Agilent

Agilent MassHunter GCMS Data Acquisition for the 5977 Series MSD System

Getting Started

Notices

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Safety Notices

CAUTION

A CAUTION 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 damage to the product or loss of important data. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met.

WARNING

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.

Contents

Getting Started

In This Book

This document contains an overview of the items included with your system. It is intended to help you get started using your GC/MSD System quickly.

In the following pages you will find:

- Details on where to find more information on MassHunter software and the Agilent 5977 Series MSD instrument
- Photos of your hardware with major parts identified
- Icons found in the MassHunter Data Acquisition software
- Procedures for common MassHunter operations
- A summarized maintenance schedule
- A brief section on operating tips, error messages, and troubleshooting

Where to Find More Information

Your system includes the **Agilent 5977 Series MSD**, an **Agilent 8890 or 7890B Series GC**, and the **Agilent MassHunter Workstation Software**.

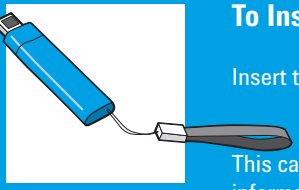
Accompanying your hardware and software is a comprehensive collection of **manuals**, **videos**, **user applications**, and **method development tools**. These are located on the:

- **Agilent GC and GC/MS Manuals and Tools** DVD set
- **Agilent GC/MS Software Information and Manuals** memory stick

To Install Your Hardware Library

Insert Disk 1 into your DVD drive and follow the prompts.

This can be installed by anyone who has authority to copy information onto the receiving computer.



To Install Your Software Library

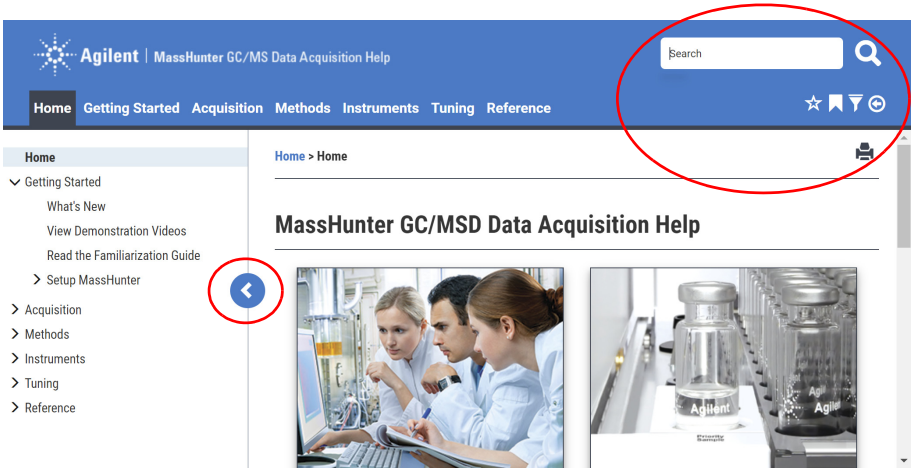
Insert the memory stick into a USB port and follow the prompts.

This can be installed by anyone who has authority to copy information onto the receiving computer.

See the **Agilent 5977 Series MSD System Quick Start** document for more details on how to install this information on your computer and a brief summary of some of the documents included with it.

Using MassHunter Online Help Files

Online Help contains extensive information on data acquisition, instrument control, data analysis, troubleshooting, and more. To access help, press **F1** to see context sensitive Help for the screen you are viewing, or click the Help icon to open the Home page of Help.

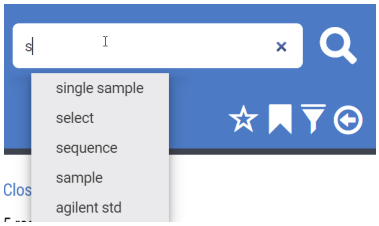


In Help, the following features are available:

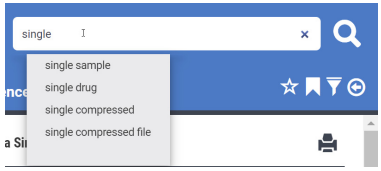
- Enter a search term, and click **Search** to search the Help content.
- Click **Set as favorite** to save the current topic to your favorites list.
- Click **Favorites** to view the topics you have set as a favorite topic.
- Click **Filter** to filter the Help content or search results based on your selection.
- Click **History** to display the Help topics you selected during the current session.
- Click **Print** to print the current topic or save it as a PDF.
- Click **Expand** or **Minimize** to show or hide the table of content.

Valuable Searching features now included predictive search, spell checking, exact match searches, frequency ranking, and PDF searching.

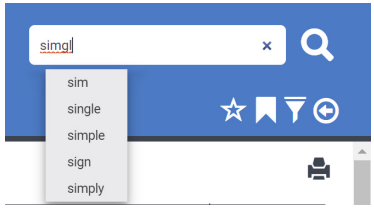
For example, when you type a single letter, a list of predictive searches is displayed instantly.



As you continue to type, the list is further refined.



Also, if you misspell a word, spelling alternative are offered.



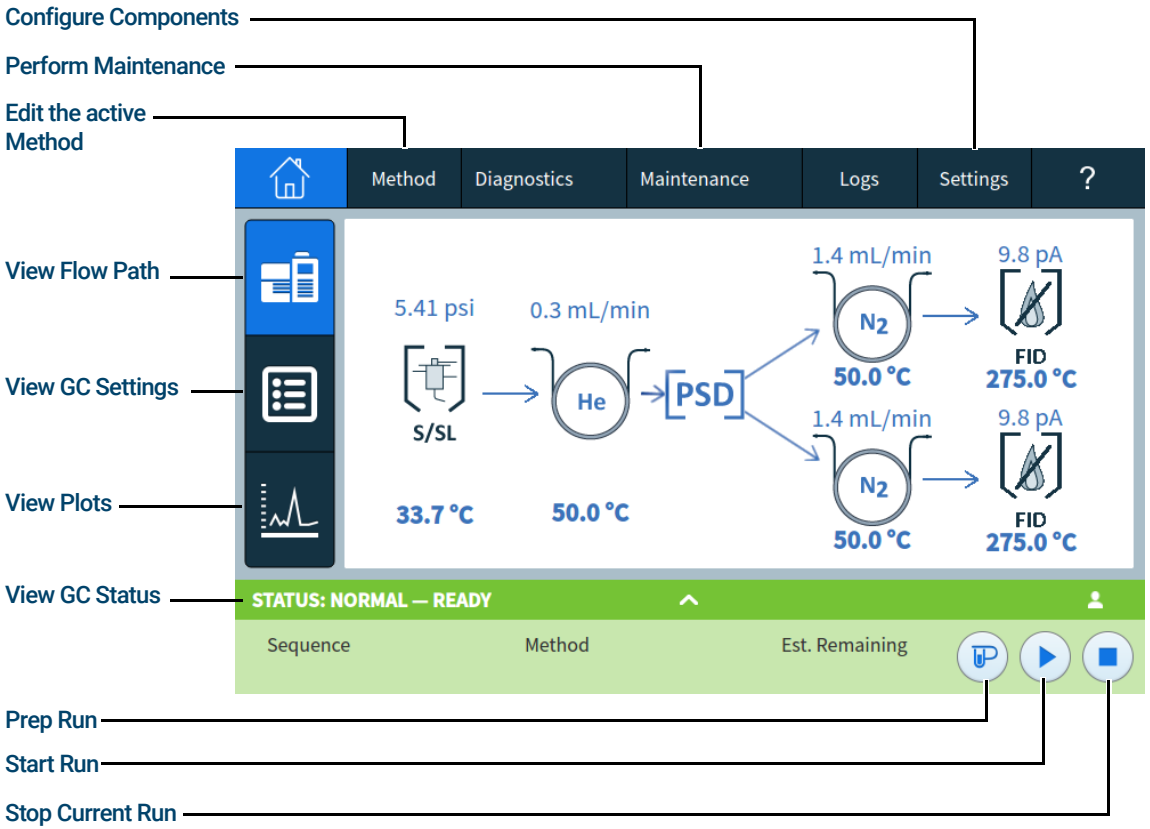
Use **quote marks** to search for an exact match. For example, **"Area Sum"** would return only those topics with Area Sum next to each other. Without the quotes, the search results would include topics with both the words Area and Sum, but they would not necessarily need to be next to each other.

Hardware

Agilent 5977 Series MSD system, shown with an Agilent 8890 GC

Touchscreen for the Agilent 8890 GC

The MassHunter Data Acquisition software provides instrument control for the Agilent 8890 GCs. This allows you to use the software, instead of the GC touchscreen, to program the instrument. However, there are times when you may want to use the touchscreen to quickly access these common functions.



Maintenance schedule

Detailed maintenance tasks are described in the hardware manuals supplied with your system. How often you need to perform system maintenance may vary for your system. Agilent recommends the following:

Every day

- ☐ Check, and if necessary, replace the septum.
- ☐ Check the tightness of the inlet liners.
- ☐ Check the tightness of the column nuts.
- ☐ Check the gas cylinders, and replace when below 500 psig.

Every week

- ☐ Check the foreline pump fluid level, if applicable.
- ☐ Change the inlet liners and O-rings.
- ☐ Check the inline gas filters, and replace when needed.

Every month

- ☐ Clean the split/splitless inlet vent line trap.
- ☐ Check for leaks (inlet, column connections).

Every six months

- ☐ Replace the foreline pump fluid, if applicable.
- ☐ Check, and if necessary, refill the calibration vial(s).

Every year

- ☐ Check, and if necessary, replace the diffusion pump fluid.
- ☐ Recondition or replace internal and external traps and chemical filters on the GC.

As needed

- ☐ Tune the MSD.
- ☐ Clean the ion source.
- ☐ Replace the carrier gas trap.
- ☐ Replace worn out parts (filaments, EM, etc.).
- ☐ Replace the column.
- ☐ Lubricate seals.

Safety warnings

WARNING

Do not perform maintenance with the MSD on or connected to its power source unless specifically instructed to by documentation supplied with the MSD.

The GC/MSD interface can be on and at a dangerously high temperature even though the MSD is off. After it is turned off, the GC/MSD interface cools very slowly. Ensure all parts have cooled before handling them.

Be careful when working behind the GC. During cool-down cycles, the GC will emit hot exhaust that could cause burns.

If you are analyzing toxic chemicals or using toxic solvents, use a hose to route the pump exhaust out of your laboratory. Note that the oil trap provided with standard foreline pumps stops foreline pump oil only, it does not trap or filter out toxic chemicals.

Use chemical-resistant gloves and safety glasses when replacing pump fluid. Avoid all contact with the fluid.

The insulation around the inlets, detectors, valve box, and insulation cups is made of refractory ceramic fibers (RCF). Avoid inhalation of RCF particles. Ventilate your work area, wear long sleeves, gloves, safety glasses, and a disposable respirator. Dispose of insulation in a sealed plastic bag. Wash your hands with soap and cold water after handling RCFs.

Operating tips

- ☐ Back up your data and methods ***regularly***.
- ☐ Ensure 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 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 two 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/min or less.
- ☐ When injecting volumes greater than 1 µL, 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.
- ☐ For a new column, check that the column nuts are still tight after the first few oven temperature cycles.
- ☐ Use the **Settings** page to display a list of parameters along with their set points and actual values (for example, **column flow** and **oven temp**).
- ☐ Rinse and refill ALS wash vials. Do not add more solvent to a partially full vial.

- Use the following table as a guide to using the SIM and/or Scan acquisition modes.

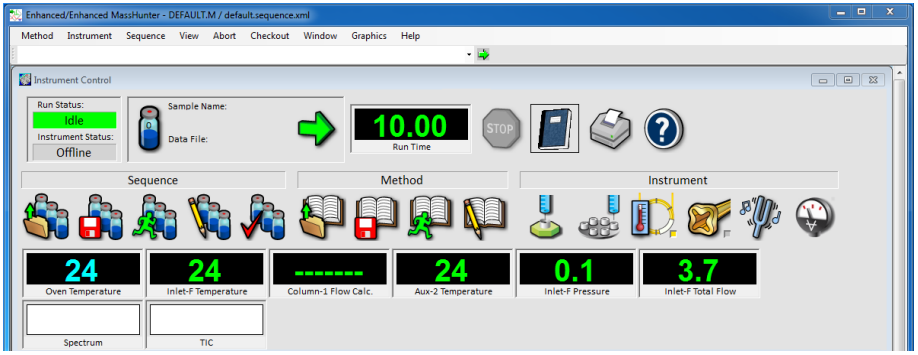
Task	Mode
Analyze a mixture with unknown components.	Scan, or SIM/Scan
Analyze a mixture with known components in unknown amounts (quantitate).	Scan, or SIM, or SIM/Scan
Identify the presence of a few known compounds at low levels within a mixture.	SIM

- 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.

Instrument Control View

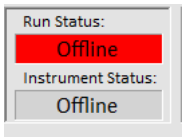
The Instrument Control view is displayed when you start up MassHunter. This is where you set and monitor instrument parameters. If you are in the Tune and Vacuum Control view, select **View > Instrument Control** when you are ready to set up the system for data acquisition.

NOTE **See the online Help for more details on the menus, buttons, or windows used in the software.**



Instrument Control view icons

Start a single sample run
Displays the sample name, vial number, and data file name. Click the green arrow to open the Start Run dialog.



Acquisition Status Indicator
Displays the status of the current run.

Run Time
When a run is in progress, this clock shows the time elapsed since the beginning of the run. Here, the clock increments. The scheduled run time is shown below the digital clock. When a run is not in progress, this clock shows the time elapsed since the last run as PostRun Time. Here, the clock decrements.

Stop
The stop sign is red when a run is in progress and gray when a run is not in progress. Use this button to stop the system when it is in PreRun, Run, or PostRun. If the system is in Run, the system will go to PostRun. If the system is in PostRun, it will go to Idle.

Logbook
Displays the logbook menu.

Print
Displays a dialog box with such printable items as sequence log, current sequence, and instrument parameters.

Help
Displays Help for the Instrument Control view, and gives access to the rest of the Help system.

	**Load Sequence** Opens the Load Sequence dialog.
	**Save Sequence** Opens the Save Sequence dialog.
	**Run Sequence** Opens the Start Sequence dialog.
	**Edit Sequence** Opens the Sequence Table dialog.
	**Simulate Sequence** Tests a sequence.
	**Load Method** Opens the Load Method dialog.
	**Save Method** Saves the current method.

Run Method
Opens the Start Run dialog.

Edit Method Information
Opens the Method Information dialog and lets you enter a method comment, select if a copy of the method is saved with the data, and specify the method sections to run.

Inlet/Injection Types
Opens the Inlet and Injection Parameters dialog. Lets you select your inlet and your injection source. From this box, you can also select whether or not you are running an MS.

ALS Parameter
Lets you edit the ALS parameters in the GC Edit Parameters dialog.

CTC Sampler
Lets you set the automatic sampler parameters for the CTC Sampler.

GC Parameters
Lets you edit the Agilent 7890A GC parameters.

MS Parameters
Lets you edit the MS parameters. The status indicator color in the lower right corner is determined by the color of the indicators in the MS Status panel.

Tune Parameters
Lets you tune the MS.
- **Tune MSD:** Performs a complete autotune
- **Quick Tune**: Adjusts peak width, mass assignment, and abundance, without changing ion ratios
- **Manual Tune**: Allows you to manually edit the Tune file

Vacuum Control
Lets you pump down or vent the analyzer.

Common MassHunter Tasks

Before you turn on the MSD

Verify the following ***before*** you turn on or attempt to operate the MSD.

- The vent valve must be closed (the knob turned all the way clockwise).
- All other vacuum seals and fittings must be in place and fastened correctly. The front side plate screw should not be tightened, unless hazardous carrier or reagent gasses 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 GC inlet, and the oven are off.
- Carrier gas of at least 99.9995% purity is plumbed to the GC with the recommended traps.
- If hydrogen is used as carrier gas, carrier gas flow must be off, and the front sideplate thumbscrew must be loosely fastened.
- The foreline pump exhaust is properly vented.

WARNING **The exhaust from the foreline pump contains solvents and the chemicals you are analyzing. If using the standard foreline pump, it also contains traces of pump oil. If you are using toxic solvents or analyzing toxic chemicals, remove the oil trap (standard pump) and install a hose (11-mm id) to take the foreline pump exhaust outside or to a fume (exhaust) hood. Comply with local regulations. The oil trap supplied with the standard pump stops only pump oil. It does not trap or filter out toxic chemicals.**

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. Read the Hydrogen Safety manual before operating the MSD with hydrogen carrier gas.**

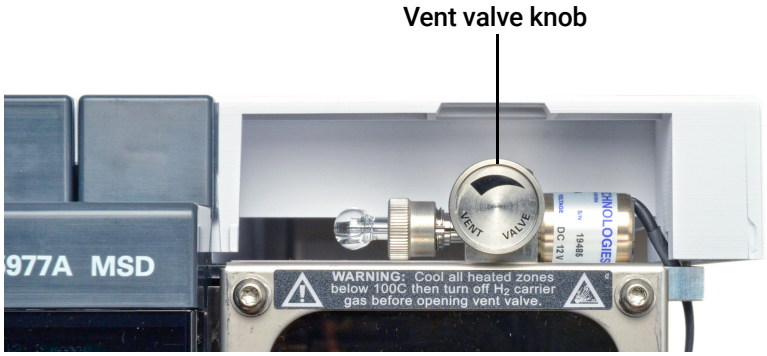
To pump down the MSD in EI mode

WARNING **Ensure your MSD meets all the conditions listed in "Before you turn on the MSD" on page 21 before starting up and pumping down the MSD. Failure to do so can result in personal injury.**

WARNING **If you are using hydrogen as a carrier gas, do not start carrier gas flow until the MSD has been pumped down. If the vacuum pumps are off, hydrogen will accumulate in the MSD and an explosion may occur. Read the Hydrogen Safety manual before operating the MSD with hydrogen carrier gas.**

1 Remove the analyzer window cover.

2 Close the vent valve by turning the knob clockwise.



3 Plug in the MSD power cord.

4 Press the **Power on** button on the front of the MSD.

5 Press lightly on the side board to ensure a correct seal. Press on the metal box on the side board.

The foreline pump will make a gurgling noise. This noise should stop within one 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.

6 Start the MassHunter Data Acquisition program.

7 In the Instrument Control view, select **Instrument > MS Vacuum Control** to display the **Vacuum Control** dialog.

8 In the Vacuum control dialog, click **Pump Down**, and follow the system prompts.

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.**

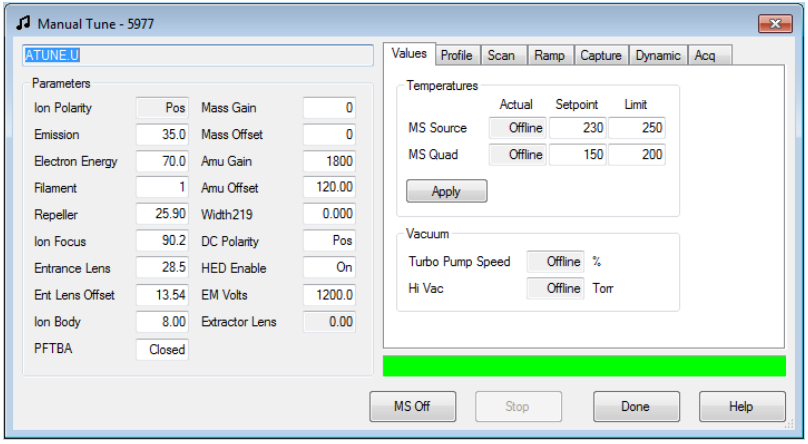
After the message **Okay to run** appears, ***wait at least two hours*** for the MSD to reach thermal equilibrium. Data acquired before the MSD has reached thermal equilibrium may not be reproducible.

To vent the MSD

1 In the Instrument Control view, select **Instrument > GC Parameters** to display the GC Edit Parameters dialog. Select **Oven**, and set the oven temperature to room temperature. Also select **Aux Heaters (MSD Transfer line)** and **Inlet**, and set those temperatures to room temperature. Click **OK** to close the dialog and send this temperature to the GC.

2 In Instrument Control view, select **Instrument > Edit Tune Parameters** to display the Manual Tune dialog.

3 Select the **Values** tab and set the MS Source and MS Quad temperatures to ambient (room temperature), and click **Apply** to download these settings to the MSD.



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 Safety manual before operating the MSD with hydrogen carrier gas.**

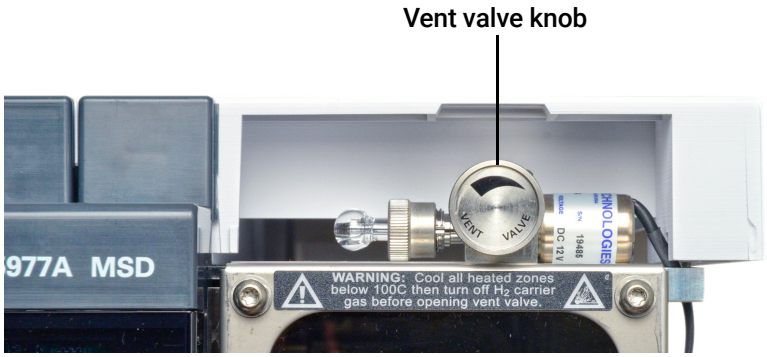
CAUTION **Ensure the GC oven and the GC/MSD interface are cool before turning off carrier gas flow to prevent damage to the column.**

4 In the Instrument Control view, select **Instrument > MS Vacuum Control** to display the Vacuum Control dialog.

5 Remove the analyzer window cover to gain access to the vent valve.

6 Click **Vent** to begin the automated shutdown of the MSD. Follow the instructions presented.

7 When prompted, turn the vent valve knob counterclockwise ***only*** 3/4 turns or until you hear the hissing sound of air flowing into the analyzer chamber.



Do ***not*** turn the knob too far or the O-ring may fall out of its groove. Close the vent valve before pumping down.

To tune the MSD in EI mode

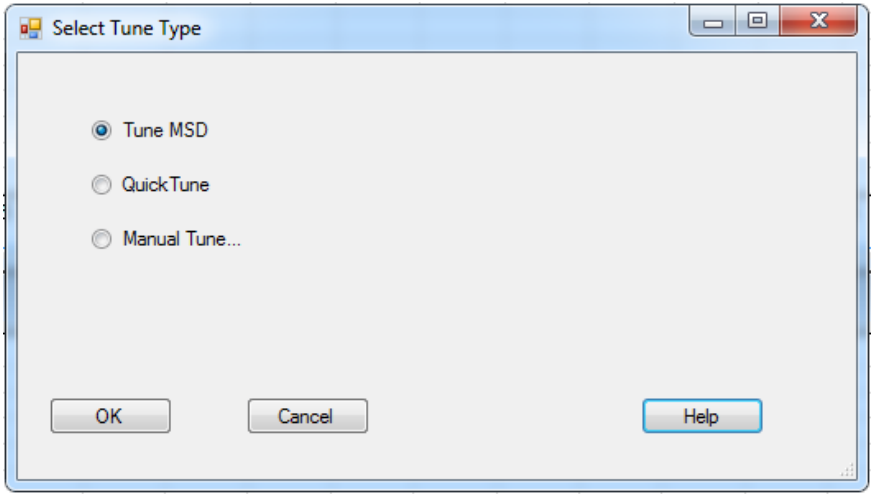
1 Load the method that will be used for data acquisition.

2 In the Instrument Control view, click the **Instrument MS Parameters** icon to display the **Quadrupole Method Editor** dialog and, in the Tune File area, verify the correct Tune file is loaded. For most applications, **ATUNE.U** (Autotune) gives good results. **STUNE.U** (Standard Tune) is not recommended, as it may reduce sensitivity.

3 To select a different tune file, press the **Browse** button to display the **Select Tune File** dialog. The **Settings** area displays the important parameters for a selected tune file.

The tune file must match the type of ion source in the analyzer. If you are using an EI ion source, select a tune file created for an EI ion source.

4 Click the **MS Tune** icon to display the **Select Tune Type** dialog.



5 Select **Tune MSD** to perform a complete autotune, or select **Quick Tune** to adjust peak width, mass assignment, and abundance, without changing ion ratios. Alternatively, select **Manual Tune** to display the Manual Tune dialog and graphs for doing manual edits to the Tune file.

6 Click **OK** to close this dialog and start the tune. If the MSD temperatures are not stable, you are prompted to wait, or override the wait by clicking **Override**.

7 Wait for the tune to complete and to generate the report.

8 To evaluate the tune results, select **Evaluate Tune** from the **Checkout** menu.

To view the history of tune results, in the Instrument Control view select **Checkout > View Previous Tunes...**.

To manually tune your MSD or to perform special autotunes, select **View >Tune and Vacuum Control**. See the manuals and online Help provided with your MassHunter Data Acquisition software for additional information about tuning.

To configure the system for GC and MS data

1 Set inlet and injection parameters, and verify that the **Use MS** check box is selected.

2 Select **Instrument > GC Parameters** to display the GC Edit Parameters dialog.

3 From the **Configuration** tab, select the **Miscellaneous** tab.

4 Verify that **Thermal Aux Type** is set to **MS Transfer Line**.

5 Click the **Columns** tab, and verify that the column attached to the MS shows **Vacuum** selected for the **Outlet**.

6 If you made any changes, click **OK**, otherwise click **Cancel** to keep the method unchanged.

To perform a single sample run

1 Select **Method > Load Method**, or click the **Load Method** icon to display the Browse for Folder dialog.

2 Select the method file for this acquisition run, and click **OK**.

3 Select **Method > Run Method** or click the large green arrow on the Instrument Control panel to display the Start Run dialog.

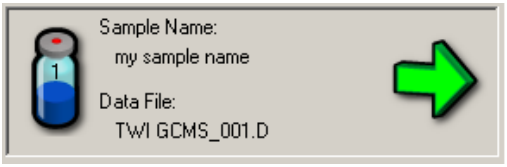
4 In the Start Run dialog enter a **Data File Name**. Enter the **Sample Name** and **Misc. Info.**, which is optional information to be stored with your data file. Enter a **Sample Amount**, **Multiplier** and the **Vial Number** location where the sample is stored in the ALS tray.

   When using the Agilent 7693 ALS with a G4515A BCR tray installed, you must enter a value in the **Expected Barcode** field. If you are not entering a barcode value, enter the noncase sensitive string **Off** to disable reading of a nonexistent barcode.

5 In the Method Sections to Run box, select **Data Acquisition**.

6 Click the **OK and Run Method**.

The vial number, sample name, and data file name are displayed in the Sample Run box, and the run time is displayed above the total run time in the Run Time box. The Status box at the top left corner shows the system in Pre-Run status, which changes to Run status after the sample is injected. The Instrument Control panel icons are grayed out and all instrument controls except the Stop Run icon are disabled until the run is over.



Perform multiple automated runs

This procedure uses a previously created sequence table.

1 From the Instrument Control panel, select **Sequence > Load Sequence** to display the Load Sequence dialog.

2 Select the sequence file, and click **Open**. If needed, browse the Look In field for the directory containing the saved sequence.

3 Click **Sequence > Run Sequence** or click the **Run Sequence** icon to display the Start Sequence dialog.

4 Enter a comment in the Sequence Comment field.

5 Click **Run Sequence** to start the automated sequence.

A yellow bar appears above the Instrument Control panel displaying the status of the sequence, with buttons that allow you to edit or pause the sequence. If you edit or pause the sequence during a sample run, the current sample run is completed but additional samples cannot be processed until the sequence table is closed or the paused sequence is resumed.

To inject a sample manually

In the Instrument Control view:

1. From the Instrument menu, select **Instrument > Inlet\Injector Types**.
2. In the Inlet and Injection Parameters dialog, select **Manual** as the injection source and click **OK** to continue.
3. On the GC touchscreen, touch  **(Prep Run)**.This cancels the gas saver flow, brings the inlet flow to its setpoint value, and closes the purge valve (for splitless injection only).
4. Select **Method > Run Method**.
5. When the Start Run dialog appears, specify the sample information as described below:
   - ☐ Specify a unnique data path for the sample.
   - ☐ Specify a unique data file name for the sample.
   - ☐ (Optional) Fill in the **Operator Name**, **Sample Name**, and **Misc Info** fields to document the injection.
   - ☐ Ensure 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.
6. Click **Run Method** to initiate the run. If the temperatures are stable, the Prepare to Inject dialog appears. Otherwise, the message Waiting for GC ready is displayed.
7. When the GC temperatures have stabilized, (the status in the bottom part of the touchscreen will read READY) inject the sample and touch  **(Start)** on the GC touchscreen.

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

To edit the entire method

Do not use this procedure when configured for Classic (ChemStation) Drug Analysis, instead use Edit Entire Method (Drug Analysis) as described in online Help.

1 Select **Method > Load Method**.

2 Select the method file that you wish to use for this acquisition and click **OK**.

When you load a method on a system configured with an MS, the software compares the actual source/quad temperatures to the setpoints stored in the method tune file. If they are not within a three-degree tolerance, you are asked if you want to download the new setpoints.

3 Select **Method > Edit Entire Method**, or click the **Edit Entire Method** icon. The Edit Method dialog is displayed.

4 Select the **Information** checkbox to edit the Method Information dialog.

5 Select **Instrument/Acquisition** checkbox to edit these dialogs:

- Inlet and Injection Parameters
- GC Edit Parameters
- Real Time Plots for GC
- MS or MSD Method Editor
- Select Monitors

6 Click **OK**. The dialogs displayed depend on the options selected in the previous steps.

7 If the Method Information dialog is displayed, optionally enter a method comment and select if a copy of the method is saved with the data. Then, specify the method sections to run. If you select the Data Analysis method section, you activate the Automated Data Analysis feature. When finished with the edits, click **OK** to display the next dialog.

8 If the Inlet and Injection Parameters dialog is displayed, set the inlet and injection parameters. When finished with the edits, click **OK** to display the next dialog.

9 If the Edit GC Parameters dialog is displayed, edit the GC parameters. Optionally, to display real-time plots of GC data, click the **Signals** icon and configure up to four GC signals to display. When finished with the edits, click **OK** to display the next dialog.

10 If the Real Time Plots for GC dialog is displayed, select the **GC signals** to display based on the signal configurations made in the previous step. When finished with the edits, click **OK** to display the next dialog.

11 If the MS Method Editor dialog is displayed, set the scanning parameters for the MS. If the Single Quadrupole MS Method Editor dialog is displayed, set the scanning parameters for the MSD.

   a Optionally, enter the required values for specifying the real-time plot of MS data in the Method Editor Real-Time Plot Display tab.

   b Optionally, enter the values for Timed Events in the Method Editor Timed Events tab.

12 When finished with the edits, click **OK** to close the Method Editor dialog.

13 If the Select Monitors dialog is displayed, Move the desired monitors from the Available Monitors column to the Selected Monitors column, and click **OK** when finished.

14 Save the method.

To set up a sequence

The sequence table is used to set up a sequence. Each line in the sequence table contains information for the analysis of one sample (one vial for an ALS).

1 Select **Sequence > Edit Sequence** or click the **Edit Sequence** icon in the tool bar to display the sequence table.

2 To use a previously saved sequence table column layout, click **Tools**, and select **Load Column Layout** from the drop-down list to display the Open dialog.

   Select the file that contains the correct layout and click **Open**.

3 To configure which columns are displayed in the sequence table, click **Tools** in the table toolbar, and select **Add/Remove Columns**.

4 If you want to add more samples than the current table can hold, with a line in the table selected, click the 

down arrow and select **1 sample** to insert one new sample or **5 samples** to insert five new samples at the bottom of the table. Select **N Samples** to specify the number of samples to add to the table. Select **Insert** to add a new row above the currently selected line.

5 Edit the sequence table by entering the required values for all parameters that apply to each sample.

When using the Agilent 7693 ALS with a G4515A BCR tray installed, you must enter a value in the Expected Barcode column. If you are not entering a barcode value, enter the noncase sensitive string **Off** to disable reading of a nonexistent barcode.

To use Sample Overlap on an ALS and tray, the sequence must use a single method defining Sample Overlap for all samples.

6 To increment the value in a selected cell to all subsequent cells, select a cell in the table, and click  .

7 When you are finished, click **OK** to close the sequence table.

8 Save the sequence.

To use retention time locking

Overview

Retention time locking (RTL) involves the collection of data for a compound whose desired retention time is known at various inlet pressures around the current method setpoint (-20%, -10%, nominal, +10%, +20%). The five resultant runs are then evaluated, and a pressure/retention time curve is generated to characterize that particular instrument. From the curve, a predicted pressure which causes the lock compound to elute at the desired time can be calculated and stored so that the method will run at that pressure.

Lock a method

This panel permits you to specify the pressures for RTL calibration runs and the ALS vial. The default parameters will be the same as the parameters currently used by the menu item. You can change the parameters to ± 20% of the current method setpoint.

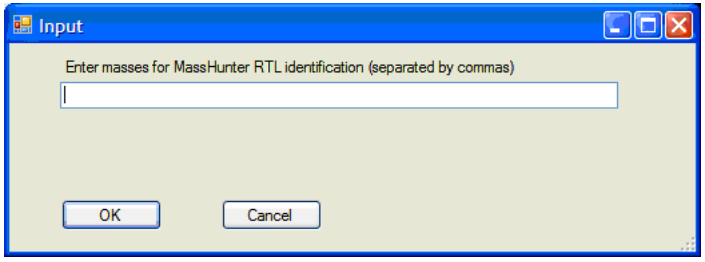
1 Load the method you would like to lock. Edit the Method if necessary.

2 Click **Method>Acquire RT Lock Calibration Data**. A dialog displays the message “This action will collect 5 data files with varying pressures. Continue?”

3 Click **Yes**.

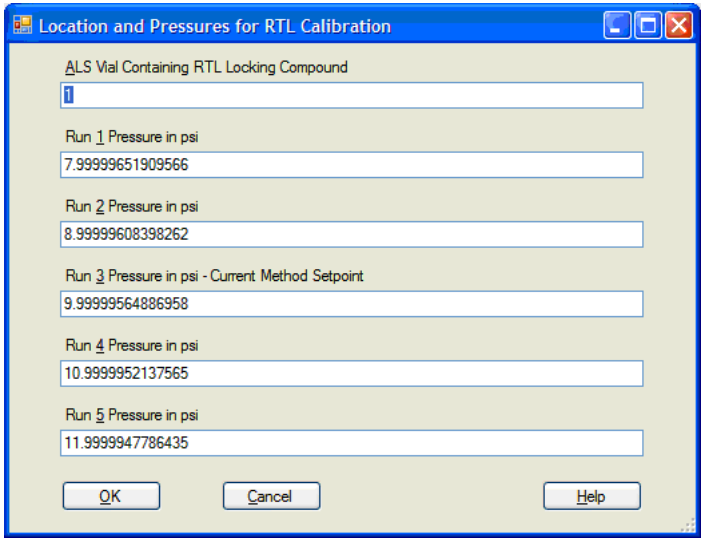
If the loaded method is already locked, an alert dialog will appear warning that all locked data will be deleted. Click **Yes** to continue and delete the existing calibration, or **No** to abort the recalibration.

4 Click **OK**. You will be asked to Enter masses for MassHunter RTL identification.



5 Enter mass values, separated by commas if there are more than one. Click **OK**.

This presents the dialog box where the pressure and the ALS vial can be set for the five RTL runs. Enter the vial position for your ALS sample.



6 Click **OK**.

A dialog box displays the message “Sample MUST be in position vial_position”. Where vial_position is the vial position entered in the previous step.

7 Click **OK** to initiate the run.

8 Acquisition starts with a cleanout run (cleanout.d) followed by five more runs (rtlock1 through rtlock5) starting with rtlock1.d. The message box that appears displays the RTL Cal times. Click **OK**.

9 Click **Yes** to Retain lock cal data files.

10 Accept or modify the data, and click **OK** to Enter the Locking RT for subsequent data files.

11 Click **OK** to Save the lock pressure to the method.

The locking process is complete when RTL Calibration Complete displays on the status line.

12 Save the method.

Unlock a method

1 Load the method you want to unlock.

2 From the Instrument Control panel, select **Method > Unlock Method**.

3 When the message box appears, click **Yes**. This will remove the lock flag from the method, allowing the user to perform manual column flow adjustments.

4 Save the method under a different name.

Relock method

1 Load the method you want to relock.

2 From the Instrument Control panel, select **Method > Relock Method**.

3 When prompted by the system, enter mass values, separated by commas if there are more than one. Click **OK**. This presents the dialog box where the pressure and the ALS vial can be set for the five RTL runs. Enter the vial position for your ALS sample.

4 Click **OK**. The message “Sample MUST be in position 1” displays.

5 Click **OK** to initiate the run.

6 Verify that acquisition starts, and that five more runs are made starting with rtlock1.d. Verify that the pressure for each individual run is almost constant. The message box that appears displays the RTL Cal times. Click **OK**.

7 The message displays the LSTSQFIT coefficients. Click **OK**.

8 Click **Yes** to Retain lock cal data files.

9 Click **OK** to Enter the Locking RT for subsequent data files.

10 Click **OK** to Save lock pressure to method.

11 Save the method.

Error Messages and Troubleshooting

Error messages

Sometimes, a problem in your MSD will cause an error message to appear in the MassHunter software. Some error messages appear only during tuning. Other messages will appear during tuning or instrument control.

Sometimes, instead of a message only a number will appear. This number can represent one or more error messages.

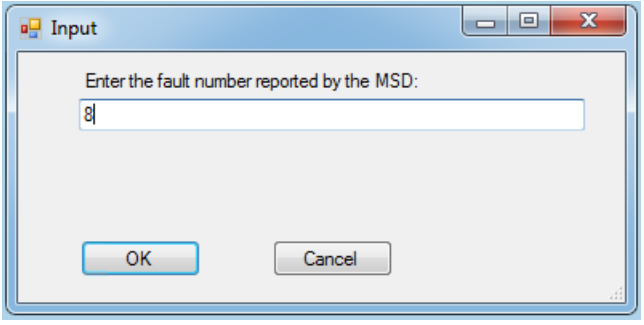
To translate a number into an error message:

1 Note the number.

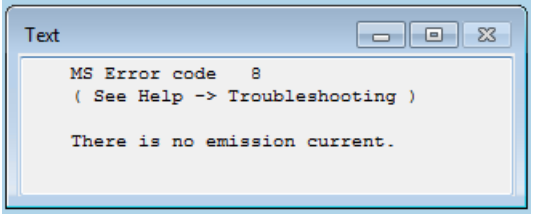
2 In Instrument Control, select **View > Tune and Vacuum Control**.

3 Select **Status > MS Error Codes**.

4 Type the error number in the box provided and click **OK**.



The corresponding error message(s) will be displayed.



Troubleshooting tips

MSD LAN error

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 MassHunter Data Acquisition software has not yet established contact with the MSD. If the flashing continues after the pumpdown is initiated:

- Temporary power failure interrupted communications.
- There is a bad connection between the MSD and MassHunter Data Acquisition and/or the Agilent Bootp service and/or the switch/hub.
- MAC and IP addresses for the MSD are not properly configured in the Agilent Bootp service for the LAN.

Baseline rising

- Column bleed
- Other contamination

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)

High background in mass spectra

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

Ions at *m/z* 18, 28, 31, 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
- Entrance lens and ion focus leads have been reversed
- Entrance lens, or focus, or extractor lens have been crossed

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

Peaks tailing

- Active sites in sample path
- Injection too large
- Inlet 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

Peak widths inconsistent

- Incorrect tuning
- No PFTBA and PFDTD 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

Poor repeatability

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

Poor sensitivity

- Incorrect tuning
- Tune file does not match type of analysis
- Incorrect temperatures
- Incorrect sample concentration
- Leaking inlet
- 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

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)
- Inlet leak (longer RT)
- Initial oven temperature changed (up = shorter RT, down = longer RT)

Refer to the Troubleshooting the MSD section of the online Help for more detailed information.

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