

**Agilent MassHunter
Data Acquisition
Software for 5975/5977
Series GC/MSD**

Familiarization Guide



Agilent Technologies

Notices

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In This Guide...

This manual will serve as a familiarization or quick reference guide for first-time users of MassHunter Data Acquisition.

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

1 Introduction

Chapter 1 provides an overview of how to start up and tune your system.

2 Create a Scan Method

In Chapter 2 you will learn how to edit, save, and run a data acquisition Scan method.

3 Create a SIM Quantitation Method

In Chapter 3 you will learn how to create a SIM method.

4 Running Samples

Chapter 4 describes how to run a single sample and how to run a sequence.

5 General Information

Chapter 5 includes miscellaneous information on commonly used features in MassHunter.

Where to Find More Information

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

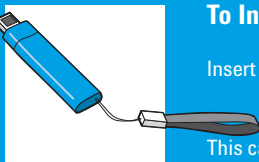
- **Agilent GC and GC/MS Manuals and Tools** DVD set
- **Agilent GC/MS Software Information and Manuals** memory stick



To Install Your Hardware Library

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

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



To Install Your Software Library

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

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

See the **Agilent 5977 Series MSD System Quick Start** document (G3870-90103) for more details on how to install this information on your computer.

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Introduction

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Included in this Chapter is a general list of steps to follow when you start up your hardware. Also included is a brief introduction to tuning and an explanation of how to run an autotune on the instrument.



Start up the Hardware

Below is a general list of steps to follow when you start up your hardware.

- 1** Review the Agilent Gas Chromatograph and MSD Operating Guides for important safety information and start up details before powering on your instruments.
- 2** Verify that the inlet septum, liner, and O-ring are clean, properly installed, and in good condition.
- 3** Install a conditioned column that is appropriate for your method in the GC. Attach the column inlet to the inlet and its outlet to the MSD transfer line. See the Agilent MSD Operation Manual for details.
- 4** Verify the ion source is installed.
- 5** Verify the carrier gas required by your method is attached to your inlet.
- 6** Power on the Agilent 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 completed automatically and does not require operator actions.

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

Tune your MS

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.


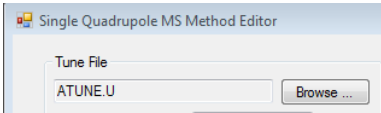

The autotune program described in this section adjusts the MS for good performance over the entire mass range and is recommended for most applications.

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

Run Autotune

- 1 From the PC desktop, select the Instrument Control shortcut icon to open MassHunter Data Acquisition.
- 2 Select **Method/Load Method** then navigate to and select the method you want to use for data acquisition (e.g., **default.m** in the MassHunter/GCMS/1/methods directory), and click **OK**.
- 3 To check which Tune file is currently associated with your method, in the Instrument Control view, click the **Instrument MS Parameters** icon to display the Quadrupole Method Editor dialog. 
- 4 In the Tune File group, verify the tune file shown is the one you wish to use for this method. (Atune.U is a good choice.) Click **Browse** and select a new one if necessary. 
- 5 Click the **MS Tune** icon to display the Select Tune Type dialog. 

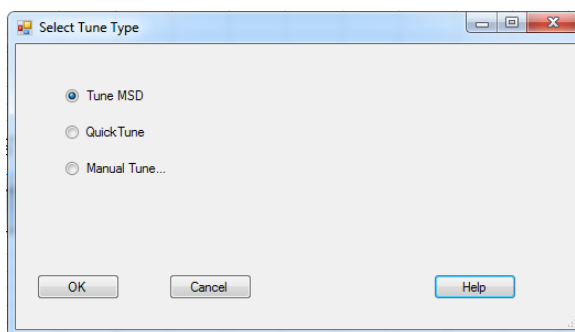
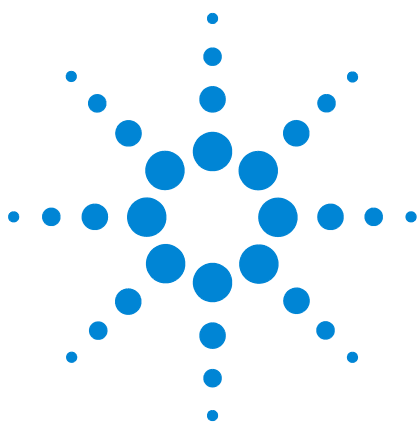


Figure 1 Select Tune Type dialog display

- 6 Select **Tune MSD** to perform a complete autotune, or select **Quick Tune** to adjust peak width, mass assignment, and abundance, without changing ion ratios. Alternatively, select **Manual Tune** to display the Manual Tune dialog and graphs for doing manual edits to the Tune file.
- 7 Click **OK** to close this dialog and start the tune.
- 8 Wait for the tune to complete and to generate the report.
- 9 To evaluate the tune results, select **Evaluate Tune** from the **Checkout** menu. To view history of tune results, in the Instrument Control view select **Checkout > View Previous Tunes....**

See the online Help provided with your MassHunter Data Acquisition software for additional information about tuning.



2 Create a Scan Method

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A good way to create a new method is to begin with an existing one, edit the settings, and save it as a new method, using the **Edit Entire Method** option.

This chapter describes how to use the **Edit Entire Method** option to open the default method and modify its Method Information and Instrument/Acquisition settings to represent a scan method, then save it as a new method name.



Edit the Entire Method Option - Overview

For this example, we will modify the Method Information and Instrument/Acquisition settings of the default method to represent a scan method. During this process you will be presented with 7 primary dialogs. Each is described on the following pages.



Figure 2 Edit the entire method option - overview

1) Edit Method Dialog

The first dialog you will see when you select the **Edit Entire Method** option is the Edit Method dialog.

Here you will identify the parts of the method you want to address as you create your method.

To begin the process:



- 1 Click the **Load Method** icon, or select **Method/Load Method**.
- 2 Navigate to and select the method you want to start with (e.g., **default.m** in the `MassHunter/GCMS/1/methods` directory), and click **OK**.
- 3 Select **Method/Edit Entire Method...**The Edit Method dialog opens.
- 4 Check each section you want included in this method.
 - **Method Information** - To provide a description of the method, specify whether or not to save a copy of the method with the collected data, and define what parts of the method sections to run.
 - **Instrument/Acquisition** - To have the Edit Entire Method process display all the dialogs required to edit the acquisition parameters for both the GC and MS parts of the currently loaded method.
 - **Data Analysis** - To display the Data Analysis portions of the method. Data Analysis settings are beyond the scope of this guide, so we are not including data analysis in our example.
- 5 Click **OK** to close the Edit Method dialog and continue.The Method Information dialog opens.

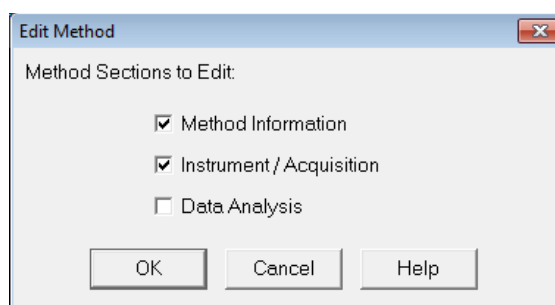


Figure 3 Edit Method dialog

2) Method Information Dialog

In the Method Information dialog you will provide a description of the method, decide whether or not to save a copy of the method with the collected data, and define what parts of the method sections to run. For example:

- 1 In the **Method Comments** field, enter a description of this method.
- 2 Mark the **Save Copy of Method With Data** checkbox. This causes MassHunter to save a copy of the method along with the sample data collected.
- 3 In the **Method Sections to Run** group box, mark the **Data Acquisition** checkbox only. This specifies that the Data Acquisition will run when this method is used, but the data analysis and post-run macros will not be run.
- 4 Click **OK** to close the Method Information dialog. Because we are running the Edit Entire Method option, and we did check the Instrument/Acquisition option in the previous step, the Inlet and Injection Parameters dialog displays next.

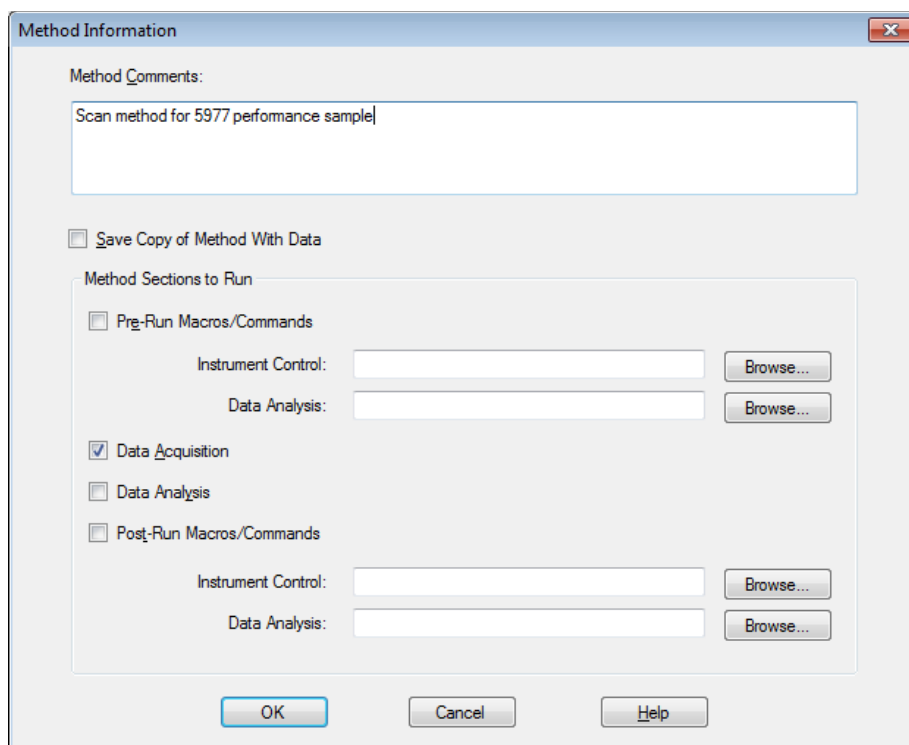


Figure 4 Method Information dialog

3) Inlet and Injection Parameters Dialog

In the Inlet and Injection Parameters dialog you will specify the Sample inlet and Injection Source and the inlet and MS locations.

- 1 From the **Sample Inlet** dropdown list, select **GC**.
- 2 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**.
- 3 Mark the **Use MS** checkbox to allow MassHunter to turn on the MS analyzer and save the MS sample data acquired during the run. Uncheck this box only if you do not want to collect Mass Spec data. For example, you would uncheck this box if you are running a GC (non-MS) detector and you were acquiring data for the GC detector only.
- 4 In the **Inlet Location** box, select the location where your inlet is attached to the MS through the column.
- 5 In the **MS Connected to** box, select the location where your inlet is attached to the MS through the column.
- 6 Click **OK** to close the Inlet and injection Parameters dialog. Because we are running the Edit Entire Method option, the **GC Edit Parameters** window displays next.

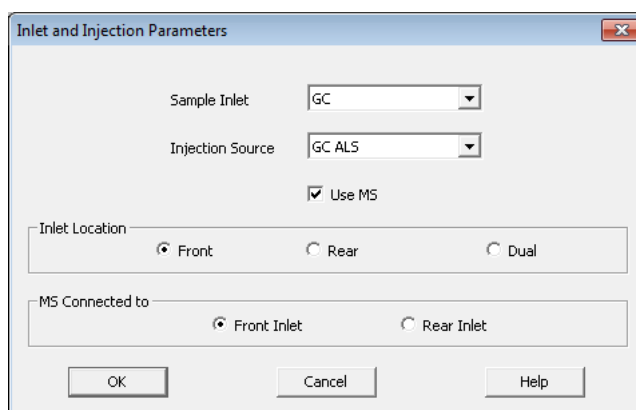


Figure 5 Inlet and Injection Parameters dialog

4) GC Edit Parameters Dialog

The **GC Edit Parameters** window contains several icons. Each icon contains one or more panels where you will define the GC instrument settings. We will go through each one here.

To begin, we will address the Configuration panel because some of the settings made here will affect settings in other panels.

GC Configuration panel

On the **Configuration** panel, there are four 4 tabs:

- **Miscellaneous**
- **Columns**
- **Modules**
- **ALS**

The following pages include a general description of the settings used in these tabs. Check the online **Help** for additional details.

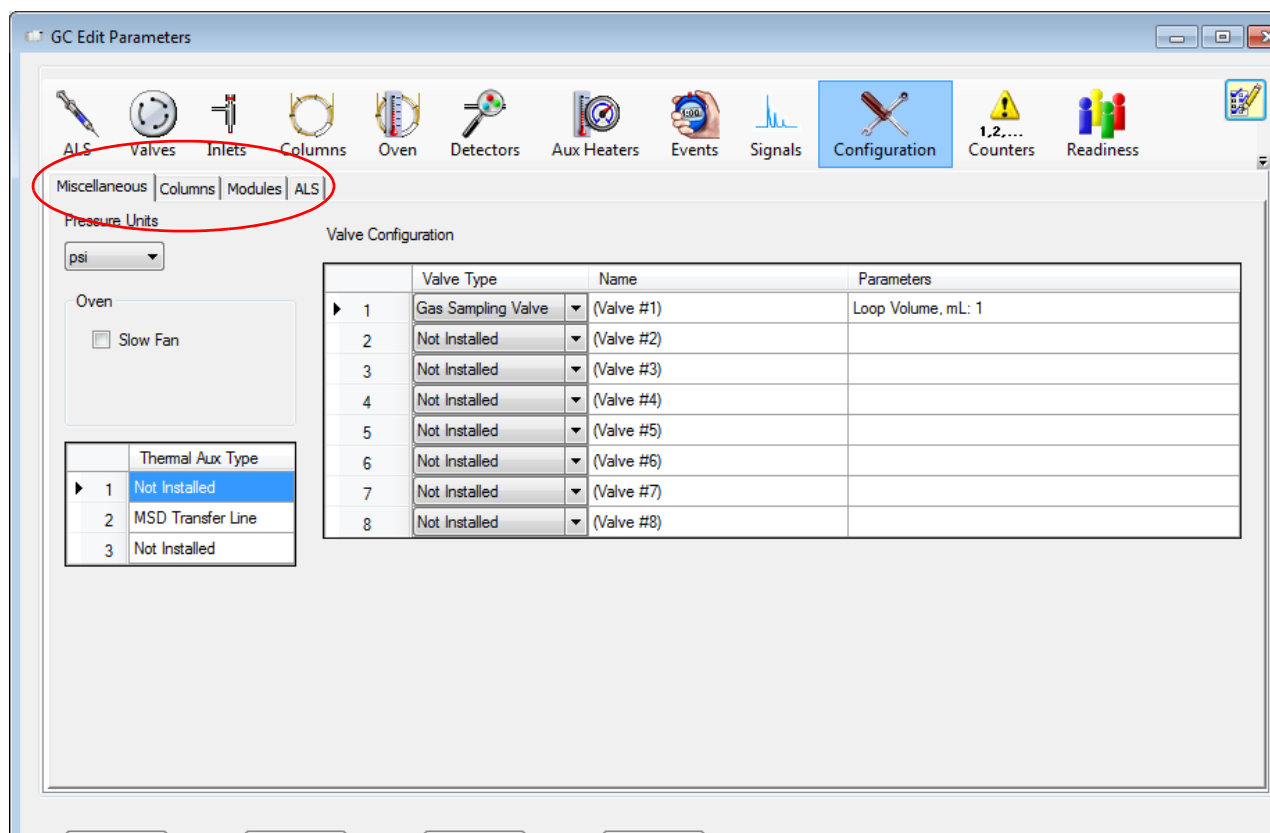


Figure 6 Configuration panel

GC Configuration panel - Miscellaneous tab

Under the **Miscellaneous** tab you will define your method's pressure units, configure valve types and oven fan parameters (slow fan_), and view the thermal aux configuration.

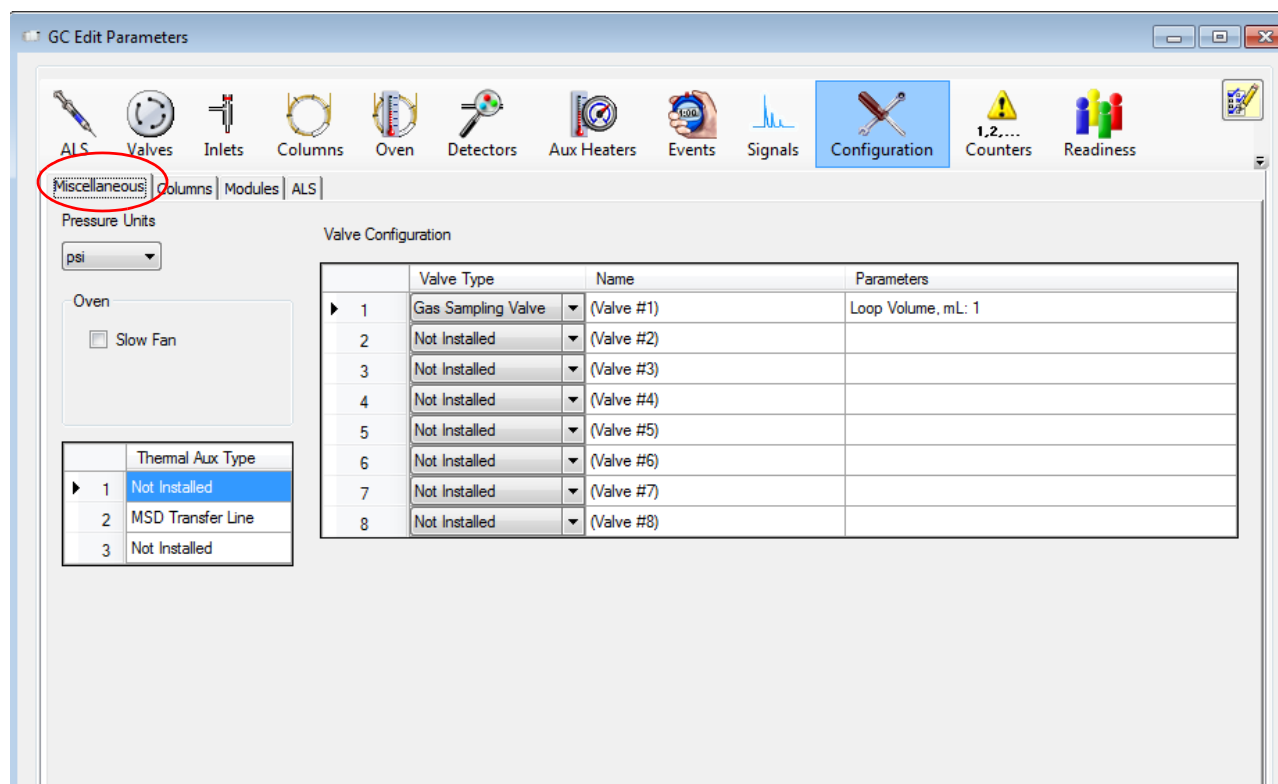


Figure 7 GC Configuration panel - Miscellaneous tab

GC Configuration panel - Columns tab

Under the **Configuration > Columns** tab you will define and configure a column for each position (1-6) in your GC. You can also calibrate a column, lock a column's configuration, describe how each column connects to GC devices, and tell the system how each column is heated.

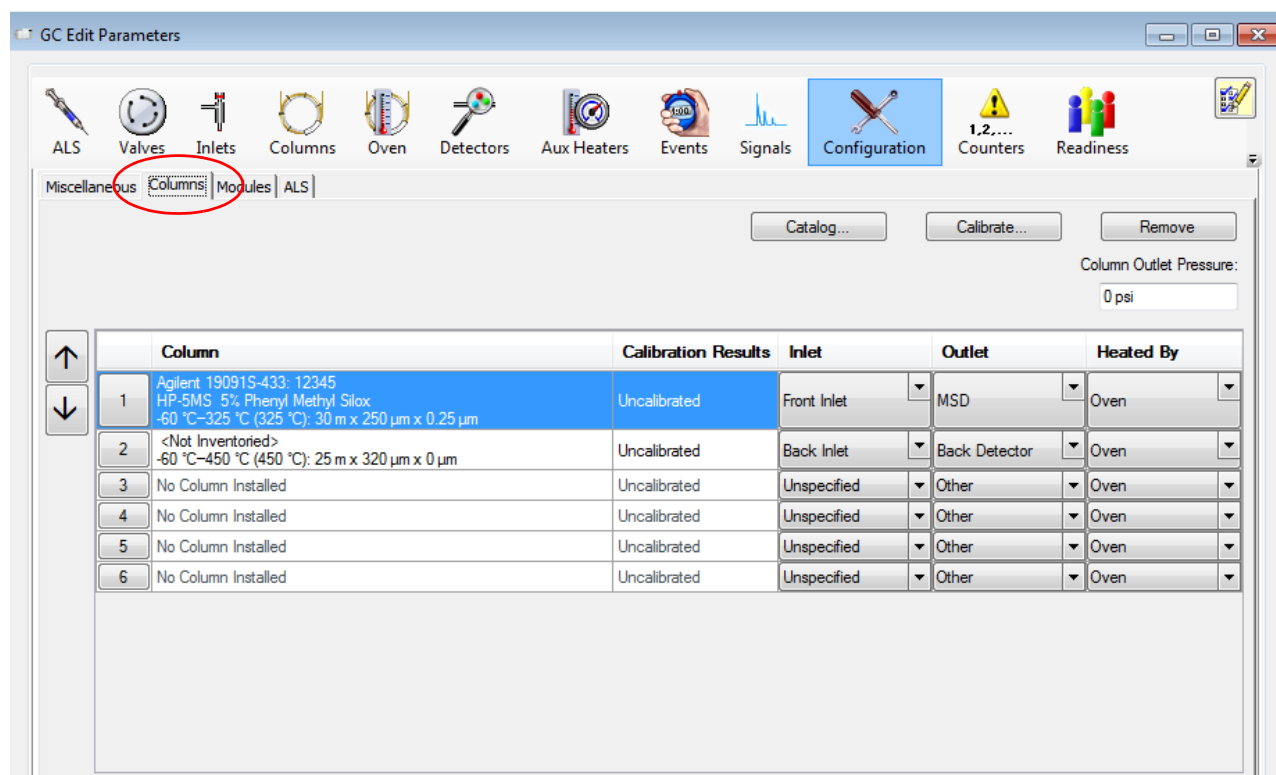


Figure 8 GC Configuration panel - Columns tab

GC Configuration panel - Modules tab

Under the **Configuration > Modules** tab you will define the gas types for your inlets, detectors, and any Aux EPC modules.

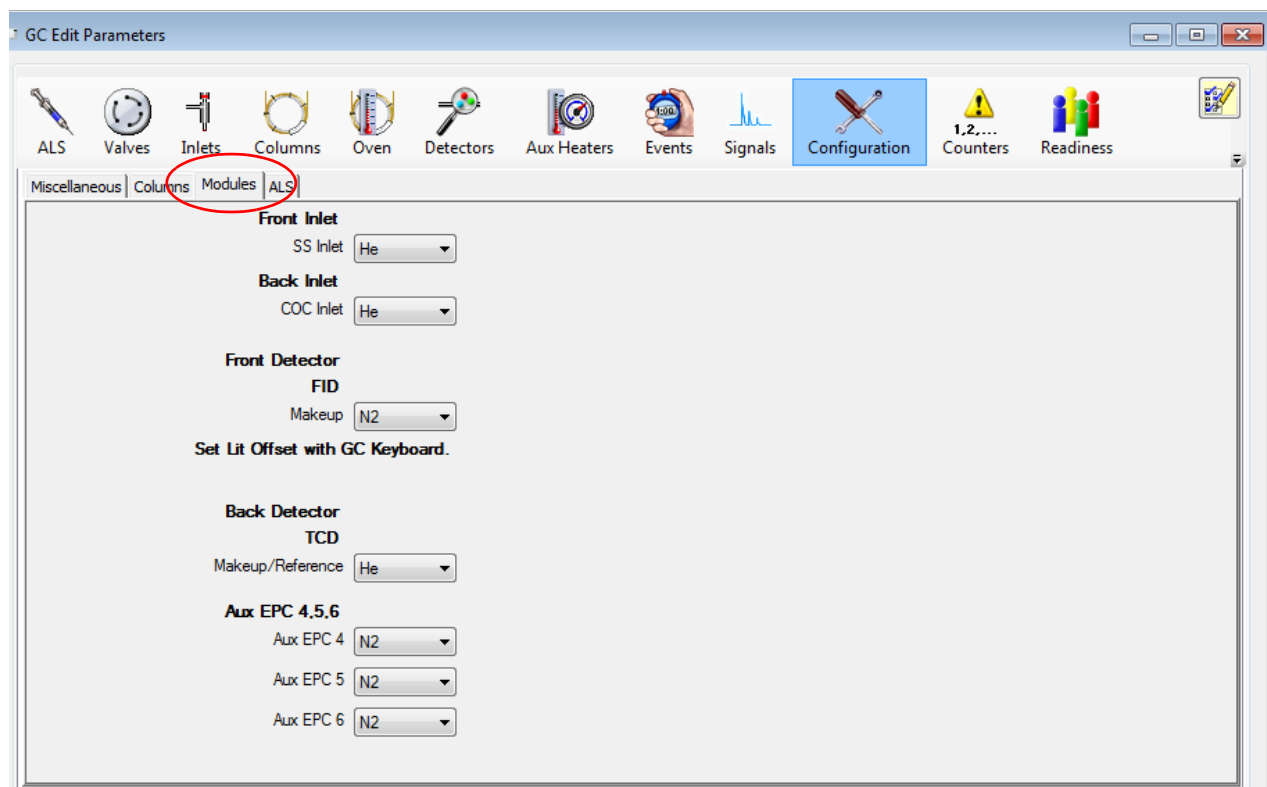


Figure 9 GC Configuration panel - Modules tab

GC Configuration panel - ALS tab

If you have a front or back injector installed, under the **Configuration > ALS** tab you will see the installed injector type and current firmware revision.

From here you will set the configuration parameters to match your installed syringe and desired wash mode.

The configured syringe information must match the actual syringe to achieve accurate injection amounts.

Depending upon the installed injector and turret, these parameters may be available to configure multiple solvent wash bottles usage. If necessary, refer to your injector user documentation for details.

CAUTION

Some syringes are labeled so that the labeled syringe size reflects the maximum injectable volume, rather than the full syringe volume. The maximum injectable volume for a syringe installed in an Agilent injector is 50% of the full syringe volume. If the highest syringe volume marking is only halfway up the syringe barrel, enter double that value as the syringe size. Agilent systems calculate injection volume based on full plunger stroke.

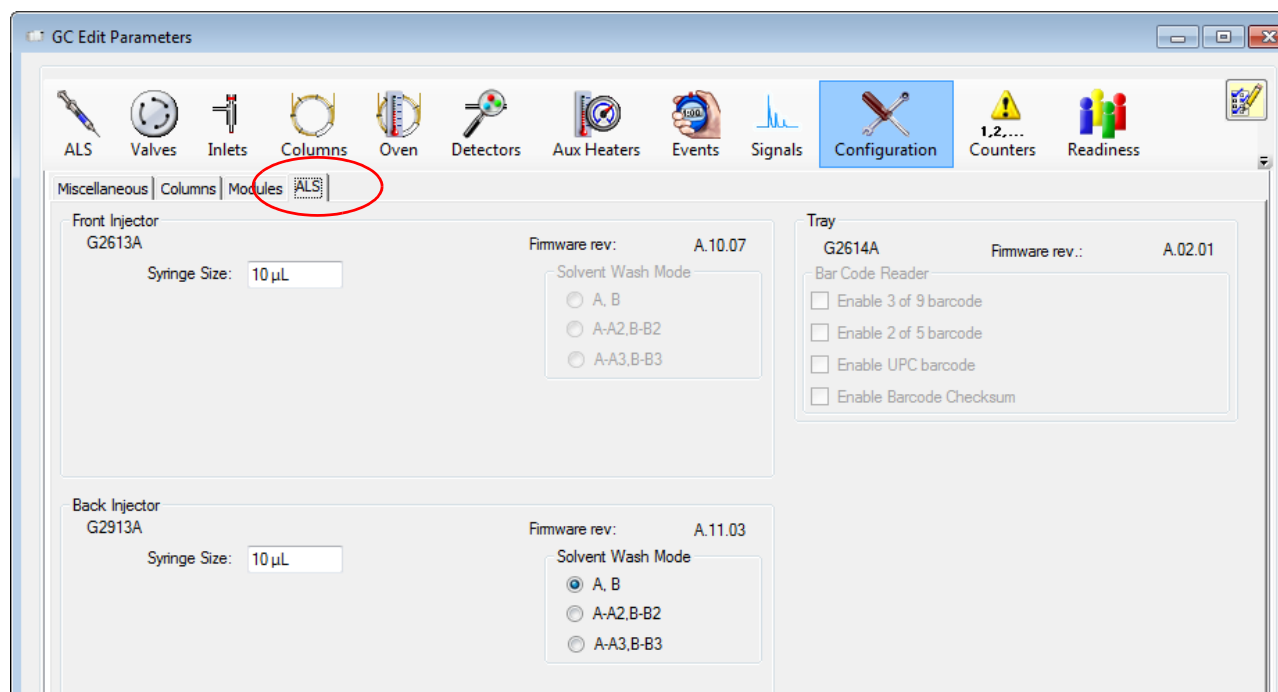


Figure 10 GC Configuration panel - ALS tab

GC ALS panel

ALS - Front/Back Injector tab

Use the **Front/Back Injector** panels to configure injection parameters, washes and pumps settings, dwell time, plunger speed, sample depth, and the type of your injections.

Note that wash behavior varies depending on syringe size:

- For 100- μ L and smaller syringes, the sampler draws the solvent volume specified in the Volume field. After the needle leaves the wash solvent, the injector draws the plunger up until the solvent slug rinses 80% of the syringe volume, then dispenses to waste.
- For 250- μ L and larger syringes, the injector draws and dispenses the exact wash volume of solvent specified in the Volume field. The injector rinses only the volume specified.

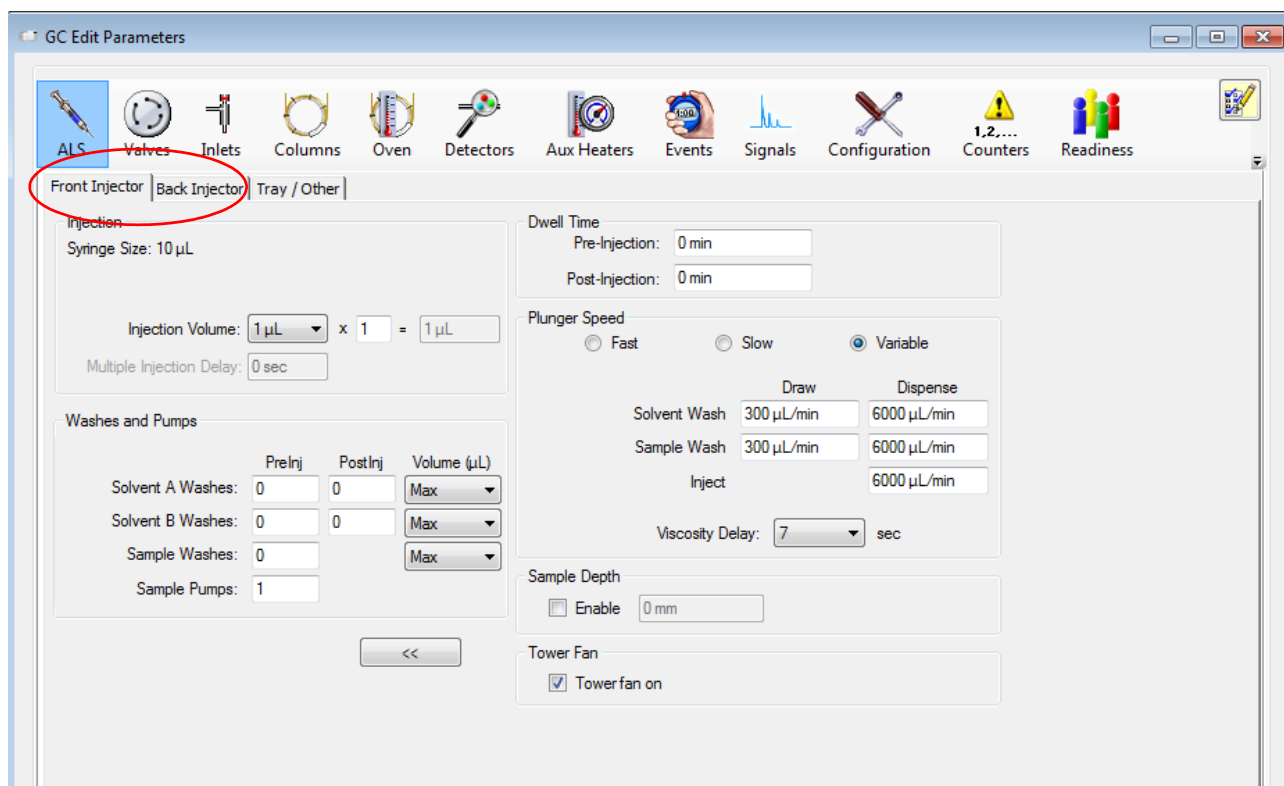


Figure 11 ALS - Front/Back Injector parameters

ALS - Tray/Other tab

Use the **Tray/Other** tab to set the barcode reader, vial mixer, and vial heater parameters, if installed and supported on your system.

Sample overlap is also set on the Tray/Other tab. Sample overlap can increase throughput by allowing the ALS to begin processing the next sample before the current run finishes. ALS sample preparation does not have to be finished before the current runs ends.

To use Sample Overlap, select **Enable Sample Overlap**, then select the preferred timing for the overlap and enter a time (in minutes) as needed.

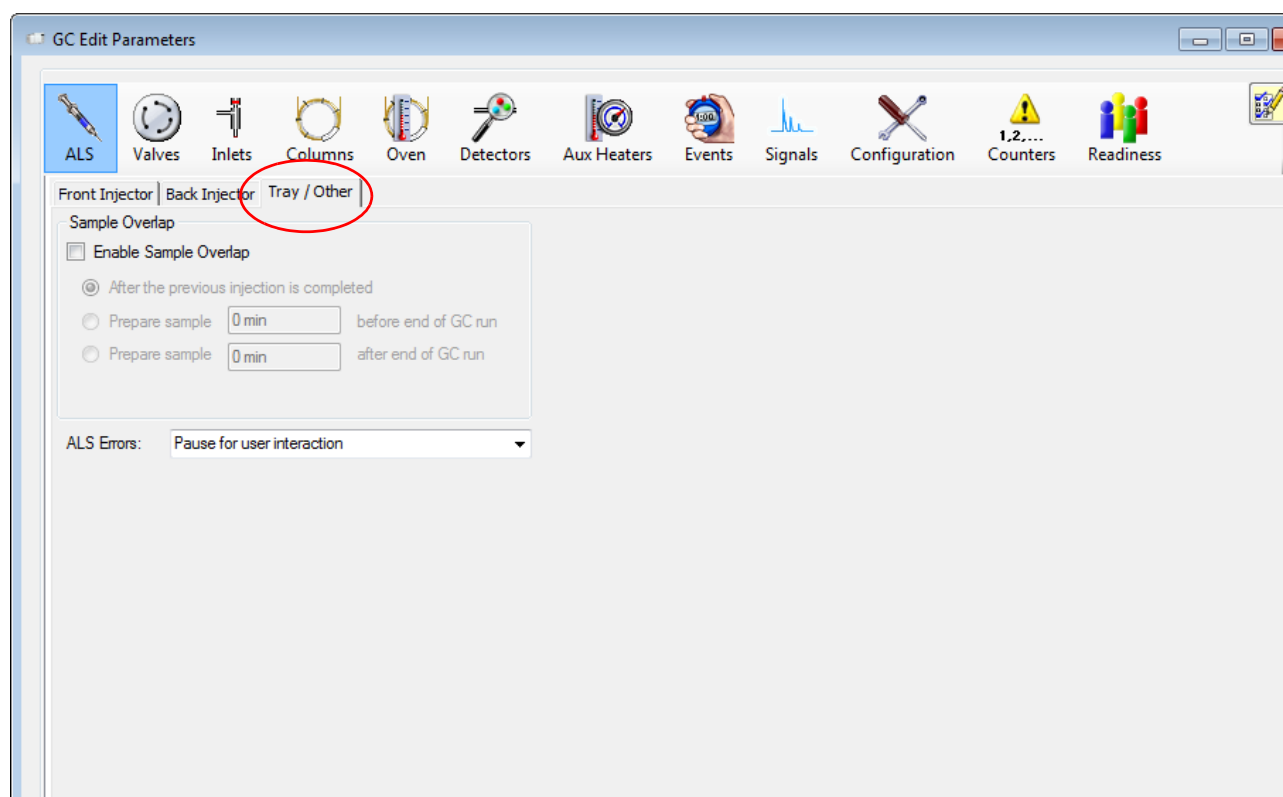


Figure 12 ALS - Tray/Other parameters

GC Valves panel

The **Valves** icon is available only if your GC and method configuration have installed and configured valves.

Refer to your GC documentation to determine the valve types and capabilities available with your GC type.

In a method you can specify the initial setting for valve parameters such as the On/Off state, Position, Load Time (min), and Inject Time (min) depending on the valves configured in your GC.

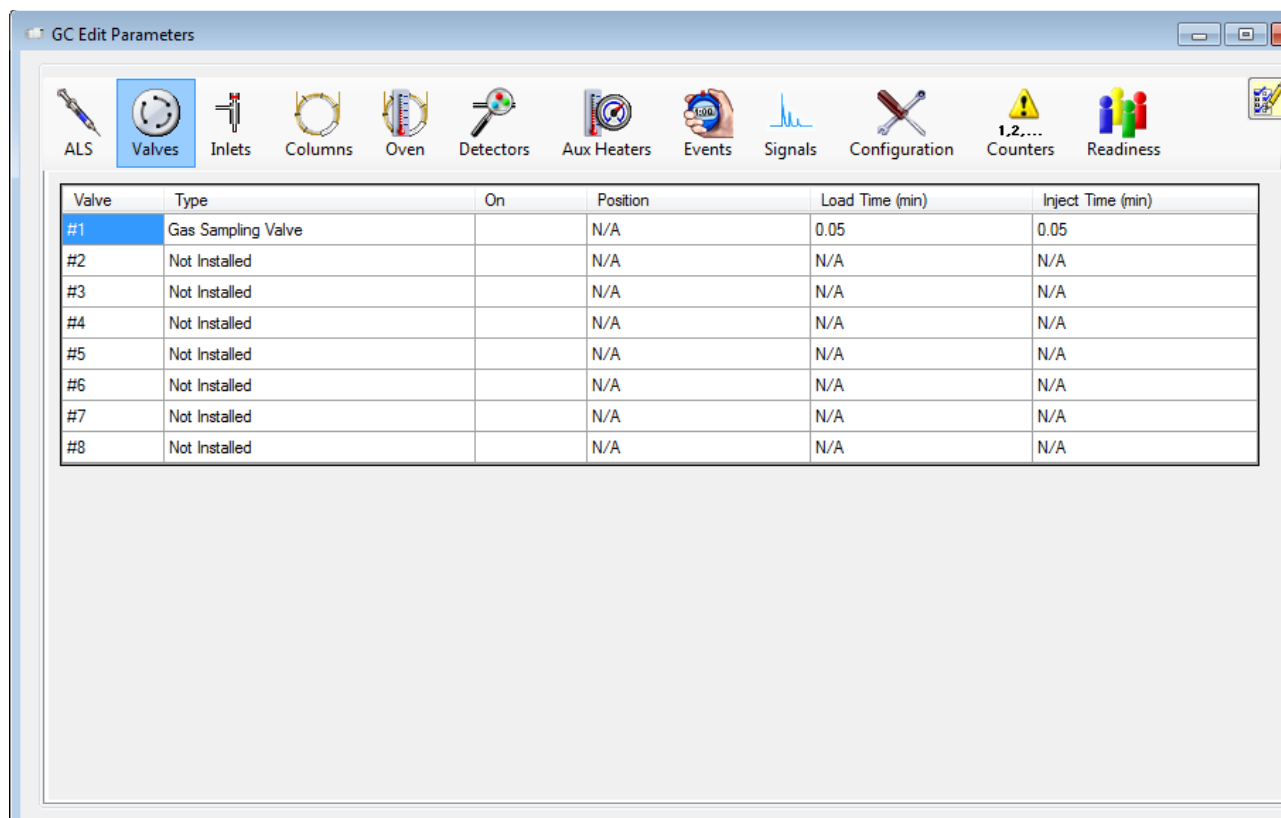


Figure 13 GC Valves panel

GC Inlets panel

Use the **Inlets** panel to set parameters for the front and back inlet installed in your GC. This example shows Split / Splitless (SSL) inlet parameters and setpoints. The column can be either pressure or flow controlled.

NOTE

Settings made in Columns Parameters automatically modify Pressure and Flow parameters in Inlet Parameters, and vice versa. For example, entering a Column panel Pressure of 30 psi sets Inlet panel Pressure to 30 psi.

The actual GC parameter value for each setpoint displays next to the setpoint field.

Gas Saver reduces the carrier gas flow rate from the split vent after a sample is within the column. The program maintains column head pressure and flow rate while purge and split vent flow rates are decreased. Gas Saver can be used in any column mode.

In general, start Gas Saver after inlet purge time for splitless injection or 2 minutes after a split injection. Reduced flow rates continue until the next run.

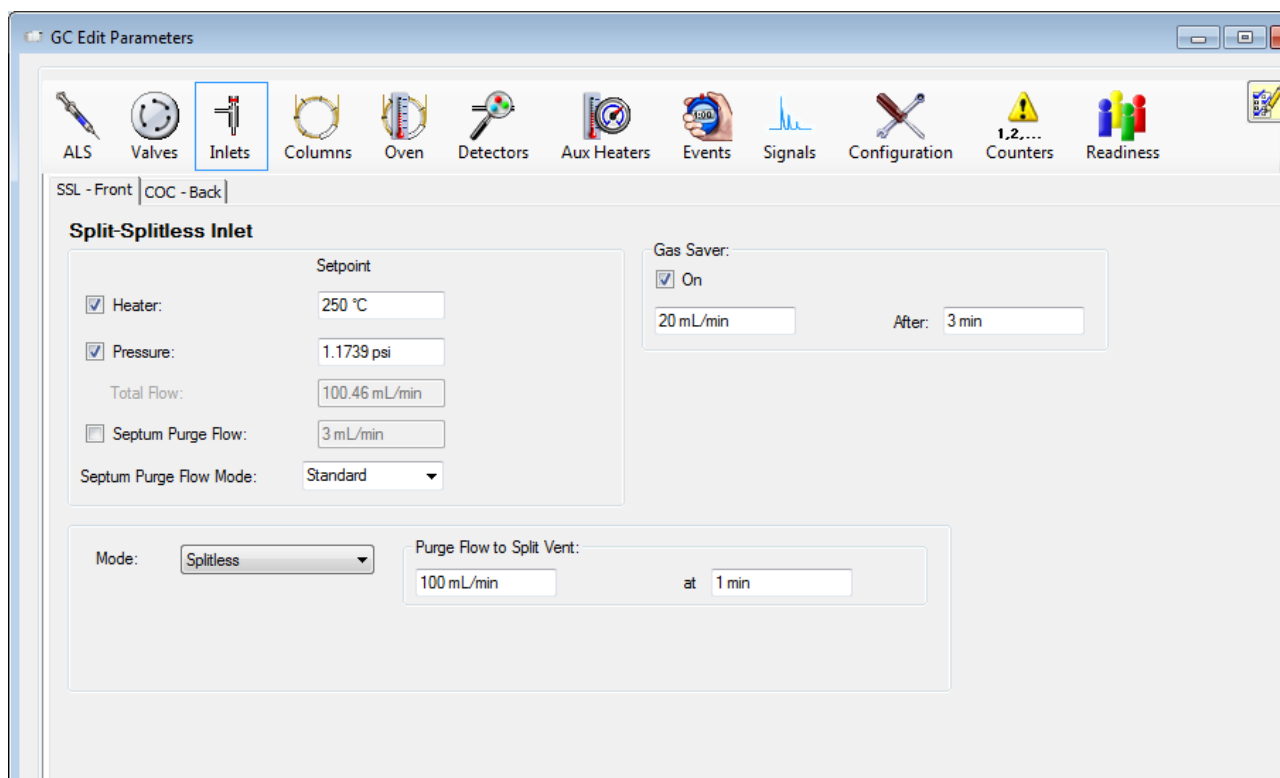


Figure 14 GC Inlet panel

GC Columns panel

Use the **Columns** panel to control column flow and pressure behavior during the run.

Column parameters are available only if your GC and method configuration have installed and configured columns.

From the Selection box, select the desired column or auxiliary pressure control device for your method.

Click the **Control Mode** checkbox to enable the inlet EPC control, then configure the column Flow, Pressure, Average Velocity, and Holdup Time parameters.

Select a constant pressure, constant flow, ramped pressure, or ramped flow program. If you selected a ramped pressure or ramped flow program, configure the ramp table.

If desired, you can change an installed column using the Change Column option.

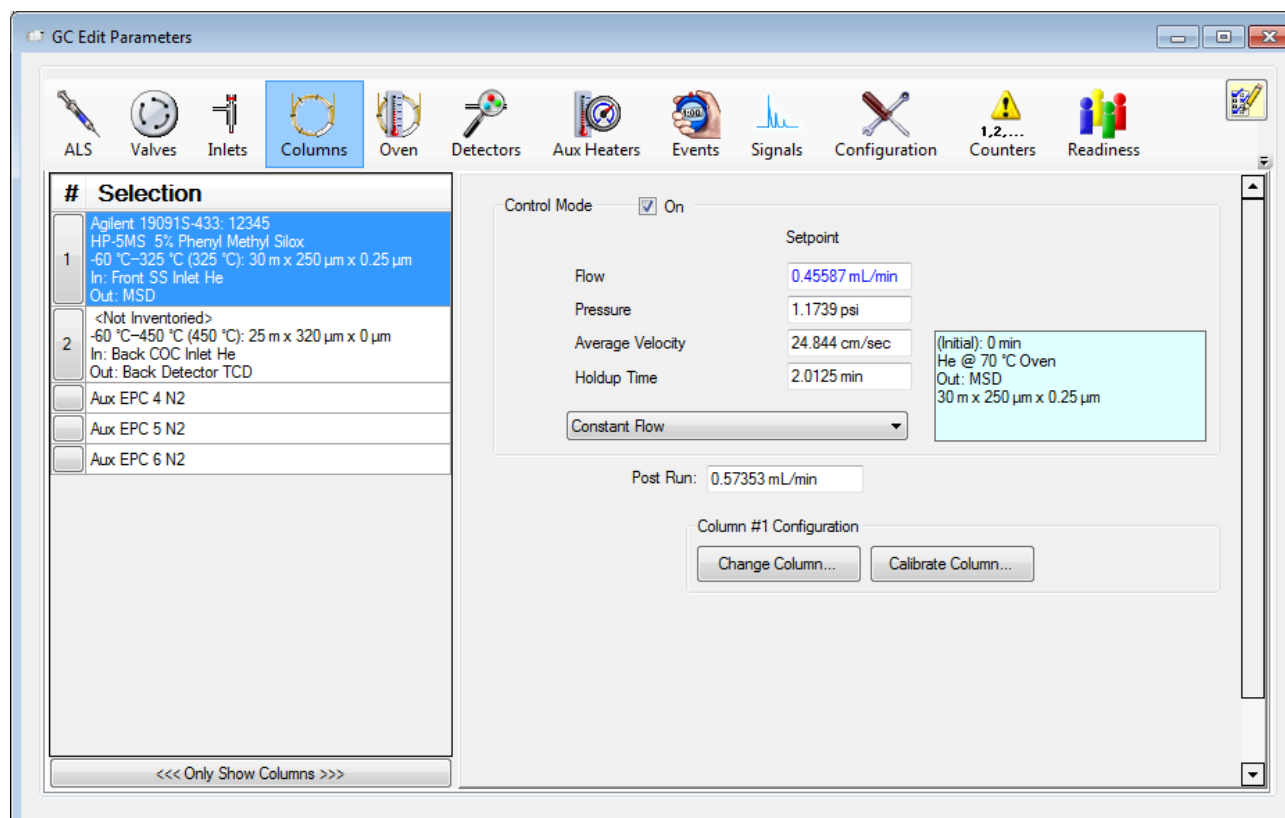


Figure 15 GC Columns panel

GC Oven panel

Use the **Oven** panel to control oven temperature parameters and to configure temperature ramps during and following a run.

If the current heater settings will adversely affect the columns you have specified for this method, they will be highlighted in yellow. Change the column selections, or your heater settings to resolve the problem before continuing.

Note that the oven total **Run Time** controls the maximum Run Time in all other flow, pressure, and temperature programs. For example, inlet and column program times cannot exceed the total oven Run Time.

Select **Override Column Max** (in °C) if you need to allow an oven temperature greater than the column maximum temperature setpoint. If the Maximum Oven Temperature is greater than the column maximum temperature, this label highlights.

The **Post Run** temperature value is the temperature at which the oven heats or cools to and holds for the specified time in the **Post Run Time** field. Set these if appropriate for your method.

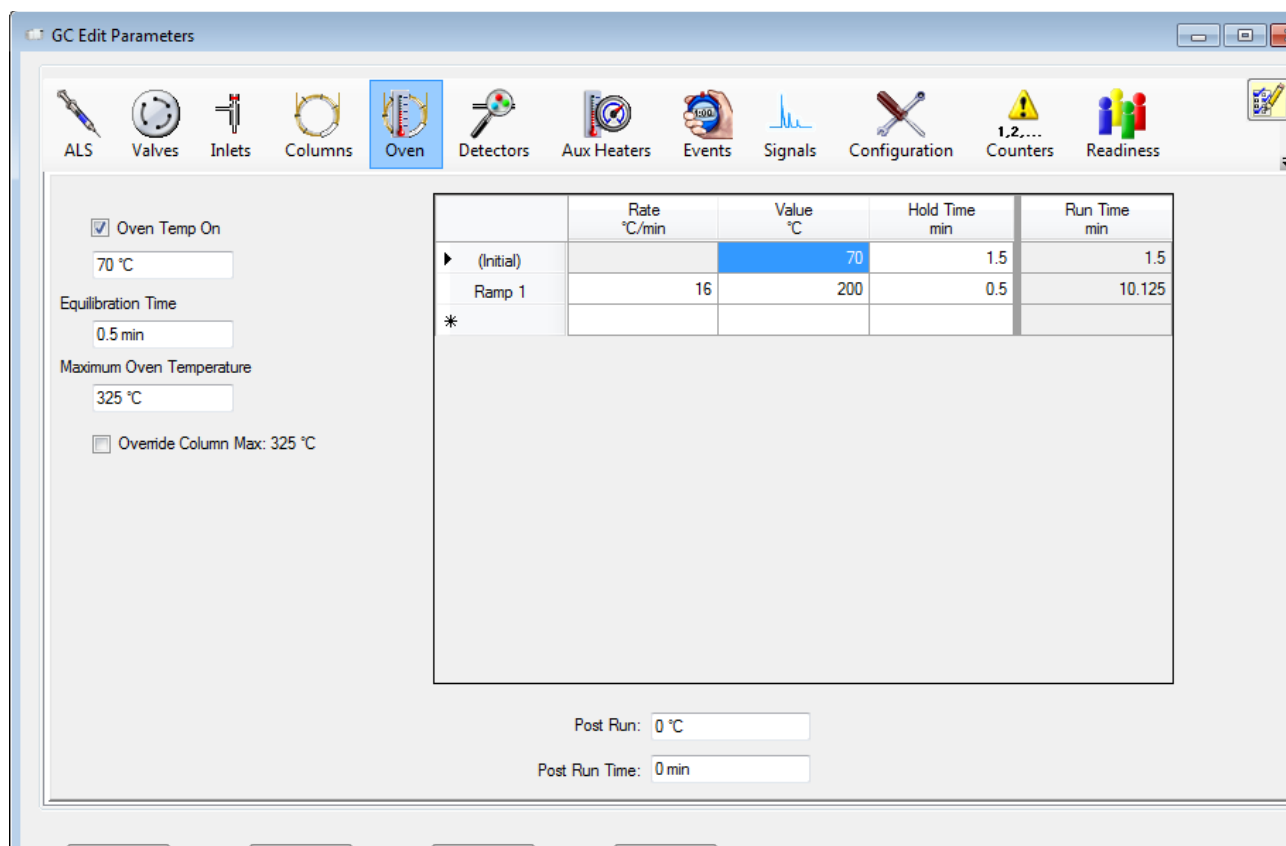


Figure 16 GC Oven panel

GC Detector panel

Select the **Detectors** icon to open the Detectors panel and set parameters that control the front, back, and auxiliary detectors on your GC. The detector control parameters you see are based upon the type of detector you have installed and configured.

For example, if you have a Flame Ionization Detector (FID) detector, you would see a screen similar to this one.

The FID passes sample and carrier gas from the column through a hydrogen-air flame. The hydrogen-air flame alone creates few ions, but, when an organic compound is burned, there is an increase in ions produced. A polarizing voltage attracts these ions to a collector. The current produced is proportional to the amount of sample being burned. An electrometer senses the current, converts it to digital form, and sends the data as output.

Complete the Heater, Air Flow, H2 Flow, Makeup Flow settings as appropriate for your method. See the online Help for more details on each of these settings.

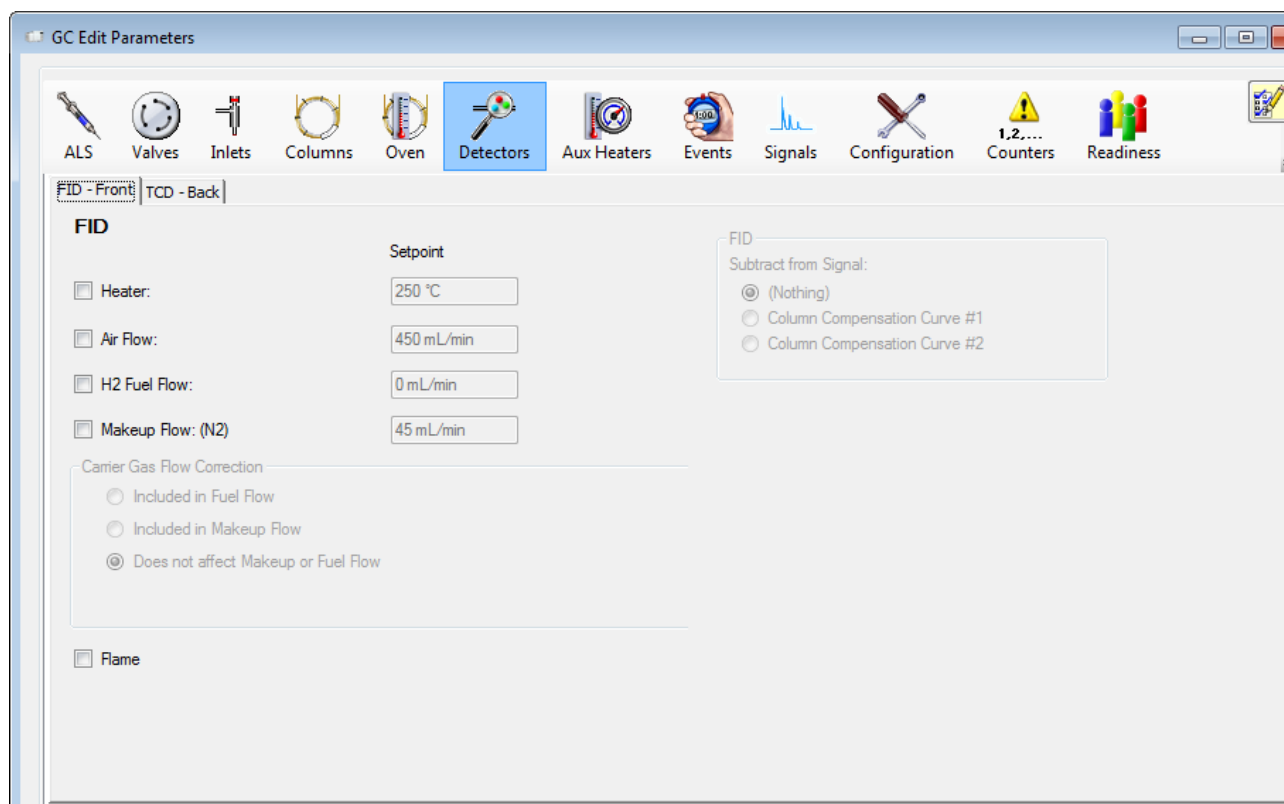


Figure 17 Detector panel

GC Aux Heaters panel

Select the **Aux Heaters** icon to open the **Aux Heaters** panel and set parameters that enable or disable an auxiliary heated zone and control its temperature.

Typically, a Thermal Aux zone is used to control a heated valve box. Other uses include heated transfer lines for a mass spectrometer (MSD) or atomic emission detector (AED).

A given device connected to an auxiliary heated zone may have an upper limit in temperature above which damage could occur. Be sure to enter a temperature that is lower than the column maximum temperature and the temperature that would degrade the compounds you are trying to analyze.

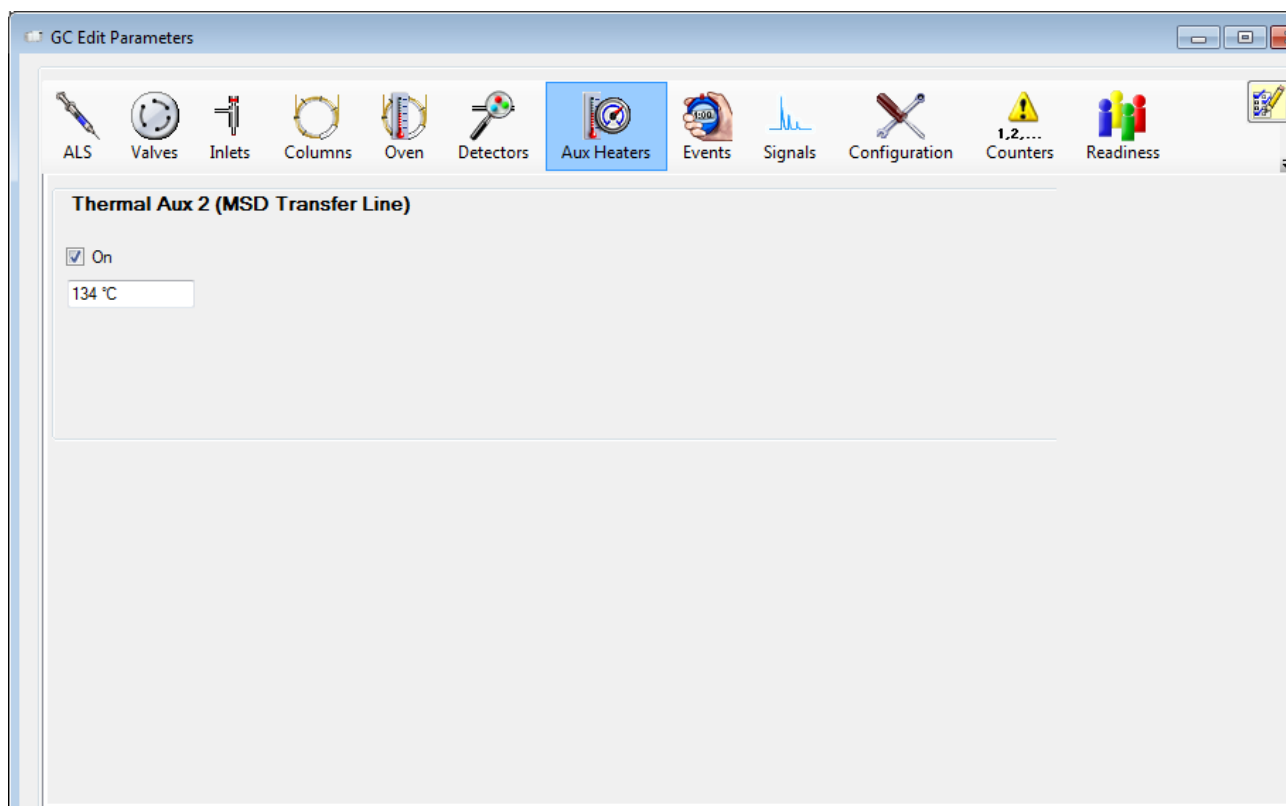


Figure 18 GC Aux Heaters panel

GC Events panel

Use **Events** panel to schedule events to occur automatically during a run. The events available depend on the hardware installed on your GC.

At the end of a run, the program automatically returns all setpoints to their original values, except for Switching Valve state changes. If you rotate a switching valve as a run time event, the program does not automatically return it to its initial setpoint position at the end of the run. You must specify this reset operation as a run time event.

For example, you could:

- Set a new detector fuel gas flow, or turn on/off (if applicable for the detector)
- Turn the valve On/Off
- Change an inlet's septum purge flow, or turn on/off (if applicable for the inlet)
- Use to change the source signal for Signal 1 and Signal 2 analog outputs

Please read online Help for more details on this option.

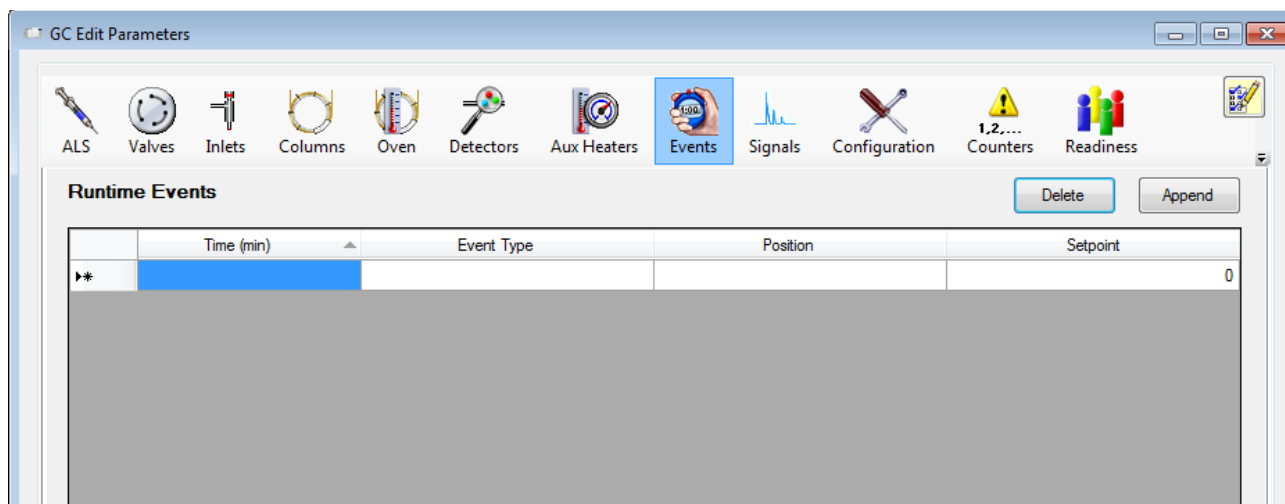


Figure 19 GC Events panel

GC Signals panel

On the **GC Signals panel**, you will identify the source of the signals and the data capture rate, plus what readings you want to display or save in a data file as output of the run.

For the Oven, for example, you can record the actual setpoint, the power, or the duty cycle of the oven. For the front inlet you can record the pressure and the actual setpoints or the inlet flow.

For every place that there are sensors recording data, you can elect to record that data. If you navigate through all of the possible settings here, you will see there are many variations from which to choose.

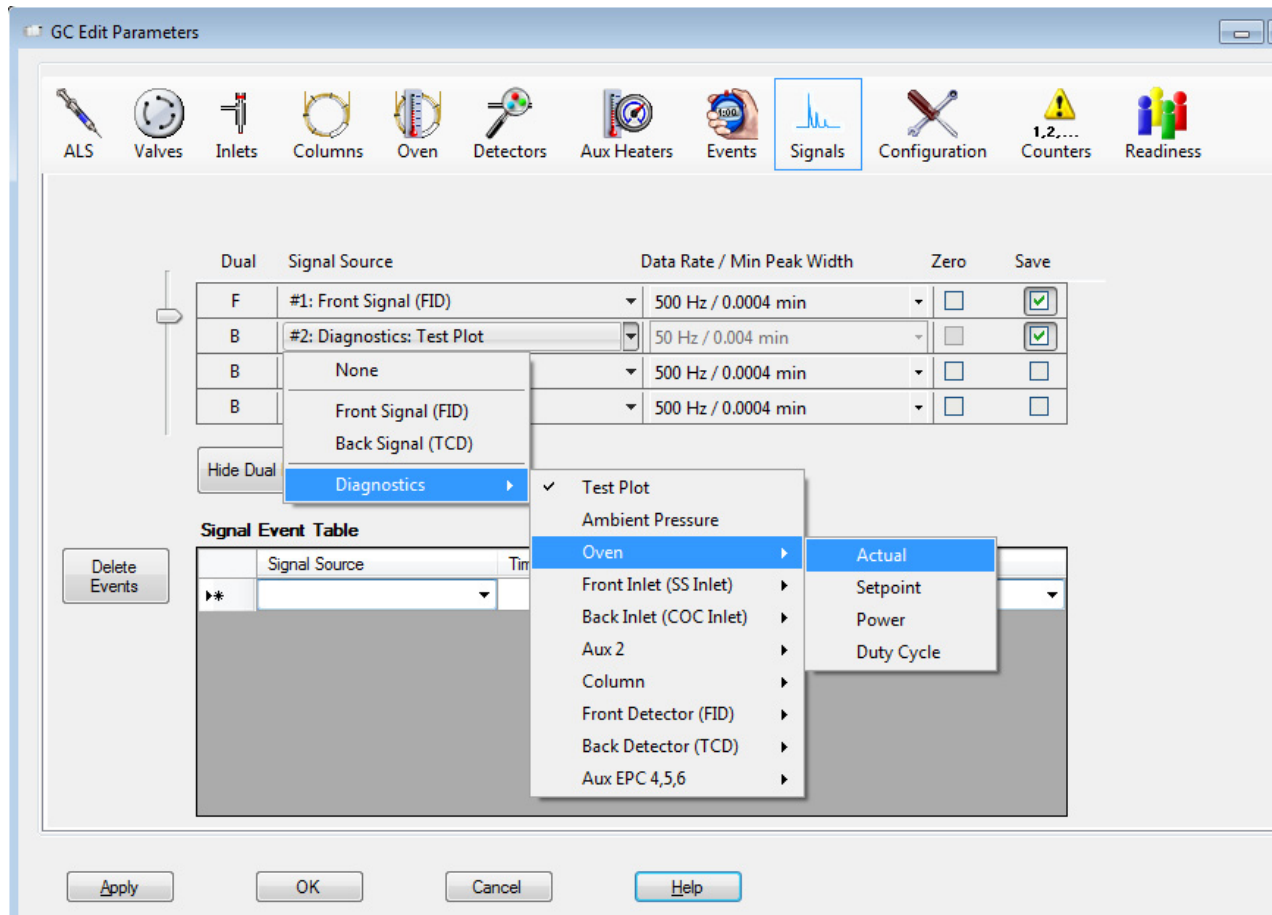


Figure 20 GC Signals panel

GC Counters panel

Use the **Counters** panel to identify the items you want to track as they are used each time this method is processed.

For example, if you are using the Front Inlet Liner and Front Inlet Gold Seal and you would like to keep track of how many times these have been used, you could check these items here. Then, each time this method is run, the total usage counters for these items is tracked by the GC.

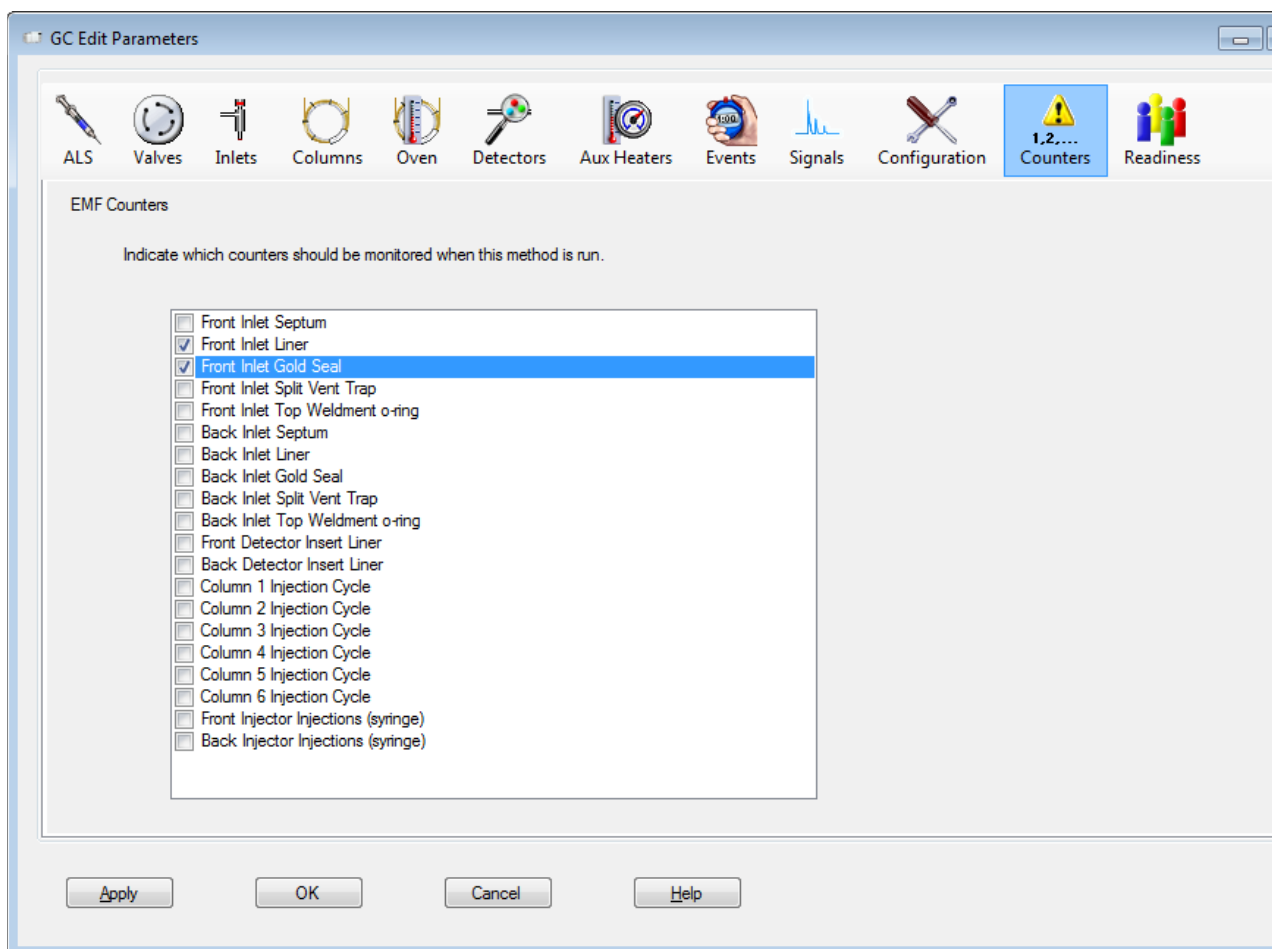


Figure 21 GC Counters panel

GC Readiness panel

Use the **Readiness** panel to select which instrument components your method requires to be *ready* before a run starts.

For example, if you are using the Oven and it needs to be at a specific temperature before the run starts, you should check the Oven checkbox here so the instrument will wait for the oven to reach the specified temperature before posting the **Ready** prompt.

This feature is useful if you wish to ignore the readiness state of hardware that is not used in your method. For example, if you use only the front detector, you do not need to wait for the back detector to reach temperature before starting the run, so you would leave the back detector checkbox unchecked.

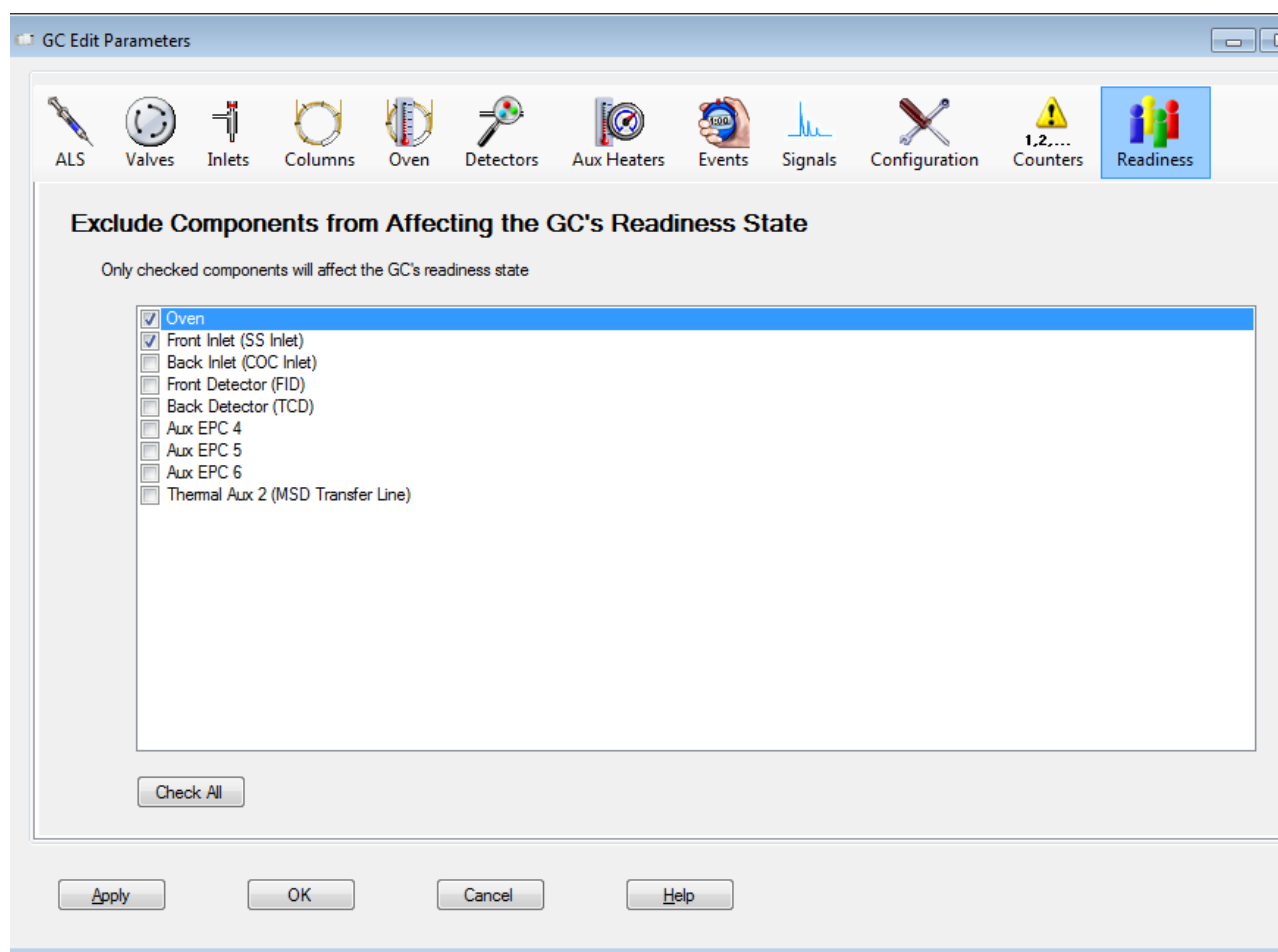


Figure 22 GC Readiness state panel

GC Calculators panel

You can use the **GC Calculator** panel to help you translate methods, pressure flows, solvent vent calculations, etc.

The calculations you receive here are for information purposes only. They are not saved as part of the method.

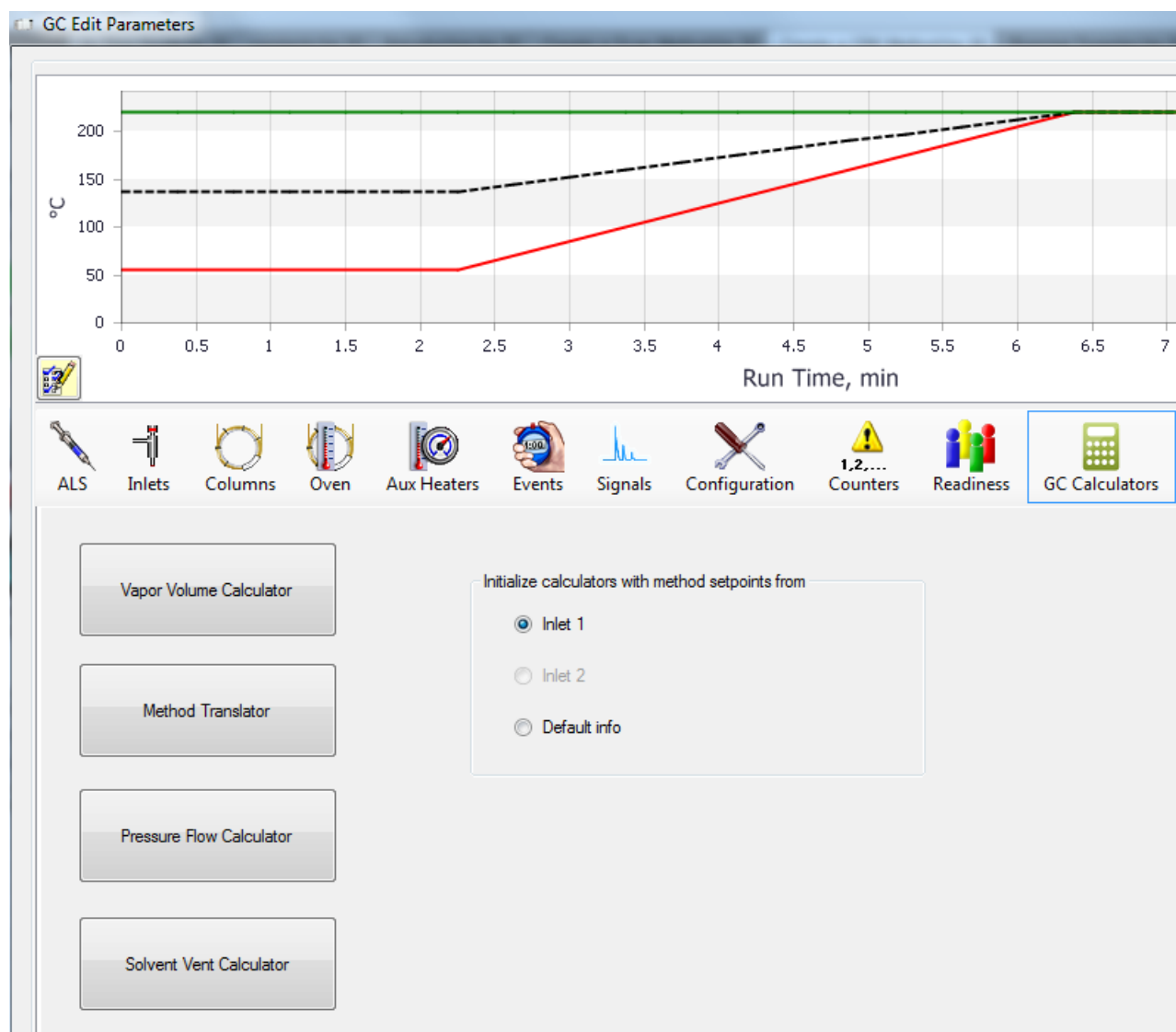


Figure 23 GC Calculators panel

5) Real-Time Plots for GC Dialog

When you have finished entering all your GC parameters, click **OK** to close the GC Edit Parameters window.

The next window that displays as part of the Edit Entire Method routine is the Real-Time Plots dialog.

Use the Real-Time Plots dialog to identify which GC signals you want displayed as real-time plots in the Instrument Control panel during the run.

Select **OK** to save the settings you make here, and close the dialog box. The MS Method Editor dialog displays next.



Figure 24 GC Real-Time Plots dialog

6) MS Method Editor Dialog - Scan Method Setup

This dialog box is used to enter instrument parameters, select parameters to record during the run, and specify the curves to appear as real-time plots for the MS part of a method.

Sections of the MS Editor dialog

The screen capture shown below, shows three general areas:

- **1** Applies to all methods; Scan, SIM, or SIM/SCAN. (See [“Sections to complete for all methods”](#) on page 38.)
- **2** Applies to Scan methods only. (See [“Settings for Scan Methods”](#) on page 42)
- **3** Applies to SIM methods only. (See [“Create a SIM Method - MS Method Editor Dialog”](#) on page 47).

In this example, we are creating a Scan method. So we will be completing the sections labeled 1 and 2.

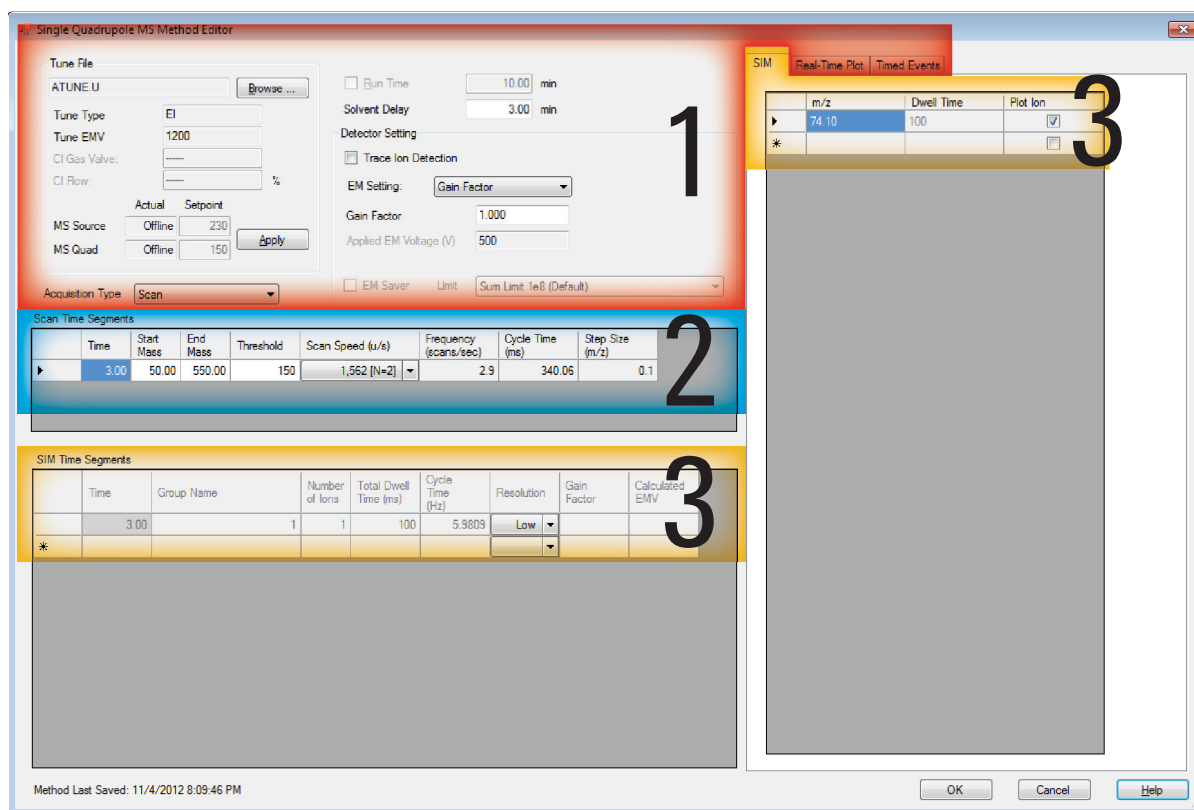


Figure 25 Sections of the MS Method Editor dialog

Sections to complete for all methods

Regardless of the type of method you are creating, SIM, Scan, or SIM/Scan, you will complete the:

Tune File group box	Detector Settings group box
Acquisition Type	Real-Time Plot tab
Run Time	Timed Events tab
Solvent Delay	

Each of these entries are described in this section.

Tune File group box

- The **Tune file name** displays the current tune file selected for the method. Autotune is a good choice. However, you may click **Browse** to select a different one.
- The **Tune Type** - Displays the ion source and polarity configured when the tune file was obtained.
- **Tune EMV**- Displays the EMV setting configured when the tune file was obtained.
- **CI Gas Valve** - Displays the channel selected for reagent gas.
- **CI Flow** - A read only field obtained from the method's tune file.
- **MS Source and MS Quad** - Displays the MS Source and MS Quad setpoint values used during the method's tune and the current temperatures. Click **Apply** to send the MS Source and MS Quad temperature setpoints to the instrument for immediate control to these setpoint values.

Check online Help for more details on the Tune File.

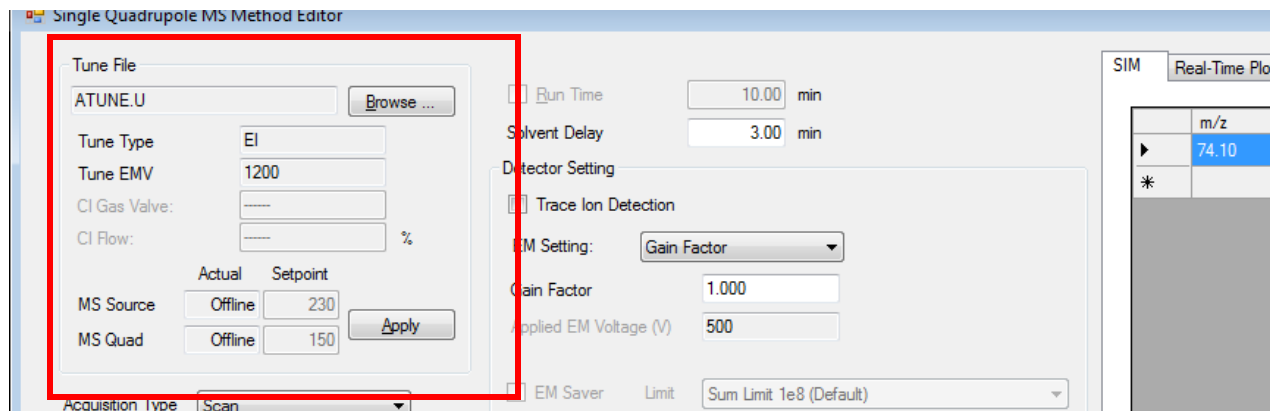


Figure 26 MS Method Editor dialog - Tune File group box

Acquisition Type

Under the **Acquisition Type** dropdown menu, select Scan (RAW) to create a Scan method. Scan stores all data.

Run Time

When the acquisition instrument is configured without a GC, the value entered in the Run time is the time span during which MS data is acquired. MS data is not collected during the solvent delay. If a GC is configured, its run time is used.

Solvent Delay

The solvent delay is the time in minutes after the start of the run when the mass spectrometer is turned on.

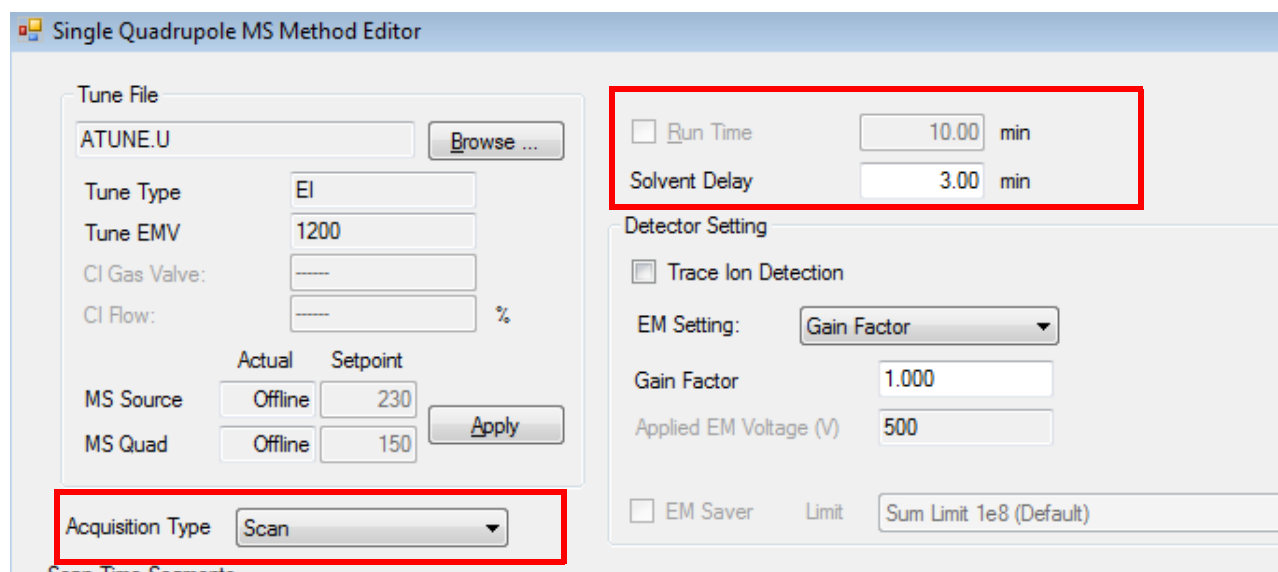


Figure 27 MS Method Editor dialog - Acquisition Type, Run Time, and Solvent Delay

Detector Settings

- Check the **Trace Ion Detection** checkbox to turn this feature ON.

Trace Ion Detection (TID) allows you to obtain lower Method Detection Limits (MDL's) during analysis. It decreases the noise content of Total and Extracted Ion Chromatograms.

The lower noise level results in more reproducible baselines for calculating the peak height and peak area for analyses and standards.

TID also reduces the number of peaks requiring manual integration in complex chromatograms.

Trace Ion Detection will be reported in Print Method.

- Under **EM Setting**, select one of the following:
 - Gain Factor - The default and also the recommended EM.
 - Delta EMV - Delta EMV is not recommended for most applications.
 - Absolute EMV

See online Help for more details on these settings.

- **Gain Factor** - If **Gain Factor** is selected in the **EM Setting** drop-down list, the Gain Factor can be specified here.

An EM gain factor adjusts the signal sensitivity of the detector. EM gain factors have the advantage of remaining constant as the multiplier ages. Therefore, the use of a gain factor produces much higher signal reproducibility for any instrument and better consistency between instruments.

- **Applied EM Voltage** is a read only field that displays the electron multiplier voltage reading of the detector.
- To use **EM Saver**, select this checkbox and then select a **Limit**. This feature is only available in SIM mode.

EM Saver is used to prevent a non-linear detector response at the upper range of a compound's calibration curve. This non-linear response can be caused by setting the gain value or dwell time too high on compounds with high ion concentrations. With EM saver, high gain values and dwell times can be used without saturating the EM.

EM Saver is used with SIM mode in a single quadrupole configuration and MRM mode in a triple quadrupole configuration. A benefit of using EM saver is a more consistent detector response and an extended EM life.

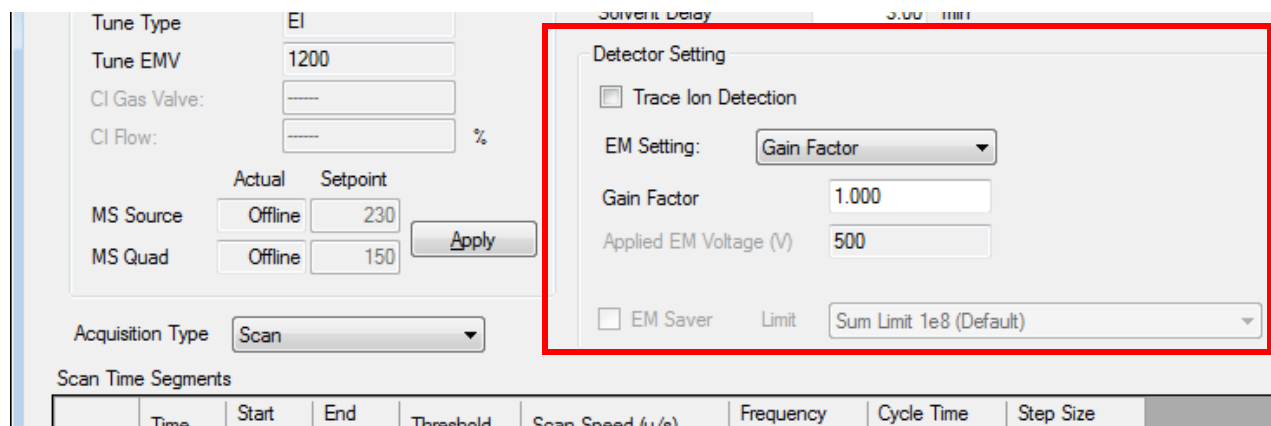


Figure 28 MS Method Editor dialog - Detector Settings

Real-Time Plot tab

Use the **Real-Time Plot** tab, to specify the Spectrum and Chromatogram for display in the real-time plots dialogs in Instrument Control view. Also specify Extracted Ion Chromatograms to display the EIC in the Instrument Control view Chromatogram panel with the label you specify here.

Timed Events tab

Use the Timed Events tab to schedule an event that you want to occur during a specified time in the data acquisition run.

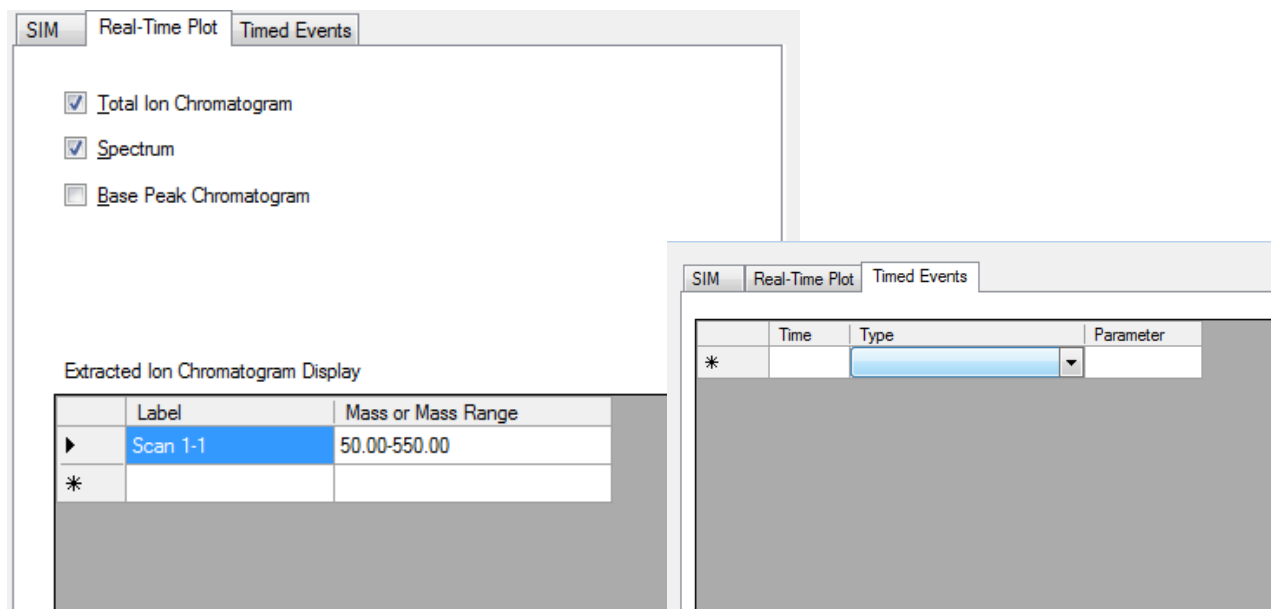


Figure 29 MS Method Editor dialog - Real-Time Plot and Timed Events tabs

Settings for Scan Methods

When you are creating a Scan method, you will complete the sections labeled 1 and 2 of the MS Editor dialog (shown in [Figure 30](#)).

- Items included in Section 1 were described in “[Sections to complete for all methods](#)” on page 38.
- The following describes how to complete the entries for Section 2, which apply to Scan methods.
- Section 3 is for creating SIM methods, and is discussed in [Chapter 3](#), “Create a SIM Quantitation Method” on page 45.

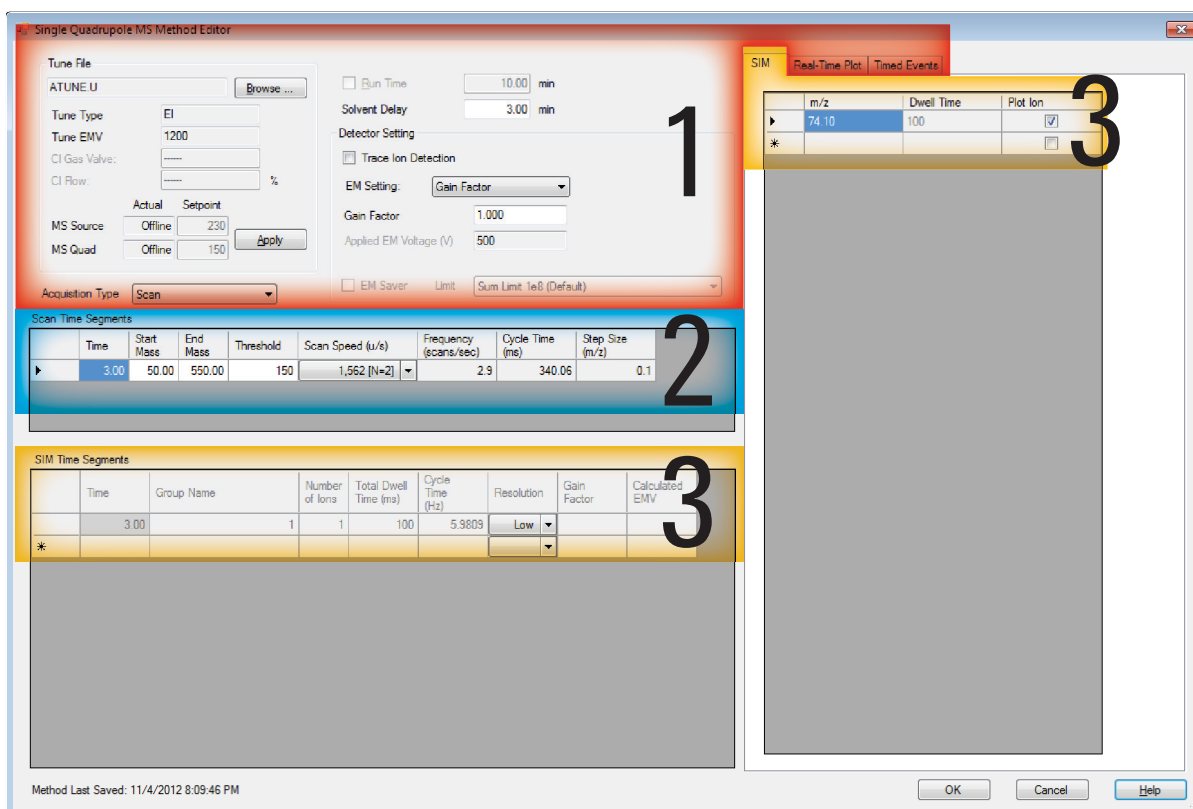


Figure 30 Sections of the MS Method Editor dialog

Scan Time Segments group box

In the **Scan Time Segments** group box (shown in [Figure 31](#)) you will define the time segments for the Scan run.

- The **Time** field displays what is entered in the **Solvent Delay** field. This is the time at which the mass spec will turn on and start gathering data.
- In the **Start Mass** and **End Mass**, enter the mass range containing your ion of interest for this time segment.
- In the **Threshold** field, enter the abundance value below which data is considered noise, and should be ignored.
- From the **Scan Speed** dropdown menu select the atomic mass units you wish to scan in a second.

Click **OK** when you have entered your MS method settings, and the Select Monitors dialog box is displayed.

Acquisition Type: Scan

☐ EM Saver Limit: Sum Limit 1e8 (Default)

Scan Time Segments

	Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
▶	3.00	50.00	550.00	150	1,562 [N=2] ▼	2.9	340.06	0.1

Figure 31 Scan Time Segments group box

7) Select Monitors Dialog

Use the **Select Monitors** dialog to identify the GC and MS monitors you wish to see displayed in the Instrument Control view.

After selecting the monitors you want to see, click **OK** to complete the process. The Save Method dialog box is automatically displayed.

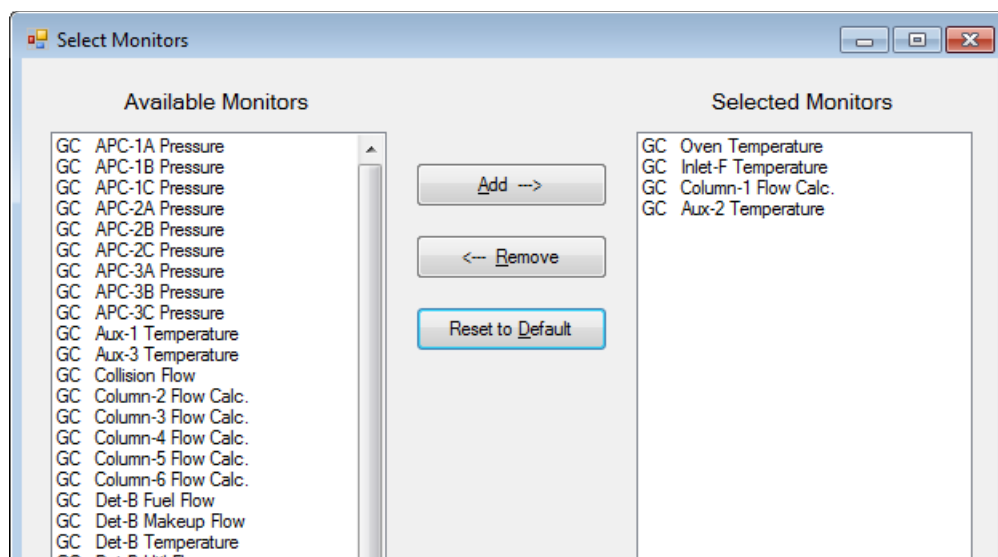


Figure 32 Select Monitors dialog

8) Save The Method

This is the final step in the Edit The Entire Method process.

Type a name for your method and click **OK**.

This completes the Edit Entire Method process. Your method is now saved.

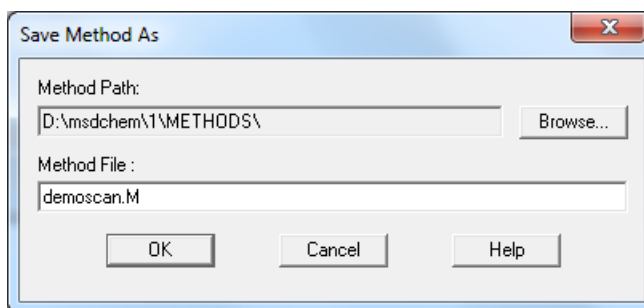
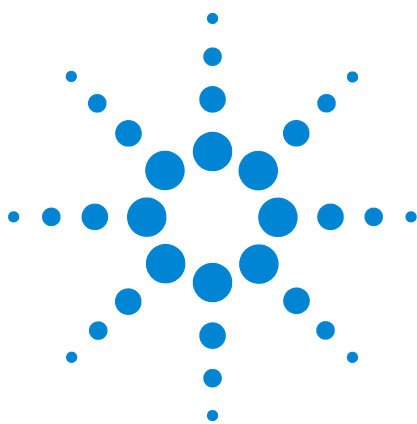


Figure 33 Save Method As dialog



3 Create a SIM Quantitation Method

Introduction	46
Create a SIM Method - MS Method Editor Dialog	47
Simultaneously Acquire Scan and SIM Data (SIM/Scan Mode)	49
SIM/Scan Mode Cycle Frequency	50

This chapter describes how to create a SIM method by revising an existing Scan method.



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 (SIM Time Segments) of up to 60 ions each for selected ion monitoring, however, Agilent recommends you use as few ions in a group 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 (Start a new Time Segment) where the peaks are well separated to avoid variations in retention time due to sample matrix effects.

Create a SIM Method - MS Method Editor Dialog

When you create a SIM method, you will complete all the common settings (labeled as 1 in [Figure 25](#) on page 37), plus the SIM Time Segments settings (labeled as 3 in [Figure 25](#) on page 37), and described here.

Previously we accessed the Single Quadrupole Method Editor dialog automatically during the Edit an Entire Method procedure. This time we will access this dialog directly since we are not changing other parts of the method.

To begin the process:

- 1 Click the **Load Method** icon or select **Method/Load Method**. 
- 2 Navigate to and select the method you want to start with (e.g., the Scan method created in Chapter 2), and click **OK**.
- 3 Click the MS Instrument Parameters icon, or select **Instrument/MS Edit Parameters...** to open the Single Quadrupole Method Editor dialog. 
- 4 See [“Sections to complete for all methods”](#) on page 38 for details on filling out the general method settings (those that are common to both SIM and Scan).
- 5 Under the **Acquisition Type** dropdown menu select SIM and then specify the SIM parameters for each time segment that you define in the table below.
- 6 In the SIM Time Segments group box enter the SIM parameters for the run's first time segment when the Acquisition type is set to SIM or SIM and Scan analysis.

	Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
	3.00	50.00	550.00	150	1,562 [N=2]	2.9	340.06	0.1

	Time	Group Name	Number of Ions	Total Dwell Time (ms)	Cycle Time (Hz)	Resolution	Gain Factor	Calculated EMV
▶	3.00		1	3	400	2.3719	Low	500
*								

Figure 34 MS Method Editor dialog- SIM Time Segments

Assign a Group Name for this time segment, select a Resolution of High or Low, and if needed enter a different value for the specified EM Setting (Gain Factor, Absolute EMV, Delta EM) for this time segment only. If you do not enter a value here the EM Setting specified in the Detector Settings area located in labeled area 1 is used. Next specify the ions to acquire in this time segment.

- 7 Select the SIM tab and specify each ion to acquire for this time segment. For each ion, in the Dwell Time column enter the time in ms to acquire the ion's abundance. Select Plot to have the EIC displayed in the Instrument Control view Chromatogram panel labeled with the ion m/z.

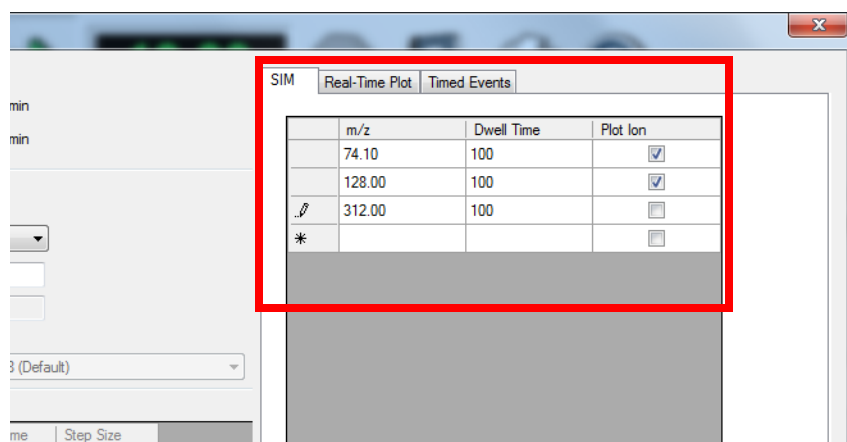


Figure 35 MS Method Editor dialog- SIM tab

- 8 To add additional SIM time segments, right-click within the SIM Time Segments area and from the context menu select Add. In the new time segments Time column enter the starting time for this segment which is also the ending time for the previous time segment.

Enter the acquisition parameters that define this time segment and enter the ions to acquire as above.

- 9 Exit the Single Quadrupole Method Editor dialog by clicking OD and then save the method.

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

An MSD can simultaneously acquire SIM and Scan data if it is equipped with fast electronics like the Agilent 5977 MSD and 5975 MSD (fast electronics model). The cycle time displayed for both Scan and SIM are not corrected for simultaneous acquisition. See [“SIM/Scan Mode Cycle Frequency”](#) on page 50.

- 1 Open the Single Quadrupole Method Editor dialog.
- 2 From the Acquisition Type dropdown list, select SIM and Scan and notice that the areas for entering both the SIM data and the Scan data are in edit mode..

The screenshot displays the Single Quadrupole Method Editor dialog. The Acquisition Type is set to "SIM and Scan". The Scan Time Segments table shows a segment from 3.00 to 550.00 with a cycle time of 340.06 ms. The SIM Time Segments table shows a segment from 3.00 to 500 with a cycle time of 3.1095 Hz. The SIM Real-Time Plot table shows peaks at m/z 74.10, 128.00, and 312.00.

Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
3.00	50.00	550.00	150	1,562 [N=2]	2.9	340.06	0.1

Time	Group Name	Number of Ions	Total Dwell Time (ms)	Cycle Time (Hz)	Resolution	Gain Factor	Calculated EMV
3.00		1	3	300	3.1095	Low	500

m/z	Dwell Time	Plot Ion
74.10	100	<input checked="" type="checkbox"/>
128.00	100	<input checked="" type="checkbox"/>
312.00	100	<input type="checkbox"/>

Figure 36 Scan and SIM data entry both active

- 3 Edit the Scan and SIM acquisition parameters, as needed. Refer to [“6\) MS Method Editor Dialog - Scan Method Setup”](#) on page 37 and [“Create a SIM Method - MS Method Editor Dialog”](#) on page 47.
- 4 At a time during the run where you require the number of peak data points generated, write down both the Scan and SIM cycle times in the time segments that bracket this time.

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 in [“SIM/Scan Mode Cycle Frequency”](#) on page 50.

- 5 Save the method.

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 37](#).

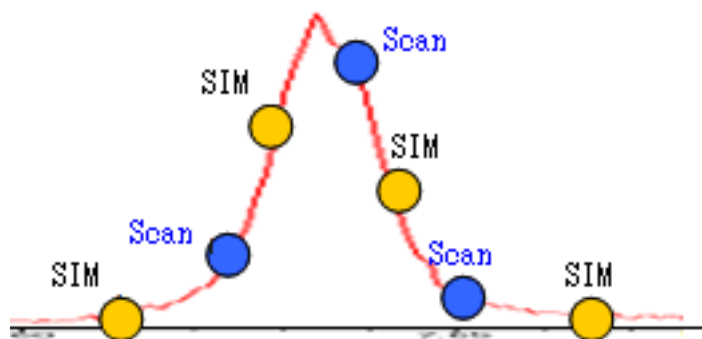


Figure 37 SIM/Scan mode

Actual cycle frequency is calculated with the equation in [Figure 38](#).

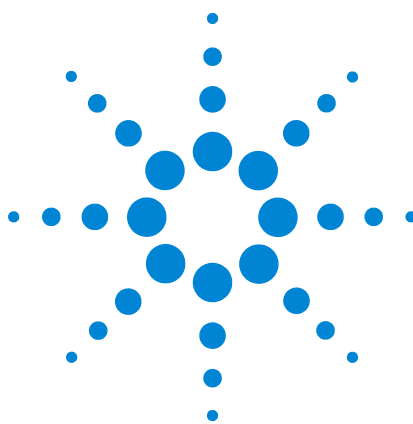
$$\text{SIM/Scan Cycle Frequency} = \frac{1}{\left(\frac{1}{A} + \frac{1}{B}\right) \times 1.05}$$

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

Figure 38 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.



4 Running Samples

Running a Single Sample [52](#)

Running a Sequence [55](#)



Running a Single Sample

The following describes how to run a single sample in MassHunter Data Acquisition.

Load the Method

- 1 From the PC desktop, click the MassHunter shortcut icon. The **Instrument Control** window below opens.
- 2 Click the **Load Method** icon (red box 1) to open the **Load Method** dialog and select the method you want to use to acquire data. Click **OK** to load the method and close the dialog. The method name is displayed in the title bar of the Instrument Control window (red box 4).

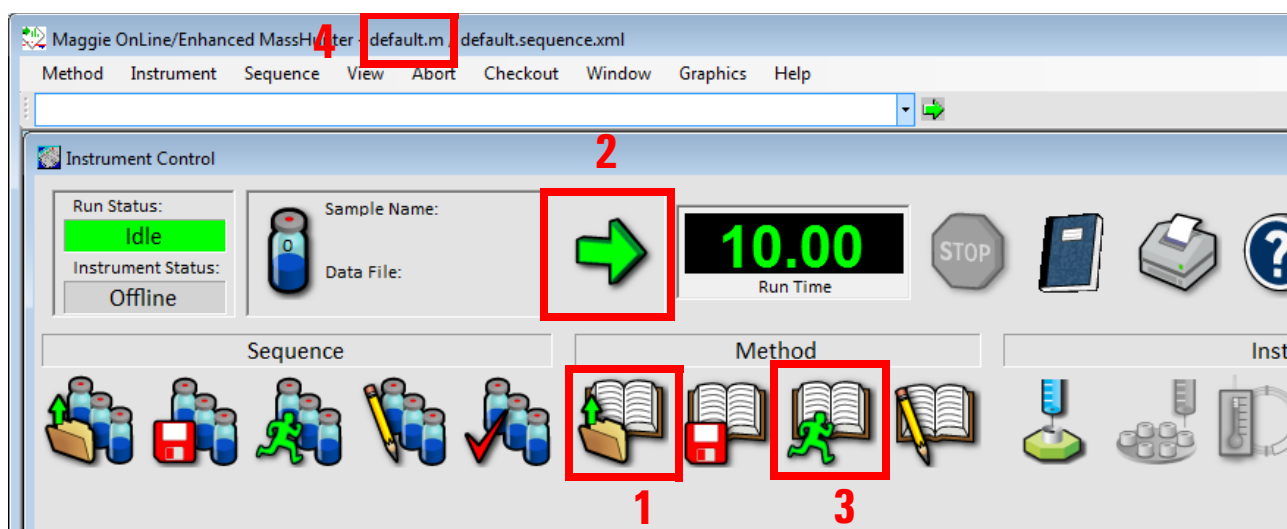


Figure 39 Instrument Control window

Run the Method

For an automated injection, with your method loaded and a sample vial loaded in the ALS, begin the run as follows:

- 1 Click the **Run Method** icon (red box 1 or 3). The **Start Run** dialog box opens.
- 2 Enter the method parameters in the start run dialog for this sample run.

Basic Tab

The top part of the dialog displays the parameters specified by the method (See [Figure 40](#) on page 53). These are read-only fields. To modify these entries you need to edit the method. Included are the:

- **Current Method Inject Style**
- **Inlet Location**
- **MS Connected To**

Start Run

Basic Advanced

Current Method Injection Style: Manual

Inlet Location: ☒ Front ☐ Rear ☐ Dual

MS Connected to: ☒ Front Inlet ☐ Rear Inlet

Operator Name: John Smith

Data Path: C:\MassHunter\GCMS\2\DATA\ Browse...

Sample

Data File Name: Maggie OnLine_001.D Browse...

Sample Name:

Misc. Info:

Expected Barcode:

Sample Amount: 0

Multiplier: 1

Vial Number: 1

Tray Name:

Injection Volume: ☐ Current Method μ L ☒ Override using 1 μ L

Data

Data File Name: EVALDEMO.D Browse...

Sample Name:

Misc. Info:

Expected Barcode:

Sample Amount: 0

Multiplier: 1

Vial Number:

Tray Name:

Injection Volume: ☐ Current Method μ L ☒ Override using μ L

Method Sections to Run:

☒ Data Acquisition

☐ Data Analysis (MassHunter DA)

OK and Run Method Exit Cancel Help

Figure 40 Start run dialog

Operator Name - Optional. Enter the name of the person performing the run. This name will be stored in the data file.

Data Path - The current data path is displayed. If you wish to have data files placed in a different data file directory, click the Browse button and select the new directory.

Data File Name - Required. The file used for data storage. You can also click the Browse button to select a different data file name.

Sample Name - Optional. This is the name you give to your sample. This field is informational only and is stored in the data file.

Misc Info - Optional. Enter any additional information about the sample or the method. This field is stored in the data file.

Expected Barcode - Optional. Enter the barcode for the current sample. This is greyed out if a barcode reader is not installed.

Sample Amount - A divisor to convert the absolute amounts of compounds to a percentage of the original sample. If you are generating a quantitative report, this item may be needed.

Multiplier - A multiplier to correct for dilution or other sample handling adjustments. If you are generating a quantitative report, this item may be needed.

Vial Number - This is the vial number containing your sample.

Tray Name - Some ALS models have multiple trays. Select a tray name from the drop-down list if configured.

Injection Volume - Click the injection volume option. You may select the volume from the current method, or specify a sample volume by clicking the Override option.

Method Sections to Run - This section allows you to specify whether you want to run a Data Acquisition method, a Data Analysis method, or both.

- 3 When the instrument is in a ready state as shown by a green **Ready** indicator in the instrument status, click **OK** and **Run Method** to close the dialog and start the run. The ready state indicator changes to **Run**.

Running a Sequence

A sequence is a list of samples to be analyzed automatically without operator intervention. Each sample is assigned a method to be used for analysis.

To begin the process you must first create a sequence.

Create the Sequence



- 1 Click the **Edit Sequence** icon . The **Sequence Table** opens. This is where you will create the list of samples to be analyzed and designate the method to be used for each analysis.
- 2 To select the cells you wish to edit in the **Sequence Table**:
 - Left-click in a cell to select it
 - Click and drag to select multiple cells
 - Select a series of cells by left-clicking the first cell, holding **Shift**, and left-clicking the final cell
 - Select specific multiple cells by holding the Ctrl key while left-clicking each cell
 - Select a rows of cells by left-clicking in the grey column on the left
- 3 To add lines to the table, with a line in the table selected, click the **New Sample(s)** button. From the drop down menu that is displayed, select **1 Sample** to insert one new sample or **5 Samples** to insert 5 new samples at the bottom of the table, or select **N Samples** to specify the number of samples to add to the table. For this example, we will just use the 3 default lines provided.

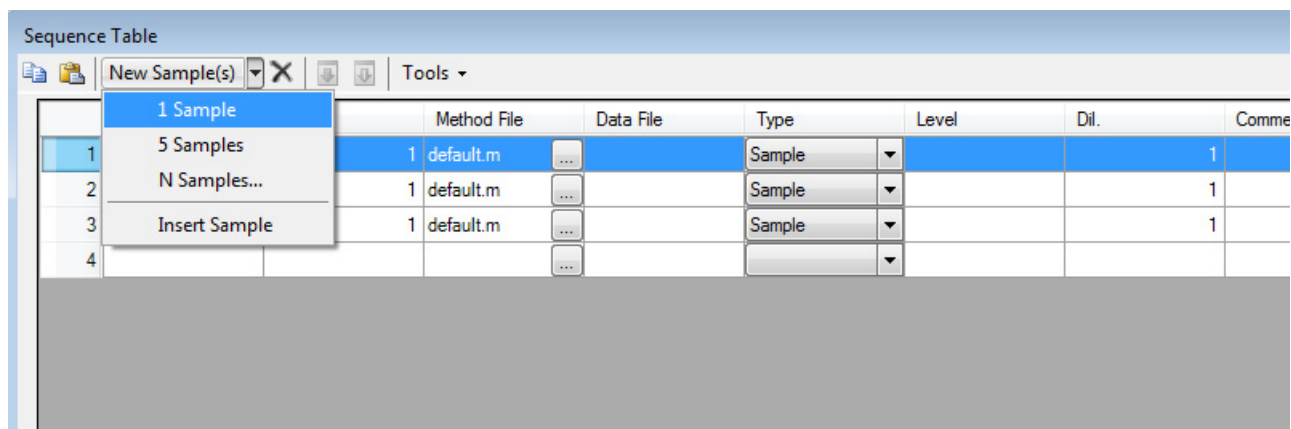

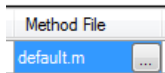

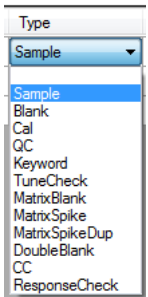


Figure 41 Sequence Table with default entries

- 4 In the **Name** column, type the name for your sample.
- 5 To increment this value down through all the subsequent lines in this table, select the cell with the information you want to increment, and click the **Increment** icon. 
- 6 Under the **Vial** column, enter the location of the sample(s) in the ALS tray. You can use the **Increment** icon here too.
- 7 Under the **Method File** column, select the ellipses and browse to the method you want to use to acquire the data from this sample. 
- 8 To copy this method name down to all the subsequent cells, select the cell and click the **Copy Down** icon. 
- 9 In the **Data File** column, enter the name you want to be used for the data collected from this sample. Use the **Increment** icon here, if desired.
- 10 From the **Type** drop down box, select the applicable sample type for each line in your table. This entry identifies how the sample is processed by the MassHunter Data Analysis programs. For this example, we are using the type called **Sample**. 








Sequence Table									
    Tools ▾									
	Name	Vial	Method File		Data File	Type	Level	Dil.	Comm
1	Benzo021409_1	1	default.m		Benzo021409_1	Sample ▾			1
2	Benzo021409_2	2	default.m		Benzo021409_2	Sample ▾			1
3	Benzo021409_3	3	default.m		Benzo021409_3	Sample ▾			1

Figure 42

- 11 When you have finished entering all the samples you want to run in this sequence, click the **OK** button to close the Sequence table. You are returned to the main MassHunter Data Acquisition window.

Save the Sequence



- 1 Select the **Save Sequence As...** icon. The **Save Sequence** dialog box opens.
- 2 In the **File name** field, type a name for your sequence.
- 3 Click **Save**. The dialog box closes and the sequence is saved.

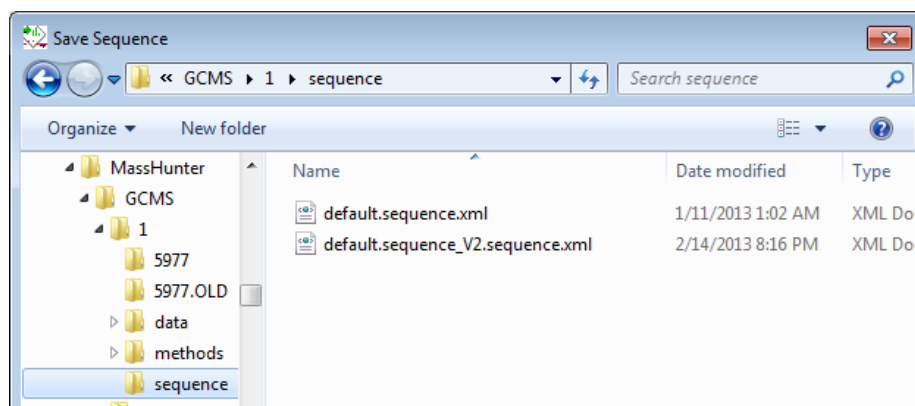


Figure 43 Save Sequence

Load the Sequence



- 1 Select the **Load Sequence** icon.
- 2 Navigate to and select the sequence you want to load, or type the name in the **File Name** field.
- 3 Click **Open** to close the dialog box and load the sequence.

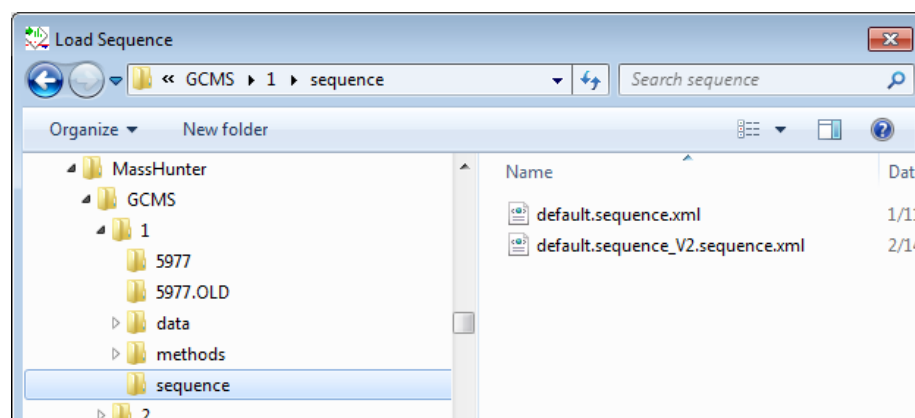

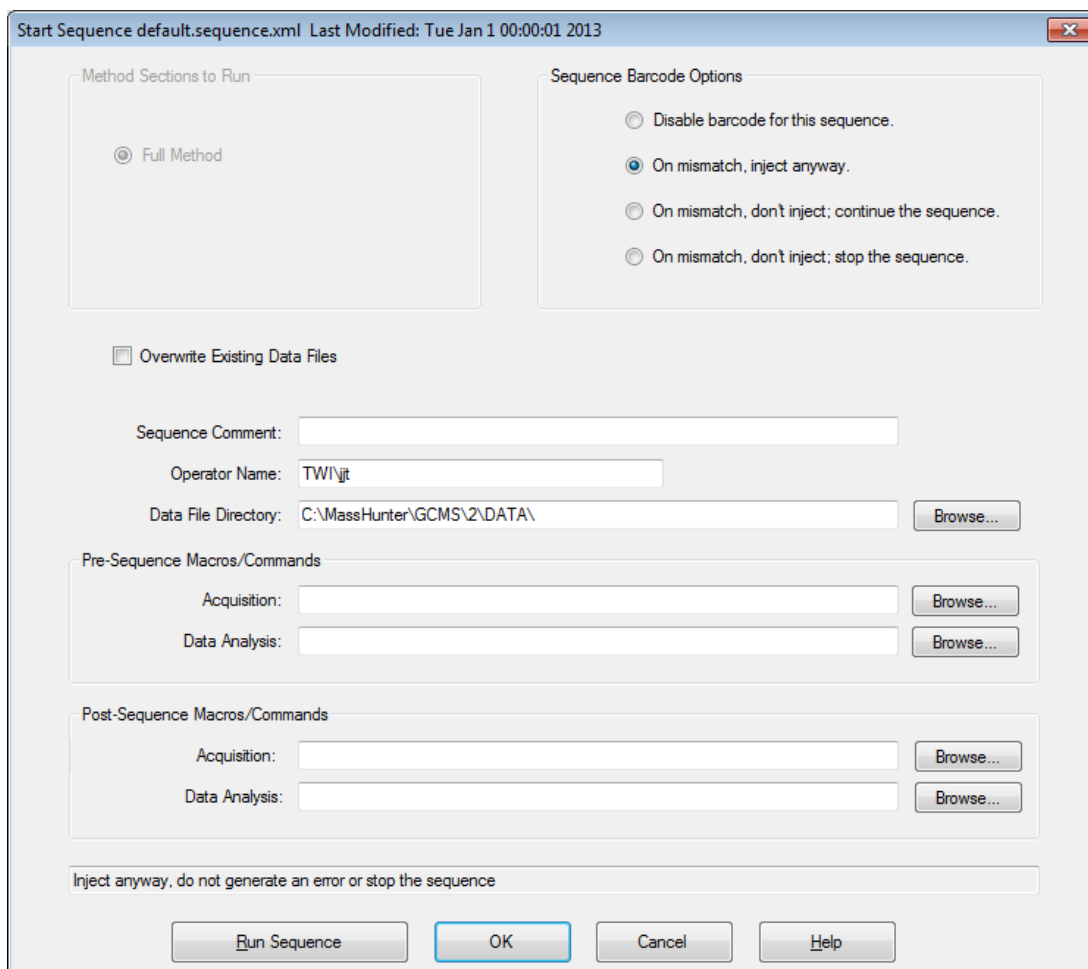


Figure 44 Load Sequence

Run the Sequence

- 1 Click the **Run Sequence** icon . The **Start Sequence** dialog box opens. This dialog is used to set some general parameters to control the sequence process.
- 2 In the **Sequence Comment** field, enter a description of the sequence.
- 3 In the **Operator Name** field, enter your name.
- 4 In the **Data File Directory** field, accept the default location for this data file, or click **Browse** to select a new path.
- 5 The **Pre-Sequence** and **Post Sequence Macros/Commands** options let you specify data acquisition or data analysis macros or commands to run before or after the sequence.



Start Sequence default.sequence.xml Last Modified: Tue Jan 1 00:00:01 2013

Method Sections to Run

☒ Full Method

Sequence Barcode Options

☐ Disable barcode for this sequence.

☒ On mismatch, inject anyway.

☐ On mismatch, don't inject; continue the sequence.

☐ On mismatch, don't inject; stop the sequence.

☐ Overwrite Existing Data Files

Sequence Comment:

Operator Name:

Data File Directory:

Pre-Sequence Macros/Commands

Acquisition:

Data Analysis:

Post-Sequence Macros/Commands

Acquisition:

Data Analysis:

Inject anyway, do not generate an error or stop the sequence

Figure 45 Start Sequence dialog

- 6 When you have completed your selections, click **Run Sequence**.

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.

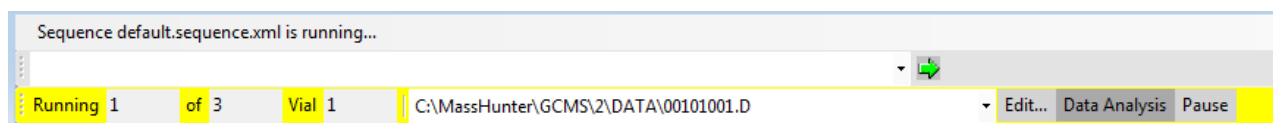



Figure 46 The sequence status bar

Print the Sequence Log



- 1 Select the **Print** button, . The **Select Items to Print** dialog box opens.
- 2 Mark the **Sequence Log** checkbox.

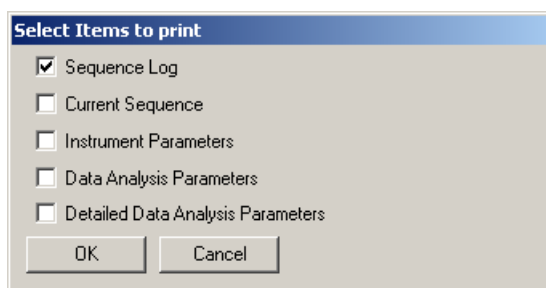


Figure 47 Select Items to print

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

4 Running Samples

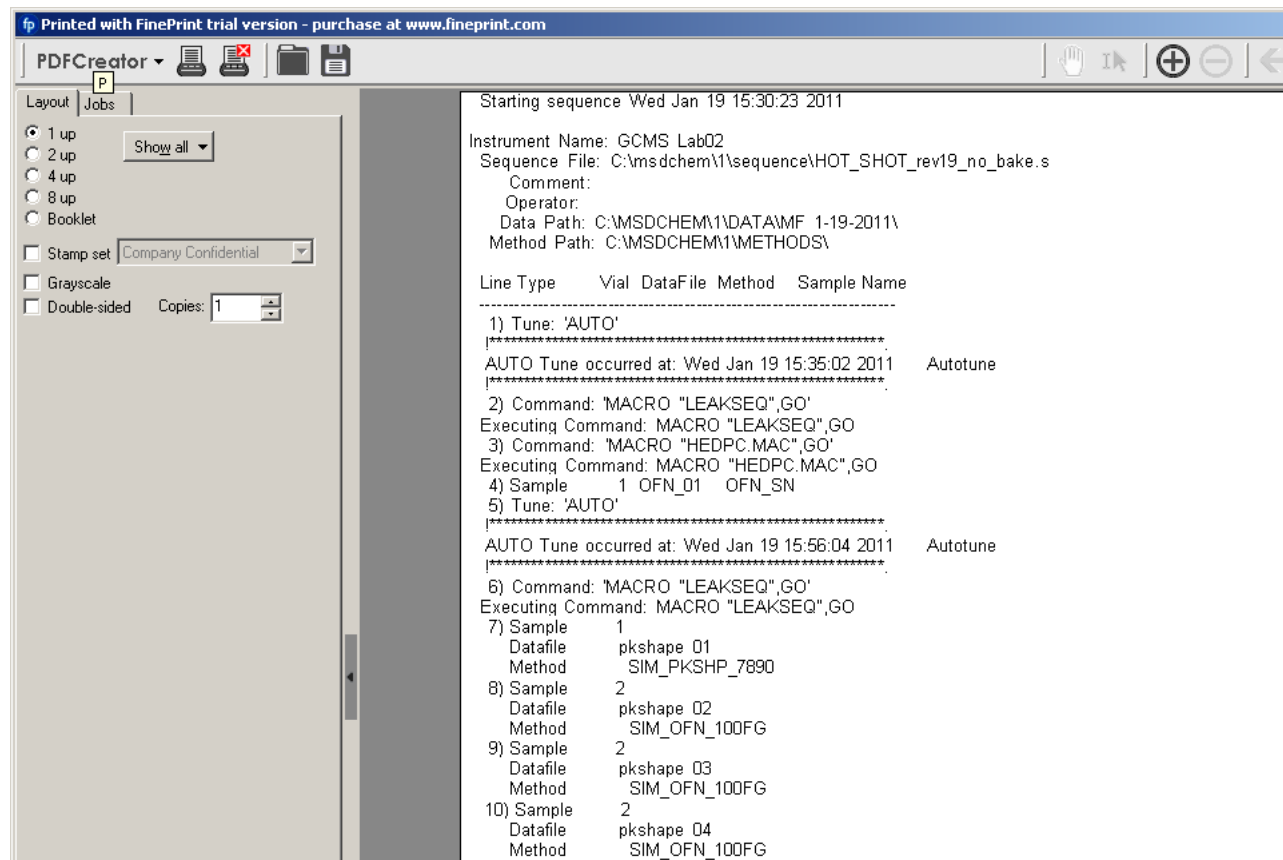
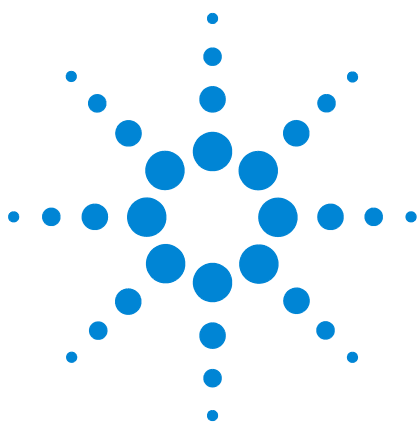


Figure 48 Printed sequence log



5 General Information

- Editing Individual GC Parameters [62](#)
- Add a Column to Your Local Column Inventory [63](#)
- Upload Parameters from the 7890X GC [66](#)
- Customize the Status Panel View [66](#)



Editing Individual GC Parameters

Although a good way to edit a method is use the Edit Entire Method option, as described in [Chapter 2](#), “Create a Scan Method”, you may edit any single part, outside the Edit Entire Method option, using the following procedure.

- 1 From **Instrument Control** select the **GC Parameters** icon to display the **GC Edit Parameters** window.
- 2 Click on any icon to open the corresponding dialog and enter changes as required.

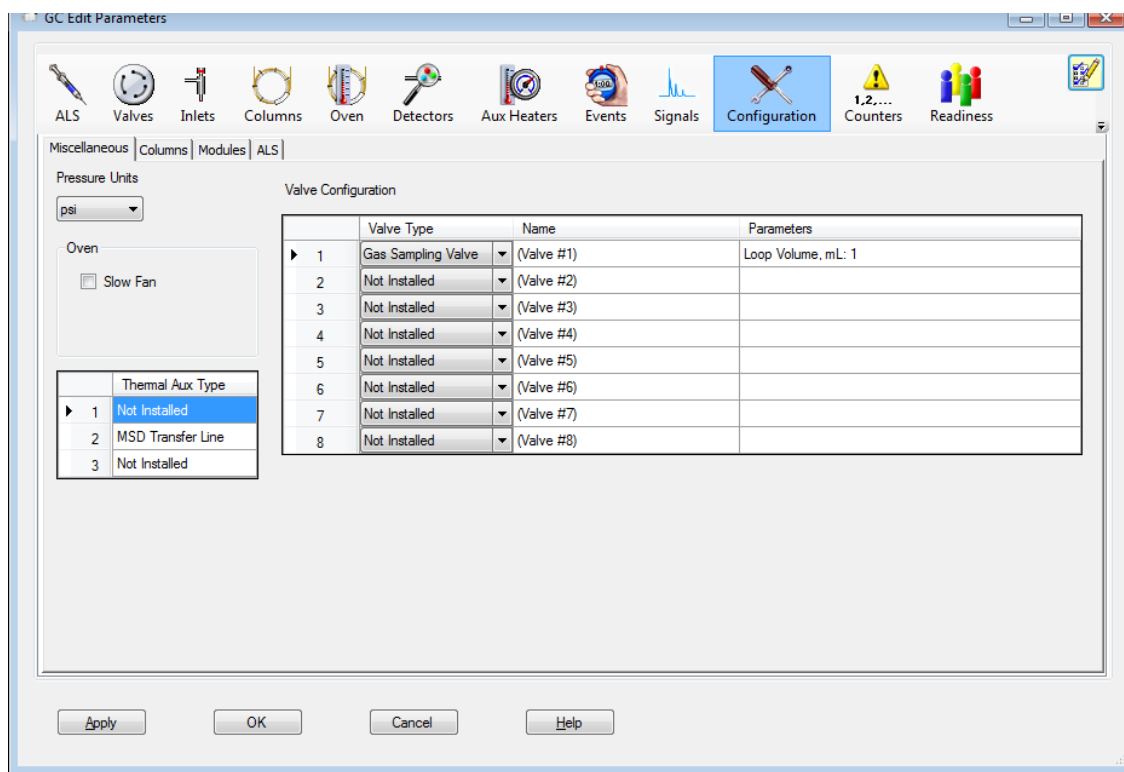


Figure 49 GC Edit Parameters window

- 3 Apply or cancel your changes as needed. See [Table 1](#) on page 63 for a description of the **GC Edit Parameters** window buttons.

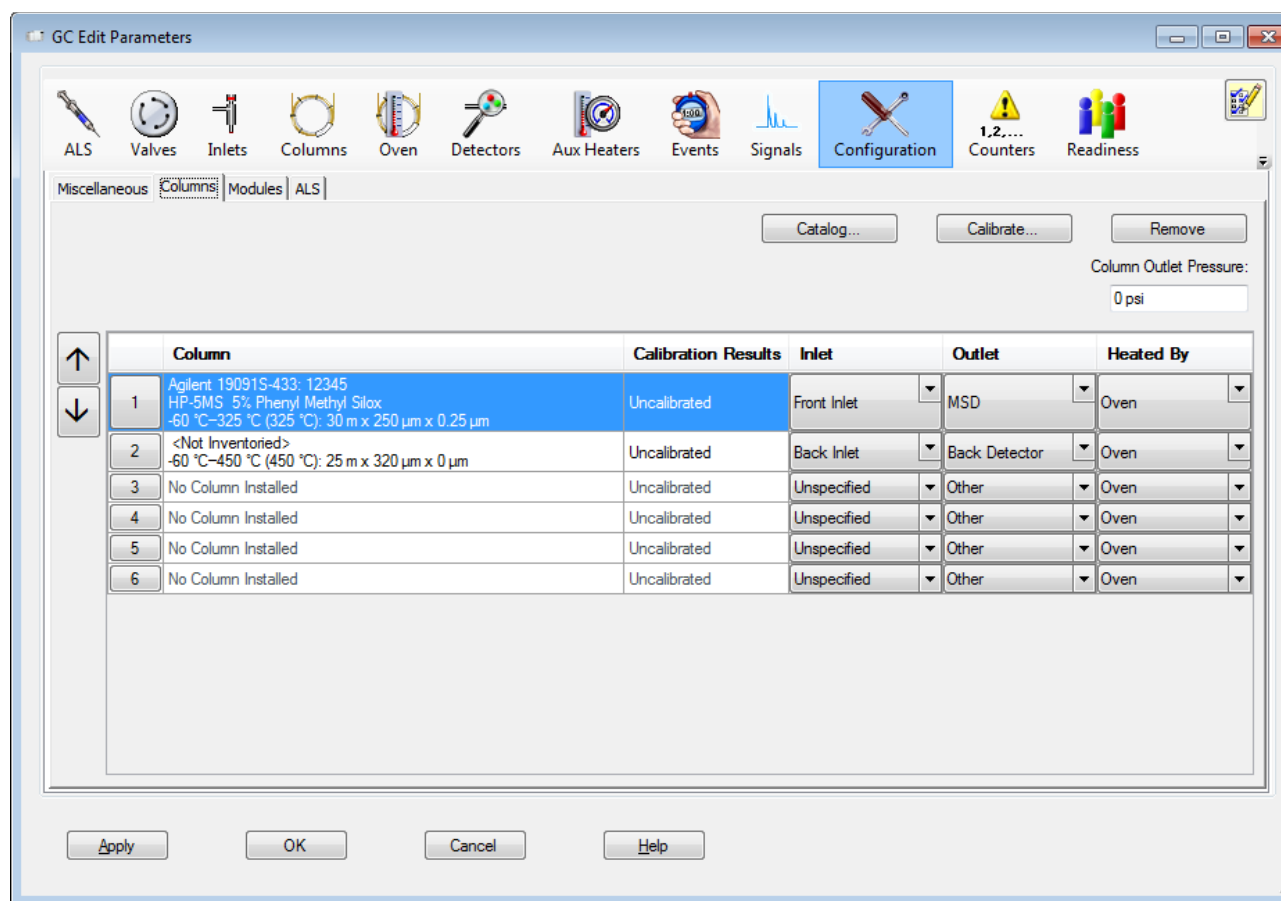
Table 1 GC Edit Parameters window buttons

Button	Action
Apply	Downloads any settings that have been changed to the GC.
OK	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 Your Local Column Inventory

Use the **Catalog** option to select a column from the **Column Catalog** and add it to your **Local Column Inventory**.

- 1 Select the **Configuration** icon, then the **Columns** tab to display the columns configured for the instrument.

**Figure 50** Columns configured for the instrument

- 2 Click **Catalog** button to display a local database that is shared across all instrument sessions on your data system. The database contains popular column models, plus any custom columns entered.
- 3 Use the GC Column Catalog to select a column model to add to your Column Inventory, or to select a column model already entered in your inventory.

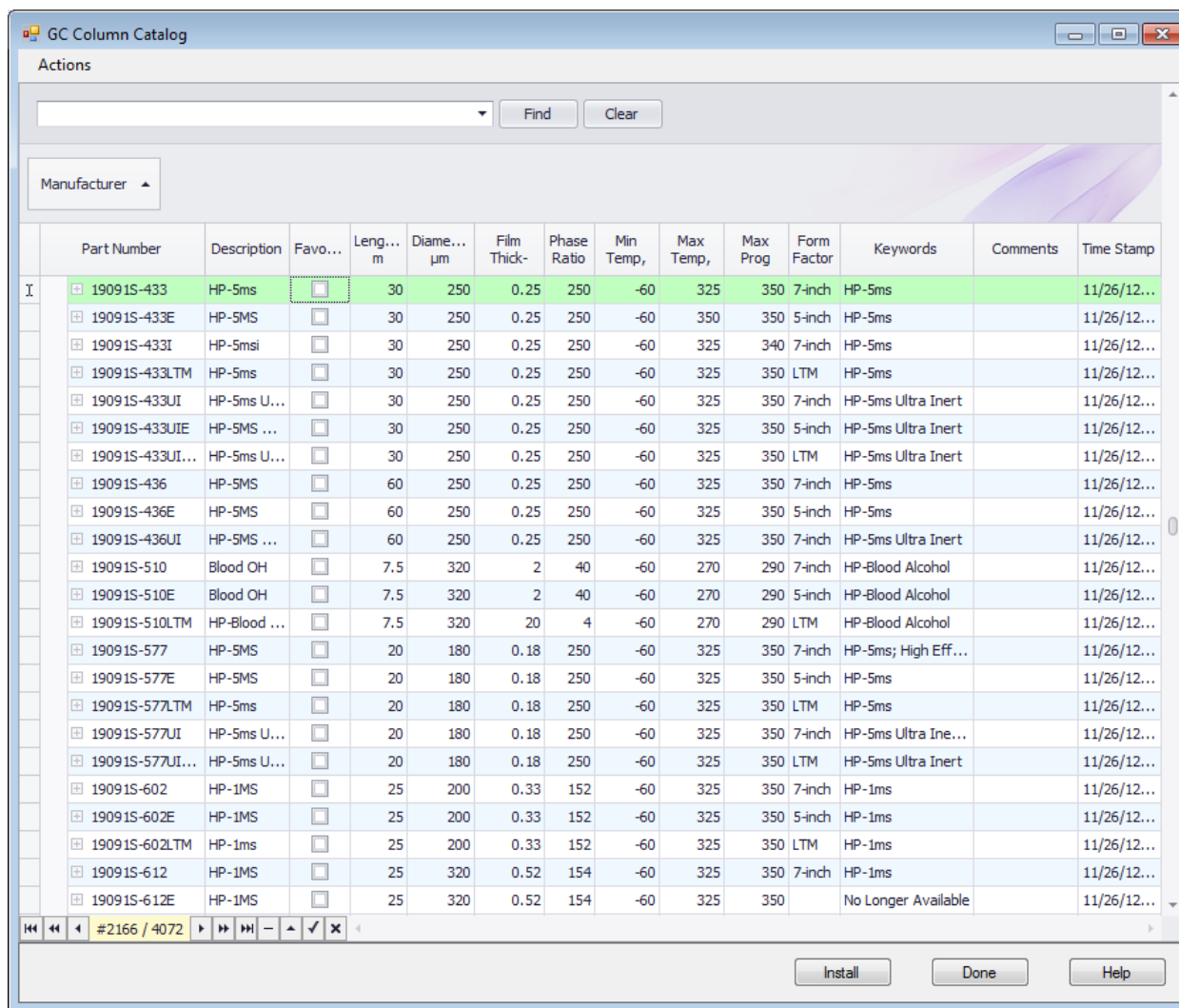


Figure 51 Column catalog

- 4 Click **Actions Menu** in the upper left corner of the screen, to display the actions available in this catalog.

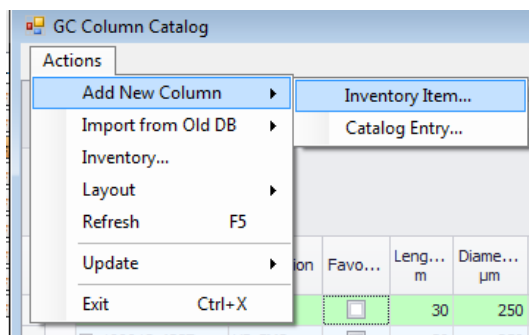


Figure 52 GC Column Catalog Action menu

- 5 Once you define a column, the column information displays in your method editor, the GC actuals, and in the report following a run.

Refer to online **Help** for details on how to complete the actions shown here.

Upload Parameters from the 7890X 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.

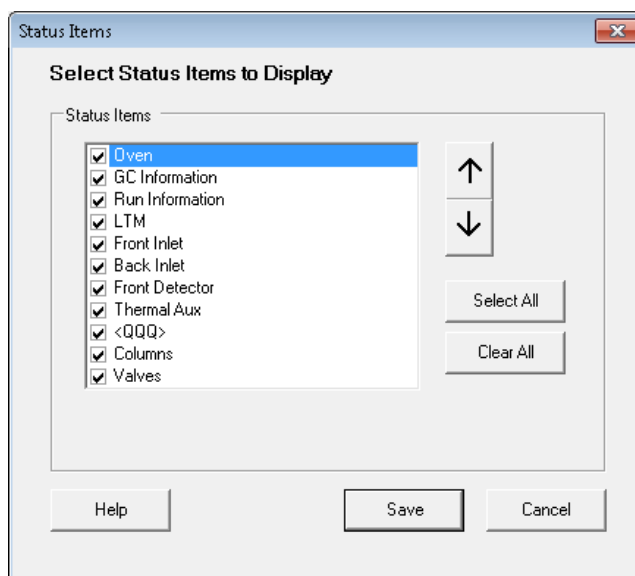


Figure 53

- 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.



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