

Agilent G1701EA MSD Productivity ChemStation

Familiarization Guide



Agilent Technologies

Notices

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In This Guide

This guide contains a step-by-step exercise to help you become familiar with your Agilent 7890A GC/5975 MSD with G1701EA MSD Productivity ChemStation software.

To successfully use this book, you need the following:

- GC Inlet: Split/Splitless Inlet with EPC (default inlet configuration)
- Column: HP-5ms 30 m x 250 μm x 0.25 μm
- Sample: 5975 MSD Sample (P/N 05970-60045) or (P/N 5074-3025 Japan only)
- MSD Tuning PFTBA (perfluorotributylamine) Calibrator:

Before operating your instruments, be sure to read all safety and regulatory information included with your instruments.

1 Start Up the System

Start up your system hardware and software for data acquisition.

2 Tune the MS

Determine whether the instrument is correctly tuned.

3 Create a Method for Qualitative Analysis

Create a new qualitative analysis scan method from the system default method.

4 Run the Scan Method

Run the method created in Chapter 3 to acquire sample data.

5 Qualitative Data Analysis

Use the Enhanced Data Analysis program to analyze the data generated in Chapter 4.

6 Create a SIM Quantitation Method

Create a SIM method from the scan method created in Chapter 3.

7 Run a Sequence

Create and run a sequence using the method created in Chapter 6.

8 Set Up a Quantitation Database

Set up a database with compounds and calibrators to identify unknown samples.

9 Generate a Report

Generate a report automatically after a run or at a later point from previously acquired data.

10 Recalibrate and Quantitate Unknowns

Modify a sequence for recalibration and then use it to quantitate an unknown sample.

11 Create a Cool Down Method

Create and store a maintenance method.

- 12 Shut Down the System
- **13 Frequently Asked Questions**

Where to Find Information

Hardware

In addition to this document, Agilent provides several learning products that document how to install, operate, maintain, and troubleshoot the 7890A GC/5975 MSD. This information can be found on the Agilent Technologies GC and GC/MS Hardware User Information and Utilities DVDs that ship with your instrument.



The Agilent Technologies GC and GC/MS Hardware User Information and Utilities DVDs that ship with your instrument provides an extensive collection of online help, videos, and books for current Agilent gas chromatographs, mass selective detectors, ion traps, and GC samplers. Included are localized versions of the information you need most, such as:

- Getting Familiar documentation
- Safety and Regulatory guides
- Site Preparation checklists
- Installation information
- Operating guides
- Maintenance information
- Troubleshooting details

Software

For an introduction to, and where to find more information on, the G1701EA MSD Productivity ChemStation see the *Agilent* G1701EA GC/MSD ChemStation Getting Started manual.

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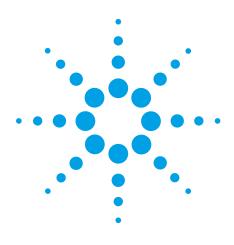
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Start Up the System

1

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In this chapter, the startup checklist is reviewed for instrument readiness. If necessary, changes are made to the instrument hardware configuration to handle the data acquisition of the samples that are run in this manual. With the instruments off and the G1701EA MSD Productivity ChemStation not running, the instruments are started and the MSD is pumped down. Finally, a method is loaded in preparation for bringing all instrument parameters to settings required for data acquisition.



Start up the Hardware

- 1 Review the Agilent 7890A Gas Chromatograph Operating Guide (P/N G3430-90011) and the Agilent 5975 Series MSD Operation Manual (P/N G3170-90036) for important safety information and start up details before powering on your instruments.
- 2 Verify that the split/splitless (S/SL) inlet septum, liner, and O-ring are clean, properly installed, and in good condition.
- Install a conditioned (HP-5ms 30 m x 250 μm x 0.25 μm) column in the GC. Attach the column inlet to the S/SL inlet and its outlet to the MSD transfer line. See the Agilent 5975 Series MSD Operation Manual for details.
- **4** Verify the EI ion source is installed.
- 5 Verify 99.9995% purity helium is attached to the carrier gas supply of the S/SL inlet.
- 6 Power on the 7890A GC.
- 7 From the GC keypad, turn off the oven, Aux 2 heated zone (GC/MSD transfer line), and inlet heater. If equipped, turn off any GC detectors.
- 8 Before you turn on or attempt to operate the MSD verify the following:
 - 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.
 - 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.
 - The foreline pump exhaust is properly vented.
- **9** Open the MSD analyzer top cover.
- **10** Close the MSD vent valve.
- **11** Press the **Power** button on the front of the MSD to power it on. The foreline pump will make a gurgling noise.

Press lightly on the metal box mounted on the MSD side board until the air noise stops to ensure a correct seal.

- 12 Close the MSD analyzer top cover.
- **13** On the MSD local control panel:
 - a Press Menu repeatedly until Maintenance appears.
 - **b** Press **Item** repeatedly until **Pumpdown** appears.
 - c Press Yes/Select to start the pumpdown.

The pumpdown is completely automatic and does not require operator actions.

After the turbo pump starts and the ion gauge value reaches 100 mTorr, allow the MSD to operate for a minimum of 2 hours before acquiring sample data.

Run the ChemStation Software

The GC and MSD must both be running before starting an online session of the ChemStation product. If reports are to be printed, a printer must be installed on the computer.

- **1** Power on the PC.
- 2 From the PC desktop, select the ChemStation Instrument Control shortcut icon, to display the Enhanced ChemStation Instrument Control window.



3 If the Actual MS temperatures have not reached their Setpoints, the **MS Temperatures** dialog box will appear. Enter new setpoints if needed and click **OK**. The screen will appear repeatedly until the temperatures are reached.

MS Temperatures			×
Zone	Actual	Setpoint	Limit
MS Source MS Quad		230 150	250 200
Apply Of		Cancel	Help

4 Set the default printer to PDF Printer, if a PDF writer like Adobe Acrobat is installed on the computer.

Select the Tune File

- From the Enhanced ChemStation main control window, select View > Tune and Vacuum Control.... to display the Tune and Vacuum Control window.
- 2 Select File > Load Tune Parameters. The Select Tune File dialog box opens.

Select Tune File		— ×
Path: C:\MSDCHEM\2\5975\ Date Last Modified: Wed Apr 06 01:10:54 2011 Files: air.u atune.u bfb.u dftpp.u NCICH4.u pcich4.u stune.u target.u	Settings: Type: EMV: Source: Quad: Emission: EleEnergy: CI Gas:	
OK Cancel	Help	

- **3** From the **Files** list, select **atune.u**. The **atune.u** file contains the optimal MSD parameter settings determined during the last autotune run.
- 4 Select **OK**. The **atune.u** tune file is loaded and the dialog box closes.

Load the Method

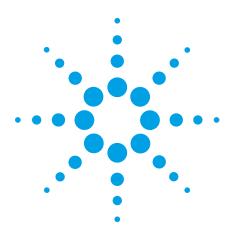
- 1 Select View > Instrument Control to close the Tune and Vacuum Control.... and display the Enhanced ChemStation Instrument Control window.
- 2 Select the **Load Method** button, dialog box opens.



3 Navigate to and select **default.m** in the msdchem/1/methods directory.

Load Method	—
Load Method	
4 🍌 msdchem	*
a 🌗 1	
Þ 퉲 5975	
⊳ 퉬 5975.OLD	
🛛 🐌 퉲 data	
4 퉬 methods	
🛛 🕒 checkout	
🛛 🕒 default	
🧧 퉲 default.m	. .
Make New Folder	OK Cancel

4 Select OK.



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Tune the MS

2

Introduction 18 Run Autotune 19 Evaluate the Autotune Results 22 Tune History Trends 24

This chapter provides a brief introduction to tuning and explains how to run an autotune on the instrument. An autotune report is generated as well as a report to evaluate the autotune results. This report is reviewed to see which items pass or fail the evaluation. Finally, we look at how we can graphically view the variation in tuned parameters that are plotted over a number of recent autotune runs.



Introduction

Tuning is the process that adjusts the MS for good performance over the entire mass range. Using a known compound as a calibrator, the tune parameters are set to achieve sensitivity, resolution, and mass assignments for the known calibration ions.

Tuning is performed using either the autotune or manual tune features.

Manual tune allows you to adjust an MS tune parameter while viewing the results easily in profile scans and spectra.

Manual tuning is used:

- To achieve maximum sensitivity by sacrificing some resolution
- To tune specifically for the very low end of the mass range (< 150 amu)
- To tune with a compound other than the standard calibrator

To access manual tune parameters select **Parameters > Manual Tune** from the **Tune and Vacuum Control** window or select **Instrument > Edit MS Tune Parameters** from the **Instrument Control** window. Please see the ChemStation online help for details on using manual tune.

The autotune program described in this section adjusts the MS for good performance over the entire mass range and is recommended for most applications.

Run Autotune

1 From the Instrument Control window select Instrument > Tune MSD... to display the Select Tune Type dialog box.

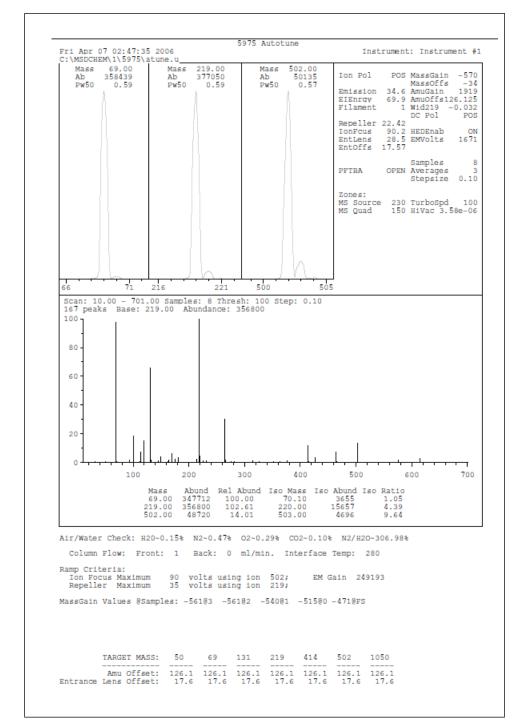
Select Tune Type		
 Tune MSD QuickTune 		
ОК	Cancel	Help

2 Select **Tune MSD** and click **OK** to close the dialog box and start the autotune procedure.

The system uses the PFTBA (perfluorotributylamine) calibrator to tune the instrument. When the tune is complete, the mass 69, 219, and 502 profile scans are displayed with abundance and peak widths noted. See Figure 1. The tune report is also generated as shown in Figure 2 on page 21.

	- EI mode - atune.u							
e <u>l</u> une V		<u>Parameters</u> <u>State</u>	is <u>V</u> iew <u>A</u> bort					
Ab	Mass 69.10 293685 Pw50) 0.68	Ab	Mass 219.00 382822 Pw50 0).74	Ab N	lass 502.20 44418 Pw50 0.69)
	ĥ			<u>A</u>			A	
				L L				
			<u>\</u>	$\sum n$				
66	68 70	72 74	214 216	218 220	222 224	498 500	502 504	506
eller mas	s = 219.00							

Figure 1 Profile scan results for mass 69, 219, and 502





Evaluate the Autotune Results

- **1** Select View > Instrument Control.
- 2 Select **Checkout > Evaluate Tune**. The system compares your tune parameter results to preset acceptable results and displays the System Verification report. See Figure 3.
- 3 Review the report. Criteria marked as OK are functioning correctly. If all criteria are marked OK, Tune portion of System Verification passed is printed on the last line of the report. See Figure 3.

If one or more criteria do not pass verification, the incorrect behavior and suggested corrective actions are described. See Figure 4 on page 23 where the report shows a high ratio of mass 18 to 69. This report warns of a high amount of water in the system and a corrective action to be taken.

	: Instrument #1 : Positive		
'ilament	: 1		
BasePeak should be 69 or 219	• +		Ok
Position of mass 69		69.00	
Position of mass 219		219.00	
Position of mass 502		502.00	Ok
Position of isotope mass 70		70.01	Ok
Position of isotope mass 220		220.00	Ok
Position of isotope mass 503		503.04	Ok
Ratio of mass 70 to mass 69(0.	.5 - 1.6%)	1.09	Ok
Ratio of mass 220 to mass 219			
Ratio of mass 503 to mass 502	(7.9 - 12.3%)	9.73	Ok
Aatio of 219 to 69 should be :		103.07	
Ratio of 502 to 69 should be :	> 2.4% and is	13.58	Ok
Mass 69 Precursor (<= 3%)		0.10	Ok
Mass 219 Precursor (<= 6%)		0.21	
Mass 502 Precursor (<= 12%)		0.26	Ok
Testing for a leak in the	system		
Ratio of 18 to 69 (<20%)		0.22	
Ratio of 28 to 69 (<10%)		0.43	Ok
Clectron Multiplier Voltage		1671	Ok
Tune portion of System Ver	rification passed.		

Figure 3 Passing system verification tune report

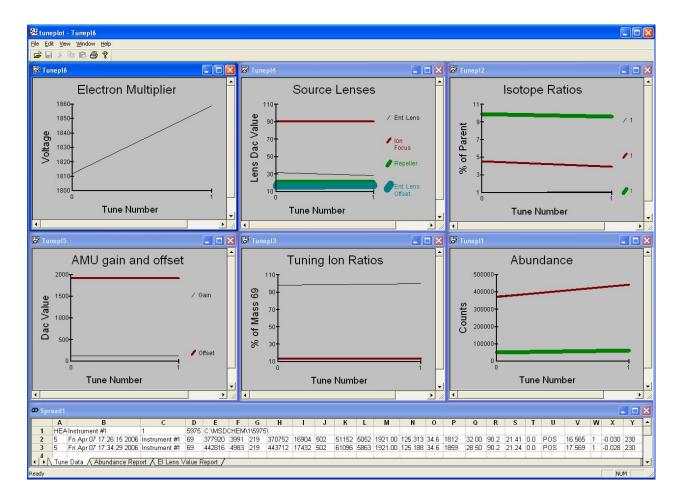
System Verification	n - Tune (Detector	r Optimization) Portion
Instrument Name DC Polarity Filament	: Instrument #1 : Positive		
Filament BasePeak should be 69 or 219 Position of mass 69 Position of mass 219 Position of mass 502 Position of isotope mass 70 Position of isotope mass 220 Position of isotope mass 503 Ratio of mass 70 to mass 69(0.1 Ratio of mass 220 to mass 219(1 Ratio of mass 503 to mass 502(7 Ratio of 219 to 69 should be > Ratio of 502 to 69 should be >	3.2 - 5.4%) 7.9 - 12.3%) 40% and is	502.95 1.34 4.33	Ok Ok Ok Ok Ok
Mass 69 Precursor (<= 3%) Mass 219 Precursor (<= 6%) Mass 502 Precursor (<= 12%)		0.42 0.26 0.45	Ok Ok Ok
Testing for a leak in the s Ratio of 18 to 69 (<20%) Ratio of 28 to 69 (<10%) There is a high amount of w Wait 24 hours for the syste	water in your syst		Ok
Electron Multiplier Voltage		1671	Ok
One or more specifications Please correct before cont:			
Failure of one or more test selecting the wrong DC Pola Please verify that the corr by removing the detector co at the top of the EID.	arity. rect DC Polarity H	has been set	

Figure 4 Failing system verification tune report

2 Tune the MS

Tune History Trends

- **1** Select View > Instrument Control.
- 2 Select **Checkout > View Previous Tunes**... to display the **Tuneplot** window plotting the results of recent tune parameters.





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Create a Method for Qualitative Analysis

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This chapter describes how to create an acquisition method that will be used later to identify all compounds in an Agilent standard sample. The method is created by editing the default method to include an MS scan that is set to identify all ions created by EI of each compound.



Introduction

The method we are creating will be used to find the known compounds in the Agilent sample P/N 05970-60045 (P/N 5074-3025 Japan only). The sample compounds are in isooctane solvent in 1 mL ampules of 10 ng/ μ L, 100 ng/ μ L, and 100 pg/ μ L concentrations and are shown in Table 1.

Compound	MW	Formula
Dodecane	170	C ₁₂ H ₂₆
Biphenyl	154	C ₁₂ H ₁₀
4-Chlorobiphenyl (PN 05970-60045 only)	188	C ₁₂ H ₉ CI
Methyl palmitate	270	C ₁₇ H ₃₄ O ₂

Table 1Sample Compound list

The MS part of the method is required to scan for all ions contained in the range that includes all the molecular weights for these compounds. As seen in the table, the range of the molecular ions is from 0 to 270 so we will scan for ions from 0 to 300 in the method.

Edit the Entire Method

1 With the default method loaded, see "Load the Method" on



page 16, select the **Edit Entire Method**... button, to edit the currently loaded method. The **Edit Method** dialog box opens.

Edit Method
Method Sections to Edit:
I ✓ Method Information
Instrument / Acquisition
🗖 Data Analysis
OK Cancel Help

2 Mark the **Method Information** and **Instrument/Acquisition** checkboxes only. Clear the **Data Analysis** checkbox.

Selecting **Instrument/Acquisition** displays all the dialog boxes required to edit the acquisition parameters for both the GC and MS parts of the currently loaded method. We are not modifying the **Data Analysis** part of the method at this time.

3 Select **OK** to close the **Edit Method** dialog box. Because **Method Information** was selected, the **Method Information** dialog box opens.

Method Information	×
Method Comments:	
Scan method for 5975 MSD performance sample	
✓ Save Copy of Method with Data	
Method Sections to Run	
Pre-Run Macros/Commands:	
Instrument Control:	
Data Analysis:	
Data Acquisition	
🗖 Data Analysis	
Post-Run Macros/Commands:	

- 4 In the **Method Comments** field, enter a description of this method.
- **5** Mark the **Save Copy of Method With Data** checkbox. When the ChemStation acquires sample data using this method, it automatically saves a copy of the method along with the data.
- 6 In the **Method Sections To Run** area, mark the **Data Acquisition** checkbox only. The data analysis will not be run at this time.
- 7 Select **OK** to close the **Method Information** dialog box and display the **Inlet and Injection Parameters** dialog box.

Inlet and Injection I	Parameters			×
	Sample Inlet Injection Source	GC GC ALS	•	
-Inlet Location	• Front	C Rear	C Dual	
MS Connected to	• Front Inle	t	O Rear Inlet	
ОК		Cancel	Help	

- 8 From the Sample Inlet dropdown list, select GC.
- 9 From the **Injection Source** dropdown list, select your source.
 - If you are injecting from the GC using the Automatic Liquid Sampler (**ALS**), select **GC ALS**.
 - If you are manually injecting or using another injection source, select **Manual**.
- 10 Mark the **Use MS** checkbox to allow the ChemStation to turn on the MS analyzer and save the MS sample data acquired during the run. You would only uncheck this box when you have a GC (non-MS) detector and you were acquiring data for the GC detector only.
- 11 In the **Inlet Location** area, select the location where your S/SL inlet is attached to the MS through the column.
- 12 In the **MS Connected to** area, select the location where your S/SL inlet is attached to the MS through the column.
- **13** Select **OK** to close the **Inlet and Injection Parameters** dialog box and display the **GC Edit Parameters** window.

Check the GC configuration



- 1 Select the **Configuration** button, . See the ChemStation Online Help for more information.
- 2 With the Miscellaneous tab selected, set the Pressure Units to psi. Under Valve Configuration set all Valve Type fields to Not Installed, and verify that the MSD transfer line is shown as a Thermal Aux Type.

ALS Valves Inlets C	V	Dven Detector	s Aux Heaters	Events	 Signals	Configuration	1,2, Counters	Readiness
fiscellaneous Columns Modules	ALS						1	
Pressure Units	ValveCo	nfiguration						
jpsi 💽		Valve Type	Name		Para	meters		
Oven	▶ 1	Not Installed	✓ Valve #1					
Slow Fan	2	Not Installed	▼ Valve #2					
	3	Not Installed	✓ Valve #3					
	4	Not Installed	✓ Valve #4					
	5	Not Installed	▼ Valve #5					
Thermal Aux Type	6	Not Installed	▼ Valve #6					
1 Not Installed	7	Not Installed	✓ Valve #7					
2 MSD Transfer Line	8	Not Installed	✓ Valve #8					
3 Not Installed								
	1							

Figure 5 Miscellaneous configuration tab

3 Select the **Columns** tab to display the columns configuration parameters. The HP-5ms checkout column supplied with the MS should be listed under **Column**.

	Column	Calibration Results	Inlet	Outlet	Heated By
1	Agilent 19091S-433: 325 °C: 30 m x 250 µm x 0.25 µm HP-5MS 5% Phenyl Methyl Silox: <not Invertoried></not 	Uncalibrated	Back Inlet	▼ Vacuum	Oven
2	No Column Installed	Uncalibrated	Unspecified 💌	Other 💌	Oven
3	No Column Installed	Uncalibrated	Unspecified -	Other 💌	Oven Oven Oven Oven Oven
4	No Column Installed	Uncalibrated	Unspecified 💌	Front Detector	Oven
5	No Column Installed	Uncalibrated	Unspecified 💌	Front Detector	Oven
6	No Column Installed	Uncalibrated	Unspecified 💌	Other 💌	Oven

Figure 6 Column configuration tab

- 4 If a different **Column** is configured to the inlet location you are using or is attached to the MS, select it and click **Remove**.
- 5 If the HP-5ms is not listed under Column, click the Inventory button and add it to inventory before listing it here. See "Add a column to ChemStation local inventory" on page 48.
- 6 If required, use the up and down arrow keys to put the HP-5ms column in the **1** position.
- 7 For the **Inlet** pressure for this column, select the **Front** or **Back Inlet** from the dropdown.
- 8 For the column **Outlet** pressure select **Vacuum** for the MS
- 9 For the column **Heated By** select **Oven** from the dropdown.

🔳 GC Ec	dit Paramet	ers									×
ALS	Valves	- ∏ Inlets	Columns	(II) Oven	Detectors	Aux Heaters	Events	Signals	Configuration	1,2, Counters	Readiness
Miscellar	neous Colu	mns Modu	les ALS							-	
			FrontInle	at .				PCM C			
			SS Inle	et He	•			PCM C-1	N2 💌	Channel B C	ontrol Mode
			Back Inle					PCM C-2	N2 -	Forward Press	sure 🔻
			SS Inle	He	•					,	_
			Front Detecto	r							
			FI	D							
			Makeu	p N2	-						
		Set	t Lit Offset w	ith GC Key	board.						
			Aux EPC 4,5,	6							
			Aux EPC		•						
			Aux EPC	5 He	•						
			Aux EPC	,							
			101210	• Ine	<u> </u>						
						III					+
He	ala I								Apply	ок	Cancel
He	εφ								Арріу		Cancel

10 Click the **Apply** button and then select the **Modules** tab.

Figure 7 Modules configuration tab

- **11** Select **He** gas from the dropdown for the inlet connected to column 1. The system uses the properties of helium to obtain an accurate flow and pressure relationship for the column.
- 12 Click the **Apply** button to download any edits to the GC.

3 Create a Method for Qualitative Analysis

Set the GC readiness state



1 Select the **Readiness** button, Readiness . The **Readiness** . The **Readiness** .

GC Edit F	Parameters		-			_	-	-		×
	inlets Colum	nns Oven	Detectors	LIO Aux Heaters	فی) Events	Signals	Configuration	1,2, Counters	Readiness	
	-	onents from		j the GC's F iness state	Readine	ss State				
,	 ✓ Oven □ Front Inlet ✓ Back Inlet ((SS Inlet)								
	Front Detect Aux EPC 4 Aux EPC 5 Aux EPC 6									
	PCM C-1 PCM C-2	x 2 (MSD Transfe	er Line)							
	Check All									
Help							Apply	ОК	Cancel	

Figure 8 Readiness state component selection

- 2 Select the Oven, SS Inlet (attached to column 1), and Thermal Aux 2 (MSD Transfer Line). These selections require the GC to wait until all setpoints related to the oven, inlet, and transfer line are held at a steady value before allowing a run to begin.
- **3** Click **Apply** to download these selections to the GC.

Set the GC oven parameters

1



The **Oven** parameters are

Select the **Oven** button, displayed.

For this example we require an oven program that initially holds the column temperature at 50 °C. When the run starts, the column temperature is increased from this temperature to 300 °C at a rate of 35 °C/min. The column is then held at 300 °C for an additional 2 minutes. At this time the oven is cooled down to 50 °C to await the next data acquisition run.

GC Edit Paramet	ters									×
ALS Inlets	Columns	Oven	Detector	rs Aux Heater	rs Events	Signals	Configuration	1,2, Counters	Readiness	
Ven Temp	Actu	al	ſ		Rate ℃/min		Value °C	Hold Time min	Run Tim min	•
50 °C	27.5	°C		(Initial)			50		0	0
Equilibration Time				Ramp 1		35	300		0	7.1429
0.5 min				*						
Maximum Oven Ten	nperature									
325 °C										
🗌 Override C	olumn Max: 3	25 °C								
Cryo:										
C On										
C Quick Cool										
Cryo Use Tempera	ature:									
0°C			L							
Timeout Deter	ction				Post Run: 3(n oct				
0 min										
Fault Detectio	n			Pos	t Run Time: 2	min				
Help							Apply	/ ОК	Cano	el

Figure 9 GC oven parameters

- 2 Mark the **Oven Temp On** checkbox and enter 50 °C in the corresponding field.
- 3 In the Equilibration Time field, enter 0.5 min.
- 4 In the Maximum Oven Temperature field, enter 325 °C. This is the maximum temperature for the HP-5ms column.
- 5 Clear the Override Column Max. 325 °C checkbox.
- 6 In the **Oven Ramp** table, enter the settings shown in Table 2.

Table 2Oven ramp settings

Oven Ramp	Rate	Value	Hold Time
	°C/min	°C	min
(Initial)		50	0
Ramp 1	35.00	300	0

- 7 In the **Post Run** field, enter 300 °C.
- 8 In the **Post Run Time** field, enter 2 min to hold the 300 °C oven temperature for 2 minutes after the run is finished before cooling down to 50 °C for the start of the next run.
- **9** Select **Apply** to download these settings to the GC.

Set the GC column parameters

- 1 Select the **Columns** button, . The **Column** parameters are displayed.
- 2 Check the Column information in the **Selection** list.
 - Column: 19091S-433 (HP-5ms 30 m x 250 μm x 0.25 μm)
 - In: front or back (split/splitless inlet position)
 - Out: Vacuum
- 3 Mark the **Control Mode** checkbox.
- 4 In the Flow Setpoint field, enter 1.0 mL/min. The Pressure, Average Velocity, and Holdup Time Setpoints will be calculated and displayed in the corresponding fields.
- **5** In the dropdown list, select **Constant Flow**.
- 6 In the **Post Run** field, enter **1.0 mL/min**.
- 7 Select **Apply** to download these settings to the GC.

GC Edit Parameters		x
ALS Inlets Columns Oven Detectors	Aux Heaters Events Signals Configuration Counters Readiness	
# Selection Agilent 19091S-433: 325 °C: 30 m x 250 µm x 0.25 µm 1 In: Back SS Inlet He Out: Vacuum	Control Mode ♥ On Setpoint Actual Flow 1mL/min 0mL/min Pressure 7.6522 psi 4.6 psi Average Velocity 36.445 cm/sec Holdup Time 1.3719 min Constant Row ♥ Post Run: 1mL/min Column #1 Configuration Change Column Calibrate Column	
<<< Show All Flows >>>	<u> </u>	$\overline{}$
Help	Apply OK Cancel	

Figure 10 GC columns parameters

Set the GC inlet parameters

- Select the **Inlets** button, displayed.
- 2 Select the **Front** or **Back** tab, depending on your hardware configuration.
- 3 Mark the **Heater** checkbox and enter **250** °C in the corresponding **Setpoint** field.
- 4 Mark the **Pressure** checkbox. The **psi** in the corresponding **Setpoint** field is automatically set when the column flow rate is set.
- 5 Mark the **Septum Purge Flow** checkbox and enter **3 mL/min** in the corresponding **Setpoint** field.
- 6 From the Septum Purge Flow Mode drop down list, select Standard.
- 7 In the **Gas Saver** area:
 - **a** Mark the **On** checkbox.
 - **b** In the field below, enter 20 mL/min.
 - c In the After field, enter 2 min.
- 8 In the **Mode** area:
 - a From the Mode drop down list, select Splitless.
- **9** In the **Purge Flow to Split vent** area:
 - a In the field, enter 50 mL/min.
 - **b** In the **Start Time** field, enter 1.
- 10 Select Apply.

GC Edit Parameters							×
ALS Inlets Columns	Oven Detectors	LIO Aux Heaters	Events Signals	Configuration	1,2, Counters	Readiness	
SSL - Front SSL - Back							
Split-Splitless Inlet							
	Setpoint	Actual	ous buven. ☑ On				
Heater:	250 °C	25.9 °C	20 mL/min		After: 2 min		
✓ Pressure:	7.6522 psi	4.7 psi	,		,		
Total Flow:	54 mL/min	0 mL/min					
Septum Purge Flow:	3 mL/min	-0.1 mL/min					
Septum Purge Flow Mode:	Standard 🗨						
Mode: Spitless	▼ Purge 50 mL	Flow to Split Vent /min	at 1 min				
Help					Ap	ply	OK Cancel

Figure 11 GC inlet parameters

Set the GC injector parameters

If you are not using the autosampler, skip this section.



- 1 Select the **ALS** button,
- 2 Select the **Front Injector** or **Back Injector** tab, depending on your hardware configuration.
- **3** In the **Injection** area:
 - **a** Verify that the **Syringe Size** matches your hardware configuration.
 - **b** In the **Injection Volume** field, enter 1.
- 4 In the Washes and Pumps area:
 - a For Solvent A Washes, enter 5 in the Postlnj field.
 - **b** For Sample Washes, enter 3 in the Prelnj field.
 - c For Sample Pumps, enter 5 in the Prelnj field.
- **5** Select the **Advanced** button, options are displayed in the window.
- 6 In the Plunger Speed area, select Fast.
- 7 In the **Sampling Depth** area,
 - **a** Mark the **Enable** checkbox.
 - **b** In the field, enter 3.6.
- 8 Select Apply.

GC Edit Parameters	×
ALS Inlets ColumnsEditor3	Image: Signals Image: Signal
Front Injector Back Injector Tray / Other	
Injection Syringe Size: $5 \ \mu L$ Injection Volume: $1 \ \mu L$ \checkmark $1 = 1 \ \mu L$	Dwell Time Pre-Injection: 0 min Post-Injection: 0 min
Multiple Injection Delay: 0 sec	Plunger Speed
Washes and Pumps PreInj PostInj Volume (µL) Solvent A Washes: 0 5 Max Image: Comparison of the second sec	Image: Fast C Slow C Variable Draw Dispense Solvent Wash 150 μL/min 3000 μL/min Sample Wash 150 μL/min 3000 μL/min Inject 3000 μL/min Viscosity Delay: 0 ▼
<<	Sample Depth Image: Sample Depth
Help	Apply OK Cancel

Figure 12 ALS parameters

Set the GC Aux heaters parameters

1

Select the **AUX Heaters** button,



- 2 For Thermal Aux 2, mark the **On** checkbox.
- 3 In the Ramps table, enter 280 in the Value °C field.
- 4 Select Apply.

🔳 GC	Edit Parame	eters		-					-				X
ALS	⊣ Inlets	Columns	(D) Oven	Detectors	LO Aux Heaters	المیں Events	Signals	X Configuration	1,2, Counters	Readiness			
The	ermal Aux		_										
N	On	Actual	Γ		Rate °C/min	\ \	/alue ℃	Hold Time min	Run Time min				
	0°C	280 °C		(Initial)			280	0		2			
			L				Final v	alue will be extend	ed by GC run ti	me.			
	Help									Apply	ОК	Cancel	

Figure 13 GC aux heaters parameters

Set the GC signals parameters



- 1 Select the **Signals** button, Signals
- 2 In the **Signal Source** dropdown list, select **None** for all the signal sources.

GC Edit Param	eters						-				X
ALS Inlets	Columns Oven	Detectors	Aux Heaters	کی Events	Ju. Signals	Configuration	1,2, Counters	Readiness			
	Signal Source			Data R	late / Min P	eak Width	Zero	Save			
	#1: None			▼ 50 H	z / .004 min						
	#2: None #3: None				z / .004 min						
	#3: None #4: None				z / .004 min z / .004 min		· 🗖				
	Show Dual Injection S			e to enable	event definiti	on.)					
Delete Events	Signal Source	+	Time, min	Signal E	vent						
	•	<u> </u>]					_			
Help								Apply	ОК	Cancel	

Figure 14 GC signals parameters

3 Select **OK** to download the selected parameters to the GC and close the **GC Edit parameters** window. The **GC Detector Data** dialog box opens. See Figure 15 on page 42.

Edit the GC real time plots to display

GC Detector Data			×					
Signal 1	Signal 2	Signal 3	Signal 4					
🗖 Display	🗖 Display	🗖 Display	🗖 Display					
Attn: 0 2^	Attn: 0 2^	Attn: 0 2^	Attn: 0 2^					
Offset: 10 %	Offset: 10 %	Offset: 10 %	Offset: 10 %					
Time: 5.0 min	Time: 5.0 min	Time: 5.0 min	Time: 5.0 min					
	OK C.	ancel Help						

Figure 15 Selecting GC signals to plot in real time

- **4** From the **GC Detector Data** dialog box, clear the checkboxes for all signals. We will not be plotting GC signals.
- 5 Select OK to save the settings and close the dialog box. The MS Tune File dialog box opens. See Figure 16.

Edit the MS parameters

MS Tune Fi	ile				x
Path:	D:\msdchem	\1\5975\			
Files:	atune.u bfb.u dftpp.u NCICH4.u pcich4.u stune.u target.u				
		ОК	Cancel	Help	

Figure 16 Selecting the method MS tune parameter file

- 1 Select atune.u from the File list.
- 2 Select OK to assign the tune file to the current method and close the MS Tune File dialog box. The MS SIM/Scan Parameters dialog box opens.
- 3 In the **MS Instrument** area enter:
 - a In the Solvent Delay field, enter 3.00 min.
 - **b** In the **EMV mode** drop down list, select **Gain Factor**.
 - **c** In the **Gain Factor** field, enter 1.00.

- d In the Acq. mode drop down list, select Scan.
- e In the Scan Speed drop down list, select Normal.
- f Clear the Acquire both Scan and SIM data checkbox.
- 4 In the **Real-Time Plot** area **Time Window** field, enter 10.
- **5** In the **MS Window 1** area:
 - a From the Plot Type dropdown, select Total.
 - **b** In the **Y-Scale** fields, enter 0 to 2000000.
- 6 In the MS Window 2 area:
 - a From the Plot Type dropdown, select Spectrum.
 - **b** In the **Y-Scale** fields, enter 0 to 1000000.

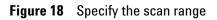
MS SIM/Scan Parameters	×
MS Instrument	Real-Time Plot
Sample Inlet: GC	Time Window: 10 min.
Solvent Delay: 3.00 min. EMV Mode: Gain Factor ▼ Gain Factor: 1.00 = 506 V Acq. Mode: Scan ▼ Scan Speed: Normal ▼ Acquire both Scan and SIM data: □	MS Window 1 Plot Type: Total Y-Scale: 0 to 2000000 MS Window 2 Plot Type: Spectrum Y-Scale: 0 to 100000
Tune Fileatune.u	
Scan Parameters Zones OK Cancel	Timed Events Help

Figure 17 Setting the MS scan parameters

- 7 Select Scan Parameters. The Edit Scan Parameters dialog box opens.
- 8 Select the Scanning Mass Range tab:
 - a Mark the Scan Group 1 checkbox.
 - **b** In the Start at Mass field, enter 50.00.
 - c In the End at Mass field, enter 300.00.

This scan range includes all the expected ions.

E	dit Scan Parame	eters		-	_		? ×			
	Scanning Mass	Range Thresh	old and Samplin	ng Rates Plotting						
		Scan Gr		art Time Start at inutes) Mass (amu 3.00 50.00						
	Summary o	of Settings								
	Group	Start Time	Low Mass	High Mass	Threshold	Samples	S			
	1	3.00	50.00	350.00	150	2	4.			
	•									
		Low to	High mass rang	je must be in ascending	order from 1.60 -	1050.00. Close	Help			



- **9** Select the **Threshold and Sampling Rates** tab:
 - **a** In the **Threshold** field, enter 40.
 - **b** In the **Sampling Rate** field, enter 3.

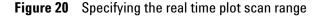
	eters		-	-	२ ×
Scanning Mass	Range Thresho	old and Sampling Rate	es Plotting		
		Thres (cou			
	Scan	Group 1	40	3	
	Scan	Group 2			
	Scan	Group 3			
Summary o	_	High Mass	Threshold	Samples	Scans/Sec
Summary of art Time 10	of Settings Low Mass 50.00	High Mass 350.00	Threshold 40	Samples 3	Scans/Sec
art Time	Low Mass				
art Time IO	Low Mass 50.00	350.00	40	3	2.44

Figure 19 Setting the sampling rate and threshold filter

- **10** Select the **Plotting** tab, In the **Plot Window #2** area:
 - **a** Under Low Mass, enter 50.
 - **b** Under High Mass, enter 350.

Plot Window #1 was set to be a TIC so no plotting entry is required. **Plot Window #2** is a spectrum including all ions found between 50 and 350 m/z.

canning Mass Range Th	hreshold and Samp	ling Rates Plotting			
	Plot Wir	ndow #1	Plot Wi	ndow #2	
	Low Mass	High Mass	Low Mass	High Mass	
Scan Group 1			50.00	400.00	
Scan Group 2					
Scan Group 3					
- Summary of Settings-	Samples	Scans/Sec	Plot 1 Plot 1	Plot 2	Plot 2
	Samples	Scans/Sec 2.44	Plot 1 Plot 1	Plot 2 50.00	Plot 2 400.00
Summary of Settings			Plot 1 Plot 1		
Summary of Settings			Plot 1 Plot 1		
Summary of Settings Threshold 40					400.00
Summary of Settings Threshold 40	3	2.44		50.00	400.00



- Select Close to save the settings and return to the MS SIM/Scan Parameters dialog box.
- 12 Select **OK** to save the parameters and close the dialog box. The **Save Method As** dialog box opens. See Figure 21.

Save the method

- 1 Enter demoscan.M in the Method File field.
- 2 Select **OK** to save the current ChemStation method as **demoscan.m** method.

Save Method As		×
Method Path:		
D:\msdchem\1\METHODS\		Browse
Method File :		
demoscan.M		
ОК	Cancel	Help

Figure 21 Saving the method

General Information for Editing the GC Parameters

Open the GC edit parameters window

1 From **Instrument Control** select the **GC Parameters** button to display the **GC Edit Parameters** window. See Figure 9 on page 33.



2 When a parameter button at the top of the screen is selected, the button is highlighted in blue and the settings for that parameter are displayed in the right panel. The GC instrument status is shown in the left panel.

Table 3 lists a description of the **GC Edit Parameters** window buttons.

Button	Action
Apply	Downloads any settings that have been changed to the GC.
ОК	Downloads any settings that have been changed to the GC and closes the GC Edit Parameters window.
Cancel	Discards any settings that have been changed and closes the GC Edit Parameters window.
Help	Displays help topics for the current parameter.

Add a column to ChemStation local inventory

Use the **Add Column to Local Inventory** dialog box to select a column from the **Column Catalog** and add it to your **Local Column Inventory**. This example adds the supplied checkout column to local inventory.

1 Select the **Configuration** icon to display the columns configured for the instrument.

GC E	idit Para	meters				×
ALS	⊣ Inlet	s Columns Oven Detectors	Aux Heaters Events Signals	Configuration	1,2, Counters	Readiness
Miscella	ineous	Columns Modules ALS				
			Inventory	Calibr	rate	Remove
		Column	Calibration Results	Inlet	Outlet	Heated By
\uparrow	1	Agilent 19091A-102: 325 °C: 25 m x 200 μm x 0.33 μm Ultra 1 Methyl Siloxane: 1528,43506	Uncalibrated	Front Inlet	/acuum 🗖	Oven 💌
$ \downarrow $	2	No Column Installed	Uncalibrated	Unspecified 💌 F	Front Detector 💌	Oven Oven
	3	No Column Installed	Uncalibrated		Other 🗾	Oven 💌
	4	No Column Installed	Uncalibrated		Dther 🗾	Oven 💌
	5	No Column Installed	Uncalibrated	Unspecified 💻 (Dther 🗾	Oven 💌
	6	No Column Installed	Uncalibrated	Unspecified 💻 (Dther 🗾	Oven 💌
Н	elp			Apply	ОК	Cancel



 Inst	all Column 1									×
Sel	ect column to ir	nstall from Local Inve	entory listed	below:						
Dr	ag a column hea	ader here to group b	y that colur	mn						
	Inventory Number	Manufacturer	Model	Description	Temperature, °C	Length, m	Diameter, µm	Film Thick- ness, µm	Calibration Information	
He	4 4 #0 / 0								>	
	*070									
	Add Column	to Local Inventory								
	Delete S	elected Column		Insta	Il Selected Column		Done		Help	

2 Click **Inventory** to display the **Install Column 1** dialog box containing a list of columns in local inventory.

Figure 23 The local inventory of columns

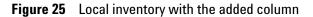
	Manufacturer	Model	Description	Temperature, °⊂	Length, m	Diameter, µm	Film Thick- ness, µm
P	Agilent	19091J-236	HP-5 5% Phenyl Methyl Siloxane	325	60	250	1
P	Agilent	19091J-313	HP-5 5% Phenyl Methyl Siloxane	325	30	320	0.1
Æ	Agilent	19091J-333	HP-5 5% Phenyl Methyl Siloxane	325	30	250	0.1
Æ	Agilent	19091J-411	HP-5 5% Phenyl Methyl Siloxane	325	15	320	0.25
F	Agilent	19091J-413	HP-5 5% Phenyl Methyl Siloxane	325	30	320	0.25
f	Agilent	19091J-416	HP-5 5% Phenyl Methyl Siloxane	325	60	320	0.25
f	Agilent	19091J-431	HP-5 5% Phenyl Methyl Siloxane	325	15	250	0.25
► A	Agilent	19091J-433	HP-5-5% Phenyl Methyl Siloxane	325	30	250	0.25
Æ	Agilent	19091J-436	HP-5 5% Phenyl Methyl Siloxane	325	60	250	0.25
Æ	Agilent	19091J-441	HP-5 5% Phenyl Methyl Siloxane	325	10	100	0.4
A	Agilent	19091J-442	HP-5 5% Phenyl Methyl Siloxane	325	20	100	0.4
A	Agilent	19091L-005	HP-50+ 50% Phenyl Methyl Siloxane	310	50	200	0.11
_	Agilent • • • #73 / 1	19091L-102 060 • • • •	HP-50+ 50% Phenyl Methyl Siloxane	310	25	200	0.33 🗸

3 Click Add Column to Local Inventory to display the Add Column to Local Inventory dialog box.

Figure 24 The catalog of columns

- 4 Scroll down the list of columns to model number **19091J-433** and enter hp5ms433 as the **New Inventory#**.
- 5 Click Add Selected Column to Inventory to display the Install
 Column 1 dialog box with the selected column now added to the local inventory list.

	1			le alacció					
'e	liect column to ir	nstall from Local Inv	entory listed	Delow:					
Drag a column header here to group by that column									
DI	rag a column he	ader here to group I	by that colur	mn					
	rag a column he	ader here to group I	by that colur						
Di	-							Film Thick-	Calibration
Di	Inventory	Manufacturer	Model	Description	Temperature, °C	Length, m	Diameter, µm	Film Thick-	Calibration
Di	-				Temperature, °C	Length, m	Diameter, µm	Film Thick- ness, µm	Calibration Information



Columns added to local inventory can be quickly added and configured for the instrument. See "Select and configure a column" on page 51.

Select and configure a column

This example selects a column previously added to local column inventory and configures it as column number 1. See "Add a column to ChemStation local inventory" on page 48.

1 Select the **Configuration** icon and click on the **Column** description for column 1 to select it. The column number selected here will be replaced with the column we are adding.

GC Eq	dit Parar	neters										
ALS	⊐ <mark> </mark> Inlets	Columns	(D) Oven	Detectors	LIO Aux Heaters	Events	Signals	Configurati	1,2, on Counter	rs R	Readiness	
1iscellan	neous	olumns Modules	ALS									
-							nventory	C	librate		Remove	
[Column			Calibration			C.	librate Outlet		Remove Heated E	
	1	Column Agilent 19091A-1 0.33 µm Ultra 1 Methyl Silv					-		Outlet	-		
↑ ↓	1	Agilent 19091A-1 0.33 μm	oxane: 152		x		-	Inlet	Outlet	-	Heated E	3y

Figure 26 Columns configured for the instrument

2 Click **Inventory** to display the **Install Column 1** dialog box containing a list of columns in local inventory.

Dra	ag a column h	eader here to gr	oup by that c	olumn					
	Invent Number	Manufact	Model	Description	Temperature, °⊂	Length, m	Diameter, µm	Film Thick- ness, µm	Calibration Information
•	hp5ms433	Agilent	19091J	HP-5 5% Phenyl Methyl Silo	325	30	250	0.25	Uncalibrated
	1528.43506	Agilent	19091A	Ultra 1 Methyl Siloxane	325	25	200	0.33	Uncalibrated
HH		Agilent	19091A	Ultra 1 Methyl Siloxane	325	25	200	0.33	

Figure 27 The local inventory of columns

3 Select a column from the local inventory list and click **Install Selected Column** to display the **Configuration** panel for **Edit GC Parameters** with the selected column replacing the previously configured column 1 for the instrument.

C Ed	lit Para	ameters								
S.	⊣į Inlet	ts Columns Columns Modules	(D) Oven	Detectors	LIO Aux Heaters	ویک Events	Signals	Configuration	1,2, Counters	Readiness
.c.idi i		Induics				I	nventory	Cali	brate	Remove
Г		Column			Calibration F	Results		Inlet	Outlet	Heated B
	1	<mark>Column</mark> Agilent 19091A-10 0.33 μm Ultra 1 Methyl Silos			Calibration F	Results		Inlet Front Inlet	Outlet Vacuum	▼ Oven
- _	1 2	Agilent 19091A-10 0.33 μm	xane: 1528		×	Results		Front Inlet	Vacuum	↓
- _	1 2 3	Agilent 19091A-10 0.33 μm Ultra 1 Methyl Silo:	xane: 1528 ed		^X Uncalibrated	Results		Front Inlet	Vacuum	■ Oven
-11	_	Agilent 19091A-10 0.33 μm Ultra 1 Methyl Silo: No Column Installe	xane: 1528 :d :d		X Uncalibrated	Results		Front Inlet	Vacuum Front Detector	✓ Oven ✓ Oven
	3	Agilent 19091A-10 0.33 μm Ultra 1 Methyl Silos No Column Installe No Column Installe	xane: 1528 ed ed		X Uncalibrated Uncalibrated Uncalibrated	Results		Front Inlet	Vacuum Front Detector Other Other	Oven Oven Oven Oven

Figure 28 Columns configured for the instrument

- 4 Under the **lnlet** heading dropdown, select the item the column inlet is attached to.
- 5 Under the **Outlet** heading dropdown, select the item the column outlet is attached to. For an MS select **Vacuum**.
- **6** Under the **Heated By** heading dropdown, select the method for controlling the column temperature.

Upload parameters from the 7890A GC

- 1 On the **Instrument > GC Edit Parameters** screen, right-click in the blank area.
- 2 From the shortcut menu, select Upload Method from GC.

Customize the status panel view

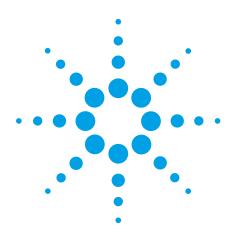
1 In the status panel, select the **Setup Actuals** button, the **Status Items** dialog box opens.



Status Items Select Status Items to Display	
Status Items GC Information GC Information LTM Front Inlet Back Inlet Front Detector Thermal Aux QQQ> Columns Valves	Select All Clear All
Help Sa	ave Cancel

- 2 Mark the checkboxes of the items in the **Status Item** list that you want to have displayed in the status panel.
- **3** To move an item up or down in the displayed list, select the item and then the up or down arrow buttons until it is in the desired position.
- 4 Select **Save** to save the settings and return to the **GC Edit Parameters** window.

3 Create a Method for Qualitative Analysis



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Run the Scan Method

Prepare the Sample56Load the Method57Run the Method58Take a Snapshot61View the Logbook62

4

In this chapter, a sample is prepared for data acquisition and the ALS is loaded with the sample, the solvent wash vial, and a solvent waste vial. The single sample is run and during the data acquisition a snapshot is taken to demonstrate how it is possible to look at partial analysis results before a run is completed. Finally, the logbook showing actions taken during the run is reviewed.



Prepare the Sample

1 Fill a sample vial with the contents of the 10 ng/mL 5975 MSD Sample (P/N 05970-60045 or P/N 5074-3025 Japan only) and cap the vial.

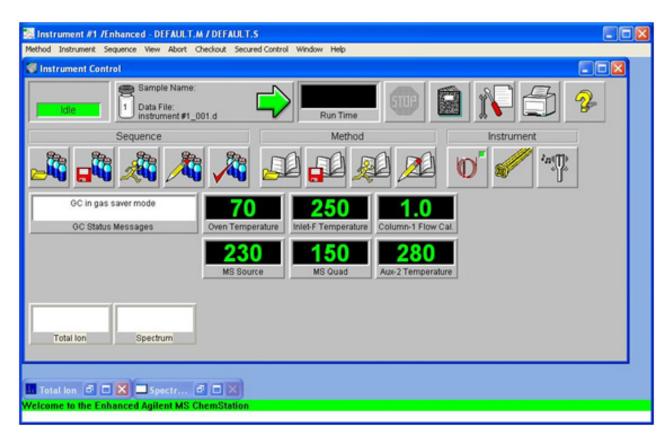
If you are not using an ALS skip the remaining steps.

- **2** Place the sample vial into position 1 of the GC sample tray.
- **3** Fill a solvent wash vial with isooctane and place it in injector turret location A for solvent wash mode A, B.
- 4 Place a waste vial in turret location B specified for solvent wash mode A, B.

Load the Method

1 From the PC desktop, select the **ChemStation** shortcut icon, the **Instrument Control** window opens.





- 2 Select the Load Method button, is to open the Load Method window. Navigate to and select demoscan.M.
- **3** Select **OK** to load the method and close the dialog box.

Run the Method



1 Select the **Run Method** button, **Select** the **Start Run** dialog box opens with the **GC ALS**, **Inlet Location**, and **MS Connected to** selections pre-populated.

Basic Advanced	ction Style: GC ALS				
- Inlet Location	© Front C Rear Operator Name: John	C Dual	MS Connected to —	Front Inlet	C Rear Inlet
-Front Inlet	Data Path: C:\M	1SDCHEM\1\DATA\	– Rear Inlet –		Browse
Data File Name: Sample Name:	EVALSCAN_1.D	Browse	Data File Name:	EVALDEMO.D	Browse
Misc Info:			Misc Info:		
Expected Barcode:			Expected Barcode:		
Sample Amount: Multiplier:	0		Sample Amount: Multiplier:	0	
Vial Number: Tray Name:	1 Agilent ALS	-	Vial Number:	igilent ALS	
Select Injection Vol	ume:	_	Select Injection Volume		
C Override	e using 1 µL		C Override us	ing µL	
Method Sections to Run -	 Data Acquisition Data Analysis 	OK and Ru	n Method	Exit	Cancel Help

Figure 29 Start a single sample run

2 In the **Operator Name** field, enter your name.

- 3 In the **Front Inlet** area:
 - a In the Data File Name field, enter EVALSCAN 1.
 - **b** In the **Sample Name** field, enter a name for your sample (optional).
 - **c** In the **Misc Info** field, enter a description of your scan (optional).
 - d In the **Expected Barcode** field, enter a barcode (optional).
 - e In the Vial Number field, enter 1.
- 4 In the Method Selections to Run area:
 - a Mark the Data Acquisition check box.
 - **b** Clear the **Data Analysis** check box.
- 5 When the instrument is in a ready state as shown by a green ldle indicator in the upper left hand corner, select OK and Run Method to close the dialog box and start the run. The ready state indicator changes to Run. See Figure 30 on page 60.

If the instrument was not in a ready state, the system will prompt for you to override. When the status is Ready, the dialog box will close automatically.

GC Acquisition
Waiting for GC ready To override ready, press Override.

During the solvent delay the system will prompt for you to override. When the time is up, the dialog box will close automatically.

Acquisition	
Override solvent o	lelay (3.00 minutes)?
	g solvent delay may ilament lifetime.
Yes	No

6 Observe the TIC real time plot and go to "Take a Snapshot" on page 61 after the second compound elutes.

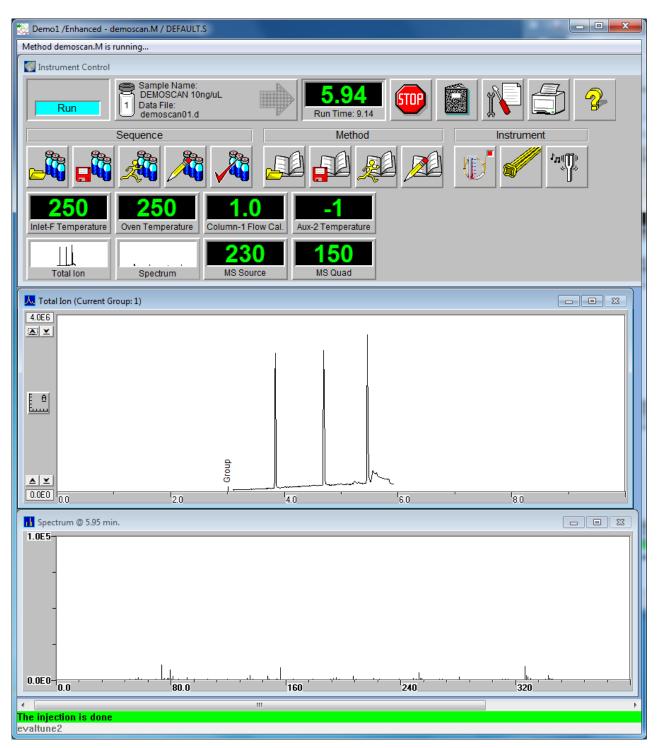


Figure 30 Instrument control window during single sample run

Take a Snapshot

Snapshot is useful when a compound of interest elutes early during a long run and you want to analyze that compound immediately. The system creates a snapshot data file with data that has been acquired up to the time the Snapshot is taken.

- During the run select View > Data Analysis to open the data analysis view.
- 2 Select **File > Take Snapshot**. The data analysis windows opens displaying the TIC obtained for the run up to this point in time.

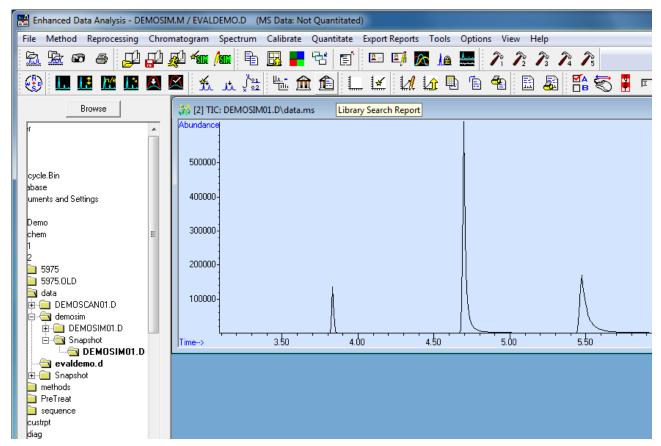


Figure 31 The TIC of the snapshot data file

Observe the location of the snapshot data file in the navigation pane. It is placed in the data directory specified for the run under the snapshot subdirectory and given the same name as the data file specified for the sample.

- **3** Analyze the compound of interest.
- 4 Exit data analysis and return to the **Instrument Control** view.

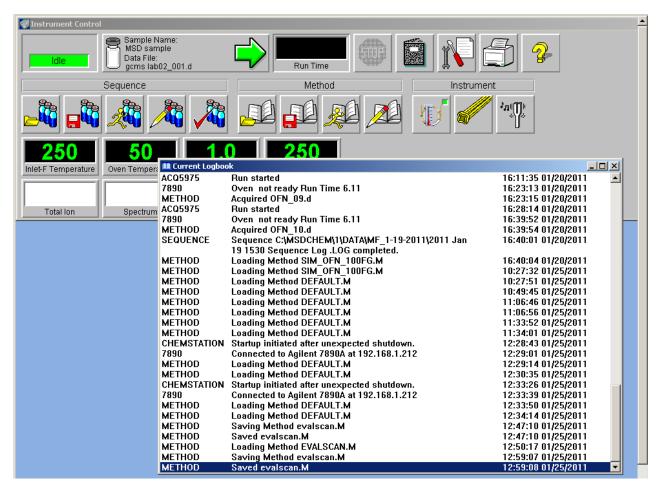
View the Logbook

The system keeps a logbook named MSLOGBK.LOG that records all instrument error and status messages prior to and during acquisition.

The Current Logbook lets you review instrument diagnostic information and any mass spec malfunctions recorded during the current and previous acquisitions. It is located in the instrument directory.



- Select the **Logbook** button, **_____**. The **Logbook** menu opens.
- 2 Select **Current Logbook** to display the active log.

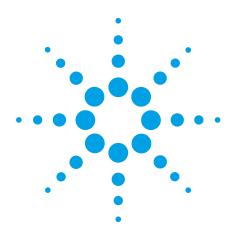


1

Figure 32 The current logbook is open

- **3** With the logbook open, select the **Logbook** button again and then from the menu select:
 - **Open Logbook** to select a a logbook to open from a list of all logbooks in the instrument directory.
 - **Clear Logbook** to delete the currently displayed logbook.
 - **Save As Logbook** to save the displayed logbook into a new file.
 - **Print Logbook** to print the displayed logbook.
- **4** Exit the Instrument Control program.

4 Run the Scan Method



5

Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Qualitative Data Analysis

Integrate Peaks 66 Edit the Method to Generate a Report 74 Display Extracted Ion Chromatograms (EIC)s 76 Enable or Disable the Right Mouse Click Context Menu 78 Analyze Data 79 Search the Spectral Library 83 Print a Window, TIC, Spectrum, or Method 86 Save the Data Analysis Method 87 Exit the Data Analysis Program 88

Qualitative data analysis identifies the compounds in your sample by:

- Integrating the peaks in your acquisition scan data
- Identifying the ions in the spectra from those peaks
- Comparing the ions from the peaks it found to ions in a library of known compounds, stored on your system
- Reporting the identity of the compound(s) found for each peak

This chapter reviews each of these processes.



5 Qualitative Data Analysis

Integrate Peaks

Integration is a tool for finding the peaks in a chromatogram and determining their size. In qualitative analysis integration is required for producing a percent report, doing a library search on integrated peaks, and producing a library search report.

1 Start the data analysis program using the desktop Data Analysis icon, _____.



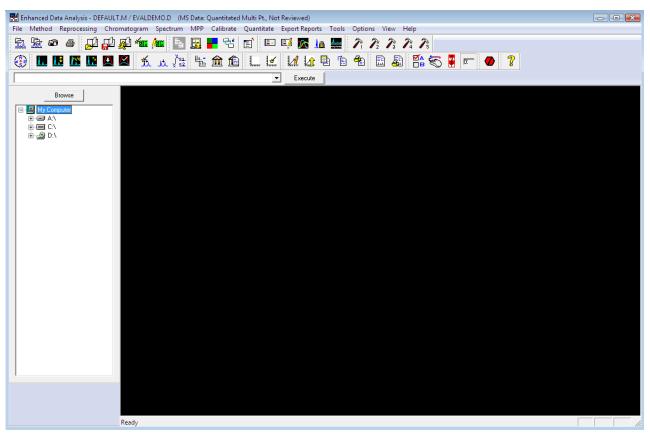


Figure 33 The initial data analysis window

2 Select the Load Data File button, dialog box is displayed.



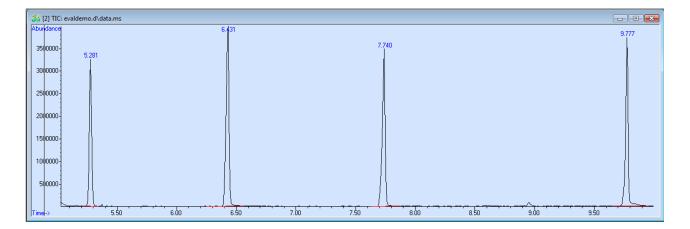
Select Data File		
Path: C:\msdchem\1\data		
Change Path evaldemo.d FAMES-2COL-SPLIT01.D L-Histofine.D RI-calibration.D	Operator: Vial: Misc Info: Sample Name:	perkins 1 10 ng per component demoscan sample
	Abundance	8.60
OK	Cancel	Help

3 Select Change Path. The Browse for Folder dialog box opens.

Browse For Folder	×
Select folder containing Datafiles	
🧮 Desktop	*
▷ My Documents	
Public	
4 🖳 Computer	
Floppy Disk Drive (A:)	
🛛 🚰 Local Disk (C:)	=
Database	
EnvDemo	
D GCMS	
🖉 🎍 msdchem	
▲ <u>↓</u> 1	
> 5975	
> 5975.OLD	
A B DATA	
i evaldemo.d	
FAMES-2COL-SPLIT01.D	
b L-Histidine.D	
II-calibration.D METHODS	
sequence	
sequence	
	Ŧ
	_
Make New Folder OK Cancel	
	H.

- **4** Navigate to **evaldemo.d**. This is the data file from the scan analysis of our sample.
- 5 Select **OK**.

6 In the **Select Data File** dialog box, select **OK**. The data file is loaded and the total ion chromatogram (TIC) is displayed.



Edit the integration events

When the data analysis part of your method is run, the chromatogram is integrated using autointegrate. Most of the chromatogram can be successfully integrated by using the ChemStation default auto integration parameters. However, you can customize the auto integration parameters and add integration events for your specific chromatograms. These events are saved and used when your method is run.

1 Select the Integration Parameters button, ∠ 22. The Edit Integration Events dialog box opens.

This example assumes the **ChemStation Integrator** is the specified integrator

Edit Integration Events			×
Possible Events	Event	Value	Time
•	Initial Area Reject	0	Initial
	Integrator Event Name Value		
	Initial Area Reject 0 Initial Peak Width 0.027 Shoulder Detection 0FF	Initial	
	Initial Threshold 18.2	Initial	
Apply Load Save	Enter Delete Of	Cance	l Help

- 2 To change Initial Area Reject, Initial Peak Width, or Initial Threshold:
 - a Select the parameter you wish to change in the **Integrator Event Name** list. The parameter is displayed in the **Event** field and the current value is displayed in the **Value** field.
 - **b** Enter the custom value in the **Value** field.
 - c Select Enter. The custom value is now listed in the Value list.
- **3** To change **Shoulder Detection**:
 - a Select Shoulder Detection in the Integrator Event Name list. The parameter is displayed in the Event field and the current setting is displayed in the Value field.
 - **b** Select the **Value** field. An **Edit Integration Events** confirmation message appears.
 - c Select **Yes** to change the setting.

- 4 To add integration events:
 - **a** From the **Possible Events** drop down list, select the event to add to your integration.
 - **b** Enter the required information in the **Value** or **Time** fields.
 - c Select Enter. The event and value or time is now listed in the Integrator Event Name, Value, Time list.
- Select **Apply** to view the results in the **TIC** window. 5
- 6 Select **Save** to save the auto integration parameters. The Save Events dialog box opens.

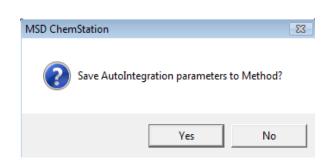
Save events	х
C:\msdchem\1\methods\evalscan.M\	_
,	
manual integration 1.e	
OK Cancel Help	

- 7 Enter a file name.
- Select **OK** to close the **Edit Integration Events** dialog box. The 8 results are displayed in the **TIC** window.

Save the integration events to the method

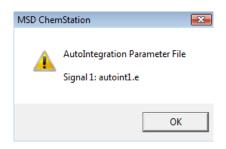


. The integration Select the **AutoIntegrate** button, 1 results appear in the **TIC** window (Figure 34) and a confirmation message appears.



2 Select **Yes** to save the integration or **No** to continue without saving this integration to the method.

If you selected **Yes**, a confirmation message appears displaying the saved auto integration parameter file name. Select **OK** to save the integration to the method.



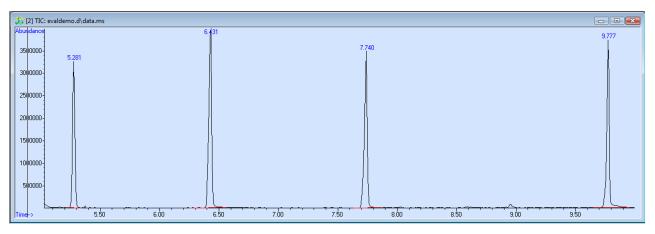
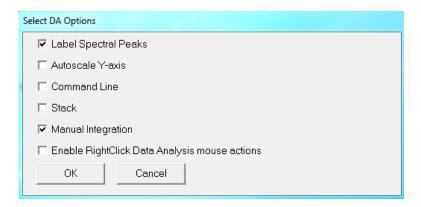


Figure 34 Integrated chromatogram

Manually integrate peaks

- 1 If required, "Edit the integration events" or load a saved integration events file.
- 2 Select Tools > Options to display the Select DA Options dialog box.



- **3** Select **Manual Integration** to turn it on and click **OK**. The mouse cursor changes to a crosshair in the **TIC** window.
- 4 Right-click the TIC to display a context menu. Select **Enable** standard Data Analysis mouse actions from the menu.
- **5** Click and drag the left mouse button to zoom in on the peak of interest in the chromatogram.
- 6 Click and drag the right mouse button to draw an integration baseline on the peak. When you release the button, the peak will be integrated, using the integrator you have selected.

If you want to delete an integrated peak, put the cursor on it and double-click the right mouse button.

View the integration results in a table

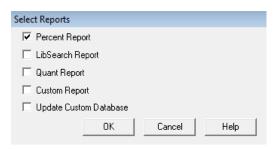
1 Select **Chromatogram > Integrate Results...**. The **Tabulate** window opens and lists the results.

Ret Time 5.281	Type	Width				
		vvium I	Area	Start Time	End Time	
	BV	0.023	44191981	5.210	5.342	
6.431	BB	0.027	69317820	6.250	6.563	
7.740	BB	0.028	59113575	7.630	7.877	
9.777	BBA	0.024	54740746	9.650	9.953	
	9.777					

- **2** To print the integration table, select **Print** and navigate to your printer.
- **3** To copy the table to your clipboard for use in another application, such as MS Excel, select **Copy**.
- 4 Select **Close** to close the dialog box.

Edit the Method to Generate a Report

1 Select Method > Edit Method. The Select Reports dialog box opens.

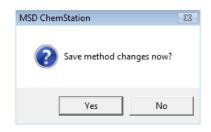


2 Check **Percent Report** and **OK**. Other report types can also be selected.

Percent Report Options		x
Sort by	Signal	
Destination		
🗖 Screen		
I Printer		
🗖 File		
Integration Parameter File:	Browse]
ОК	Cancel Help	

The **Percent Report Options** dialog box opens.

- 3 In the **Destination** pane, check where you want the report to be generated.
- 4 Select **OK**. A confirmation message appears.



5 Select Yes. The Save Method As dialog box opens.

Save Method As	×
Method Path:	
C:\msdchem\1\METHODS\	Browse
Method File :	
default.m	
OK Cancel H	elp

- 6 Select **OK** to save the setting to the current method.
- 7 To interactively generate a report, select **Chromatography** > **Percent Report**. The report is displayed in a new window.

Data	Path								
Data	Path							A	rea Perce
Opera Sampl Misc	File In Itor .e	: C:\r : eva] : 7 { : [BSE : demo : 10 r : 1	Ldemo Sep 19 31]pei oscan ig pei	.d 989 kins samp r com	13:59 Le poner)			
Integ Metho Title	jrator Id	:	nStat: nsdchi	ion em\1\I	METHO)DS\defau]			
eak	R.T.		nax	last	РК		corr. area		
1 5	.281	24	32	40	 BB	 2921136	 42153440	 86.85%	 29.439%
26	.431	170	176	182	BB	1638563	15421309	31.77%	10.770%
3 7	.740	330	339	352	BB	2286751	37079934	76.40%	25.896%
49	.777	578	594	6 08	BB	3379976	48534958	100.00%	33.896%
				Sum	of c	orrected	areas:	143189640	
fault	.n Tu	e Jan	04 09	9:47:1	04 20	911			

5 **Qualitative Data Analysis**

Display Extracted Ion Chromatograms (EIC)s

1 Chromatograms dialog box opens.

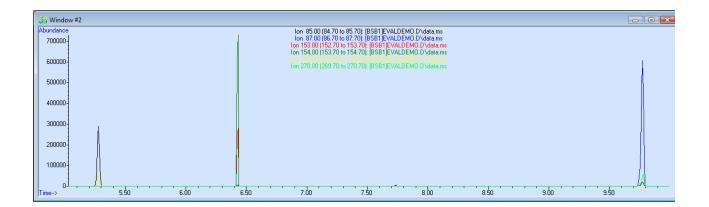


Extracted Ion	Chromatograms		
Time Range:	5.030 to	9.997	minutes
_ lons			
1:	85.00	4: 154.00	
2:	87.00	5: 170.00	
3:	153.00	6: 270.00	
	Use m/z range from - 0.30	to + 0.7	0
	OK Cancel	H	telp

- 2 In the **Time Range** fields, enter the range you wish to extract. The complete time range of the data file is initially displayed. You can specify a shorter time range by entering the appropriate starting and ending values.
- 3 In the lons area, enter the ion masses of interest. You can specify up to six ions.
- 4 In the **Use** *m*/*z* range from fields, enter the range of interest. The default m/z range for each ion is -0.3 to +0.7 of the ion mass specified. You can change the range by entering the appropriate starting and ending values.
- 5 Select **OK**. A window opens displaying a chromatogram for each ion.

🎎 Window #	[‡] 2									, • 💌
Abundance 7305593	Ion 85.00 (84.70 to 85.70); [BSB1]EVALDEM0.D\data.ms									
0≟_, Time>		6.00	6.50	7.00	7.50	8.00	8.50	9.00	9.50	-, , ,
Abundance 730559a		0.00			0 to 87.70): [BSB1]EVAL			0.00	0.00	
01 Time>	5.50	6.00	6.50	7.00	7.50	8.00	8.50	9.00	9.50	д
Abundance 730559	3.30	0.00	0.30		'0 to 153.70): [BSB1]EV/		0.30	3.00	3.30	
01	5.50	6.00	6.50	7.00	7.50	8.00	8.50	9.00	9.50	
Time> Abundance 730559a	5.50	6.00	6.50		7.50 70 to 154.70): [BSB1]EV/		0.00	3.00	3.50	
01			A							
Time> Abundance 730559g	5.50	6.00	6.50	7.00 Ion 170.00 (169.7	7.50 70 to 170.70): [BSB1]EV/	8.00 ALDEMO.D\data.ms	8.50	9.00	9.50	
03										
Time> Abundance 7305595	5.50	6.00	6.50		7.50 70 to 270.70): [BSB1]EV/	8.00 ALDEMO.D\data.ms	8.50	9.00	9.50	
730559										
Time>	5.50	6.00	6.50	7.00	7.50	8.00	8.50	9.00	9.50	

6 Select the **Merged Format** button **b** to toggle from a chromatogram that displays the ions separately to one that displays the ions superimposed.



Enable or Disable the Right Mouse Click Context Menu

A right mouse click context menu can be enabled to allow you easy access to common data analysis tasks directly from a chromatogram or spectrum window rather than from using the main menu or toolbar buttons.

Selecting the **Switch Data Analysis Mouse Actions** button from the toolbar toggles between enabling and disabling the context menu. When the enhanced data analysis context menu is enabled, the standard right button mouse actions are disabled. The enabled context menu is shown in Figure 35.



	Integrate	
	Tabulate	
	Print	
	Select	
	Select and Search	
	Create Metafile	
	Сору	
	Redraw	
	Annotate	
	Enable Standard Data Analysis mouse actions	
	· · · · · · · · · · · · · · · · · · ·	
		.^.
7	, , , , , , , , , , , , , , , , , , , 	

Figure 35 Right mouse click context menu

Certain mouse actions like averaging peak spectra and manually editing the baseline of a peak require the standard mouse actions.

Analyze Data

To perform these actions you must be using the standard mouse actions. See the preceding topic for details.

1 Enlarge the first peak using a left mouse click and drag to create a rectangle around the peak. The chromatogram is enlarged for the selected area. This is the peak for the compound Dodecane.

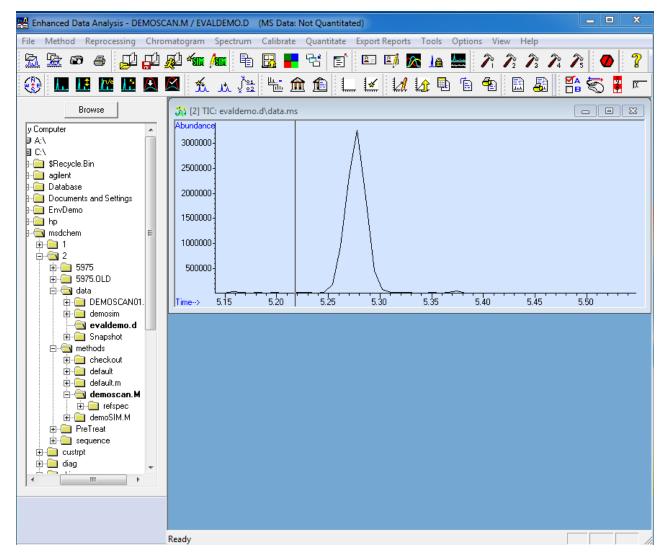
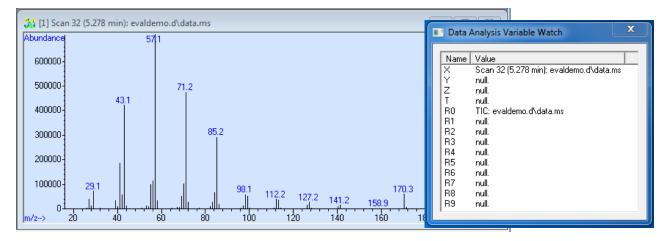


Figure 36 The enlarged peak

- **2** Enable the stack window:
 - a From the main menu, select **Tools > Options**.
 - b In the Select DA Options dialog box, check Stack and OK. The Data Analysis Variable Watch window opens.

Data Analysis Variable Watch
Name Value X null. Y null. Z null. T null. R0 TIC: evaldemo.d\data.ms R1 null. R2 null. R3 null. R4 null. R5 null. R6 null. R7 null. R8 null. R9 null.

3 Position the cursor at the highest point of the first peak and double right mouse click to display the spectrum.



You must be using the standard mouse actions.

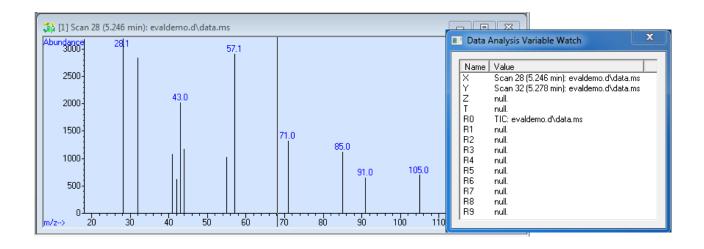
Figure 37 The spectrum at the peak apex

The **Data Analysis Variable Watch Window** now shows the peak spectrum in the \boldsymbol{X} register.

Subtract the baseline noise from the spectra

Use spectral subtraction to improve the quality of your spectra by subtracting the baseline signal (noise) from peaks of interest.

1 With the peak apex stored in the Stack X register, position the cursor on the peak at its baseline and double right mouse click. The spectrum is displayed and placed in the X register in the Data Analysis Variable Watch window. The previous spectrum (peak apex) in the X register is moved to the Y register.



2 Select the Subtract button, . The difference (Y - X) will be displayed as a spectrum labeled with a [-] following its title. See Figure 38.

μ.-

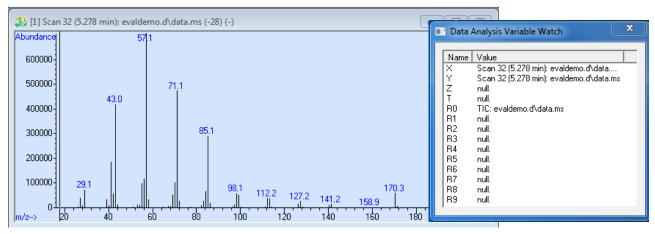


Figure 38 Subtracted spectrum for Dodecane

Select target and qualifier ions

Target ion

One target ion must be selected for each compound to be quantified (target compound). Ideally, the target ion is characteristic of the target compound and distinguishes it from other compounds with similar retention times.

Qualifier ions

Qualifier ions are secondary characteristic ions present in the mass spectrum of the target compound. The presence and correct amounts relative to the target ion of these ions support the identification of the correct target compound

Selection of peak and qualifier ions for Dodecane

Examination of the spectrum for Dodecane in Figure 38 on page 81 shows that Dodecane (mw = 170) molecular ion of 170 is present and will be used as the target ion. The 85 ion at half the mw of Dodecane is also significant and will be used as the qualifier ion.

Selection of peak and qualifier ions for the other compounds

Repeat the procedures under "Analyze Data" on page 79 selecting the other compound peaks in our sample and determining the target and qualifier ions for these compounds. Suggested selections are shown in Table 4 and will be used to set up a SIM acquisition and quantitative analysis later.

Compound	Target lon	Qualifier Ion	Dwell time
Biphenyl	154	153	60
Dodecane	170	85	60
Chlorobiphenyl	188	152	60
Methyl Palmitate	270	87	60

Table 4Target and qualifier ion selections

Search the Spectral Library

A library search compares the spectrum of an unknown compound against a library of reference spectra. The search identifies those spectra from the reference library that are most similar to the spectrum of the unknown compound.

You can do a search on an individual peak (spectrum) or on all integrated peaks in the TIC.

Search for an individual spectrum

1 Select a spectrum to search (X in Data Analysis Variable Watch window). See Figure 38 on page 81.



2 Select the Select Library button, _____. The Library Search Parameters dialog box opens.

		\
Library S	earch Parameters	
Search Order	Library Name	Search Next Library If Match Quality <
1		Browse 0
2		Browse 0
3		Browse
	OK Cancel	Help

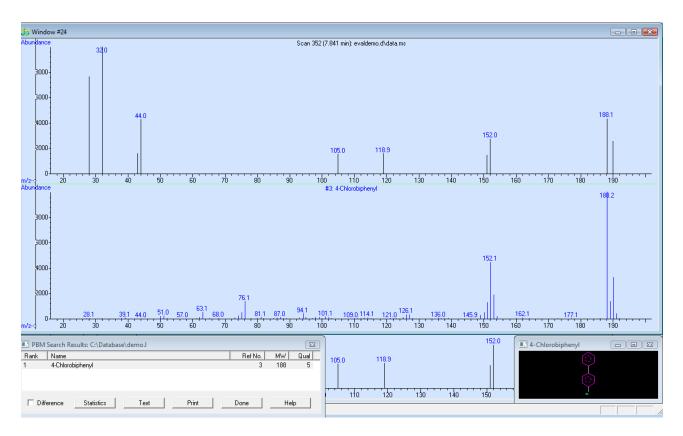
3 Select **Browse** to open the **Browse for Folder** window. Navigate to the demonstration library **demo.l** and select it.

Browse For Folder	x
Select Library	
🧾 Desktop	*
Lisa Hutter	
🛛 🔑 Public	
🛛 🖳 Computer	=
Floppy Disk Drive (A:)	
🛛 🚢 Local Disk (C:)	
b J agilent	
a 🌗 Database	
🔒 demo.l	
Description Descripti Description Description Description Description Descr	
EXCELDIR	
D 퉲 hp	-
Make New Folder OK Cancel	

4 Select **OK**. The file path is entered and this library will be searched first. Use positions 2 and 3 to add any additional libraries you have purchased and installed.

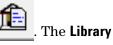
Í	Library S	earch Parameters	
	Search Order	Library Name	Search Next Library If Match Quality <
	1	C:\Database\demo.l	Browse 0
	2		Browse 0
r	3		Browse
5		OK Cancel	Help

- **5** Select **OK** to save selections.
- **6** Double right-click on the spectrum. A search is performed and the results are displayed.



Generate an automated library search report

1 Open the data file.



2 Select the Library Search Report button, Search Report Options dialog box opens.

ibrary Search Report Options		X
Style	Summary	
Destination		
C Screen		
Printer		
🗖 File		
Integration Parameter File	Browse	
1		
Spectrum to Use		
Apex - Start of Peak	•	
	OK Cancel Help	

- **3** From the **Style** drop down list select **Summary**.
- 4 In the destination area, check **Printer**.
- 5 From the Spectrum to Use drop down menu, select Apex- Start of Peak. This selection automatically subtracts the spectrum at the start of the peak from the spectrum at the peak apex which you performed manually in the previous section "Subtract the baseline noise from the spectra" on page 81.
- 6 Select **OK** to generate the report.

C:\ms	sdchem	1\data\evalder	no.a\tmplib	ipion.						
					Library	Search	Repo	ort		
Data	Path	: C:\msdcl	hem\1\dat	ta\						
Data	File	: evaldem	o.d							
Acq (On	: 7 Sep	1989 13	:59						
Opera	ator	: perkins								
Sampl	le	: demosca	n sample							
		: 10 ng p								
ALS (Vial	:1 Sam	ple Mult:	iplier: 1						
C		raries:	0				ы.	nimum Qual	i+	-
seard		raries:	C:\Data	base\demo.	L		LITI	ITHUM QUAT	rty	-
							1111	ттылы қаат	rty	
Unkna	own Sp	ectrum:	Apex mi	nus start	of peak	autoint [.]		ттылы блат	rty	
Unkna	own Sp	ectrum:	Apex mi		of peak	autoint		IIMUM QUAI	ILY	
Unkna	own Sp gratio	ectrum:	Apex min ChemStat	nus start	of peak				-	ual
Unkno Integ k#	own Sp gratio RT	ectrum: n Events: Area%	Apex min ChemStat Lil	nus start tion Integ brary/ID	of peak		1.e		-	
Unkno Integ k#	own Sp gratio RT	ectrum: n Events: Area% 19.44 C:\\	Apex min ChemStat Lin Database'	nus start tion Integ brary/ID	of peak		1.e Ref#	CAS#	Qu	ual
Unkno Integ k#	own Sp gratio RT	ectrum: n Events: Area%	Apex min ChemStat Lin Database'	nus start tion Integ brary/ID	of peak		1.e Ref#		Qu	ual
Unkno Integ k# 1 9	own Sp gratio RT 5.278	ectrum: n Events: Area% 19.44 C:\\	Apex min ChemStat Lil Database' cane	nus start tion Integ brary/ID \demo.l	of peak		1.e Ref#	CAS#	Qu	ual
Unkno Integ k# 1 9	own Sp gratio RT 5.278	ectrum: n Events: Area% 19.44 C:\\ Dode 30.49 C:\\	Apex min ChemStat Lin Database' cane Database'	nus start tion Integ brary/ID \demo.l	of peak		1.e Ref# 1	CAS#	Qu -3 9	ual 96
Unkno Integ k# 1 5	own Sp gratio RT 5.278	ectrum: n Events: Area% 19.44 C:\\ Dode	Apex min ChemStat Lin Database' cane Database'	nus start tion Integ brary/ID \demo.l	of peak		1.e Ref# 1	CAS# 000112-40	Qu -3 9	ual 96
Unkno Integ k# 1 5 2 0	own Sp gratio RT 5.278 6.431	ectrum: n Events: Area% 19.44 C:\\ Dode 30.49 C:\\	Apex min ChemStat Lin Database cane Database enyl	nus start tion Integ brary/ID \demo.l \demo.l	of peak		1.e Ref# 1	CAS# 000112-40	Qu -3 9	ual 96
Unkno Integ k# 1 5 2 0	own Sp gratio RT 5.278 6.431	ectrum: n Events: Area% 19.44 C:\ Dode 30.49 C:\ Biph 26.00 C:\	Apex min ChemStat Lin Database cane Database enyl	nus start (tion Integ brary/ID \demo.l \demo.l	of peak		1.e Ref# 1 2	CAS# 000112-40	Qu -3 9	ual 96 95
Unkno Integ k# 1 9 2 0 3 7	own Sp gratio RT 5.278 6.431 7.737	ectrum: n Events: Area% 19.44 C:\ Dode 30.49 C:\ Biph 26.00 C:\	Apex min ChemStat Lil Database cane Database enyl Database lorobipho	nus start (tion Integ brary/ID \demo.l \demo.l enyl	of peak		1.e Ref# 1 2	CAS# 000112-40 000092-52	Qu -3 9	ual 96 95

Figure 39 The library search report

Print a Window, TIC, Spectrum, or Method

Once you set your printer you can print a window, scan, spectrum, or method for the data file you are viewing on the screen.

Select a printer

- 1 Select File > Select Printer.
- 2 Select printer from the list of printers on your system.
- 3 Select OK.

To change the page orientation

- 1 Select File > Printer Setup.
- 2 Select Orientation.
- 3 Select OK.

Select an item to print

1 Select **File > Print**. The Print dialog box is displayed.

Print	
C Selected Win C TIC & Spectru C Method I Select Printer	um
	OK Cancel

2 Select:

1

- **Selected Window** to print an open window and enter the window number from the window header in the Input dialog box.
- **TIC & Spectrum** to print these graphs.
- **Method** to print the method parameters.
- **Select Printer** to select a printer from the list of printers on your system.
- **3** Select **OK** to print your selection.

dialog box opens.

Save the Data Analysis Method

Г	Ċ)

Select the Save Method button, I . The Save Method As

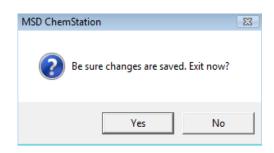
Save Method As	×
Method Path:	
C:\msdchem\1\METHODS\	Browse
Method File :	
default.m	
OK Cancel	Help

2 Enter a name for the method and select **OK** to save the updated parameters to this method.

5 Qualitative Data Analysis

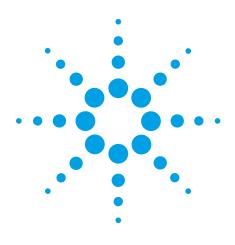
Exit the Data Analysis Program

1 Select **File > Exit**. A warning message appears.



2 Select **Yes** to close the program.

If you have not saved your method, you will lose changes if you click **Yes** to exit now.



6

Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Create a SIM Quantitation Method

Introduction 90 Create a SIM Method 91 Simultaneously Acquire Scan and SIM Data (SIM/Scan Mode) 96 SIM/Scan Mode Cycle Frequency 98

This chapter describes how to create a SIM method for our standard sample using the target and qualifier ions found during qualitative analysis. We also examine how to set up a method that performs simultaneous SIM and scan data acquisition.



Introduction

Selected ion monitoring (SIM) mode is a data acquisition technique where only selected ion fragments are monitored in order to obtain maximum sensitivity.

To find appropriate conditions for the SIM data acquisition, analyze your scan data for:

- **Ions** (*m*/*z*) **monitored for each peak** MS SIM parameters allow you to define up to 100 groups of up to 60 ions each for selected ion monitoring, however, Agilent recommends you use as few ions as possible to maximize the signal to noise ratio.
- The best time to switch groups Agilent recommends that you choose a time to switch groups where the peaks are well separated to avoid variations in retention time due to sample matrix effects.

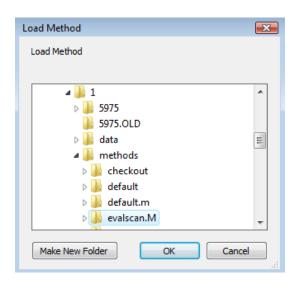
Create a SIM Method

1 From the **Instrument View**, select the **Load Method** button, the **Load Method** dialog box opens.



2 Navigate to and select evalscan.M.

Since the GC acquisition parameters in this method were set for good chromatographic data resolution, use this method as a starting point and only change the MS parameters in the method.



3 Select **OK** to load the method and close the dialog box.



4 Select the MS Parameters button, Scan Parameters dialog box opens.

MS SIM/Scan Parameters	
MS Instrument Sample Inlet: GC	Real-Time Plot Time Window: 10 min.
Solvent Delay: 3.00 min. EMV Mode: Relative ▼ Relative Voltage: 0 = 1200 V Acq. Mode: SIM	MS Window 1 Plot Type: Total Y-Scale: 0 to 2000000 MS Window 2 Plot Type: None Y-Scale: 0 to 100000
Tune Fileatune.u	
SIM Parameters Zones	Timed Events Help

5 From the Acq. Mode dropdown box, select SIM.

- 6 Select SIM Parameters. The Edit SIM Parameters dialog box opens. See Figure 40.
- 7 In the **Group** field, enter 1. Group 1 appears in the right panel table.
- 8 For **Resolution**, select **High**.
- **9** In the **Edit lon** area enter the values for all 4 ions in the group 1 ions time segment.
 - a In the *m/z* and **Dwell** fields enter the ion values for these compounds from Table 5 on page 93.
 - **b** After each ion addition, select **Add/Modify lon**.

Group 1 Start Time: 3.00	Resol	ution: C Low I High s/Sec = 3.80	Group 1	Start Time 3.00	# of lons
Edit Ion		✓ Plot this Ion	∢ Add New	III • Group D	elete Group
m/z 85.00 153.00 154.00 170.00	Dwell 60 60 60 60 00 Delete lon(s)	Plot YES YES	the at Group then s left. single	lit a Group, just se bove list box. To b, click "Add New start editting the fi To delete a Grou e or multiple select	add a /Group'', elds on the p(s), pick a ion from the
955			list ab Group	ove , then press b(s)''.	"Delete

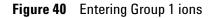


Table 5SIM ion selection

Compound	Target lon	Qualifier Ion	Dwell time
Biphenyl	154	85	60
Dodecane	170	85	60
Chlorobiphenyl	188	152	60
Methyl pamitate	270	87	60

- Select Close to save settings and return to the MS SIM / Scan Parameters dialog box.
- 11 Select **OK**.

|--|

- . The Save Method As
- 12 Select the **Save Method** button, dialog box opens.
- 13~ In the Method File field, enter demosim and select OK.

Save Method As		×
Method Path:		
C:\msdchem\1\methods		Browse
Method File :		
DEMOSIM.M		
ОК	Cancel	Help



- 14 Select the Edit Entire Method... button, Method dialog box opens.
- 15 Mark the Method Information check box only. Clear the Data Analysis and Instrument/Acquisition check boxes.

Edit Method			×	
Method Sections to Ed	dit:			
🔽 Met	hod Information			
Instrument / Acquisition				
🗆 Dat	a Analysis			
ОК	Cancel	Help		

- 16 Select **OK**. The **Method Information** dialog box opens.
- **17** In the **Method Comments** field, enter a description of this method.
- 18 In the Method Sections To Run area, mark the Data Acquisition check box.

Method Information	×
Method Comments:	
This is the SIM method for 5975 MSD Sample (P/N 05970-60045)	
✓ Save Copy of Method with Data.	
Method Sections to Run	
Pre-Run Macros/Commands:	
Instrument Control:	
Data Analysis:	
✓ Data Acquisition	
🗖 Data Analysis	
Post-Run Macros/Commands:	
Instrument Control:	
Data Analysis:	
OK Cancel Help	

19 Select **OK**. The **Save Method As** dialog box opens.

Save Method As	×
Method Path:	
C:\msdchem\1\methods	Browse
Method File :	
DEMOSIM.M	
OK Cancel	Help

20~ Confirm that demosim is entered in the Method File field and select 0K.

Simultaneously Acquire Scan and SIM Data (SIM/Scan Mode)

If we start with a method containing Scan parameters and then also enter SIM parameters like we did for the evalsim.m method, our method already contains all parameters required except one. We only need to check a box that specifies that we want to acquire both types of data simultaneously.

In SIM/Scan mode the number of data points taken in each mode is reduced and we will see how that impacts the total cycle frequency.



- 1 Select the **MS Parameters** button, **Scan Parameters** dialog box opens.
- . The MS SIM /
- 2 Mark the Acquire Scan and SIM data check box.
- 3 From the Acq. Mode dropdown box, select Scan.

MS SIM/Scan Parameters	×
MS Instrument	Real-Time Plot
Sample Inlet: GC	Time Window: 10 min.
	MS Window 1
Solvent Delay: 3.00 min.	Plot Type: Total
EMV Mode: Gain Factor 💌	Y-Scale: 0 to 2000000
Gain Factor: 1.00 = 1471 V	
Acq. Mode: Scan	MS Window 2
	Plot Type: None
	Y-Scale: 0 to 100000
Acquire both Scan and SIM data: 🔽	
Tune File	
atune.u	
<u> </u>	
Scan Parameters Zones	Timed Events
OK	Help

4 Select **Scan Parameters**. The **Edit Scan Parameters** dialog box opens and we can view our previous settings.

canning Mass	Range Thres	hold and Samplin	g Rates Plotting			
			rt Time Start at inutes) Mass (an	Endat hu) Mass (amu)		
	Scan Gr	roup 1 🗹 📔	3.00 50.0	10 <u>350.00</u>		
	Scan Gr	roup 2 🗖 📔				
	Scan bi	roup 3 🗖 📔				
Summary		roup 3	J			
Summary of Group		Low Mass	 High Mass	Threshold	Samples	s
	of Settings	,	High Mass 350.00	Threshold 40	Samples 3	<u>s</u> 2.
Group	of Settings	Low Mass				
Group 1	of Settings	Low Mass 50.00		40	3	2.

5 Select the Mass Range tab and note the asterisk.

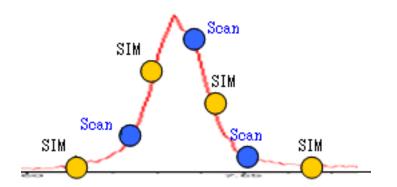
The asterisk in the **Summary Of Settings** table, **(Scans/Sec*)** denotes that the **Scans/Sec** displayed here does not represent the actual cycles. See "SIM/Scan Mode Cycle Frequency" on page 98, for more information.

- **6** Write down the cycle frequency for the scan mode.
- 7 Select **Close** to return to the **MS SIM/Scan Parameters** dialog box.
- 8 From the Acq. Mode dropdown box, select SIM.
- **9** Select **SIM Parameters**. The **Edit SIM Parameters** dialog box opens where we can view our previous settings.
- **10** Select the **Mass Range** tab and write down the cycle frequency for the SIM mode.
- 11 Select **Close** to return to the **MS SIM/Scan Parameters** dialog box.
- 12 Select **OK** to save the parameters and close the dialog box.
- **13** Save the method with the name **sim_scan.M**.

The individual cycle frequencies recorded here will be used to calculate the actual cycle frequency in the next section "SIM/Scan Mode Cycle Frequency" on page 98.

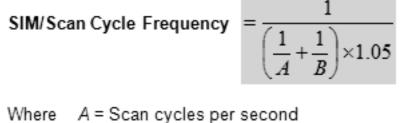
SIM/Scan Mode Cycle Frequency

In SIM/Scan mode, to complete one cycle the MSD acquires a single group of SIM data followed by a single group of Scan data. It may be necessary to increase the Scan speed or decrease the SIM dwell time to achieve the desired number of data points for effective chromatographic integration. See Figure 41.





Actual cycle frequency is calculated with the equation in Figure 42.

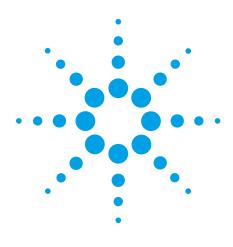


Where A = Scan cycles per second B = SIM cycles per second

Figure 42 SIM/Scan cycle

When switching from the SIM data acquisition mode to the Scan mode, about 5% of the available run time will be consumed.

For our example, Scan = 2.44 cycles/sec and SIM = 1.97 which results in an actual cycle time of 1.04 cycles/sec. To improve the number of data points, we could reduce the SIM dwell time, and increase the scan speed.



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Run a Sequence

7

Prepare the Samples100Create the Sequence101Save the Sequence103Load the Sequence104Run the Sequence105Print the Sequence106

This chapter describes how to create and run a sequence.

A sequence is a list of samples to be analyzed and a designated method to be used for each analysis. Once defined, the sequence may run unattended, automatically processing the samples defined in the sequence.

When an ALS is installed, the entire analysis, from injection of the sample through reporting of results, can be automated to save you time.

The data files generated when running this sequence will be used later for developing a quantitate analysis.



Prepare the Samples

- Prepare 1:2 serial dilutions of the 100 ng/mL 5975 MSD Sample (P/N 05970-60045 or P/N 5074-3025 Japan only) in hexane to make a 50 ng/mL and a 25 ng/mL method calibration sample.
- 2 Prepare 1:2 serial dilutions of the 10 ng/mL 5975 MSD Sample (P/N 05970-60045 or P/N 5074-3025 Japan only) in hexane to make a 5 ng/mL and 2.5 ng/mL method calibration sample.
- 3 Fill the vials with approximately 500 μ L of each standard (2.5, 5, 10, 25, and 50 ng/mL).

If you are not using an ALS skip the remaining steps.

- **4** Place the sample vials in increasing order of concentration into positions 1 through 5 of the GC sample tray.
- **5** Fill a solvent wash vial with isooctane and place it in injector turret location A for solvent wash mode A, B.
- **6** Place an empty waste vial in turret location B specified for solvent wash mode A, B.

Create the Sequence

1 Select the Edit Sequence button,



. The Sample Log

Table opens.

- 2 In sample row 1 under the **Type** column, click in the cell to activate the dropdown list, and select **Sample**.
- **3** Under the **Vial** column, enter 1 if you placed the lowest concentration sample in the ALS tray position 1.
- 4 Under the Sample column, enter Standard 5 ng/mL.
- **5** Under the **Method/Keyword** column:
 - a Right mouse click and select **Browse for Method**. The **Browse for Folder** dialog box opens.
 - **b** Navigate to and select **demoSIM**.
 - c Select **OK**. The method name appears in the column.
- 6 In the Data File column, enter STD01.

Data Pa	ath: C:\msdchem\1\DAT	A	Brov	vse Method Path:	C:\msdchem\1\METHODS	Browse.
	Туре	Vial	Sample	Method / Keyword	Data File	Co Keyv
1	Sample 🚽	1	Standard 5 ng/mL	demoSIM	STD01	
2	Sample	1	Sample 2	DEFAULT		
3	Sample	1	Sample 3	DEFAULT		
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16 17						
						•

7 Highlight rows 1 to 5.

8 Right mouse click and select **Repeat Row & increment**. Four lines are added to the table with incremented vial number and data file names.

	,	0110111121	DATA	Brow	se	Method C:\MSDCH	EM\2\METHODS
4	Туре	Vial	Sample	Method / Keyword	Data File	Comment / KeywordString	Multiplier
1	Sample 🖵	1	Standard 5ng/mL	demoSIM	STD01		1.00000
2	Sample	2	Standard 5ng/mL	demoSIM	STD02		1.00000
3	Sample	3	Standard 5ng/mL	demoSIM	STD03		1.00000
4	Sample	4	Standard 5ng/mL	demoSIM	STD04		1.00000
5	Sample	5	Standard 5ng/mL	demoSIM	STD05		1.00000

- 9 In row 1, under Sample column, change the value to 2.5 ng/mL.
- 10 In row 3, under Sample column, change the value to 10 ng/mL.
- 11 In row 4, under Sample column, change the value to 25 ng/mL.
- 12 In row 5, under Sample column, change the value to 50 ng/mL.

					,	
Туре	Vial	Sample	Method / Keyword	Data File	Comment / KeywordString	Multiplier
Sample 🖵	1	Standard 2.5ng/mL	demoSIM	STD01		1.00000
Sample	2	Standard 5ng/mL	demoSIM	STD02		1.00000
Sample	3	Standard 10ng/mL	demoSIM	STD03		1.00000
Sample	4	Standard 25ng/mL	demoSIM	STD04		1.00000
Sample	5	Standard 50ng/mL	demoSIM	STD05		1.00000
		_				
	Sample 🖵 Sample Sample Sample	Sample 🗨 1 Sample 2 Sample 3 Sample 4	Sample Texture 1 Standard 2.5ng/mL Sample 2 Standard 5ng/mL Sample 3 Standard 10ng/mL Sample 4 Standard 25ng/mL	Type Vial Sample Keyword Sample 1 Standard 2.5ng/mL demoSIM Sample 2 Standard 5ng/mL demoSIM Sample 3 Standard 10ng/mL demoSIM Sample 3 Standard 25ng/mL demoSIM Sample 4 Standard 25ng/mL demoSIM	Type Vial Sample Keyword File Sample 1 Standard 2.5ng/mL demoSIM STD01 Sample 2 Standard 5ng/mL demoSIM STD02 Sample 3 Standard 10ng/mL demoSIM STD03 Sample 4 Standard 25ng/mL demoSIM STD04	Type Vial Sample Keyword File KeywordString Sample 1 Standard 2.5ng/mL demoSIM STD01 Sample 2 Standard 5ng/mL demoSIM STD02 Sample 3 Standard 10ng/mL demoSIM STD03 Sample 4 Standard 25ng/mL demoSIM STD04

13 Select OK to close the Sample Log Table.

Save the Sequence



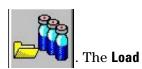
1 Select the Save Sequence As... button, Sequence dialog box opens. L. The Save

2 In the File name field, enter eval.

Save Sequence					<u>?</u> ×
Save in:	🔁 sequence		•	+ 🗈 💣 🎟	
My Recent Documents Desktop My Documents My Computer	default.s HOT_SHOT_re HOT_SHOT_re HOT_SHOT_re HOT_SHOT_re HOT_SHOT_re RSD only.s RSD with EI Of RSD with EI of SSD sp_01.s	v18.s v19.s v19_no_bake.s TN Runs.s			
My Network	File name:	eval		•	Save
Places	Save as type:	Sequence Files (*.s)		•	Cancel

3 Select **Save**. The dialog box closes and the sequence is saved.

Load the Sequence



- 1 Select the Load Sequence button, Sequence dialog box opens.
- 2 In the File Name field, enter eval.s.

Load Sequenc	e				<u>? ×</u>
Look in: ն	sequence	•	+ E	d 🏹	•
default.s eval.s HOT_SHOT HOT_SHOT HOT_SHOT HOT_SHOT		EI OFN single E		n.s	
File name:	eval.s		•	Se	lect
Files of type:	Custom (*.S)		•	Ca	ncel
	Dpen as read-only				

3 Click **Select** to close the dialog box and load the sequence.

Run the Sequence



1 Select the **Run Sequence** button, **Sequence** dialog box opens. . The Start

- 2 In the Method Sections to Run area, select Full Method.
- **3** In the **Sequence Comment** field, enter a description of the sequence.
- 4 In the **Operator Name** field, enter your name.
- 5 In the Data File Directory field, add demosim to the path.
- 6 Select Run Sequence.

	28 02:27:44 2011
Method Sections to Run	Sequence Barcode Options
Full Method	C Disable Barcode for This Sequence
C Reprocessing Only	On Mismatch – Inject Anyway, Continue Sequence
	C On Mismatch – Don't Inject, Continue Sequence
Overwrite Existing Data Files	
Sequence Comment SIM Aquis	tion
Operator Name: John Smit	h
Data File Directory: C\msdch	em\1\DATA\demoSIM Browse
Data i ne Directory. <u>Jet</u> insden	
Pre-Seq Macros/Commands	
Pre-Sed Macros/Commands	
Instrument Control:	
Data Analysis:	
Daia Analysis.	
Post-Seq Macros/Commands	
Instrument Control:	
instanient control.	
Data Analysis:	
Data Analysis:	
Data Analysis:	OK Cancel Help

The **Sequence Status** bar is displayed. During the sequence run, you can monitor the number of the samples run, the number of samples remaining, and the current sample vial being processed. Use the controls on the bar to pause the sequence, access data analysis, or edit sequence sample entries that have not yet run.

Sequence Status: SIM demo			
Running 1 of 5 Vial 2	D:\MSDCHEM\2\DATA\demosim\DEMOSIM01.D	▼ Edit Data Analysis Pause	

Figure 43 The sequence status bar

Print the Sequence Log



The Select Items to Print

2 Mark the Sequence Log checkbox.

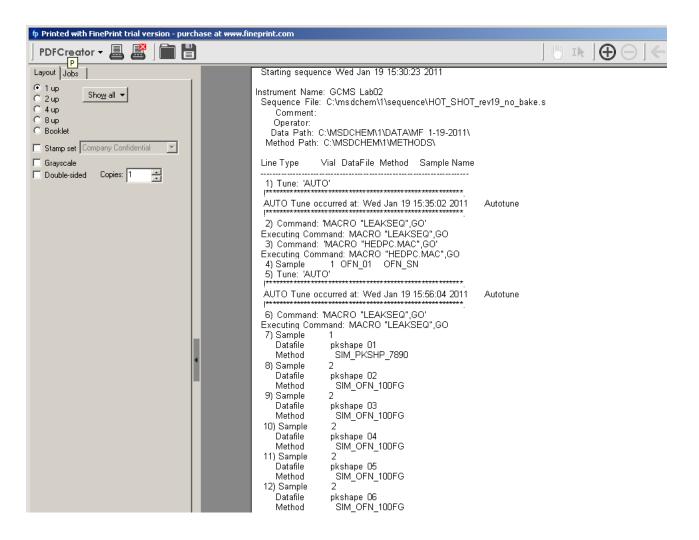
Select the **Print** button,

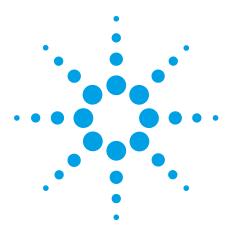
dialog box opens.

1

Select Items to print
Sequence Log
Current Sequence
Instrument Parameters
🔲 Data Analysis Parameters
🔲 Detailed Data Analysis Parameters
OK Cancel

3 Select **OK**. The **Sequence log** is displayed for printing.





8

Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Set Up a Quantitation Database

Add Compound Entries for the Database 108 Add the Calibration Curve 115 View or Edit an Existing Database 120

This chapter describes how to add compounds to the database. After a compound is identified, quantitative data analysis determines the amount of the compound in your sample by comparing the response from an unknown amount of compound with the response from a known measured amount of the compound stored in the quantitation database.



Add Compound Entries for the Database

1 Start the Enhanced Data Analysis program.



2 Select the Load Method button, . A confirmation message dialog box may open. If so, select Yes. The Load Method window opens.

Load Method	×
Load Method	
🔺 🌗 msdchem	~
▲ <u>]]</u> 1	
⊳ 🍌 5973N	
Þ 퉲 5975	_
> 🌗 5975.OLD	=
🛛 🐌 data	
4 🍌 methods	
Deckout	
Image: Provide the second s	
default.m	
b b demoscan.M	
demoSIM.M	
sequence	-
Make New Folder OK Cancel	

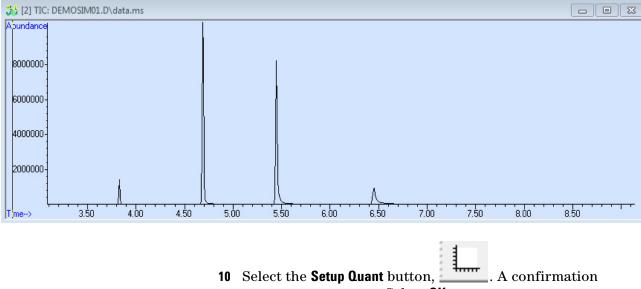
- **3** Select the demosim method and click **OK**.
- 4 Select the Load Data File button, dialog box opens.
- 5 Select Change Path. The Browse for Folder window opens.
- 6 Navigate to and select C:\msdchem\1\data\demosim.
- 7 Select **OK**. The path is displayed in the **Path** field.

Select Data File			
Path: C:\msdchem\2\data\demosim Change Path DEMDSIM01.D DEMDSIM02.D DEMDSIM02.D DEMDSIM02.D		Operator: Vial: Misc Info: Sample Name:	twi 6 50 ng/uL Standard A 50 ng/uL
DEMOSIM04.D DEMOSIM05.D		Abundance 1e+07	
	OK	Cancel	Help

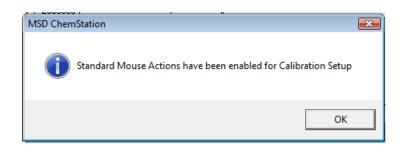
8 From the list of files, select **DEMOSIM01.D**.

Later we will use the load next file function. It remembers this data directory and the last file selected from it and automatically loads the next data file with the click of an icon.

9 Select **OK**. The **TIC** window opens.



message may appear. Select **OK**.



- 11 Select **OK**. The standard right mouse buttons are enabled.
- **12** The **Quantitation Database Globals** dialog box opens.

ç	uantitation Database Globals					
	Calibration Title					
	MSD Sample					
	Locating Peaks					
	Reference Window	2.000	Minu	tes	•	
	Non-Reference Window	1.000	Minu	tes	•	
	Correlation Window	0.100	minute	BS		
	(signal-to-signal retention time m	natch)			🔽 Usel	RTEINT
	New Compound Info					
	Integration Parameter File					Browse
	Measure	Area	•			
	Default +/-	0.500	min arour	nd exp RT		
	Curve Fit		Linear Re	egression		•
	Data point weight for linear regre	ssions		Equal	weighting	•
	Units of concentration	ng/uL				
	ISTD concentration	0.000000				
		ОК	Cancel		Help	

- **13** Enter the following information to set parameters that will initially be set for all compounds in this database. If some compounds need different parameters they can be changed later in the database.
 - **a** Calibration Title-MSD Sample.
 - **b** Units of Concentration ng/uL
 - **c** Select **Use RTEINT**. The RTE integrator is recommended for MS data.

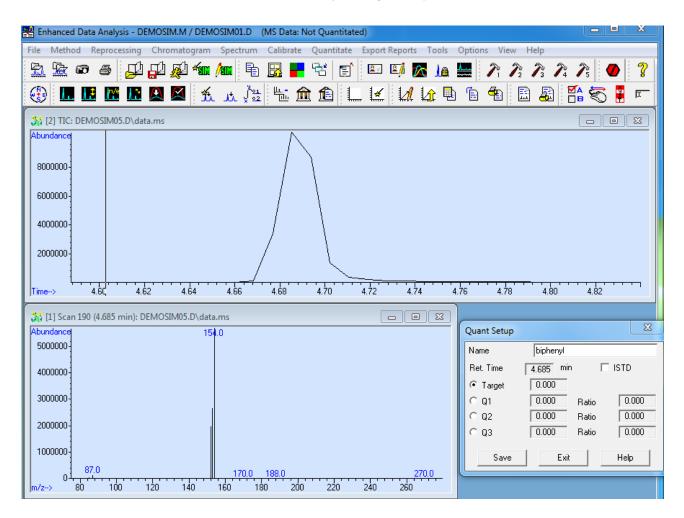
14 Select **OK** to save the settings and open the **Edit Compounds** dialog box.

ſ	Edit Compou	nds				×		
	Index	Ret. Time	Signa	al Compound Name				
		ID LIST]						
	• ∟					•		
	* before Compound Name denotes ISTD							
		Insert Above		Exit	Help			
			-					

Identify compounds

The first part of setting up a Quantitation database is identifying and naming the compounds by selecting target and qualifier ions from a known sample.

1 From the Edit Compounds dialog box, select Insert Above. The Quant Setup dialog box opens.



- 2 In the name field, enter the first compound name, biphenyl.
- **3** In the TIC window, enlarge the biphenyl peak (near RT 4.7).
- 4 Position the cursor at the highest point of the peak and double right mouse click. The RT is added to the Ret. Time field. The Scan is displayed in the lower window and the RT is displayed for the Ret. Time in the Quant Setup dialog box.

Target is selected in the Quant Setup dialog box.

5 In the scan window, position the bulls eye cursor on the target ion (154) and click both mouse buttons simultaneously. The m/z is displayed for the Target.

Q1 is selected in the **Quant Setup** dialog box.

6 In the scan window, position the cursor on the first qualifier ion (153) and click both mouse buttons simultaneously. The m/z is added to the **Q1** field and the ratio is calculated and added to the **Ratio** field.

Quant Setup	X
Name	biphenyl
Ret. Time	4.685 min 🗌 ISTD
C Target	154.000
C Q1	153.000 Ratio 46.734
€ Q2	0.000 Ratio 0.000
C Q3	0.000 Ratio 0.000
Save	Exit Help

To clear an incorrect ion selection, select the radio button for that ion. Next, simultaneously click both mouse buttons with the cursor positioned on an area not containing an ion.

- 7 Select **Save** to add the biphenyl peak to the database and clear the **Quant Setup** dialog box.
- 8 Add the remaining compounds using the target and qualifier ions identified in qualitative analysis.

Compound	Target lon	Qualifier lon	Dwell time
Biphenyl	154	153	60
Dodecane	170	85	60
Chlorobiphenyl	188	152	60
Methyl pamitate	270	87	60

Table 6Target and qualifier ion selections

- 9 When all compounds are added, select Exit to return to the Edit Compounds dialog box.
- **10** Review the compound list. If any corrections need to be made, double-click on the compound and reenter the information in the **Quant Setup** dialog box.

Edit Compour	nds			×
Index	Ret. Time	Signal	Compound Name	
1 2 3	3.831 5.472 6.542	170.00 188.00 270.00	dodecane chlorobiphenyl methylpamitate	
4	4.685	154.00	biphenyl [END OF COMPOU	
•				- F
* before Comp	oound Name denotes ISTD		Exit	Help

11 Select Exit. A confirmation message appears.

MSD Chen	nStation 🛛 🕅	Ĵ
?	File has not been quantitated. Quantitate now?	
	Yes No	

This procedure continues with the next section "Add the Calibration Curve" on page 115.

Add the Calibration Curve

The second part of setting up a Quantitation database is entering the compound concentrations from a group of samples. Each sample in the group contains a different compound concentration used to create the calibration curve.

Add calibrator level 1

1 Select **Yes** to the confirmation message that appears in step 11 of "Identify compounds" above. The **Update Calibration** dialog box opens.

Upd	ate Calibration	_	-		23				
	Calibration Data File (Selection ignored by Seq			_					
	C:\msdchem\2\data\demosim\DEMOSIM01.	D							
	 Add Level (supply new Calibration Level I 	D)		Level IDs-					
	Compound Concentration:	2.500000		New Level IE	>				
	ISTD Concentration:	0.000000		2.5					
				Existing Level	ID				
					-				
	O Update Level (select existing Calibration L	evel ID)							
	Responses	C Average	C Replace						
	Retention Times	C Average	C Replace						
	🔲 Replace Qualifier Ion Relative Re	sponses							
	🔲 Update Mass Assignments								
	O Delete Level (select existing Calibration Le								
	C Delete Level (select existing Calibration Le	wend)							
	Do Update	e Cancel	Help						

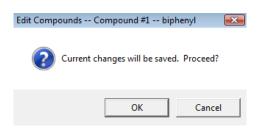
- **2** For the first calibrator,
 - a Select Add Level.
 - **b** Compound Concentration enter 2.500000.
 - c In the Level IDs area, enter 2.5 in the New Level ID field.
- **3** Select **Do Update**. The **Edit Compounds** dialog box opens and displays the first calibration point.

8 Set Up a Quantitation Database

Search by: 📀 Ret Time	e 🔿 Name	C Index		Find Compound	
Compound Database	Identification Calibration	Jser-Defined Advanced Rep	porting		
External Standard Compour dodecane chlorobiphenyl	Name biphenyl		Concentration Units ng/uL	Compound Type	
methylpamitate	Signals to Be Used for Qu Ret Time 4.685	RRT 0.000	Quantitation Options		
opnenyn	Extract signals from	RRT [0.000	Quantitation type	Target compound	
	-	0.500 © Min © %	Sample ISTD Concentration	0.000000	
	1	to 5.185 minutes	Measure response by	Area	
		-	Identify by	All Hits	
	Quant signal Target	Ion V Wuncertainty	Maximum number of hits	9	
	m/z Relative Re	sponse Rel 💌	Subtraction Method	Avg first & last	
	Target 154.00	100.00	Curve Fit	Linear Regression	
	· · · · · · · · · · · · · · · · · · ·	46.70 20.00	Weight	Equal weighting	
		0.00 20.00		lah ang d	
		0.00 20.00	Response	iphenyl	
	Q3 0.00	,	4.00e+005- 2.00e+005- 0	1 2 Concentration	

Figure 44 The first calibration point is added to the calibration curve

- 4 Select the **Identification** tab.
- **5** In the **Quantitation Options** area, select:
 - a Identify by All Hits
 - b Subtraction Method Avg first & last
- 6 Select **OK**. A confirmation message appears.



7 Select **OK** to save the changes.

The Quantitation Report window opens. To add additional calibration levels, see the next section.

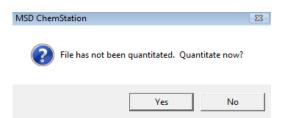
Add calibrator levels 5, 10, 25, and 50 to the calibration curve

Repeat this procedure for DEMOSIM02 (level = 5), DEMOSIM03 (level = 10), DEMOSIM04 (level = 25), and DEMOSIM05 (level = 50).

- 1 Select the **Load Next Data File** button, . The next data file is automatically loaded.
- Select the Update Calibration button, _____. The Select Update Option dialog box opens.

Select Update Option					
• Update One Level					
O Quick Levels Update					
O Global Update					
OK Cancel					

3 Select **Update One Level** and **OK**. A confirmation message appears.



- 4 Select Yes. The Update Calibration dialog box opens.
- **5** For this calibrator,
 - a Select Add Level.
 - b Compound Concentration for DEMOSIM02 (level = 5), DEMOSIM03 (level = 10), DEMOSIM04 (level = 25), and DEMOSIM05 (level = 50).
 - c In the Level IDs area, enter for DEMOSIM02 (level = 5), DEMOSIM03 (level = 10), DEMOSIM04 (level = 25), and DEMOSIM05 (level = 50) the New Level ID field.

- **6** Select **Do Update**. The **Edit Compounds** dialog box opens and displays the new calibration point.
- 7 Select the **Identification** tab.
- 8 In the **Quantitation** area, select:
 - a Identify by All Hits
 - **b** Subtraction Method Avg first & last
- 9 Continue with the above steps under "Add calibrator levels 5, 10, 25, and 50 to the calibration curve" until all concentration levels are added. The completed calibration curve is shown in Figure 45.

Edit Compounds Compound #1	dodecane	X
Edit Compounds Compound #1 Search by: Compound Database E- Compound Database Compound Com	ne C Name C Index Identification Calibration User-Defined Advanced Re Name dodecane Signals to Be Used for Quantitation Ret Time 3.831 RRT Extract signals from - 0.500 + 0.500 • Min C % This is 3.331 to 4.331 minutes	Find Compound
	Quant signal Target Ion % Uncertainty m/z Relative Response Rel Target 170.00 100.00 Q1 85.00 1453.10 20.00 Q2 0.00 0.00 20.00 Q3 0.00 0.00 20.00 Level Concentration Response 4	Maximum number of hits 9 Subtraction Method Avg first & last Curve Fit Linear Regression Weight Equal weighting dodecane Response
< Þ	Level Concentration 1357.00000 5 5.00000 1357.000000 10 10.00000 50554.000000 25 25.00000 110707.000000 50 50.00000 181745.000000	
	OK Cancel Help	Print Calibration Curve Copy Calibration Curve

Figure 45 The completed quant database

10 Select **OK** to close the window.

Save the database

- 1 Select the Save Method button, . The Save Method As dialog box opens with the name of the current method displayed in the Method Path and Method File fields.
- 2 Select **OK**.

View or Edit an Existing Database

- 1 Select the Edit Compounds button, _____. The Edit Compounds dialog box opens.
- 2 Select a compound in the navigation tree. The corresponding information is displayed in each tab.
- **3** To copy the calibration curve to your clipboard for use in another application, select **Copy Calibration Curve**.
- 4 To print the calibration curve, select **Print Calibration Curve**.

Identification tab

- Name of the compound
- Concentration units
- Compound type
- Retention time information
- Signals to be used for quantitation
- Calibration information
- Quantitation parameters

Calibration tab

- Concentration units
- Response for each level ID

User-Defined tab

- A1 through A3 alphanumeric items with a maximum of 19 characters
- N5 through N9 numeric items

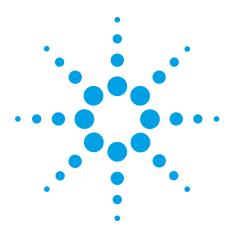
Advanced tab

- Area Correction Mass
- Correction Factor
- Integration parameter files for target and qualifier compound quantitation. The **Sum?** field allows you to add the response of the designated qualifier ion to the response of the target ion. This method is valid only in area quantitation using the extended area quantitation method.

Reporting tab

- CAS # designed for a Chemical Abstract Service number. However, you may use this for any other number or information about the compound.
- Surrogate / Matrix Spike Amount
- Matrix A and B concentrations
- Signal level minimum and maximum
- MS database name
- Reference Spectrum number

8 Set Up a Quantitation Database



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Generate a Report

9

Generate a Report Automatically After the Run 124 Generate a Detailed Report for Previously Acquired Data 129

This chapter explains how to modify your method to generate a report at the end of each sample run and how to interactively generate a report from the **Data Analysis** view.



Generate a Report Automatically After the Run

Load the method

1 From the Instrument View, select the Load Method button,



. The **Load Method** window opens.

- 2 Navigate to and select **demosim.m**.
- **3** Select **OK** to close the dialog box and load the method.

Edit the method to generate a report

1 From the Instrument View, select the Edit Entire Method...

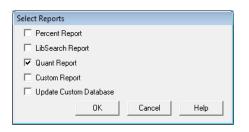


Edit Method	×							
Method Sections to Edit:								
Method Information								
Instrument / Acquisition								
🔽 Data Analysis								
OK Cancel Help								

- 2 Mark the **Method Information** and **Data Analysis** check boxes only. Clear the **Instrument/Acquisition** check box.
- **3** Select **OK**. The **Method Information** dialog box opens.

Method Information	×
Method Comments:	
This is the SIM method for 5975 MSD sample	
Save Copy of Method with Data	
Method Sections to Run	
Pre-Run Macros/Commands:	
Instrument Control:	
Data Analysis:	
✓ Data Acquisition	
🔽 Data Analysis	
Post-Run Macros/Commands:	
Instrument Control:	
Data Analysis:	
OK Cancel Help	

- 4 In the **Method Comments** field, enter a description of this method.
- 5 In the Method Sections To Run area, mark the Data Acquisition and Data Analysis check boxes, and clear the Post-Run Macro/Commands check box.
- 6 Select **OK**. The **Select Reports** dialog box opens.



- 7 Mark the **Quant Report** check box and clear all other check boxes.
- 8 Select **OK**. The **Quant Report Options** dialog box opens.

Quant Repo	ort Options	×
Style	Summary	•
_ Destinatio	n	
C Scree	en	
🔽 Printe	a	
🗖 File		
	OK Cancel Help	

- **9** From the **Style** drop down list, select **Summary**.
- **10** In the **Destination** area, mark the **Printer** check box and clear all the other check boxes.
- 11 Select OK. The Select RUNMETHOD printer dialog box opens.

Select RUNMETHOD printer
Microsoft XPS Document Writer HP Color LaserJet 4700 Adobe PDF
Select Cancel Help

- **12** Select a printer and click **Select**. The **Save Method As** dialog box opens.
- **13** Select **OK** to save the setting to the current method or enter a new file name for the method.

Run the method and generate the report

 With the method modified to print a quantitation summary report loaded, click on the green arrow to display the Start Run dialog box.

Basic Advanced Current Method Injection Style: GCALS	
Inlet Location Front C Rear C Dual Operator Name:	MS Connected to Science International Intern
Data Path: C:\MSDCHEM\1\DATA\EVAL1	Rear Inlet
Data File Name: EVALUNKN.D Browse Sample Name: Demo QReport Misc Info:	Data File Name: EVALDEMO.D Browse Sample Name:
Sample Amount: 0 Multiplier: 1	Sample Amount: Image: Description of the second
Vial Number: 1 Tray Name: Agilent ALS Select Injection Volume: ▼ Current Method □ µL 	Vial Number: Tray Name: Agilent ALS Select Injection Volume: © Current Method 0 μL
Ο Override using 0 μL	O Override using
Method Sections to Run	fethod Exit Cancel Help

- 2 In the Data Path field, add eval1 to the path.
- 3 In the Data File Name field, enter evalunkn.d.
- 4 In the **Operator Name** field, enter your name.
- 5 In the **Sample Name** field, enter a sample name.
- 6 Enter the **Vial** number for your sample location in the ALS.
- 7 In the Method Sections to Run area, select Data Acquisition and Data Analysis.

8 Select **OK and Run Method**. The method is run and the summary quantitation report is automatically generated after the run is completed.

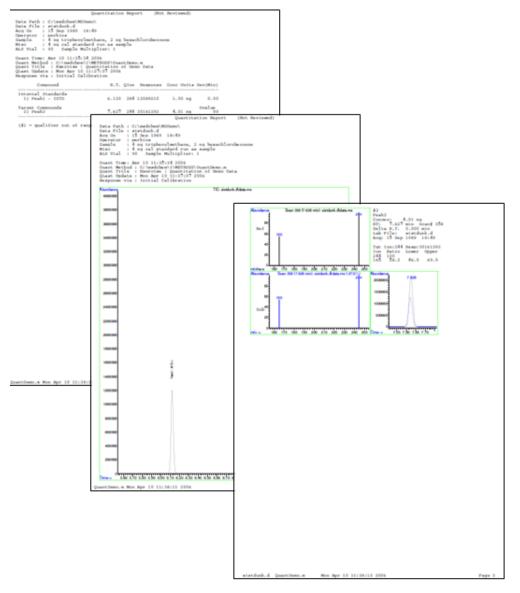


Figure 46 Summary quantitation report

Generate a Detailed Report for Previously Acquired Data

Load the method

1 Start the data analysis program by using the desktop icon,



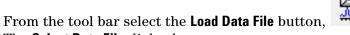
From the Instrument View, select the Load Method button. 2



The Load Method dialog box opens.

3 Navigate to and select **demosim**.**M** and then **OK**.

Load the data file



- 1 The Select Data File dialog box.
- From the list, select evalunkn.d. 2
- 3 In the **Path** field, enter C:\msdchem\1\DATA\eval1.
- Select **OK** to load the file and close the dialog box. 4

Generate a detailed quantitation report

Select the Generate Reports button, . The Quant Reports 1 Options dialog box opens.

Quant Repor	t Options			X
Style	Detailed			•
Destination				
C Screen				
Printer				
File File				
	ок	Cancel	Help	

- From the Style drop down list, select Detailed. 2
- In the **Destination** area, mark the **Printer** check box and clear 3 all the other check boxes.
- 4 Select **OK**. The dialog box closes and the report is printed.

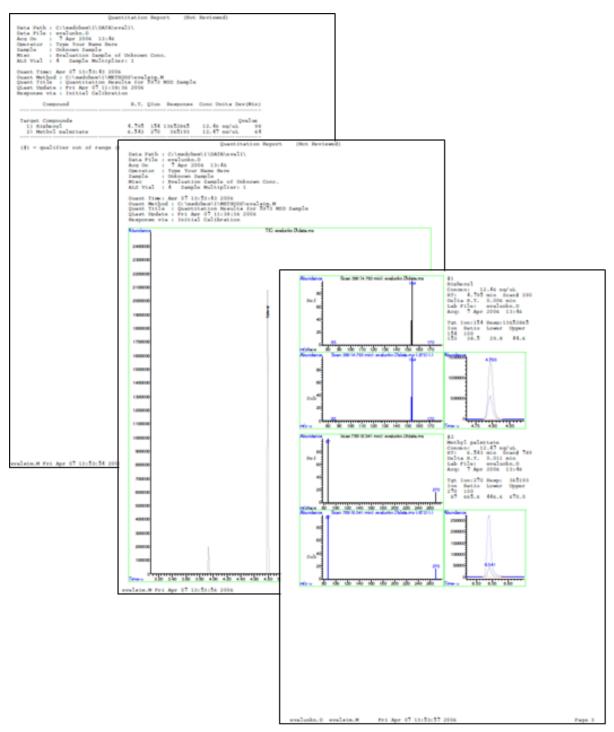
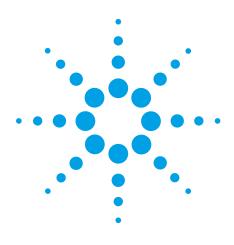


Figure 47 Detailed quantitation report



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

10 Recalibrate and Quantitate Unknowns

Create a Recalibration Sequence 132 Save the Sequence 134 Run the Sequence 135

Regular recalibration is required to account for changes in your system. The ChemStation can perform this recalibration automatically using the recalibration sequence described here. This is normally done on a scheduled basis that precedes the running of samples.



Create a Recalibration Sequence

1 Select the **Edit Sequence** button,



The Sample Log

Table opens.

- 2 In sample row 1 under the **Type** column, click in the cell to activate the drop down list and select **Calibration**.
- **3** Under the **Vial** column, enter 1 if you place the lowest concentration sample in the ALS tray position 1.
- 4 Under the Sample column, enter Std 2.5ng.
- **5** Under the **Method/Keyword** column:
 - a Right mouse click and select **Browse for Method**. The **Browse for Folder** dialog box opens.
 - **b** Navigate to and select **demosim.M**.
 - c Select **OK**. The method name appears in the column.
- 6 Under the Data File column, enter Stdupdate01.

Т	Sam	ple Log Table								l	- 0	х
Data Path: C:\msdchem\2\DATA Browse Browse Method C:\MSDCHEM\2\METHODS												
	4	Туре	Vial	Sample	Method / Keyword	Data File	Multiplier	Level	Update RF	Update RT	Update QI	
	1	Calibration 🖵	1	Std 2.5ng	demoSIM	stdupdate01	1.00000	2.5	Replace 🖵	Replace 🖵	Replace 🕳	
	2	Sample	1	Sample 2	DEFAULT		1.00000		No Update	No Update	No Update	
	3	Sample	1	Sample 3	DEFAULT		1.00000		No Update	No Update	No Update	
	4											
	5											
	ę											
	•											
								OK	Cancel	Help		

- 7 Under Level column, enter 2.5.
- 8 Under **Update RF** column, click in the cell to activate the drop down list and select **Replace**.
- 9 Under **Update RT** column, click in the cell to activate the drop down list and select **Replace**.
- **10** Under **Update QI** column, click in the cell to activate the drop down list and select **Replace**.
- **11** Highlight rows 1 to 5.

- **12** Right mouse click and select **Repeat Row & increment**. Four lines are added to the table with incremented vial number and data file names.
- 13 In row 2, under Sample column, change the value to Std 5 ng.
- 14 In row 3, under Sample column, change the value to Std 10 ng.
- 15 In row 4, under Sample column, change the value to Std 25 ng.
- 16 In row 5, under Sample column, change the value to Std 50 ng.
- 17 In row 2, under Level column, change the value to 5.
- **18** In row 3, under **Level** column, change the value to **10**.
- 19 In row 4, under Level column, change the value to 25.
- 20 In row 5, under Level column, change the value to 50.

Data	a Path: C:\msdc	hem\2\D	ATA		Browse	N	fethod C:	MSDCHEM\2\	METHODS	
	Туре	Vial	Sample	Method / Keyword	Data File	Multiplier	Level	Update RF	Update RT	Update QI
1	Calibration	1	Std 2.5ng	demoSIM	stdupdate01	1.00000	2.5	Replace	Replace	Replace
2	Calibration	2	Std 5ng	demoSIM	stdupdate02	1.00000	5	Replace	Replace	Replace
3	Calibration	3	Std 10ng	demoSIM	stdupdate03	1.00000	10	Replace	Replace	Replace
1	Calibration	4	Std 25ng	demoSIM	stdupdate04	1.00000	25	Replace	Replace	Replace
5	Calibration	5	Std 50ng	demoSIM	stdupdate05	1.00000	50	Replace	Replace	Replace
6	Sample 룾	6	unknown01	demoSIM	unknown01	1.00000		-	-	-
•						1	II			

- **21** In row 6, enter an unknown sample for analysis as shown in the figure.
- 22 Select **OK** to close the **Sample Log Table**.

Save the Sequence



- 1 Select the Save Sequence As... button, **Sequence** dialog box opens.
- 2 In the File name field, enter updatequant.
- 3 Select Save. The dialog box closes and the sequence is saved.

Run the Sequence



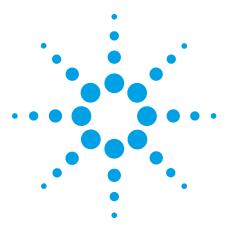
. The Start Sequence

1 Select the **Run Sequence** button, **1**. The dialog box opens.

 Sequence Barcode Options Disable Barcode for This Sequence On Mismatch – Inject Anyway, Continue Sequence On Mismatch – Don't Inject, Continue Sequence
On Mismatch – Inject Anyway, Continue Sequence
C On Mismatch – Don't Inject, Continue Sequence
-
1\DATA\eval2 Browse
Cancel Help

- 2 In the Method Sections to Run area, select Full Method.
- **3** In the **Sequence Comment** field, enter a description of the sequence.
- 4 In the **Operator Name** field, enter your name.
- 5 In the Data File Directory field, add eval2 to the path.
- 6 Select **Run Sequence**. The calibration table of the demoSIM method is updated and the unknown sample results are calculated/reported with the recalibrated calibration curve.

10 Recalibrate and Quantitate Unknowns



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Create a Cool Down Method

Create the Cool Down Method 138 Use the Cool Down Method 139

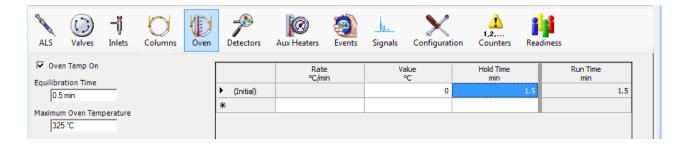
11

This chapter describes how to create and store a method to use for instrument maintenance tasks. Using this type of method helps prevent damage to the instrument electronics and columns and avoid injuries such as burns or shocks.



Create the Cool Down Method

- **1** Select View > Instrument Control.
- 2 Select the GC Edit Parameters button, . The GC Edit Parameters window opens.
- . The oven parameters are
- 4 In the **Oven Ramp** table, clear the **Rate** and **Value** entries.



Select the **Oven** button,

displayed.

3

- 5 Select the **lnlets** button, . The inlet parameters are displayed.
- 6 Select the **front** or **back** tab, depending on your hardware configuration.
- 7 Mark the **Heater** check box and enter 35°C in the corresponding field.
- 8 Mark the **Pressure** check box. Column flow must be maintained to prevent damage to the column when hot.



- 9 Select the AUX button,
- 10 Clear the **On** check box for the **Aux 2 Heater**.



11 Select **OK**.

F1

The Save Method

13 In the Method File field, enter cool down.

12 Select the Save Method button,

As dialog box opens.

14 Select OK.

Use the Cool Down Method

To use the cool down method, load the method, access the Edit **GC Parameters** window, and right mouse click in the right panel. Select **Download Method to GC** from the context menu. A confirmation message is displayed.

Agilent	7890A	×
Metho	d Download Con	npleted.
	ок	

Select **OK** to close the message and return to the **GC Edit Parameters** window.

When the GC enters the Ready state, perform the maintenance.

11 Create a Cool Down Method



Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

12 Shut Down the System

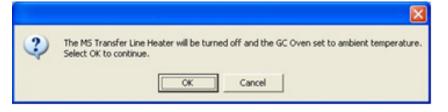
Shut Down the MS142Shut Down the GC143

This chapter describes how to shut down the MS and GC.



Shut Down the MS

- **1** Select View > Tune and Vacuum Control....
- 2 Select Vacuum > Vent... A confirmation message appears.



- 3 Select OK.
- 4 The Vent Cycle dialog status window opens and remains open until the vent is complete. You can close the dialog box by selecting Exit, however, the process continues. To reopen the Vent Cycle status window, select View > Vacuum Status.

Vent Cycle							
VENT CYCLE IN PROGRESS							
Cycle started: Wed Apr 06 17:15:09 2011 Time remaining: 36:46 min (approx.)							
Turbo pump:	Actual On	Criteria	Status				
Turbo pump speed:	0%	< 50%	Ready				
MS Source:	230 °C	< 100	°C				
MS Quad: 150 °C < 100 °C							
Turbo pump is off and MS is cooling.							
			×				
	cle comple the mass		Power Switch.				

OK

5 Select **OK** to close the dialog box.

Do not turn off the MS at this time if you are first cooling down the instrument. The **Instrument Control** window will close when a configured instrument is powered off.

6 Select Close.

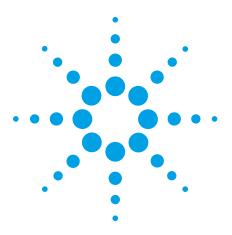
Shut Down the GC

- 1 In **Instrument Control**, load the GC cool down method.
- 2 Access the Edit GC Parameters window.
- **3** Right mouse click in the right panel and select **Download Method to GC** from the context menu. A confirmation message is displayed.

Agilent 7890A	×
Method Download Complet	ed.
ОК	

- 4 Select **OK** to close the message and return to the **GC Edit Parameters** window.
- 5 Close the **Edit GC Parameters** window and exit the ChemStation.
- **6** When the GC enters the Ready state turn off the power to the GC and the MS.
- **7** Turn off the carrier gas.
- 8 Power off the PC and all peripheral equipment.

Shut Down the System



13

Agilent G1701EA MSD Productivity ChemStation Familiarization Guide

Frequently Asked Questions

- Q. How often should the MSD be tuned?
- A. Perform an Autotune on a regular basis: weekly or monthly depending on use of the MSD. Perform a Check Tune daily to validate the performance of your instrument. If needed, perform a Quick Tune.
- Q. There are two autotune options: Tune MSD and Quick Tune. What are the differences between them?
- A. The Tune MSD maximizes the instrument sensitivity over the calibrant (PFTBA) mass range (69, 219, and 502). Quick Tune updates the peak width, mass assignment, and abundance.
- Q. An analyte elutes before the solvent peak. How can data be acquired before the solvent peak as well as after?
- A. Method parameters that control the MSD can be modified to update the method to capture data prior to the solvent peak. To update the method, use the timed events table to turn off the filament and data detection after the analyte elutes but before the solvent elutes. Set an event to turn the filament and the detector back on after the solvent peak has eluted.
- Q. The sensitivity for some analytes has become reduced while some are not being detected at all. How can this be corrected?
- A. Decreased sensitivity with the GC/MSD system may be caused by the following situations:
 - Sample: Analytes have evaporated or deteriorated in the sample.
 - Column: Column may be contaminated; column maintenance is recommended.
 - GC Inlet: Inlet liner, split vent, or septum may be dirty, damaged or contaminated; inlet maintenance is required.
 - Column Connection: Loose injection port ferrule or MSD transfer line ferrule, column installed incorrectly at the inlet or transfer line.
 - Injector: The syringe is plugged with septum material or is using an incorrect sampling volume.



- Ion Source: The ion source has become contaminated or dirty; clean the source or replace the necessary parts.
- Method Parameters:
 - MSD Parameters: Incorrect mass assignments are being used with your method.
 - GC Parameters: Method uses incorrect split ratio or requires a longer purge time.

To improve your sensitivity:

- Perform autotune to verify MSD performance.
- Refer to the hardware manual for step by step troubleshooting procedures.
- Call Agilent Technologies Customer Support.
- Q. When loading a data file, the error message **"No MS Data"** appears. What does this mean and what is the cause?
- A. **"No MS Data"** means that the data file selected does not contain the data.ms within the datafile.d. Typically this occurs when the user forgets to save the MS data file within the method parameters, the remote start/stop cable is not connected, or the acquisition was aborted or terminated.
- Q. When right-clicking on the TIS, the spectrum does not display, and the cursor is a (+) instead of a line. What causes this?
- A. This is generally caused by the manual integration feature turned ON in Data Analysis. In this mode, to turn OFF manual integration, use the **Manual Integration** option in the data analysis option dialog box. The cursor in the chromatogram window should return to a vertical line.
- Q. How does the Match Quality of library search results relate to the compound?
- A. The Match Quality of the unknown is identified as the reference. Values greater than 90 are very good matches. Values less than 50 mean that substantial differences exist between the unknown and reference and the match should be regarded as uncertain. Differences in probability values of ±5 are generally not significant. An asterisk (*) before the probability value indicates that the molecular ion was used in the match. Because many factors affect the match quality and ordering of the compounds in the hit list, the list should be viewed as an interpretative guide to the unknown's identity. It is the chemist's responsibility to

determine whether the match identity is correct. For example, graphical comparison of the unknown's mass spectrum with that of an authentic sample, knowledge of the sample's history, and other pertinent information should be considered.

- Q. Why does the library search list different spectra for the same compound?
- A. Commercially available databases such as NIST or WILEY libraries contain MSD data for instruments from several manufacturers for one compound. This means search results may list duplicate compounds. To avoid this duplication, edit your Search Strategy to remove duplicate CAS numbers. See the online help for instructions on how to do this.
- Q. Can a compound in a spectral library be viewed manually?
- A. Yes it can. The Parametric Retrieval feature allows you to manually specify search criteria for your spectra. It retrieves a spectrum from the specified library based on those criteria and displays the results. The online help contains instructions for how to set up the criteria for your manual search.
- Q. Can a chromatogram be redrawn to a different scale? How?
- A. There are three ways to redraw the image of a chromatogram:
 - Zoom in the area of interest in the existing chromatogram. Click within the area of interest and drag the cursor to define the area for the new chromatogram.
 - Using the Data Analysis menu, click Chromatogram > Chromatogram Scaling... Select the chromatogram to be rescaled, and specify the scaling method to be used.
 - Use the **DRAW** command to rescale the chromatogram and define the window location of the image. Refer to the online help for more detailed instructions on how to perform this action.
- Q. After column maintenance or replacement, the chromatographic peak is missing. How can the peak be recovered?
- A. The chromatographic peak is normally determined by the retention time window where the peak of interest would display. After a column change or maintenance, this retention time will shift. Perform a retention time update.

- Q. Why would an Extracted Ion Chromatogram (EIC) be used instead of a Total Ion Chromatogram (TIC) for quantitation?
- A. An EIC gives more stable results compared to the TIC.
- Q. If autointegration does not work on a peak, can it still be integrated?
- A. Yes, the peak can be integrated manually. For some cases, manual integration mode is the only method to use. Turn on manual integration under **Tools > Options > A/B**. Select the area of the peak that you would like to integrate. Refer to the online help for complete instructions on how to manually integrate.
- Q. How can integration results be exported?
- A. Click **Chromatogram > Integration Results**... Tabulation of the integration results associated with the current data file is displayed. Click the **Copy** button to save tabulated data to the clipboard. Now the results can be pasted into another application package.
- Q. How can chromatogram graphics be exported?
- A. To copy a selected Data Analysis window to the clipboard use the Tools > Copy Window menu. Answer the prompt for the number of the graphics window to be copied ('1' for spectrum, '2' for TIC). Click OK to copy the selected window to the clipboard. Now the graphics can be pasted into another application package. Alternatively, right-click Data Analysis, right-click in the window of interest, and copy and paste the image into another application.
- Q. Why are the integration results on the quantitation report and my integration results different?
- A. Integration results on the quantitation report are generated using the extracted ion chromatogram (EIC) of the target ion specified in the compound on the first page of the quantitation database while the integration results generated manually are based on the total ion chromatogram (TIC). Specific integration events can be used to integrate if the compound data file is specified on the third page of the quantitation results database. If the extracted ion chromatogram and the total ion chromatogram use the same integration event file, you may get the same integration results.
- Q. Why would the chromatogram show a peak if the quantitation report shows a N.D?

- A. There are two possible reasons. First, incorrect integration events may be used for quantitation. To check this, open the **Edit Compound** dialog with **Calibration >Edit Compound**....Select the compound of interest on the left panel. Click the **Advanced** tab to show the Integration Parameter File being used. The second cause may be that the concentration of the peak was lower than the quantitation limit. Integration parameters are set to integrate at least the lowest concentration standard sample peak. The area reject or other event may restrict small peaks from being integrated. In this case, the peak is lower than the quantitation limit; therefore, N.D. is appropriate. Please refer to the online help for additional information.
- Q. Why does the quantitated data file show different results for the qualifier ion ratio?
- A. The qualifier ion ratio on the first page of the quantitation database is calculated using the abundance of the qualifier ion relative to the abundance of the target ion when the compound was registered (=abundance ratio). The qualifier ion ratio can also be calculated using the integration of the spectrum (area of the curve) of the qualifier ion relative to the integration of the target ion spectrum (=area ratio). You can specify which way you would like the qualifier ion ratio to be calculated. See the online help for instructions.

13 Frequently Asked Questions



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