	End (min.)	Scan Description
1	FIL/MUL DELAY	0.00	4.00	Ionization Off
2	Coltest	4.00	18.00	EI Auto - Full
3				
4				
5				
6				

Buttons: Add, Insert, Delete, Defaults, Restore

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal

General Parameters | Ionization Control | External EI Parameters | Full Scan Parameters

Mass Range: Low Mass: 50 m/z High Mass: 350 m/z

Tune Type: Auto

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	150	20	100
2	151	300	25	100
3	301	399	35	100
4	400	1000	35	100
5				
6				

Buttons: Customize, Insert, Add, Delete

Setting a Full-Screen Display

If you are going to do a thorough editing operation, to click **Hide Directory** on the toolbar so that you can see the MS Method Editor full-screen.



Instrument Configuration: External EI

Acquisition Data Type: Centroid (selected), Profile

Chromatographic Time Segments:

	Segment Description	Start (min.)	End (min.)	Scan Description
1	FIL/MUL DELAY	0.00	4.00	Ionization Off
2	Coltest	4.00	18.00	EI Auto - Full
3				
4				
5				
6				

Method Segment:

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints... Defaults

General Parameters | Ionization Control | External EI Parameters | Full Scan Parameters

Mass Range: Low Mass: 10 m/z, High Mass: 350 m/z

Tune: Type: Auto Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	150	8	100
2	151	300	25	100
3	301	399	35	100
4	400	1000	35	100
5				
6				

Customize Insert Add Delete

Setting the MS Instrument Configuration

Instrument Configuration:

- External EI
- Internal EI and CI
- External EI (highlighted)
- External CI
- Hybrid HPS
- Hybrid LPS

Configuration Menu

There are three hardware configurations: Internal, External, and Hybrid. The configuration defines what ionization modes can be used for data acquisition. The choices are Electron Ionization (EI) or Chemical Ionization (CI). The instrument Configuration is set in the upper-left corner of the MS Method Editor by selecting from the drop-down list box.

When you change from one ionization configuration to another, for example, from Internal ionization to External ionization, you must either activate an existing method or build a new method appropriate to the new ionization configuration.

The ionization configuration is selected in the MS Acquisition Method section of the method in the Instrument Configuration list box. If you change the ionization configuration in a method, all the previous Time Segment information will be replaced with a default section appropriate to the new ionization configuration.

Internal Configuration Options

In Internal configuration you can create a method to collect EI or CI data alone, or you can build a method with multiple time segments to collect either EI **or** CI data. CI in internal configuration can **only** be performed on positive ions. This is also known as PCI.

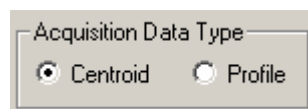
External Configuration Options

In External configuration, create an External EI method **or** an External CI method but **not** a method with both EI and CI segments. However, in CI mode, you can collect either positive CI (PCI) **or** negative CI (NCI) data on a segment-by-segment basis. When an External CI method is active, the CI ion volume in the external ion source is automatically inserted except when Auto Tune methods are being run.

Hybrid Configuration Options

Hybrid methods can be performed **only** in positive or negative ion CI modes (PCI or NCI) except when Auto Tune methods requiring EI mode are being run. For both these methods, CI reagent ions are formed in the external ion source and drawn into the ion trap to react with compounds eluting from the GC column. Note the Hybrid HPS and LPS options in the selection menu. Hybrid HPS (High Pressure Source) is performed with the CI volume inserted into the ion source whereas the LPS (Low Pressure Source) option occurs in the EI source configuration.

Choosing the MS Acquisition Data Type



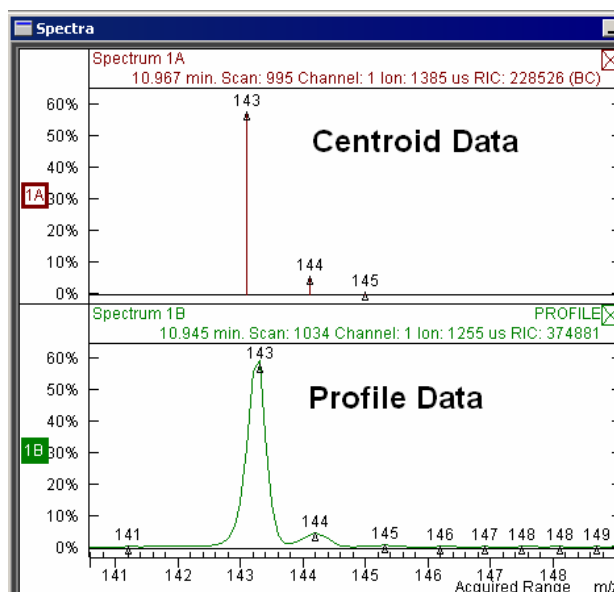
In a given method, only Profile or Centroid data may be collected.

Centroid Data

The standard data acquisition type is Centroid. The apex of each mass peak is reported with as ion and intensity data this is reported to one decimal place, for example, m/z 231.2. Centroid data are required for library searches and quantitation.

Profile Data

Profile data may also be acquired; the data files are much larger because ten data points are collected at each m/z. Profile data is useful for determining the charge state of multiply charged ions. It is also used to diagnose difficulties with mass resolution or mass assignment.



Editing Chromatographic Time Segments

The mass spectrometer can be programmed to acquire data in different ways by dividing the analysis into chromatographic time segments. In the simplest implementation, the default method always has a 3-minute filament/multiplier delay in Time Segment 1 before the instrument is turned on to collect data in Segment 2. This protects the system during the elution of the sample solvent front. However, for more complicated analyses, such as GC/MS/MS on multiple analytes, you can define up to 250 time segments for a single analysis. The maximum run time is 650 minutes.

Chromatographic Time Segments						
	Segment Description	Start (min.)	End (min.)	Scan Description		
1	FIL/MUL DELAY	0.00	3.00	Ionization Off	Add Insert Delete	
2		3.00	10.00	El Auto - Full		
3						
4						
5						
6						

Highlight rows by clicking on the row numbers on the left side of the segment table.

- **Add** - adds a new segment with the same MS parameters of the last segment in the table (except for the segment description).
- **Insert** - inserts a new row before the currently selected row with the same parameter values.
- **Delete** - delete the currently selected row.
- **Default button** - inserts system default parameters to the current segment.
- **Restore** - restores current segment parameters that were present when the method was first opened or last saved.

You can enter a segment description of up to 60 characters into the Segment Description field for each segment. Enter the desired time in minutes in the Start and End Times fields.

The Scan Description field is entered automatically based on the choice of Scan Type and Ionization mode in the Method Segment field below.

Chromatogram Time Gaps in Multi-Segment Methods

Depending upon the instrument configuration, data can be acquired in different Ion Modes (EI/CI - Auto/Fixed) and/or using different Ion Preparation techniques. When multiple segments have been acquired, MS Data Review displays each segment independently in the Chromatogram display. There will be no connection between the last point in one segment and the first point of the next segment. There is a small time gap between segments. This gap will be less than one second.

Programming Individual Time Segments

Method Segment | Checks and Adjustments | Module Attributes

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints... Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

Mass Range

Low Mass: 50 m/z High Mass: 1000 m/z

Tune

Type: Auto Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	99	35	100
2	100	249	35	100
3	250	399	35	100
4	400	1000	35	100
5				
6				

Customize Insert Add Delete

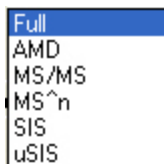
Features selected in the top of the Method Segment dialog determine which tab dialogs will be displayed in the remainder of the screen.

Ionization On Selection

If Ionization On is not checked, the filament and multiplier will be OFF during the segment. This is the default setting for Segment 1 when new methods are created to protect the filament and multiplier from damage by the solvent front during GC/MS runs. Ionization mode None can be used for other time segments as well.

NOTE: For all data acquisition segments the Ionization On box must be checked.

Scan Type/Group



The scan type is either full-scan or an ion preparation techniques. The default is *Full*.

Full: Full-scan data for the selected mass range

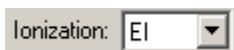
AMD: Automated Method Development of MS/MS conditions. Up to ten voltages for collision-assisted dissociation of the MS/MS precursor ion can be tested on a single chromatographic peak using this feature. Once the best dissociation condition has been identified, it can be selected for MS/MS, MRM, or MSⁿ.

MS/MS: MS/MS, or tandem mass spectrometry, is the isolation of a precursor ion formed during ionization, dissociation of the precursor ion by applying energy, trapping and scanning the resulting product ions. You can select multiple precursor ions.

MSⁿ: This is multistage MS/MS, in which a product ion from one tandem MS experiment becomes the precursor ion for the next stage. MS³, MS⁴, up to MSⁿ. The practical analytical range of MSⁿ has not yet been evaluated at introduction of the 4000 MS system.

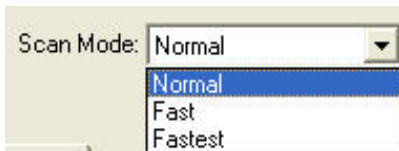
SIS: Selected Ion Storage allows selection of up to 6 ion ranges for monitoring.

Ionization



The available ionization modes depend on instrument configuration. Internal EI and CI offers either option; External and Hybrid offer but one ionization mode for a given configuration choice.

Scan Mode



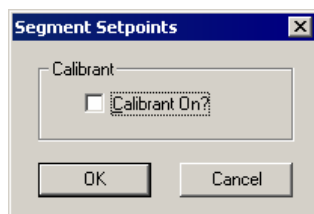
The default scan mode is normal.

Normal: This Scan Mode uses a prescan in Automatic Gain Control mode to determine optimum ionization time, and then ions are scanned at 5000 u/sec to collect the mass spectrum.

Fast: This Scan Mode also uses a prescan in Automatic Gain Control mode to determine optimum ionization time, but ions are scanned at 10000 u/sec to collect the mass spectrum.

Fastest: This Scan Mode uses *no prescan* and ions are scanned at 10000 u/sec to collect the mass spectrum. This mode is only available in **Full** scan type.

Setpoints



Check **Calibrant On?** to turn on the FC-43 calibration gas during an acquisition segment.

Overview of Ionization Methods

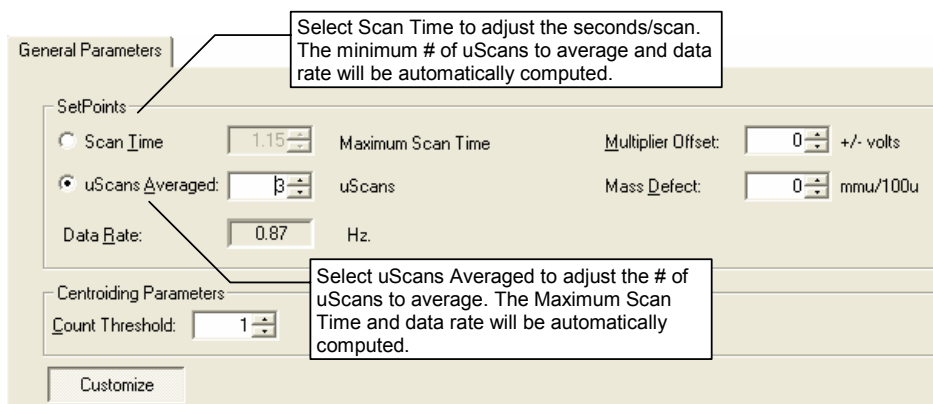
There are five sets of ionization parameters depending upon instrument configuration. In this section, the parameters for controlling each mode are discussed.

- “Internal EI Method Parameters” on page 110
- “Internal PCI Method Parameters” on page 115
- “External EI Method Parameters” on page 119
- “External PCI and NCI Method Parameters” on page 122
- “Hybrid PCI and NCI Method Parameters” on page 125

Internal EI Method Parameters

This section details how to set parameters for an electron ionization method in Internal configuration. “Internal Electron Ionization” on page 191 has more information about the theory of electron ionization and how it is implemented in this instrument.

General Parameters



The SetPoints dialog box contains three settings:

- Scan Time:** A radio button is selected, with a value of 1.15 in the adjacent field and the unit "Seconds/scan" to the right.
- uScans Averaged:** A radio button is selected, with a value of 3 in the adjacent field and the unit "Minimum # of uScans Averaged" to the right.
- Data Rate:** A value of 0.87 is shown in the field with the unit "Hz."

Scan Time, uScans Averaged, and Data Rate are linked. The number of microscans averaged is updated when the scan time is adjusted and vice versa.

There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections.

Setting up to average three scans is a good recommendation to get good spectral quality along with an acceptable data rate.

The Mass Defect allows for a systematic correction of the difference between the nominal mass of an ion and its exact mass. Its importance arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect.

The Multiplier Offset adjusts the EM voltage as much as $\pm 300V$ relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis.

The Count Threshold is normally 1 count; a value of 2-3 counts will reduce the number of low-level ions reported in the mass spectrum. This approach may improve library searches and reduce data file size at the cost of somewhat less detailed information in the mass spectra. The count threshold is shown only if the **Customize** button is active.

Ionization Control

The Ionization Control dialog box has three tabs: "General Parameters", "Ionization Control" (which is selected), and "Internal EI F". Under the "Automatic Gain Control" section, there is a "Target TIC:" label followed by a value of 20000 in a field and the unit "counts" to the right.

The Target Total Ion Current, or TIC, is specified here. The Automatic Gain Control (AGC) algorithm uses the ion count from a prescan at fixed ion time, along with this target value, to calculate an ion time necessary to fill the ion trap with the target number of ions during the analytical scan. The objective is to fill the trap with an optimal number of ions during each analytical scan. The Target

TIC is usually not set below 10,000 for full scan acquisitions, but it should also not be set too high or spectral distortions due to space charge (loss of MS resolution and/or shift in mass assignments for strong chromatographic peaks) may result. Typically, a Target TIC between 20,000 and 40,000 counts gives the best results.

General Parameters | Ionization Control | Internal EI Parameters

Automatic Gain Control

Target TIC: 20000 counts

Ionization Time

☐ Fixed Ionization Time

Ionization Time: 100 useconds

Max Ion Time: 25000 useconds

Customize

If one activates the Customize features, it becomes possible to run fixed ion time experiments and/or to change the Maximum Ion Time for Automatic Gain Control. Fixed ion time experiments are generally limited to diagnostic tests and are not advised for qualitative or quantitative analysis in the ion trap.

Changing the Maximum Ion Time can improve the sensitivity of techniques such as MS/MS or SIS. This parameter can be increased to 65,000 μ sec. If the ion time in the baseline of the chromatogram is not reduced below the maximum ion time, the ion trap is not being filled with ions to the level designated by the Target TIC. One can do three things in Internal EI mode to increase the ion signal: 1) raise the emission current, 2) raise the multiplier voltage, 3) increase the Maximum Ion Time. If the third approach is used and you are collecting data for quantitation, make sure that enough data points are collected across the chromatographic peaks for reliable quantitation. At least five, but preferably seven or more data points are needed for good quantitative data. Remember that using Fast Data Rate may allow the use of higher Maximum Ion Times without compromising quantitative data quality.

Internal EI Parameters

General Parameters | Ionization Control | Internal EI Parameters

Internal EI Parameters

Emission Current: 10 uamps

The emission current is the current of electrons produced by the filament and it controls the number of electrons that enter the trap. It is a good rule for full scan data acquisition to adjust the emission current so that the AGC ion time of the baseline is approximately the maximum ion time. By doing this, the system will have its maximum dynamic range. The number of ions stored in baseline scans will require the Maximum Ion Time and the AGC Ion Time will be cut back for any chromatographic peaks. In EI/MS/MS, where the MS/MS isolation step eliminates

the higher chemical background that would normally arise from a high emission current, emission currents of 50-100 μA can proportionately increase sensitivity over that of the default 10 μA current.

Full Scan Parameters

The lower and upper limits of the mass range to be acquired are entered in this tab dialog.

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	150	20	100
2	151	300	25	100
3	301	399	35	100
4	400	1000	35	100
5				
6				

With Customize active, it is possible to program up to six different segments of the acquisition mass range. You can use the **Insert**, **Add**, and **Delete** buttons to adjust the number of segments. Notice that the RF Storage Level can be programmed for each segment as well as the Ion Time Factor. When a scan is performed in Auto mode, the prescan is followed by an ionization time and scan of each segment needed to cover the Mass Range designated to the left of the segment table. The actual Ion Time for each segment is the AGC Ion Time multiplied by the percentage in the Ion Time Factor field.

Setting Up the Method for DFTPP or BFB Tuning

Use the menu in the Tune Type field to designate BFB Tune or DFTPP Tune. This displays a segment table in which mass ranges and Ion Time Factors have been suggested to meet the US EPA tune requirements for determination of environmental contaminants. BFB tune is used for volatiles analysis and DFTPP tune for semi-volatile analyses. Following is the set of factors for DFTPP Tune.

Method Segment
☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints...
 Defaults

General Parameters Ionization Control Internal EI Parameters Full Scan Parameters

Mass Range
 Low Mass: 50 m/z
 High Mass: 1000 m/z

Tune
 Type: DFTPP Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	75	35	80
2	76	130	35	140
3	131	399	35	70
4	400	1000	35	25
5				
6				

Customize Insert Add Delete

DFTPP Tune Parameters

To meet DFTPP tune requirements, use these parameters as a starting point and check the relative ratios of ions in the spectrum of DFTPP compared to the requirements of the particular semi-volatile method. If needed, adjust the Ion Time Factor for particular segments to increase or decrease the abundance of key ions. You can use Varian's EnviroPro software to generate DFTPP Tune and other reports needed for environmental analyses.

Similarly, one can designate BFB Tune to display a table of parameters that is a good starting point toward meeting US EPA tuning requirements for volatiles analyses.

General Parameters Ionization Control Internal EI Parameters Full Scan Parameters

Mass Range
 Low Mass: 35 m/z
 High Mass: 300 m/z

Tune
 Type: BFB Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	70	27	120
2	71	78	35	70
3	79	150	35	100
4	151	1000	35	70
5				
6				

Customize Insert Add Delete

DFTPP Tune Parameters

NOTE: The recommended starting parameters for DFTPP and BFB are somewhat different for Internal and External EI modes.

Internal PCI Method Parameters

This section details how parameters may be set for a positive chemical ionization method in Internal configuration. For more details on the theory of positive chemical ionization and how it is implemented in this instrument, see the 4000 MS Ionization Techniques section, "Internal Positive Chemical Ionization" on page 194.

General Parameters

General Parameters

Adjust the seconds/scan

SetPoints

☒ Scan Time: 1.15 Maximum Scan Time Multiplier Offset: 0 +/- volts

☐ uScans Averaged: 3 uScans Mass Defect: 0 mmu/100u

Data Rate: 0.87 Hz

Centroiding Parameters

Count Threshold: 1

Adjust the # of uScans to average

Customize

SetPoints

☒ Scan Time: 1.15 Seconds/scan

☐ uScans Averaged: 3 Minimum # of uScans Averaged

Data Rate: 0.87 Hz

Scan Time, uScans Averaged, and Data Rate are all linked. The number of microscans averaged is updated when the scan time is adjusted and *vice versa*.

There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections.

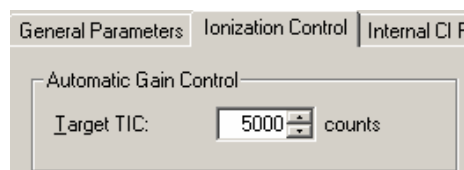
Set up to average three scans to get good spectral quality along with an acceptable data rate.

The Mass Defect allows for a systematic correction of the difference between the nominal mass of an ion and its exact mass. Its importance arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect.

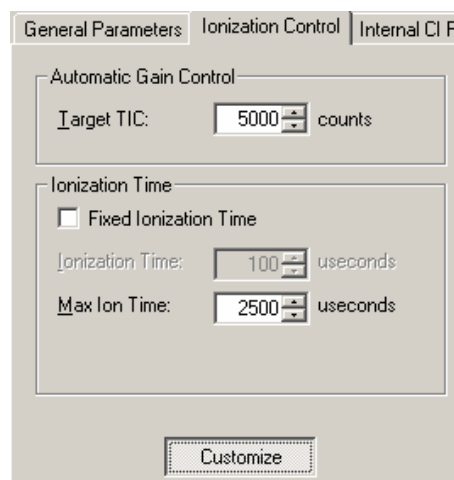
The Multiplier Offset adjusts the EM voltage as much as $\pm 300V$ relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis.

The Count Threshold is normally 1 count; a value of 2-3 counts will reduce the number of low-level ions reported in the mass spectrum. This approach may improve library searches and reduce data file size at the cost of somewhat less detailed information in the mass spectra. The count threshold is shown only if the **Customize** button is active.

Ionization Control



The default Target TIC for positive chemical ionization is 5000. The target can be set as high as 65,000.



When you click **Customize**, you can run fixed ion time experiments with ion times as high as 65,000 μsec or to change the Maximum Ion Time for CI Auto experiments. You can turn on the CI Gas and the ion trap in Manual Control and check the ion time in CI Auto mode. If this time is less than 1,000 μsec , lowering the Max Ion Time may improve performance. The closer the actual ionization time is to the Max Ion time, the closer the actual reaction time will be to the Max Reaction Time.

Internal CI Parameters

Internal CI Parameters

Emission Current: 10 uAmps

Reagent Parameters

Reagent: Methanol Save...

Reagent Low Mass: 25 m/z

Reagent High Mass: 50 m/z

Customize

Set the emission current set between 10 and 100 μ Amps. Settings above 10 μ Amps do not improve performance unless the ion time in the baseline of the chromatogram is at the Max Ion Time; this value can be viewed by clicking the **Customize** button in the Ionization Control tab dialog. Setting the emission current so that the ion time in the baseline is just below the Max Ion Time will give the widest dynamic range for the CI method.

Internal CI Parameters

Emission Current: 10 uAmps

Reagent Parameters

Reagent: Methanol

Reagent List:

- Acetonitrile
- Isobutane
- Methane
- Methanol

Select Parameters for any of four internal PCI reagents from list. If you design parameters for a new reagent or change the parameters for one of these reagents, you may save these changes under a new name that will then be added to your list of selections.

Internal CI Reagent Parameters

Create, update, or delete user defined parameter sets:

Name: MyMethanol

Create Delete Cancel

The default values for Reagent Low Mass and Reagent High Mass vary depending on the selected CI reagent. When the **Customize** button is clicked, note that the Reaction Storage Level and the Ejection Amplitude are also reagent-dependent. These features are all discussed in more detail in "Internal Positive Chemical Ionization" on page 194 in the section 4000 MS Ionization Techniques. Briefly, Reagent Low Mass must be low enough so that the reagent ions can be created and stored. For example, formation of the methane reagent

ions at m/z 17, 29, and 41 requires that the CH_3^+ and CH_4^+ precursor ions be stored. Therefore, the default Reagent Low Mass for methane is 15u.

Methanol has its reagent ions at m/z 33 and 47, so a higher default Reagent Low Mass value of 25u is appropriate.

The Reagent High Mass value may be lowered to exclude one of the CI reagent ions. For example, you could set the value to 40u to exclude the 41u reagent ion of methane. However, since there are still neutral methane molecules in the ion trap, the m/z 41 ion can be recreated by reactions of stored 29u ions with methane.

Internal CI Parameters

Emission Current: 10 uAmps

Reagent Parameters

Reagent: Methanol Save...

Reagent Low Mass: 25 m/z

Reagent High Mass: 50 m/z

Reaction Storage Level: 25 m/z

Ejection Amplitude: 15 Volts

Max Reaction Time: 100 mSecs

Customize

With the **Customize** button active, you can change the Reaction Storage Level or the Ejection Amplitude. The Ejection Amplitude is applied at the end of the ionization time to eject unwanted high mass EI artifact ions. The default value for each reagent usually does not need adjustment. The Reaction Storage Level affects the storage efficiency for high mass ions. When this value is set below 25u, storage of ions above m/z 200 will be poor. This is one reason that ammonia PCI is not a default option for the 4000 MS in Internal PCI; its reagent ion is at m/z 18.

The final adjustable parameter in the Customize screen area is the Maximum CI Reaction Time. The operating range is from 1-2000 μsec . Depending on the CI reagent and the target compound of the analysis, sensitivity may be affected significantly by varying this parameter.

Full Scan Parameters

Full Scan Parameters

Mass Range

	Low Mass (m/z)	High Mass (m/z)
1	50	1000
2		
3		
4		
5		
6		

Insert Add Delete

It is typical to use only a single mass range segment in PCI, in which case one simply enters the desired values for Low Mass and High Mass of the acquisition range. However, as shown below, one can enter up to six non-contiguous mass ranges (separated by at least 10u). This feature can also be time programmed on a chromatographic segment basis. It is then possible to tailor the CI acquisition ranges for CI to different target analytes depending on the mass spectrum of each compound. An example a three-range acquisition is shown here:

	Low Mass (m/z)	High Mass (m/z)
1	150	200
2	300	350
3	400	425
4		
5		
6		

Insert Add Delete

External EI Method Parameters

This section details how parameters may be set for an electron ionization method in External configuration. For more details on the theory of electron ionization and how it is implemented in this instrument, go to “External Electron Ionization” on page 199 in the section 4000 MS Ionization Techniques.

General Parameters

General Parameters

SetPoints

☐ Scan Time: 1.15 Maximum Scan Time Multiplier Offset: 0 +/- volts

☒ uScans Averaged: 3 uScans Mass Defect: 0 mmu/100u

Data Rate: 0.87 Hz

Centroiding Parameters

Count Threshold: 1

Customize

SetPoints

☒ Scan Time: 1.15 Seconds/scan

☐ uScans Averaged: 3 Minimum # of uScans Averaged

Data Rate: 0.87 Hz

Scan Time, scans Averaged, and Data Rate are all linked. The number of microscans averaged is updated when the scan time is adjusted and *vice versa*.

There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections.

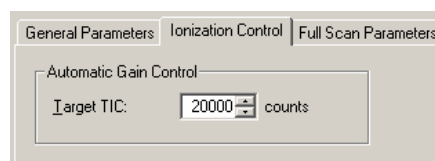
Setting up to average three scans is a good recommendation to get good spectral quality along with an acceptable data rate.

The Mass Defect allows for a systematic correction of the difference between the nominal mass of an ion and its exact mass. Its importance arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect.

The Multiplier Offset adjusts the EM voltage as much as $\pm 300V$ relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis.

The Count Threshold is normally 1 count; a value of 2-3 counts will reduce the number of low-level ions reported in the mass spectrum. This approach may improve library searches and reduce data file size at the cost of somewhat less detailed information in the mass spectra. The count threshold is shown only if the **Customize** button is active.

Ionization Control



The Target Total Ion Current, or TIC, is specified here. The Automatic Gain Control (AGC) algorithm uses the ion count from a prescan at fixed ion time, along with this target value, to calculate an ion time necessary to fill the ion trap with the target number of ions during the analytical scan. The objective is to fill the trap with an optimal number of ions during each analytical scan. The Target TIC is usually not set below 10,000 for full scan acquisitions, but it should also not be set too high or spectral distortions due to space charge (loss of MS resolution and/or shift in mass assignments for strong chromatographic peaks) may result. Typically, a Target TIC between 20,000 and 40,000 counts gives the best results.

General Parameters | Ionization Control | Full Scan Parameters

Automatic Gain Control

Target TIC: 20000 counts

Ionization Time

☐ Fixed Ionization Time

Ionization Time: 100 useconds

Max Ion Time: 25000 useconds

Customize

If one activates the Customize features, it becomes possible to run fixed ion time experiments and/or to change the Maximum Ion Time for Automatic Gain Control. Fixed ion time experiments are generally limited to diagnostic tests and are not advised for qualitative or quantitative analysis in the ion trap.

Changing the Maximum Ion Time can improve the sensitivity of techniques such as MS/MS or SIS. This parameter can be increased to 65,000 μsec . If the ion time in the baseline of the chromatogram is not reduced below the maximum ion time, the ion trap is not being filled with ions to the level designated by the Target TIC.

To increase the ion signal in External EI mode, either raise the multiplier voltage or increase the Maximum Ion Time. If using Maximum Ion Time, at least five, but preferably seven or more data points are needed for reliable quantitative data. Using Fast Data Rate may allow the use of higher Maximum Ion Times without compromising quantitative data quality.

Full Scan Parameters

General Parameters | Ionization Control | Full Scan Parameters

Mass Range

Low Mass: 40 m/z

High Mass: 300 m/z

Customize

The lower and upper limits of the mass range to be acquired are entered in this tab dialog.

Method | Active Segment | Checks and Adjustments | Module Attributes

Method Segment
☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints...
 Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

Mass Range
 Low Mass: 50 m/z
 High Mass: 1000 m/z

Tune
 Type: Auto Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	99	35	100
2	100	249	35	100
3	250	399	35	100
4	400	1000	35	100
5				
6				

Customize Insert Add Delete

When **Customize** is enabled, it is possible to program up to six different segments of the acquisition mass range. You can use the **Insert**, **Add**, and **Delete** buttons to adjust the number of segments. Notice that the RF Storage Level can be programmed for each segment as well as the Ion Time Factor. If excessive fragmentation is observed in the mass spectrum, it may be useful to lower the RF storage level below 35u for a given segment of the acquisition range. Note that lowering the RF storage level below 35u will decrease the storage efficiency for high-mass ions, particularly above m/z 250.

When a scan is performed in Auto mode, the prescan is followed by an ionization time and scan of each segment needed to cover the Mass Range designated to the left of the segment table. The actual Ion Time for each segment is the AGC Ion Time multiplied by the percentage in the Ion Time Factor field.

External PCI and NCI Method Parameters

This section details how parameters may be set for positive or negative chemical ionization methods in External configuration. For more details on the theory of chemical ionization and how it is implemented in this instrument, go to "External Positive or Negative Chemical Ionization" on page 202 in the section 4000 MS Ionization Techniques.

General Parameters

General Parameters | Adjust the seconds/scan to average

SetPoints
☒ Scan Time: 1.15 Maximum Scan Time Multiplier Offset: 0 +/- volts
☒ uScans Averaged: 3 uScans Mass Defect: 0 mmu/100u
 Data Rate: 0.87 Hz Adjust the # of uScans to average

Centroiding Parameters
 Count Threshold: 1

Customize

The SetPoints dialog box contains three settings:

- Scan Time:** A radio button is selected, with a value of 1.15 in the adjacent field and the unit "Seconds/scan" to the right.
- uScans Averaged:** A radio button is selected, with a value of 3 in the adjacent field and the unit "Minimum # of uScans Averaged" to the right.
- Data Rate:** A value of 0.87 is in the field with the unit "Hz." to the right.

Scan Time, uScans Averaged, and Data Rate are all linked. The number of microscans averaged is updated when the scan time is adjusted and *vice versa*.

There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections.

Setting up to average three scans is a good recommendation to get good spectral quality along with an acceptable data rate.

The Mass Defect allows for a systematic correction of the difference between the nominal mass of an ion and its exact mass. Its importance arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect.

The Multiplier Offset adjusts the EM voltage as much as $\pm 300V$ relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis.

The Count Threshold is normally 1 count; a value of 2-3 counts will reduce the number of low-level ions reported in the mass spectrum. This approach may improve library searches and reduce data file size at the cost of somewhat less detailed information in the mass spectra. The count threshold is shown only if the Customize is active.

Ionization Control

The Ionization Control dialog box has three tabs: "General Parameters", "Ionization Control" (which is selected), and "External CI". Under the "Automatic Gain Control" section, the "Target TIC:" is set to 5000 counts.

The default Target TIC for positive or negative chemical ionization is 5,000. The target can be set as high as 65,000.

The screenshot shows the 'Ionization Control' tab. It contains two main sections: 'Automatic Gain Control' and 'Ionization Time'. In the 'Automatic Gain Control' section, the 'Target TIC' is set to 5000 counts. In the 'Ionization Time' section, there is a checkbox for 'Fixed Ionization Time' which is currently unchecked. Below this, the 'Ionization Time' is set to 100 useconds, and the 'Max Ion Time' is set to 25000 useconds.

When the **Customize** button is clicked, one has the option to run fixed ion time experiments with ion times as high as 2,500 μsec or to change the Maximum Ion Time for CI Auto experiments. One can turn on the CI Gas and the ion trap in Manual Control and check the ion time in CI Auto mode.

External CI Parameters

The screenshot shows the 'External CI Parameters' tab. It contains two main sections: 'External CI Ionization Parameters' and 'Reagent Parameters'. In the 'External CI Ionization Parameters' section, the 'Polarity' is set to 'Positive'. In the 'Reagent Parameters' section, the 'Reagent Gas' is set to 'Methane' and the 'CI Background' is set to 45 m/z. There is a 'Save...' button next to the 'Reagent Gas' dropdown.

Although there is a menu to allow you to choose from several CI reagents, initial evaluation indicates that methane is the reagent of choice for chemical ionization when the instrument is in External configuration.

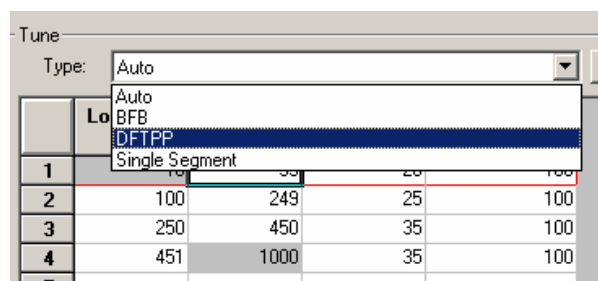
The screenshot shows a dropdown menu for 'Reagent Parameters'. The 'Reagent Gas' is set to 'Methane'. The 'CI Background' dropdown menu is open, showing a list of reagents: Acetonitrile, Ammonia, Isobutane, Methane (which is highlighted), and Methanol.

Full Scan Parameters

The screenshot shows the 'Full Scan Parameters' tab. It contains a 'Mass Range' section with two input fields: 'Low Mass' set to 50 m/z and 'High Mass' set to 1000 m/z.

Only the start and end mass for the acquisition range are entered in this tab dialog. Do not use a start mass below 50 for External PCI with methane. This causes reagent ions to be counted in the prescan and leads to ion times that are too short.

Setting up the Method for DFTPP and BFB Tuning



From the list, select BFB Tune or DFTPP Tune. Doing this displays a segment table in which mass ranges and Ion Time Factors have been suggested to meet US EPA tune requirements for determination of environmental contaminants. BFB tune is used for volatiles analysis and DFTPP tune for semi-volatiles analysis. Here is the set of factors for BFB tune in External EI mode:

Full Scan Parameters

Tune Type: BFB

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	70	27	200
2	71	78	35	50
3	79	170	35	100
4	171	270	35	100
5	271	1000	35	100
6				

To meet tune requirements, use the provided parameters as a starting point and check the relative ratios of ions in the spectrum of BFB or DFTPP, as relevant. If needed, adjust the Ion Time Factor for particular segments to increase or decrease the abundance of key ions. You can use Varian's EnviroPro software to generate BFB or DFTPP Tune reports and other reports needed for environmental analysis.

Hybrid PCI and NCI Method Parameters

This section details how parameters may be set for positive or negative chemical ionization methods in Hybrid configuration. For more details on the theory of chemical ionization and how it is implemented in this instrument, go to "Hybrid Positive or Negative Chemical Ionization" on page 206 in the section 4000 MS Ionization Techniques.

General Parameters

General Parameters

SetPoints

☐ Scan Time 1.15 Maximum Scan Time Multiplier Offset: 0 +/- volts

☒ uScans Averaged: 3 uScans Mass Defect: 0 mmu/100u

Data Rate: 0.87 Hz

Centroiding Parameters

Count Threshold: 1

Customize

SetPoints

☒ Scan Time 1.15 Seconds/scan

☐ uScans Averaged: 3 Minimum # of uScans Averaged

Data Rate: 0.87 Hz

Scan Time, uScans Averaged, and Data Rate are all linked. The number of microscans averaged is updated when the scan time is adjusted and *vice versa*.

There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections.

Setting up to average three scans is a good recommendation to get good spectral quality along with an acceptable data rate.

The Mass Defect allows for a systematic correction of the difference between the nominal mass of an ion and its exact mass. Its importance arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect.

The Multiplier Offset adjusts the EM voltage as much as $\pm 300V$ relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis.

The Count Threshold is normally 1 count; a value of 2-3 counts will reduce the number of low-level ions reported in the mass spectrum. This approach may

improve library searches and reduce data file size at the cost of somewhat less detailed information in the mass spectra. The count threshold is shown only if the **Customize** button is active.

Ionization Control

The screenshot shows the 'General Parameters' tab of the 'Ionization Control' dialog. It features a section titled 'Automatic Gain Control' with a 'Target TIC' input field set to '5000' and the unit 'counts'.

The default Target TIC for positive or negative chemical ionization is 5,000. The target can be set as high as 65,000.

The screenshot shows the 'Ionization Time' tab of the 'Ionization Control' dialog. It includes a 'Fixed Ionization Time' checkbox (unchecked), an 'Ionization Time' input field set to '100' with the unit 'useconds', and a 'Max Ion Time' input field set to '2500' with the unit 'useconds'. A 'Customize' button is located at the bottom.

Click Customize, to run fixed ion time experiments with ion times as high as 65,000 μ sec, or to change the Maximum Ion Time for CI Auto experiments. One can turn on the CI Gas and the ion trap in Manual Control and check the ion time in CI Auto mode.

Hybrid Parameters

The screenshot shows the 'Hybrid Parameters' dialog box. It has tabs for 'General Parameters', 'Ionization Control', 'Hybrid Parameters', and 'Full Scan Parameters'. The 'Hybrid Ionization Parameters' section includes:

- 'Reagent Gas' dropdown set to 'Acetonitrile' with a 'Save...' button.
- 'Reagent Low Mass' input set to '35' m/z.
- 'Reagent High Mass' input set to '60' m/z.
- 'Ejection Amplitude' input set to '15' Volts.
- 'Max Reaction Time' input set to '100' mSecs.
- 'Reaction Storage Level' input set to '33' m/z.
- 'CI Background Mass' input set to '60' m/z.
- 'Polarity' section with 'Positive' selected.
- 'Reagent Ions' section with 'View' checked, 'Start Mass' set to '35' m/z, and 'End Mass' set to '60' m/z.

The Reagent Low Mass and Reagent High Mass values are set to bracket the CI reagent ion mass range of interest. The Reagent Low Mass must be set to at

least 10u below the mass of the lowest reagent ion of interest without a loss of intensity for that reagent ion. It is helpful to make adjustments of these parameters in Manual Control with the View box checked in the field to the right of this dialog. The Reagent Low Mass parameter sets the RF storage level to exclude ions below the selected m/z. This is not a precise way to perform isolation. By contrast, the Reagent High Mass isolation step occurs after the ionization time, when resonant waveforms are applied to the ion trap end caps to eliminate ions with m/z above the selected Reagent High Mass.

Ejection Amplitude is the voltage of the waveforms for high mass isolation of CI reagent ions. The default value is 15V.

Max Reaction Time is the maximum time in μsec allowed for CI reaction. If the ion time is reduced below the maximum based on the results of the prescan, the ion time will be scaled back proportionately. The allowed range for this parameter is 1-2000 μsec .

The Reaction Storage Level is the RF storage level in the ion trap during CI reaction, following the ionization period. It should not be set above the m/z of the CI reagent ion with which you wish to perform the CI reaction, else these ions will be ejected from the ion trap.

The CI Background Mass is the lowest m/z counted during the CI prescan. It can be higher than the low mass of the acquisition range but is usually set to be at or below the Start Mass value.

The Polarity is selected here to perform either positive or negative hybrid CI.

Set the Start and End Mass ions to View in this dialog. Click the **View** box when the method is opened in Manual Control to observe the effects of Reagent Start Mass and Reagent End Mass isolation adjustments. Remember you must manually turn on the ion trap and CI gas icons to observe the CI reagent ions in this way.

Full Scan Parameters

The screenshot shows a software dialog box titled 'Full Scan Parameters'. It has four tabs: 'General Parameters', 'Ionization Control', 'Hybrid Parameters', and 'Full Scan Parameters' (which is selected). Inside the dialog, there is a section labeled 'Mass Range' containing a table with two columns: 'Low Mass (m/z)' and 'High Mass (m/z)'. The table has six rows, numbered 1 to 6. Row 1 contains the values 50 and 1000. Rows 2 through 6 are empty. Below the table are three buttons: 'Insert', 'Add', and 'Delete'.

	Low Mass (m/z)	High Mass (m/z)
1	50	1000
2		
3		
4		
5		
6		

It is typical to use only a single mass range segment in CI, in which case one simply enters the desired values for Low Mass and High Mass of the acquisition range. However, as shown below, one can enter up to six non-contiguous mass ranges (separated by at least 10u). This feature can also be time programmed on a chromatographic segment basis. It is then possible to tailor the CI acquisition ranges for CI to different target analytes depending on the mass spectrum of each compound. An example of a three-range acquisition is shown here:

Mass Range

	Low Mass (m/z)	High Mass (m/z)
1	150	200
2	300	350
3	400	425
4		
5		
6		

Insert Add Delete

Overview of Ion Preparation Methods

Ion Preparation Methods

The two fundamental processes in mass spectrometry are ion creation and ion analysis. Because the ion trap stores ions before the ion analysis step, it is possible to insert ion preparation steps to manipulate the ion population before the mass spectrum is collected. This manipulation can consist of simply ejecting certain ions or ranges of ions from the ion trap (Selected Ion Storage, or SIS). More involved ion preparation is required for tandem mass spectrometry (MS/MS). In this process, Precursor Ions of a selected m/z are first isolated by application of waveforms to the ion trap end cap electrodes. Then the isolated ions are dissociated by using additional waveforms to induce energetic collisions with helium in the ion trap. For some applications, ion preparation methods improve both selectivity and sensitivity significantly. The 4000 MS ion trap performs SIS and MS/MS by means of *ion preparation methods* that modify the scan function. Advantages associated with ion preparation methods are similar to those of other sample preparation methods, e.g., reduction of noise, increased selectivity, and improved sensitivity.

MS/MS Ion Prep Method

General Parameters Ionization Control Internal EI Parameters MS/MS Parameters

MS/MS

	Precursor Ion (m/z)	Isolation Window (m/z)	Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Product Ion Start Mass (m/z)	Product Ion End Mass (m/z)	More Ranges
1	131.0	3.0	Resonant	57.7	0.20	58	141	

Insert Add Delete "q" Calculator Customize

The MS/MS parameters are set out in tabular form. Each of the three steps of MS/MS-isolation, dissociation, and scanning, follow across the table.

Isolation of the Precursor Ion

Precursor Ion (m/z)	Isolation Window (m/z)
131.0	3.5

Precursor Ion Mass (m/z) has a range is 50 to 1000 m/z.

NOTE: Integer mass values may be used if the mass isolation window (next step) is greater than 1.5 m/z. If the window size is less than 1.5 m/z, or if the mass defect of the precursor ion is greater than approximately 0.3 m/z, use the exact fractional mass.

The Isolation Window (m/z)

The full mass isolation window range is 1.0 to 14.0 m/z; the actual range is dependent on the precursor ion. The default value is 3.0 m/z. Integral and fractional mass isolation windows are both accepted. If an isolation window smaller than 1.5 m/z is used, then the exact mass of the precursor ion should be entered in Precursor Ion Mass.

Maximum Isolation Window as a Function of Precursor Mass

Precursor Ion (m/z)	<80	<100	<120	<180	<280	<420	>420
Maximum Isolation Window (m/z)	3.0	4.0	5.0	7.0	10.0	12.0	14.0

NOTE: When beginning method development, start with a mass isolation window of 3 m/z to ensure that the ion is centered in the window. For example for a precursor ion mass of 502 m/z and a mass isolation window of 3 m/z, ions of masses 501, 502, and 503 m/z would be isolated. If there is a need to reduce the window because of interfering ions, this should be done in a later step because the customized parameters may require optimization.

NOTE: Some ions are more easily dissociated during the isolation step. Therefore, increasing the window may reduce the ion loss by dissociation at the expense of selectivity.

Dissociation of the Precursor Ion

Select the Waveform Type by selecting **Non-Resonant** or **Resonant** excitation in the drop-down list box.

Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)
Resonant	57.7	0.20
Non-Resonant		
Resonant		

Excitation Storage Level (Resonant or Non-Resonant excitation)

Definition: the RF storage level in m/z when the dissociation waveform is applied following isolation.

The excitation storage level range depends on the precursor mass, but the lowest product ion must be more than several mass units above the excitation storage level. A starting excitation storage level for a precursor ion can be calculated using the “q” calculator. The “q” calculator is accessed by right clicking on any of the fields in the MSMS parameters table.

Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Product Start M_z (m/z)
57.7	100	100
"q" Calculator		
What's This?		

The optimum excitation storage level is a tradeoff between a storage level high enough to allow fragmentation of the precursor ion and a storage level low enough to allow efficient trapping of the lowest m/z product ion.

A higher excitation storage level allows more energy to be imparted to the precursor ions by allowing a higher excitation amplitude.

The storage efficiency for product ions will be reduced if the storage level is greater than about 70% of the m/z of the ion.

For some analyses, a compromise may be needed to observe any product ions. An excitation storage level may need to be chosen which will result in reduced trapping efficiency of the product ions.

As a rule of thumb, the highest recommended excitation storage level will be the product ion m/z divided by 1.4. For example, if the product ion m/z is 140, then the excitation storage level should be no higher than m/z 100 for optimum trapping efficiency of the product ion.

The MS “q” Calculator

For convenience, The MS “q” Calculator is available in all Ion Preparation Options.

"q" Calculator

Specify a desired "q" value and click Calculate to determine the Excitation Storage Level corresponding to the "q" value. Click Update and Close to copy the calculated Excitation Storage Level value back into the table. Click Cancel to discard any calculation and return to the table.

Precursor Ion Mass: 450.0 m/z

Desired "q" Value: 0.40000

Excitation Storage Level: 198.2 m/z

Calculate

Update and Close

Cancel

The “q” value can be changed and a new excitation storage level calculated for a given precursor ion.

NOTE: Set the excitation storage level to avoid ejection of the lowest mass product ion. If, for example, a precursor ion of m/z 403 dissociated to product ions with m/z values of 350, 200, and 131, you would not want to use a storage level value above 94 m/z ($131/1.4=94$).

Storage RF values are often reported in terms of the Mathieu “q” parameter in the scientific literature. The value of the “q” parameter determines the stability of the precursor ion trajectory. More stable trajectories allow higher excitation voltages to be applied before ions are ejected from the trap. It has been empirically observed that a “q” value of 0.4 provides an optimum yield of product ions. This calculator utility determines the corresponding CID storage RF value (m/z) following user entry of the desired “q” value.

Excitation Amplitude in volts

The amplitude range for non-resonant excitation is 0 to 120 volts. For resonant excitation, the range is 0 to 60 volts. The default values are 20 volts for the non-resonant excitation method and 0.2 volts for the resonant excitation method

NOTE: If the excitation amplitude used is too large, the precursor ion and product ion spectra will be absent because both ions will be ejected from the trap. If the value is too small, the precursor ion spectrum will be dominant and the product ion spectrum will be weak or missing.

Scanning the Product Ions

Product Ion Start Mass (m/z)	Product Ion End Mass (m/z)	More Ranges
198	460	

Set the Product Ion Start and End Masses to cover the mass range required.

Customize a Non-Resonant Method

The parameters in the MS/MS Ion Prep Method Editor window are the basic ones needed to create a MS/MS Ion Prep Method file. For the vast majority of applications, no other information is required to use the MS/MS technique successfully. However, some applications may require further customization.

Customize Parameters is separated into three sections: Ionization, Isolation and Dissociation. These parameters are hidden by default but can be modified by clicking the **Customize** button.

Precursor Ion (m/z)	Ionization Storage Level (m/z)	Isolation Window (m/z)	Low Offset (m/z)	High Offset (m/z)	High Mass Ejection (volts)
450.0	35	3.5	0.0	0.0	35.0

Customize Ionization and Isolation Parameters

Ionization

Precursor Ion (m/z): The precursor ion is the desired ion m/z that will be isolated in the MS/MS isolation step. This Precursor Ion m/z value is used in both Resonant and Non-Resonant Methods of MS/MS. For more info, please review the previous table titled **Maximum Isolation Window as a Function of Precursor Mass**.

Ionization Storage Level: The m/z value at which the RF is maintained during ionization and the coarse isolation step. The range is 30 to 100 m/z, with a default value of 35 m/z.

Isolation

Low Edge Offset: The mass offset to optimize the ejection of the mass just below the precursor ion mass. The **Low Edge Offset** range is -0.5 m/z to 0.5 m/z. The default value is 0.

Low Edge Offset affects the isolation window on the low mass side of the precursor ion. Increasing the mass offset (increasing the default from 0 to 0.1 m/z) makes the isolation window on the low mass side of the precursor ion larger. Decreasing the offset (decreasing from the default 0 to -0.5 m/z) decreases the window on the low mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments.

High Edge Offset: The mass offset to optimize the ejection of the mass just above the precursor ion mass. The **High Edge Offset** range is -0.5 m/z to 0.5 m/z. The default value is 0.

High Edge Offset affects the isolation window on the high mass side of the precursor ion. Increasing the mass offset (increasing the default from 0 to 0.1 m/z) makes the isolation window on the high mass side of the precursor ion larger. Decreasing the offset (decreasing from the default 0 to -0.1 m/z) decreases the window on the high mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments.

If the **Low** or **High Edge Offset** range is not sufficient to completely isolate the desired ions, either increase (in the case of desired ions not being present) or decrease (in the case of unwanted ions being isolated) the isolation window.

High Edge Amplitude: Amplitude of broadband waveform used to eject masses above the isolated precursor ion. Default value is 35 volts. If loss of precursor ions due to dissociation is observed, reducing this amplitude may help. However, some ions with a higher m/z than the precursor ion may not be ejected.

Dissociation

Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Excitation Time (msec)
Non-Resonant ▾	198.2	20.00	20

Customize Dissociation Parameters (Non-Resonant)

Waveform Type: The waveform type is either resonant or non-resonant. Resonant waveforms are harmonious with the frequencies of electrons held in

the ion trap. Non-resonant waveforms are not harmonious with the frequencies of the electrons held in the ion trap.

Excitation Storage Level (m/z): The RF storage level in m/z when the dissociation waveform is applied following isolation. The excitation storage level range depends on the precursor mass, but the lowest product ion must be more than several mass units above the excitation storage level. A starting excitation storage level for a precursor ion can be calculated using the “q” calculator. The “q” calculator is accessed by right-clicking on any of the fields in the MS/MS parameters table.

The optimum excitation storage level is a tradeoff between a storage level high enough to allow fragmentation of the precursor ion and a storage level low enough to allow efficient trapping of the lowest m/z product ion. A higher excitation storage level allows more energy to be imparted to the precursor ions by allowing a higher excitation amplitude.

Excitation Amplitude (volts): Voltage used to excite the precursor ion causing it to dissociate into product ions. The amplitude range for non-resonant excitation is 0 to 120 volts. For resonant excitation, the range is 0 to 60 volts. The default values are 0.2 volts for the resonant excitation method and 20 volts for the non-resonant excitation method.

If the excitation amplitude used is too large, the precursor ion and product ion spectra will be absent because both ions will be ejected from the trap. If the value is too small, the precursor ion spectrum will be dominant and the product ion spectrum will be weak or missing.

Excitation Time: The excitation time is the time required for collision-induced dissociation (CID) by ion excitation. The excitation time range is 0 to 650 msec. The default excitation time is 20 msec for both resonant and non-resonant excitation.

Customize a Resonant Method

The parameters in the MS/MS Ion Prep Method Editor window are the basic ones needed to create a MS/MS Ion Prep Method file for most application. However, some applications may require further customization.

Customize Parameters is separated into three sections: Ionization, Isolation and Dissociation. These parameters are hidden by default but can be modified by clicking the **Customize** button.

Precursor Ion (m/z)	Ionization Storage Level (m/z)	Isolation Window (m/z)	Low Offset (m/z)	High Offset (m/z)	High Mass Ejection (volts)
450.0	35	3.5	0.0	0.0	35.0

Customize Ionization and Isolation Parameters

Ionization

Precursor Ion (m/z): The precursor ion is the desired ion m/z that will be isolated in the MS/MS isolation step. This Precursor Ion m/z value is used in both Resonant and Non-Resonant Methods of MS/MS. For more info, please review the previous table titled **Maximum Isolation Window as a Function of Precursor Mass**.

Ionization Storage Level: The m/z value at which the RF is maintained during ionization and the coarse isolation step. The range is 30 to 160 m/z , with a default value of 35 m/z .

Isolation

Isolation Window (m/z): The full mass isolation window range is 1.0 to 14.0 m/z . The actual range is dependent on the precursor ion. The default value is 3.0 m/z . Integral and fractional mass isolation window values are both accepted. If an isolation window smaller than 1.5 m/z is used, then the exact mass of the precursor ion should be entered in the Precursor Ion Mass field.

If the Low or High Edge Offset range is not sufficient to completely isolate the desired ions, either increase (in the case of desired ions not being present) or decrease (in the case of unwanted ions being isolated) the Isolation Window value.

Low Edge Offset: The mass offset to optimize the ejection of the mass just below the precursor ion mass. The **Low Edge Offset** range is -0.5 m/z to 0.5 m/z . The default value is 0.

Low Edge Offset affects the isolation window on the low mass side of the precursor ion. Increasing the mass offset (increasing the default from 0 to 0.1 m/z) makes the isolation window on the low mass side of the precursor ion larger. Decreasing the offset (decreasing from the default 0 to -0.5 m/z) decreases the window on the low mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments.

High Edge Offset: The mass offset to optimize the ejection of the mass just above the precursor ion mass. The **High Edge Offset** range is -0.5 m/z to 0.5 m/z . The default value is 0.

High Edge Offset affects the isolation window on the high mass side of the precursor ion. Increasing the mass offset (increasing the default from 0 to 0.1 m/z) makes the isolation window on the high mass side of the precursor ion larger. Decreasing the offset (decreasing from the default 0 to -0.1 m/z) decreases the window on the high mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments.

High Mass Ejection: Amplitude of broadband waveform used to eject masses above the isolated precursor ion. Default is 35 volts. If precursor ions are lost due to dissociation, reducing this amplitude may help. However, some ions with a m/z higher than the precursor ion may not be ejected.

Dissociation

Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Excitation Time (msec)	Modulate RF	Number of Frequencies	CID Frequency Offset (kHz)
Resonant ▼	198.2	0.20	20		1	0.0

Customize Dissociation Parameters (Resonant)

Waveform Type: The waveform type is either resonant or non-resonant. Resonant waveforms are harmonious with the frequencies of electrons held in

the ion trap. Non-resonant waveforms are not harmonious with the frequencies of the electrons held in the ion trap.

Excitation Storage Level (m/z): The RF storage level in m/z when the dissociation waveform is applied following isolation. The excitation storage level range depends on the precursor mass, but the lowest product ion must be more than several mass units above the excitation storage level. A starting excitation storage level for a precursor ion can be calculated using the “q” calculator. The “q” calculator is accessed by right-clicking on any of the fields in the MS/MS parameters table.

The optimum excitation storage level is a tradeoff between a storage level high enough to allow fragmentation of the precursor ion and a storage level low enough to allow efficient trapping of the lowest m/z product ion. A higher excitation storage level allows more energy to be imparted to the precursor ions by allowing a higher excitation amplitude.

Excitation Amplitude (volts): Voltage used to excite the precursor ion causing it to dissociate into product ions. The amplitude range for non-resonant excitation is 0 to 120 volts. For resonant excitation, the range is 0 to 60 volts. The default values are 0.2 volts for the resonant excitation method and 20 volts for the non-resonant excitation method.

If the excitation amplitude used is too large, the precursor ion and product ion spectra will be absent because both ions will be ejected from the trap. If the value is too small, the precursor ion spectrum will be dominant and the product ion spectrum will be weak or missing.

Excitation Time: The excitation time is the time required for collision-induced dissociation (CID) by ion excitation. The excitation time range is 0 to 650 msec. The default excitation time is 20 msec for both resonant and non-resonant excitation.

Modulate RF: Whether the RF storage field is modulated during resonant CID. This modulation greatly reduces any effect of changing secular frequency with concentration.

Number of Frequencies: The number of frequencies at which the CID excitation waveform is applied. The range is 1 to 119 frequencies, odd numbers only. The default value is 1 frequency applied at the end caps. A value of 3 generates three frequencies: the resonance frequency and frequencies 500 Hz above and below that frequency.

CID Frequency Offset: For certain applications it may be desirable to offset the dissociation frequency. The CID frequency can be adjusted between ± 10 kHz. CID frequency offset can only be applied to a single frequency. If the number of frequencies is greater than 1 this field is hidden.

Customize Precursor Ion Isolation

If you wish to further optimize precursor ion isolation by reducing the isolation window, first build a method with a mass isolation window of 3 m/z, an excitation time of 0 μ sec, and a CID (excitation) amplitude of 0 volts. This permits you to observe only the isolation of the precursor ion without the influence of CID. The exact fractional mass of the ion should be used for the precursor ion mass.

For practice, use a fixed sample source, such as perfluorotributylamine (PFTBA) calibration gas, or the column bleed obtained by raising the gas chromatograph oven temperature. Adjust the isolation parameters to isolate a single ion.

Select an Excitation Technique

The compound and matrix you are using determine which excitation technique you should use. Resonant excitation works well with most compounds, but non-resonant excitation has been found to work better with some. You are advised to begin your analysis with resonant excitation because it gives better results. If the results desired cannot be obtained using resonant excitation, switch to non-resonant excitation.

Optimize the Non-Resonant (or Resonant) CID Parameters

Follow these steps to optimize the non-resonant or resonant excitation CID parameters. Determine whether the CID (excitation) amplitude used was too large or too small.

NOTE: If the CID (excitation) amplitude used was too large, the precursor ion and product ion spectra will be absent because both ions will have been ejected from the trap. If the value was too small, the precursor ion spectrum will be dominant and the product ion spectrum will be weak or missing.

Increase or decrease the CID (excitation) amplitude until a change in the spectra is observed. In the case of non-resonant CID, use 10 volt steps; for resonant CID, use 0.2 volt steps. Continue to increase or decrease the CID (excitation) amplitude, but in smaller increments until suitable ion spectra are obtained. Adjust the excitation time in 10-ms steps to optimize the spectra.

NOTE: If the precursor ion is ejected instead of forming a product ion, increase the excitation storage level and adjust the excitation amplitude. In general, increasing the excitation storage level necessitates the use of higher amplitude to dissociate the ions. Do not raise the excitation storage level above the storage level where the product ions will not be stored.

Customize the Resonant Excitation Method

For practice, you can use a fixed sample source such as perfluorotributylamine (PFTBA) calibration gas or the column bleed obtained by raising the gas chromatograph oven temperature.

Iteratively adjust the excitation storage level, excitation amplitude, and excitation time to optimize precursor-to-product ion conversion. The optimum excitation amplitude will be slightly different when RF modulation is selected.

NOTE: If the amplitude used is sufficiently large, the precursor ion should be ejected. Precursor ion ejection indicates that the CID frequency applied to the end caps is properly resonant and that the trap function has been correctly calibrated.

To excite multiple ions at once, increase the CID bandwidth by increasing the Number of Frequencies from 1 to 3 frequencies. This increases the mass range around the precursor ion that is excited. Several frequencies may be used with or without modulation, and may be useful when exciting isotopic clusters.

Automated Method Development

Automated Method Development (AMD) is used to optimize CID conditions for product ion formation in a MS/MS method. AMD uses up to 10 different sets of conditions for the same precursor ion.

When using Automated Method Development (AMD) option, set the ion time for 1 microscan per analytical scan by adjusting Scans Averaged (General Parameters). This will ensure that the maximum number of cycles through the different voltages (scan segments) across each peak is performed.

Below is a strategy for using AMD to determine the optimum excitation storage level and excitation voltage:

- Determine the precursor ion and retention time for each compound from an injection using normal EI mode. Build a multi-segment acquisition method, with one segment per compound.
- Use the default conditions with Automatic Method Development to determine the mass of the product ion(s). AMD allows the CID voltage to be incremented on a scan-by-scan basis (20v, 40v, 60v) for up to 10 scans and then the cycle repeats.
- Once the m/z value of the product ion is determined, make the following calculation. Product ion m/z divided by 1.4 = the highest CID RF excitation level. For example, if the product ion is m/z 140, then the excitation storage level should be no higher than 100 to ensure good trapping efficiency of the product ion.
- Once the excitation storage level is set, the only variable to optimize is the CID voltage. This can be done with two injections using AMD. The first injection can use voltage increments of 10 volts (Non-resonant) or 0.2 volts (Resonant). The second injection uses smaller increments of the excitation amplitude near the optimal level.

The optimal excitation amplitude and excitation storage level are dependent on the compound and the analysis. Optimal excitation amplitude may be such that the precursor ion has been completely dissociated and only the product ions are scanned, or a small fraction of the precursor is still present and both precursor and product ions are scanned.

Determine the Voltage for Non-Resonant Excitation (AMD)

Follow these steps to determine the optimum voltage for non-resonant excitation by using AMD while injecting the precursor compound.

	Precursor Ion (m/z)	Isolation Window (m/z)	Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Product Ion Start Mass (m/z)	Product Ion End Mass (m/z)	More Range
1	414.0	3.0	Non-Resonant	48.0	10.00	50	420	☐
2	414.0	3.0	Non-Resonant	48.0	20.00	50	420	☐
3	414.0	3.0	Non-Resonant	48.0	30.00	50	420	☐
4	414.0	3.0	Non-Resonant	48.0	40.00	50	420	☐
5	414.0	3.0	Non-Resonant	48.0	50.00	50	420	☐
6	414.0	3.0	Non-Resonant	48.0	60.00	50	420	☐
7	414.0	3.0	Non-Resonant	48.0	70.00	50	420	☐
8	414.0	3.0	Non-Resonant	48.0	80.00	50	420	☐

Buttons: Insert, Add, Delete, "q" Calculator, Customize

Example of 20 V steps in AMD.

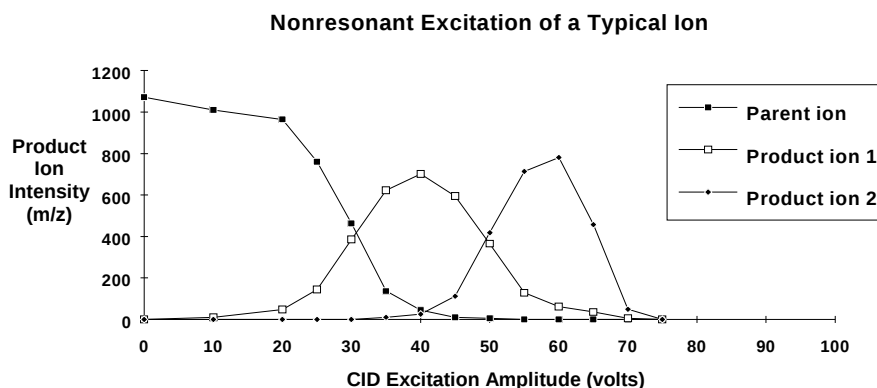
Setting the excitation amplitude for scan segment 1 to 0 volts will verify that the precursor ion is isolated without a large loss in intensity. Inject the sample and examine the spectra across the peak.

Set the excitation RF equal to the lowest mass product ion expected divided by 1.4. If the product ions are not yet known, start with the excitation RF = 48 m/z. Set up the AMD method to cycle through excitation voltages using increments of 10 or 20 volts.

If you require a more accurate value for the excitation voltage, change the excitation amplitude steps to 2 or 3 volts for each group near the best values found with the 10-volt increments and re-inject the sample.

NOTE: If the RF level was raised to trap the lowest product ion and you do not have a satisfactory number of product ions either try another precursor ion or switch to resonant excitation. Some ions are sufficiently stable that they are ejected from the trap before they can acquire enough energy to dissociate. Also try increasing the excitation time.

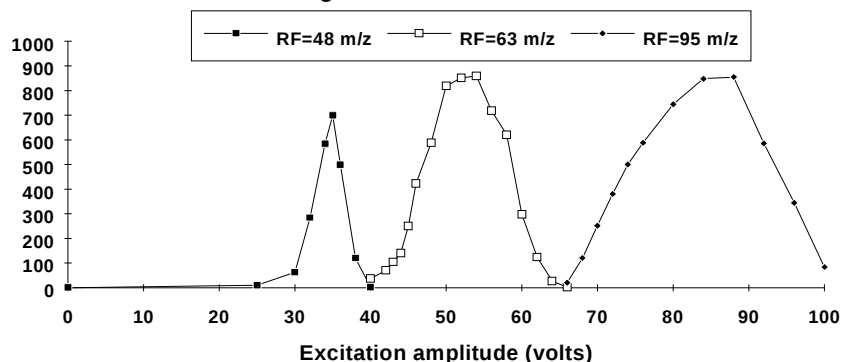
Determine the optimum voltage by plotting the product ion intensity data as a function of CID (excitation) amplitude as shown below.



In the case above, the optimal excitation amplitude would be about 40V. This is because there is still some precursor ion present, the primary product ion intensity is at the maximum, and the secondary product ion has just started to form.

Determine the optimum storage level by plotting the product ion intensity data as a function of CID (excitation) amplitude for different RF levels as shown below.

Product Ion Formation vs. Excitation RF for a Typical Ion Using Nonresonant Excitation



In the case shown above, the storage level of 48 m/z has a very narrow optimal excitation range. This may result in a change in dissociation over time as the excitation energy output drifts.

NOTE: The maximum yield of product ions shifts to higher CID (excitation) amplitudes as the RF level is increased.

Save AMD conditions to MS/MS.

How to Determine the Optimum Voltage for Resonant Excitation Using Automated Method Development (AMD)

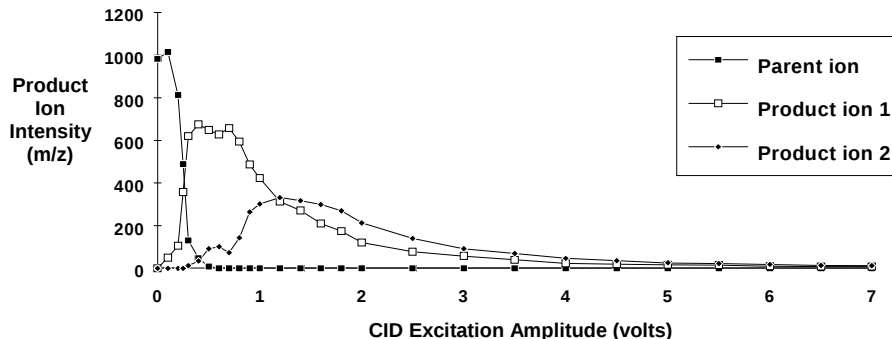
Follow these steps to determine the optimum voltage for resonant excitation using AMD while injecting the precursor compound.

	Precursor Ion (m/z)	Isolation Window (m/z)	Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Product Ion Start Mass (m/z)	Product Ion End Mass (m/z)	More Range
1	414.0	3.0	Resonant	48.0	0.00	50	420	☐
2	414.0	3.0	Resonant	48.0	0.10	50	420	☐
3	414.0	3.0	Resonant	48.0	0.20	50	420	☐
4	414.0	3.0	Resonant	48.0	0.30	50	420	☐
5	414.0	3.0	Resonant	48.0	0.40	50	420	☐
6	414.0	3.0	Resonant	48.0	0.50	50	420	☐
7	414.0	3.0	Resonant	48.0	0.60	50	420	☐
8	414.0	3.0	Resonant	48.0	0.70	50	420	☐

Set the excitation storage level equal to the lowest mass product ion expected divided by 1.4. If the product ions are not yet known, start with the excitation RF=48 m/z and an excitation time of 20 ms. Set up the AMD method to cycle through excitation voltages using increments of 0.2 or 0.5 volts.

Determine the optimum voltage by plotting the product ion intensity data as a function of CID (excitation) amplitude as shown below.

Resonant Excitation of a Typical Ion



This figure shows a typical breakdown curve for resonant excitation using modulation. The optimal excitation amplitude for maximizing product ion 1 is around 0.5 volts. The optimal excitation amplitude for producing both product ions 1 and 2 is around 1.2 volts.

Save AMD to MS/MS method.

MS/MS with Different Precursor Ions

Multiple Reaction Monitoring (MRM) is using MS/MS with different precursor ion masses and different dissociation parameters. MRM is capable of doing MS/MS of up to ten different precursor ions. This is useful, for example, for a target compound and a labeled standard which have a mass difference larger than the maximum allowed isolated window and which co-elute.

When using MRM, set up the 4000MS acquisition method for 3 microscans per analytical scan. Each scan function will have its own data channel. When doing quantitation, MS Data Review can analyze these channels separately.

Each of the MRM scan functions must be optimized individually using AMD. The optimal AMD conditions can be saved to a MS/MS method by highlighting the line and changing the scan type from AMD to MS/MS. You can copy a MS/MS (or AMD) line by highlighting the line and typing <ctrl> c (copy). This line can be copied into the MRM (or MS/MS) method by <ctrl> v (paste).

NOTE: If you are doing linearity studies using MRM, you may need to change the default prescan ionization time of 300 μ sec to obtain optimal linearity. This value depends on the compound and the concentration range.

MS/MS/MS (or MSⁿ)

MSⁿ uses sequential MS isolation, dissociation and trapping steps to gain information about a given precursor ion. In this operation a product ion from a previous CID step is isolated and becomes the precursor ion for the next CID step. The additional dissociation step can be used to increase selectivity of analyte versus background or for additional structure information. MS³ uses two precursor ions for sequential dissociation. MS¹⁰, or MS/MS/MS/MS/MS/MS/MS/MS/MS/MS, is 9 isolation and dissociation steps before the final scan of product ions.

General Parameters | Ionization Control | External EI Parameters | MSⁿ Parameters

MSⁿ

	Precursor Ion (m/z)	Isolation Window (m/z)	Waveform Type	Excitation Storage Level (m/z)	Excitation Amplitude (volts)	Product Ion Start Mass (m/z)	Product Ion End Mass (m/z)	More Ranges
1	496.0	5.0	Resonant	57.7	0.35	425	428	☐
2	426.0	5.0	Resonant	57.7	0.42	355	358	☐
3	356.0	3.0	Resonant	57.7	0.38	58	141	☐
4								☐
5								☐
6								☐
7								☐
8								☐
9								☐

Insert Add Delete "q" Calculator Customize

The optimization of MSⁿ is similar to MS/MS. First, work out optimized conditions for MS/MS of the first precursor ion. AMD will make this process go faster.

Then optimize the conditions for dissociation of that product ion (entered as the second precursor ion). You may need to make sequential injections using the MS³ method. If the second (or third)-generation product ion is in the original EI or CI spectrum, then you can use AMD to find the optimum conditions.

Selected Ion Storage

Selected Ion Storage (SIS) may be used with either External EI or Internal EI.

Using the Selected Ion Storage (SIS) option, you may selectively accumulate or store specific ions in the ion trap. Success with this technique requires that there be no excessive space-charge build up. Optimum mass resolution is achieved by controlling the number of ions in the trap.

SIS eliminates unwanted ions by ejecting them from the trap. Given the optimum number of ions that can be stored in the trap, SIS enriches the sample ions relative to the unwanted matrix ions by ejecting the latter throughout ionization. The unwanted ions are ejected from the trap by a technique known as *resonant ion ejection*.

Operational Modes for SIS

SIS is enabled using the scan Type/Group drop-down list box.

SIS can operate in several mode; Single-Ion, Multiple-Ion, Range, and Cluster. Each is described below.

General Parameters | Ionization Control | Internal EI Parameters | **SIS Parameters**

SIS

	Low Mass (m/z)	High Mass (m/z)	Ionization Storage Level (m/z)	Target Factor (%)	Waveform Scale Factor (%)
1	200	208	28	100	100
2	370	382	35	150	100
3	440	458	35	100	75
4	468	469	35	100	150
5					
6					

☒ AutoScale Waveform

SIS mode; Customize

The Ionization Storage Level, Target Factor (Ion Time Factor), and Waveform Scale Factor can be adjusted for each of the mass segments. The Waveform Scale Factor can be autoscaled—based on the calibration—or explicitly set.

NOTE: There is a minimum gap of 10 m/z between the end of one segment and the start of the next segment.

Single-Ion Mode

Single-Ion Mode is where a single ion is selected for storage.

SIS

	Low Mass (m/z)	High Mass (m/z)
1	276	276
2		

SIS; Single-Ion Mode

The Single-Ion mode of SIS has the advantage of removing all of the background noise from the analysis. However, since there is only one ion, there is no secondary information, and therefore no confirmation of the compound.

NOTE: Unit mass isolation can only take place at low mass; at higher masses, a range of ions is stored even when a single mass is entered.

Multiple-Ion Mode

SIS		
	Low Mass (m/z)	High Mass (m/z)
1	276	276
2	414	414
3	890	890

SIS; Multiple-Ion Mode

The Multiple-Ion Mode is used to collect several ions from a single analyte. This allows for confirmation of compound identity through ion ratio confirmation. Up to six (6) ions can be specified in a single time segment.

Range Mode

SIS		
	Low Mass (m/z)	High Mass (m/z)
1	150	350
2	450	500

SIS; Range Mode

The Range Mode is good at eliminating sections of the spectra where high background ions are present, such as column bleed at 207 m/z. Up to 6 ranges can be analyzed in a single time segment.

Cluster Mode

SIS		
	Low Mass (m/z)	High Mass (m/z)
1	200	208
2	370	382
3	440	458

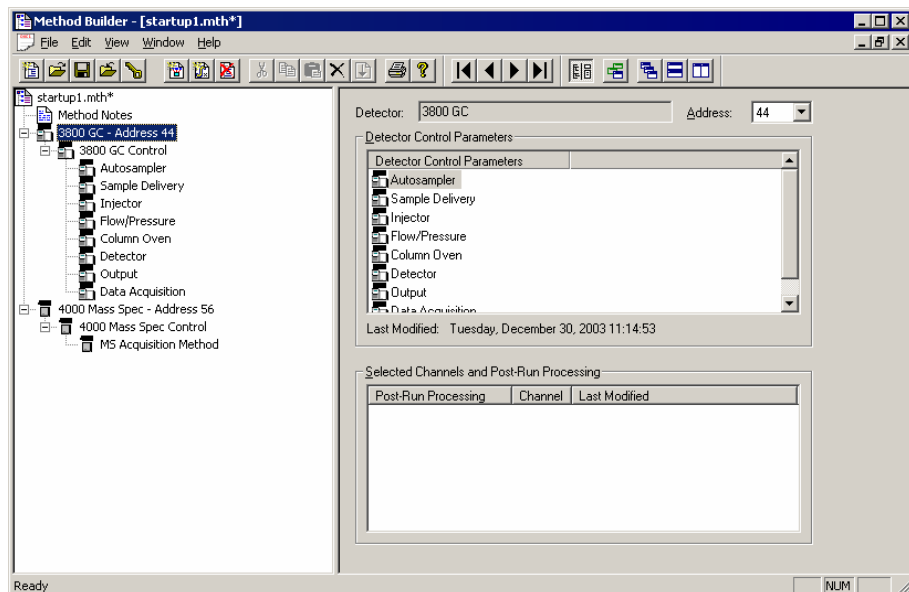
SIS; Cluster Mode

The Cluster Mode is a combination of the Range and the Multiple-Ion Modes. The Cluster Mode is set up to store only the ion clusters of a selected compound. This is useful when looking at chlorinated or brominated compounds. The Cluster Mode retains all of the isotope ratio information but removes most of the background ions for better sensitivity. The clusters of octachlorobiphenyl ether at 201, 372, and 442 are shown in the above example.

3800 GC Method Section

The 3800 GC Method Windows

The 3800 GC entry specifies the Varian MS Workstation module address of the GC that the method uses. The module address in the parameter window can be changed so that a method developed for an instrument using one module address can be easily modified for use on another instrument at a different module address.



The 3800 Method section contains eight Method windows: Autosampler, Sample Delivery, Injector, Flow/Pressure, Column Oven, Detector, Output, and Data Acquisition. These windows contain all parameters that control the operation of the 3800 GC during a chromatographic run. If the 4000 MS is the only detector and samples are injected by syringe, the Sample Delivery, Detector, Output, and Data Acquisition sections are irrelevant and will not be discussed in this manual. For information on these sections of the 3800 GC method, see the 3800 GC Operator's Manual.

Checking Method End Times

All editing windows other than the Column Oven window and Detector window show the current end time of the column oven program above the **Save** and **Cancel** buttons.

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	50		1.00	1.00
2	200	200	20.00	21.75
3				
4				
5				

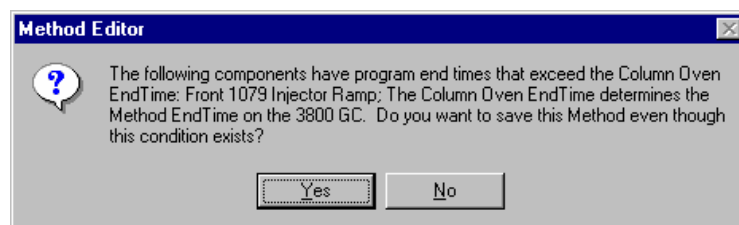
Add

Insert

Delete

Column Oven End Time: 20.00 min

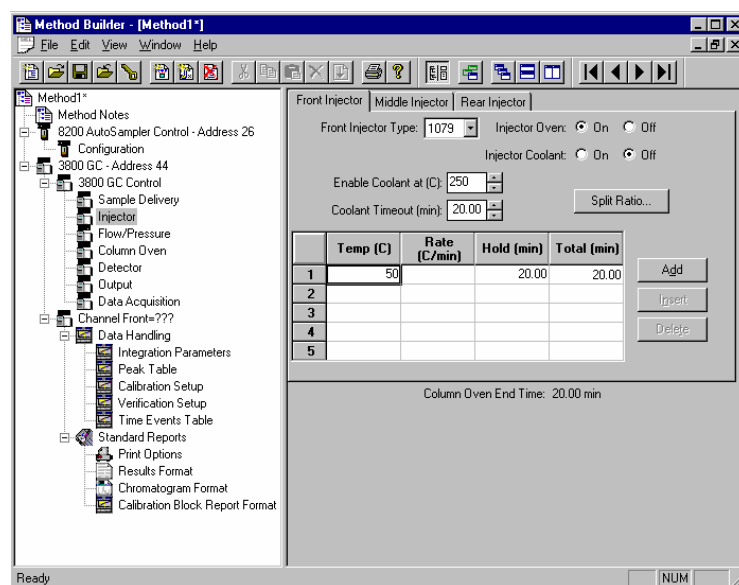
If you enter program end times for components other than the column oven that exceed the column oven end time, the following warning is displayed.



When the Method is downloaded to the 3800 GC, component program end times that exceed the column oven end time will be truncated.

Injector Window

The Injector window contains up to three Injector Programs (Front, Middle, Rear). Only the isothermal 1177 and temperature-programmable 1079 injectors are available on the 4000 GC/MS system. Use the Injector Programs to specify the temperature set point and split ratio of the isothermal 1177 Injectors, and to specify the temperature ramp and split ratio of the programmable 1079 Injector.



Use the tabs to select which Injector Program you wish to edit.

Select the type of injector (1177 or 1079) at each position, or select "None" if no injector is installed at that position.

1079 Injector

If you indicate that a 1079 Injector is installed, an Oven Power switch and a Coolant switch are displayed.

Front Injector | Middle Injector | Rear Injector

Front Injector Type: 1079 Injector Oven: ☒ On ☐ Off

Injector Coolant: ☐ On ☒ Off

Enable Coolant at (C): 250

Coolant Timeout (min): 20.00 Split Ratio...

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	50		20.00	20.00
2				
3				
4				
5				

Add Insert Delete

Column Oven End Time: 20.00 min

Indicate whether or not you will use the installed 1079 injector by clicking on the **On** or **Off** radio buttons (buttons relate to Injector Oven and Injector Coolant).

If you indicate that Coolant is turned on, specify the desired “Enable Coolant at” temperature and “Coolant Timeout” time.

Use the spreadsheet to build a temperature ramp program. The first row of the spreadsheet contains the initial temperature and hold time for the 1079 injector. The 1079 will equilibrate to this setting when the method is activated, and will be restored to this setting when each chromatographic run is completed.

Rows after the first row of the spreadsheet contain the programmed settings for each of the ramp segments. Each segment will ramp to the specified temperature at the specified rate (assuming the rate is achievable), and then hold the temperature for the specified time.

NOTE: The **Rate** in the first row is always blank and cannot be edited. The **Total** column cannot be edited either.

1079 Split Ratio

Front Injector | Middle Injector | Rear Injector

Front Injector Type: 1079 Injector Oven: ☒ On ☐ Off

Injector Coolant: ☐ On ☒ Off

Enable Coolant at (C): 250

Coolant Timeout (min): 20.00 Split Ratio...

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	50		20.00	20.00

Add

If the 1079 Injector is connected to a Type 1 EFC, press the **Split Ratio** button to build the Split Ratio Time Program.

NOTE: You must have already configured the Type 1 EFC in the corresponding position in the Flow/Pressure Section before you can program the Split Ratio. Specifically, to build a 1079 Split Ratio Program, a Front 1079 must have a corresponding Front Type 1 EFC, a Middle 1079 must have a corresponding Middle Type 1 EFC, and a Rear 1079 must have a corresponding Rear Type 1 EFC.

Use the Split Ratio spreadsheet to build a Split Ratio Time Program to control the 1079 injector / Type 1 EFC combination.

	Time	Split State	Split Ratio
1	Initial	On	20
2	0.01	Off	Off
3	1.00	On	100
4			
5			
6			

Column Oven End Time: 20.00 min

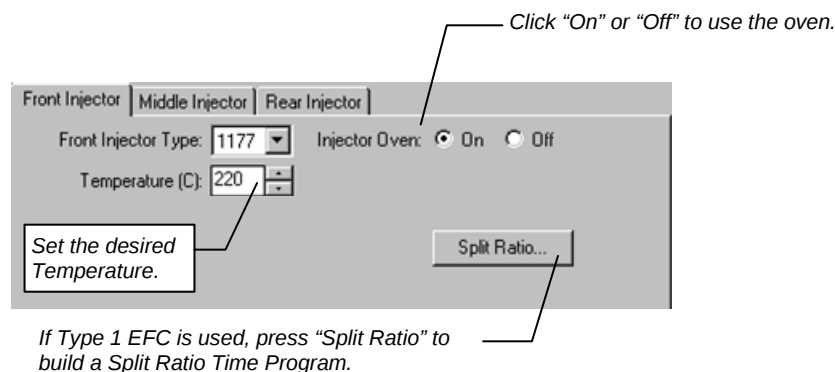
This is a typical program for a splitless injection. A low split ratio conserves carrier gas while keeping contaminants from building up in the injector. The injector is kept splitless for about 1 minute so that all analytes injected can focus on the column. A split ratio of 100 after injection flushes residual solvent from the injector.

The first row of the spreadsheet contains the Initial Split State and Split Ratio for the 1079 injector. The 1079 will equilibrate to this setting when the method is activated, and will be restored to this setting when each chromatographic run is completed.

Rows after the first row of the spreadsheet contain the time-programmed settings for the 1079 Split Ratio. The Split Valve will be switched to the ratio in each row at the indicated time.

1177 Injector

If you indicate that an 1177 Injector is installed, an Injector Oven Power switch, a Temperature setting and a Split Ratio button are displayed.



NOTE: The Type 1 EFC in the corresponding position in the Flow/Pressure Section must be configured before you can program the Split Ratio. Specifically, to build an 1177 Split Ratio Program, a Front 1177 must have a corresponding Front Type 1 EFC, a Middle 1177 must have a corresponding Middle Type 1 EFC, and a Rear 1177 must have a corresponding Rear Type 1 EFC.

Use the Split Ratio spreadsheet to build a Split Ratio time program to control the 1177 injector / Type 1 EFC combination.

Split Ratio for Front 1177 + Front Type 1 EFC

	Time	Split State	Split Ratio
1	Initial	On	20
2	0.01	Off	Off
3	1.00	On	100
4			
5			
6			

Column Oven End Time: 20.00 min

Buttons: Add, Insert, Delete, Sort, Save, Cancel

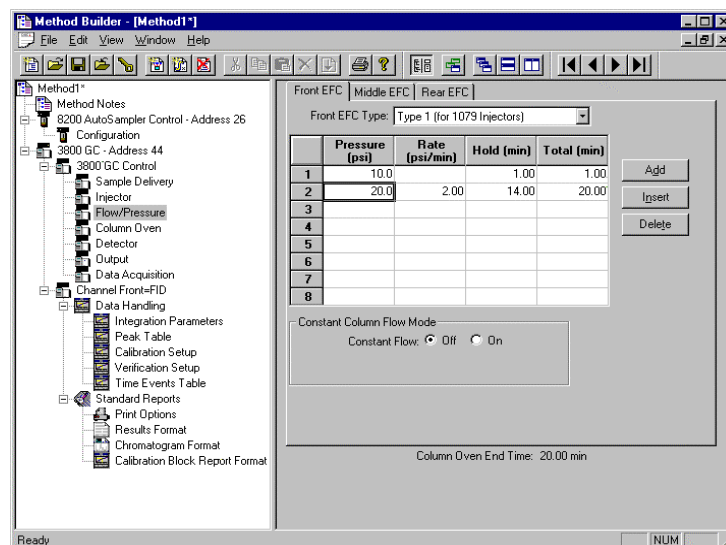
This is a typical program for a splitless injection. A low Split Ratio conserves carrier gas while keeping contaminants from building up in the injector. The injector is kept splitless for about 1 minute so that all analytes injected can focus on the column. A Split Ratio of 100 after injection flushes residual solvent from the injector.

The first row of the spreadsheet contains the Initial Split State and Split Ratio for the 1177 injector. The 1177 will equilibrate to this setting when the method is activated, and will be restored to this setting when each chromatographic run is completed.

Rows after the first row contain the time-programmed settings for the 1177 Split Ratio. The Split Valve switches to the ratio at the indicated time.

Flow/Pressure Window

The Flow/Pressure window contains up to three Electronic Flow Control (EFC) Programs (Front, Middle, Rear). Only Type 1 and Type 5 EFC modules can be configured on the 4000 GC/MS system. Type 5 EFC is used for applications like purge-and-trap or static headspace sample introduction.



Use the tabs to select which EFC Program you wish to edit.

Select the type of EFC (Type 1, Type 3, or Type 4) at each position, or select "None" if no EFC is installed at that position.

Type 1 EFC (for 1079/1177 Injectors)

If you indicate that a Type 1 EFC is installed, a pressure ramp spreadsheet will appear.

The screenshot shows a software window titled 'Front EFC | Middle EFC | Rear EFC'. The 'Front EFC' tab is active, and a dropdown menu shows 'Type 1 (for 1079 Injectors)'. Below this is a table with 5 columns: an index column (1-8), 'Pressure (psi)', 'Rate (psi/min)', 'Hold (min)', and 'Total (min)'. The first row contains values 10.0, blank, 1.00, and 1.00. The second row contains 20.0, 2.00, 14.00, and 20.00. To the right of the table are buttons for 'Add', 'Insert', and 'Delete'. Below the table is a section for 'Constant Column Flow Mode' with a 'Constant Flow' label and two radio buttons, 'Off' (selected) and 'On'. At the bottom of the window, it says 'Column Oven End Time: 20.00 min'.

	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
1	10.0		1.00	1.00
2	20.0	2.00	14.00	20.00
3				
4				
5				
6				
7				
8				

Constant Column Flow Mode
Constant Flow: ☒ Off ☐ On

Column Oven End Time: 20.00 min

Use the spreadsheet to build a pressure ramp program to control the Type 1 EFC.

The first row of the spreadsheet contains the Initial pressure and hold time for the Type 1 EFC. The EFC will equilibrate to this setting when the method is activated, and will be restored to this setting when each chromatographic run is completed.

The Rows after the first row of the spreadsheet contain the programmed settings for each of the ramp segments. Each segment will ramp to the specified pressure at the specified rate (assuming the rate is achievable), and then hold the pressure for the specified time.

Note that the *Rate* in the first row is always blank and cannot be edited. Also note that the entire *Total* column cannot be edited.

Constant Column Flow mode for Type 1 EFC is enabled in the Flow/Pressure window.

Front EFC
Middle EFC
Rear EFC

Front EFC Type:
Type 1 (for 1079 Injectors)

	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
1				
2				
3				
4				
5				
6				
7				
8				

Add
Insert
Delete

Constant Column Flow Mode

Constant Flow:
Off
On

Column Flow (ml/min):
5.0

Column Oven End Time: 20.00 min

Click on the Radio Button to turn on the Constant Column Flow Mode. This mode disables the spreadsheet and reveals the Constant Flow rate value.

Enter the desired Column Flow.

When the method is activated in System Control, the Varian MS Workstation will generate and download the appropriate pressure program corresponding to the Column Temperature program, the Column Setup Length and Diameter, the Carrier Gas, and the ambient Barometric Pressure. These parameters are specified at the 3800 GC front panel, and the Barometric Pressure is measured by the 3800 at the time the Method is downloaded. The 3800 will run the pressure program to hold the Column Flow at the specified value as the Column Temperature is ramped during each run.

Column Oven Window

The Column Oven window contains the Column Oven Program. Use the Column Oven Program to specify the Coolant Parameters and Stabilization Time of the Column Oven, and to specify its programmable temperature ramp.

Method1*
File Edit View Window Help

Method1*
Method Notes
8200 AutoSampler Control - Address 26
Configuration
3800 GC - Address 44
Sample Delivery
Injector
Flow/Pressure
Column Oven
Detector
Output
Data Acquisition
Channel Front=???
Data Handling
Integration Parameters
Peak Table
Calibration Setup
Verification Setup
Time Events Table
Standard Reports
Print Options
Results Format
Chromatogram Format
Calibration Block Report Format

Column Oven Coolant: ☐ On ☒ Off
Enable Coolant at (C): 50
Coolant Timeout (min): 20.00
Stabilization Time (min): 2.00

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	50		20.00	20.00
2				
3				
4				
5				
6				
7				
8				

Add
Insert
Delete

Ready NUM

Indicate whether or not you will use the Coolant by clicking on the **On** or **Off** radio buttons.

Use the spreadsheet to build a temperature ramp program to heat and/or cool the Column Oven.

Specify the desired temperatures at which to Enable Coolant and then set the times for Coolant Timeout and Stabilization Time.

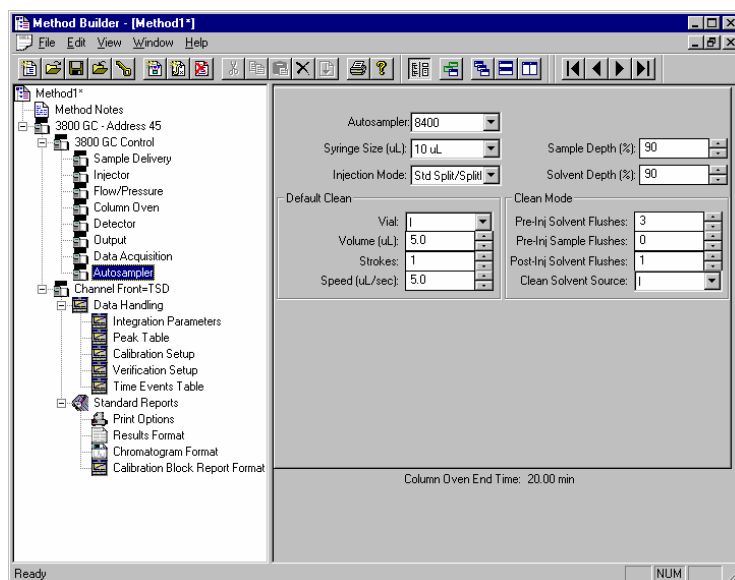
The first row of the spreadsheet contains the Initial temperature and hold time for the Column Oven. The Column Oven will equilibrate to this setting, and stabilize for the specified Stabilization Time, when the method is activated, and will be restored to this setting when each chromatographic run is completed.

Rows after the first row of the spreadsheet contain the programmed settings for each of the ramp segments. Each segment will ramp to the specified temperature at the specified rate (assuming the rate is achievable), and then hold the temperature for the specified time.

NOTE: The *Rate* in the first row is always blank and cannot be edited. Also note that the entire *Total* column cannot be edited.)

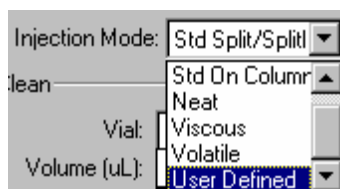
Autosampler Window

The Autosampler window contains sampling parameters for the 8400/8410 Autosampler when attached to the 3800 GC. The parameters include selection of the sampling syringe size, the penetration depth for sample and solvent wash vials, the default clean and clean modes, and the injection modes.



Default clean steps are used in the case that automation is stopped or a serious fault is detected. Select Pre and Post injection syringe clean modes.

Injection Mode

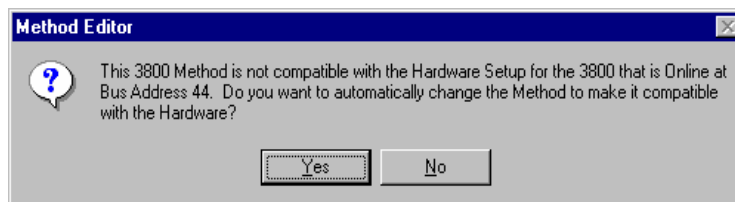


The Injection Mode selection consists of five predefined injection modes: Standard Split/Splitless, Standard On-Column, Neat, Viscous and Volatile. If any of these modes cannot satisfy your injection needs, a sixth User Defined mode is available. Selecting User Defined mode allows you access to all 8400 autosampler parameters to fine-tune your injection.

Consult the Online Help for a detailed description of all Injection Mode parameters.

Auto-Configuring the Method to Match the 3800 GC Hardware

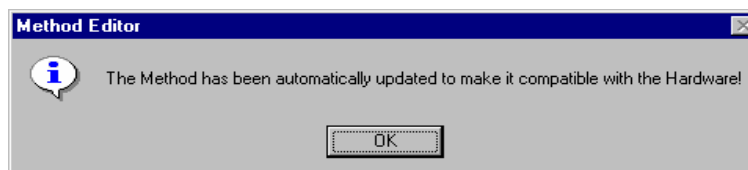
When you edit a 3800 GC Method while the corresponding GC is online in System Control, you are warned if the Method configuration does not match the hardware on the GC.



Click on **Yes** to change the Method to match the GC.

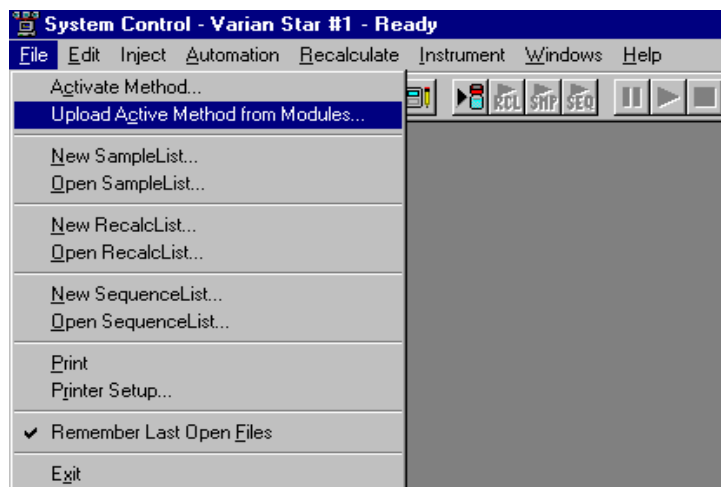
Click on **No** to accept the Method as is.

If you choose to auto-configure the Method, the following dialog is displayed.



Uploading the Method from the 3800 GC

You can get the Method from the GC and save it in the Varian MS Workstation. This allows you to do local Method editing on the 3800 GC front panel and then save this Method for later use on the Workstation.



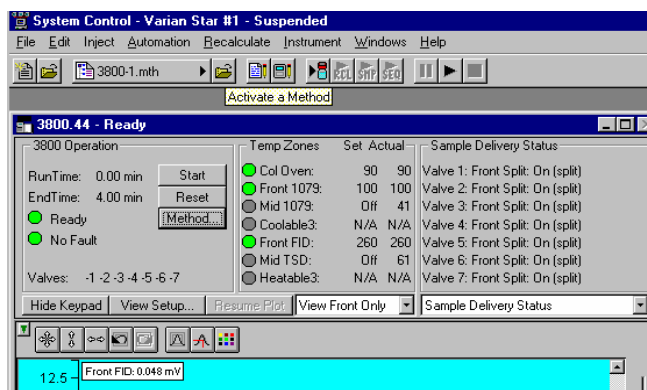
To upload the Method from the 3800 GC to System Control, select **Upload Active Method from Modules** from the File menu.

You are then prompted for the name of the file under which to save the Method. The active Method in System Control is updated to reflect the parameters obtained from the 3800 GC.

The Startup Method

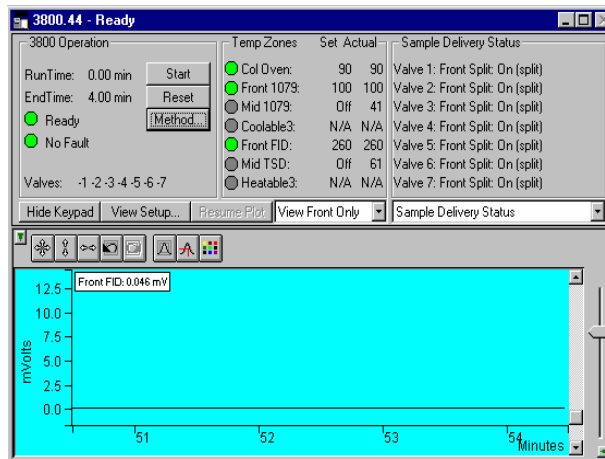
When you start System Control and display an Instrument Window, the last active Method for that instrument is activated. When System Control is started, it will return to the initial settings in the Method that was last used on the instrument.

To change Methods, simply click on the **Activate a Method** button on the System Control Toolbar or choose **Activate Method** from the File menu.



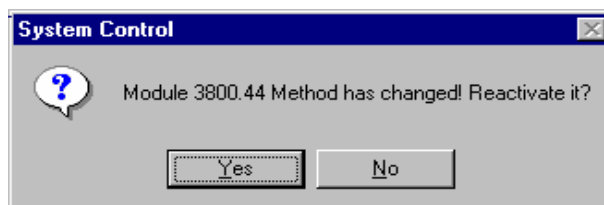
Editing Methods from the 3800 Status and Control Window

The 3800 GC Status and Control Window contains a Method... button that allows you to directly edit the active Method corresponding to that GC.



When you click on this button, Method Builder is opened with the active Method loaded and the corresponding Instrument Module selected. You can then edit that section or any other section of the Method as described previously.

Once you have edited the Method and closed the Method Builder window, you are prompted to reactivate the Method. Reactivating the Method downloads the changes to the Module.



Acquiring GC/MS Data

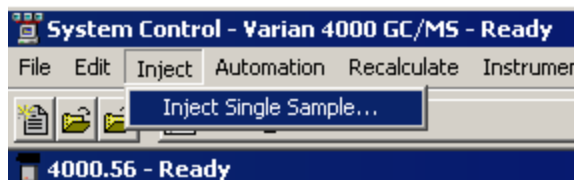
Injecting a Single Sample

Overview

If you are doing method development or are just looking at one sample, you may prefer to acquire data one sample at a time. If you want to run multiple samples, automation using a SampleList is the most efficient route. If the 3800 GC and 4000 MS are configured and a GC/MS Method has been built for data acquisition, you are ready to acquire data with either of these approaches. If this has not been done, please see the previous sections for instructions.

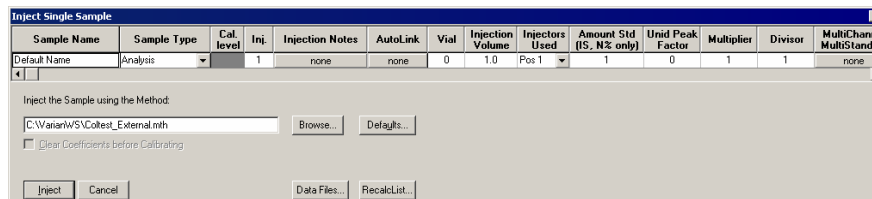
Using the Inject Single Sample Dialog Box

You can inject a single sample from System Control by using the Inject Single Sample dialog box.



Display the Inject Single Sample dialog by selecting it from the Inject menu or by clicking on the **Inject Single Sample** button on the toolbar .

The Inject Single Sample dialog box is displayed. The dialog will be automatically configured to match the sampling device installed in the instrument.

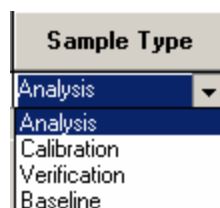


Defining the Fields and Buttons

The top of Sample List window has fields. The following are definitions.

Sample Name Enter the sample name here. The final name of the file depends on settings accessed by clicking the **Data Files** button. If only %s is listed in the Data Files dialog, the sample name is as entered.

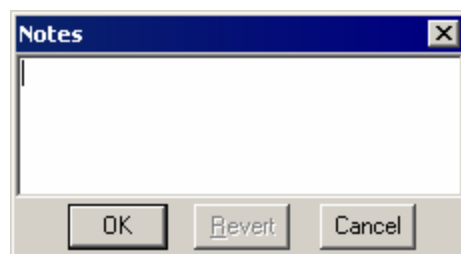
Sample Type There are several choices available from the Sample Type drop-down list box:



Cal. level If you designate the Sample Type as calibration, you can designate which calibration level you are injecting. You can enter up to ten levels.

Inj. Specify here the number of injections of this sample. You may make up to nine replicate injections at each calibration level.

Injection Notes Enter notes about the sample.



AutoLink See the Specifying Data Handling Parameters section in this manual.

Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
0	1.0	Pos 1 ▾	1	0	1	1	none

In the Vial, Injection Volume, and Injectors Used fields, enter information about the sample. The fields beginning with Amt. Std. are used only for data files acquired with standard GC detectors.

Select the Method to use for the run. Click the **Browse** key to select a file using Windows Explorer.

Click here to change the default entries in the table above, such as Injection Volume, Injector Used, etc. After defining new defaults, click the **Save** button to activate the new defaults.

Change the location and name of the Data Files here.

Click to generate automatically a Recalc list associated with single sample acquisitions.

Click **Inject** to start the run.

Specifying the Data File Name and Path

Data File names can be up to 255 characters long. Sample ID, injection date, module name, and injection number can be embedded in the file name making the Data File name correlate with each sample injection. When you click on the **Data Files** button, the Data File Generation dialog box is displayed. The left side of the Data File Generation dialog box allows you to select the drive letter and subdirectory (path) where the data files are to be stored.

The Data File names field allows you to create a file name “specification.” Follow the directions in the text shown below this field to specify file names that contain sample injection specific information. In the Example field, you will see a dynamic update of an example file name as you type in the specification. This makes it easy to see how a Data File created with this file name specification will appear.

Specifying Per-Sample Data Handling Parameters

NOTE: Data Handling Parameters in the Sample List are applied only to data collected with standard GC detectors!

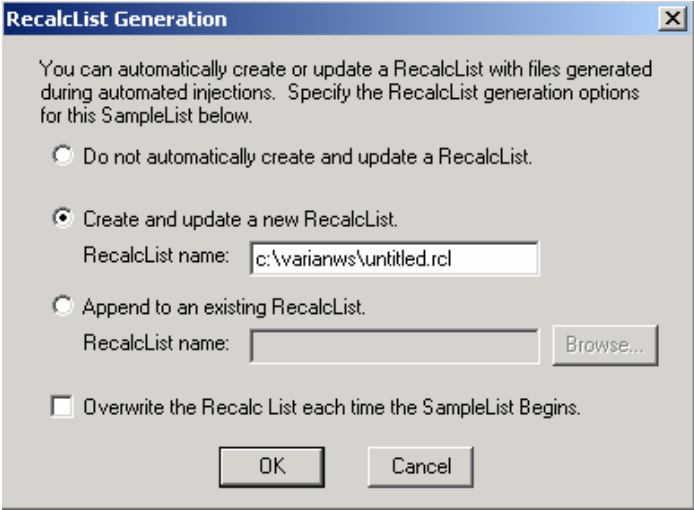
Most Data Handling parameters are specified in the Method used during the injection. Some parameters may vary on a per-sample basis, and are therefore specified when you perform the injection. The following Data Handling parameters can be specified on a per-sample basis:

- Unidentified Peak Factor
- Multiplier
- Divisor
- Amount Standard when one Internal Standard is being used

See the GC Data Handling and Reports Manual for a brief description of these parameters. See the Star Workstation Regulatory Compliance Manual for a complete description of how these parameters are used to calculate results.

Specifying a RecalcList

You can choose to create a new RecalcList, append to an existing RecalcList, or neither create nor update a RecalcList. To select an option that you want, click on the **RecalcList** button and the related dialog box will be displayed.



The image shows a dialog box titled "RecalcList Generation". It contains the following text and controls:

You can automatically create or update a RecalcList with files generated during automated injections. Specify the RecalcList generation options for this SampleList below.

☐ Do not automatically create and update a RecalcList.

☒ Create and update a new RecalcList.

RecalcList name:

☐ Append to an existing RecalcList.

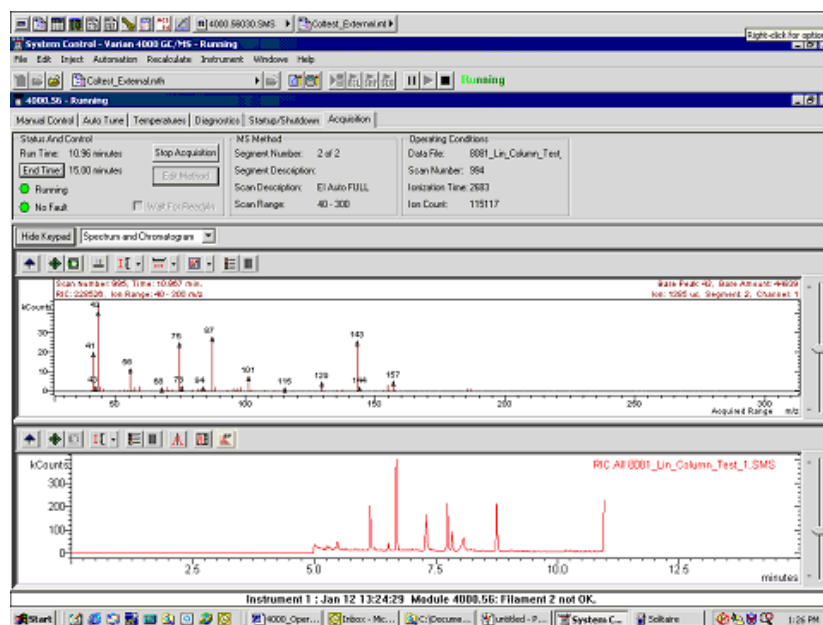
RecalcList name:

☐ Overwrite the Recalc List each time the SampleList Begins.

If you choose to create a new RecalcList, this automatically-generated RecalcList will not overwrite an existing RecalcList. If a RecalcList with the same file name exists, the newly created RecalcList will have number appended to its file name to make it unique and to prevent the older RecalcList from being overwritten. Alternatively, you may select the checkbox to overwrite the RecalcList each time the SampleList begins.

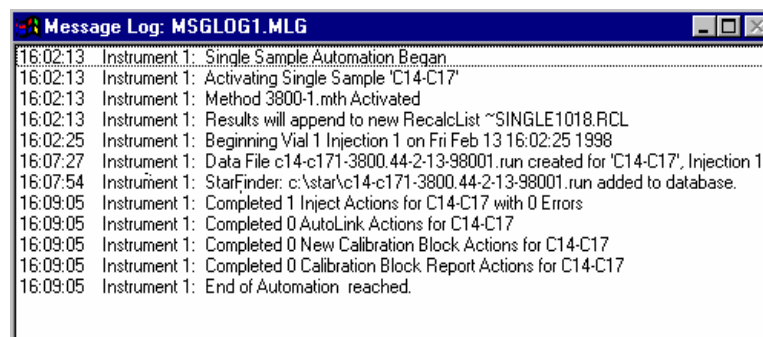
Monitoring the Status of the Run

After an injection is performed, the status of the run can be monitored in the instrument window.



The module status is shown in the status and control windows and on the Toolbar.

Automation actions and errors are logged in the Message Log; the latest line of this log is shown at the bottom of the screen. When you double-click on the status bar at the bottom of the instrument window, the entire Message Log window is displayed.



All Message Log entries are stamped with the time they occurred.

Using QuickStart to Acquire a Single Sample

QuickStart is a fast way to inject a single sample without using System Control directly. QuickStart can be customized and is ideal when setting up instruments for routine use. See the on-line help in QuickStart for further details.



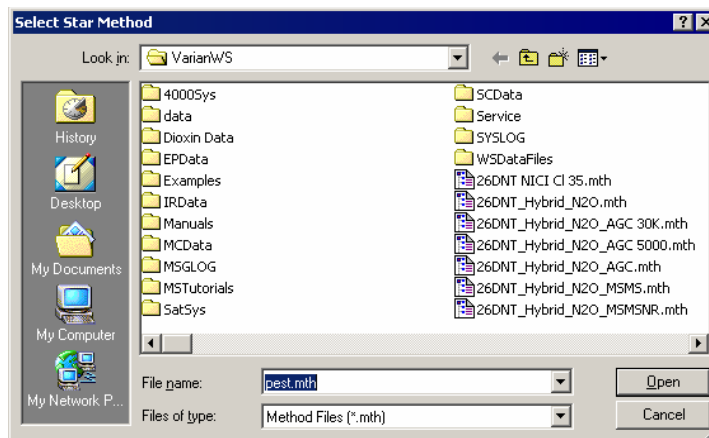
QuickStart starts System Control and waits for the modules to log in. When the instrument is ready, the QuickStart dialog is displayed.

Entering Information in the QuickStart Dialog

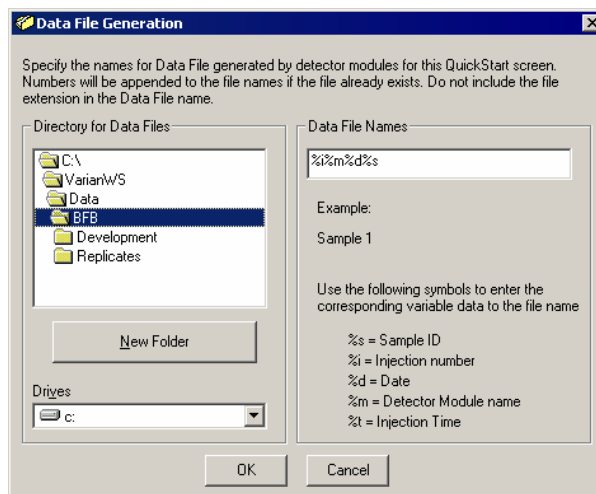
The instrument number, name, and operator are entered in the top three fields.

Enter sample name and descriptions, if desired, in the next three fields.

Click the mouse in the Primary Method field. Either modify the name here or click the **Browse** button to select from the Windows Explorer menu:



In a similar manner, select a path for data file storage by first clicking the mouse within the Folder for Data File Storage field and then either typing the path or clicking the **Browse** button to open the Data File Generation dialog and choose the path. To tailor the file name, follow the instructions in the Data File Names field.



Choose the Sample Type: Analysis, Calibration, Baseline, or Verification.

NOTE: Selecting Baseline will actually create an Analysis file type for GC/MS data.

Click **AutoLink** to add any post-run processing commands you want to use (optional). For example, you can Browse for a Custom MS Report Editor software template and insert it as an AutoLink command. Then, in System Control processing, you can have automated printing of Custom MS Report Editor reports at the end of a run.

The **AutoLink Commands** dialog box contains three sections for configuring commands:

- Start of SampleList:** Fields for **Command/Method** and **Other Parameters**, with a **Browse** button.
- Injections:** Fields for **Command** and **Other Parameters**, with a **Browse** button.
- End of SampleList:** Fields for **Command/Method** and **Other Parameters**, with a **Browse** button.

At the bottom are **OK** and **Cancel** buttons.

Click **Hardware** to select parameters for the AutoSampler. This is the dialog if an 8400 AutoSampler is configured:

The **8400 Hardware & Configuration** dialog box includes the following settings:

- Sampling Device:** Radio buttons for **5 uL Syringe**, **10 uL Syringe** (selected), and **100 uL Syringe**.
- Injectors Used:** A dropdown menu showing **Pos 1**.
- Between Injections:** A dropdown menu showing **Duplicate**.
- 2nd Inj. Volume:** A text field with the value **1.0**.
- Injection Delay:** A text field with the value **0.0**.
- Second Sample Name:** A text field containing **%s #2**.
- Second Sample Notes:** A large text area for additional notes.

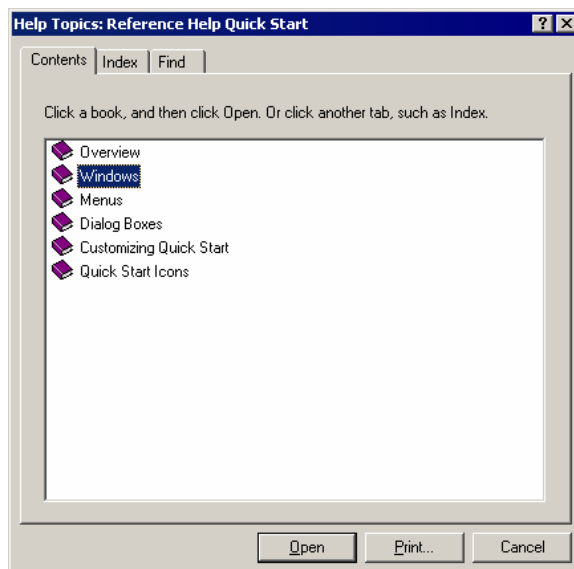
OK and **Cancel** buttons are at the bottom.

Enter sampling information. This information is specific to the type of sampling device installed. Note that Factor, Multiplier, and Divisor fields apply only to data acquired with standard GC detectors, not MS data files. Level is the Calibration level if that is the sample type.

A vertical stack of input fields for sampling parameters, each with a label and a text box containing the value **1**:

- Vial**
- # Injects**
- Volume**
- Amount**
- Factor**
- Multiplier**
- Divisor**
- Level**

Click **Help** for more information about QuickStart.



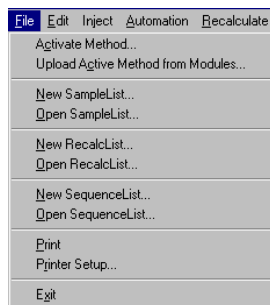
Injecting Multiple Samples

Introduction

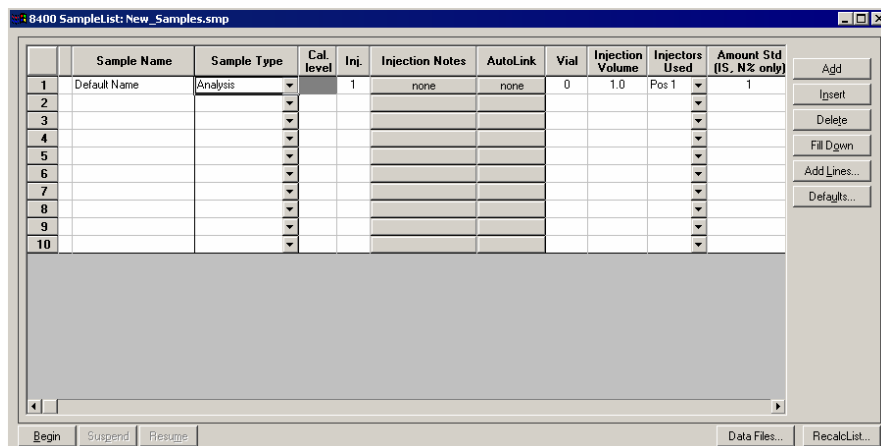
Before injecting multiple samples, the 3800 GC, 4000 MS, and AutoSampler must be configured and a GC/MS Method must be created.

Using a SampleList in System Control

You can create and edit a SampleList in the Automation File Editor or in System Control. In this section you will learn how to edit a SampleList and inject multiple samples from System Control.



Choose to open either a New SampleList or an existing SampleList from the File menu. The SampleList window for the open SampleList is displayed. It contains fields that are specific to the sampling device configured in the instrument. In this case, if a sampling device such as the 8400 AutoSampler is configured, the corresponding SampleList is used.



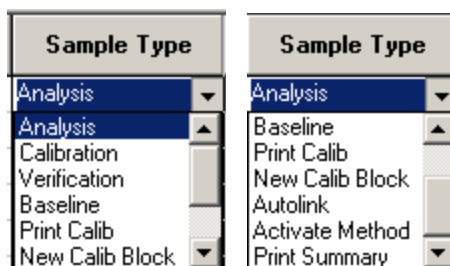
Size spreadsheet columns by dragging their border using the left mouse button. Right-click on the column headers for formatting options. When the table is scrolled to the right, the Sample Name column does not scroll so you can easily tell for which sample you are entering additional parameters.

Defining the Fields in the Table

The top of the SampleList window you see fields where you can enter information about the sample. The following define the fields and buttons in this dialog:

Sample Name Enter the sample name here. The final name of the file depends on settings accessed by clicking the **Data Files** button. If only %s is listed in the Data Files dialog the sample name will be as you entered it here.

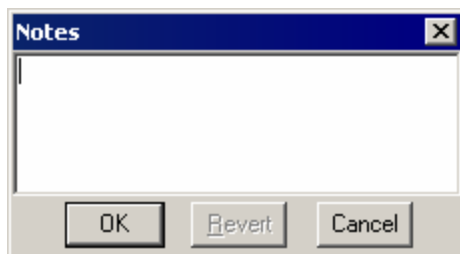
Sample Type There are several choices available from the Sample Type drop-down list box. Use the scroll bar to see all the options.



Cal. level If you designate the sample type as calibration, you can designate which calibration level you are injecting. You can enter up to ten levels.

Inj. Specify here the number of injections of this sample. You may make up to nine replicate injections at each calibration level.

Injection Notes Enter notes about the sample.



AutoLink See the Specifying Data Handling Parameters section in this manual.

Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
0	1.0	Pos 1	1	0	1	1	none

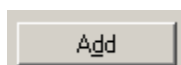
In the Vial, Injection Volume, and Injectors Used fields, enter information about the sample. The fields beginning with Amt. Std. are used only for data files acquired with standard GC detectors.

Inject the Sample using the Method:

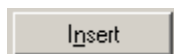
C:\Varian\WS\Coltest_External.mth Browse...

Select the Method to use for the run. Click the **Browse** key to select a file using Windows Explorer.

What the Buttons Do



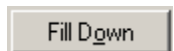
Adds a new line to the end of the list.



Inserts a new line before the currently active line.



Deletes the currently active line.



When sections of columns or entire lines are highlighted, the information in the top highlighted row is repeated to the lowest selected row.



Add several similar lines to the Sample list, You can then enter the common information in the dialog box.

Add Lines to 8400 SampleList

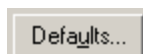
Sample Name	Sample Type	Cal level	Inj	Injection Notes	AutoLink	Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Name	Analysis		1	none	none	0	1.0	Pos 1	1	0	1	1	none

Number of Lines to Add:

☒ Number Sample Names from: ☒ Number Vials from:

Add Insert Cancel

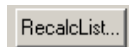
To sequentially number the Sample names, check the box and enter the starting number and the number of entries to add. The Sample Names will have these numbers appended to them.



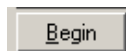
Change the default entries in the table above, such as Injection Volume, Injector Used, etc. After defining new defaults, click the **Save** button to activate the new defaults.



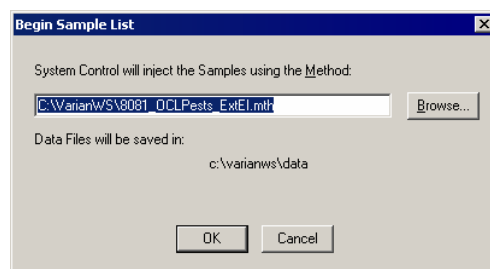
Change the location and name of the Data Files.



Select to automatically generate a Recalc list for with single sample acquisitions.



Click and a prompt for the Method opens.

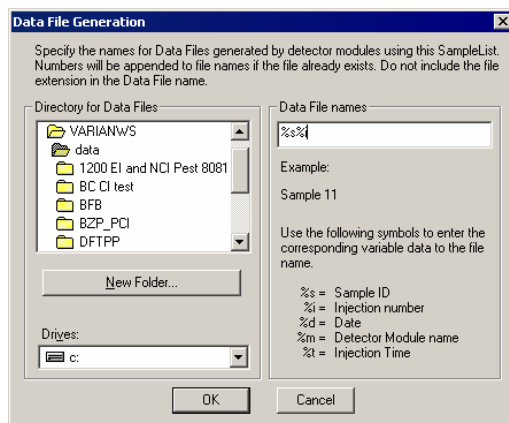


Enter the method name directly or Browse for the Method file. Click **OK**, the Method downloads to the 3800 GC, and the run begins. If using a manual injector or a sampling device not controlled by the Varian MS Workstation, start the device manually.

Specifying the Data File Name and Path

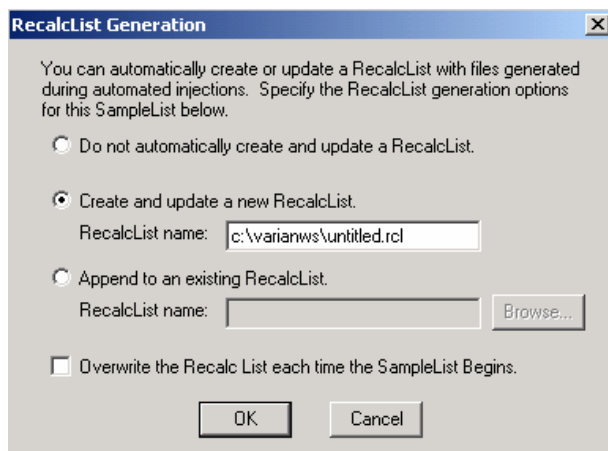
Data File names can be up to 255 characters long. Sample ID, injection date, module name, and injection number can be embedded in the file name making the Data File name correlate with each sample injection. When you click on the **Data File** button, the Data Files Generation dialog box is displayed. Select the path and the file name specification for the data file. The left side of the Data File Generation dialog box has the drive letter and subdirectory (path) where the data files stored.

Use the right side to create a file name specification. Combine text entry with the “%” variable symbols to specify file names that contain sample injection specific information. An example of the file name is dynamically updated as the file name specification is typed. This feature makes it easy to see how a Data File created with this file name specification will appear.



Specifying a RecalcList

You can choose to create a new RecalcList, append to an existing RecalcList, or not create nor update a RecalcList. To select the RecalcList option that you want, click on the **RecalcList** button. The RecalcList Generation dialog box is displayed.



If you choose to create a new RecalcList, this automatically generated RecalcList will not overwrite an existing RecalcList. If a RecalcList with the same file name exists, the newly created RecalcList will have number appended to its file name to make it unique and to prevent the older RecalcList from being overwritten. Alternatively, you may select the checkbox to overwrite the RecalcList each time the SampleList begins.

Changing Default SampleList Entries

When you add a new row into a SampleList, default values are used for each cell. To change the default values, click on the **Default** button in the open SampleList window. The following dialog box will be displayed. Enter the desired default values and click on **Save**.

Sample Name	Sample Type	Cal. level	Inj.	Injection Notes	AutoLink	Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unit Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Name	Analysis		1	none	none	0	1.0	Pos 1	1	0	1	1	none

Save Cancel

Monitoring the Status of Runs

Monitor the status of a run in the instrument window.



The module status is shown in the status and control windows and on the Toolbar.

Keypad Display

For information on “Status and Control” see page 59, “MS Method” see page 65, and “Operating Conditions” see page 69. For fields in the keypad display, go to the relevant section in “System Control” on page 17.

Monitoring the Chromatogram

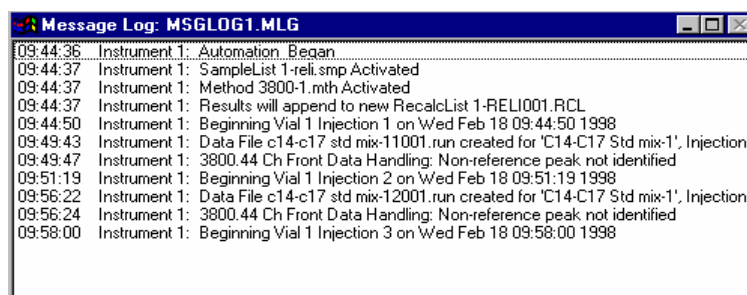
For information on chromatogram display options, go to “Chromatogram Toolbar” on page 29 and “Configuring the Real-Time Chromatogram Display” on page 34 in the System Control section.

Monitoring the Mass Spectra

For information on spectrum display options, go to “Spectrum Toolbar” on page 32 and “Configuring the Real-Time Chromatogram Display” on page 34 in the System Control section.

Message Log

Automation actions and errors are logged in the Message Log; the latest line of this log is shown at the bottom of the screen. When you double-click on the status bar at the bottom of the instrument window, the entire Message Log window is displayed.



The chromatogram is displayed as it is acquired.

Saving SampleLists for Later Use

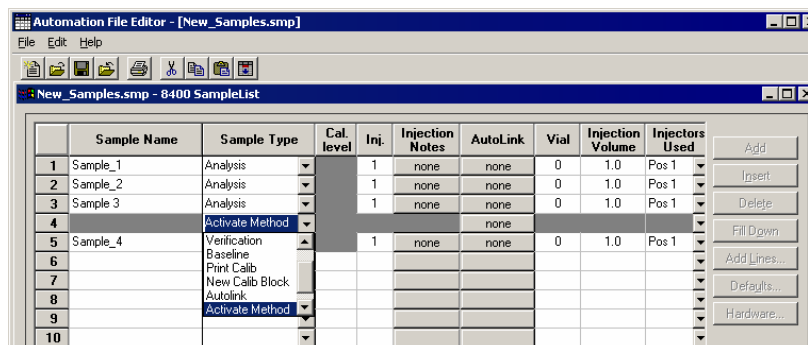
When you make changes to the open SampleList, the changes are automatically saved to the SampleList file and will be used for the automated runs that are in progress. If you want to edit a SampleList other than the open SampleList, use the offline Automation File Editor application described in the next section of this manual.

Using More Than One Method for Injections

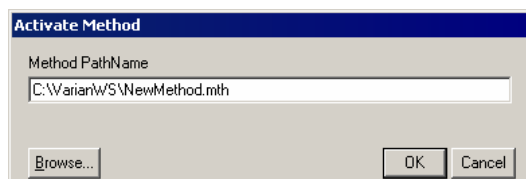
The Varian MS Workstation allows you to perform automated injections using more than one Method. There are two ways in which this can be accomplished. The first is by changing the active Method from within the SampleList. The second is by using a Sequence.

Changing the Method in the SampleList

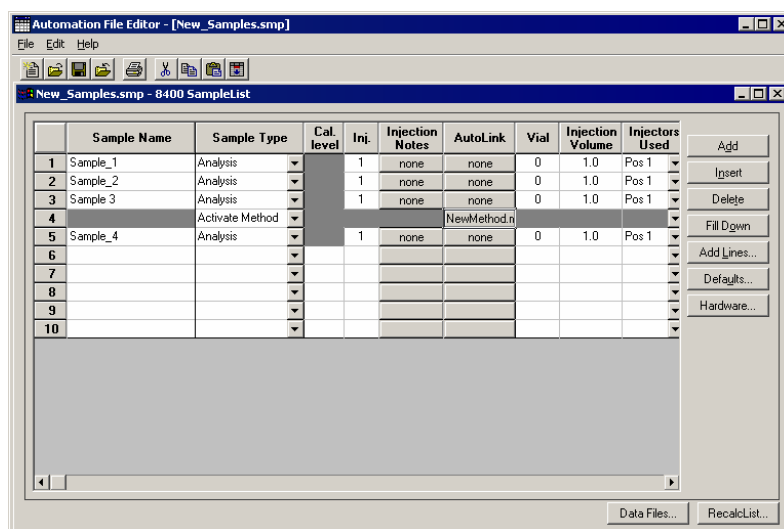
Change the Method used during injections by activating a Method in a SampleList row.



1. Select Activate Method from the Sample Type cell.
2. Click on the **AutoLink** button in the row that you are working. The Activate Method dialog box is displayed.



3. Enter the name of the Method or use **Browse** to pick from a list of files.

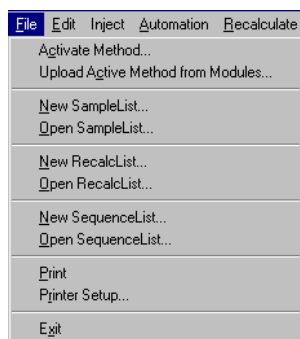


When this line is encountered during automated injections, the specified Method is activated.

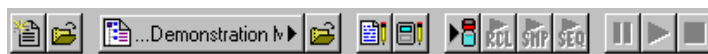
You may specify any number of Methods to be used in the SampleList.

Using the Sequence Window

The Sequence window allows you to specify multiple Methods and SampleLists to be processed during automation.



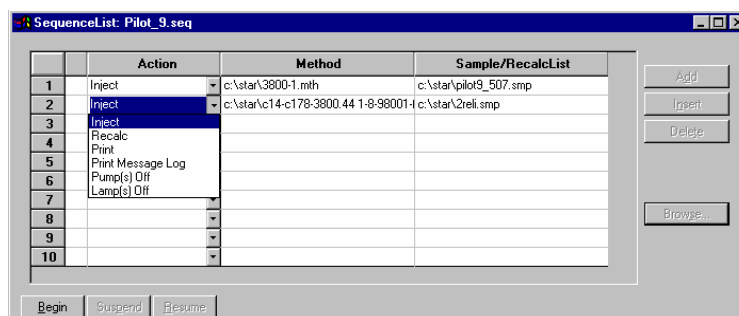
Choose to open either a New SequenceList or an existing SequenceList from the File menu...



...or by clicking on the **New** or **Open Automation File** button on the toolbar



The Sequence window for the open Sequence is displayed.



Click **Add** to add lines to Sequence List. You may enter any number of Sequence lines.

Choose the action to be done in that step of the Sequence from the drop down box.

Browse for a Method and SampleList file in the active cells.

Press **Begin** to start the automation.

Automation File Editor

Overview

The Automation File Editor is used for creating and editing SampleLists, RecalcLists, and SequenceLists outside of the Varian MS Workstation System Control application. The off-line Automation File Editor allows access to these files without disrupting automated runs that may be occurring in System Control.

NOTE: SampleLists and Sequences that are active and running in System Control cannot be accessed simultaneously in the off-line Automation File Editor.

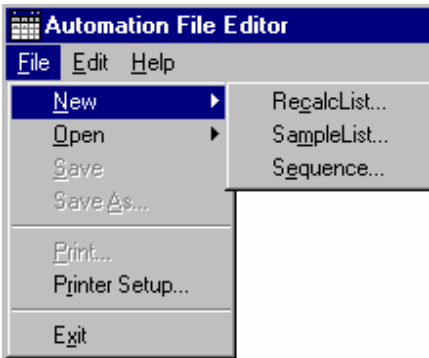
Accessing the Automation File Editor



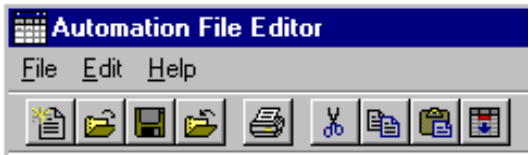
Click on the **Automation File Editor** icon on the Workstation Toolbar

The Automation File Editor is displayed.

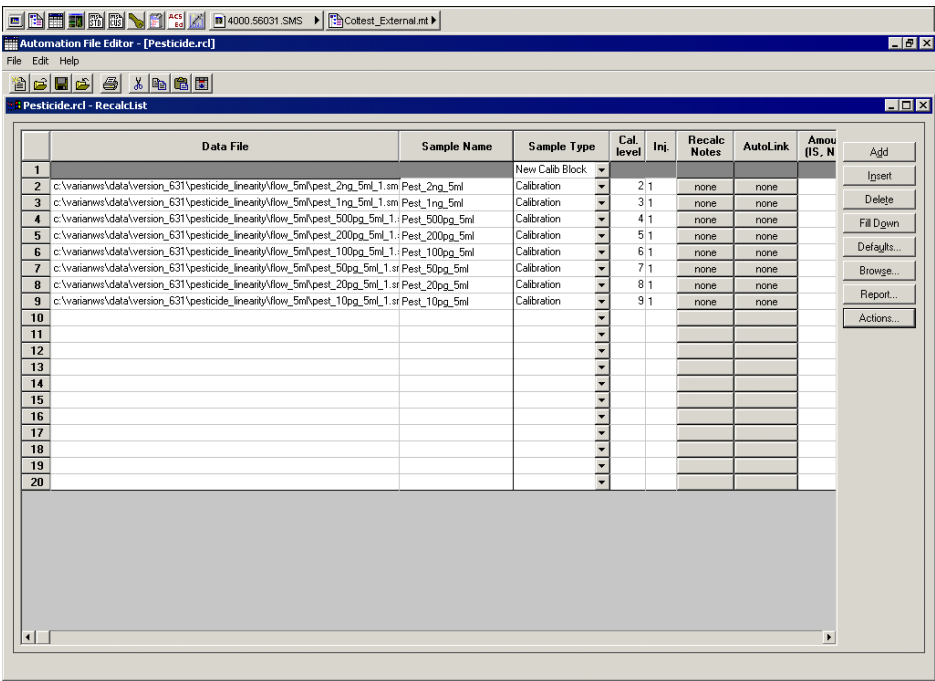
Creating or Editing a RecalcList



Either choose a New RecalcList or Open an existing RecalcList from the File menu.



The RecalcList window for the open RecalcList is displayed. It contains most of the same fields contained in the SampleList. Where the SampleList may contain AutoSampler and sample specific data handling information, the RecalcList contains the Data File name and data file specific data handling information. The fields that are common to both the RecalcList and the SampleList are described in the sections following the creation of the SampleList.



Recalc
Notes

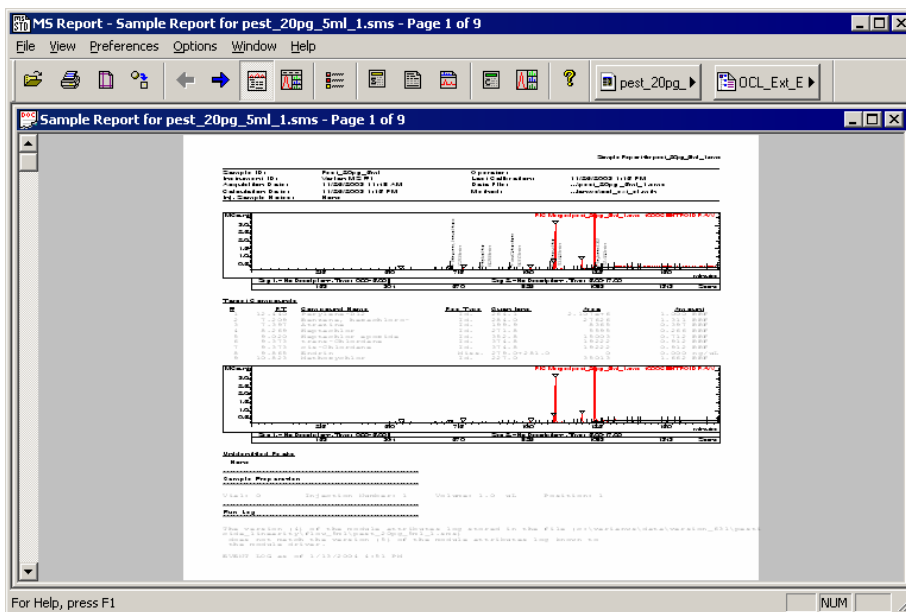
Enter notes about the Recalculation of the Data File.

AutoLink

Enter post calculation operations to be performed.

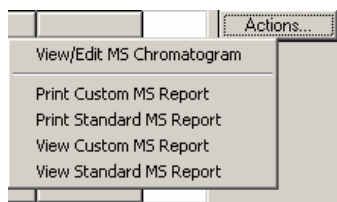
Report...

Press the **Report** button to generate a report for the currently selected data file. Note that if a data file is not highlighted in the Data File column, no report will be generated.

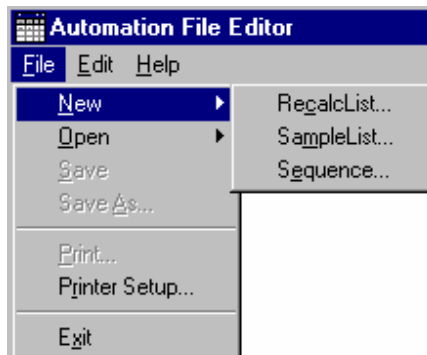


Actions...

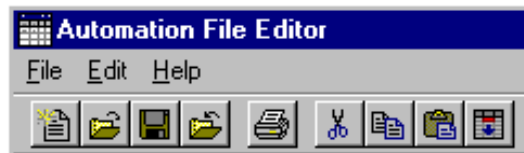
If a report was generated with the **Report** button, clicking on the **Actions** button shows a selection of actions that may be done.



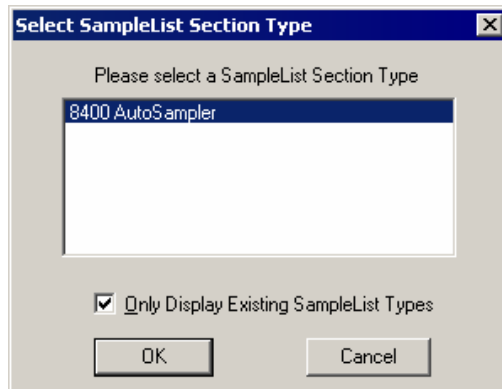
Creating or Editing a SampleList



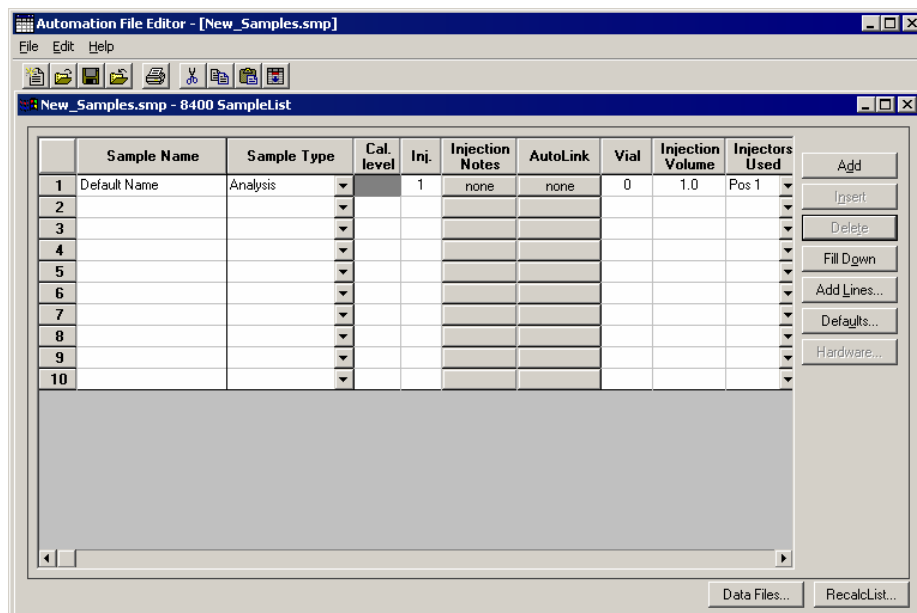
Either choose a New SampleList or Open an existing SampleList from the File menu



Select a list type:



The SampleList window for the open SampleList type is displayed.



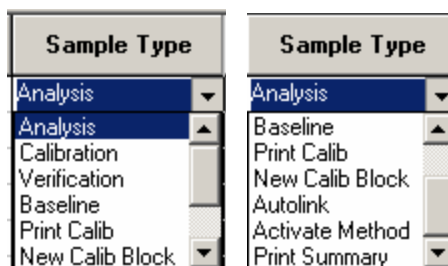
Spreadsheet columns can be sized by dragging their border using the left mouse button. Right-click on the column headers for formatting options. When the table is scrolled to the right, the Sample Name column does not scroll so you can easily tell for which sample you are entering additional parameters.

Defining the Fields in the Table

Along the top of the Inject Single Sample window you see fields where you can enter information about the sample. Here is a quick definition of the various fields and buttons in this dialog:

Sample Name Enter the sample name here. The final name of the file depends on settings accessed by clicking the **Data Files** button. If only %s is listed in the Data Files dialog the sample name will be as you entered it here.

Sample Type There are several choices available from the Sample Type drop-down list box. Use the scroll bar to see all the options.



Cal. level If you designate the sample type as calibration, you can designate which calibration level you are injecting. You can enter up to ten levels.

Inj. Specify here the number of injections of this sample. You may make up to nine replicate injections at each calibration level.

Injection Notes

Enter notes about the sample.

A dialog box titled "Notes" with a close button (X) in the top right corner. Below the title bar is a large empty text area for entering notes. At the bottom are three buttons: "OK", "Revert", and "Cancel".

AutoLink

See the Specifying Data Handling Parameters section in this manual.

Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
0	1.0	Pos 1	1	0	1	1	none

In the Vial, Injection Volume, and Injectors Used fields, enter information about the sample. The fields beginning with Amt. Std. are only used for data files acquired with standard GC detectors.

A dialog box titled "Inject the Sample using the Method:". It contains a text field with the path "C:\Varian\WS\Coltest_External.mth" and a "Browse..." button to the right.

Select the Method to use for the run. Click the **Browse** key to select a file using Windows Explorer.

What the Buttons Do

A button labeled "Add".

Adds a new line to the end of the list.

A button labeled "Insert".

Inserts a new line before the currently active line.

A button labeled "Delete".

Deletes the currently active line

A button labeled "Fill Down".

When sections of columns or entire lines are highlighted, the information in the top highlighted row is repeated to the lowest selected row.

A button labeled "Add Lines...".

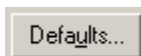
If you need to add several similar lines to the Sample list, click on the **Add Lines** button. You can then enter the common information in the dialog box.

A dialog box titled "Add Lines to 8400 SampleList". It contains a table with the same headers as the one above. Below the table are input fields for "Number of Lines to Add:" (set to 10), "Number Sample Names from:" (set to 1), and "Number Vials from:" (set to 0). At the bottom are "Add", "Insert", and "Cancel" buttons.

Sample Name	Sample Type	Cal. level	Inj.	Injection Notes	AutoLink	Vial	Injection Volume	Injectors Used	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Name	Analysis		1	none	none	0	1.0	Pos 1	1	0	1	1	none

For sequentially numbered Sample names, check the box and enter the starting

number and the number of entries to add to the SampleList. The Sample Names will have these numbers appended to them.



Click here to change the default entries in the table above, such as Injection Volume, Injectors Used, etc. After defining new defaults, click the **Save** button to activate the new defaults.



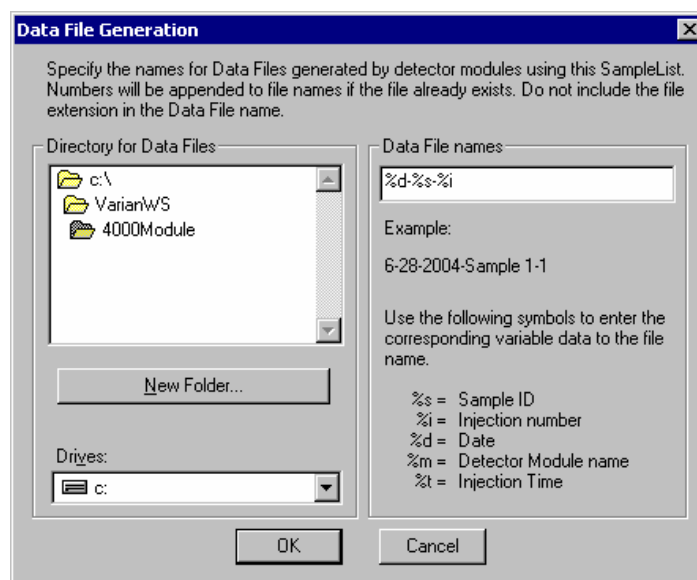
Change the location and name of the Data Files.



Click here to select whether to generate automatically a RecalcList associated with single sample acquisitions.

Specifying the Data File Name and Path

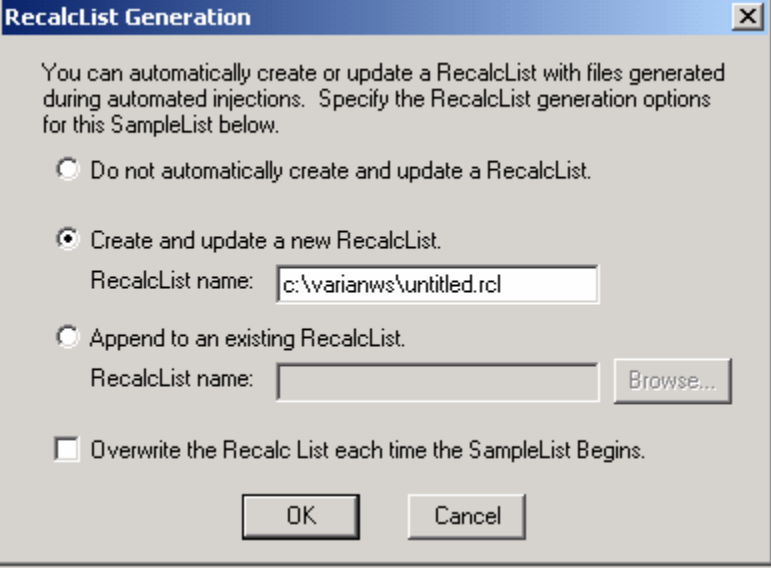
Data File names can be up to 255 characters long. Sample ID, injection date, module name, and injection number can be embedded in the file name making the Data File name correlate with each sample injection. When you click on the **Data File** button, the Data Files Generation dialog box is displayed. This dialog box allows you to select the path and the file name specification for the data file. The left side of the Data File Generation dialog box allows you to select the drive letter and subdirectory (path) where the data files are to be stored.



The right side of this dialog box allows you to create a file name specification. You can combine text entry with the “%” variable symbols shown to specify file names that contain sample injection specific information. An example of the file name is dynamically updated as you type in the file name specification. This feature makes it easy to see how a Data File created with this file name specification will appear.

Specifying a RecalcList

Create a new RecalcList, append to an existing RecalcList, or not create nor update a RecalcList. To select the RecalcList option that you want, click the **RecalcList** button. The RecalcList Generation dialog box opens.



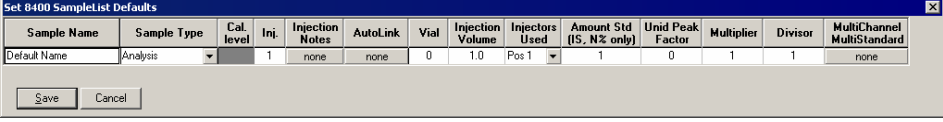
The RecalcList Generation dialog box contains the following elements:

- Instructional text: "You can automatically create or update a RecalcList with files generated during automated injections. Specify the RecalcList generation options for this SampleList below."
- Three radio buttons:
 - ☐ Do not automatically create and update a RecalcList.
 - ☒ Create and update a new RecalcList.
 - RecalcList name:
 - ☐ Append to an existing RecalcList.
 - RecalcList name:
- ☐ Overwrite the Recalc List each time the SampleList Begins.
- OK and Cancel buttons at the bottom.

If you choose to create a new RecalcList, this automatically generated RecalcList will not overwrite an existing RecalcList. If a RecalcList with the same file name exists, the newly created RecalcList will have number appended to its file name to make it unique and to prevent the older RecalcList from being overwritten.

Changing Default SampleList Entries

When you add a new row into a SampleList, default values are used for each cell. To change the default values, click on the **Default** button in the open SampleList window. The following dialog box will be displayed. Enter the desired default values and click on **Save**.



The Set 8400 SampleList Defaults dialog box features a table with the following columns and values:

Sample Name	Sample Type	Cal. level	Inj.	Injection Notes	AutoLink	Vial	Injection Volume	Injectors Used	Amount Std (IS, M% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Name	Analysis		1	none	none	0	1.0	Pos 1	1	0	1	1	none

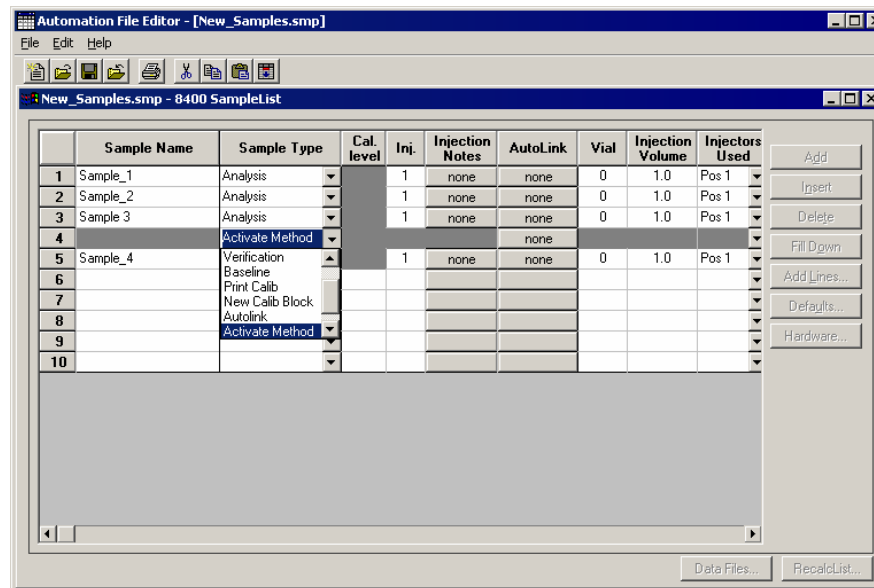
Save Cancel

Using More Than One Method for Injections

The Varian MS Workstation allows you to perform automated injections using more than one Method. There are two ways in which this can be accomplished. The first is by changing the active Method from within the SampleList. The second is by using a Sequence.

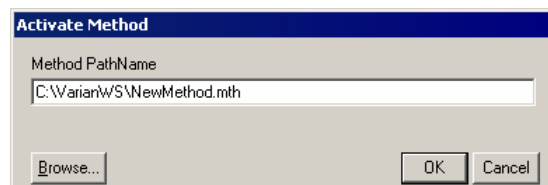
Changing the Method in the SampleList

You may change the Method used during injections by activating a Method in a SampleList row.



Select **Activate Method** from the Sample Type cell.

Click on the **AutoLink** button in the row that you are working. The Activate Method dialog box is displayed.

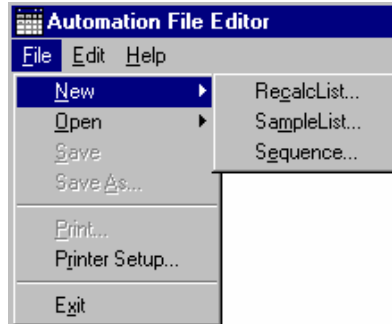


Enter the name of the Method or use the **Browse** button to pick the Method from a list of files.

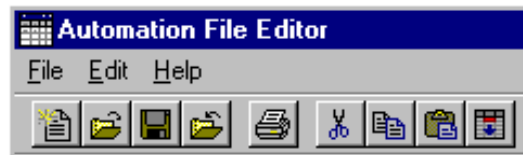
When this line is encountered during automated injections, the specified Method is activated.

You may specify any number of Methods to be used in the SampleList.

Creating or Editing a Sequence

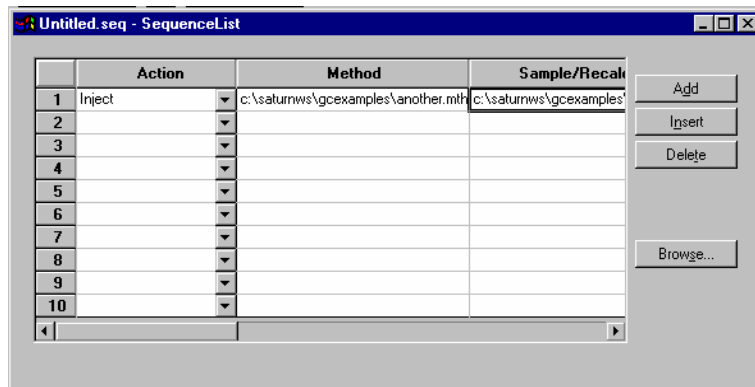


Click **New Sequence** or **Open** to open an existing Sequence from the File menu...



...or click **New** or **Open Automation File** button on the toolbar.

The Sequence window opens.



Click **Add** to add lines to the SequenceList. You may enter any number of Sequence lines.

Choose the action for the selected step of the Sequence from the drop down box.

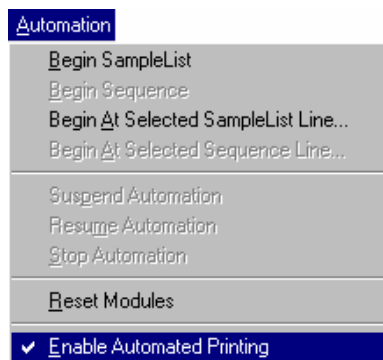
Browse for a Method and SampleList file in the active cells.

Automated MS Report Generation

Overview

Once you have added Report sections to your Method, automated reports can be generated from System Control after each injection, after Recalculations, or by the use of any of the Print actions in the Sequence window.

You may choose to disable automated Report printing during the course of an automated sequence of injections or Recalculations. You can do this by disabling automated printing from the Automation menu in the Instrument window.



When this item is not checked, report printing is disabled. ASCII file generation is still performed if specified in the Report Method section.

Disabling automated printing is analogous to disconnecting the printer—automation continues but no reports are printed. This is useful if you are about to run out of printer paper but do not wish to suspend automation.

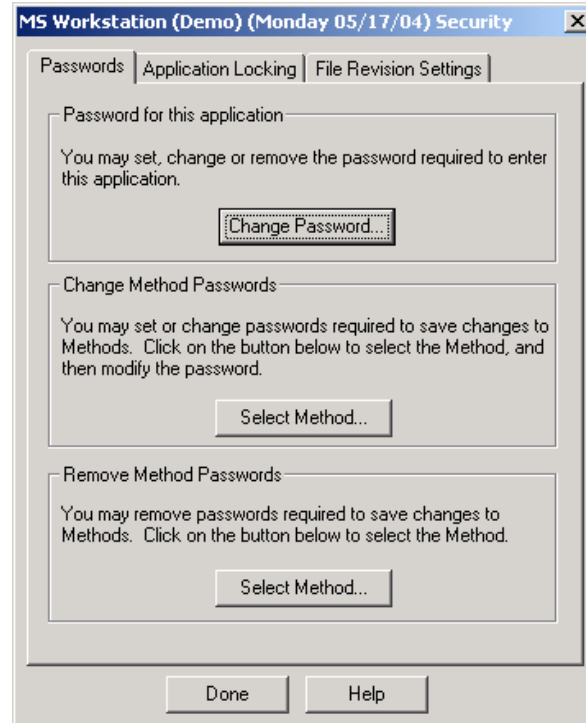
Security Administration

Overview

The Security Administration window contains three window tabs that can be used to select the categories of security parameters that can be specified for Varian MS Workstation applications. Click on the category names below for more information on each:

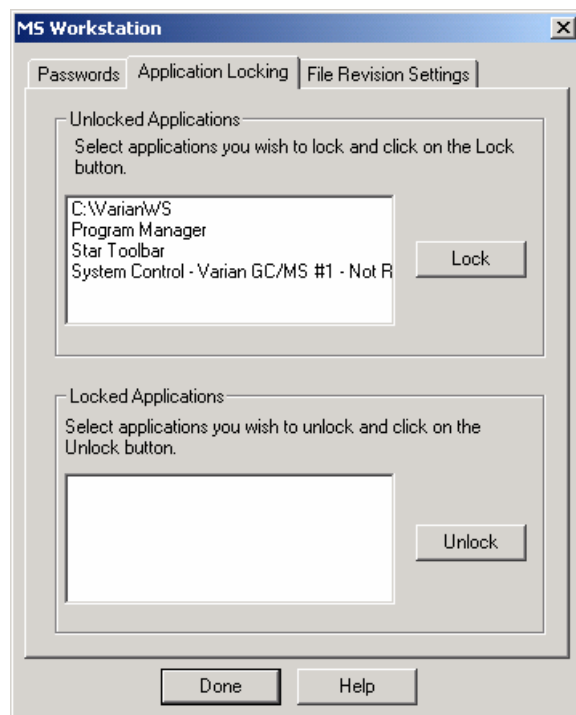
- Passwords
- Application Locking
- File Revision Settings

Passwords



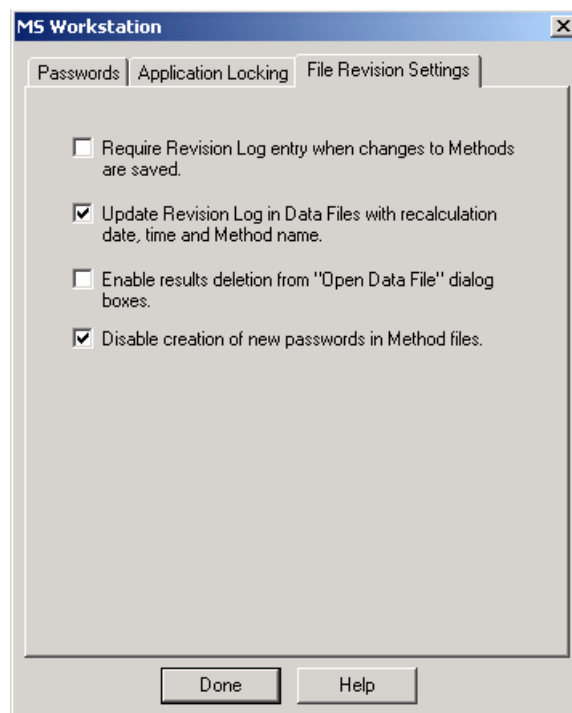
Item	Description
Password for this application	Allows you to specify a password required to enter the Security Administration application. <i>Note that there is no way to recover this password if it is lost, so it is recommended that you document the password in a secure location.</i>
Change Method Passwords	Method passwords can be added, changed and removed from the Security Administration application. Click on the Select Method button to browse for and select the desired Method. After the Method has been selected, you are either prompted for a new password (if no previous password exists for the Method), or are prompted for the old and new password (if the Method already contains a password). <i>Note that if creation of new passwords is disabled in the File Revision tab, you will not be able to add a new password to a Method that does not already contain one.</i> Method passwords can also be added and changed in the Method Builder and Interactive Graphics applications.
Remove Method Passwords	Method passwords can be removed by the Security Administration application without entering the existing password. This is useful if the password for a Method has been lost. Click on the Select Method button, select the desired Method, and the password is removed.

Application Locking



Item	Description
Unlocked Applications	This list box shows all top level windows currently running that are not locked. You may select any number of them and click on the Lock button. When you do, they are moved to the Locked Applications list box and their windows are disabled (they will not respond to mouse or keyboard input). Note that applications that are locked by the Security Administration application remain locked even after the Security Administration application is closed.
Locked Applications	This list box shows all top level windows that are currently running and are locked. You may select any number of them and click on the Unlock button. When you do, they are moved to the Unlocked Applications list box and their windows are enabled (they respond to mouse or keyboard input).

File Revision Settings



Item	Description
Require Revision Log entry when changes to Methods are saved.	When checked, the user is prompted for a description of changes when a Method file is altered and saved. This Revision Log appears in the Notes portion of the File Open dialog box, is listed in the Method Builder application window when the Method is open, and is included in the Method printout.
Update Revision Log in Data Files with Recalculation date, time and Method name.	When checked, Data Files are updated with a time stamp and Method name when they are recalculated (either from System Control or from Interactive Graphics). The Data File Revision Log can be included in printed reports.
Enable results deletion from "Open Data File" dialog boxes.	When checked, a button appears in the Open Data File dialog box allowing results to be deleted from a specified channel of a Data File. Results deletion is logged in the Data File's Revision Log. This option only affects standard GC results. GC/MS results will not be deleted.
Disable creation of new passwords in Method files.	When checked, new passwords cannot be added to Methods. Methods already containing passwords will still prompt you for the password before changes are saved.

4000 MS Ionization Techniques

Choices of Ionization Mode

Choose from the following list to go to a specific topic on the particular ionization mode:

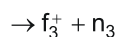
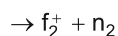
- “Internal Electron Ionization” on page 191
- “Internal Positive Chemical Ionization” on page 194
- “External Electron Ionization” on page 199
- “External Positive or Negative Chemical Ionization” on page 202
- “Hybrid Positive or Negative Chemical Ionization” on page 206

Internal Electron Ionization

About Internal Electron Ionization

Electron ionization is the most common mode of operation of the 4000 MS. All of the sample ionization, fragmentation, storage, and scanning steps occur within the confines of the ion trap. Electron ionization generates fragments that are specific to the compound, providing fingerprint spectra. It is, however, not selective and will show ions for all compounds present. EI is sensitive but the sensitivity can be improved by reducing the interfering ions using ion preparation techniques.

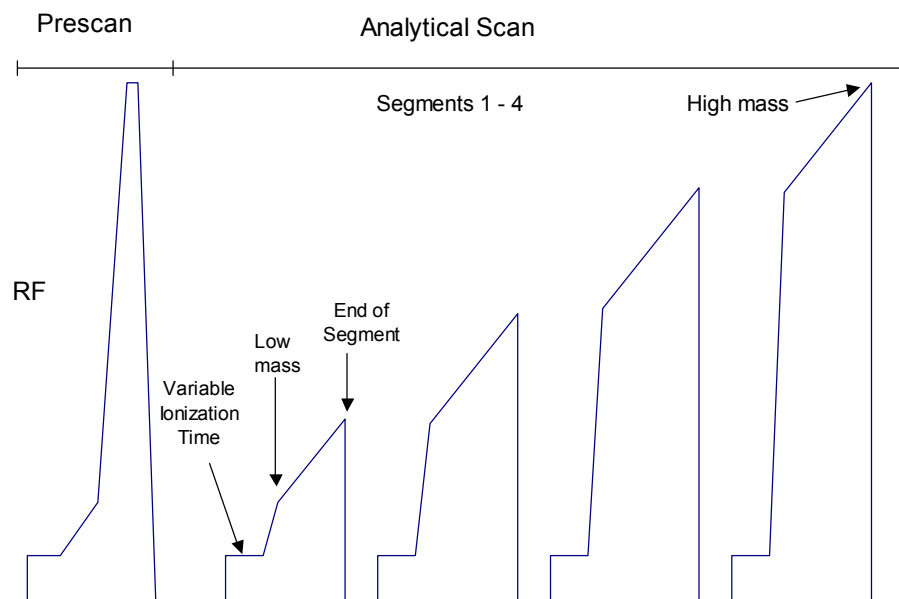
In the EI mode, electrons are gated into the ion trap cavity during the ionization period. These electrons collide with the neutral sample molecules A, removing an additional electron to create energetically-excited molecular ions A^{+*} . Some of the excited molecular ions equilibrate through collisions with helium but others undergo unimolecular decomposition to create various fragment ions, f_i^+ . This set of fragment ions, characteristic of the sample molecule, makes up the mass spectrum.



Internal EI Scan Function

In electron ionization mode, electrons emitted by a filament interact with sample molecules to form ions. For internal ionization mode, the ion formation occurs within the trap. For external ionization mode (electron & chemical), the ion formation occurs within the source with subsequent transfer of ions to the trap by a set of lenses. For both internal and external ionization, the ions are stored and stabilized in the ion trap cavity by an RF field applied to the ring electrode of the ion trap. The ion trap has a maximum storage capacity for ions, beyond which mass resolution and spectral quality deteriorate. The number of ions created is proportional to the ionization time, with more ions being produced the longer the ionization time. Automatic Gain Control (AGC) controls the ionization time in order to maintain the number of ions in the trap at the optimum level.

Four modes of operation (Internal EI, External EI, External CI, and Hybrid CI) use a similar scanning sequence involving Automatic Gain Control (AGC) to scan ions out of the trap. The AGC scan function consists of a prescan and up to six scan segments. The prescan uses a fast scan rate and the number of ions detected in the prescan is used to calculate the ionization time for the succeeding scan segments. The figure below illustrates the AGC prescan and four scan segments corresponding to the following four portions of the mass range (default values): 10u to 99u, 100u to 249u, 250u to 399u, and 400u to 1000u. One scan involves one complete ramping of the RF voltage over the mass range selected by the user. The RF voltage is ramped at a constant rate of approximately 5000 daltons/sec for normal scan and 10000 daltons/sec for fast scan. One complete scan at this fixed rate produces a microscan.



The AGC Scan Function

Scan Segment Setup Period

The prescan is followed by the first of the scan segments. Each segment is divided into two parts: a setup period and a scanning period. Each setup period is subdivided into the following four stages:

First stage: The RF voltage is set to near 0V for 2 milliseconds. This setting allows the RF voltage generator circuit to stabilize while clearing the trap cavity of all ions.

Second stage: The RF voltage is set to, and stabilizes at, the storage voltage selected to store, or trap, all ions of interest in the ion trap cavity.

Third stage: Energetic electrons from the heated filament are accelerated into the ion trap, where they interact with sample molecules to form sample ions. For external ionization mode, the ion formation occurs within the source with subsequent transfer of ions to the trap by a set of lenses.

The newly formed sample ions are then permitted to stabilize in their orbits at the storage voltage. This stabilization period is referred to as the cool time. The length of this period is fixed.

Fourth stage: The dynode and electron multiplier are made ready, and the RF voltage is ramped quickly from the storage voltage to a value appropriate to begin the mass scanning of the segment.

Scan Segment Scanning Period

During the scanning period the RF voltage is steadily increased, causing ions of increasing m/z to be ejected from the trap, to be detected by the dynode and electron multiplier.

The AGC software automatically selects an ionization time from 10 μsec to 65 μsec for each of the scan segments of the microscan, depending on the amount of analyte in the ion trap and the tuning parameters. An estimate of the number of ions formed in the ion trap is provided by a prescan with a varying ionization period (10 to 2500 μsec). For low levels (e.g., for baseline or small GC peaks) the maximum ionization time of 65 milliseconds is selected, which offers maximum sensitivity. As the amount of analyte increases, the ionization time is decreased automatically in order to prevent overloading of the ion trap with ions.

The ion signals in each microscan are scaled automatically to compensate for the variation in ionization time. Microscans may be averaged to improve the spectral quality.

To optimize trapping of the ions of interest, a notched waveform is applied during ionization for internal EI mode. For external EI & external CI mode, a notched waveform is applied after the ions are trapped. The ions are analyzed by applying a radio frequency (RF) voltage to the ring electrode encircling the trap cavity. As the voltage increases on the ring electrode, ions are sequentially ejected from the trap according to their mass-to-charge ratio. Supplemental waveform voltages are applied to the end cap electrodes during the analysis to improve mass resolution and mass axis stability. The ions scanned out of the trap strike the conversion dynode, ejecting electrons which are repelled to the electron multiplier. The conversion dynode is held at -10 KV and the voltage on the multiplier is adjusted to provide an enhancement of $\sim 10^5$. The signal is then sent through an integrator to collect an intensity reading for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

Ion Preparation Options

The 4000 MS can use a combination of waveforms applied to ion trap electrodes to isolate or remove specific ions once they have been formed and are stored in the trap.

Options like Selected Ion Storage (SIS) and MS/MS can be performed on the ions stored in the ion trap before mass analysis takes place. In MS/MS a parent ion is isolated and then dissociated by energetic collisions with helium buffer gas to form product ions. In SIS, resonant waveforms are applied to eject unwanted ions within the stored mass range and fill the trap only with ions in the mass range(s) of interest.

The Internal configuration can have SIS, MS/MS, MSⁿ, and MRM as ion preparation options. SIS is included with all instruments, while MS/MS, MSⁿ, and MRM are available when the MS/MS option is installed.

Internal Positive Chemical Ionization

Chemical ionization (CI) provides mass spectral data that complement electron ionization (EI) data for the analysis of complex compounds. The generation of ions in chemical ionization is a two-step process. A CI reagent gas is introduced into the ion trap analyzer. The reagent gas is ionized by EI. Then, the sample molecules are ionized by ion-molecule reactions with the reagent gas ions.

CI is a softer ionization technique than EI. That is, CI imparts less energy to the sample molecules than does EI. Thus, the ionized sample molecule undergoes less fragmentation, and an ion indicative of the molecular weight is more likely to be observed. In addition to molecular weight confirmation, CI mass spectra often provide other significant structural information that may not be available from EI mass spectra.

Adjusting CI Pressure

1. Connect a reservoir containing the chosen reagent, liquid or gas, to the CI Gas inlet block using the correct restrictor. Use the 50 mL/min CI restrictor for liquid CI reagents when you are in Internal configuration.
2. Open the CI needle valve 6-7 turns counterclockwise.
3. Open the CI gas valve solenoids by clicking on the CI Gas Control on the System Control page and allow the vapor flow from the reservoir to equilibrate. If, after allowing some time for equilibration, there is not enough CI gas entering the trap, further open the needle valve by turning it clockwise.
4. While observing the spectrum using Adjust CI Gas (checks and adjustments tab), turn the CI needle valve to increase or decrease the amount of reagent entering the trap until the resolution between M and M+1 just starts to degrade.

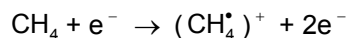
Forming Reagent Ions

In the first step, reagent gas ions are formed as the reagent gas is ionized by interaction with electrons emitted by the filament. The ion trap operates in a pulsed mode. The supply of reagent ions is created during the ionization pulse and consumed during the reaction period to form analyte ions.

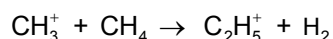
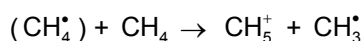
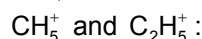
In internal mode, the CI reagent can be either liquid or gas. The most common reagents are methane, methanol, acetonitrile, and isobutane.

NOTE: Reagent ion formation can be a complex process. For example, when methane is used as the reagent gas, reagent gas ions are formed as follows:

First, methane is ionized to form two primary ions:



These primary ions then react very rapidly to form predominantly the secondary ions,

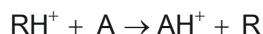


PCI Reactions in the Ion Trap

Four main types of reactions occur in positive chemical ionization. Symbolizing the sample analyte by A with mass M and the neutral reagent by R, these are:

Proton Transfer

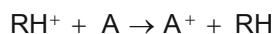
The reagent ion donates a proton to the sample molecule to produce a protonated analyte, sometimes called a pseudomolecular ion with mass M+1.



This is the most commonly-recognized PCI reaction. Note that the AH^+ ion may fragment to lower-mass ions because of excess energy deposited during the PCI reaction.

Charge Transfer

The CI reagent may transfer only charge to the analyte molecule by electron transfer to the reagent.



Adduct Ion Formation

The CI reagent ion may bond in its entirety to the sample molecule to create an adduct ion.



Hydride Abstraction

Some reagent ions have a tendency to remove a hydrogen atom to create an analyte product ion that is still positively-charged but having a weight of M-1.



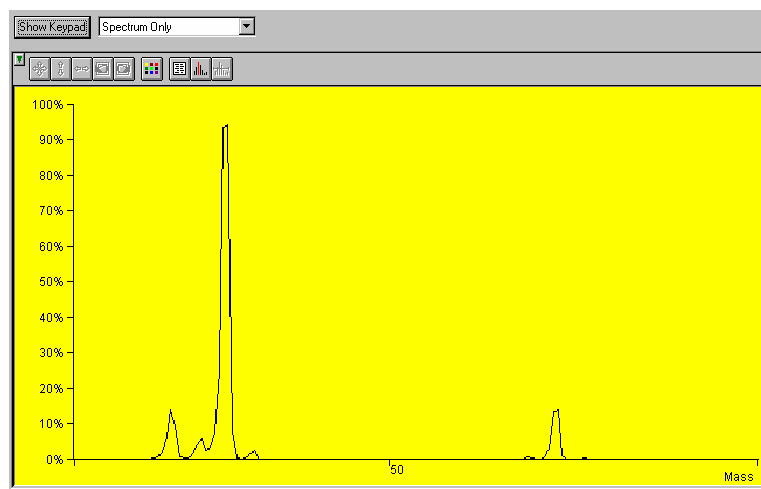
Recommended Internal PCI Parameters

PCI with CH₄

The pressure of methane is adjusted so the ratio of the peak heights at mass 17 (CH₅⁺) to mass 16 (CH₄⁺) is about 10:1; the ratio of the peak heights at mass 17 to 29 (C₂H₅⁺) is about 1:1; and mass 41 (C₃H₅⁺) is visible.

Reagent Gas	Methane
Maximum Ionization Time (μsec)	2000
Maximum Reaction Time (μsec)	60
CI Storage Level (m/z)	13
CI Background Mass (m/z)	45
Ejection Amplitude (v)	9.0

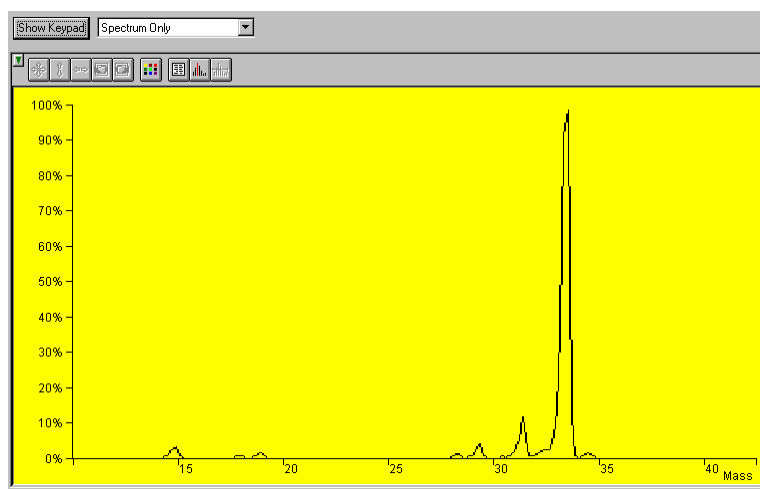
PCI with CH₃CN



Properly Adjusted Acetonitrile Reagent Spectrum

Reagent Liquid	Acetonitrile
Maximum Ionization Time (μsec)	2000
Maximum Reaction Time (μsec)	40
CI Storage Level (m/z)	19
Background Mass (m/z)	65
Ejection Amplitude (v)	15
Target TIC	5000
Prescan Ion Time (μsec)	100

PCI with CH₃OH



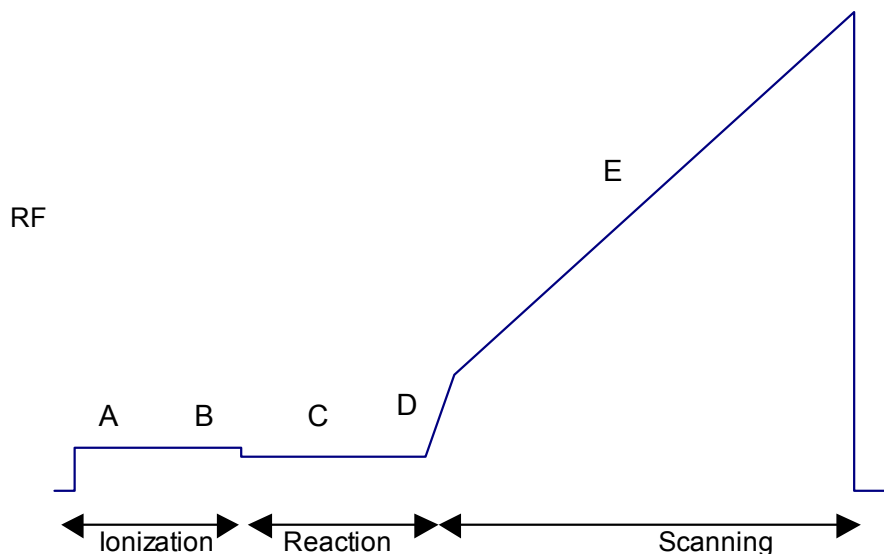
Properly Adjusted Methanol Reagent Spectrum

Reagent Liquid	Methanol
Maximum Ionization Time (μsec)	2000
Maximum Reaction Time (μsec)	40
CI Storage Level (m/z)	19
Background Mass (m/z)	55
Ejection Amplitude (v)	15
Target TIC	5000
Prescan Ion Time (μsec)	100

Internal PCI Scan Function

El space charge control uses the integrated ion current from a prescan to calculate the analytical scan ionization time. By contrast, Internal CI space charge control does not use a prescan to measure the charge formed. Internal CI uses a portion of the ions detected during the previous analytical scan to calculate the ionization and reaction times for the next analytical scan.

The following figure shows the scan function for internal chemical ionization.



Internal CI Scan Function

- A Reagent ionization, time determined by the previous scan
- B Ejection of unwanted ions (i.e., non-reagent ions) using waveform
- C Reaction of reagent ions with sample molecules, time determined from previous scan
- D Ejection of ions with masses lower than the start mass of scan range
- E Acquisition of CI mass spectrum

Chemical ionization occurs by forming reagent ions by electron impact. These reagent ions then react with the sample to form product ions by ion-molecule reactions. The ionization and reaction storage RF can be set at the same level or different levels. Unwanted sample ions formed by electron ionization during the formation of the reagent ions are ejected by a broadband waveform prior to the chemical ionization step.

The ion trap operates in a pulsed mode. The supply of reagent ions is created during the ionization pulse and consumed during the reaction period to form analyte ions. The number of analyte ions depends on the concentration of the analyte, the initial reagent ion intensity, and the reaction time.

Because the spectral intensity is proportional to sample concentration and reaction time, linear calibration curves can be obtained.

During the internal CI scan, the following steps occur:

- The reagent gas is ionized for the length of time determined by the previous scan. (A)
- All ions with a mass greater than the reagent ions are ejected, i.e., all EI ions from the sample. (B)
- Reagent ions react with sample molecules to form sample ions. (The reaction time was determined by the previous scan.) (C)
- Reagent ions are ejected. (D)
- The CI mass spectrum is acquired for the sample ions. (E)

Ejection of all EI artifact ions above Reagent High Mass is accomplished by applying a broadband waveform between the ionization and reaction periods. It should be noted that fragmentation can still be observed in CI spectra due to energy imparted when the proton is transferred from the reagent ion to the sample molecule.

Scanning Ions to Collect Mass Spectra

The scanning process for chemical ionization is similar to that for electron ionization. After ionization, trapping, and ion preparation steps, ions are scanned out to the conversion dynode and electron multiplier. Mass scanning is implemented by increasing the RF voltage on the ring electrode; the mass spectrum is collected in order from low to high mass over the user-designated scan range. In positive mode, electrons are ejected from the conversion dynode held at -10000V and repelled to the electron multiplier. In negative mode, positive ions are ejected from the +10000V dynode and repelled toward the multiplier. The signal is enhanced by $\sim 10^5$ by the multiplier and sent through an integrator to collect an intensity for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

External Electron Ionization

Chemical Information for External EI

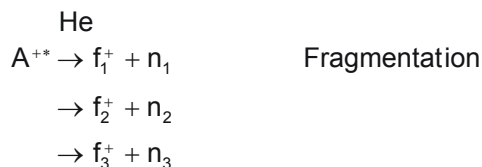
Ionization Energy

Positive ions are formed in electron ionization by the impact of energetic electrons (70 eV) on neutral molecules. The minimum energy necessary to remove an electron from the neutral to create a positive molecular ion is called the ***ionization energy*** or the *ionization potential* of the molecule. Thus for molecule A:



Unimolecular Decomposition

For most organic molecules, the ionization potential is between 10-11 electron volts (eV). If the energy of the electrons striking molecule A is higher than this by several eV, the collision of the electron with the target molecule may deposit enough internal energy to the molecular ion to cause fragmentation. If there are several chemical bonds in the molecule, different fragment ions and neutrals will be produced:



The minimum energy required to create a fragment ion is called the **appearance energy** or the *appearance potential* for the fragment ion. The fragmentation of molecular ions is on a very fast time scale compared to other processes in the mass spectrometer. In fact, it happens so fast that the molecular ion fragments before there is time to collide with other atoms, molecules or surfaces in the external ion source.

Energetics of the Trapping Processes

Ions are transferred to the ion trap by applying voltages of the opposite polarity to the three lenses between the ion source and the ion trap. Lens voltages are negative for PCI and positive for NCI. The voltages on the lenses are tuned in Auto Tune to optimize focusing of the ions toward the ion trap. The Trap DC offset voltage applied to the ion trap creates a potential well to trap all ions above a mass determined by the RF Storage Level.

The default RF storage level is 35u, so only ions above this m/z are stored in the ion trap. Several performance issues are related to the setting for the RF storage level. First, the efficiency for storing high mass ions (> 300u) seems to reach its peak for an RF storage level of about 45-50u. If this were the only consideration in the process the default value in the Method Builder would be set at this value. However in looking at the NIST library search results for a wide variety of compounds, it is found that using a lower RF storage level of 35u as the default gives better search results. Upon closer examination we found that fragile ions in the spectra of some compounds, particularly ions at low mass, can decompose due to energetic collisions with Helium buffer gas as they enter the ion trap from the external source. Whereas the RF storage level of 35u has been chosen as default and reduces or eliminates decomposition during ion transport for most ions, sometimes it is useful to use RF storage values as low as 20-30u in the lower mass segments to reduce these effects even further.

Link to NIST Database

The NIST Chemistry Web Book contains a wealth of information on organic thermochemistry, including the mass spectra found in the NIST 2002 Library:

- <http://webbook.nist.gov/chemistry/>

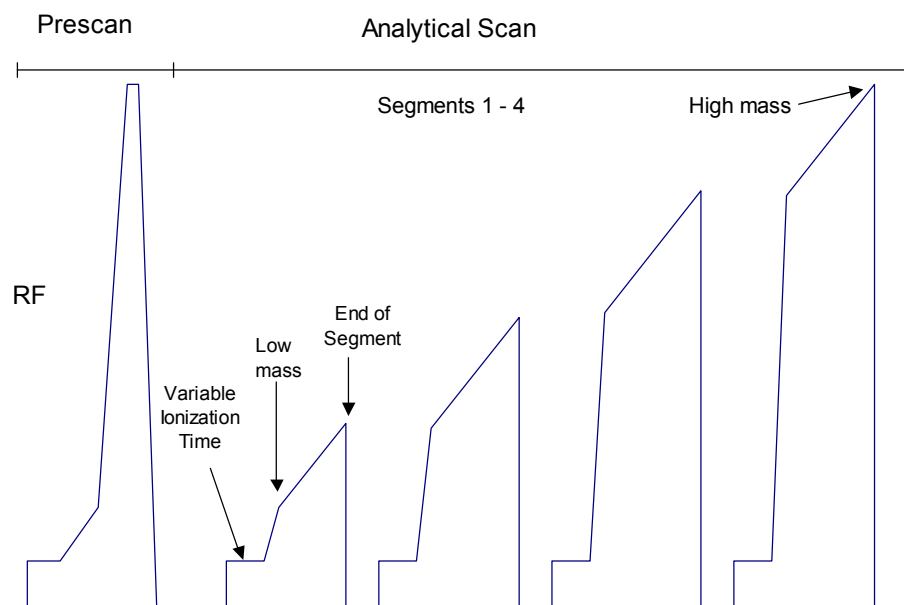
The subsection on Gas-Phase Ion Thermochemistry by Lias and Bartmess gives a detailed explanation of the gas phase energetics relevant to mass spectrometry.

- <http://webbook.nist.gov/chemistry/ion/>

External EI Scan Function with Automatic Gain Control (AGC)

In External EI mode, electrons emitted by a filament interact with sample molecules to form ions in the ion source where unimolecular fragmentation occurs immediately. The sample molecular and fragment ions are transferred to the ion trap by a set of lenses. The ions are stored and stabilized in the ion trap cavity by an RF field applied to the ring electrode of the ion trap. The ion trap has a maximum storage capacity, beyond which mass resolution and spectral quality deteriorate. The number of ions created is proportional to the ionization time, with more ions being produced the longer the ionization time. AGC controls the ionization time in order to maintain the number of ions in the trap at the optimum level.

Four modes of operation (Internal EI, External EI, External CI, and Hybrid CI) use a similar scanning sequence involving AGC to scan ions out of the trap. The AGC scan function consists of a prescan and up to six scan segments. The prescan uses a fast scan rate and the number of ions detected in the prescan is used to calculate the ionization time for the subsequent scan segments. The figure below illustrates the AGC prescan and four scan segments corresponding to the following four portions of the mass range (default values): 10 to 99 m/z, 100 to 249 m/z, 250 to 399 m/z, and 400 to 1000 m/z. One scan involves one complete ramping of the RF voltage over the mass range selected by the user. The RF voltage is ramped at a constant rate of approximately 5000 daltons/sec for normal scan and 10000 daltons/sec for fast scan. One complete scan at this fixed rate produces a microscan.



The AGC Scan Function

Scan Segment Setup Period

The prescan is followed by the first of the scan segments. Each segment is divided into two parts: a setup period and a scanning period. Each setup period is subdivided into the following four stages:

First stage: The RF voltage is set to near 0V for 2 milliseconds. This setting allows the RF voltage generator circuit to stabilize while clearing the trap cavity of all ions.

Second stage: The RF voltage is set to, and stabilizes at, the storage voltage selected to store, or trap, all ions of interest in the ion trap cavity.

Third stage: Energetic electrons from the heated filament are accelerated into the ion source to ionize sample molecules with subsequent transfer of these ions to the trap by a set of lenses.

The newly formed sample ions are then permitted to stabilize in their orbits at the storage voltage. This stabilization period is referred to as the cool time. The length of this period is fixed.

Fourth stage: The dynode and electron multiplier is made ready, and the RF voltage is ramped quickly from the storage voltage to a value appropriate to begin the mass scanning of the segment.

Scan Segment Scanning Period

During the scanning period the RF voltage is steadily increased, causing ions of increasing m/z to be ejected from the trap, to be detected by the dynode and electron multiplier.

The AGC software automatically selects an ionization time from 10 μsec to 65 μsec for each of the six scan segments of the microscan, depending on the amount of analyte in the ion trap and the tuning parameters. An estimate of the number of ions formed in the ion trap is provided by a prescan with a varying ionization period (10 to 2500 μsec). For low levels (e.g., for baseline or small GC peaks) the maximum ionization time of 65 milliseconds is selected, which offers maximum sensitivity. As the amount of analyte increases, the ionization time is decreased automatically in order to prevent overloading of the ion trap with ions.

The ion signals in each microscan are scaled automatically to compensate for the variation in ionization time. Microscans are averaged to improve the spectral quality.

To optimize trapping of the ions of interest for external EI & external CI mode, a notched waveform is applied after the ions are trapped. The ions are analyzed by applying a radio frequency (RF) voltage to the ring electrode encircling the trap cavity. As the voltage increases on the ring electrode, ions are sequentially ejected from the trap according to their mass-to-charge ratio. Supplemental waveform voltages are applied to the end cap electrodes during the analysis to improve mass resolution and mass axis stability. The ions scanned out of the trap strike the conversion dynode, ejecting electrons which are repelled to the electron multiplier. The conversion dynode is held at -10 KV and the voltage on the multiplier is adjusted to provide an enhancement of $\sim 10^5$. The signal is then sent through an integrator to collect an intensity reading for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

External Positive or Negative Chemical Ionization

About External Chemical Ionization

Chemical ionization (CI) provides mass spectral data that complement electron ionization (EI) data for the analysis of complex compounds. The generation of ions in chemical ionization is a two-step process. A CI reagent gas is introduced into the ion source. For chemical ionization, a special CI ion source volume of the reagent gas is ionized by EI. Then, the sample molecules are ionized by ion-molecule reactions with the reagent gas ions.

CI is a softer ionization technique than EI. That is, CI imparts less energy to the sample molecules than does EI. Thus, the ionized sample molecule undergoes less fragmentation, and an ion indicative of the molecular weight is more likely to be observed. In addition to molecular weight confirmation, CI mass spectra often provide other significant structural information that may not be available from EI mass spectra.

Adjusting CI Pressure

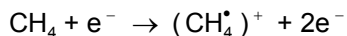
We recommend setting flows by observing the ion gauge pressure in Manual Control. Adjust the flow of reagent gas so that the ion gauge reading is between 50 and 80 μ Torr.

Forming Reagent Ions

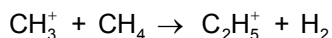
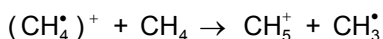
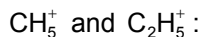
In the first step, reagent gas ions are formed as the reagent gas is ionized by interaction with electrons emitted by the filament. In external mode, the CI reagent can be either liquid or gas. The most common reagents are methane and ammonia. For ammonia the reagent ion is simply the protonated reagent, NH_4^+ .

NOTE: Reagent ion formation can be a complex process. For example, when methane is used as the reagent gas, reagent gas ions are formed as follows:

First, methane is ionized to form two primary ions:



These primary ions then react very rapidly to form predominantly the secondary ions,



The function of the reagent in negative CI is to act as a source of electrons and then to “thermalize”, or lower the kinetic energy of the electrons. Only electrons with energies less than 1-2 eV can attach and form negative ions by electron capture.

Transferring Analyte Ions to the Ion Trap

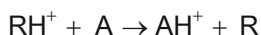
As chemical ionization occurs in the ion source, product ions are continuously being directed toward the ion trap by the lenses between the source and the ion trap. Loss of electrons from negative ions or fragmentation of positive ions can occur during this transfer process. Lowering RF storage levels may reduce this effect but remember that using too low an RF storage level will reduce trapping efficiency for high-mass ions.

PCI Reactions in the Ion Source

Four main types of reactions occur in positive chemical ionization. Symbolizing the sample analyte by A with mass M and the neutral reagent by R, these are:

Proton Transfer

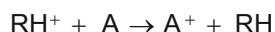
The reagent ion donates a proton to the sample molecule to produce a protonated analyte, sometimes called a pseudomolecular ion, with mass M+1.



This is the most commonly-recognized PCI reaction. Note that the AH^+ ion may fragment to lower-mass ions because of excess energy deposited during the PCI reaction.

Charge Transfer

The CI reagent may transfer only charge to the analyte molecule by electron transfer to the reagent.



Adduct Ion Formation

The CI reagent ion may bond in its entirety to the sample molecule to create an adduct ion.



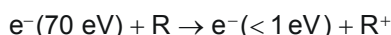
Hydride Abstraction

Some reagent ions have a tendency to remove a hydrogen atom to create an analyte product ion that is still positively-charged but having a weight of $M-1$.



NCI Reactions in the Ion Source

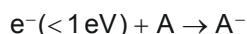
A variety of reactions occur in negative chemical ionization. First, the reagent is ionized by Electron Ionization to create thermalized, or low-energy electron:



Using CH_4 (Methane) as an example:



These low-energy electrons react with the analyte A:



Recommended External CI Reagents

Methane is the most frequently used CI reagent for both positive and negative chemical ionization. Ammonia can also be used for External PCI.

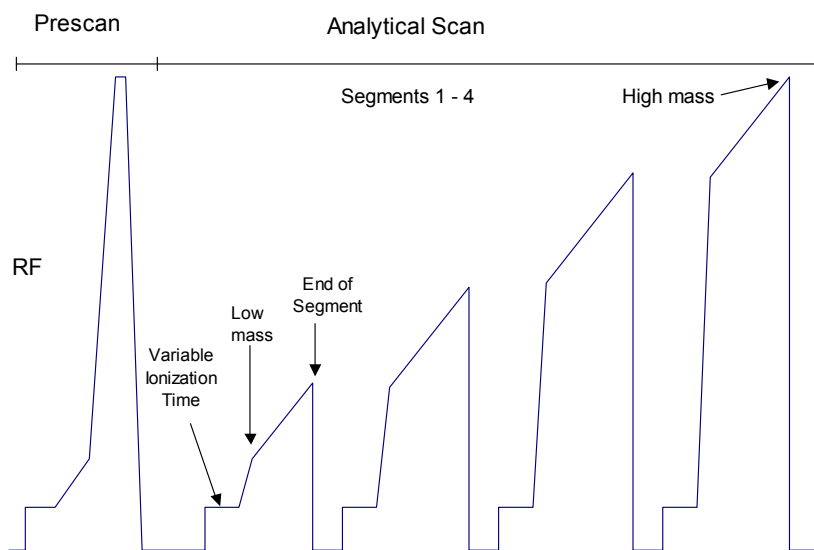
Liquid CI reagents are not recommended for External CI. Liquid reagents have insufficient vapor pressure to be used; the pressure of reagents in the ion source is not adequate for the high-pressure reactions required.

External CI Scan Function

For external chemical ionization mode, ion formation occurs within the source with subsequent transfer of ions to the ion trap by a set of lenses. The ions are stored and stabilized in the ion trap cavity by an RF field applied to the ring electrode. The ion trap has a maximum storage capacity, beyond which mass resolution and spectral quality deteriorate. The number of ions created is proportional to the ionization time, with more ions being produced the longer the ionization time. Automatic Gain Control (AGC) controls the ionization time in order to maintain the number of ions in the trap at the optimum level.

Four modes of operation (Internal EI, External EI, External CI, and Hybrid CI) use a similar scanning sequence involving Automatic Gain Control (AGC) to scan ions out of the trap. The AGC scan function consists of a prescan and up to six scan segments. The prescan uses a fast scan rate and the number of ions detected in the prescan is used to calculate the ionization time for the six scan segments. The figure below illustrates the AGC prescan and four scan segments corresponding to the following four portions of the mass range (default values): 10 to 99 m/z, 100 to 249 m/z, 250 to 399 m/z, and 400 to 1000 m/z.

One scan involves one complete ramping of the RF voltage over the mass range selected by the user. The RF voltage is ramped at a constant rate of approximately 5000 daltons/sec for normal scan and 10000 daltons/sec for fast scan. One complete scan at this fixed rate produces a microscan.



The AGC Scan Function

Scan Segment Setup Period

The prescan is followed by the first of the scan segments. Each segment is divided into two parts: a setup period and a scanning period. Each setup period is subdivided into the following four stages:

First stage: The RF voltage is set to near 0V for 2 milliseconds. This setting allows the RF voltage generator circuit to stabilize while clearing the trap cavity of all ions.

Second stage: The RF voltage is set to, and stabilizes at, the storage voltage selected to store, or trap, all ions of interest in the ion trap cavity.

Third stage: Energetic electrons from the heated filament are accelerated into the ion trap, where they interact with sample molecules to form sample ions. For external ionization mode, the ion formation occurs within the source with subsequent transfer of ions to the trap by a set of lenses.

The newly formed sample ions are then permitted to stabilize in their orbits at the storage voltage. This stabilization period is referred to as the cool time. The length of this period is fixed.

Fourth stage: The dynode and electron multiplier are made ready, and the RF voltage is ramped quickly from the storage voltage to a value appropriate to begin the mass scanning of the segment.

Scan Segment Scanning Period

During the scanning period the RF voltage is steadily increased, causing ions of increasing m/z to be ejected from the trap, to be detected by the dynode and electron multiplier.

The AGC software automatically selects an ionization time from 10 μsec to 65,000 μsec for each of the scan segments of the microscan, depending on the amount of analyte in the ion trap and the tuning parameters. An estimate of the number of ions formed in the ion trap is provided by a prescan with a varying ionization period (10 to 2,500 μsec). For low levels (e.g., for baseline or small GC peaks) the maximum ionization time of 65 milliseconds is selected, which offers maximum sensitivity. As the amount of analyte increases, the ionization time is decreased automatically in order to prevent overloading of the ion trap with ions.

The ion signals in each microscan are scaled automatically to correct for the variation in ionization time. Microscans are averaged to improve the spectral quality.

To optimize trapping of the ions of interest, a notched waveform is applied during ionization for internal EI mode. For external EI & external CI mode, a notched waveform is applied after the ions are trapped. The ions are analyzed by applying a radio frequency (RF) voltage to the ring electrode encircling the trap cavity. As the voltage increases on the ring electrode, ions are sequentially ejected from the trap according to their mass-to-charge ratio. Supplemental waveform voltages are applied to the end cap electrodes during the analysis to improve mass resolution and mass axis stability. The ions scanned out of the trap strike the conversion dynode, ejecting electrons which are repelled to the electron multiplier. The conversion dynode is held at -10 KV and the voltage on the multiplier is adjusted to provide an enhancement of $\sim 10^5$. The signal is then sent through an integrator to collect an intensity reading for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

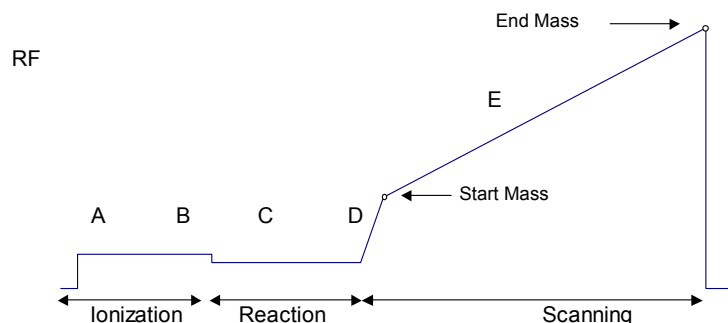
Hybrid Positive or Negative Chemical Ionization

Hybrid CI Scan Function

In the Hybrid CI configuration the external ion source is installed, but the transfer line directs the sample into the ion trap. CI reagent ions are generated in the external source and only those reagent ions which are selected are stored in the ion trap. These trapped reagent ions are allowed to react with sample molecules as they enter the ion trap, forming CI product ions by ion-molecule reactions. Hybrid CI may be used with either positively-charged or negatively-charged reagent ions.

The ion trap operates in a pulsed mode. Reagent ions are created only during the ionization pulse and are consumed during the reaction period to form analyte ions. The number of analyte ions depends on the concentration of the analyte, the initial reagent ion intensity, and the reaction time.

Space charge control in the Hybrid CI mode is achieved by using the results from an AGC prescan to calculate the ionization time and reaction time for the analytical scan. Because the spectral intensity is proportional to sample concentration and reaction time, linear calibration curves can be obtained.



Hybrid CI Scan Function (Analytical scan only)

During the Hybrid CI analytical scan, the following steps occur:

- A The reagent gas is ionized for the length of time determined by the prescan.
- B The selected reagent ions are stored in the ion trap. Ejection of ions above the Selected Reagent High Mass is accomplished by applying a broadband waveform between the ionization and reaction periods.
- C Reagent gas ions react with sample molecules to form sample ions. (The reaction time is determined by the prescan.)
- D Reagent ions are ejected.
- E The Hybrid CI mass spectrum is acquired for the sample ions.

The ionization and reaction storage RF can be set at the same level or different levels.

The “View Reagent Ions” function can be used to tune the isolation of individual reagent ions.

Scanning Ions to Collect Mass Spectra

The scanning process for hybrid chemical ionization is similar to that for electron ionization. After ionization, trapping, and ion preparation steps, ions are scanned out to the conversion dynode and electron multiplier. Mass scanning is implemented by increasing the RF voltage on the ring electrode; the mass spectrum is collected in order from low to high mass over the user-designated scan range. In positive mode, electrons are ejected from the conversion dynode held at -10000V and repelled to the electron multiplier. In negative mode, positive ions are ejected from the +10000V dynode and repelled toward the multiplier. The signal is enhanced by $\sim 10^5$ by the multiplier and sent through an integrator to collect an intensity for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

Ion Preparation Techniques

MS/MS - Tandem Mass Spectrometry

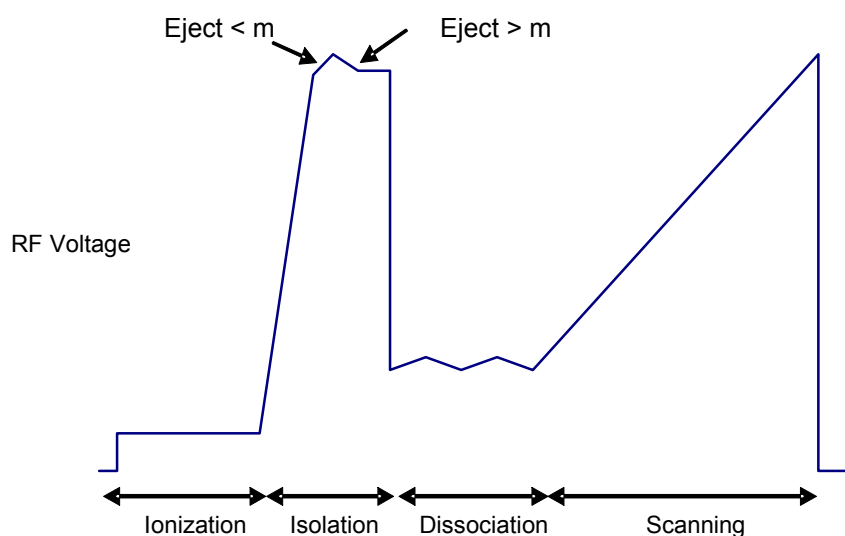
Overview

MS/MS is the isolation of a precursor ion formed by either EI or CI ionization, dissociation of the precursor ion by energetic collisions with helium atoms, and trapping and scanning of the resulting product ions.

The utility of the MS/MS technique derives from the following:

- Optimally filling the ion trap with the selected precursor ion;
- Obtaining qualitative structural information about the sample by forming the product ion spectrum; and
- Increasing the signal-to-noise ratio by eliminating interfering matrix ions in the product ion spectrum through isolation and dissociation - the only ions in the product ion mass spectrum originate from ions that fall into the isolation window.

The MS/MS Scan Function



MS/MS Scan Function (Analytical scan only)

This section describes the four basic operations in 4000 MS Ion Trap Tandem Mass Spectrometry (MS/MS):

- Ion formation and matrix ion ejection
- Precursor ion isolation
- Product ion formation through collision-induced dissociation
- Product ion mass scanning.

Ion Formation and Matrix Ion Ejection

Ionization of the sample and any co-eluting matrix occurs by electron or chemical ionization. A notched waveform is applied to the end caps of the ion trap as described above to resonantly eject ions above and below the specified precursor ion mass. Removal of most of the space charge from the trap improves mass resolution in subsequent MS/MS steps. The ions remaining in the trap include the optimum number of precursor ions, along with any ions which fall into the coarse isolation window.

Precursor Ion Isolation

The precursor ions are then isolated from any remaining matrix ions in a two-step process.

The first step ejects the ions with masses below the precursor ion mass by ramping the RF amplitude while applying the supplemental waveforms to the end cap electrodes. This step resonantly ejects all ions having masses up to, and including, the mass just below the precursor ion mass. The supplemental waveforms are then turned off.

In the second step, a broadband waveform (WF2) is applied to the trap to simultaneously eject all masses above the precursor ion mass. Isolation occurs at elevated RF levels where mass resolution is optimal. Thus, it is possible to achieve unit mass isolation over the entire mass range of the ion trap, which may be useful in isolating a single mass in an isotopic cluster, or in separating the precursor ion from interfering matrix ions.

Product Ion Formation

Product ions are formed from the precursor ions by collision-induced dissociation (CID). The precursor ions constantly undergo collisions with helium gas in the ion trap. Normally these collisions involve relatively small energies, but if the translational energy of the precursor ion is increased, the collisions may convert the translational kinetic energy into internal vibrational energy.

If the precursor ion acquires enough vibrational energy, one or more chemical bonds in the ion may be broken, forming ions of lower m/z than the original precursor ion. The ion distribution that results from the CID process depends on the characteristics of the precursor ion and the amount of energy that has been converted into internal vibrational energy.

The amount of energy that is converted to internal energy in the precursor ion depends on the number of collisions (excitation time), the relative energy of the collisions (CID excitation amplitude), and the rate that the internal energy is removed by collisional deactivation (related to the excitation method). Collision-induced dissociation in the ion trap is always in competition with ion ejection. If the CID excitation amplitude selected is too large, the precursor ion will be

ejected to the trap electrodes before it can collide with background helium atoms. If the CID excitation amplitude selected is too small, the energy of the precursor ion will not exceed the internal energy threshold required to break the chemical bonds and form product ions. This is because energy is constantly being removed by low energy collisional deactivation. Therefore, the CID excitation amplitude and the excitation time are both used to optimize the CID process in conjunction with an appropriate choice of excitation method.

The translational kinetic energy of the precursor ion can be increased using either of two methods. Each method requires application of a waveform to the ion trap. The waveform amplitude is called the CID excitation amplitude, and the length of time that the waveform is applied is called the excitation time.

The first method involves *non-resonant excitation*. In this approach, a low frequency supplemental dipole field is applied to the end caps of the trap, resulting in an instantaneous change in the potential energy of the ion in the trapping field.¹ The restoring force of the trapping field converts the newly increased potential energy of the ion into increased translational kinetic energy. A portion of this kinetic energy is then converted into internal vibrational energy upon subsequent collisions. This process is repeated during each oscillation cycle of the low frequency dipole field.

An advantage of using non-resonant excitation is that it is not critical to match the applied dipole frequency to that of the ion. Consequently, electronic drift, space charge effects, and sample concentration do not affect the method. This results in reproducible product ion spectra. This method is often useful with precursor ions that fragment by the breakage of a single weak chemical bond to form highly stable ions containing functional groups that do not undergo significant rearrangements. A disadvantage of the method is that it is not selective with respect to excitation of ions in the trap. Therefore, the method cannot be selectively tuned to excite only ions having a particular m/z . The method is also less useful with precursor ions in which multiple chemical bonds are broken and with precursor ions that undergo complex rearrangements following collision-induced dissociation.

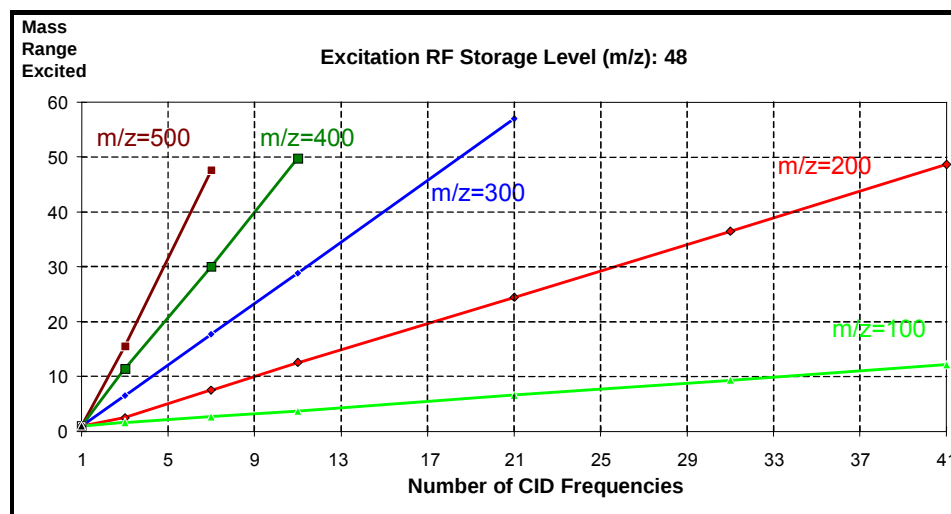
The second method for increasing the precursor ion kinetic energy involves *resonant excitation*. This method requires application of a high frequency supplemental dipole field to the end caps of the ion trap. The frequency must match the oscillation frequency of the trapped ion. The resonant frequency of the trapped ion depends on ion mass, space charge within the ion trap, RF trapping field amplitude, and other instrumental factors. Thus, it is difficult to precisely calculate its value. The amplitude of the RF trapping field is therefore modulated over a fixed range. As the resonant frequency of the trapped ion depends on the magnitude of the RF field, modulating the RF field amplitude results in a modulation of the resonant frequency of the ion. Modulation of this frequency causes the frequency of the ion to periodically match that of the applied supplemental dipole field. Thus, the energy coupled to ion motion is maximized, and the effects of shifts in the ion resonant frequency are minimized.

The resonant excitation method is selective to the mass range that is excited. It permits coupling of energy to the motion of an ion having a particular m/z value in a very controlled way. Consequently, at the correct excitation amplitude, the rate that the amplitude of the ion motion increases is just balanced by the rate that energy is removed by collisions. The ion is not ejected and energy can be deposited as internal energy by increasing the number of collisions. Increasing

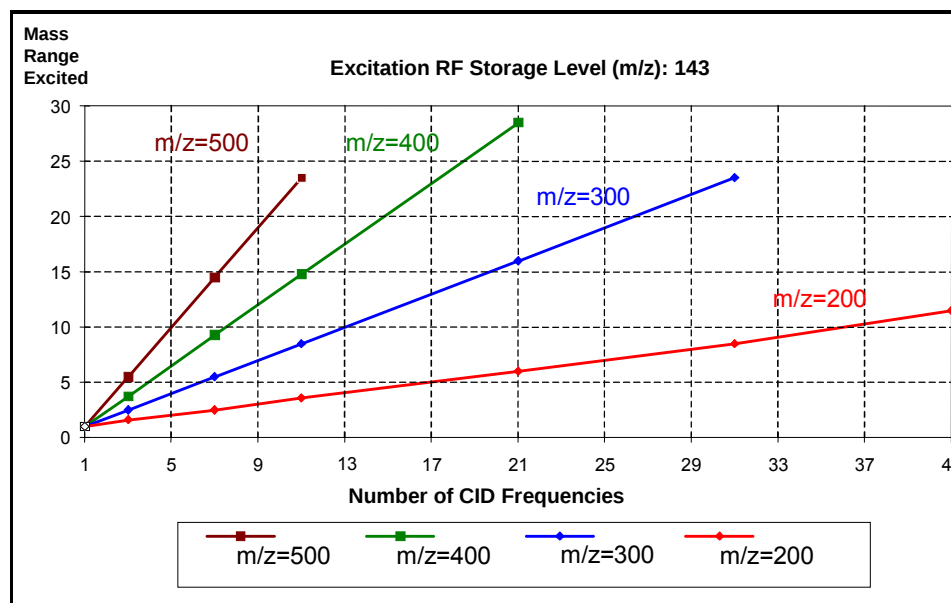
¹ The dipole field is simply an electric field oriented along the axis of the trap.

the excitation time increases the number of collisions. Thus, it is possible to fragment precursor ions that require the breakage of multiple chemical bonds or that undergo significant rearrangements. Periodic modulation through ion resonance results in an averaging of the energy coupled into the ion motion and provides a reproducible product ion spectrum, even with changes in concentration.

An additional parameter is available with resonant excitation. Increasing the number of CID frequencies permits simultaneous excitation of ions within a range of masses. This may be useful when trying to excite an entire isotopic cluster at once. As the number of CID frequencies increases, the range of excited masses also increases. At a given number of CID frequencies, the range of excited masses increases with increasing ion mass, and decreases with increasing excitation RF storage level.



Mass range excited vs. number of CID frequencies for an excitation RF storage level of 48 m/z



Mass range excited vs. number of CID frequencies for an excitation RF storage level of 143 m/z

Note that at a storage level of 48 m/z and with 9 CID frequencies, the mass range increases from 3 to 40 m/z when the precursor ion mass increases from 100 to 400 m/z. Given a precursor ion mass of 400 m/z and 9 CID frequencies, the mass range decreases from 40 m/z for a storage level of 48 m/z to a range of 12 m/z for a storage level of 143 m/z.

Product Ion Mass Scanning

Following formation of the product ions by collision-induced dissociation, an RF ramp is used along with the supplemental waveforms to resonantly scan ions from the trap to the conversion dynode and electron multiplier. This process generates the product ion mass spectrum.

Chemical Ionization/MS/MS

Chemical ionization (CI) may also be used to selectively fill the ion trap. In this case, the ion formation and matrix ion ejection steps described above are modified. Insertion of the three subsequent MS/MS operations, i.e., precursor ion isolation, product ion formation, and product ion mass scanning, into the basic CI scan function (following the ionization and reaction steps) creates the CI/MS/MS scan function.

EI/MS/MS and CI/MS/MS Automatic Space Charge Control

Space charge control for EI/MS/MS and CI/MS/MS uses a prescan to measure the number of precursor ions obtained using the same scan function as the analytical scan up through the fine isolation step. Based on the signal intensity of the precursor ions in the prescan, an ionization time is calculated for the analytical scan which will maintain the optimum number of ions in the trap, even as the sample and matrix levels change, resulting in consistent, reproducible product ion spectra.

When using MS/MS, lower target values may be required to prevent adverse space charging effects. Based on the isolated precursor ion, the following target values are recommended.

Precursor Ion	Target
60 < m/z < 150	10000
150 < m/z < 500	5000
500 < m/z < 1000	2000

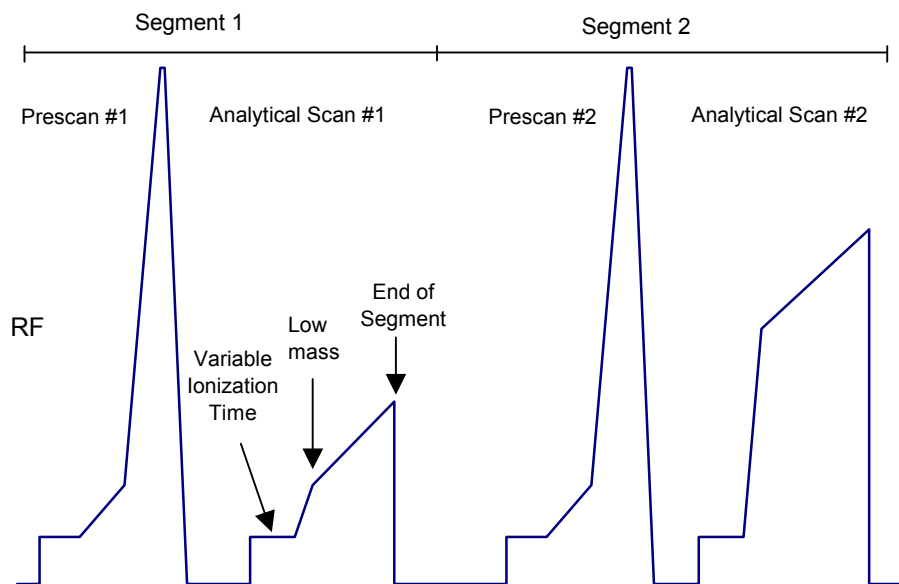
Selected Ion Storage

Selected Ion Storage Scan Function

Selected Ion Storage (SIS) may be used with external EI or internal EI to store and scan narrow and/or discontinuous mass ranges. Using waveforms, unwanted parts of the mass range are ejected. When used for target compound analysis, SIS can provide library-searchable EI spectra. However, because the isolation waveform is applied at a low q value, unit mass isolation is not possible across the mass range.

Space charge control in the SIS mode is achieved by using the results from an AGC prescan to calculate the ionization time for each analytical scan. Each SIS segment uses its own prescan, collecting ions only from the range specified in that segment. The stored spectrum is the sum of the individual spectra from each individual scan segment.

NOTE: Because each SIS segment has its own prescan and analytical ionization time, the reported ion time for each analytical scan can only reflect a subset of the ion time data. The reported ion time is the shortest ion time among the set of SIS segment ion times. This allows users to identify situations where overloading may distort mass spectra. Distortion is likely when the ion time becomes very short - approaching the minimum ion time of 10 μsec .



SIS Scan Function

During the SIS scan, the following steps occur:

- The prescan measures the number of ions within the specified scan segment and calculates an ionization time for the associated analytical scan segment.
- The selected range of ions is stored in the ion trap.
- The SIS mass spectrum is acquired for the range of sample ions.

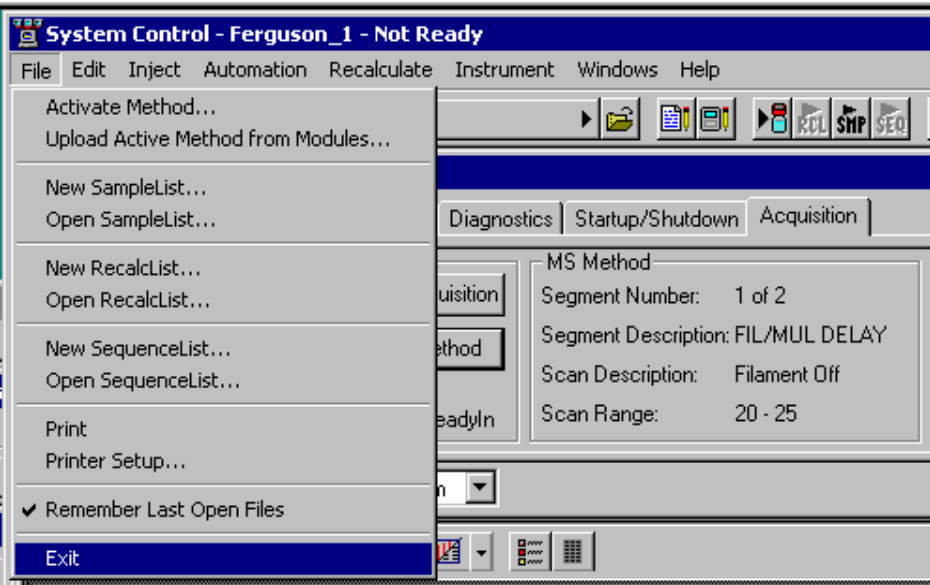
Scanning Ions to Collect Mass Spectra

The SIS scanning process is very similar to that for full scan electron ionization. After ionization, trapping, and ion preparation steps, ions are scanned out to the conversion dynode and electron multiplier. Mass scanning is implemented by increasing the RF voltage on the ring electrode; the mass spectrum is collected in order from low to high mass over the user-designated scan range. In positive mode, electrons are ejected from the conversion dynode held at -10000V and repelled to the electron multiplier. In negative mode, positive ions are ejected from the +10000V dynode and repelled toward the multiplier. The signal is enhanced by $\sim 10^5$ by the multiplier and sent through an integrator to collect an intensity for each m/z . MS data are stored as sets of ion-intensity pairs for each m/z over the acquired mass range. A complete mass spectrum is stored for each analytical scan.

System Control Command Reference

System Control Menus

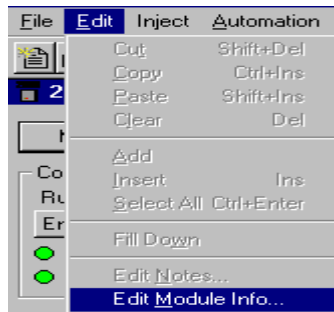
File Menu



Menu Item	Description
Activate Method...	Displays the Activate a System Control Method File dialog box to activate a Method and download the Instrument Control sections to the modules.
Upload Active Method from Modules...	Displays the Save a System Control Method File dialog box to allow you to name the Method that will receive the instrument control sections uploaded from all modules connected to the instrument.
New SampleList...	Displays the Create a New System Control SampleList File dialog box to name a new SampleList.
Open SampleList...	Displays the Open a System Control SampleList File dialog box to open an existing SampleList.

Menu Item	Description
New RecalcList...	Displays the Create a New System Control RecalcList File dialog box to name a new RecalcList.
Open RecalcList...	Displays the Open a System Control RecalcList File dialog box to open an existing RecalcList.
New SequenceList...	Displays the Create a New System Control Sequence File dialog box to name a new Sequence.
Open SequenceList...	Displays the Open a System Control Sequence File dialog box to open an existing Sequence.
Print	Prints the contents of the active window in System Control.
Printer Setup...	Opens the Star Print Setup Dialog Box to select a printer and set options for it. You can open the Print Setup dialog box from the Star Printer Setup dialog box. The Varian MS Workstation only uses the printer for instrument 1.
Remember Last Open Files	When checked, upon startup System Control will restore the active Method and any automation files that were open when System Control was last closed.
Exit	Closes System Control. If System Control is in the process of performing a critical operation, you may be prompted before the application is closed.

Edit Menu



These commands are for use with SequenceLists, SampleLists, and RecalcLists.

Menu Item	Description
Cut	Deletes a selection and copies it to the Clipboard. Used to remove or move a selected part of a spreadsheet or entered text.
Copy	Copies a selection to the Clipboard. Used to duplicate a selection and place the duplicate in a new place (using Paste).
Paste	Inserts previously cut or copied information that was stored in the Clipboard into a spreadsheet.
Clear	Deletes a selection but leaves the Clipboard unchanged.

Menu Item	Description
Add	Adds a new line in a SequenceList, SampleList or RecalcList.
Insert	Inserts a new line in a SequenceList, SampleList or RecalcList.
Select All	Selects all lines in a SequenceList, SampleList or RecalcList.
Fill Down	Causes the contents of the top cell in a series of highlighted cells to be copied to the cells below it. Used to edit all the cells in a column quickly.
Edit Notes...	Opens the Edit Notes Dialog Box to permit editing of the notes associated with the SequenceList, SampleList, RecalcList, or Method file displayed in the active window.
Edit Module Info...	Opens the Module Information Editor Dialog Box to permit editing of the Module Information associated with the module displayed in the active window.

Inject Menu



Menu Item	Description
Inject Single Sample...	Opens the appropriate Inject Single Sample Dialog Box for your system. Used to inject a single sample without building a SequenceList and SampleList manually.

Inject Single Sample

Sample Name	Sample Type	Cal. level	Inj.	Injection Notes	AutoLink	Amount Std (IS, N% only)	Unid Peak Factor	Multiplier	Divisor	MultiChannel MultiStandard
Default Sample	Analysis		1	none	none	1	0	1	1	none

Inject the Sample using the Method:

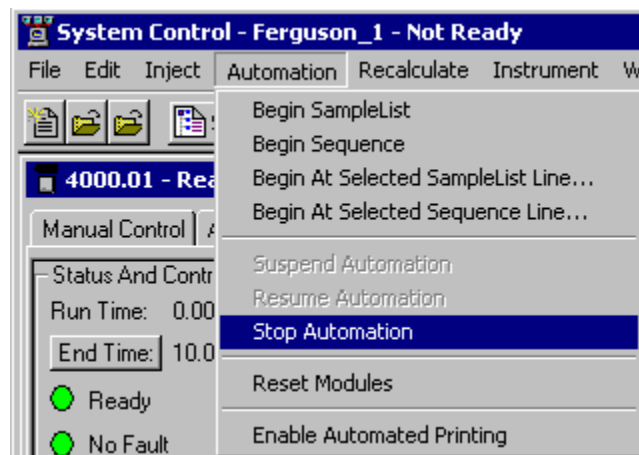
C:\Varian\WS\MP10\SPME2.mth Browse... Defaults...

☐ Clear Coefficients before Calibrating

Inject Cancel Data Files... RecalcList...

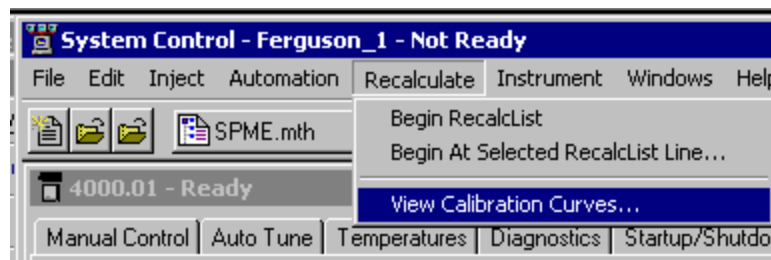
For more information on the Inject Single Sample dialog, see Acquiring GC/MS Data, "Injecting a Single Sample" on page 159.

Automation Menu



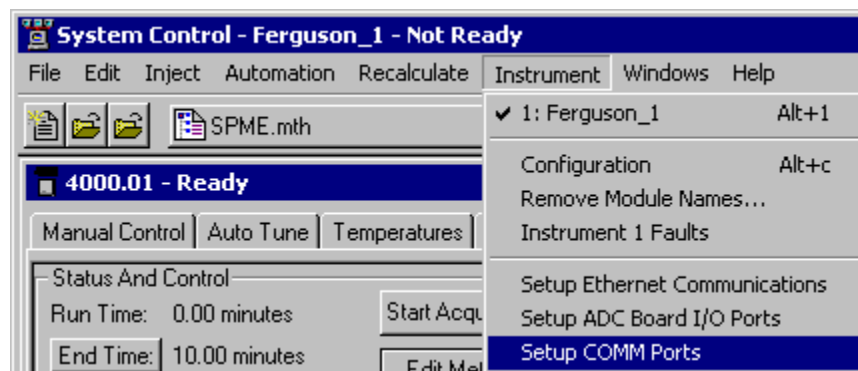
Menu Item	Description
Begin SampleList	Begins execution of the currently open SampleList. The currently active method will be downloaded to the instrument(s), which will begin to equilibrate and stabilize.
Begin Sequence	Begins execution of the currently open Sequence. The method specified in the first line of the sequence will be downloaded to the instrument(s) and the corresponding Sample List will be activated.
Begin at Selected SampleList Line...	Begins the open SampleList at the selected SampleList line. This item is enabled when a SampleList is open. Upon selecting this menu item, a prompt for the line in the SampleList appears. This is useful when recovering from a power failure that interrupted automation in progress.
Begin at Selected Sequence Line...	Begins the open Sequence at the selected Sequence line. This item is enabled when a Sequence is open. Upon selecting this menu item, a prompt for the line in the Sequence appears. This is useful when recovering from a power failure that interrupted automation in progress.
Suspend Automation	Suspends execution of automation after the current run has been completed.
Resume Automation	Resumes execution of automation after it has been suspended.
Stop Automation	Stops the current run, resets all modules simultaneously and suspends automation.
Reset Modules	Stops the current run, resets all modules simultaneously. Automation proceeds to the next injection after all the modules go to the Ready state.
Enable Automated Printing	Turns on or off the printing of reports automatically generated during any automation action. This is useful when you wish to disable printing (if the printer is out of paper, for example) without having to modify the Print Options portion of the Report Method.

Recalculate Menu



Menu Item	Description
Begin RecalcList...	Begins recalculation of the open RecalcList using the active Method.
Begin at Selected RecalcList Line...	Begins the open RecalcList at the selected RecalcList line. This item is enabled when a RecalcList is open. Upon selecting this menu item, a prompt for the line in the RecalcList appears. This is useful when recovering from a power failure that interrupted automation in progress.
View Calibration Curves...	Opens the Open a Method File to View Curves dialog box to view calibration curves that are stored in a Method file.

Instrument Menu

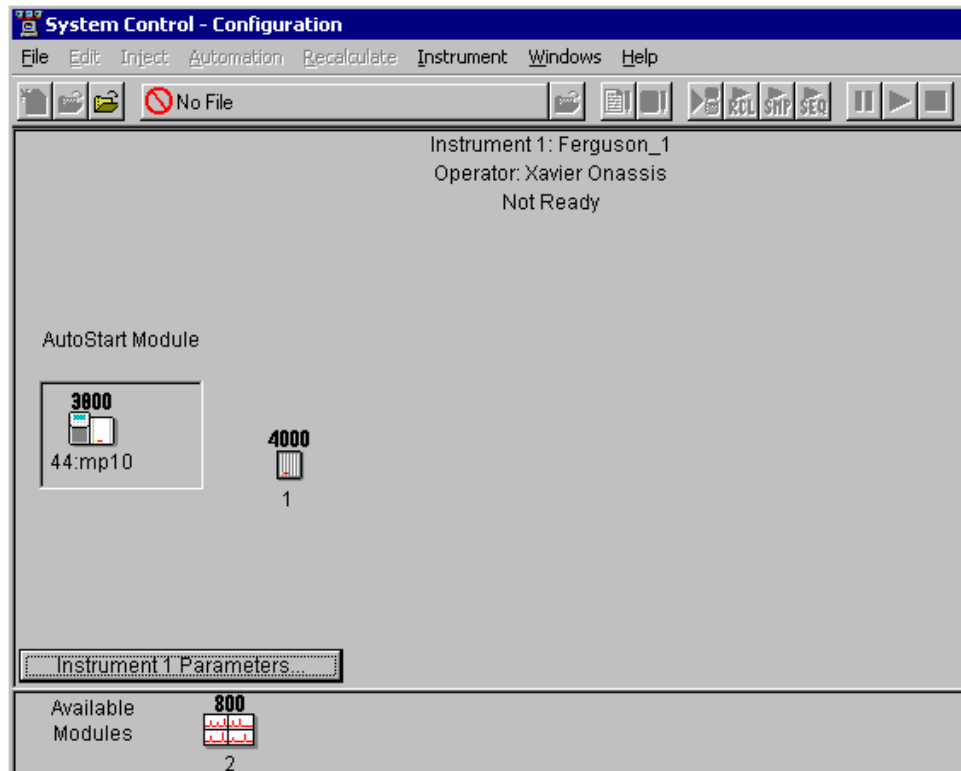


The Instrument Menu commands are used to switch the display between the configuration screen and the instrument.

Menu Item	Description
#:instrument name	Displays the Instrument Status window and Status and Control windows for the named Instrument, e.g., 4000 GC/MS #1
Configuration	Displays the System Control Configuration window.
Remove Module Names...	Brings up a dialog listing any associations you have set up. You may select items from this list to remove individual associations, in preparation for renaming LC modules.
Instrument # Faults	Displays the Instrument Faults dialog box, showing recent and self-test messages and faults. The list of faults may be printed from this dialog box.

Menu Item	Description
Setup Ethernet Communications	Available if an optional module driver is installed that uses Ethernet to communicate with the device. See the Operation Manual for your module driver for a detailed description if applicable. Allows set-up of the communication between the 3800 GC and System Control.
Setup ADC Board I/O Ports	Opens the Setup ADC Board I/O Ports Dialog Box to configure the ADC Board base address and determine the Switch S1 settings for each ADC Board. Applicable only if the ADC Board module driver is installed.
Setup COMM Ports	Available if an optional module driver is installed that uses Serial I/O to communicate with the device. See the Operation Manual for your module driver, such as the 3400 GC, for a detailed description if applicable.

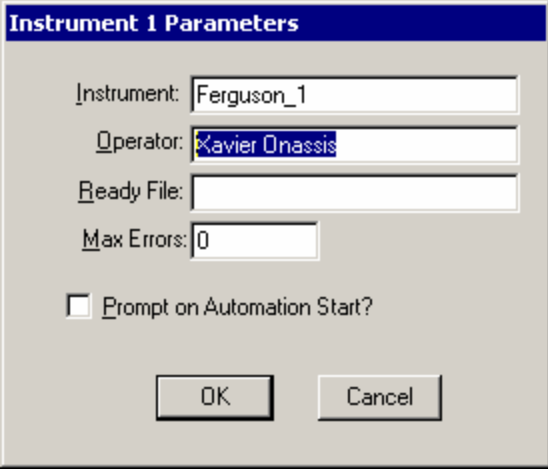
Configuration Window



The Configuration screen is divided into two parts: the Instrument window and the Available Modules window. Initially, modules are displayed in the Available Modules window as they log in, and can then be added to the Instrument by clicking and dragging the module icon into the Instrument window. The configuration of modules is remembered when System Control closes, and modules will be restored automatically to their proper locations when System Control is opened.

The AutoStart Module area is used to designate which module will start the system in Automation.

- If you are not using an AutoSampler or other automated injection device, do not place any module in this area.
- If you are using a CP-3800 GC with an 8400 AutoSampler, place the GC icon in the Autostart area.
- If you are using an 8200 or a Combi PAL AutoSampler, place the AutoSampler icon in the AutoStart area.
- If you are using any other device which will start the system through the Sync signal inputs of the GC, leave the AutoStart area empty.



The image shows a dialog box titled "Instrument 1 Parameters". It contains four text input fields: "Instrument:" with the value "Ferguson_1", "Operator:" with the value "Xavier Onassis", "Ready File:" which is empty, and "Max Errors:" with the value "0". Below these fields is a checkbox labeled "Prompt on Automation Start?" which is currently unchecked. At the bottom of the dialog are two buttons: "OK" and "Cancel".

The Instrument 1 Parameters dialog allows you to enter an instrument name and operator name.

The Ready File is used only by earlier versions of the Saturn GC/MS, and should be left blank for operation with 4000 MS.

The Max Errors number sets the allowable number of non-fatal errors during Automation. When the limit is reached, Automation halts. An entry of 0 defeats this feature.

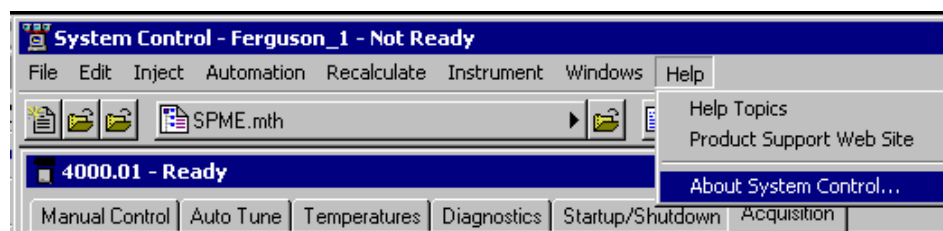
The Prompt on Automation Start checkbox allows you to view and edit information in this dialog each time Automation is started.

Windows Menu



Menu Item	Description
Show Module Windows	Opens the Status and Control windows for all the modules in the Instrument. It is disabled when all the windows are open, even if some are hidden under others.
Iconize Module Windows	Iconizes the Status and Control windows for all the modules in the Instrument. It is disabled when all the windows are iconized.
Show Automation Windows	Displays the windows for all the automation files open in the Instrument. It is disabled when all the automation windows are open, even if some are hidden under others.
Iconize Automation Windows	Iconizes the windows for all the automation files in the Instrument. It is disabled when all the windows are iconized.
Arrange Icons	Arranges the icons, if any, in a row at the bottom of the System Control window.
SequenceList:, SampleList:, RecalcList:	Opens the window for the selected file or moves it to the front. Used to display the open SequenceList, SampleList, or RecalcList.
Instrument Status, Message Log, Configuration Log (only if Configuration window is open)	Displays the corresponding window for the instrument or moves it to the front. Used to see the instrument status, Message Log, or Configuration Log.
Module Window (name varies depending on module)	Displays the Status and Control window for the chosen module or brings it to the front.

Help Menu

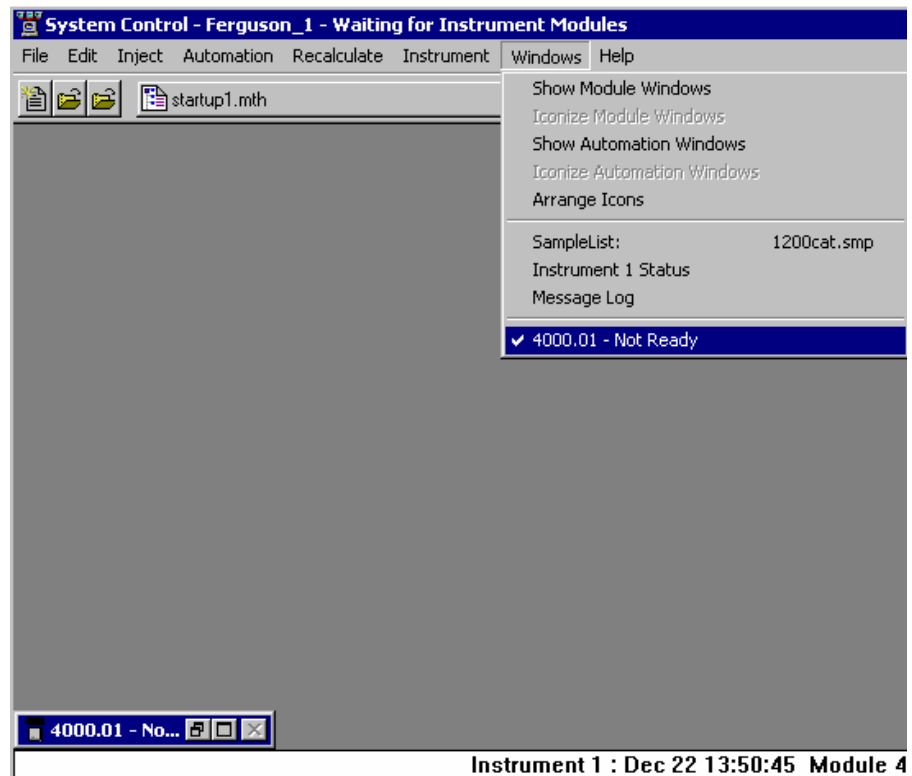


Menu Item	Description
Help Topics	Displays the Help Topics window for System Control.
Product Support Web Site	If you have Internet access and a web browser installed on your computer, this option will automatically open the Varian MS Workstation Product Support Web Site. Here you will find the latest software and documentation updates for the Varian MS Workstation suite of products, along with additional notes, tips, and answers to frequently asked questions. You may wish to visit this site periodically to see if new information is available that may be pertinent to you.
About System Control	Displays the About Box for the System Control application. The About Box contains information about the software version, installation information, and a list of the instrument control modules that you have installed. You can also validate the installed Workstation files from this dialog.

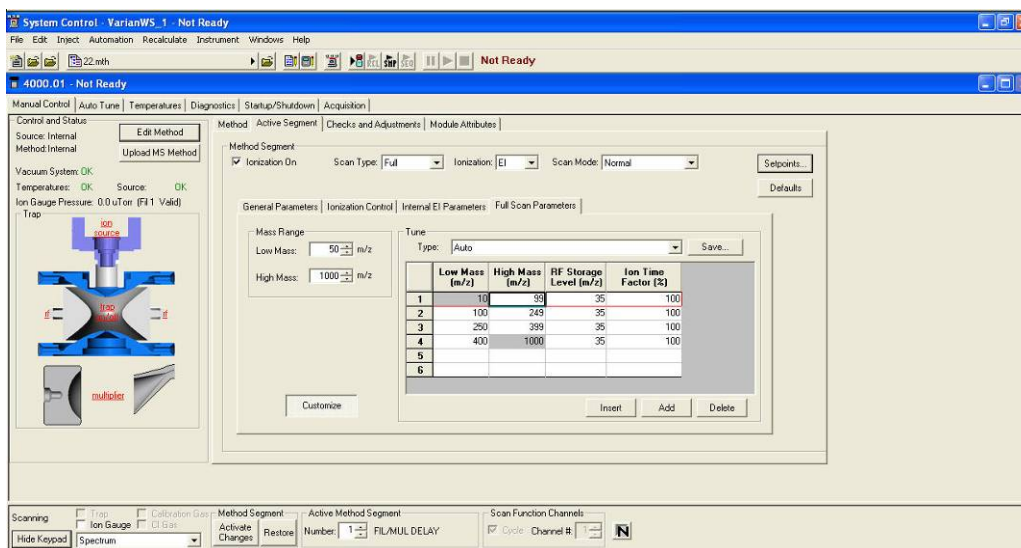
4000 MS System Control Command Reference

Display

The 4000 MS status and control window is available for display whenever a 4000 MS module is configured in System Control. It may be displayed in the System Control Status window by selecting the Windows menu item and clicking the 4000.xx menu item, or by expanding the 4000.xx icon which appears at the lower left of the System Control window.



System Control Tabs



Access most of the 4000 MS functions through Control and Status. The various functions are organized into six tabs.

Manual Control - Interactive adjustment of all the method parameters of the currently active method. The Checks and Adjustments tab allows adjustment of CI gas and Cal gas flow, RF ramp, and a check of air/water levels. The Module Attributes tab permits the selection of filament 1 or 2, and the setting of electron multiplier voltage and Trap Damping Gas Flow (External mode).

Auto Tune – Run the built in tuning routines of the 4000 MS.

Temperatures - Set trap, manifold, and transfer line temperatures for normal operation and for Bakeout, and set the timer for the Bakeout period.

Diagnostics allows the user to run built in diagnostic routines to assess the state of the vacuum system and electronics.

Startup/Shutdown allows the user to start or shut down the vacuum system and heated zones in an orderly manner.

Acquisition - Run samples and acquire data files, often in conjunction with the Inject and Automation menus of the System Control window.

Hide/Show Keypad

The Control and Status window can be hidden by clicking the Hide Keypad. This becomes a Show Keypad button to restore the Control and Status display. While the keypad is hidden, the On/Off controls for the trap, Cal Gas, and CI gas are still accessible, as well as the method segment selector and the Profile/Centroid control.

Manual Control

Manual Control is very closely linked with the Method Builder function of the software. A method may be built in Method Builder, activated in System Control, then manipulated segment by segment in Manual Control. Conversely, a method may be modified and fine tuned in Manual Control, then uploaded and saved under the same name or as a new named method. The important distinction is that the effect of changes made in Manual Control can be seen immediately in the real time spectrum display, using Cal Gas or background ions as a surrogate sample, or using a device such as a Chromatoprobe to provide a continuous supply of a sample.

Control and Status

Control and Status displays a graphic of the ion trap system and status of the vacuum system and temperatures. Clicking the trap in the graphic display to turn on/off the ion source, RF system and the multiplier. Clicking on the ion sources, RF and multiplier will turn these parameters on/off individually.

Edit Method

Edit Method opens Method Builder with the current method in System Control.

Upload MS Method

Upload MS Method opens a Save Method dialog to allow the MS method parameters currently active in System Control to be copied to an existing method, or to a new method name. Changes made in Manual Control must be Activated using the Activate Changes before they can be uploaded.

Checkboxes

Clicking the Trap checkbox to turn on the entire ion trap system. Click the Ion Gauge checkbox to monitor the pressure inside the trap. Clicking Calibration Gas and CI Gas to open the valves to allow those gasses into the trap.

Method Segment: Activate Changes/Restore

These become active only after one or more changes have been made through Manual Control. Any changes made through Manual Control may be made a part of the active method by clicking Activate Changes. Before this, changes may be reversed and the method segment restored to its previous condition by pressing the Restore button. To make changes a permanent part of the active method, click Activate Changes, then click Upload MS Method, select a folder and file name, and save the method. You will be prompted to save any unactivated changes when you leave the Manual Control tab or change Segment number.

Active Method Segment Number

The active method segment control allows you to select any existing segment of the active method for review or modification under Manual Control. Existing segments cannot be deleted, nor can additional segments be added through Manual Control.

Scan Function Channels

Some of the 4000 MS methods are broken up into multiple channels. The channels can be thought of as a subset of a segment, where the scan conditions can be different than in other channels. These are most frequently used in the case of MSⁿ, MRM, etc. In Manual Control, there is the ability to continuously scan a single channel, or to rotate through all of them sequentially, as happens in the acquisition mode.

Mass Data Type

Centroid data is the default acquisition type, as data handling, library searching, and spectral comparison can be done from this type of data.

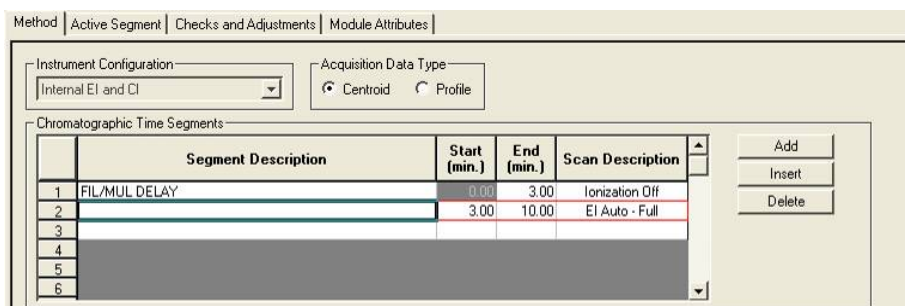
Profile data is typically used mainly for diagnostic purposes. Profile files are also approximately 10 times larger, but can be converted to centroid format after acquisition.

Intensity Normalization

Click  to turn normalization on or off. When it is on, the intensities are normalized by the ionization time. The normalization is different for EI and CI. Intensities collected under CI conditions are multiplied by the square of the ion time ratio. Intensities collected under EI conditions are multiplied by the ion time ratio. The ion time ratio is the maximum ion time specified, divided by the actual ionization time for that scan segment.

Method

The Method functions are illustrated here using the internal EI mode of operation. Details of other ionization modes and ion preparation options are in the Method Builder section of the manual. While the contents of the segments can be modified, the begin and end times of the segments cannot be changed from the Method Segments window. You can add, insert or delete segments using the buttons to the left of the segments.



	Segment Description	Start (min.)	End (min.)	Scan Description
1	FIL/MUL DELAY	0.00	3.00	Ionization Off
2		3.00	10.00	EI Auto - Full
3				
4				
5				
6				

Full Scan EI General Parameters: The Scan Time and Scans Averaged fields are linked with each other and with the Scan Type, Ionization, and Scan Mode fields. If you adjust the Scans Averaged value, the Scan Time and Data Rate values will also change, the values obtained depending upon the Scan Type, Ionization mode, and Scan Mode selected. Changing the Scan Time changes the values for Scans Averaged and Data Rate. Defaults returns all values to defaults for the selected Scan Type, Ionization mode, and Scan Mode. If the Ionization On box is not checked, all other controls become inactive, indicating that the segment is a Filament/Multiplier Delay segment.

Method | Active Segment | Checks and Adjustments | Module Attributes

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints... Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

SetPoints

☐ Scan Time 1.15 Maximum Scan Time Multiplier Offset: 0 +/- volts

☒ uScans Averaged: 3 uScans Mass Defect: 0 mmu/100u

Data Rate: 0.87 Hz

Customize

Full Scan EI Ionization Control: Use Ionization Control to set the Automatic Gain Control (AGC) target, the total number of ions the system will attempt to generate within the trap, using up to the Maximum ionization time selected. A lower target reduces space charge effects on resolution, while a higher target may improve quantitation. Click Customize to display Ionization Time. If the Fixed Ionization Time box is checked, the AGC adjustment of ionization time is replaced by the fixed ionization time selected. Click Defaults to restore the default times for the segment Scan Type, Ionization mode, and Scan Mode.

Method | Active Segment | Checks and Adjustments | Module Attributes

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints... Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

Automatic Gain Control

Target TIC: 20000 counts

Ionization Time

☐ Fixed Ionization Time

Ionization Time: 100 useconds

Max Ion Time: 25000 useconds

Customize

Full Scan EI – Internal EI Parameters: Use Internal EI Parameters to set the filament emission current. Higher current leads to more ionization and thus higher sensitivity, but may also lead to shorter filament lifetime.

Method | Active Segment | Checks and Adjustments | Module Attributes

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints...
Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

Internal EI Parameters

Emission Current: 10 uAmps

Full Scan EI – Full Scan Parameters: Use Full Scan Parameters to select the mass range for this segment. You can customize the mass range, RF storage level, and ion time factor for each segments, to optimize the sensitivity for selected compounds, or match the desired spectral characteristics required for tuning with BFB or DFTPP.

Method | Active Segment | Checks and Adjustments | Module Attributes

Method Segment

☒ Ionization On Scan Type: Full Ionization: EI Scan Mode: Normal Setpoints...
Defaults

General Parameters | Ionization Control | Internal EI Parameters | Full Scan Parameters

Mass Range

Low Mass: 50 m/z
High Mass: 1000 m/z

Tune

Type: Auto Save...

	Low Mass (m/z)	High Mass (m/z)	RF Storage Level (m/z)	Ion Time Factor (%)
1	10	99	35	100
2	100	249	35	100
3	250	399	35	100
4	400	1000	35	100
5				
6				

Customize Insert Add Delete

Checks and Adjustments

The Checks and Adjustments tab allows adjustment of CI gas and Cal gas flow, RF ramp, and a check of air/water levels.

The screenshot shows the 'Checks and Adjustments' tab selected. On the left, a list of options includes 'Cal Gas Adjustment', 'CI Gas Adjustment', 'RF Ramp Adjustment', and 'Air/Water Check'. The 'Cal Gas Adjustment' option is highlighted. Below this list are 'Start' and 'Cancel' buttons, and an 'Action: Idle' status indicator. On the right, a sub-window titled 'Cal Gas Adjustment' displays the following information:

Last Set Date:	Not Applicable
Result:	Not Adjusted
Ionization Time:	0
Total Ion Count:	0

For the Cal Gas and CI reagent adjustments, the MS parameters are set up to produce and collect characteristic ions of the gas being adjusted. The software then monitors the amplitude and resolution of these ions as the needle valve controlling gas flow is adjusted. The system contains built-in criteria for adjustment of the common CI reagents: methane, iso-butane, ammonia, methanol, and acetonitrile. A gauge display shows whether the flow is too high, too low, or OK, and the spectrum of the gas is displayed to allow the user to judge for himself based on any special needs of his analysis.

Use RF Ramp Adjustment to fine tune the RF by adjusting the tuning screw, that is in the front panel of the 4000 MS, while observing a graphical display of the RF ramp. Proper adjustment will give a smooth linear ramp with minimum amplitude.

Use the air/water check to diagnose the state of the vacuum system. Generally, a narrow peak for the nitrogen ion at m/z 28 indicates you do not have a vacuum leak problem. A broad nitrogen peak may indicate an air leak, but it might also indicate nitrogen contamination in the helium carrier supply. The water level in the system can be estimated from the relative amounts of the m/z 18 and 19 peaks. The more water in the system, the larger the m/z 19/18 ratio.

Module Attributes

The Module Attributes tab has three sections. In the first section select a filament and set the voltage for the electron multiplier. In the second section adjust the high mass calibration for either the normal or fast scan mode. In the third section, enable low mass enhancement, which requires a new mass calibration.

The screenshot shows the 'Module Attributes' tab. It is divided into three main sections:

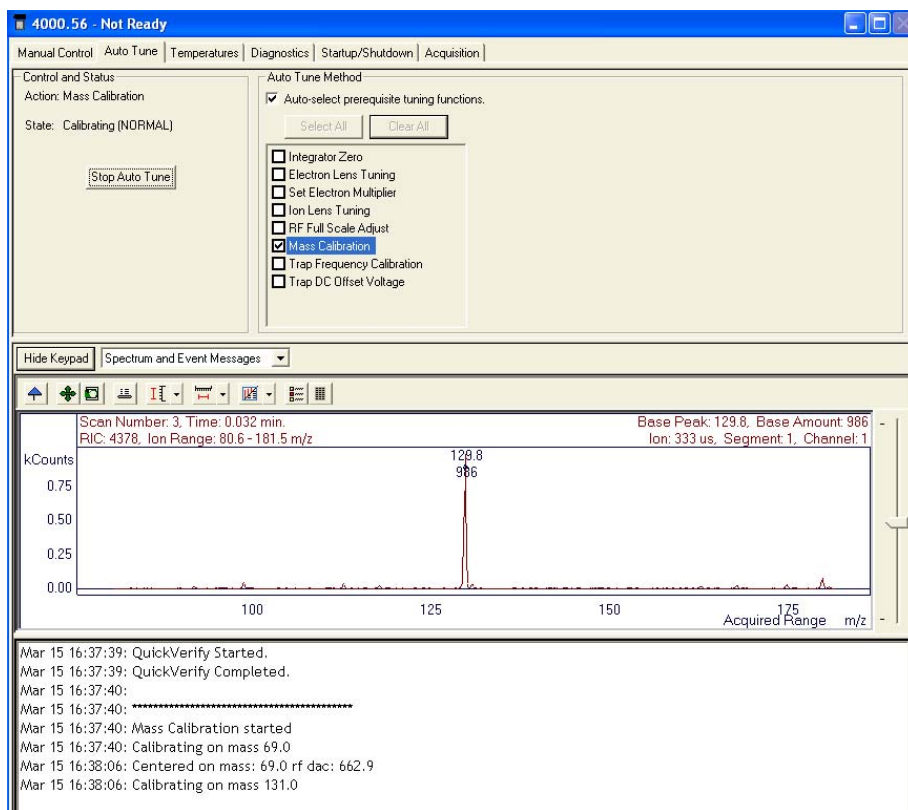
- Filament:** Radio buttons for '1' and '2'.
- Electron Multiplier:** Fields for 'Last Set: Not Applicable', 'Result: Default Values', and 'Voltage: 1200 volts'.
- High Mass Calibration:** A checkbox 'Adjust high mass calibration?' is unchecked. Below it, 'Scan Mode' is set to 'Normal'. 'Expected Mass' and 'Observed Mass' are both set to '1000.00 m/z'.
- Low Mass Enhancement:** A checkbox 'Enable' is unchecked. Below it, a caution message reads: 'CAUTION: Enabling/Disabling Low Mass Enhancement requires new Mass Calibration'.

A 'Defaults' button is located at the bottom center of the tab.

Auto Tune

Auto Tune is an automated process for optimizing the MS 4000 performance over the entire mass range. It is necessary to perform Auto Tune when the instrument is first set up. **Each time the system is shut down, Auto Tune should be performed when it is restarted and after running a bakeout.** Before doing Auto Tune it is advisable to check RF ramp, air/water, and cal gas settings under the Adjustments tab in Manual Control mode. Many tuning features involve using Perfluorotributylamine (FC-43) ions to adjust MS4000 parameters (voltages, gain values, etc.) to optimize the signal. Most Auto Tune functions are performed in EI mode, even if the active method is Internal CI, External CI, or one of the Hybrid CI modes. For the last two cases, the CI volume is automatically retracted from the external ionization source during most Auto Tune routines.

After doing major maintenance, such as cleaning the ion trap or source or switching between internal and external ionization modes, run all Auto Tune routines. Some routines should be run after changing operating conditions, such as trap temperature or helium buffer gas flow rate. Specific routines may be run periodically as part of a system check.



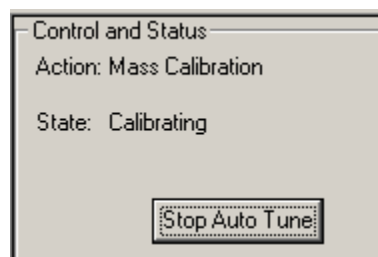
There are three major sections to the Auto Tune window: the Control and Status display, the Spectrum and Event Message windows, and the Auto Tune Method display.

Control and Status

Click Start Auto Tune to start the selected Auto Tune routines. Click Stop Auto Tune to stop Auto Tune.

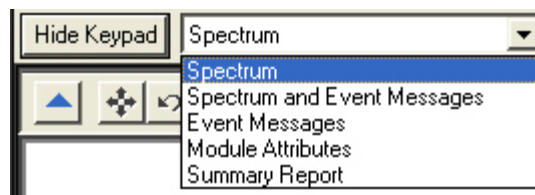
The Action field shows the tuning routine that is being performed.

The State field displays whether the 4000 MS is currently executing an auto tune routine or is idle.

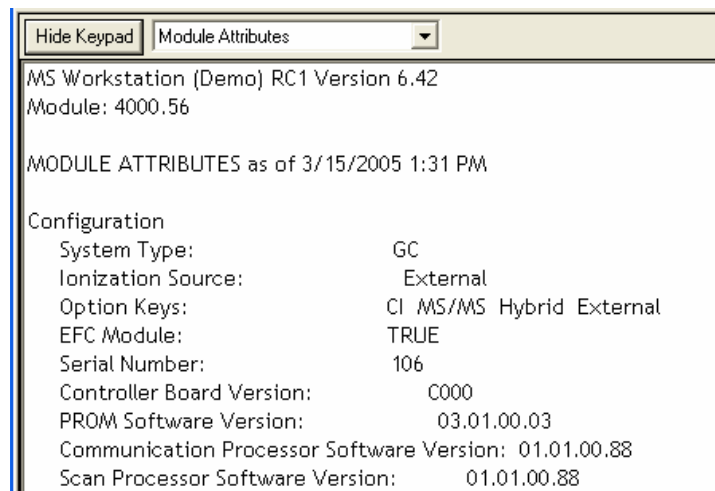


Spectrum and Event Message Window

Select the display for the auto tune tests by selecting from the list. Hide Keypad causes the upper portion of the screen to disappear and the lower portion expand.

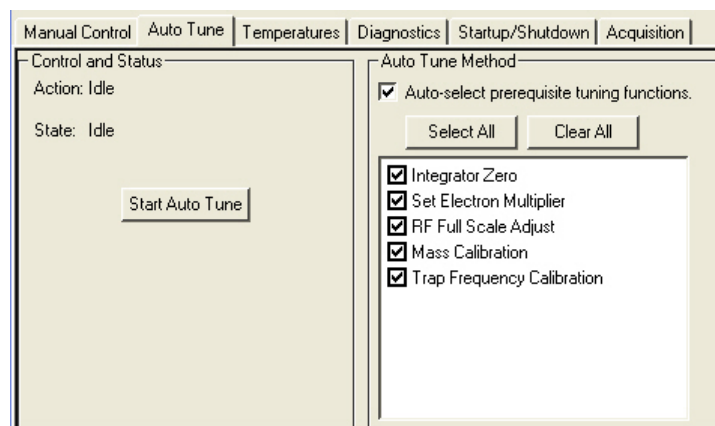


The current Module Attributes is the information stored from the last auto tune. When you are finished performing the auto tune you can view this information, which is stored in an auto tune log.



Auto Tune Methods

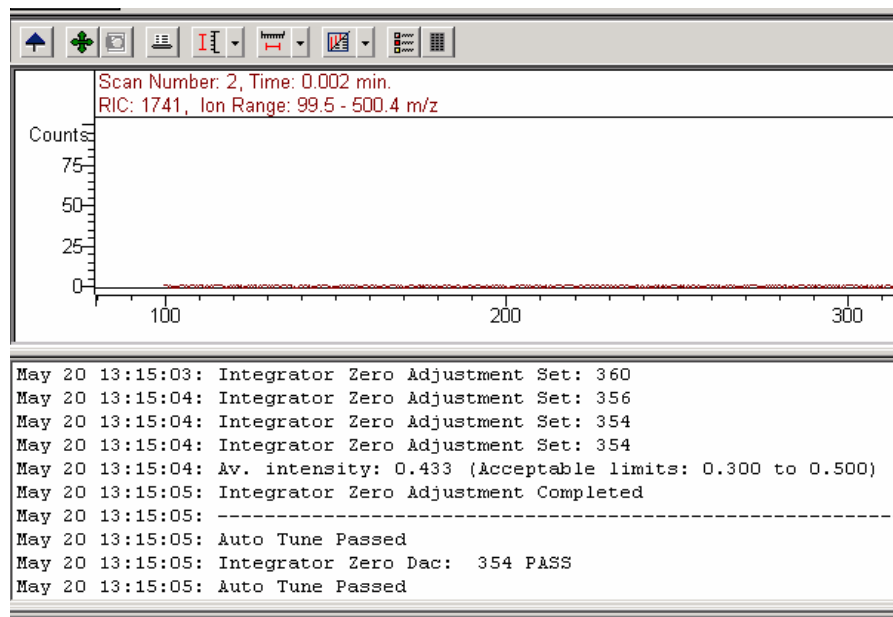
There are various checks that automatically set up, tune and calibrate the mass spectrometer. Each instrument configuration uses different checks.



Auto Tune is generally performed in the order listed, beginning with Integrator Zero. All auto tune methods are selected if you click Select All or cleared if you click Clear All.

To perform Auto Tune select the desired routines, and click Start Auto Tune. Stop auto tune at any time by clicking the Stop Auto Tune.

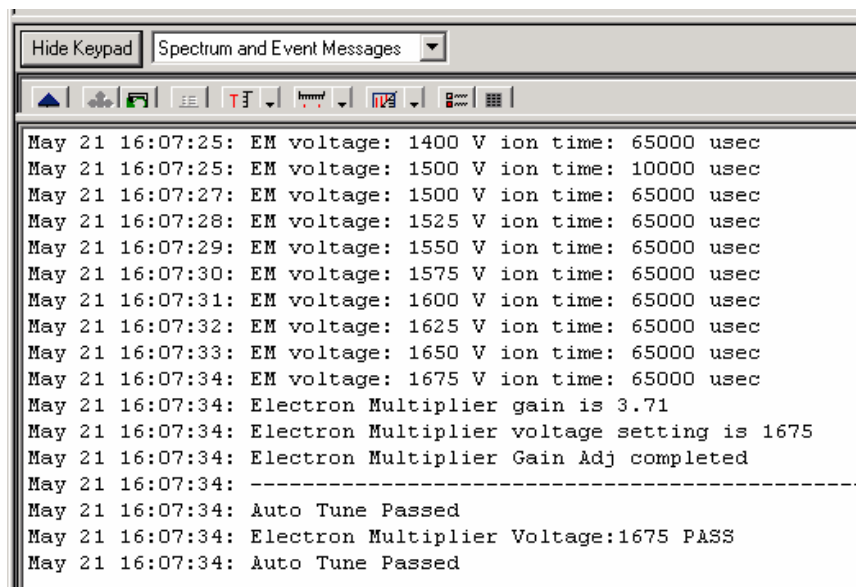
Integrator Zero



The Integrator Zero Adjustment routine sets the signal level coming from the Integrator Circuitry with the filament off. When the filament is off, the major source of signal coming from this circuitry is electronic noise. The Integrator zero is adjusted such that the average value of electronic noise is slightly greater than zero. The acceptance range is between 0.3 and 0.5 counts. The integrator

should then record signals only when ions actually strike the dynode and electrons emitted from the dynode strike the electron multiplier.

Set Electron Multiplier

A screenshot of a software window titled "Spectrum and Event Messages". The window has a toolbar with various icons and a text area displaying a log of events. The log shows a series of "EM voltage" and "ion time" readings, followed by a sequence of messages indicating the completion of the "Electron Multiplier Gain Adj" process and the "Auto Tune" procedure.

```
May 21 16:07:25: EM voltage: 1400 V ion time: 65000 usec
May 21 16:07:25: EM voltage: 1500 V ion time: 10000 usec
May 21 16:07:27: EM voltage: 1500 V ion time: 65000 usec
May 21 16:07:28: EM voltage: 1525 V ion time: 65000 usec
May 21 16:07:29: EM voltage: 1550 V ion time: 65000 usec
May 21 16:07:30: EM voltage: 1575 V ion time: 65000 usec
May 21 16:07:31: EM voltage: 1600 V ion time: 65000 usec
May 21 16:07:32: EM voltage: 1625 V ion time: 65000 usec
May 21 16:07:33: EM voltage: 1650 V ion time: 65000 usec
May 21 16:07:34: EM voltage: 1675 V ion time: 65000 usec
May 21 16:07:34: Electron Multiplier gain is 3.71
May 21 16:07:34: Electron Multiplier voltage setting is 1675
May 21 16:07:34: Electron Multiplier Gain Adj completed
May 21 16:07:34: -----
May 21 16:07:34: Auto Tune Passed
May 21 16:07:34: Electron Multiplier Voltage:1675 PASS
May 21 16:07:34: Auto Tune Passed
```

Before starting the EM gain algorithm, the RF Full Scale is adjusted so that the cal gas ion at m/z 502 is calibrated. The Set Electron Multiplier method adjusts the multiplier voltage and ion time to look at low-mass fragments from the unstable m/z 502 ion. The objective is to set the multiplier voltage such that each electron emitted by the dynode will create a known signal from the multiplier. The gain in this example is 3.71×10^5 .

When the 10^5 gain voltage of your electron multiplier reaches 2000V you should order a new multiplier to have on hand. When the 10^5 gain voltage exceeds 2500V you should install a new multiplier.

Electron Lens Tuning

In typical external ion sources, electrons are continuously directed into an ion volume containing the sample to be ionized. Eventually the interior surface of the ion volume becomes contaminated by material that can charge up and disturb the extraction of ions from the ion source. To minimize ion volume contamination, the 4000 MS pulses electrons into the ion volume only when needed for ionization. Two plates bracket the filament to control electron pulsing. The plate closest to the ion volume is the electron lens, and the plate away from the ion volume is the repeller lens. The electron lens is set positive and the repeller lens negative to admit electrons to the ion volume only during the ionization period. Otherwise the voltages are reversed to exclude electrons from the ion volume. Tuning of electron/repeller lens voltages is important for linear signal response and filament lifetime.

The four variables to deal with are electron lens ON and OFF voltages, and repeller lens ON and OFF voltages. ON and OFF refer to ionization ON and OFF. The electron lens ON voltage is set to optimize electron transmission to the ion volume, electron lens OFF voltage is set to thoroughly exclude electrons from the

ion volume. The Repeller Lens ON and OFF voltages balance the emission current.

Electron/Repeller Lens Adjustment: Electron lens tuning involves measuring the transient behavior of the emission current immediately after the lenses have been switched on or off. If the lenses are unbalanced, the emission current will change in time and be proportional to the imbalance. If the balance is outside the range of 200 to 300 μA , the algorithm will search the optimal values by changing values of four variables one at a time. If it fails to find the best voltage setting for lens tuning, auto tune will generate an error message, and restore the last values in the instrument.

When the Electron Lens Tuning Box is clicked, an additional “Turn on CI gas flow during tune” option appears. For CI methods in External or Hybrid mode, the electron/repeller lens must be tuned with the CI plunger (CI volume) in place and the CI gas turned on. The user should adjust the CI gas flow in Manual Control before this tune function is done.

Ion Lens Tuning

The Ion Lens system consists of three lenses (Lens 1, 2 & 3). These lenses are tuned using Cal Gas ions at m/z 131 and 414. Optimum voltages are determined based on weighted intensities of the two ions.

RF Full Scale Adjust

This routine should always be run before mass calibration and trap function calibration. The RF Full Scale Adjust routine coarsely adjusts the mass calibration of the ion trap. Successive adjustments are performed on PFTBA calibration gas ions at m/z 69, 414 and 614. For this algorithm to work properly, it is important that the specified ions are stable and present in sufficient intensity (>20 counts) under normal scanning conditions.

Mass Calibration

When you initiate this routine, the calibration gas valve is opened and the electron multiplier, filament, and RF voltage are turned on. The algorithm then uses the calibration compound ions at m/z 69, 131, 264, 414, 464, and 614 to create a calibration curve. The nitrogen peak (m/z 28) is not used for calibration. A fixed offset at m/z 0 has been empirically determined to give proper mass assignments between m/z 10 and 69. The calibration is extrapolated from the highest observed calibration ion to the maximum mass (m/z 1000). Mass calibration is performed in both normal scanning (5,000 u/sec) and fast scanning (10,000 u/sec) modes.

Factors Affecting Mass Calibration

For this routine to work properly, the calibration ions must be present in sufficient intensity (> 20 counts) and stable under normal scanning conditions.

Temperature changes can shift the mass calibration and this procedure should be initiated after the trap has reached its equilibrium point (approximately 2 hours after the set-point temperature has been established). For External and Hybrid modes, changing the trap damping gas flow may require both Mass Calibration Trap Frequency Calibration. There could also be subtle effects on mass assignments after ion source temperature changes.

Trap Frequency Calibration

When the mass calibration has been completed, the trap frequency calibration must be performed. This routine determines resonant frequencies required for the generation of notched waveforms used in most modes of operation. In particular, the resonant frequencies for FC-43 ions at m/z 69 and 414 are determined. The method takes several minutes to complete.

NOTE: Trap Frequency Calibration should be performed each time a mass calibration is done.

Upon completion of Trap Frequency Calibration, the Event Message Window displays the secular frequency values for the two trap function calibration points. Nominal values are 249 kHz and 248 kHz, respectively. These values can vary up to ± 4 kHz between instruments.

Trap DC Offset Voltage

The trap offset is a DC voltage applied to the ion trap electrodes (end caps and ring electrode) to facilitate the injection of the externally generated ions into the trap. The intensity of the m/z 414 ion from FC-43 is used to maximize the signal, thus optimizing for the midpoint of the mass range. The trap offset has a range from 3 to 15 volts. Lower voltages are useful if an increase in the relative abundance at the low end of the mass range is desired. Furthermore, if your application involves more fragile compounds, then a lower setting can produce a situation where less fragmentation of the molecular ion is observed.

Temperature(s)

The Temperatures tab allows you to set the temperature zones of the mass spectrometer for both normal operation and Bakeout, and to initiate and terminate Bakeout. The Status display can be set to display either Event messages regarding the execution of the temperature commands, or Module Attributes detailing the instrument configuration, temperature setpoints, and results of manual and Autotune procedures.

The screenshot shows a software window titled "4000.56 - Not Ready". It has a menu bar with "Manual Control", "Auto Tune", "Temperatures", "Diagnostics", "Startup/Shutdown", and "Acquisition". The "Temperatures" tab is active, showing two sub-tabs: "Analysis" and "Bakeout".

Control and Status:

- Conditions: Analysis
- State: Equilibrating
- Hold Time: 0.00 hours
- Buttons: Start Bakeout, Save, Restore

Analysis / Bakeout Settings:

Parameter	Value	Unit
Trap Temperature:	150	C
Manifold Temperature:	50	C
Transfer Line Temperature:	170	C

Operating Conditions:

Parameter	Value	Unit
Trap Temperature:	0	C
Manifold Temperature:	0	C
Transferline Temperature:	0	C

Analysis

Temperature setpoints entered in the Analysis dialog will be executed at all times when the instrument is not in Bakeout mode. It is recommended that temperatures be allowed to stabilize for 2 to 3 hours in Analysis mode before tuning or acquiring data.

Trap Temperature

A temperature between 50 and 250 °C may be entered. The default is 150 °C. It is recommended that the trap be operated at the lowest temperature consistent with proper chromatographic performance, typically about 50°C less than the maximum column temperature employed.

Manifold Temperature

A temperature between 0 and 120 °C may be entered. However, a setpoint near or below the ambient temperature will not be achieved, as there is no cooling available for the manifold. The default setpoint is 35 °C. Manifold heating is used primarily for Bakeout.

Transfer Line Temperature

A temperature between 0 and 350 °C may be entered. However, a setpoint near or below the ambient temperature will not be achieved, as there is no cooling available for the transfer line. The default setpoint is 170 °C. The transfer line should not be heated above the maximum operating temperature of the capillary column installed in the GC.

Save/Restore

These buttons become active only after a change has been made in the Temperatures dialog. Pressing Save will execute any changes to setpoints made since the last Save or the opening of the dialog. Pressing Restore will discard any changes made since the last Save or the opening of the dialog, and will restore setpoints to the *status quo ante*. If changes are pending when you leave the Temperature dialog, you will be prompted to save or discard the changes.

Bakeout

The Bakeout tab allows you to set temperatures and a timer to control the instrument in Bakeout mode. These parameters may be changed only while Bakeout is not active, or stopped.

Bakeout Trap Temperature

A temperature between 50 and 250 °C may be entered. The default is 150 °C, and a typical setpoint for Bakeout is 220 °C.

Bakeout Manifold Temperature

A temperature between 0 and 120 °C may be entered. The default setpoint is 35 °C.

Transfer Line Temperature

A temperature between 0 and 350 °C may be entered. The default setpoint is 170 °C. The transfer line should not be heated above the maximum operating temperature of the capillary column installed in the GC.

Hold Time

The Hold Time establishes the duration of the Bakeout, starting when the system components reach their Bakeout temperature setpoints. Values from 0 to 72 hours may be entered. The default is 8 hours.

Start/Stop Bakeout

The **Start Bakeout** button activates the Bakeout temperature setpoints and enables the Bakeout timer when the setpoints are reached. Stop Bakeout resets the temperature setpoints to the Analysis values and resets the Bakeout timer.

Diagnostics

The Diagnostics tab provides control and status for monitoring and diagnosing nearly every system in the Mass Spectrometer.

The screenshot shows the '4000.56 - Not Ready' software window with the 'Diagnostics' tab selected. The interface is divided into several sections:

- Control and Status:** Shows 'State: Idle' and 'Function: Monitoring Status'. A 'Start Diagnostic' button is present.
- Monitoring / Diagnostic Tests:** Contains two sub-sections:
 - Trap - On/Off:** Includes checkboxes for 'Ion Source' (checked), 'Multiplier/Dynode' (checked), and 'RF Control' (checked). Under 'Ion Source', 'Filament 1' is selected. Under 'RF Control', 'Waveform' is selected.
 - Ion Gauge:** Includes a checked 'Ion Gauge' checkbox and 'Filament 1' is selected.
- Monitor Window and Event Messages:** A dropdown menu.
- Vacuum System:** Displays 'Turbo Pump' status as 'Normal' with 'Speed: 100 %', 'Current: 242 mAmps', 'Power: 10 Watts', and 'Temp: 26 C'.
- Ion Gauge:** Displays 'Pressure: 21.8 uTorr', 'Filament 1: OK', and 'Filament 2: Untested'.
- Roughing Line Pressure:** 18 mTorr.
- Trap Damping Gas Flow:** 1.0 mL/min.
- Trap:** Displays 'Multiplier: -2152 Volts' and 'Dynode: -10374 Volts (+ ions)'.
- Waveform System:** Displays 'Entrance Endcap Trapping Field Dipole: 0 %', 'Exit Endcap Trapping Field Dipole: 0 %', 'Dipole Supplemental Waveform: 103 %', and 'Quadrupole Supplemental Waveform: 101 %'.
- Temperatures:** Displays 'Entrance Endcap: 100 C', 'Exit Endcap: 100 C', 'Manifold: 53 C', 'Transferline: 174 C', and 'Source: 154 C'.
- Ion Source:** Displays 'Emission: 24 uAmps', 'Balance: 1244', 'Deviation: 1.4 uA', 'Filament Bias: -70.6 Volts', 'Filament 1: OK', 'Filament 2: Untested', 'Lens 2: -90 Volts / 120 Volts', 'Electron Lens: 95 Volts / -95 Volts', 'Repeller Lens: -171 Volts / 159 Volts', 'Lens 1: -26.4 Volts', 'Lens 3: -17.8 Volts', 'Ring Offset: -7.4 Volts', and 'Endcap Offset -7.5 Volts'.

Control and Status

The Control and Status area indicates the state of the Diagnostics system (Idle or Running) as well as the function the Diagnostics system is currently in. The normal function is Monitoring Status but cycles through various test functions once the Start Diagnostics button is clicked.

Monitoring Tab

This tab provides the user with choices for which Ion Trap and Ion Gauge components will be monitored. Selecting the Ion Source checkbox activates the Emission, Balance and Deviation parameters in the Ion Source data area (lower Monitor Window section). Selecting the Multiplier/Dynode checkbox activates the Multiplier and Dynode parameters in the Trap data area (lower Monitor Window section). Selecting the RF Control checkbox activates the Waveform System data area (lower Monitor Window section). Underneath the RF Control checkbox, there are two radio buttons (RF and Waveform) that indicate which elements of the Waveform System the user wishes to monitor. Selecting the RF radio button activates the Entrance End Cap Trapping Field Dipole percentage and Exit End Cap Trapping Field Dipole percentage. Selecting the Waveform radio button activates the Dipole Supplemental Waveform percentage monitor and the Quadrupole Supplemental Waveform percentage monitor. Selecting the Ion Gauge checkbox adds the Ion Gauge to the components that will be monitored and tested. The user may also select which filament is to be monitored (Filament 1 or Filament 2) by selecting the appropriate radio button.

Diagnostic Tests Tab

The Diagnostic Tests Tab provides a checklist of which diagnostic tests should be run. There are currently three diagnostic tests that can be run: System Verification, Waveform Generator, and the Scan and Acquisition Test. There are Select All and Clear All buttons.

Display

You may choose to display either Monitor Window and/or Event Messages by choosing the desired display from the drop-down menu next to the Keypad button. Event Messages are a log of actions and events which have occurred during the Diagnostic and Monitoring process. In most cases, there will be messages indicating if certain tests Started, Passed and Completed. The Monitor Window displays the status and values of the following systems: Vacuum, Trap, Waveform, Temperatures, and Ion Source. Have both Monitor Window and Event Messages selected at the start of any diagnostics and monitoring.

After you select which components to activate and diagnose, click Start Diagnostic in the Control and Status Area. The Monitor Window has the following areas that indicate the value and status of their respective components.

Vacuum System

This indicates the status of the Turbo Pump, Ion Gauge, and Foreline Pressure. The Turbo Pump area indicates the pump status (Stopped, Running), the pump speed, the pump current (in mAmps) and pump power (in Watts). The Ion Gauge

area indicates the ion gauge pressure (in μTorr) and the status of the Filaments (OK, open, broken). The Foreline Pressure is indicated in mTorr.

Trap

The Trap area in the Monitor Window is active only if the Multiplier/Dynode checkbox has been selected in the Monitoring Tab (top level). The Trap Area (if active) indicates the Multiplier voltage and Dynode voltage (in Volts).

Waveform System

The Waveform System area is active when the user has chosen RF Control in the Monitoring Tab (top area). The percentages of the Entrance End Cap Trapping Field Dipole and Exit End Cap Trapping Field Dipole are indicated when the RF radio button is chosen underneath the RF Control checkbox. The percentages of the Dipole Supplemental Waveform and Quadrupole Supplemental Waveform are indicated when the Waveform radio button is chosen underneath the RF Control checkbox.

Temperatures

The Temperatures area indicates the temperatures of the Entrance End Cap, Exit End Cap, Manifold, and Transferline (in degrees C).

Ion Source

The Ion Source Area indicates the values and status for the following components:

Emission current (in μAmps) indicates the Ion Source emission current.

Balance of the ion source (in $\mu\text{Amps per second}$). This is a proprietary diagnostic used by Varian to determine the quality of the ion source and for instrument tuning and troubleshooting.

Deviation of the Ion Source Balance (in μAmps) relates to the proprietary diagnostic used to determine the quality of the ion source and for instrument tuning and troubleshooting.

Filament Bias of the ion source (in Volts).

Filament 1, Filament 2 status will indicate either OK or open (broken or faulty filament).

Ion Source **Gate** status and voltage indicates the voltage for the gate when it is On and Off.

Emission, Balance, and Deviation will only be active if the Ion Source checkbox has been selected in the Trap area of the Monitoring Tab.

Startup/Shutdown

The Startup/Shutdown tab provides status and control for the vacuum system, the heated zones, and the damping gas pneumatics.

The screenshot shows a software interface titled "4000.56 - Not Ready" with a tabbed menu at the top: Manual Control, Auto Tune, Temperatures, Diagnostics, Startup/Shutdown (selected), and Acquisition. The main area is divided into three columns: Status and Control, Current Set Points, and Operating Conditions. The Status and Control column contains a "Shut Down" button, a "Vacuum System" status of "Ready", "Pneumatics" with "Damping Gas" set to "Off" and a "Turn On" button, and "Getter Control" with "Heater" set to "Off" and a "Turn On" button. The Current Set Points column shows "Heated Zones" with "Trap Temperature: 150 C", "Manifold Temperature: 35 C", and "Transferline Temperature: 170 C"; "Vacuum System" with "Pump Spin Speed: 100 %"; "Pneumatics" with "Flow Rate: OFF"; and "Getter Control" with "Temperature: OFF". The Operating Conditions column shows "Heated Zones" with "Trap Temperature: 150 C", "Manifold Temperature: 34 C", and "Transferline Temperature: 170 C"; "Vacuum System" with "Pump Spin Speed: 100 %" and "Current: 220 mAmps"; "Pneumatics" with "Flow Rate: 0.0 mL/min." and "Inlet Pressure: 101 PSI"; and "Getter Control" with "Temperature: 42 C". At the bottom, there is a "Hide Keypad" button, an "Event Messages" dropdown menu, and a log showing "Jan 13 13:45:43: Turning Damping Gas ON." and "Jan 13 13:46:00: Turning Getter ON."

Start Up/Shutdown

When the system is shut down, the Startup button is displayed. Pressing the **Startup** button starts the pumping system and the zone heaters. The status display shows the heated zone setpoints and current temperatures, along with information on the turbo pump spinup and current draw.

After the system has been started, the ShutDown button is displayed. Pressing this button begins an orderly shutdown of the vacuum system and the heated zones. The progress of the shutdown can be monitored on the status displays.

Vacuum System status indicating whether the Vacuum system is Ready. To adjust damping gas, see the Manual Control Tab instructions. See Startup/Shutdown Tab instructions to turn on the damping gas.

Pneumatics Damping Gas Turn On/Turn Off

If you have the Damping gas option installed, the **Turn On/Turn Off** button on this dialog will allow you to control the damping gas flow. The flow rate is set in the Manual Control section under the Module Attributes tab.

Getter Control Heater Turn On/Turn Off

If you have the Damping Gas option installed, the **Heater Turn On/Turn Off** button allows you to control the heater. The heater cannot be turned on until the Damping Gas is turned on. The getter temperature setpoint is fixed at 400 °C.

Current Set Points

The Current Set Points area displays the set points for Heated Zones, the Vacuum System, Pneumatics and Getter Control.

Heated Zones area indicates what temperature set points have been set for the Trap, Manifold and Transferline (in degrees C).

Vacuum System indicates what Pump Spin Speed has been set. It is displayed as a percentage of the ideal pump spin speed (100%).

Pneumatics indicates if the Flow Rate is set or not (ON or OFF).

Getter Control indicates if the temperature of the Getter Control has been set or not (ON or OFF).

Operating Conditions

The Operating Conditions area displays the actual conditions so you can compare the desired conditions (current set points) with the actual operating conditions.

Heated Zones area indicates the actual temperatures of the Trap, Manifold and Transferline (in degrees C).

Vacuum System area indicates the actual Pump Spin Speed and current of the vacuum system (in mAmps).

Pneumatics area displays the actual Flow Rate (in mL/min) and Inlet Pressure (in psi).

Getter Control area displays the actual temperature of the Getter Control (in degrees C).

Display

You may choose to display either Event Messages or Module Attributes. Event Messages are a log of actions and events which have occurred during the Startup or Shutdown process. Module Attributes displays the current instrument configuration, temperature setpoints, date and results of the most recent manual adjustments, date and results of the most recent Autotune procedures, and a log of any tuning errors encountered.

Acquisition

Selecting the **Acquisition** button activates the 4000 module for analysis. The module loads the information for the active method so that it is ready to collect data according to the conditions prescribed in the method. You will see a screen similar to the following.

4000.01 - Ready		
Manual Control Auto Tune Temperatures Diagnostics Startup/Shutdown Acquisition		
Status And Control	MS Method	Operating Conditions
Run Time: 0.00 minutes	Segment Number: 1 of 2	Data File:
End Time: 10.00 minutes	Segment Description: FIL/MUL DELAY	Scan Number: 0
Start Acquisition	Scan Description: Filament Off	Ionization Time: 0
Edit Method	Scan Range: 20 - 25	Ion Count: 0
☒ Ready		
☒ No Fault		
☐ Wait For ReadyIn		

The red lights turn to green to indicate that the 4000 module is ready for analysis. If the GC and/or AutoSampler are not ready you will see a Not Ready message at the top of the screen. Once the GC and AutoSampler come to a ready state, the Not Ready message will change to Ready. To determine the individual ready states of the components you can go to the top pull-down menu under Windows and see the states for the 4000, 3800, and AutoSampler modules. Once all components are ready, you are ready to start an analysis.

Start Acquisition

Click Start Acquisition to immediately start the 4000 MS collecting data with the currently active method.

Edit Method

Click Edit Method to open the Method Builder application and loads the currently active method.

Wait for ReadyIn

If this box is checked, the state of the ReadyIn sync port is included in the checks the system performs to determine if the system is Ready. The ReadyIn port typically would be used to interface to an external device such as a Purge and Trap sampler.

End Time

The End Time is initially set equal to the end time of the active method. You may change the end time to either shorten or prolong the end time of the data acquisition. Note that this does not affect the end time of the GC method.

Status Display

Spectrum Toolbar



Click the green arrow to hide the toolbar. Clicking on the upper left of the full screen display restores the toolbar.



Click the full scale button to cause the spectrum to be normalized in the vertical and horizontal directions. Double clicking on the lower left corner of the scale has the same effect.



Click the previous scale button to restore previous scalings.



Click the spectrum plot button to open a window with the spectrum.



Click the scale button to choose between Auto Scale Intensity and Fixed Scale Intensity.



Click the mass range button to choose between Acquired Mass Range, Fixed Mass Range, or Maximum.



Click this to switch between graphics and text displays of ion intensity and status information.

Chromatogram Toolbar



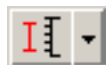
Clicking on the green arrow will hide the toolbar. Clicking on the upper left of the full screen display restores the toolbar.



Clicking on the full scale button will cause the chromatogram to be normalized in the vertical and horizontal directions. Double clicking on the lower left corner of the scale has the same effect.



Clicking on the previous scale button will restore previous settings.



Clicking on the scale button allows you to choose between Auto Scale Intensity and Fixed Scale Intensity.



When selected, this button allows you to click on a feature in the chromatogram and have the spectrum at that point displayed in the Spectrum window. When this button is not selected, the Spectrum window will display and continuously update the most recently acquired spectrum.



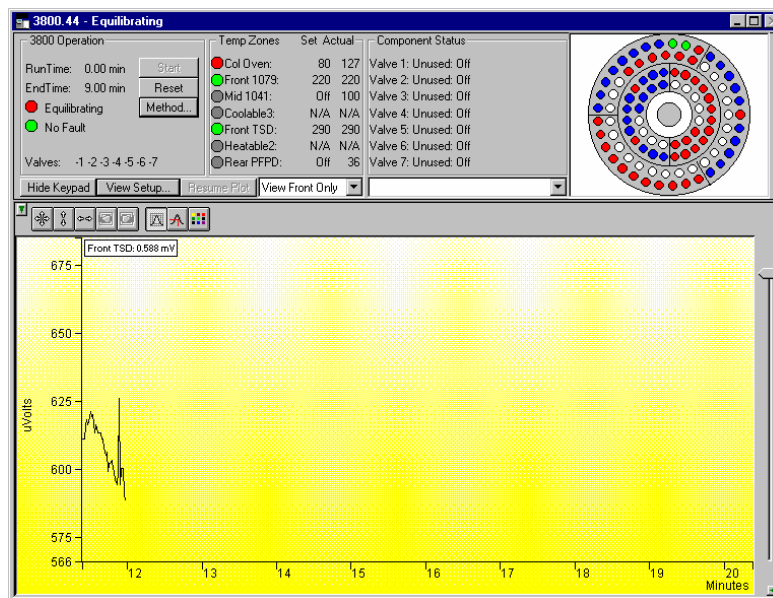
This links MS Data Review. If an acquisition is running when the button is pressed, MS DR will be opened with the current acquisition as the active chromatogram; if MS DR was already open, the current acquisition will be added as the active chromatogram. If no acquisition is running and the chromatogram window is blank when this button is pressed, MS DR will be opened with a file select dialog to select the active file.



Click to display a plot of the Intensity of the Base Peak. To display the RIC trace and the Intensity of Base Peak plot at same time, enter IBP in the Ions field. Clicking the Base Peak icon does not affect the display of the extracted ion chromatogram.

3800 GC System Control Command Reference

Status and Control Window



The 3800 Status and Control window is divided into five display areas. These are the 3800 Operation display, the Temp. Zones Status display, the Component Status display, the 8400/8410 Carrousel display, and the Chromatogram display.

The left-most display area is the 3800 Operation display, which contains information about the current run and the overall state of the GC.

The next display area to the right is the Temperature Zones Status display, which shows the setpoint and current actual temperature of each heated zone in the GC.

Next is the Component Status display. You select the component for which you wish to see more detailed status information, including ready and fault status for the selected component. You can change the component being displayed using the selection box below the display area, or by clicking on the corresponding temperature zone entry in the temperature zone status display.

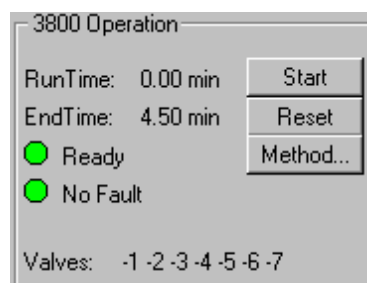
3800 GCs that have an 8400 AutoSampler or 8410 AutoInjector installed will have another display area, the 8400/8410 Carrousel display, that shows a

graphical representation of the 8400 or 8410 Carrousel. This display graphically shows which vials are to be sampled, which vials have been sampled, which vial is currently being sampled.

Below these display areas is the Chromatogram display. A selection box above the chromatogram allows you to select the channels for which you want to view the real-time chromatogram.

3800 Operation Display

The 3800 Operation display is in the left portion of the 3800 Status and Control window.



Item	Description
Run Time	Shows the elapsed time in minutes since the beginning of the run. The maximum run time is 999.99 minutes.
End Time	Shows the time at which the run will end.
State	Ready, Running, Stabilizing, Equilibrating, Computing, Needs Monitor, Monitoring, Sampling, Connecting, or Not Ready Shows the state of the GC. The light appears green if the GC is Ready or Running. Otherwise, the light is red.
Fault/No Fault indicator	No Fault (green light) or Fault (red light). When a hazardous or disabling fault occurs, any run in progress will halt and the affected component will shut down. If the fault is recoverable, then the run is not terminated.
Valves	Displays the 3800 GC valve status. A plus sign ('+') precedes the valve number if its state is energized. A minus sign ('-') precedes the valve number if the valve is in the default state or if the valve is unused.
Start	If the system is Ready, starts the GC Method and sends a start signal to the GC. Equivalent to pressing Start on the front panel of the GC. The Start button is disabled unless the 3800 GC is in the Ready state.
Reset	Resets the GC, advancing it to the next state. If it is RUNNING, the GC aborts the current run, goes to the READY state, and continues with the next injection in the Sequence. Equivalent to pressing Stop on the front panel of the GC.
Method...	Opens the Method Builder application for editing the active 3800 GC method.

Temperature Zones Status Display

Temp Zones	Set	Actual
Col Oven:	57	57
Front 1079:	50	50
Mid 1079:	50	50
Rear 1079:	50	50
Front TCD:	50	50
Mid FID:	50	50
Rear ECD:	50	50

The temperature zones status display is in the middle portion of the 3800 Status and Control window. The display includes the setpoints and actual temperatures of the column oven, the three coolable zones, and the three heatable-only zones of the 3800 GC. The display is "Fault" if a fault occurs in the zone component or "Off" if the zone oven is off. If the zone has not been configured by the 3800 GC, then "N/A" is displayed for the setpoints and actual temperature. Clicking on one of the temperature zone entries will display the corresponding component status in the Component Status Display.

The LEDs indicate the status of each temperature zone. If the zone is not configured or the zone oven is off, the LED is gray. If the temperature zone is Not Ready or there is a fault, then the LED is red. Otherwise, the LED is green.

Component Status Display

Column Oven Status

Ready: Yes Fault: No
Oven Power: On Coolant: Off
Setpoint: 65 C Actual: 65 C
Stabilization Time 0.50 min

Enable Column Oven Coolant at 50 C
Timeout: 20.00 min Timed Out: No

Column Oven Status

Front Injector Status
Middle Injector Status
Rear Injector Status
Front Flow/Pressure Status
Middle Flow/Pressure Status
Rear Flow/Pressure Status
Column Oven Status
Front Detector Status
Middle Detector Status
Rear Detector Status
Front Detector EFC Status
Middle Detector EFC Status
Rear Detector EFC Status
Autosampler Status

The Component Status display is in the right portion of the 3800 Status and Control window. Select the component status to view with the drop down list box below the display area.

Column Oven Status

Item	Description
Ready	Yes or No. The column oven is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the column oven component has occurred.
Oven Power	On or Off. Displays if the column oven component is turned off or on at the 3800 GC.
Coolant	On or Off. Displays if the column oven coolant is enabled in the active method.
Setpoint	-99 to 450 °C Displays the column oven programmed temperature setting in the active method.
Actual	-99 to 450 °C Displays the actual temperature of the column oven at the 3800 GC.
Stabilization Time	0.00 to 10.00 min Shows the 3800 GC column oven stabilization time in minutes. The stabilization time counts down from the programmed method value and then displays 0.00 minutes when the stabilization period is over.
Enable Column Oven Coolant at	30 °C to 450 °C Displays the programmed column oven temperature at which the column oven coolant is enabled.
Timeout	0.01 min to 999.99 min Displays the programmed coolant time-out value in the active method for the column oven.
Timed Out	Yes or No. Displays Yes if the coolant time-out period has elapsed for the column oven.

AutoSampler Status

This option is only shown on 3800 GCs that have an 8400 AutoSampler or 8410 AutoInjector connected to them.

Item	Description
8400/8410 State	Ready or Running
Current Vial	0 through 99 for 8400 AutoSampler 1 through 21 for 8410 AutoInjector
Plunger Strokes	Displays the number of times that the syringe plunger has been stroked up and down. This can be used to help monitor syringe wear and plan for syringe replacements.
Injection Mode	Shows the injection mode specified in the active 3800 GC method.
Injection number: x of y	where x and y are any number between 1 and 9. Shows the number of the injection that is currently being run and the total number of injections scheduled for that sample.
Injectors used	Pos 1 Pos 2 Pos 1 then 2 Pos 2 then 1 Shows the injector positions that the 8400/8410 will use for current run.
2nd Injection	Duplicate or Advance This field is only displayed when the 8400/8410 is in Dual Mode or Duplicate Mode.
Tray Orientation (8410 AutoInjector Only)	Vials 1 through 10, Vials 11 through 16, or Vials 17 through 21 Shows the range of vials accessible from the current 8410 carousel location.

Sample Delivery Status

Item	Description		
Valve 1 through Valve 7	If the valves are configured in the 3800 GC, displays the assigned valve name and current state at the 3800 GC. The possible valve names, default state of the valve, and the energized state of the valve are listed below.		
	Name	Default State	Energized State
	Unused	Off	On
	Front Split	On (split)	Off (splitless)
	Middle Split	On (split)	Off (splitless)
	Rear Split	On (split)	Off (splitless)
	Gas Sampling	Fill	Inject
	Liquid Sampling	Fill	Inject
	Sample	Off	On
	Internal Std	Off	On
	Surrogate Std	Off	On
	Series Bypass	Series	Bypass
	Backflush to Detector	Forward	Backflush
	Backflush to vent	Forward	Backflush
	Column Selection	Column 1	Column 2
	Injection to detector	Backflush	Inject
	Injection to vent	Backflush	Inject
	Alternate injection	Column 1	Column 2
	Simultaneous Injection	Fill	Inject
	Methanizer Bypass	Series	Bypass
	Sample Preconcentration Trap	SPT Desorb	SPT Trap
	External Event A	Off	On
	External Event B	Off	On
	External Event C	Off	On
	External Event D	Off	On

1177 Injector Status

Item	Description
Ready	Yes or No. The 1177 injector oven is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the 1177 injector component has occurred.
1177 Oven	On or Off. Displays if the 1177 injector oven component is turned off or on at the 3800 GC. The 1177 injector is an isothermal injector.
Setpoint	50 to 450 °C Displays the 1177 injector oven programmed temperature setting in the active method.
Actual	50 to 450 °C Displays the actual temperature of the 1177 injector oven at the 3800 GC.
Split State	On or Off. Displays the current split state of the 1177 injector method.
Split Ratio	1 to 10000 or Off Displays the current split ratio of the 1177 injector method. The split ratio is defined as the (Column Flow + the Split Flow) / the Column Flow.

1079 Injector Status

Item	Description
Ready	Yes or No. The 1079 injector oven is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the 1079 injector component has occurred.
1079 Oven	On or Off. Displays if the 1079 injector oven component is turned off or on at the 3800 GC.
Coolant	On or Off. Displays if the 1079 injector oven coolant is enabled in the active method.
Setpoint	-99 to 450 °C Displays the 1079 injector oven programmed temperature setting in the active method.
Actual	-99 to 450 °C Displays the actual temperature of the 1079 injector oven at the 3800 GC.
Split State	On or Off. Displays the current split state of the 1079 injector method.

Item	Description
Split Ratio	1 to 10000 or Off Displays the current split ratio of the 1079 injector method. The split ratio is defined as the (Column Flow + the Split Flow) / the Column Flow.
Enable 1079 Injector Coolant at	30 °C to 450 °C Displays the programmed injector oven temperature at which the 1079 injector oven coolant is enabled.
Timeout	0.01 min to 999.99 min Displays the programmed coolant time-out value in the active method for the 1079 injector oven.
Timed Out	Yes or No. Displays Yes if the coolant time-out period has elapsed for the 1079 injector oven.

1041/1061 Injector Status

Item	Description
Ready	Yes or No. The 1041/1061 injector oven is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the 1041/1061 injector component has occurred.
1041/1061 Oven	On or Off. Displays if the 1041/1061 injector oven component is turned off or on at the 3800 GC. The 1041 and 1061 injectors are isothermal injectors.
Setpoint	50 to 450 °C Displays the 1041/1061 injector oven programmed temperature setting in the active method.
Actual	50 to 450 °C Displays the actual temperature of the 1041/1061 injector oven at the 3800 GC.

SPT Injector Status

Item	Description
Ready	Yes or No. The SPT injector oven is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the SPT injector component has occurred.
SPT Oven	On or Off. Displays if the SPT injector oven component is turned off or on at the 3800 GC.
Coolant	On or Off. Displays if the SPT injector oven coolant is enabled in the active method.

Item	Description
Setpoint	-99 to 450 °C Displays the SPT injector oven programmed temperature setting in the active method.
Actual	-99 to 450 °C Displays the actual temperature of the SPT injector oven at the 3800 GC.
Enable SPT Coolant at	30 °C to 450 °C Displays the programmed injector oven temperature at which the SPT injector oven coolant is enabled.
Timeout	0.01 min to 999.99 min Displays the programmed coolant time-out value in the active method for the SPT injector oven.
Timed Out	Yes or No. Displays Yes if the coolant time-out period has elapsed for the SPT injector oven.

Type 1 Injector EFC Flow/Pressure Status

Item	Description
Ready	Yes or No. The Type 1 Injector EFC component is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the Type 1 EFC component has occurred.
Type 1 Pressure Setpoint	0.1 to 100 psi Displays the programmed column head pressure setting in the active method.
Actual Pressure	0.1 to 100 psi Displays the actual column head pressure at the 3800 GC.
Column Flow	Displays, in mL/min, the column flow rate calculated from the column head pressure, column temperature, and column parameters (carrier gas, column length, and internal diameter).
Linear Velocity	Displays, in cm/sec, the column linear velocity calculated from the column head pressure, column temperature, and column parameters (carrier gas, column length, and internal diameter).
Total Flow	Displays, in mL/min, the total flow rate through the system.
Split State	On or Off. Displays the current split state of the 1079/1177 injector method.
Split Ratio	1 to 10000 Displays the current split ratio of the 1079/1177 injector method. The split ratio is defined as the (Column Flow + the Split Flow) / the Column Flow.

Type 3 Injector EFC Flow/Pressure Status

Item	Description
Ready	Yes or No. The Type 3 Injector EFC component is Ready (Yes) if the setpoints have been achieved and stabilized
Fault	Yes or No. Displays if a fault in the Type 3 EFC component has occurred.
Type 3 Flow Setpoint	0.1 to 100 mL/min Displays the programmed column flow rate setting in the active method.
Actual Flow	0.1 to 100 mL/min Displays the actual column flow rate calculated from the measured head pressure and column parameters (carrier gas, column length, and internal diameter).
Pressure	Displays, in psi, the measured column head pressure.
Linear Velocity	Displays, in cm/sec, the column linear velocity calculated from the column head pressure, column temperature, and column parameters (carrier gas, column length, and internal diameter).

Type 4 Injector EFC Flow/Pressure Status

Item	Description
Ready	Yes or No. The Type 4 Injector EFC component is Ready (Yes) if the setpoints have been achieved and stabilized.
Fault	Yes or No. Displays if a fault in the Type 4 EFC component has occurred.
Type 4 Pressure Setpoint	0.1 to 100 psi Displays the programmed column head pressure setting in the active method.
Actual Pressure	0.1 to 100 psi Displays the actual column head pressure at the 3800 GC.
Column Flow	Displays, in mL/min, the column flow rate calculated from the column head pressure, column temperature, and column parameters (carrier gas, column length, and internal diameter). This value is not displayed if the column length has not been set in the 3800 GC column parameters setup.
Total Flow	1 to 1000 mL/min Displays the programmed total flow rate in the active method.
Linear Velocity	Displays, in cm/sec, the column linear velocity calculated from the column head pressure, column temperature, and column parameters (carrier gas, column length, and internal diameter).

TCD Status

Item	Description
Ready	Yes or No.
Fault	Yes or No. Displays if a fault in the TCD detector component has occurred.
TCD Electronics	On or Off Displays the TCD Electronics setting in the active method.
Range	0.05, 0.5, or 5.0 Displays the current TCD Range setting
Time Const	Fast (50 μ sec) or Slow (200 μ sec) Displays the electrometer time constant setting from the TCD Adjustments in the active method.
Filament Temp	0 to 490 °C Displays the TCD Filament Temperature setting in the active method.
Current	Displays, in mA, the actual TCD current.
Balance Pct	Displays, in %, the TCD bridge balance. On the 3800 GC, the bridge is balanced automatically each time an Autozero is performed and should normally set around 0%. A large offset in bridge balance (+ or -) could indicate a potential problem.
Polarity	Positive or negative Displays the current TCD Polarity setting
Detector Signal	Displays, in mV, the actual TCD detector signal at the 3800 GC.
Bunch Size	Displays the data acquisition bunch size.
Freq	Displays the data acquisition bunch rate, in Hz.

FID Status

Item	Description
Ready	Yes or No. The FID detector is Ready (Yes) if the setpoints have been achieved and stabilized. The FID detector is Not Ready (No) if readiness to start a run has not been achieved or if, during a run, the FID detector becomes not ready or goes "out of tolerance". Some reasons the GC may go Not Ready during a run are: the temperature program is too aggressive, the oven door is opened, the coolant runs out, the power line voltage drops, etc.
Fault	Yes or No. Displays if a fault in the FID detector component has occurred.
FID Electronics	On or Off Displays the FID Electronics setting in the active method.
Range	9, 10, 11, or 12 Displays the FID Range initial condition setting or the time-programmed setting in the active method.
Time Const	Fast (50 μ sec) or Slow (200 μ sec) Displays the electrometer time constant setting from the FID Adjustments in the active method.

Item	Description
Detector Signal	Displays, in mV, the actual FID detector signal at the 3800 GC.
Bunch Size	Displays the data acquisition bunch size.
Freq	Displays the data acquisition bunch rate, in Hz.

ECD Status

Item	Description
Ready	Yes or No.
Fault	Yes or No. Displays if a fault in the ECD detector component has occurred.
ECD Electronics	On or Off Displays the ECD Electronics setting in the active method.
Range	1 or 10 Displays the current ECD Range setting.
Time Const	Fast (50 μ sec) or Slow (200 μ sec) Displays the electrometer time constant setting from the ECD Adjustments in the active method.
Detector Signal	Displays, in mV, the actual ECD detector signal at the 3800 GC.
Bunch Size	Displays the data acquisition bunch size.
Freq	Displays the data acquisition bunch rate, in Hz.

TSD Status

Item	Description
Ready	Yes or No.
Fault	Yes or No. Displays if a fault in the TSD detector component has occurred.
TSD Bead Power	On or Off Displays the current TSD Bead Power setting
Range	9, 10, 11, or 12 Displays the current TSD Range setting
Time Const	Fast (50 μ sec) or Slow (200 μ sec) Displays the electrometer time constant setting from the TSD Adjustments in the active method.
Bead Current	2.4 to 3.8 A or Off Displays the TSD Bead Current setting in the active method or Off if the Bead Power is off.
Detector Signal	Displays, in mV, the actual TSD detector signal at the 3800 GC.
Bunch Size	Displays the data acquisition bunch size.
Freq	Displays the data acquisition bunch rate, in Hz.

PFPD Status

Item	Description
Ready	Yes or No.
Fault	Yes or No. Displays if a fault in the PFPD detector component has occurred.
PFPD Electronics	On or Off Displays the PFPD Electronics setting in the active method.
Range	8, 9, or 10 Displays the current PFPD Range setting.
Square Root Mode	On or Off Displays the PFPD Square Root Mode setting in the active method.

Type 11 Detector EFC Status

Item	Description
Type	11 Displays the Detector EFC type. Detector EFC Type 11 has 3 channels and is used for FID detector gas control.
Ready	Yes or No.
Fault	Yes or No. Displays if a fault in the Detector EFC component has occurred
Ch 1 (Make-up) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate.
Ch 2 (H2) Set, Actual	Displays, in mL/min, the programmed and actual channel 2 flow rate.
Ch 3 (Air) Set, Actual	Displays, in mL/min, the programmed and actual channel 3 flow rate.

Type 12 Detector EFC Status

Item	Description
Type	12 Displays the Detector EFC type. Detector EFC Type 12 has 3 channels and is used for TSD detector gas control.
Ready	Yes or No.
Fault	Yes or No.
Ch 1 (Make-up) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate.
Ch 2 (H2) Set, Actual	Displays, in mL/min, the programmed and actual channel 2 flow rate.
Ch 3 (Air) Set, Actual	Displays, in mL/min, the programmed and actual channel 3 flow rate.

Type 13 Detector EFC Status

Item	Description
Type	13 Displays the Detector EFC type. Detector EFC Type 13 has 1 channel and is used for either ECD or TCD detector gas control.
Ready	Yes or No.
Fault	Yes or No.
Ch 1 (Make-up) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate for ECD or TCD detector gas control.

Type 14 Detector EFC Status

Item	Description
Type	14 Displays the Detector EFC type. Detector EFC Type 14 has 2 channels and is used for TCD detector gas control for Helium, Nitrogen, or Argon.
Ready	Yes or No.
Fault	Yes or No.
Ch 1 (Make-up) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate.
Ch 2 (Reference) Set, Actual	Displays, in mL/min, the programmed and actual channel 2 flow rate.

Type 15 Detector EFC Status

Item	Description
Type	15 Displays the Detector EFC type. Detector EFC Type 15 has 3 channels and is used for PFPD detector gas control.
Ready	Yes or No.
Fault	Yes or No.
Ch 1 (Air 1) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate.
Ch 2 (H2) Set, Actual	Displays, in mL/min, the programmed and actual channel 2 flow rate.
Ch 3 (Air 2) Set, Actual	Displays, in mL/min, the programmed and actual channel 3 flow rate.

Type 16 Detector EFC Status

Item	Description
Type	16 Displays the Detector EFC type. Detector EFC Type 16 has 2 channels and is used for TCD detector gas control for hydrogen.
Ready	Yes or No.
Fault	Yes or No.

Item	Description
Ch 1 (Make-up) Set, Actual	Displays, in mL/min, the programmed and actual channel 1 flow rate.
Ch 2 (Reference) Set, Actual	Displays, in mL/min, the programmed and actual channel 2 flow rate.

Chromatogram Display



The Chromatogram Display toolbar contains the following controls:



Full Scale button: Resets the X axis scale from 0 to the end time of the current chromatogram and the Y axis to include the full range of the selected detector signal. This effect may also be achieved by double clicking in the lower left corner outside the chromatogram display.



Vertical Full Scale: Resets the Y axis to include the full range of the selected detector signal within the selected range of the X axis. This effect may also be achieved by double clicking in the left margin outside the chromatogram display.



Horizontal Full Scale resets the X axis from 0 to the end time of the currently displayed chromatogram. This effect may also be achieved by double clicking in the margin below the chromatogram display.



Previous Scaling restores the scaling previously applied to the chromatogram display. Repeated use of this button allows you to step back through a number of sequential scaling changes.



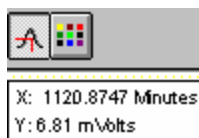
Next Scaling is only active after one or more Previous Scaling selections have been made. This button allows you sequentially to restore chromatogram scaling previously accessed by the **Previous Scaling** button.



Auto Scaling Selecting this button allows the system to adjust the scaling to display the full range of the selected detector signal while the chromatogram is running.

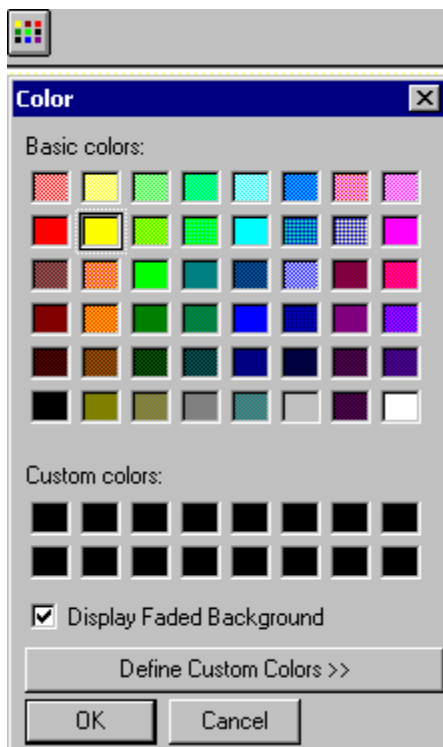
In addition to these scaling controls, you can scale vertically using the Attenuation Control to the right of the chromatogram display window. This adjusts the amplitude range on a logarithmic scale. When using the attenuation control, the lowest point of the active trace is fixed, and the amplitude range is adjusted to a smaller range as you slide the position indicator up and to a larger range as you slide it down.

If the detector signal exceeds the range of the display, a vertical offset control is added to the right border of the display. This control allows you to move the chromatogram up or down in the display window.



Cursor Display - turns the cursor on or off. The cursor follows the chromatogram trace and displays the time and amplitude of its current position in an "info-panel".

Hold the control key down to allow the cursor to move anywhere on the screen without following the active trace.



Background Color: Allows you to select any of the standard colors, or a custom color, as the background for the chromatogram display. The Display Faded Background checkbox allows you to use either a uniform color or an artistic faded color effect as the background.

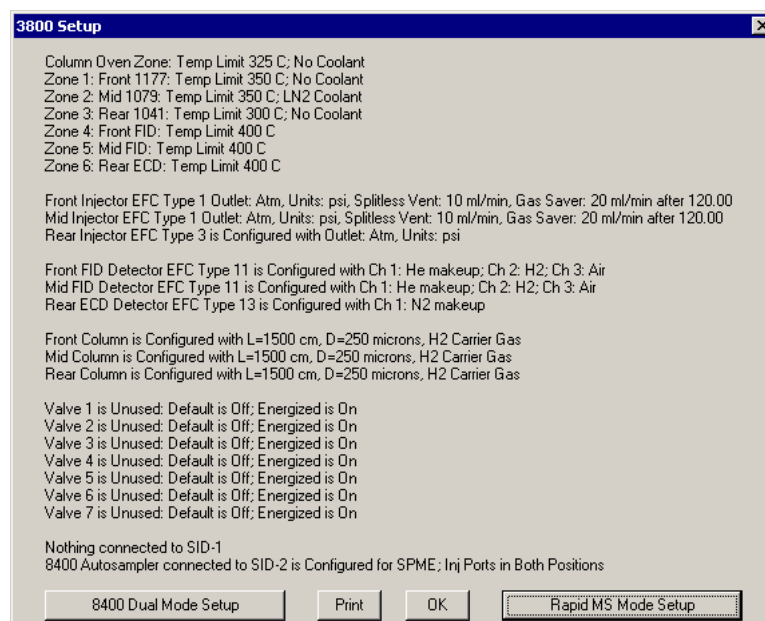
Interactive Scaling

The following mouse actions are possible for the chromatogram display window:

Action	Effect of Action
Left mouse button click and drag a selection.	Any section of the chromatogram display may be expanded to fill the chromatogram window by clicking and dragging the cursor to enclose the area of interest on the display. Whenever the display scaling is less than full scale in either axis, the appropriate scroll bar(s) will be displayed at the right or bottom of the display to allow scrolling of the display.
Left mouse button double click.	Zooms out to full scale.

Left mouse click and hold	Zooms isometrically from the mouse position, or if you are holding the control key down, zooms out from that point. Once PowerZooming begins, you can move the mouse around while the mouse button is still down to change the point at which you are zooming.
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3800 Setup Dialog



The 3800 Setup dialog displays all the hardware configuration information input through the GC keyboard and currently saved in the GC memory. The **Print** button allows you to make a printed copy of this information for your records. The **OK** button closes the dialog. The remaining two buttons allow you to input additional configuration information, detailed below.

8400 Dual Mode Setup

8400 Dual Mode Setup

8400 Mounting Position: Front

	8400 Injection Position	Injector	Front FID Detector	Mid FID Detector	Rear ECD Detector
1	Pos 1	Front 1177	yes	no	no
2	Pos 2	Mid 1079	no	yes	no
3	N/A	Rear 1041			

Indicate which Detectors are connected to each Injector that is reachable by the 8400 Syringe Tower. This information is used to manage Data Handling for Dual-Sample Injections only (those using 'Advance' and 'Clean & Adv' modes for 2nd Injection).

OK Cancel

The 8400 AutoSampler has the ability to inject into two contiguous injectors in the same chromatographic run. The 8400 Dual Mode Setup dialog allows you to define the configuration to be used when that capability is invoked. The 8400 can inject into front and middle, or middle and rear, injectors, and each of the injectors can be connected through an analytical column to any of the installed detectors.

When the 8400 AutoSampler or 8410 AutoInjector is used in Dual Mode, this dialog box is used to assign the detector channels to the datafile for the corresponding sample. In Dual Mode operation, two datafiles are created; one for the first sample injected, the other for the second sample injected. The samples that are injected are identified by their 8400/8410 Injection Position as either Position 1 or Position 2. The table in this dialog box allows the user to specify which detector information to associate with each 8400/8410 Injection Position.

8400/8410 SampleList Window Extensions

8410 SampleList: 8410-a.smp

	Sample Name	2nd Sample Name	Injection Notes	2nd Injection Notes	Vial	Injection Volume	2nd Inj Volume	Injectors Used	2nd Injection	Injection Delay
1	Standard Sample		none		1	1.0		Pos 1		
2	Standard Sample		none		2	1.0		Pos 2		
3	Same Sample Vial		none		5	1.0	1.0	1 then 2	Duplicate	0.0
4	First Sample Vial	Second Sample Vial	none	none	9+10	1.0	1.0	2 then 1	Advance	10.0
5	Same Sample Vial		none		4	1.0	1.0	2 then 1	Clean & Dup	0.0
6	First Sample Vial	Second Sample Vial	none	none	7+8	1.0	1.0	1 then 2	Clean & Adv	10.0
7										
8										
9										

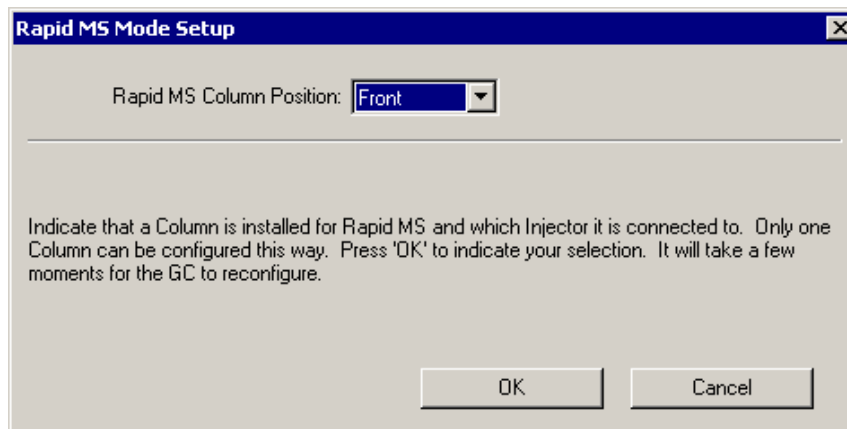
Begin Suspend Resume Data Files... RecalList...

When a 3800 GC controls an 8400 AutoSampler or 8410 AutoInjector, the SampleList for that instrument contains the following device-dependent fields. Many of these SampleList fields are only shown and available for editing when the 8400 AutoSampler or 8410 AutoInjector is used in Duplicate Mode or Dual Mode. Refer to the help on the generic SampleList Window for a description of the fields not listed below.

Field	Description
2nd Sample Name (Dual Mode operation only)	Up to 19 characters Sets the name of the second sample injected. This column is only displayed when the 2 nd Injection is "Advance" or "Clean & Adv".
2nd Inject Notes (Dual Mode operation only)	Up to 180 characters Opens the Notes window for the selected second sample to edit or create a note about the sample. This column is only displayed when the 2 nd Injection is "Advance" or "Clean & Adv".
Vial	0 through 99 for 8400 AutoSampler 1 through 21 for 8410 AutoInjector Sets the AutoSampler vial number of each sample in the SampleList. When the 2nd Injection is "Advance" or "Clean & Adv", two sequentially numbered vials will be shown. The first vial will be injected on the first injection and the next vial location will be sampled for the second injection.
Injection Volume	0.1 to 5.0 μ L or 0.1 to 10 μ L or 1 to 100 μ L depending on syringe volume. Sets the injection volume of the sample in microliters. When the SampleList is edited in System Control, the maximum value allowed is set to the syringe size currently configured in the 3800 GC setup. During operation, if the sample volume plus solvent volume, internal standard addition volume, and air gaps volume specified in the active method on the 3800 exceed the syringe volume, a 3800 hardware fault will occur. This will cause the Star Workstation to halt automation and reset the 3800. After changing the method settings and/or the injection volume to reduce the total below the installed syringe volume shown in the 3800 setup screen, you should begin the SampleList at the selected line to bypass the samples that have already been run.
2nd Inj Volume (Duplicate Mode or Dual Mode operation only)	0.1 to 5.0 μ L or 0.1 to 10 μ L or 1 to 100 μ L depending on syringe volume. Sets the injection volume in microliters for the second injection of the sample in Duplicate Mode or the injection of the second sample in Dual Mode. When the SampleList is edited in System Control, the maximum value allowed is set to the syringe size currently configured in the 3800 GC setup. During operation, if the sample volume plus solvent volume, internal standard addition volume, and air gaps volume specified in the active method on the 3800 exceed the syringe volume, a 3800 hardware fault will occur. This will cause the Star Workstation to halt automation and reset the 3800. After changing the method settings and/or the injection volume to reduce the total below the installed syringe volume shown in the 3800 setup screen, you should begin the SampleList at the selected line to bypass the samples that have already been run.
Injectors Used	Pos 1, Pos 2, 1 then 2, or 2 then 1 Selects the 8400 Injection position to be used for the sample. If the 8400 is not configured for both injection positions, entering the wrong position will cause the sample

	line to be skipped and a "Bad Injector" message entered into the message log. To run samples in Duplicate Mode or Dual Mode, you must select either "1 then 2" or "2 then 1".
2nd Injection (Duplicate Mode or Dual Mode operation only)	Duplicate, Advance, Clean & Dup, or Clean & Adv Specifies the action that the 8400 will take before making the second injection during a run. If you choose "Duplicate" or "Clean & Dup", the same sample will be used for both injections. This is called Duplicate Mode. If you choose "Advance" or "Clean & Adv", the 8400 will advance to the next vial location and will sample the vial, if any, that is there for the second sample injection. This is called Dual Mode.
Injection Delay (Duplicate Mode or Dual Mode operation only)	0.0 and 0.1 through 10.0 minutes Sets the time delay between the first injection and the second injection in Duplicate Mode and Dual Mode operation. When the value is 0.0, the injection time delay is turned off and not used. For values other than zero, the 8400 will prepare and load the syringe for the second injection. When the syringe is loaded, it will move to the inject position and wait for the injection delay to time out before injecting. If the injection delay times out before the sample is injected, an error message saying that the injection delay timed out before injection is placed in the message log.

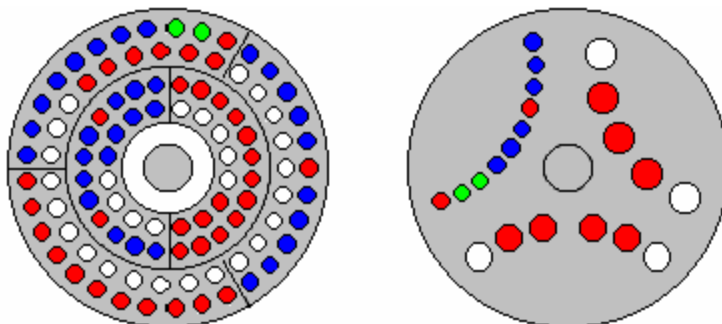
Rapid MS Mode Setup



RapidMS is a patented technology which allows fast separations on large bore capillary columns. This list box lets you identify the injector equipped with a RapidMS column. Pressing **OK** accepts the selection, and pressing **Cancel** closes the dialog without making any change.

8400/8410 Carrousel Display

When an 8400 AutoSampler or 8410 AutoInjector is connected to your 3800, the Carrousel display will be visible in the 3800 GC Status and Control window.



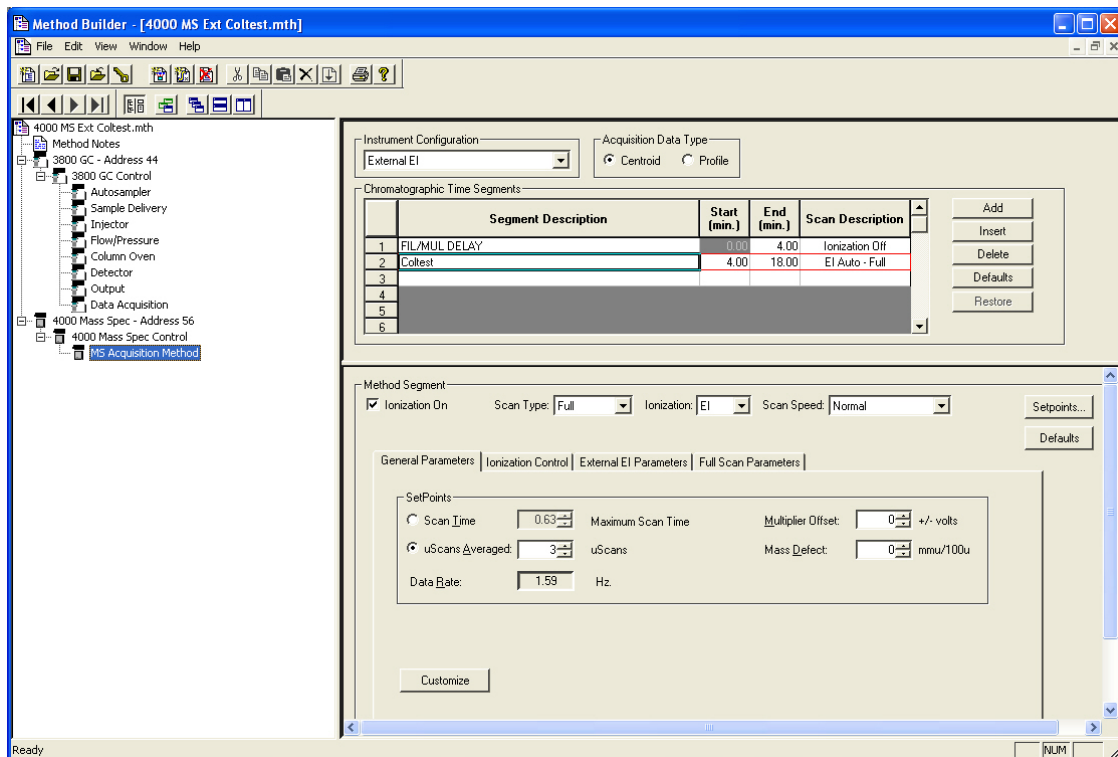
8400 AutoSampler Carrousel 8410 AutoInjector Carrousel

Item	Description
Red fill	Indicates vials listed in the active SampleList that have not yet been sampled or are missing.
Green fill	Indicates the vial (vials in Dual Mode) currently being sampled.
Blue fill	Indicates the vials in the active SampleList that have been sampled.

Action	Description
Click on a vial position	Displays the vial number for that position for 5 seconds
Double-click on a vial position	Opens the Inject Single Sample dialog box to inject a single sample.
Double click in center	Displays the active SampleList, if one is selected
Double click outside carrousel	Displays the active RecalcList, if one is selected.

4000 MS Method Builder Command Reference

The Method Builder commands available to you depend on which Instrument Configuration you have selected. In the following graphic, the Instrument Configuration is External EI.



Internal EI and CI

Item	Description
Instrument Configuration	The GC and MS will be set to the configuration of the instrument connected to the Varian MS Workstation. For the 4000 MS Internal Configuration, both EI and CI can be performed during the same run and so there is only one internal setting. For the 4000 MS External Configuration, you may select either to perform External EI or External CI. It is not possible to do both EI and CI segments in a single run, unlike Internal EI and CI methods.
Acquisition Data Type	The Acquisition Data Type can either be Centroid or Profile. Centroid is the preferred and default acquisition data type, as data handling, library searching, and spectral comparison can be done from this type of data. Profile data is typically used for diagnostic purposes. Profile files are also approximately 10 times larger, but can be converted to centroid after acquisition.
Chromatographic Time Segments	The Chromatographic Time Segments table allows you to time-program analysis conditions to get the best results for each segment in the analysis. Up to 250 time segments can be created for runs up to 650 minutes in length. By default, there is a Filament/Multiplier Delay segment at the start of the run so that the system will not be stressed during the elution of the chromatographic solvent. Following this segment, you could just acquire the mass spectra in full-scan with a single analysis segment. However, you can tailor variables such as acquired mass range, insert MS/MS segments for individual analytes, and otherwise set up the instrument to acquire the best data for each analyte. Double-click on the required field to edit the Segment Description, Start and End times. The Scan Description information is automatically entered based on the choice of Scan Type and Ionization mode in the Method Segment field below.
Segment Description	This describes the nature of the particular segment in question and usually pertains to a stage in the scan or a name of a compound perceived to be eluting during the segment. Clicking the mouse in the Segment Description cell changes the lower window contents so the Method Segment details for that particular segment are displayed in the lower window area. These are an Ionization On checkbox, Scan Type, Ionization Type, and Scan Mode.
Start (min.)	This cell indicates the time at which the particular segment will begin. Clicking in this cell allows you to edit the Start Time. The Start Time you enter will also be entered as the End Time of the preceding segment, if one exists.
End (min.)	This cell indicates the time at which the particular segment will end. Clicking in this cell allows you to edit the End Time. The End Time you enter will also be entered as the Start Time of the succeeding segment, if one exists.
Scan Description	This indicates the Scan Type and Ionization Type. You don't edit this cell but instead edit the Scan Type and Ionization Type in the lower Method Segment window by choosing the desired Scan Type and Ionization from the appropriate drop-down menus. Changing these drop-down menu values will cause the Scan Descriptions in the upper window to change.

Item	Description
Add	This button adds a chromatographic time segment to the end of the Chromatographic Time Segment Table. Adding a segment will copy all of the parameters from the last segment into the newly created segment. The default duration for the newly created segment is 1.00 minute.
Insert	This button inserts a chromatographic time segment before the currently selected row of the Chromatographic Time Segment table. Inserting a segment will copy all of the parameters from the currently selected segment into the newly created segment. Inserting a segment will essentially split the currently selected segment into two segments of equal duration.
Delete	This button deletes one or more selected segments from the Chromatographic Time Segment table. However, there must always be one segment in the method. To delete multiple segments, the entire row for each segment must be selected.
Defaults	This button sets the selected segment to default values for the selected instrument configuration. Every field of the segment is set to default values.
Restore	This button is used to restore a chromatographic time segment to its values from the original method file or from the last time the file was saved within the current editing session. Restore is only valid for saved or existing method files. For a new method file, the Defaults button is used to set the segment values to default values.
Method Segment	The 4000 MS Method Segments control what MS scan function is performed. The different scan functions in the Internal configuration are EI and CI, Full scan, AMD, MS/MS, MRM, MS ⁿ , SIS, and uSIS. For each Scan Segment selected in the upper Chromatographic Time Segments table, you will see a plethora of drop-down menus and tabs which provide you with the options to create the exact method required for each Chromatographic Time Segment. Only if the Ionization On radio button is chosen will you be able to view all the available options.
Ionization On (radio button)	If the Ionization On radio button is not checked, the filament and multiplier will be OFF during the segment. This is the default setting for Segment 1 when a new method is created, to protect the filament and multiplier from damage by the solvent front during GC/MS runs. This disabling of the Ionization can be used for other time segments as well. NOTE: For all data acquisition segments, the Ionization On box must be checked.
Scan Type	This drop-down menu has the seven Scan Types to choose from: Full, AMD, MS/MS, MRM, MS ⁿ , SIS, and uSIS. Selecting a scan type loads the default values for that Scan Type into the Parameters and Control tabs in the lower section.
Ionization	The available ionization options, EI and CI, depend on the Instrument Configuration. Internal EI and CI offers either option on a segment by segment basis. External and Hybrid require that all segments of the method be acquired by a selected ionization mode.
Scan Mode	This drop-down menu may have 1 or more options depending on which Scan Type and Ionization type has been selected. The available Scan Modes are Normal, Fast, and Fastest.
Setpoints...	The Setpoints... button, when clicked, opens a Segment Setpoints dialog box with an option to turn the Calibrant On. Choose this option to turn on the FC-43 calibration gas during an acquisition segment.

Item	Description
General Parameters (Tab)	The General Parameters tab has editable fields which indicate the Set Points you want to establish for that particular time segment. The Set Point option default values vary depending on which Scan Type and Ionization type you have selected. There are five set points: Scan Time , Scans Averaged , Data Rate , Mass Defect , and Multiplier Offset . An additional parameter, Count Threshold, becomes available when the Customize button is selected.
Scan Time, Scans Averaged, Data Rate	Scan Time, Scans Averaged and Data Rate are all linked. The number of scans averaged is updated when the scan time is adjusted and vice versa. The best way to set the scan time is to set the mass range from the Scan Parameters tab and then change the scans averaged to three (3). Three (3) scans averaged gives the best compromise between a high chromatographic data rate and good spectral averaging. There is a trade-off between how rapidly spectra are collected and the reproducibility of mass spectra or peak areas. As more microscans are averaged, the relative intensities of ions in the mass spectrum become more reproducible. However, one needs to keep in mind that collecting more data points across a chromatographic peak will improve peak area reproducibility on replicate injections. San Time Range: 0.02 – 30 seconds (full range) Scans Averaged Range: 1 to 99
Mass Defect	The Mass Defect allows for a systematic correction of the difference between the nominal mass of an atom (or ion) and its exact mass. The importance of the Mass Defect arises from the fact that the NIST library reports molecular weights to the nearest integer mass unit only, whereas the 4000 MS stores ion/intensity pairs with a mass assignment to the nearest tenth of a mass unit. For library searching, ion masses are rounded to the nearest integer. If the m/z of an ion happens to fall close to the 0.5 u crossover point, it may be assigned to the wrong mass. This is more likely for molecules with higher molecular weights, since the mass defects for several atoms may add together to produce a sizable mass defect. Range: -500 to +5000 milli mass units per 100 mass units (mmu/100u)
Multiplier Offset	The Multiplier Offset adjusts the EM voltage as much as +/- 300V relative to the current multiplier setting in the Module Attributes tab dialog in Manual Control (this is usually the 10^5 gain value from Auto Tune). Sometimes better sensitivity is achieved, particularly in techniques such as MS/MS, when the multiplier voltage is increased. Note that this adjustment can be made on a segment-by-segment basis. The same multiplier gain must be used for determining the calibration curve and analyzing unknown samples. Range: -300 to +300 volts
Ionization Control (Tab)	The Ionization Control Tab has either 1 or 2 editable fields depending on the Scan Type selected. The Automatic Gain Control Target TIC (Total Ionization Count) always appears, albeit with differing default values depending on the Scan Type. When either AMD or MRM has been selected, you will see a field to set the Prescan Ion Time (in microseconds) for that particular time segment. Also, you may click the Customize button which will display additional options to configure Fixed Ionization time, Max Ionization Time as well as the Prescan Ion Time, if applicable.

Item	Description
Automatic Gain Control Target TIC	The AGC Target TIC (Total Ion Current) is specified here. The Automatic Gain Control (AGC) algorithm uses the ion count from a prescan at a fixed ion time, along with this target value, to calculate an ion time necessary to fill the ion trap with the target number of ions during the analytical scan. The objective is to fill the trap with an optimal number of ions during each analytical scan. The Target TIC is usually not set below 10000 for full scan acquisitions, but it should also not be set too high or spectral distortions due to space charge (loss of MS resolution and/or shift in mass assignments for strong chromatographic peaks) may result. Typically, a Target TIC between 20000 and 40000 gives the best results. This component is disabled if you choose the Fixed Ion Time checkbox in the Customize area.
Fixed Ionization Time	If Fixed Ionization Time is checked, it becomes possible to run fixed ion time experiments. Fixed ion time experiments are generally limited to diagnostic tests and are not advised for qualitative or quantitative analysis in the ion trap. Choosing this check box disables both the Automatic Gain Control AND the Prescan Ion Time functions and uses your Fixed and Max Ionization times entered. Range (Internal EI): 10 – 65,000 microseconds Range (Internal PCI): 10 – 2,500 microseconds
Max Ionization Time	Changing the Max Ionization time can improve the sensitivity of techniques such as MS/MS or SIS. This parameter can be increased to 65 milliseconds for Internal EI. If the ion time in the baseline of the chromatogram is not reduced below the maximum ion time, the ion trap is not being filled with ions to the level designated by the Target TIC. You can do three things in Internal EI mode to increase the ion signal: 1) raise the emission current, 2) raise the multiplier voltage, and 3) increase the Maximum Ionization Time. If the third approach is used and you are collecting data for quantitation, make sure that enough data points are collected across the chromatographic peaks for reliable quantitation. At least five, but preferably seven or more data points are needed for good quantitative data. Remember that using Fast Data Rate may allow the use of higher Maximum Ionization Times without compromising quantitative data quality. When the Customize button is clicked in CI ionization mode, you will have the option to run fixed ion time experiments with ion times as high as 2500microseconds or to change the Maximum Ion Time for CI Auto experiments. You can turn on the CI Gas and the ion trap in Manual Control and check the ion time in CI Auto mode. If this time is less than 1,000 microseconds, lowering the Max Ion Time may improve performance. Range (Internal EI): 10 – 65,000 microseconds Range (Internal PCI): 10 – 2,500 microseconds
Internal EI/CI Parameters (Tab)	The Internal EI/CI Parameters Tab will display an editable field for Emission Current. When in CI mode, additional fields for editing Reagent Gas parameters are available.

Item	Description
Emission Current	The Emission Current (μAmps) is the current of electrons produced by the filament and it controls the number of electrons that enter the trap. By increasing the emission current so that the ion time for the baseline is approximately the maximum ion time, the maximum number of ions will be present. In certain cases, a high emission current can be used to increase the sensitivity. One of these is EI/MS/MS where the MS/MS isolation step eliminates the higher background that would normally arise from a high emission current. This option appears in both EI and CI Methods. Range: 10 – 100 microAmps (Internal EI/CI) Range: 5 – 250 microAmps (External EI/CI, Hybrid CI)
Reagent Parameters	When you select a standard reagent (i.e., Acetonitrile, Isobutane, Methane, or Methanol), the CI parameters for the selected reagent are set automatically, and you do not need to change the default values of the CI parameters. However, the CI parameters Reaction Storage Level, Ejection Amplitude (V), and Max Reaction time can be optimized for sensitivity by clicking the Customize button. You may also edit the parameters to accommodate another CI reagent, e.g. d_3-acetonitrile, and then Save these parameters under a user-selected name.
Reagent Low Mass	Reagent Low Mass is the lowest mass stored in the trap during ionization. The combination of Reagent Low and Reagent High Mass and Reaction Storage Level allows for the selection of a specific reagent ion in the CI process. For example, only mass 29 of methane could be used as the reagent ion by ejecting mass 41 during ionization and by reacting at a mass greater than 19 in the reaction step. Range: 15 – 149 m/z
Reagent High Mass	Reagent High Mass is the highest mass stored in the trap during ionization. All masses above this mass are ejected during the ionization step. This reduces the number of EI generated ions that are in the trap. The combination of Reagent Low and Reagent High Mass and Reaction Storage Level allows for the selection of a specific reagent ion in the CI process. For example, only mass 29 of methane could be used as the reagent ion by ejecting mass 41 during ionization and by reacting at a mass greater than 19 in the reaction step. Range: 16 – 150 m/z
Customize	When in CI, the Customize button is visible. Clicking on the Customize button displays additional components pertinent to the time segment method being edited. There are options to edit the Reaction Storage Level, Ejection Amplitude, and Max Reaction Time. There are no Custom features when in EI.
Reaction Storage Level	Reaction Storage Level is the value of the smallest mass stored in the ion trap during the reaction period. The Reaction Storage Level is typically set to a value that is lower than the mass of the reagent ion of interest. The optimum Reaction Storage Level will depend on the molecular ion of the analyte. Generally, you should use higher Reaction Storage levels for higher molecular ions without raising the storage level to cause ejection of CI reagent ions. For example, the molecular ion of the analyte is 352 m/z. Using acetonitrile, raising the RF storage level to 25 m/z may give better sensitivity than the default of 19 m/z. If the CI storage level is raised, the Ejection amplitude will have to be increased. Range: 10 – 150 m/z (full range)

Item	Description
Ejection Amplitude	Ejection Amplitude (V) is a voltage that corresponds to a low mass ejection cutoff that is slightly higher than the Reagent High Mass. This voltage actively ejects unwanted ions (i.e., not reagent ions) that are produced during ionization. Generally, higher CI storage values require higher ejection voltages. The voltage should not be set so high as to cause the ejection of CI reagent ions. The Ejection amplitude is applied at the end of the ionization time to eject unwanted high mass EI artifact ions. The default value for each reagent usually does not need adjustment. Range: 0 – 59 volts.
Max Reaction Time	Maximum Reaction Time is the maximum time that reagent gas ions are allowed to react with sample molecules to form ions. The typical reaction time is 100 milliseconds. Depending on the CI reagent and the target compound of the analysis, sensitivity may be affected significantly by varying this parameter. Range: 1- 2000 milliseconds
<Scan Type> Parameters	This tab will appear differently depending on the Scan Type that is selected. For example, if AMD is the Scan Type selected, then the tab will read AMD Parameters. If Full is the selected Scan Type, then the tab reads Full Scan Parameters.
Full Scan Parameters	You can change the values of the Full Scan Parameters Low Mass (m/z) and High Mass (m/z). If you click the Customize button, then you can edit the Tune Type (Auto, BFB, DFTPP, or Single Segment) as well as enter Low Mass, High Mass, RF Storage Level, and Ion Time Factor for each segment.
Low Mass (m/z) High Mass (m/z)	Unless the Customize button is active, the lower and upper limits of the mass range to be acquired are entered here. If in Customize mode, then the Low Mass and High Mass will be entered into the table by clicking on each cell to edit. Range: 10 – 1000 m/z (full range)
RF Storage Level (or Ionization Storage Level)	This is the m/z value at which the RF is maintained during ionization and the coarse isolation step. The typical range is 35 to 100 m/z with a default value of 35 m/z. Range: 8 – 150 m/z (full range)
Ion Time Factor (Target Factor)	When a scan is performed in Auto mode, the prescan is followed by an ionization time and scan of each segment needed to cover the Mass Range designated to the left of the segment table. The actual Ion Time for each segment is the AGC Ion Time multiplied by the percentage in the Ion Time Factor field. If needed, adjust the Ion Time Factor for particular segments to increase or decrease the abundance of key ions. For Full Scan, the Ion Time Factor is specified for both fixed ionization and AGC ionization. For SIS, the Ion Time Factor is specified for fixed ionization and the Target Factor is specified for AGC ionization. Range: 1 – 999%
Save	Save allows any modification to the tune parameters to be saved so that the tune parameters can be referenced in other methods.
Add	This button adds a segment to the end of the Scan Segment table. The range of the previously final segment is divided between that segment and the added segment. For example, if the final segment covered m/z 400 to 1000, that segment will now cover m/z 400 to 700, and the added segment will cover m/z 701 to 1000.

Item	Description
Insert	This button inserts a segment after the currently selected row of the Scan Segment table. Inserting a segment will essentially split the currently selected segment into two segments of equal mass range.
Delete	This button deletes one or more selected segments from the Scan Segment table. However, there must always be at least one segment in the table. To delete multiple segments, the entire row for each segment must be selected.
Customize	Clicking the Customize button displays the Scan Segment table and the associated controls. Clicking the button again will hide the customizable parameters.
AMD, MRM, MS/MS, MS ⁿ Scan Parameters	If you choose any of these MS/MS-type ion preparation modes, you will see a Table that shows all of the available MS/MS parameters, set out in tabular form. Each of the three steps of MS/MS (isolation, dissociation, and scanning) follows across the table.
Precursor Ion	Integer mass values may be used if the mass isolation window (next column) is greater than 1.5 m/z. If the window size is less than 1.5 m/z, or if the mass defect of the precursor ion is greater than approximately 0.3 m/z, use the exact fractional mass. Range: 50 – 1000 m/z
Isolation Window	The full mass isolation window range is 1.0 to 14.0 m/z. The actual range is dependent on the precursor ion. The default value is 3.0 m/z. Both integral and fractional mass isolation windows are accepted. If an isolation window smaller than 1.5 m/z is used, then the exact mass of the precursor ion should be entered in the Precursor Ion column. When first developing a method, you may wish to start with a mass isolation window of 3 m/z to ensure that the ion is centered in the window, e.g., given a precursor ion mass of 502 m/z and a mass isolation window of 3 m/z, ions of masses 501, 502, and 503 m/z would be isolated. If there is a need to reduce the window because of interfering ions, this should be done in a later step because the customized parameters may require optimization. Some ions are easily dissociated during the isolation step. Increasing the width of the isolation window will reduce the ion loss by premature dissociation at the expense of selectivity. Range: 1.0 – 14.0 m/z (full range)
Waveform Type	Select the Waveform Type by selecting non-resonant or resonant excitation from the list box. The non-resonant method is usually the first choice for reasons of simplicity of operation. Resonant excitation involves exciting the target ions by an RF field of the resonant frequency of the trapped ions. Non-resonant excitation involves exciting the precursor ions using an RF field not at the resonant frequency of the trapped ions.
Excitation Storage Level (m/z)	The Excitation Storage Level is the RF storage level in m/z applied while the dissociation waveform is applied following isolation. The Excitation Storage level depends on the precursor mass, but the lowest product ion must be more than 2 mass units above the excitation storage level. The excitation storage level for the precursor ion can be calculated using the “q” calculator. The “q” calculator is accessed by right clicking on any of the fields in the MS/MS parameters table. NOTE: Set the excitation storage level to avoid ejection of the lowest mass product ion. If, for example, a precursor ion of 403 m/z dissociated to product ions with m/z values of 350, 200, and 131, you would not want to use an excitation storage level above 126 m/z.

Item	Description
Excitation Amplitude (volts)	The amplitude range for non-resonant excitation is 0 to 120 volts. For resonant excitation, the range is 0 to 60 volts. The default values are 20 volts for non-resonant excitation and 0.2 volts for resonant excitation. Range (Resonant Excitation): 0 – 59.79 volts Range (Non-resonant Excitation): 0 – 119.58 volts
Product Ion Start Mass (m/z), Product Ion End Mass (m/z)	Set up the Product Ion Start and End Masses (or Low and High masses) to cover the mass range required.
More Ranges	More Ranges allows you to have multiple Product Ion Start and End Masses in a single segment. Clicking on the More Ranges square spawns the Product Ion Mass Ranges Dialog box with a Mass Ranges table, and buttons to Add, Insert, and Delete Product Ion Start and End Masses.
Add	This button adds a line to the end of the Mass Ranges Table.
Insert	This button inserts a line after the currently selected row of the Mass Ranges table.
Delete	This button deletes one or more selected rows from the Mass Ranges table.
“q” Calculator	In scientific literature, storage RF values are often reported in terms of the Mathieu “q” parameter. The value of the “q” parameter determines the stability of the precursor ion trajectory. More stable trajectories allow higher excitation voltages to be applied before ions are ejected from the trap. It has been empirically observed that a “q” value of 0.4 provides an optimum yield of product ions. This calculator utility determines the corresponding CID storage RF value (m/z) following user entry of an ion mass (m/z) and desired “q” value.
Customize	Clicking the Customize button displays parameters to customize user settings for set points, ionization parameters, or scan function parameters. Clicking the button again will hide the customizable parameters.
Ionization Storage Level (RF Storage Level)	This column appears when you click on the Customize button. This value is the m/z value at which the RF is maintained during ionization and the coarse isolation step. The typical range is 35 to 100 m/z with a default value of 35 m/z. Range: 8 – 150 m/z (full range)
Low Offset (Low Edge Offset)	This column appears when you click on the Customize button. The Low Edge Offset is the mass offset to optimize the ejection of the mass just below the precursor ion mass. The default value is 0. Low Edge Offset affects the isolation window on the low mass side of the precursor ion. Increasing the mass offset increases the isolation window on the low mass side of the precursor ion. Decreasing the offset decreases the window on the low mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments. Range: -0.5 to 0.5 m/z

Item	Description
High Offset (High Edge Offset)	This column appears when you click on the Customize button. The High Edge Offset is the mass offset to optimize the ejection of the mass just above the precursor ion mass. The default value is 0. High Edge Offset affects the isolation window on the high mass side of the precursor ion. Increasing the mass offset increases the isolation window on the high mass side of the precursor ion. Decreasing the offset decreases the window on the high mass side. The offset should be adjusted to minimize the amplitude of the adjacent masses below the precursor ion. Initially, adjust in 0.2 m/z increments. Range: -0.5 to 0.5 m/z
High Mass Ejection	This column appears when you click on the Customize button. The High Mass Ejection is the amplitude of the broadband waveform used to eject masses above the isolated precursor ion. The default High Mass Ejection value is 35 volts. Range: 0 – 59.7 volts
Excitation Time (msec)	This column appears when you click on the Customize button. The Excitation Time is the time allotted for collision-induced dissociation (CID) by ion excitation. The default excitation time is 20 msec for both resonant and non-resonant excitation. Range: 0 – 1000 msec
Modulate RF	This parameter appears when you click on the Customize button. This parameter (checked = true) specifies whether the RF storage field is modulated during resonant CID. This modulation greatly reduces any effect of changing secular frequency with concentration.
Number of Frequencies	This parameter appears when you click on the Customize button. This is the number of frequencies at which the CID excitation waveform is applied. The default value is 1 frequency applied at the end caps. A value of 3 generates three frequencies: the resonance frequency and frequencies 500 Hz above and below that frequency. Range: 1 – 119 frequencies (odd numbers only)
CID Frequency Offset	This parameter appears when you click on the Customize button. For certain applications it may be desirable to offset the dissociation frequency. The CID frequency can be adjusted between +/- 10 kHz. CID frequency offset can only be applied to a single frequency. If the number of frequencies is greater than 1, this field is hidden. Range: -10 to +10 kHz
Defaults	The Defaults button is used to set the parameters of a method segment to their default values. Generally, any user-modified mass values are preserved.
Target Factor (%)	This parameter only appears in SIS Scan Type while in Customize mode. The Target factor is also known as Ion Time Factor. When a scan is performed in Auto mode, the prescan is followed by a scan of each segment needed to cover the Mass range designated to the left of the segment table. The actual Ionization Time for each segment is the AGC Ion Time multiplied by the percentage in the Target Factor field. If needed, adjust the Target Factor for particular segments to increase or decrease the abundance of key ions. For SIS, the Ion Time Factor is specified for fixed ionization and the Target Factor is specified for AGC ionization.

Item	Description
Waveform Scale Factor (%)	This parameter only appears in SIS Scan Type while in Customize mode. The Waveform Scale Factor is a relative scaling factor applied to the amplitude of the SIS ejection waveform. Range: 1 – 999%
Count Threshold	The Count Threshold setting appears in the General Parameters Customize option of all Scan Types. Any ions with less intensity than the selected Count Threshold will be regarded as noise and will not be saved during the run.

External EI

- 25 μ Amps emission current default
- 10,000 count AGC target default

External CI

- No SIS
- Fewer options in Reagent Parameters
- Positive/Negative CI option
- Single scan segment default

Hybrid CI

- Emission current default 25 μ Amps in positive mode, 250 μ Amps in negative mode.

3800 GC Method Command Reference

3800 GC AutoSampler

Autosampler: 8400

Syringe Size (uL): 10 uL

Injection Mode: User Defined

Sample Depth (%): 90

Solvent Depth (%): 90

Default Clean

Vial: I

Volume (uL): 5.0

Strokes: 1

Speed (uL/sec): 5.0

Clean Mode

Pre-Inj Solvent Flushes: 3

Pre-Inj Sample Flushes: 0

Post-Inj Solvent Flushes: 1

Clean Solvent Source: I

Internal Standard

Use: no

Vial: II

Volume (uL): 1.0

Drawup Speed (uL/sec): 5.0

Pause Time (sec): 0.0

Air Gap: yes

More User Defined...

The 8400 AutoSampler and 8410 AutoInjector operate in liquid injection mode or Solid Phase Micro-Extraction (SPME) mode. When the liquid injection mode is selected, the AutoSampler window may contain up to five sections. The topmost section is common to all modes of operation. In addition, all modes of operation also include a Default Clean section and a Clean Mode section. When Viscous, Volatile, or User Defined modes are selected, the Internal Standard section is displayed.

In User Defined mode, the **“More User Defined...”** button is displayed to provide access to the dialog box to set the User Defined parameters. The Default Settings for Predefined AutoSampler Modes contains a table showing the parameters that are used by the 8400/8410 for each of the predefined modes of operation.

Item	Description
AutoSampler Type	8400, 8410, or None Specifies whether or not an 8400 AutoSampler or 8410 AutoInjector is installed on the 3800 GC. If an 8400 or 8410 is not installed on the GC or is turned off, select None. When 8400 or 8410 is selected, the following items are displayed for editing.
Syringe Size (µL)	5 µL, 10 µL, or 100 µL Selects the size of the syringe that is installed on the 8400/8410 for use with this method.
Injection Mode	Std Split/Splitless, Std On Column, Neat, Viscous, Volatile, or User Defined. Sets the 8400/8410 parameters for the predefined modes of operation. Note that changing the injection mode will change the parameters displayed for editing. It is not advisable to use the User Defined mode unless you are developing a special injection program.
Sample Depth (%)	0 to 100% Specifies how far the syringe needle is to go down into the sample vial. 100% is the bottom of the vial. 0% is the bottom of the vial septum.
Solvent Depth (%)	0 to 100% Specifies how far the syringe needle is to go down into the solvent vial. 100% is the bottom of the vial. 0% is the bottom of the vial septum.

Default Clean Section

The screenshot shows a dialog box titled "Default Clean". It contains four rows of settings, each with a label and a value field with a dropdown arrow on the right:

- Vial: 1
- Volume (µL): 5.0
- Strokes: 1
- Speed (µL/sec): 5.0

Default Cleaning occurs when the **Stop** button on the front panel of the 3800 GC, the **Reset** button on the 3800 Status Window in System Control, or the **Stop Automation menu item** in the System Control Automation menu is pressed after the 8400/8410 has started its run and before it has finished its sampling and post-injection washing operations. Default Cleaning also occurs when the 3800 GC has a fatal automation fault after the 8400/8410 has started its run and before it has finished its sampling and post-injection washing operations.

Item	Description
Vial	I, II, or III Specifies which of the three solvent vials will be used by the Default Cleaning.
Volume (µL)	0 to 100 microliters with 100 µL syringe 0 to 10.0 microliters with 10 µL syringe 0 to 5.0 microliters with 5 µL syringe Specifies the amount of cleaning solvent that will be drawn up with each syringe cleaning stroke.
Strokes	0 to 10 Specifies the number of times the cleaning solvent will be drawn up into the syringe and expelled into the waste cup.
Speed (µL/sec)	1 to 100 µL/per sec with 100 µL syringe 0.1 to 50.0 µL/per sec with 10 µL syringe 0.1 to 25.0 µL/per sec with 5 µL syringe Specifies the speed at which the cleaning solvent will be drawn up into the syringe and the speed at which it is expelled into the waste cup.

Clean Mode Section

Clean Mode

Pre-Inj Solvent Flushes: 3

Pre-Inj Sample Flushes: 0

Post-Inj Solvent Flushes: 1

Clean Solvent Source: I

Item	Description
Pre-Inj Solvent Flushes	0 to 99 Specifies the number of times each selected cleaning solvent will be drawn up into the syringe and expelled into the waste cup before flushing with the sample or the next cleaning solvent (if more than one cleaning solvent source is specified).
Pre-Inj Sample Flushes	0 to 99 Specifies the number of times the sample will be drawn up into the syringe and expelled into the waste cup before the syringe is loaded for injection.
Post-Inj Solvent Flushes	0 to 99 Specifies the number of times each selected cleaning solvent will be drawn up into the syringe and expelled into the waste cup before flushing with next cleaning solvent (if more than one cleaning solvent source is specified).
Clean Solvent Source	I, II, III, I & II, I & III, II & III, or I & II & III Selects which solvent vial or sequence of solvent vials will be used to flush the syringe before and/or after injection.

Internal Standard Section

Internal Standard

Use: no

Vial: II

Volume (uL): 1.0

Drawup Speed (uL/sec): 5.0

Pause Time (sec): 0.0

Air Gap: yes

Item	Description
Use	Yes or No If Yes, an internal standard addition will be used. When internal standard addition is used, the internal standard solution will be drawn up into the syringe from the specified solvent vial before the sample is drawn up.
Vial	I, II, or III Selects which solvent vial contains the internal standard.
Volume (uL)	0 to 99 uL with 100 uL syringe 0 to 9.0 uL with 10 uL syringe 0 to 4.9 uL with 5 uL syringe Specifies the amount of internal standard to be drawn into the syringe before it draws the sample.
Drawup Speed (uL/sec)	1 to 100 uL/sec with 100 uL syringe 0.1 to 50.0 uL/sec with 10 uL syringe 0.1 to 25.0 uL/sec with 5 uL syringe Specifies the speed at which the internal standard will be drawn up into the syringe.
Pause Time (sec)	0 to 9.9 seconds Specifies how long the syringe is to remain in the internal standard vial after drawing up the internal standard.
Air Gap	Yes or No If Yes, a 1 uL of room air will be drawn into the syringe to create an air gap between the internal standard and the sample.

More User Defined Settings Dialog Box

More User Defined Settings

Solvent Plug

Vial: [Dropdown]

Volume (uL): 1.0

Drawup Speed (uL/sec): 5.0

Pause Time (sec): 0.0

Air Gap: yes

Viscosity

Viscosity Delay (sec): 0.0

Fill Speed (uL/sec): 5.0

Inject Speed (uL/sec): 50.0

Pre-Inj Delay (sec): 0.0

Post-Inj Delay (sec): 0.0

User Defined

Fill Volume (uL): 5.0

Fill Strokes: 0

Sample Air Gap: no

Air Plug after Sample: 1.0

OK Cancel

Solvent Plug Settings

Solvent Plug

Vial: [Dropdown]

Volume (uL): 1.0

Drawup Speed (uL/sec): 5.0

Pause Time (sec): 0.0

Air Gap: yes

Item	Description
Vial	I, II, or III Selects which solvent vial to use for the solvent plug.
Volume (μL)	0 to 100 μL with 100 μL syringe 0 to 10.0 μL with 10 μL syringe 0 to 5.0 μL with 5 μL syringe Specifies the amount of solvent to be drawn into the syringe before the sample or internal standard.
Drawup Speed (μL/sec)	1 to 100 μL/sec with 100 μL syringe 0.1 to 50.0 μL/sec with 10 μL syringe 0.1 to 25.0 μL/sec with 5 μL syringe Specifies the speed at which the solvent will be drawn up into the syringe.
Pause Time (sec)	0 to 9.9 seconds Specifies how long the syringe is to remain in the solvent vial after drawing up the solvent.
Air Gap	Yes or No If Yes, a 1 μL of room air will be drawn into the syringe to create an air gap before the solvent plug.

User Defined Settings

User Defined

Fill Volume (uL):	5.0
Fill Strokes:	0
Sample Air Gap:	no
Air Plug after Sample	1.0

Item	Description
Fill Volume (uL)	0 to 100 uL with 100 uL syringe 0 to 10.0 uL with 10 uL syringe 0 to 5.0 uL with 5 uL syringe Specifies the sample volume that will be used for each fill stroke.
Fill Strokes	0 to 99 Specifies the number of the times the sample will be “pumped” in and out of the syringe before loading the sample volume into the syringe.
Sample Air Gap	Yes or No If Yes, a 1 uL of room air will be drawn into the syringe to create an air gap before the sample plug.
Air Plug after Sample	0 to 100 uL with 100 uL syringe 0 to 10.0 uL with 10 uL syringe 0 to 5.0 uL with 5 uL syringe Specifies the volume of room air that will be drawn into the syringe after it is loaded with sample.

Viscosity Settings

Viscosity	
Viscosity Delay (sec):	0.0
Fill Speed (uL/sec):	5.0
Inject Speed (uL/sec):	50.0
Pre-Inj Delay (sec):	0.0
Post-Inj Delay (sec):	0.0

Item	Description
Viscosity Delay (sec)	0 to 9.9 seconds Specifies how long the syringe is to remain in the sample vial after drawing up the sample.
Fill Speed (µL/sec)	1 to 100 µL/sec with 100 µL syringe 0.1 to 50.0 µL/sec with 10 µL syringe 0.1 to 25.0 µL/sec with 5 µL syringe Specifies the speed at which the sample will be drawn up into the syringe.
Inject Speed (µL/sec)	1 to 100 µL/sec with 100 µL syringe 0.1 to 50.0 µL/sec with 10 µL syringe 0.1 to 25.0 µL/sec with 5 µL syringe Specifies the speed at which the contents of the syringe will be expelled into the injector.
Pre-Inj Delay (sec)	0 to 99.9 seconds Specifies the length of time the syringe needle resides in the injector before expelling the syringe contents.
Post-Inj Delay (sec)	0 to 99.9 seconds Specifies the length of time the syringe needle remains in the injector after expelling the syringe contents.

Default Settings for Predefined AutoSampler Modes

The following table lists the parameter settings used for each of the Predefined 8400/8410 modes using a 10 µL syringe. If the predefined modes do not work acceptably for your samples, use the User Defined mode to enter settings that will work better with your samples. The values listed in this table provide a starting point for setting the various parameters.

Parameter	Std Split/ Splitless	Std On- Column	Neat*	Volatile	Viscous
Solvent Plug Settings:	Solvent plug is not used with predefined modes. If you wish to use solvent plug injections, you must use User Defined mode.				
PreDefined Settings:					
Fill Volume	7.5 µL	7.5 µL	Not used	Not used	Not used
Fill Strokes	5	5	0*	0	0
Sample Air Gap	No	No	No	No	No
Air Plug after Sample	1 µL	1 µL	1 µL	1 µL	1 µL
Viscosity Settings:					
Viscosity Delay	0 sec	0 sec	0 sec	6 sec	9.9 sec
Fill Speed	2 µL/sec	2 µL/sec	2 µL/sec	1 µL/sec	1 µL/sec
Inject Speed	50 µL/sec	2 µL/sec	50 µL/sec	1 µL/sec	5 µL/sec
Pre-Inj Delay	0 sec	0 sec	0 sec	0 sec	0 sec
Post-Inj Delay	0 sec	6 sec	0 sec	0 sec	12 sec

*Instead of fill strokes, Neat mode fills the syringe with sample at 2 µL/sec then expels it into the waste cup at 50 µL/sec a total of six times. Then it fills the syringe with sample at 2 µL/sec and expels it back into the sample vial at 50 µL/sec. This “pumping” action is done three times. The sample is then loaded into the syringe and injected using the parameters in the table.

Solid Phase Micro-Extraction (SPME) Sampling Mode

Autosampler: 8400	Adsorb Time (min): 0.00
Agitation Strokes: 1000	Desorb Time (min): 2.00
Injection Mode: SPME mode	Sample Depth (%): 85
	Solvent Depth (%): 0
Derivatization	
Use: no	
Position: III	
Adsorb Time (min): 1.00	
Clean Mode	
Pre-Inj Solvent Strokes: 0	
Post-Inj Solvent Strokes: 0	
Clean solvent source: III	
Adsorb Time (min): 1.00	
Desorb Time (min): 1.00	
Use Prep Ahead: yes	Prep Ahead Delay (min): 5.9
Column Oven End Time: 19.75 min	

SPME mode is designed to use the special Solid Phase Micro-Extraction fibers and fiber holder.

Note that you cannot select the SPME Sampling mode from the CP-3800 keypad. In order to configure the system to use SPME you must set a switch on the syringe sled to the correct position and install the special SPME fiber holder block. This process of setting up the system to use SPME was adopted to avoid unintentional damage to either SPME hardware or syringes.

SPME mode actions are summarized below.

Clean Mode



CAUTION

Do not use the clean mode actions unless you have experience with SPME or have very dirty samples with insoluble materials that need to be removed. Indiscriminate use of cleaning can introduce contaminants into your analytical run and degrade your results.

Item	Description
Pre-Injection Solvent Strokes	0 to 99 The number of pre-injection solvent clean flushes specified in the method will be performed by dipping the fiber into the solvent and waiting for each clean stroke the time specified in the Adsorb Time for Cleaning. Once the cleaning strokes are completed the tower will rotate over the waste station, the fiber holder will descend and the fiber will be exposed for drying/desorbing (desorbing requires a heated gas stream to be gently blown over the fiber).
Post-injection solvent strokes	0 to 99 Executed the same as pre-inject strokes, following the end of the sample desorb time.
Clean Solvent Source	I, II, or III The solvent vial location in which the solvent clean will be done.
Adsorb Time	0 to 999.99 minutes The time the fiber will be exposed to the solvent during each clean stroke.
Desorb Time	0 to 999.99 minutes The time the fiber will be exposed in the waste cup area following each clean stroke.

Derivatization

Item	Description
Use	Yes/No If Derivatization is specified, after pre-injection cleaning, if any, the fiber will be introduced into the Derivatizing Agent vial specified in the Derivatization page. The fiber will penetrate into the Derivatizing agent vial the depth set for the Solvent penetration depth on the main SPME parameters page. The fiber will be exposed for the specified Adsorb time and then be withdrawn.
Position	I, II, or III The position in the solvent vial rack of the Derivatization vial to be used. The default is position III.
Adsorb Time	0 to 999.99 minutes The time period during which the fiber is exposed to the Derivatizing reagent.
Solvent Depth (%)	0 to 100 This parameter is found in the main control section of the window. Use the solvent depth parameter to determine whether the fiber is immersed in the Derivatization reagent solution or is exposed to reagent vapors in the headspace above the reagent.

Prep-ahead

Item	Description
Use Prep-ahead	Yes/No If prep-ahead is used, the AutoSampler will begin the sampling process after the prep-ahead timer has expired. This allows you to sample for extended lengths of time, while the previous sample chromatogram is running, without adversely affecting the throughput of your system. The prep-ahead timer starts after the Auto Sampler has finished all injection actions for the previous injection.
Prep-ahead Delay	0 to 999.99 minutes Adjust this time such that sample adsorption is complete just as the GC becomes Ready following the previous sample run.

Sampling

Item	Description
Sample Depth (%)	0 to 100% Sample depth determines whether the sample is adsorbed from the liquid sample or from the headspace above the sample. For liquid sampling, carefully adjust the sample depth so that the fiber holder does not contact the sample liquid, to prevent potential sample carryover.
Agitation Strokes	0 to 500000 If Headspace sampling is used, no agitation is required, so a value of 0 should be entered. For liquid sampling, agitation is generally desirable to speed adsorption of the sample. Assume approximately 100 agitation strokes per minute of adsorption required. 1,000 agitation strokes would be typical.
Adsorb Time	0 to 999.99 minutes The adsorb time is executed for <i>each</i> Agitation Stroke specified. For Headspace sampling or liquid sampling without agitation, enter the total desired adsorption time, for example 10.0 minutes, divided by the number of agitation strokes, typically 1. For liquid sampling with agitation, enter a value of 0, which will allow the maximum agitation rate.

3800 GC Sample Delivery

Front Valve Oven | Middle Valve Oven | Rear Valve Oven

Front Valve Oven Installed: ☒ Yes ☐ No

Valve Oven: ☒ On ☐ Off Temperature (C): 50

	Time	Valve 1	Valve 2	Valve 3
1		Front Split	none	none
2	Initial	ON (split)		
3	1.00	OFF (s/less)		
4				
5				
6				
7				
8				
9				
10				

Add
Insert
Delete
Sort

Column Oven End Time: 20.00 min

Item	Description
Front Valve Oven Middle Valve Oven Rear Valve Oven	The 3800 GC can have up to three valve ovens installed (front, middle, and rear). The tabs at the top of the window select the valve oven position.
Valve Oven Installed	Indicate whether or not the Valve Oven at each position is installed by clicking on the Yes or No radio buttons.
Valve Oven on/off	Indicate whether or not you will use the installed Valve Oven by clicking on the ON or Off radio buttons.
Temperature (°C)	Specifies the isothermal temperature of each Valve Oven.
Time	Time settings to program how each valve is used initially and at the indicated time.
Valve 1-7	Use the first row to indicate how each valve is used. Click on the arrow in the top of each Valve column, and select from the choices displayed in the combo box. The second row contains the initial setting for each Valve when the Method is activated. The following rows contain the time-programmed settings for each of the seven Valves.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.

3800 GC Injector

Front Injector | Middle Injector | Rear Injector

Front Injector Type: 1041 Injector Oven: ☒ On ☐ Off

Temperature (C): 50

Item	Description
Front Injector Middle Injector Rear Injector	The 3800 GC can have up to three injectors installed (front, middle, and rear). The tabs at the top of the window select the injector position.
Injector Type	1079, 1177 Specifies the type of injector installed in the selected position. Select "None" if no injector is installed at that position.
Injector Oven	On or off. Turns the injector oven in the selected position on or off.
Temperature	See individual injector

Click on the following injector types for a description of the parameters that appear when the injector is selected.

1079 Injector

Front Injector | Middle Injector | Rear Injector

Middle Injector Type: 1079 Injector Oven: ☒ On ☐ Off
 Injector Coolant: ☒ On ☐ Off

Enable Coolant at (C): 250
 Coolant Timeout (min): 20.00 Split Ratio...

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	50	200	1.00	1.00
2	250	200	15.00	17.00
3				
4				
5				

Add
Insert
Delete

Column Oven End Time: 20.00 min

Item	Description
Injector Coolant	On or Off. Turns the coolant valve on when the injector is cooling down. The Coolant valve will not be used if the Off position is selected.
Enable Coolant at (°C)	30° to 450 °C. Specifies the temperature at which the coolant valve turns on.
Coolant Timeout (min)	0.01 to 999.99 min. Disables coolant flow to save coolant after the specified time, if either the injector fails to reach its set temperature or the GC does not go into run.
Split Ratio...	If the appropriate EFC type has been configured, displays the Split Ratio dialog box.

Item	Description
Temp (°C)	-99° to 450 °C.
Rate (°C /min)	1° to 200 °C/min. The Rate in the first row is always blank and cannot be edited.
Hold (min)	0.01 to 999.99 min. Total program time cannot exceed 999.99 minutes.
Total (min)	0.01 to 999.99 min. Calculated from the injector temperature program. Cannot be edited.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.

1177 Injector

Front Injector | Middle Injector | Rear Injector

Middle Injector Type: 1177 Injector Oven: ☒ On ☐ Off

Temperature (C): 200

Split Ratio...

Split Ratio for Middle 1177 + Middle Type 1 EFC

	Time	Split State	Split Ratio
1	Initial	On	100
2	0.00	Off	Off
3	1.00	On	100
4			
5			
6			

Column Oven End Time: 20.00 min

Save Cancel

Add
Insert
Delete
Sort

Item	Description
Temp (°C)	50° to 450 °C.
Split Ratio...	If the appropriate EFC type has been configured, displays the Split Ratio dialog box.

Split Ratio Dialog Box

This dialog is displayed from the 3800 GC Injector window when a 1079 or 1177 injector is configured with type 1 EFC.

Split Ratio for Middle 1079 + Middle Type 1 EFC [X]

	Time	Split State	Split Ratio
1	Initial	On	50
2	0.00	Off	Off
3	1.00	On	50
4			
5			
6			

Column Oven End Time: 20.00 min

Buttons: Add, Insert, Delete, Sort, Save, Cancel

Item	Description
Time	0.00 to 999.99 minutes
Split State	On/Off. If the split state is ON, then the sample is split according to the split ratio specified. If the split state is OFF, then all the sample enters the column.
Split Ratio	Off, 1 to 10,000. Use a split ratio of 100 after injection to vent the injector. Use a very low split ratio after flushing to conserve carrier gas.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.

3800 GC Flow/Pressure

Electronic Flow Control (EFC)

Front EFC | Middle EFC | Rear EFC

Front EFC Type: None

Item	Description
Front EFC Middle EFC Rear EFC	The 3800 GC can have up to three injector EFC modules installed (front, middle, and rear). The tabs at the top of the window select the EFC position.
EFC Type	None, Type 1 (for 1079/1177 injectors) Type 4 (for valved systems). Specifies the type of EFC installed in the selected position.

Click on the following EFC types for a description of the parameters that appear when the EFC type is selected.

Type 1 EFC (for 1079 and 1177 injectors)

Front EFC
Middle EFC
Rear EFC

Front EFC Type:
Type 1 (for 1079/1177 Injectors)

	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
1	6.3		1.00	1.00
2	9.7	2.00	0.00	2.70
3	13.3	0.43	8.93	20.00
4				
5				
6				
7				
8				

Add
Insert
Delete

Constant Column Flow Mode
Constant Flow:
☒ Off
☐ On

Item	Description
Pressure (psi)	0.1 to 100.0 psi.
Rate (psi/min)	0.01 to 400.00 psi/min. The first line is devoted to initial conditions, so a rate cannot be entered.
Hold (min)	0.01 to 999.99 min.
Total (min)	0.01 to 999.99 min. Calculated from the pressure program, cannot be edited.
Constant Flow	On or off. Click on Constant Flow to disable the spreadsheet and allow the system to calculate a pressure program which will maintain a specified constant column flow rate. See figure below.
Column Flow (mL/min)	Specifies the desired constant Column Flow.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.

Pressure Pulse Injection

Front EFC
Middle EFC
Rear EFC

Front EFC Type:
Type 1 (for 1079/1177 Injectors)

	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
1				
2				
3				
4				
5				
6				
7				
8				

Add
Insert
Delete

Constant Column Flow Mode

Constant Flow:
Off
On

Pressure Pulse:
No
Yes

Column Flow (ml/min):
2.0

Pulse Pressure (psi):
22.3

Pulse Duration (min):
0.95

Column Oven End Time: 20.00 min

If Constant Column Flow mode is selected, the pressure table is grayed out, since the pressure will be calculated by the system to increase with column temperature to maintain the selected column flow. In addition, the option of a Pressure Pulse injection will be presented. Pressure Pulse injection is used to minimize the expansion of the solvent vapor cloud in the injector and enhance transfer of the sample to the column, usually in conjunction with a splitless injection. If you click on the **Yes** button, you may enter a pressure and pulse duration for the injection. The system will stabilize all the temperature zones, then increase the pressure to the pulse pressure, stabilize, and hold until the injection is made and the run starts. The pulse pressure will be held for the pulse duration period, and will then be dropped to the pressure computed for the constant column flow selected.

Type 4 EFC (for valved systems)

Front EFC

Middle EFC

Rear EFC

Middle EFC Type:
Type 4 (for Valved Systems)

	Pressure (psi)	Rate (psi/min)	Hold (min)	Total (min)
1	10.0		1.00	1.00
2	18.7	0.51	2.00	20.06
3				
4				
5				
6				
7				
8				

Add
Insert
Delete

	Time	Total Flow (ml/min)
1	Initial	20
2	0.90	250
3	1.10	20
4		
5		

Add
Insert
Delete
Sort

Item	Description
Pressure (psi)	Pressure spreadsheet. 0.1 to 100.0 psi.
Rate (psi/min)	0.1 to 100.0 psi/min. The first line is devoted to initial conditions, so a rate cannot be entered. For subsequent lines the rate defines how quickly the pressure entered in the Pressure column will be attained.
Hold (min)	0 to 999.99 min. Total program time cannot exceed 999.99 min. Defines how long the pressure entered in the Pressure column will be held constant once that pressure is achieved.
Total (min)	Calculated by system based upon user entries in the (previous) 3 columns.
Time	Flow program spreadsheet. Initial to 999.99 min. Flows are executed as step changes at the programmed times. Commonly used to increase flow just before a valve is switched.
Total Flow (mL/min)	1 to 1000 mL/min. Be sure that total flow is adequate to support any pressure program steps you have entered.
Add	Adds a line to the spreadsheet. This button appears next to both the top and bottom spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet. This button appears next to both the top and bottom spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet. This button appears next to both the top and bottom spreadsheet.
Sort	Sorts the spreadsheet rows by time. This button appears next to the bottom spreadsheet.

3800 GC Column Oven

Column Oven Coolant: ☒ On ☐ Off

Enable Coolant at (C):

Coolant Timeout (min):

Stabilization Time (min):

	Temp (C)	Rate (C/min)	Hold (min)	Total (min)
1	80		1.00	1.00
2	180	20.0	0.00	6.00
3	250	5.0	10.00	30.00
4				
5				
6				
7				
8				

Add

Insert

Delete

Item	Description
Column Oven Coolant	On or Off. Specifies whether or not the column coolant will be used.
Enable Coolant at (°C)	30° to 450 °C. Specifies the temperature at which to enable the coolant.
Coolant Timeout (min)	0.01 to 999.99 min. Provides a safety measure to save coolant after the specified time, if either the column fails to reach its set temperature or the GC does not go into run.
Stabilization Time (min)	0 to 10.0 min. Specifies the column Stabilization Time. Adequate stabilization time will improve retention time precision in temperature programmed analyses.
Temp (°C)	-99° to 450 °C.
Rate (°C/min)	0.01° to 100.0 °C/min. The Rate in the first row is always blank and cannot be edited.
Hold (min)	0.01 to 999.99 min. Total program time cannot exceed 999.99 minutes.
Total (min)	0.01 to 999.99 min. Cannot be edited. Column Oven End Time (Total) is displayed in all time-programmed windows of the Method.
Add	Add a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.

3800 GC Detector

Front Detector

Middle Detector

Rear Detector

Front Detector Type: FID
Detector Oven: ☒ On ☐ Off

Temperature (C): 250
Electronics: ☒ On ☐ Off

	Time	Range	Autozero
1	Initial	12	yes
2			
3			
4			
5			

Add
Insert
Delete
Sort

Adjustments

Time Constant: ☐ Slow ☒ Fast
EFC Type: Type 11

Make up Flow (ml/min): 28

H2 Flow (ml/min): 30

Air Flow (ml/min): 300

Item	Description
Front Detector Middle Detector Rear Detector	The 3800 GC can have up to three detectors installed (front, middle, and rear). The tabs at the top of the window select the detector position.
Detector Type	None, FID, TCD, TSD, ECD, PFPD Specifies the type of detector installed in the selected position.
Detector Oven	On or off. Turns the detector oven in the selected position on or off.
Electronics	On or off.
Temperature (°C)	50° to 450 °C.
Methanizer...	Displays the Methanizer dialog box for the selected position.

Click on the following detector types for a description of the parameters that appear when the detector is selected.

FID Detector

Front Detector

Middle Detector

Rear Detector

Front Detector Type: FID
Detector Oven: ☒ On ☐ Off

Temperature (C): 250
Electronics: ☒ On ☐ Off

	Time	Range	Autozero
1	Initial	12	yes
2			
3			
4			
5			

Add

Insert

Delete

Sort

Adjustments

Time Constant: ☐ Slow ☒ Fast
EFC Type: Type 11

Make up Flow (ml/min): 28

H2 Flow (ml/min): 30

Air Flow (ml/min): 300

Item	Description
Detector Oven	On or off. Turns the detector oven on or off.
Electronics	On or Off. Indicates whether the installed FID will be used or not. Electronics must be ON to enable the ignitor.
Temperature (°C)	50° to 450 °C.
Time	Initial: 0.0 to 999.99 min.
Range	9, 10, 11, 12.
Autozero	Yes or No. Yes at initial time means that the FID Autozero is ON continuously before the run starts.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.
Time Constant	Slow or Fast.
EFC Type	None or Type 11 for the FID detector.
Make-up Flow (mL/min)	0 to 50 mL/min.
H2 Flow (mL/min)	0 to 50 mL/min.

Item	Description
Air Flow (mL/min)	0 to 500 mL/min.
Methanizer Installed	Yes or No.
Methanizer Oven	On or Off.
Temperature (°C)	50° to 450 °C.

TCD Detector

Front Detector

Middle Detector

Rear Detector

Middle Detector Type: TCD
Detector Oven: ☒ On ☐ Off

Temperature (C): 200
Electronics: ☒ On ☐ Off

Filament Temp (C): 266

	Time	Range	Autozero	Polarity
1	Initial	0.05	yes	positive
2		0.05		
3		0.5		
4		5.0		
5				

Add
Insert
Delete
Sort

Adjustments

Time Constant: ☐ Slow ☒ Fast
EFC Type: Type 14

Carrier Gas: He
Make up Flow (ml/min): 25

Filament Temp Limit (C): 390
Ref/Makeup Flow (ml/min): 30

Item	Description
Detector Oven	On or off. Turns the TCD oven on or off.
Electronics	On or Off. Indicates whether the installed TDC will be used or not.
Temperature (°C)	50° to 450 °C. Specifies the TCD Temperature.
Filament Temp (°C)	0° to 390 °C or 0° to 490 °C, depending on the filament temperature limit selected in the Adjustment section of this dialog. Specifies the Filament Temperature. For best lifetime, operate the TCD at the lowest practical filament temperature.
Time	Initial: 0.00 to 999.99 min.
Range (mV)	0.05, 0.5, and 5.0 mV.

Item	Description
Autozero	Yes or No. Yes at initial time means that Autozero is on continuously before the run starts. Autozero also enables the automatic cell balance.
Polarity	Positive or Negative. Allows peaks of either polarity to be processed by Data Handling.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.
Time Constant	Slow or Fast.
Carrier Gas	He or N ₂ /Argon. The automatic filament protection feature is disabled when N ₂ /Argon is selected.
Filament Temp Limit (°C)	390° or 490 °C. Automatic Filament Protection will limit filament temperature to this value during solvent peaks, etc.
EFC Type	None, Type 13, Type 14 , Type 16. Specifies the installed type. Type 13 is a single channel EFC used with packed columns. Type 14 is a dual channel EFC used with capillary columns and helium or nitrogen carrier. Type 16 is a dual channel EFC used with capillary columns and hydrogen carrier.
Make-up Flow (mL/min)	0 to 50 mL/min
Ref/Make-up Flow (mL/min)	0 to 100 mL/min

TSD Detector

Front Detector

Middle Detector

Rear Detector

Middle Detector Type: TSD
Detector Oven: ☒ On ☐ Off

Temperature (C): 250

Bead Current (A): 3.017

	Time	Range	Autozero	Bead Power
1	Initial	12	yes	on
2				
3				
4				
5				

Add
Insert
Delete
Sort

Adjustments

Time Constant: ☐ Slow ☒ Fast
EFC Type: Type 12

Make up Flow (ml/min): 28

H2 Flow (ml/min): 4.5

Air Flow (ml/min): 175

Item	Description
Detector Oven	On or off. Turns the detector oven on or off.
Temperature (°C)	50° to 450 °C. Specifies the TSD temperature.
Bead Current (A)	2.4 to 3.8 A.
Time	Initial: 0.00 to 999.99 min.
Range	9, 10, 11, 12.
Autozero	Yes or No. Yes at initial time means that the TSD Autozero is on continuously before the run starts.
Bead Power	On or Off.
Time Constant	Slow or Fast.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.
EFC Type	None or Type 12 for the TSD detector.
Make-up Flow (mL/min)	0 to 50 mL/min.
H2 Flow (mL/min)	0 to 6.0 mL/min.
Air Flow (mL/min)	0 to 200 mL/min.

ECD Detector

Front Detector

Middle Detector

Rear Detector

Middle Detector Type: ECD
Detector Oven: ☒ On ☐ Off

Temperature (C): 300
Electronics: ☒ On ☐ Off

	Time	Range	Autozero
1	Initial	1	yes
2	9.73	1	yes
3			
4			
5			

Add
Insert
Delete
Sort

Adjustments

Time Constant: ☐ Slow ☒ Fast
EFC Type: Type 13

Cell Current: CAP
Make up Flow (ml/min): 25

Contact Potential (mV): -213

Item	Description
Detector Oven	On or off. Turns the ECD oven on or off.
Electronics	On or Off. Indicates whether the installed ECD will be used or not.
Temperature (°C)	50° to 450 °C.
Time	Initial: 0.00 to 999.99 min.
Range	1 or 10.
Autozero	Yes or No. Yes at initial time means that the ECD Autozero is on continuously before the run starts.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.
Time Constant	Slow or Fast.
Cell Current	N2 High, N2 Std, CAP, Ar-CH4, Zero.
Contact Potential (mV)	-800 to +800 mV
EFC Type	None, Type 13 for the ECD.
Make-up Flow (mL/min)	0 to 50 mL/min.

PFPD Detector

Front Detector | Middle Detector | **Rear Detector**

Rear Detector Type: **PFPD** Detector Oven: ☒ On ☐ Off

Temperature (C): **300** Electronics: ☒ On ☐ Off

Square Root Mode: ☐ On ☒ Off

	Time	Range	Autozero
1	Initial	10	yes
2			
3			
4			
5			

Add
Insert
Delete
Sort

Adjustments:

Photomultiplier Voltage (V): **585** EFC Type: **Type 15**

Gate Delay (msec): **4.5** Air 1 Flow (ml/min): **17.0**

Gate Width (msec): **10.0** H2 Flow (ml/min): **13.0**

Trigger Level (mV): **200** Air 2 Flow (ml/min): **10.0**

Gain Factor: **12**

Use Auto Gain Factor? ☐ Yes ☒ No

Item	Description
Detector Oven	On or off. Turns the PFPD oven on or off.
Electronics	On or Off. Indicates whether the installed PFPD will be used or not.
Temperature (°C)	50° to 450 °C. Specifies the PFPD Temperature.
Square Root Mode	On or Off.
Time	Initial: 0.00 to 999.99 min.
Range	8, 9, 10.
Autozero	Yes or No. Yes at initial time means that the PFPD Autozero is on continuously before the run starts.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.
Photomultiplier Voltage (V)	300 to 900V.
Gate Delay (msec)	0.5 to 20 msec.
Gate Width (msec)	0.5 to 20 msec.
Trigger Level (mV)	10 to 2000 mV.

Item	Description
Use Auto Gain Factor	Yes, No Yes enables the built in compensation for gate width. No allows the user to fine tune gain to optimize dynamic range of the amplifier.
Gain Factor	2-63 Used when Use Auto Gain Factor = NO
EFC Type	None or Type 15 for the PFPD detector.
Air 1 Flow (mL/min)	0-50 mL/min.
H2 Flow (mL/min)	0-30 mL/min.
Air 2 Flow (mL/min)	0-30 mL/min.

3800 GC Output

	Time	Signal Source	Attenuation
1	Initial	Front	1
2			
3			
4			
5			

Use these fields to control the analog outputs from the GC. These parameters have no effect on the digital signal transmitted to the Varian Workstation.

Item	Description
Port A Port B Port C	The 3800 GC can have up to three output ports installed (A, B, and C). The tabs at the top of the window select the output port position.
Port Installed?	Yes or No. Indicates whether or not the Port at each position is installed. For the installed Port, the Output time program is used to program the detector signal source and attenuation.
Time	Initial: 0.00 to 999.99 min.
Signal Source	Front, Middle, Rear.
Attenuation	Powers of 2 from 1 to 1024 and Infinite.
Add	Adds a line to the spreadsheet.
Insert	Inserts a line above the currently selected row in the spreadsheet.
Delete	Deletes the currently selected row(s) in the spreadsheet.
Sort	Sorts the spreadsheet rows by time.

3800 GC Data Acquisition

Detector Bunch Rate: points (10.0 Hz)

Noise Monitor Length: bunched points
(6.4 sec)

The following settings will be ignored for all detectors other than the FID and TSD.

FID/TSD Detector Full Scale

Front:

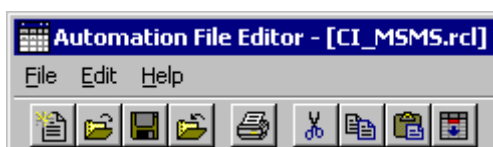
Middle:

Rear:

Item	Description
Detector Bunch Rate	Powers of 2 from 1 to 128 points. Sets the detector bunch rate in numbers of points; shows the resulting frequency in Hz. The base frequency is 40 Hz. Bunching points causes the frequency to decrease by $40 \div \text{bunch rate}$. With increased bunch rates, less disk space is required for storing the data. With decreased rates, more data is collected. Bunch rate should be set appropriately for the narrowest peak of interest in your chromatogram (~20 points/peak).
Noise Monitor Length	16 to 128 bunched points. Sets the number of points collected during the monitoring period, shows the resulting time in seconds. Used to set the number of data points used to calculate the baseline noise. Generally, a larger value entered here will yield a lower noise calculation.
FID/TSD Detector Full Scale (Font, Middle, Rear)	1V, 10V, 100V, 1000V. Specifies the maximum detector value that will be processed by the Varian MS Workstation. The dynamic range of the FID and TSD detectors are equivalent to 1000V full scale, but if your peaks of interest fall significantly below this value, you may wish to limit the full scale value here. <i>Since the attenuation setting used by the Standard Report application to scale the chromatogram printout is based on the detector's full scale value, a lower full scale value will allow you to scale smaller peaks with greater resolution.</i>

Automation File Editor Command Reference

Menus



File Menu

Menu Item	Description
New	A sub-menu displays RecalcList, SampleList and Sequence. Selecting one of them displays the Create a New Automation File dialog box to name a new file of the selected type.
Open	A sub-menu displays RecalcList, SampleList and Sequence. Selecting one of them displays the Open an Automation File dialog box to open an existing automation file of the selected type.
Save	Saves the currently displayed automation file with the existing name.
Save As	Displays the Save Automation File As dialog box, allowing you to save the currently displayed automation file with a new name.
Print	Prints the currently displayed automation file.
Printer Setup	Opens the Printer Setup. Select a printer and set options. You can open the Print Setup dialog box from the Printer Setup dialog box.
Exit	Quits the Automation File Editor. If any file is open and changes have not been saved, you will be prompted to do so.

Edit Menu

Menu Item	Description
Cut	Deletes a selection and copies it to the Clipboard. Used to remove or move a selected part of a spreadsheet.
Copy	Copies a selection to the Clipboard. Used to duplicate a selection and place the duplicate in a new place (using Paste).
Paste	Inserts previously cut or copied information that was stored in the Clipboard into a spreadsheet.
Clear	Deletes a selection but leaves the Clipboard unchanged.

Menu Item	Description
Add	Adds a new line in a SequenceList, SampleList or RecalcList.
Insert	Inserts a new line in a SequenceList, SampleList or RecalcList.
Select All	Selects all lines in a SequenceList, SampleList or RecalcList.
Fill Down	Causes the contents of the top cell in a series of added or existing, highlighted cells to be copied to the cells below it. Used to edit all the cells in a column quickly.
Edit Notes	Opens the Edit Notes dialog box to permit editing of the notes associated with the currently open automation file.

Help Menu

Menu Item	Description
Help Topics	Displays the help you are now viewing.
Product Support Web Site	If you have Internet access and a web browser installed on your computer, this option will automatically open the Varian MS Workstation Product Support Web Site. Here you will find the latest software and documentation updates for the Varian MS Workstation suite of products, along with additional notes, tips, and answers to frequently asked questions. You may wish to visit this site periodically to see if new information is available that may be pertinent to you.
About Automation File Editor	Displays the About Box for the Automation File Editor application. The About Box contains information about the software version, installation information, and a list of the instrument control modules that you have installed. You can validate the installed workstation files from this dialog.

Main Toolbar



New Automation File A sub-menu displays RecalcList, SampleList and SequenceList. Selecting one of them displays the Create a New Automation File dialog box to name a new file of the selected type.



Open Automation File A sub-menu displays RecalcList, SampleList and SequenceList. Selecting one of them displays the Open an Automation File dialog box to open an existing automation file of the selected type.



Save Automation File Saves the currently displayed automation file with the existing name.



Save Automation File As Displays the Save Automation File As dialog box, allowing you to save the currently displayed automation file with a new name.



Print Automation File Prints the currently displayed automation file.



Cut Deletes a selection and copies it to the Clipboard. Used to remove or move a selected part of a spreadsheet.



Copy Copies a selection to the Clipboard. Used to duplicate a selection and place the duplicate in a new place (using Paste).



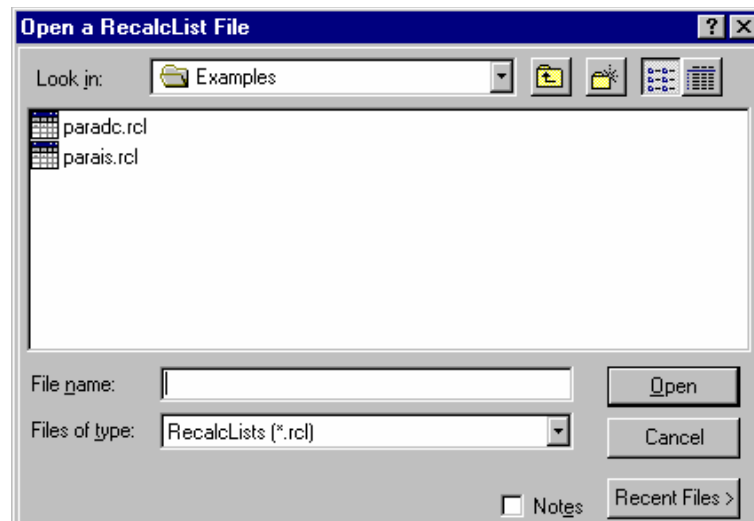
Paste Inserts previously cut or copied information that was stored in the Clipboard into a spreadsheet.



Fill Down Causes the contents of the top cell in a series of highlighted cells to be copied to the cells below it. Used to edit all the cells in a column quickly.

Open Automation File Dialog Box

This dialog box is used to specify or open an automation file. The Open Automation File dialog box appears with several different titles, depending upon how you access it. In all cases, it contains the fields listed below in forms appropriate for the type of file you are about to open.

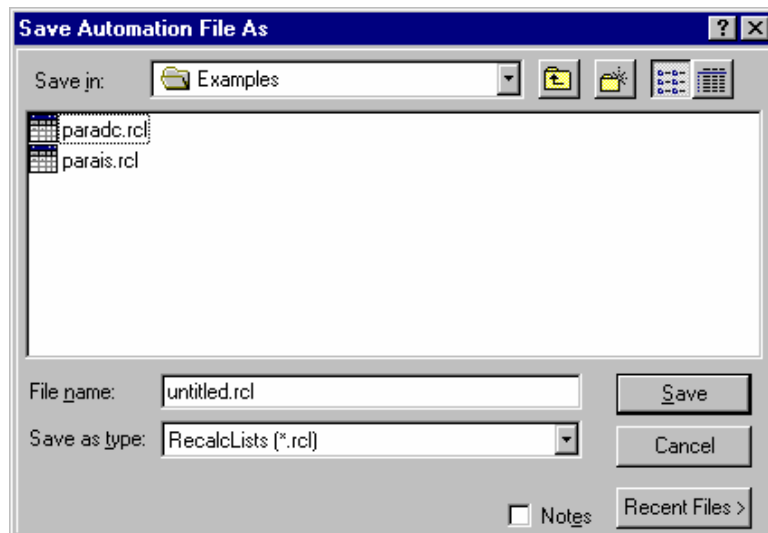


Item	Description
Look in	Lists the available folders and files. To see how the current folder fits in the hierarchy on your computer, click the down arrow . To see what is inside a folder, click it. The box below shows the folders and files in the selected location. You can also double-click a folder or file in that box to open it. To open the folder one level higher, click the up arrow button on the toolbar.
File list	Lists the folders and files in the selected location. To see what's inside a folder, double-click it. You can also use the Look In box to see the hierarchy of folders. To open the folder one level higher, click the up arrow button on the toolbar.
File name	Shows the currently selected file.
Files of type	Restricts the list of files to only those matching the selected type.

Item	Description
Recent Files	Click on this button to display a list of recently selected files. When you select a file from this list, its name is displayed in the File name box.
Notes	When checked, displays any notes and/or revision log associated with the currently selected file.
Open	Opens the selected file.
Cancel	Cancels file selection.

Save Automation File As Dialog Box

This dialog box is used to name an automation file. The Save Automation File As dialog box appears with several different titles, depending upon how you access it. In all cases, it contains the fields listed below in forms appropriate for the type of file you are about to save.

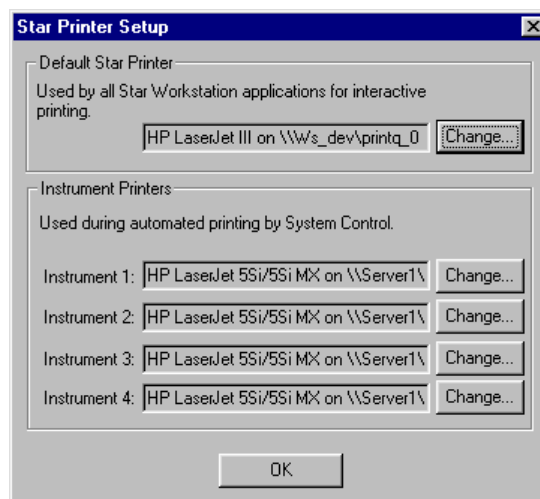


Item	Description
Save in	Lists the available folders and files. To see how the current folder fits in the hierarchy on your computer, click the down arrow . To see what's inside a folder, click it. The box below shows the folders and files in the selected location. You can also double-click a folder or file in that box to open it. To open the folder one level higher, click the up arrow button on the toolbar. To open a new folder in the current directory, click on the Create New Folder button.
File list	Lists the folders and files in the selected location. To see what's inside a folder, double-click it. You can also use the Save In box to see the hierarchy of folders. To open the folder one level higher, click the up arrow button on the toolbar.
File name	Shows the currently specified file.
Save as type	Specifies the type of file to save.
Recent Files	Click on this button to display a list of recently selected files. When you select a file from this list, its name is displayed in the File name box.

Item	Description
Notes	When checked, displays any notes and/or revision log associated with the currently selected file.
Save	Saves the file with the specified name.
Cancel	Cancels file saving.

Print Setup Dialog Box

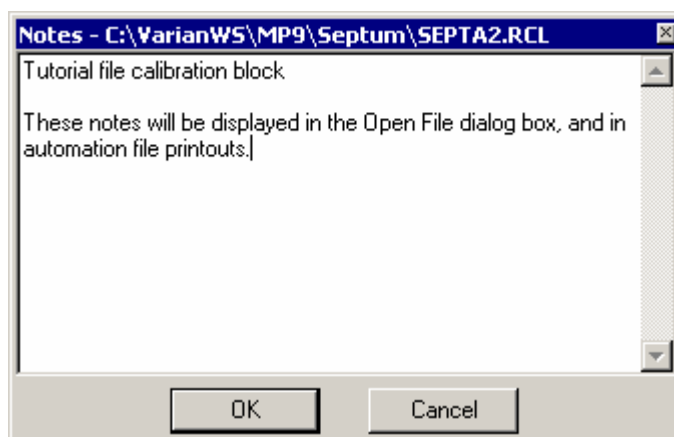
This dialog box is used to specify the printers that will be used by Varian MS Workstation applications both interactively and during automation.



Item	Description
Default Printer	Displays the printer that is used when printing interactively from Varian MS Workstation applications. Click on the Change button to select a different printer.
Instrument Printers	Displays the printer that is used when printing under automation from System Control. Since the Varian MS Workstation is a single instrument workstation, the printer specifications for instruments 2-4 will be ignored.

Edit Notes Dialog Box

This dialog box allows you to enter descriptive notes associated with an automation file. These notes can be viewed when selecting the file, and are included in the file's printout.

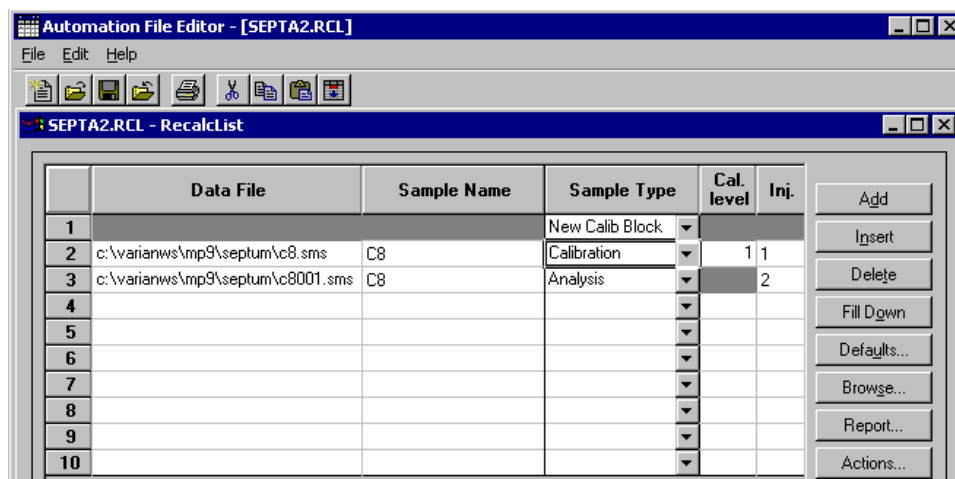


Item	Description
Notepad Area	Shows any notes attached to the file. The notes may describe the contents of the file, its uses, restrictions, etc. The Notepad Area is scrollable if the notes exceed the available space. The notes can be edited here, either by typing text directly, or by pasting text from the Clipboard by pressing the keys: CTRL V simultaneously.

RecalcList Window

Data Files can be added to a RecalcList by clicking on a cell in the Data File column and typing the file name, or by pressing the **Browse...** button and selecting the file name. Another way to quickly add one or more Data Files to a RecalcList is to select the files of interest in the Explorer and drag them over the RecalcList Window. When you release the mouse button, the Data Files are automatically appended to the RecalcList.

Note that a Recalc List may contain standard GC data files (.run extension) as well as MS data files (.sms extension). Some of the Recalc List items described below are handled differently for the two data file types. When the list is processed, the appropriate data handling will be used automatically for each data file.



Item	Description
Data File	Shows the name and path of the Data File for each sample.
Sample Name	Cannot be edited. Shows the name of each sample in the RecalcList.
Sample Type	Baseline, Analysis, Calibration, Verification, Print Calib, New Calib Block, AutoLink, Activate Method Sets the sample type, or automation action, of each line in the RecalcList.
Cal. Level	1 to 10 Sets the calibration level of each calibration or verification sample in the RecalcList.
Inj	Cannot be edited. Shows the number of injections made of each sample in the RecalcList.
Recalc Notes...	Up to 180 characters Opens the Notes window for the selected record to edit or create a note about the sample. This note is stored separately from the original injection notes.
AutoLink...	Two AutoLink commands when Sample Type is Baseline, Analysis, Calibration, or Verification. One AutoLink command when Sample Type is AutoLink. One Activate Method Command when Sample Type is Activate Method. Opens the AutoLink Parameters dialog box to set the options for linking to a remote application during automation through System Control. Opens the Activate Method Dialog Box to set the path name for activating a new method during automation of a RecalcList through System Control.
Unid Peak Factor	0 to 1,000,000.0 Sets a calibration factor for unidentified peaks. Not used by calibration samples.
Multiplier	0.000001 to 1,000,000.0 Sets a value for the multiplier. Results for the sample are multiplied by this value. Not used by calibration samples.
Divisor	0.000001 to 1,000,000.0 Sets a value for the divisor. Results for the sample are divided by this value. Not used by calibration samples.
Amount Standard	0.000001 to 1,000,000.0 GC Files: Sets the amount of the first internal standard. Used to calibrate results for Internal Standard and Normalized Percent calculations. Not used by calibration samples. MS files: Sets an ISFactor which is used by Analysis and Verification samples. It will be multiplied by the appropriate Compound Calibration Level Amount that is in the DH Method being used. Note that internal standards in Analysis and Verification samples always use the amount that is specified in Calibration Level 1.

Item	Description
MultiChannel MultiStandard	None, multiple, specific channel GC files: Opens the Data Handling Channels dialog box to specify the calibration parameters for up to four different Detector Channels. MS files: Not used by MS data handling. These are GC detector channels which are different from the scan function channels that may be specified in the MS method.
Add	Adds a line to the end of the RecalcList.
Insert	Adds a new line before the highlighted line.
Delete	Deletes the highlighted line in the RecalcList.
Fill Down	Causes the contents of the top cell in a series of highlighted cells to be copied to the cells below it. Used to edit all the cells in a column quickly.
Browse...	Opens the Open Data File dialog box to select a Data File to add to the RecalcList.
Report...	Shows the results report for the selected Data File.
Defaults...	Displays the Set Defaults dialog box, allowing you to specify default values for each applicable field in the RecalcList.
Actions...	Displays a list of applications which can be opened with the selected data file, e.g., Print Standard Report.
Begin	Opens the Begin RecalcList Dialog Box to specify a method to be used while recalculating or printing all RUN files in this RecalcList, and to then begin recalculating or printing the files. Only available in the System Control version of the RecalcList.
Suspend	Suspends execution of this RecalcList after the current file has been completed. Only available in the System Control version of the RecalcList.
Resume	Resumes execution of this RecalcList after it has been suspended. Only available in the System Control version of the RecalcList.

Data Handling Channels Dialog Box

NOTE: This dialog box is only used to specify standard GC data handling. MS data handling uses only one detector channel.

You can specify the Multiplier, Divisor, and Unidentified Peak Factor on a channel-by-channel basis for up to four different detector channels. And you can specify up to eight internal standards for each of the four channels, for a total of 32 internal standards. ***If you only want to specify a single Multiplier, Divisor, Unidentified Peak Factor, and Amount Standard to be used for all channels of all detectors, you may do so directly in the SampleList, RecalcList or Inject Single Sample dialog box and do not need to use this dialog box.***

If you plan to specify parameters on a channel-by-channel basis, before opening this dialog box make sure that the active method contains the data handling sections for each channel, and that their peak tables contain the standard peaks (if any) properly named and checked. The dialog box uses information from the active method to help you make the proper specifications for the type of calibration method that you will be using. ***If you later change the standard peak names in the peak table, you will need***

to revisit this dialog for each sample to update the names and amounts.

Data Handling Channels For Method Another.mth, Sample 'First analysis'

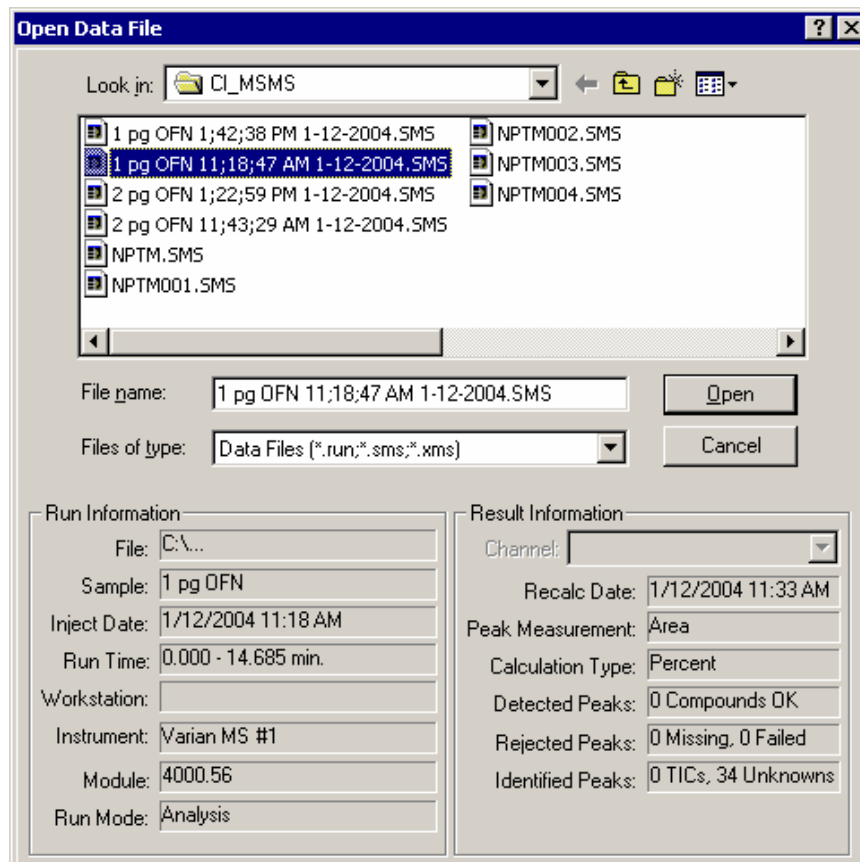
	Detector Channel	Calculation Type	Unid Peak Factor	Multiplier	Divisor	Standard Peak 1	Amount Standard 1
1	ADCB 16 Channel A	Internal Std	0	1	1	Peak 1.000	1
2							
3							
4							

Buttons: Add, Insert, Delete, Ok, Cancel, Edit Calibration Setup..., Edit Standard Peak(s)...

Item	Description
Detector Channel	Indicates the detector channel that is specified by the remaining fields in the row. Channels are indicated by the detector module name, bus address, and channel identifier. The combo box contains entries for all channels having data handling sections in the active method. If the desired channel is not visible in the combo box, make sure that the correct method is active and that it contains data handling sections for the desired channel.
Calculation Type	Internal Std External Std Normalized % No Calibration Indicates the calibration method specified for the detector channel in the active method. To change the calibration method for a specific channel, first click on the channel's row, then click on the "Edit Calibration Setup" push-button. If the Calculation Type is not Internal Std or Normalized %, the "Edit Standard Peak(s)" push-button will be disabled.
Unid Peak Factor	0 to 1,000,000 Sets a calibration factor for unidentified peaks.
Multiplier	0.000001 to 1.000,000 Sets a value for the multiplier. Results for the sample are multiplied by this value.
Divisor	0.000001 to 1,000,000 Sets a value for the divisor. Results for the sample are divided by this value.
Standard Peak 1	40 character standard peak name from peak table Indicates the name of the first Internal Standard Peak. The Amount Standard 1 will be applied to the Internal Standard Peak having the same name. To change the standard peak name for a specific channel, first click on the channel's row, then click on the "Edit Standard Peak(s)" push-button.

Item	Description
Amount Standard 1	0.000001 to 1,000,000 Sets the amount of the first internal standard. Used to calibrate results for Internal Standard and Normalized Percent calculations.
Standard Peak 2-8	40 character standard peak name from peak table Indicates names of the multiple internal standard peaks present in the specific channel's peak table. . To change the standard peak names for a specific channel first click on the channel's row, then click on the "Edit Standard Peak(s)" push-button. If the peak table does not contain multiple internal standards, these fields will be disabled.
Amount Standard 2-8	0.000001 to 1,000,000 Sets the amounts of the second through eighth internal standards. Used to calibrate results for Internal Standard and Normalized Percent calculations involving multiple internal standards. If the peak table does not contain multiple internal standards, these fields will be disabled.
Add	Adds a line to the end of the list.
Insert	Adds a new row before the highlighted line.
Delete	Deletes the highlighted row in the list.
Edit Calibration Setup	Opens the Calibration Setup Dialog Box to permit inspection and editing of the calibration parameters in the method for the indicated detector channel.
Edit Standard Peak(s)	Opens the Peak Table Dialog Box to permit inspection and editing of the Standard Peak names in the method for the indicated detector channel. Open the Peak Table and press OK to transfer the standard peak names and amounts from the Peak Table to the indicated detector channel row.

Open Data File Dialog Box

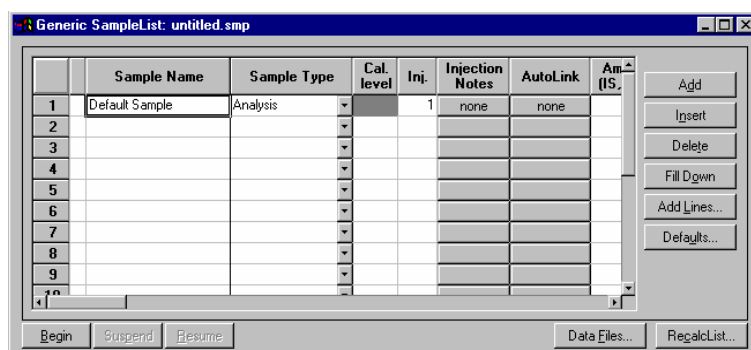


Item	Description
Look in	Lists the available folders and files. To see how the current folder fits in the hierarchy on your computer, click the down arrow . To see what is inside a folder, click it. The box above shows the folders and files in the selected location. You can also double-click a folder or file in that box to open it. To open the folder one level higher, click the up arrow button on the toolbar.
File list	Lists the folders and files in the selected location. To see what's inside a folder, double-click it. You can also use the Look In box to see the hierarchy of folders. To open the folder one level higher, click the up arrow button on the toolbar.
File name	Shows the currently selected file.
Files of type	Restricts the list of files to only those matching the selected type.
Run Information	Shows information about the currently selected file.
Result Information	Shows information about the results, if any, calculated from the current data file.
Channel	Specifies the detector channel to show Result Information for. Note that this applies only to standard GC results.

Item	Description
Delete Results	Allows you to delete results from the currently select channel of the currently selected data file. This button does not appear if it has been disabled from the Varian MS Workstation Security Administration application. Note that this only affects standard GC results. MS results are not deleted.
Open	Opens the selected data file using the selected channel.
Cancel	Cancels file selection.

SampleList Window

The SampleList window contains injection parameters that are specific to the sample introduction device you are using on your instrument. This section describes the generic SampleList fields. Refer to the appropriate Instrument Control help topic in the System Control Reference Help for a description of the device-specific extensions to the SampleList.

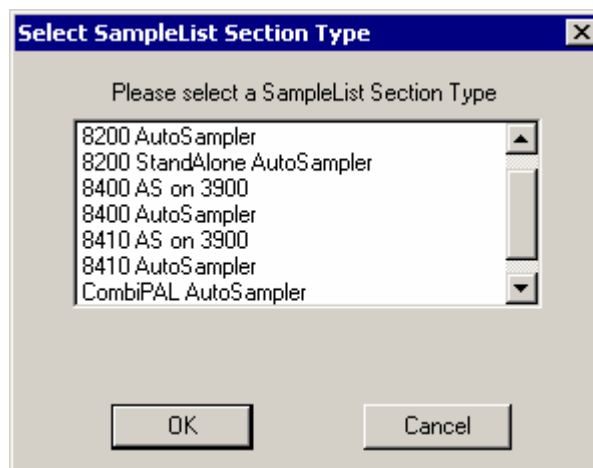


Item	Description
Sample Name	Up to 19 characters Sets the name of each sample in the SampleList
Sample Type	Baseline, Analysis, Calibration, Verification, Print Calib, New Calib Block, AutoLink, Activate Method Sets the sample type, or automation action, of each line in the SampleList.
Cal. Level	1 to 10 Sets the calibration level of each calibration or verification sample in the SampleList.
Inj	1 to 9 Sets the number of injections to be made of the sample.
Injection Notes	Up to 180 characters Opens the Notes window for the selected sample to edit or create a note about the sample.

Item	Description
AutoLink	Two AutoLink commands when Sample Type is Baseline, Analysis, Calibration, or Verification. One AutoLink command when Sample Type is AutoLink. One Activate Method Command when Sample Type is Activate Method. Opens the AutoLink Parameters dialog box to set the options for linking to a remote application during automation through System Control. Opens the Activate Method dialog box to set the path name for activating a new method during automation of a SampleList through System Control.
Unid Peak Factor	0 to 1,000,000.0 Sets a calibration factor for unidentified peaks. Not used by calibration samples.
Multiplier	0.000001 to 1,000,000.0 Sets a value for the multiplier. Results for the sample are multiplied by this value. Not used by calibration samples.
Divisor	0.000001 to 1,000,000.0 Sets a value for the divisor. Results for the sample are divided by this value. Not used by calibration samples.
Amount Standard	0.000001 to 1,000,000.0 GC Files: Sets the amount of the first internal standard. Used to calibrate results for Internal Standard and Normalized Percent calculations. Not used by calibration samples. MS files: Sets an ISFactor which is used by Analysis and Verification samples. It will be multiplied by the appropriate Compound Calibration Level Amount that is in the DH Method being used. Note that internal standards in Analysis and Verification samples always use the amount that is specified in Calibration Level 1.
MultiChannel MultiStandard	none, multiple, specific channel GC files: Opens the Data Handling Channels dialog box to specify the calibration parameters for up to four different Detector Channels. MS files: Not used by MS data handling. These are GC detector channels which are different from the scan function channels that may be specified in the MS method.
Add	Adds a line to the end of the SampleList.
Insert	Adds a new line before the highlighted line.
Delete	Deletes the highlighted line in the SampleList.
Fill Down	Causes the contents of the top cell in a series of highlighted cells to be copied to the cells below it. Used to edit all the cells in a column quickly.
Add Lines...	Displays the Add Lines dialog box, allowing you to specify the number of lines to either insert or append to the spreadsheet, along with the values to use for each applicable field. Certain fields such as Sample ID and vial number can be automatically incremented.
Defaults...	Displays the Set Defaults dialog box, allowing you to specify default values for each applicable field in the SampleList.

Item	Description
Data Files...	Opens the Data File Generation dialog box to specify the naming scheme being used for Data Files generated from injections. Note that if the method contains both MS and standard GC DH method sections, then both sms and run files will be generated.
RecalcList...	Opens the RecalcList Generation dialog box to specify the options for generating or updating RecalcLists after injections. Note that if the method used contains both MS and standard GC DH sections, then the generated Recalc List will contain both sms and run files.

Select SampleList Section Type Dialog Box

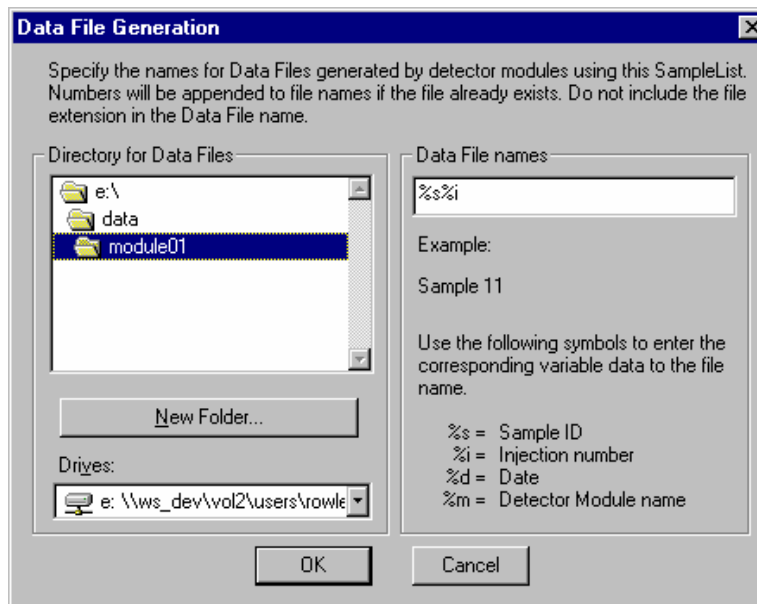


A SampleList File may contain multiple SampleList Sections. Each installed AutoSampler can contribute its own unique type of section, having special features that correspond to its special hardware. Generally each installed AutoSampler has its own unique SampleList with special columns for its features. A SampleList File can contain a section for each type of AutoSampler, similar to the way that a Method file can contain a section for each type of Instrument Module.

This dialog box lets you pick the specific section that you want to edit in a SampleList file that may contain more than one section.

Item	Description
SampleList section type list box	Selects a section type to use in building a new SampleList; opens the appropriate SampleList window. SampleLists may contain sections for multiple types of sample handling devices, depending upon which module drivers have been installed.
OK	Creates a SampleList section of the specified type.
Cancel	Cancels the SampleList section creation.

Data File Generation Dialog Box



Item	Description
Directory for Data Files	Shows the currently selected directory where Data Files will be generated.
New Folder	Allows you to create and name a new folder in the currently displayed directory.
Drives	Allows you to specify the drive where Data Files will be generated.
Data File Names	Allows you to enter the Data File name specification. You may enter as many characters as you wish, but the final file name (including path) must not exceed 255 characters in length. You may embed the special symbols listed in the window to represent sample-specific information in the Data File name. As you enter the specification, an example file name is shown.
OK	Accepts the name specification.
Cancel	Cancels the name specification.

RecalcList Generation Dialog Box

RecalcList Generation

You can automatically create or update a RecalcList with files generated during automated injections. Specify the RecalcList generation options for this SampleList below.

☐ Do not automatically create and update a RecalcList.

☒ Create and update a new RecalcList.

RecalcList name:

☐ Append to an existing RecalcList.

RecalcList name:

☐ Overwrite the Recalc List each time the SampleList Begins.

Item	Description
Do not automatically create and update a RecalcList.	No RecalcList will be generated or updated when you create Data Files as a result of injections.
Create and update a new RecalcList	Allows you to enter the name of a new RecalcList that will be created when Data Files are generated as a result of injections. If the RecalcList name that you specify already exists when the injections are performed, a number will be appended to the name so that it is unique.
Append to an existing RecalcList	Allows you to browse for an existing RecalcList file. When Data Files are generated as a result of injections, their information will be appended to this file.
OK	Accepts the RecalcList specification.
Cancel	Cancels the RecalcList specification.

AutoLink Parameters Dialog Box

With AutoLink, you can specify a program to be executed after all injections of a sample have been performed, or two programs to be executed after each injection of that sample. When AutoLink appears as a sample type in a SampleList or RecalcList, the associated program is executed at that point in the SampleList / RecalcList after the previous SampleList / RecalcList line has completely finished. The AutoLink can also be specified as part of a SampleList line that has a Baseline, Analysis, Calibration, or Verification sample type. In such cases, the two AutoLink programs are executed after each injection of the sample.

AutoLink Parameters

Command

Other parameters

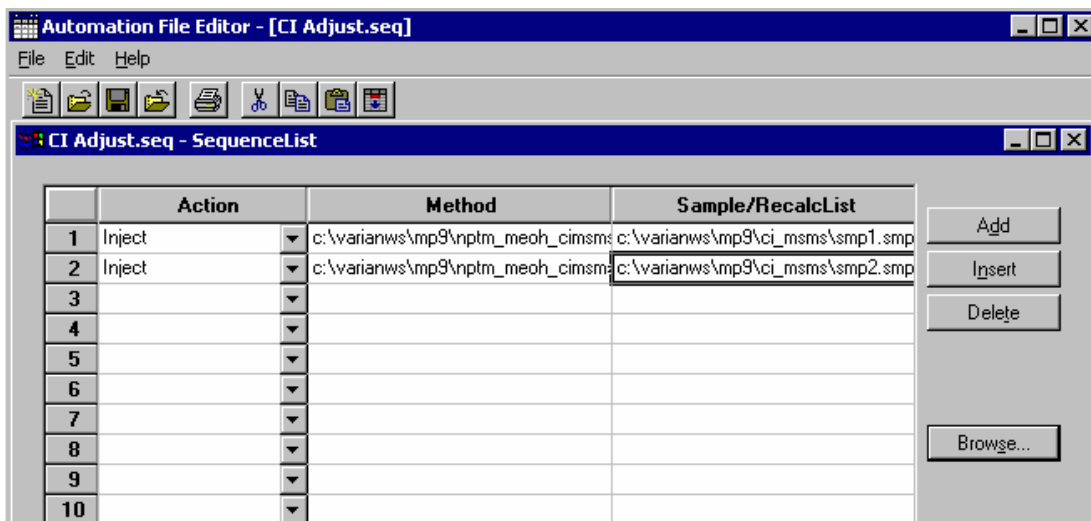
Browse...

OK

Cancel

Item	Description
Command	DOS command lines to open DDE applications Sets the command line that will be used when AutoLink is invoked.
Other Parameters	Commands understood by the remote application. Provides information to the remote application when it is invoked.
Browse...	Opens the Select an AutoLink Program dialog box to select the name of an AutoLink program and enter it into the Command field.

SequenceList Window



Item	Description
Action	Shows the action chosen for each line in the Sequence.
Method	Shows the Method chosen for each line in the Sequence.
Sample/RecalcList	Shows the SampleList or RecalcList chosen for each line in the Sequence.
Add	Adds a new line to the end of the Sequence.
Insert	Adds a new line above the highlighted line.
Delete	Deletes the highlighted line in the Sequence.
Browse	Opens the Select a Method File or Select a Sample/RecalcList window to browse through directories and find a Method or Sample/RecalcList file to add to the Sequence.