

Agilent G1732AA MSD Security ChemStation

User Guide



Notices

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A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

In this manual...

This manual contains condensed information that is often needed by MSD Security ChemStation users.

1 Introduction

This chapter describes the rest of the manual.

2 Online Help

This chapter describes the controls and icons that control online help.

3 Instruments

This chapter describes the controls that set and monitor instrument parameters.

4 Menus

This chapter displays the menus that control the software.

5 Procedures

This chapter contains a number of frequently used procedures, both hardware and software.

6 Tips

This chapter contains brief descriptions of operation and troubleshooting tips.

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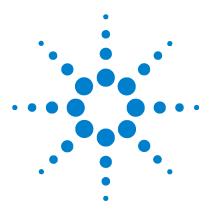
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Introduction

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NOTE

The G1732AA MSD Security ChemStation is software that should be installed on a computer dedicated to this specific application. This software can support either GPIB or local LAN acquisition from one MSD instrument. Other applications and/or wide area network access should be performed on other computers.

This guide is a collection of information about the system that has proven useful to other users. Use the online help capability, described in Chapter 2, for additional information.



Special Screens

These screens are used throughout the MSD Security ChemStation software.

The MSD Security Monitor screen

This window appears in the lower right corner of the screen after installation (Figure 1).

MSD Security Monitor			
Current User:			
Change Password	Frivate Lock		
Help	Lock Session		

Figure 1 The MSD Security Monitor

It can be moved to other positions but must remain visible whenever the MSD Security ChemStation software is in use. If it is not present, restart it using **Start > All Programs > MSD Security ChemStation > MSD Security Monitor**.

The monitor displays the Full Name of the current user, as required by the FDA regulations. It also provides these functions:

- **Change Password**—Used at first login to set the user password, and to change password when the current one times out.
- Lock Session—Locks the session until an authorized user who is a member of one or more of the MSD Security ChemStation user groups unlocks it.
- **Private Lock**—By checking this function and pressing **Lock Session**, the session is locked until the current user or a Manager unlocks it.
- Help–Opens an online help screen.

The non-private lock is typically used when operators change while the system is running, for example, during shift changes.

The login screen

This screen (Figure 2) appears when a user attempts to run one of the three secure applications.

Enter Login Name	×
UserID	
Password	
OK Cancel Help	

Figure 2 Login screen

It requires the user id, which is assigned by a Windows Administrator, and the user password, which is chosen by the user and known only to the user.

The signature screen

Whenever any form of information (new or modified method or sequence, run data, reports, etc.) is to be saved, the Signature screen appears (Figure 3).

Signature Panel			
Saving Method Version for C:\MSDChem\1\data\DEFAULT.M			
User ID: Password:			
Comment: (use CTRL-ENTER to enter multiple lines of text)			
			
-			
Sign Help			

Figure 3 Signature screen

1 Introduction

After identifying the user and providing a password to confirm the identity, the user *must* enter a comment. This comment becomes a permanent part of the audit trail system. We recommend that it be a brief note explaining why the information is being saved.



Use online help to learn about instrument control, data acquisition, data analysis, methods, sequencing, and tuning.



Help Controls

To access online help, select **Help Topics** from the Help menu in any window or click **Help** on any dialog box. See Table 1 for the available controls.

ltem	Description	
• Hide/Show	Lets you turn on or off the display of the list of help topics	
• Back	Goes back to the previous help topic	
• Print	Lets you print the current book or help topic	
Contents	Displays the list of help topics	
• Index	Lets you use keywords to search the help index for a particular topic	
• Search	Lets you type a word or phrase and then displays a list of al the topics in the online help that contain those words	

Table 1Help controls

Help Icons

The icons shown in Table 2 appear in some of the Help topic listings.

•	٠	Indicates a book containing more help topics. To open a book, select it, then double-click.
•	(<u>n</u>	Indicates an open book of help topics. To close an open book, select it, then double-click.
•	?	Indicates a help topic. To jump to a help topic, select it, then double-click.

Table 2Help icons

2 Online Help



The Instrument Control view is where you set and monitor instrument parameters.



Instrument Control View

The Instrument Control View (Figure 4) is displayed when you start up the MSD ChemStation. If you are in a different view, select View / Instrument Control when you are ready to set up the instrument for data acquisition.

See the online help for more details on the menus, buttons (Table 3), or windows used in the software.

Instrument Contro	I	
	Sample Name: Data File: evaldemo.d	me 💮 📓 😵
Sample	Chromatography	Detectors
Injector	Columns Oven	Aux MS
	GC Status Messages 🛛 💉 💌	Total Ion EM Volts
	70 Oven Temperature Column-1 Flow Cal.	
	230 150 MS Source MS Quad	TurboSpd

Figure 4 Instrument Control View

Instrument Control Buttons

-

Injector

The buttons that appear in the Instrument Control view are described in Table 3.

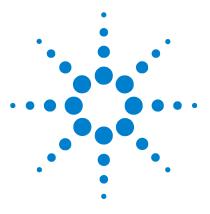
Offline	Acquisition Status Lets you tell at a glance the status of the current instrument or run (if any).		
Sample Name: sb1021 Data File: evaldemo.d	Start Lets you start a run. When a run is not in progress, the arrow is green. To start a run, click the green arrow, fill in the Sample Information, and click Run Method. When a run is in progress, the arrow is gray. This button also shows the current data file name, sample information (if any), and the vial number (1 if no ALS).		
0.71 Run Time: 12.00	Run Time Clock Shows the elapsed time since the beginning of the run if a run is in progress. The scheduled run time is shown below the digital clock. When a run is not in progress, the Run Time clock shows the elapsed time since the last run.		
(STOP)	Stop Lets you stop the system when it is in Pre-Run, Run, or Post-Run mode. The stop sign is red when a run is in progress and gray when a run is not in progress.		
	Logbook Displays a menu where you can choose to view, open, save, or print a particular logbook.		
?	Help Displays a menu where you can select an online help topic for the Instrument Control view. Choose Help Topics to go to the online help for the entire system.		
N.	Injector Lets you set parameters for the injector when you have configured your system with an ALS.		

Table 3 Instrument Control buttons

3 Instruments

	Inlets Lets you set parameters for the GC inlet (injection port) that
	is configured on your system.
Columns	Columns Lets you configure columns and set up ramped flow and pressure programs.
Oven	Oven Displays a menu that lets you edit GC oven parameters or GC monitor parameters. When the GC is ready, the small square in the upper right corner is green. When the GC is not ready, the square is red.
Aux	Aux Lets you set the temperature of the GC/MSD interface (usually Thermal Aux 2).
MS	MS Displays a menu that lets you edit MS acquisition parameters, select another tune file, or edit MS monitors.
Total Ion	Monitor Each monitor displays one instrument parameter. See the online help for a description of the instrument monitors.

Table 3 Instrument Control buttons (continued)



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Menus

4

System Configuration Menus 22 Top Menus 23 Reprocess Top Menus 24 Instrument Control Menus 25 Tune and Vacuum Control Menus 26 Data Analysis Menus 27 EasylD Menus 28 QEdit Menus 29 Parametric Retrieval Menus 30 RTLock Menus 31

The major control menus are shown in Figure 5 through Figure 14 for an offline instrument.



System Configuration Menus

File	Configure	Archive/Restore
Delete Instrument Exit	Instrument 1	Archive Master Method Archive Sequence Archive Results (Data Files)
		Archive All Archive All Results (Data Files)
		Restore Archive
Help		
Help		
Network Information Check Networking Check Networking and	Update MSD Firmware	
Review Revision Histor View Revision Readme About		

Figure 5System Configuration menus

Menus 4

Top Menus

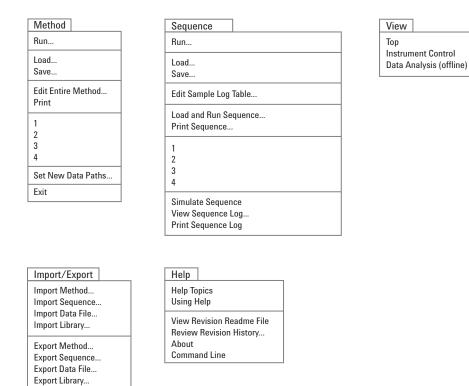


Figure 6 Top Menus

Reprocess Top Menus

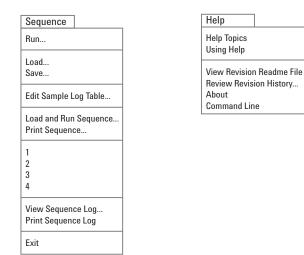


Figure 7 Reprocess menus

Instrument Control Menus

Method	Instrument	View
Run	Inlet/Injection Types	Top Instrument Control
Load Save	GC Edit Parameters GC Plot	Data Analysis (offline)
Edit Entire Method	GC Monitors	Tune and Vacuum Control
Additional Method Information Print	Acquire RTLock Calibration Data Unlock Method	
1 2	Disable Method Resolution	Abort
3 4 Exit	Select MS Tune File MS SIM/Scan Parameters Change MSD Temperatures MS Monitors	
	Perform MS Autotune	
	Pump Down Vent	
	EMF Utilities	
Window	Qualify	Help
Cascade Tile Horizontal	Checkout Tune Tune Evaluation	Help Topics Using Help
Tile Vertical Arrange Icons Arrange Plots and Monitors	View Tunes Sensitivity Check Software Validation	Review Revision History View Revision Readme file About
1 Instrument Control 2 Total Ion	Installation Checklist Familiarization Checklist	Command Line

- 2 Total Ion
- 3 Spectrum @ 0.00 min.

Figure 8 Instrument Control menus

Familiarization Checklist

Tune and Vacuum Control Menus

File	Tune
Reset to Defaults Load Tune Values Save Tune Values	Tune N QuickT
Trace on	Autotu Low M
View Trace Instrument Trace	Standa Tune V
Print Printer Orientation Generate Report	Air and Tune E
View Tunes Set View Tunes Report Options	
Restore Previous Tune(s) Exit	
Execute	
MS ON MS OFF	-
Profile scan Repeat profile Detailed profile	-
Ramp Lenses	
Spectrum scan Find mass	
Determine Mass Axis Correction Factors Optimize Quadrupole Frequency	
Status	ļ
MS Status MS I/O Test MS Temperature Controller Status	
View Instrument Macro Log	L

MS Error Codes... Logbook/Sequence Log Error Codes...

View Tune Log...

MSD Tune

ine (Atune.U) lass Autotune (Lomass.U)

ard Spectra Tune (Stune.U) Nizard...

d Water Check Evaluation

Vacuum

Vacuum Status...

Pump Down... Vent...

Purge Calibrant Valve **Power-on Temperatures** Initialize MS Interface

Calibrate		
Mass axis		
Mass #2 widths		
Mass #3 widths		
Peakwidth Linearity		
Adjust Abundances		
Ramp Repeller		
Ramp Ion Focus		
Ramp Entrance Lens		
Ramp Entrance Lens Offset		
Maximize Sensitivity		
View		

Parameters

Manual Tune...

Edit Tune and Display Parmeters... Edit MS Temperatures... Edit Ramp Parameters...

Edit Dynamically Ramped Parameters

View	
Тор	
Instrument Control	
Data Analysis (offline)	

Abort

Abort

Help
Help Topics Troubleshooting Using Help
About

Figure 9 View/Tune and Vacuum Control in Instrument Control

Data Analysis Menus

File	Method	Chromatogram
Load Data File List Header	Run Method	Chromatogram Scaling Draw Chromatogram - No Labels
Printer Setup Print	Load Method Save Method	Draw Chromatogram With Labels Select Chromatogram Labels
Abort	Edit Method	Extract Ion Chromatograms Display Ion Chromatograms in Merged Format
1 2 3	1 2 3	MS Signal Integration Parameters GC Signal Integration Parameters AutoIntegrate
Exit		Integrate Percent Report
Se e eterrer	O -librate	
Spectrum	Calibrate	Quantitate
Tabulate Select Library	Set Up Quantitation AotoQuant Setup	Calculate Generate Report
Edit Strategy	Edit Compounds	· · · · · · · · · · · · · · · · · · ·
Edit Library	Update	Report Options
Library Search Report	List Clear	Report Non Target Peaks
Tools	View	Help
Set Overlay Parameters Overlay Chromatograms	EasyID QEdit Quant Result	Help Topics Using Help
Signal to Noise check	Edit Non-Target Peaks Parametric Retrieval	Review Revision History View Revision Readme file
Change Data State Options	RTLock Setup	About
Copy Window Reset Windows		
View Audit File		
View Datafile Tune Parameters		
CFR Report		

Figure 10 View/Data Analysis (offline)

EasyID Menus

EasyID

CO Next Compound Goto Compound # Xpand Chromatographic Window +/-2 Minutes Original Chromatographic Window GRaphics Report to the Printer

Restart Quick EasyID

Set Extract Windows Start Set Extract Windows Stop

Abort Changes and Exit EXIT and Save Changes

Spectrum
Draw Next Draw Previous Tabulate
Select Library Edit Strategy Edit Library
Display Reference Spectrum
Update Reference Spectrum

Help

Help Topics

Integrate		_	View	
	eters pecific integration parameters d specific integration parameters		Data Ar	nalysis
Integrate		1		

Figure 11 View/EasyID in Data Analysis

QEdit Menus

QEdit	Spectrum	Display
CO Next Compound Goto Compound # Xpand Chromatographic Window	Draw Next Draw Previous Tabulate	Normal Qualifiers
ORIginal Chromatographic Window Graphics Report to the Printer	Select Library Edit Strategy	View
QDEL Compound	Edit Library	Data Analysis
Next File Load Data File	Display Reference Spectrum	
Restart Quick EasyID	-	Help Help
Abort Changes and Exit EXIT and Save Changes		Help Topics

Figure 12 View/QEdit Quant Result in Data Analysis

Parametric Retrieval Menus

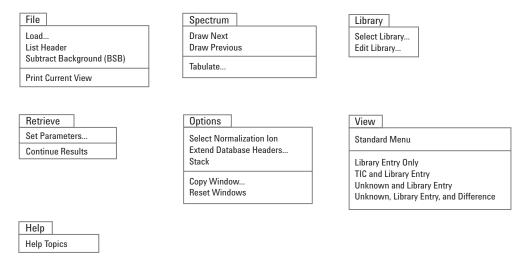


Figure 13 View/Parametric Retrieval in Data Analysis

RTLock Menus

RTLock	View	Help
View Current Method Setpoints Calculate New Curve from Selected Peaks Restore Nominal Chromatogram	Data Analysis	Help
Report RTLock Calibration Print		
Relock Method Use AutoLock		
Unlock Method		

Figure 14 View/RTLock menus

4 Menus



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This chapter contains a set of commonly-used procedures for the GC/MSD system.



To Pump Down (Start) the MSD

- **1** Make sure your system meets all of the following conditions before you pump down:
 - The vent valve is closed. The knob is turned all the way clockwise.
 - All other vacuum seals and fittings are in place and fastened correctly. (The front side plate screw should not be tightened unless hazardous carrier or reagent gases are being used.)
 - The MSD is connected to a grounded power source.
 - The GC/MSD interface extends into the GC oven.
 - A conditioned capillary column is installed in the GC inlet and in the GC/MSD interface.
 - The GC is on, but the heated zones for the GC/MSD interface, the injection port, and the oven are off.
 - Carrier gas of at least 99.999% purity is plumbed to the GC with the recommended traps.
 - If hydrogen is used as carrier gas, carrier gas flow is off and the front sideplate thumbscrew is loosely fastened.
 - The foreline pump exhaust is properly vented.

WARNING Make sure your MSD meets ALL the conditions listed above. Failure to do so can result in personal injury.

2 From instrument control, select **Pump Down** from the Instrument menu.

Note: Analysts and managers in the Tune view can select **Pump Down** from the Vacuum menu.

- **3** When prompted, switch on the MSD.
- **4** Press lightly on the side board to ensure a correct seal.

The rough pump will make a gurgling noise. This noise should stop within a minute. If the noise continues, there is a **large** air leak in your system, probably at the side plate seal, the interface column nut, or the vent valve.

5 Once communication with the PC is established, click **OK**. Within 10 to 15 minutes the diffusion pump should be hot, or the turbo pump speed up to 80%. The turbo pump should eventually reach 95%.

CAUTION If these conditions are not met, the foreline pump will be shut off. You must then power cycle the MSD. If the MSD does not pump down correctly, see the online help for information on troubleshooting air leaks and other vacuum problems.

6 When prompted, turn on the GC/MSD interface heater and GC oven. Click **OK** when you have done so. The software will turn on the ion source and mass filter (quad) heaters. The temperature setpoints are stored in the current autotune (*.u) file.

CAUTION

Do not turn on any GC heated zones until carrier gas flow is on. Heating a column with no carrier gas flow will damage the column.

7 After the message **Ok to run** appears, wait 2 hours for the MSD to reach thermal equilibrium.

CAUTION

Data acquired before the MSD has reached thermal equilibrium might not be reproducible.

To Vent (Shut Down) the MSD

- **1** If your system is equipped with a gauge controller, switch off the triode gauge controller.
- **2** Before venting a CI MSD, press **Gas Off** (turns off the reagent gas flow and closes the isolation valve.

WARNING On a CI MSD, the Gas Off light must be on when the MSD is venting.

3 From the Instrument view, select **Vent** from the Instrument menu in the software. Follow the instructions presented.

Note: Analysts and managers in the Tune view can select **Vent** from the Vacuum menu.

4 Set the GC/MSD interface heater and the GC oven temperatures to ambient (25 °C).

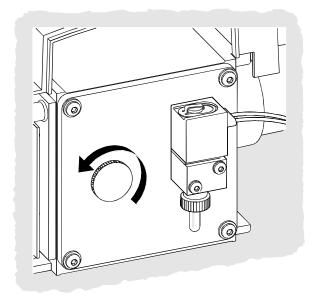
WARNING

If you are using hydrogen as a carrier gas, the carrier gas flow must be off before turning off the MSD power. If the foreline pump is off, hydrogen will accumulate in the MSD and an explosion may occur. Read the Hydrogen Carrier Gas Safety Guide (5955-5398) before operating the MSD with hydrogen carrier gas.

CAUTION

Be sure the GC oven and GC/MSD interface are cool before turning off carrier gas flow.

- **5** When prompted, turn off the MSD power switch.
- **6** Unplug the MSD power cord.
- **7** Remove the analyzer cover.



8 Turn the vent valve knob counterclockwise **only** 3/4 turns or until you hear the hissing sound of air flowing into the analyzer chamber.



CAUTION Do not turn the knob too far, or the O-ring may fall out of its groove. Be sure to retighten the knob before pumping down.

WARNING Allow the analyzer to cool to near room temperature before touching it.

CAUTION Always wear clean gloves while handling any parts that go inside the analyzer chamber.

WARNING

When the MSD is vented, do not put the ChemStation into the Top view. Doing so will turn on the interface heater.

5 Procedures

To Tune the MSD

You should tune the MSD periodically to maintain its optimum performance. Tuning is the process of adjusting MSD parameters so the instrument meets certain performance criteria. How often you should tune is determined by the number and type of samples you are running, as well as the overall condition of your system.

Always tune the MSD with the same GC oven temperature, column flow, and analyzer temperature that will be used for data acquisition.

Keep the Tune reports in a notebook so that successive reports can be easily compared.

To use Autotune

1 From the Instrument Control view, select **Perform MS Autotune** from the Instrument menu.

Note: Analysts and managers in the Tune view can select **Autotune** from the Vacuum menu.

- **2** Review the Tune report.
- 3 To view the history of tune results, select View Tunes from the Qualify menu.

To use Manual Tune

Manual tuning lets you interactively set the MSD parameters, such as lens voltages and tuning masses, to values that meet the needs of your particular analysis. You can often obtain greater sensitivity than you can with autotune.

Manual tuning allows you to ramp individual parameters and to specify the range and step size for the ramp. The results of the ramp are displayed visually with the optimum value for the parameter clearly marked on the plot.

You can acquire two types of data in manual tune: profile scans (plots the abundance and peak shape of the tune masses) and spectrum scans (scans plot response across the entire mass range).

See the online help for more details about manual tuning.

5 Procedures

To Acquire Data

To set up the GC for use with the MSD

- 1 Select Instrument/Inlet/Injection Types. Select the appropriate injection source and select the Use MS checkbox. Click OK.
- 2 Click Aux. Verify that you are using auxiliary channel 2, the heater is on and set to the desired temperature, and that MSD is selected as the Type. Click OK.
- **3** Click **Columns**. Verify that the detector is **MSD** and that **Vacuum** is selected for Outlet psi. Click **OK**.

To inject a sample with the autosampler

- **1** Place the autosampler vial containing the sample into the autosampler tray.
- 2 Select **Run** from the Method menu in the Top or Instrument Control view.
- **3** When the Start Run box appears, specify the sample information:
 - Specify a unique data file name for the sample.
 - Enter the position number of the sample vial in the **Vial** field (1–100).
 - (optional) Fill in the **Sample Name** and **Misc Info** fields to document the injection.
 - Make sure that the **Data Acquisition** option is selected. Select the Data Analysis option if you want to generate any of the reports specified in the method.
- 4 Click **Run Method** to initiate the run.

CAUTION

Do not use the Start button on the GC to start a run when using the autosampler.

To inject a sample manually

- **1** Select **Manual** as the injection source on the Inlet and Injection Parameters box.
- **2** Press the **[Prep Run]** key on the GC keypad. This cancels the gas saver flow, brings the inlet flow to its setpoint value, and closes the purge valve (for splitless injection only).
- **3** Select **Run** from the Method menu from the Top or Instrument Control view.
- **4** When the Start Run box appears, specify the sample information as described below:
 - Specify a unique data file name for the sample.
 - (optional) Fill in the **Sample Name** and **Misc Info** fields to document the injection.
 - Make sure that the **Data Acquisition** option is selected.
 - (optional) Select the **Data Analysis** option if you want to generate any Data Analysis reports specified in the method.
- **5** Click **Run Method** to initiate the run. If the temperatures are stable, the Prepare To Inject box appears. Otherwise, the message **Waiting for GC ready** is displayed.
- **6** When the GC temperatures have stabilized (the **Pre Run** light on the GC is steady), inject the sample and press **[Start]** on the GC.

CAUTION Do not inject before the GC is ready. This will cause inconsistent results.

To Analyze MS Data

To load a data file

- 1 In Data Analysis, select **Load** from the File menu.
- **2** Select a data file (double-click on a file name or type a name and click **OK**). The chromatogram for the data file is loaded and displayed in window [2].

A data file must be loaded to perform any of the following tasks.

To integrate a chromatogram

Select Integrate from the Chromatogram menu.

To select a spectrum

Double-click the *right* mouse button on the time point of interest in the chromatogram. The spectrum appears in window [1].

To zoom in

- **1** Position the pointer at one corner of the area you wish to expand in a chromatogram or spectrum.
- **2** Press and hold the *left* mouse button while dragging the mouse to select the area you wish to expand.
- **3** Release the mouse button. The selected area expands to fill the existing window.

To zoom out

- **1** Position the pointer anywhere in the zoomed window.
- **2** Double-click the *left* mouse button.

To average spectra

- **1** Position the pointer in the chromatogram at the starting time for the range you want to average.
- **2** Press the *right* mouse button while dragging the mouse to the end of the range you want to average.

3 Release the mouse button. The spectra in the selected range are averaged and the averaged spectrum is displayed in window [1].

To Use Spectral Libraries

To integrate and search peaks

Use the following procedure to integrate a total ion chromatogram (TIC) and automatically generate a library search report for each peak detected.

- **1** In Data Analysis, load a data file. The TIC is displayed.
- 2 Select Spectrum / Library Search Report.
- **3** When the Library Search Report Options dialog box appears, select the options you want for the library search report:
 - Select either Summary or Detailed to determine the report format.
 - Select one or more destinations (Screen, Printer, and File).
 - Select an **Integration Parameter File** (leave the field blank to autointegrate using the ChemStation integrator).
 - Select which spectrum from each peak to use (Apex, Apex Start of Peak, Apex Background at time, or Peak Average).
- 4 Click **OK** to initiate the search.

The chromatogram is integrated and a spectrum from each peak is searched. The results of the integration appear on the screen. The library search report is sent to the destinations selected in Step 4.

To search a single spectrum

- 1 In Data Analysis, load a data file. The TIC is displayed.
- **2** Select a spectrum. The selected spectrum appears in a window below the chromatogram.
- **3** Initiate the library search by double-clicking the right mouse button in the window containing the spectrum.
- **4** When the search is complete, the search results appear on the screen. The spectrum for the unknown, the reference spectrum you select from the list of hits, and, if available, the chemical structure of the reference compound is displayed.
- **5** To view other spectral data:
 - Click on another compound in the hit list to display a different reference spectrum.

- Select **Difference** to display the difference between the unknown and the reference spectra.
- **6** To view other information:
 - Click **Statistics** to display information about the quality of each hit found in the list.
 - Click **Text** to view the header information stored in the library for the current reference spectrum.
- 7 Click **Print** to print a copy of the displayed spectra.
- 8 Click **Done** to clear the library search results from the screen.

To Reprocess a Sequence

The MSD Security ChemStation provides a new tool for reprocessing sequences: **Reprocessing**. **Reprocessing** runs independently of the online instrument, so reprocessing a previously-run sequence can occur while collecting new data. It can also reprocess a sequence using an edited Sample Log Table, such as one containing different sample amounts. When the modified sequence is run, any revised file versions are saved under the sequence's run directory path.

If reprocessing requires any changes to the method parameters for a run, use Data Analysis instead.

Note that during reprocessing, the program creates a subdirectory named $\2$ under the results directory path. This directory contains temporary files that are deleted when reprocessing ends.



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Tips

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This chapter is a collection of assorted facts about system operation and problems.



Operating Tips

- Back up your data and methods **regularly** to avoid loss of data if the files are accidentally overwritten or deleted, or if a hardware problem develops with your disk drive.
- Make sure the tune file you are using is appropriate for your samples.
- Save Tune reports in a notebook for future reference.
- Perform system maintenance as indicated by the maintenance schedule in the GC and MSD hardware documentation. Keep a record of all maintenance performed.
- When venting the MSD, take advantage of the cool GC to do maintenance such as replacing inlet liners, septa, etc.
- After pumpdown, wait *at least 2 hours* for the MSD to reach thermal equilibrium before tuning or acquiring data.
- Optimum sensitivity generally occurs at column flow rates of 1.2 mL/minute or less.
- When injecting volumes greater than one microliter, use the pulsed splitless mode and increase the initial oven temperature 10–20°C.
- For splitless injections, pulsed splitless mode gives more quantitative sample transfer onto the column. A pulse pressure of twice the initial inlet pressure is typical.
- Selecting Constant Flow mode will provide the most efficient separation in most cases. It also results in constant sensitivity throughout the temperature program.
- For a new column, check that the column nuts are still tight after the first few oven temperature cycles.
- Use the Config Status keys on the GC keypad to set the three display items most important to you (time remaining, oven temp, etc.). These are then always visible regardless of which ChemStation view is on top.
- Rinse and refill autosampler wash vials. Do not add more solvent to a partially full vial.
- Use Table 4 as a guide to using the Selected Ion Monitoring (SIM) or Scan acquisition modes.

 Table 4
 Choosing the acquisition mode

ask	Mode
Analyze a mixture with unknown components	Scan
Analyze a mixture with known components in unknown amounts (quantitate)	Scan or SIN
Identify the presence of a few known components at low levels in a mixture	SIM

- When choosing masses for SIM, use the exact mass printed in the Tabulation report, not the nominal mass annotated on the spectrum display. This provides more accurate data.
- When doing SIM analysis, use low resolution mode unless you are trying to determine the ratios of masses one amu apart. Low resolution provides maximum sensitivity and repeatability.
- Choose the narrowest scan range that still produces good library search results. This allows more spectra across the peak and better quantitation.

Troubleshooting Tips

MSD is on, but status flashing "Server not found! Check LAN connection"

This is normal when the MSD is initially turned on. It means the ChemStation has not yet established contact with the MSD. If the flashing continues after the pumpdown is initiated:

- Temporary power failure interrupted communications
- Bad connection between the MSD and the ChemStation and/or the hub

No peaks

- Incorrect sample concentration
- No analytes present
- Syringe missing or not installed correctly (ALS only)
- Empty sample vial
- Injection in split mode instead of splitless mode

Tailing peaks

- Active sites in sample path
- Injection too large
- Injection port too cool
- Column flow too low
- GC/MSD interface or ion source too cool

Peaks with flat tops

- Solvent delay time too short
- Display scale is wrong
- Injection too large
- Electron multiplier voltage too high

Peaks with split tops

- Bad injection technique
- Injection too large

Rising baseline

- Column bleed
- Other contamination

Retention time (RT) drift

- Column has been shortened (shorter RT)
- Old column (shorter RT)
- Active sites in sample path (longer RT)
- Reduced column flow (longer RT)
- Injection port leak (longer RT)
- Initial oven temperature changed (up = shorter RT, down = longer RT)

Poor sensitivity

- Incorrect tuning
- Tune file does not match type of analysis
- Incorrect temperatures
- Incorrect sample concentration
- Leaking injection port
- Incorrect split ratio
- Purge off time in splitless mode too short
- Excessive pressure in the MSD
- Dirty ion source
- Air leak
- Detector is not working correctly
- Poor filament operation
- Incorrect mass filter polarity

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Poor repeatability

- Dirty syringe needle
- Leaking injection port
- Mismatched injection port liner and injection size
- Loose column connections
- Variations in pressure, column flow, and temperature
- Dirty ion source
- Loose connections in the analyzer
- Ground loop

Inconsistent peakwidths

- Incorrect tuning
- No PFTBA in calibration vial
- Calibration valve failure
- Dirty ion source
- Worn out electron multiplier
- MSD has not had enough time to reach thermal equilibrium
- Large variations in the temperature of the lab

High background in mass spectra

- Air leak
- Foreline or vacuum manifold pressure too high
- Other contamination

lons at m/z 18, 28, 32, and 44

- Detector vented recently (residual air and water)
- Air leak

Isotopes missing or isotope ratios incorrect

- Incorrect tuning
- Dirty ion source
- · High background
- · Electron multiplier voltage too high
- Repeller voltage too high
- High scan speed (Scan mode)
- Low dwell time (SIM mode)
- Peaks too wide or too narrow
- Repeller and ion focus leads have been reversed

Foreline or vacuum manifold pressure too high

- Excessive column flow
- Air leak
- Diffusion pump fluid level too low
- Diffusion pump fluid is contaminated
- Foreline pump oil level too low
- Foreline pump oil is contaminated
- Constricted foreline hose (this would cause the vacuum manifold pressure to be too high but the foreline pressure to be too low)

Refer to Troubleshooting the MSD in the online help for more detailed information.

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