



Agilent JetClean

Operating Manual



Notices

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Introduction

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The Agilent JetClean system allows you to greatly increase the number of samples you can process before you must manually clean your ion source. This chapter provides a brief concept of how the JetClean system works.

General Concept

During the JetClean process a small amount of hydrogen is introduced into the ion source's ion volume while the filament is emitting electrons: reactive hydrogen species are created. Each time this process runs, contamination is removed from the ion source depending on the conditions and the nature of the contamination. Consequently:

- Background is reduced (chemical noise)
- "Lost" compound detection limits can be recovered
- Manual cleaning will be required less frequently

However, over time, in spite of the JetClean process, less reactive contaminants may build up on the ion source optics, and the source will then have to be manually cleaned. By careful application of the process and use of the parameters for JetClean, this manual process can become much less frequent.

Two Modes of Operation

The JetClean application can be applied in one of two modes:

- **Clean Only** mode – In this mode, the hydrogen is introduced into the system after the sample(s) have been processed. When you use this mode, you would create a new method which will be used in the JetClean process, while your existing sample processing methods would remain unchanged.
- **Acquire & Clean** mode – In this mode, samples are analyzed via GC/MS EI while a small amount of hydrogen is simultaneously introduced into the ion volume to remove and reduce contaminants. When you use this mode, you would modify your existing acquisition methods to include a low flow of hydrogen suitable to your application.

Each mode offers advantages and disadvantages, based on the variables in your system, such as:

- The samples you are processing and their degree of cleanup, contamination, and matrix
- The analytes you target
- Your existing Standard Operating Procedures
- Your current workflow or sample batches

Other considerations that will become clear as you gain an understanding of JetClean.

Which Mode Should You Use?

To decide which JetClean mode (Clean Only or Acquire & Clean) is best for you, you must consider the variables specific to your system (such as sample type and cleanup effectiveness, and the analyte's chemistry, for example) and evaluate the impact each mode would have on your work flow.

Although each situation is different, some of the major items to consider are listed below.

1 What compounds are you analyzing?

Polar compounds, compounds containing oxygen, nitrogen, sulphur, or phosphorus, may react with trace (trickle) hydrogen flow provided by the JetClean process to compromise your detection limits or spectral matches. In this case, the Acquire & Clean mode may not be acceptable. The Clean Only mode may be a better solution.

Non-polar compounds, and other highly stable compounds (PAHs, PCBs, etc.) on the other hand, will have limited reactivity in hydrogen, and therefore may work well with the Acquire & Clean mode.

2 Will you need to modify your Standard Operating Procedures?

When you use the Acquire & Clean mode, you will need to modify your GC/MS analytical methods by including hydrogen during sample analysis. If your methods are included in validated SOPs, you may, therefore, need to modify and revalidate your SOPs.

On the other hand, the Clean Only mode will not require a new acquisition method, and, depending on your in-house operations, it may only be an addition to your system maintenance SOPs. However, the existing acquisition methods and SOPs could remain unchanged, and would not need to be revalidated.

1 Introduction

Configure the Instrument for Operating the JetClean System

Configure the Instrument for Operating the JetClean System

Once you know which mode you will be using, you must configure your system as described here.

- 1 In the Instrument control view select **Instrument > Configure MS Gases** to display the Gas Control Configuration dialog. (See [Figure 1.](#))

Gas Control Configuration

Controller Type

None

JetClean - EI only system

CI/JetClean - EI/CI system

Gas Configuration

Channel A gas Other

Channel B gas Other

Turn off hydrogen gas if inactive (min)

Figure 1. Gas Control Configuration panel

- 2 Select a **Controller Type**.
- 3 Select **Jet Clean - EI only system** if your flow control system does not support Chemical Ionization (CI). This setting configures hydrogen as the Channel B gas.
- 4 Select **CI / JetClean - EI/CI system** if your system is designed to support both CI and JetClean systems. This setting allows you to configure the Channel B gas for hydrogen use with the JetClean system and Channel A for a CI reagent gas.
- 5 Configure the hydrogen gas supplied:

1 Introduction

Configure the Instrument for Operating the JetClean System

- For a Jet Clean - EI only system controller for JetClean system use, enter a time in the **Turn off hydrogen gas if inactive** field. This is the time to wait before closing the hydrogen supply to the JetClean system after the JetClean method run is completed.
- For a CI / JetClean - EI/CI system controller for JetClean system operation, select hydrogen from the Channel B gas drop down menu and enter a time in the **Turn off hydrogen gas if inactive** field. Be sure that this time exceeds the time between GC runs so the hydrogen remains on.

1 Introduction

Set the Mode for the JetClean System in MassHunter

Set the Mode for the JetClean System in MassHunter

Once you have decided on which mode you will be using, and you have configured your system, you may select the applicable mode as follows:

- 1 Click the **MS Parameters** icon in the **Instrument Control** view.
- 2 Click **JetClean**. (See **Figure 2**.)
- 3 Select a mode from the **Operation** dropdown menu. (See **Figure 2**.)
 - **No Cleaning** – The JetClean system is off. Close the manual hydrogen shutoff valve for the JetClean system. The MFC must be purged prior to use after the manual valve has been closed for any period.
 - **Clean Only** – During the “Clean Only” mode of operation no sample analysis takes place. Hydrogen is introduced to the source only during this maintenance period. Sample analysis takes place in pure helium carrier gas as usual. (See **“Set the JetClean Parameters for the Clean Only Mode”** on page 17.)
 - **Acquire & Clean** – Continuously introduces hydrogen to the sample arriving at the ion source. (See **“Set the JetClean Parameters for the Acquire & Clean Mode”** on page 32.)

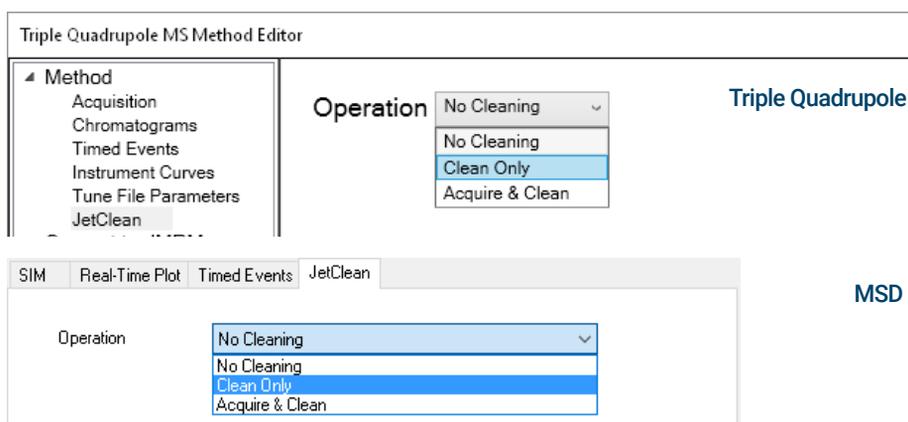


Figure 2. Method editor – JetClean mode selection panel

Complete the screens that are displayed as applicable for your method. (See **“Set the JetClean Parameters for the Clean Only Mode”** on page 17 and **“Set the JetClean Parameters for the Acquire & Clean Mode”** on page 32 for more details.)

2

Clean Only Mode

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Concept

The JetClean **Clean Only** mode is independent of your standard sample acquisition, and uses a completely different and separate method than your sample acquisition method(s). You do not need to modify your existing sample acquisition methods at all.

The JetClean **Clean Only** mode requires an investigation to determine the number of samples that can be run before running the JetClean **Clean Only** method and what parameters to apply in the JetClean method. In general, all analysts experience the same general trend. When the system is clean and leak-tight, compound responses and detection limits remain suitable as samples are running. At some point detection limits begin to suffer and maintenance is required. This may be the replacement of the septum and liner, column cut back, tightening of the ferrule, etc., and detection limits are restored. Careful examination of autotune files can reveal issues specific to the source beyond leaks or GC related problems. Typically, this is the time the source would require baking, updating the gain factors, or manual cleaning. Before the system has reached this stage the analyst, based on their experience, could apply a short and light JetClean **Clean Only** method to head off source degradation. In fact, one way to consider the JetClean **Clean Only** method is as an additional tool analogous to baking the source to remove contamination. The JetClean **Clean Only** method is far more effective than baking alone. An illustration of the situation is given in **Figure 3**.

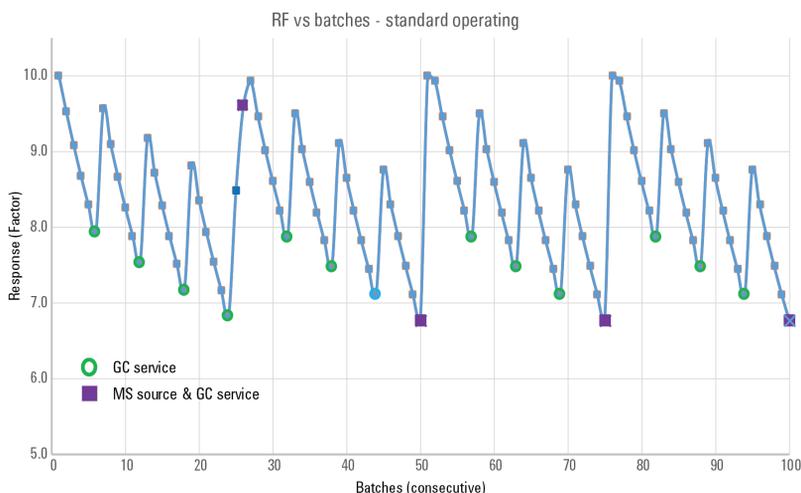


Figure 3. Response factor versus batches – Standard operating conditions

2 Clean Only Mode Concept

Figure 3 on page 12 shows the analytical situation for the results of the response of a standard as the course of sequential sample batches are being run. From the left to the right, as the number of sample batches are acquired, there is a clear decay in response (factor) proceeding from the starting response (at 0) and through to the 6th set. By the 6th set, the response had dropped > ~20% and indicates GC servicing is necessary which is marked in the figure as a (green) circle (probably not using rapid intra-column backflush).

After the GC maintenance, response has returned but not to the prior value (of 10 for batch 0). This signifies that some degradation of the source (and its contribution to) response has occurred.

Another 6 sets are acquired and another GC maintenance takes place (at batch 12), and this continues through batches 18, and 24.

However, after maintenance at batch 24, the 25th batch shows a lower response by >~15% than initial response which is deemed unacceptable and so the MS is vented and the source cleaned, which is indicated by the square marker at batch 26.

Response nearly returns and as the source is “shot in” by continued injections it returns to the initial response at batch 0.

The sequence of batches continues with GC servicing every 6 batches consistent with the response loss until both a GC service and MS source cleaning must be conducted at batch 50, 75 and so on.

Figure 4 shows the situation as before, but with the JetClean **Clean Only** mode operating after each batch.

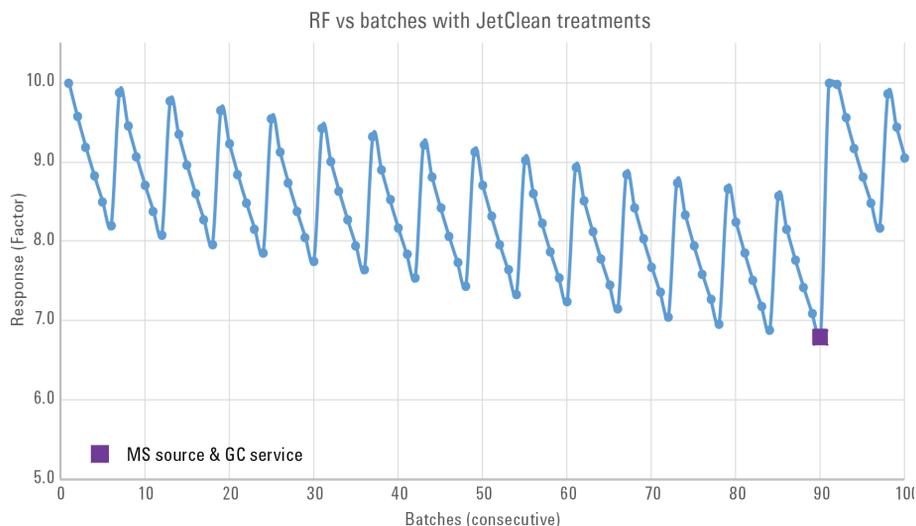


Figure 4. Response factor versus batches with JetClean Clean Only mode after each batch

2 Clean Only Mode Concept

GC maintenance is still required every 6 batches (not indicated but visible as before), but now GC and MS maintenance is not required until after batch 90; from every 25 batches to 90 batches – a substantial decrease in MS servicing. This illustrates what JetClean can offer.

It is important to note that this control chart approach, although widely applied, is not strictly correct and often misleading. In the same way that Signal-to-noise is not a good metric, this approach should be replaced by one that focuses on method detection limits.

Depending on the results of the investigations of your samples and required maintenance frequency, you may implement the JetClean **Clean Only** mode:

- After running a batch of samples
- After running a single sample (if degraded performance is seen immediately after such a sample is acquired)

The next section describes a little bit more about this concept. Investigative testing will help determine a solution that is best for extending the ion source manual cleaning cycle when running specific sample types.

It is important to recognize that JetClean is not a replacement for things such as proper sample preparation or maintenance of GC issues such as leaks, and users should consider implementing (rapid, intra-column) backflushing (Agilent G1472A Universal GC/MS PCT Backflush Kit) which has shown the greatest reliability in preventing source related degradation of compound detection limits.

Running the Clean Only Mode, After a Batch of Samples – Concept

The following describes the general procedure you would follow to apply the JetClean **Clean Only** mode after running a batch of samples.

- 1 Create a standard JetClean **Clean Only** acquisition method.
Start with the lightest conditioning method, which will be described below.
- 2 Run your samples as usual with your standard sample acquisition method.
- 3 When you observe signal loss, or background noise, perform your standard troubleshooting routine (GC liner and column maintenance, leak test, gain factor update, analyzer bakeout, autotune, etc.). (See **“Troubleshooting”** on page 35.)
 - a If, after routine troubleshooting and maintenance, the signal is restored, or the background noise is removed, resume processing samples as usual.
 - b If the troubleshooting did not improve the results, run the JetClean **Clean Only** method.
- 4 After running the JetClean **Clean Only** method, run a sample to see how effective the JetClean application was.
 - a If the results have improved satisfactorily, resume processing samples as usual.
 - b If the results have improved, but not sufficiently, adjust the setpoints of the JetClean **Clean Only** method, very slightly, and re-run the JetClean method. (For example, increase the amount of hydrogen added or the exposure time.)
 - c If the results have gotten worse, it may be time to perform a manual cleaning. It may also be another issue which is common to cleaned sources and will be discussed later in the document.

It is important to note that “the results” refers to the ability of the system to detect compounds of interest (not signal alone nor S:N!), as well as considerations of spectral fidelity, system background, etc.

2 Clean Only Mode

Running the Clean Only Mode, After Running Each Sample – Concept

Running the Clean Only Mode, After Running Each Sample – Concept

Generally speaking, to run the JetClean **Clean Only** mode after running each sample, you would do the following:

- 1 Create a very short running JetClean **Clean Only** method intended to run after each sample run.
- 2 Run sample 1 with your normal sample acquisition method, as usual.
- 3 Run the JetClean method.
- 4 Run sample 2, as usual.
- 5 Run the JetClean method.
- 6 Continue alternately running the JetClean method and normal samples until you observe detection limits rising.
- 7 When you observe this, perform your standard troubleshooting and maintenance routine (leak test, gain factor update, bakeout, autotune). (See **“Troubleshooting”** on page 35.)
 - **If your results improved**, resume alternately processing samples and running the JetClean method, as above.
 - **If your results did not improve**, it may be time to manually clean your ion source or decrease or increase the intensity of the JetClean method.

2 Clean Only Mode

Set the JetClean Parameters for the Clean Only Mode

Set the JetClean Parameters for the Clean Only Mode

If you are using the JetClean **Clean Only** mode, access the JetClean **Clean Only** parameters, and complete them as appropriate for your method.

- 1 Click the **MS Parameters** icon in the **Instrument Control** view.
- 2 Select **JetClean**. (See **Figure 2** on page 10.)
- 3 Select **Clean Only** from the **Operation** dropdown menu. (See **Figure 2** on page 10.)
- 4 Enter the parameters supplied by your method developer. (See **Figure 5**)
- 5 Save the method.

Triple Quadrupole

Triple Quadrupole MS Method Editor

Method

- Acquisition
- Chromatograms
- Timed Events
- Instrument Curves
- Tune File Parameters
- JetClean
- Convert to dMRM

Operation: Clean Only

Cleaning

Hydrogen Flow (mL/min): 0.67
(Limited to steps of 0.0666 mL/min)

Filament: 2

Emission (μA): 10

Source Temperature (°C): 230

Quadrupole Temperature (°C): 150

Duration (min): 1.3

Post Cleaning

For Post Cleaning, the Source and Quadrupole temperatures are derived from the temperature settings in the Tune File associated with this method.

Stabilization Duration (min): 10

Apply Ok Reset Cancel Help

MSD

JetClean

Operation: Clean Only

Cleaning

Hydrogen flow: 0.13 mL/min
(limited to steps of 0.0666 mL/min)

Filament: Filament 2

Emission: 10 μA

Source Temperature: 230 °C

Quadrupole Temperature: 150 °C

Duration: 1.3 min

Post Cleaning

For Post Cleaning, the Source and Quadrupole temperatures are derived from the temperature settings in the Tune File associated with this method.

Stabilization Duration: 10.0 min

Figure 5. JetClean Clean Only mode

Optimize Parameters (Setpoints)

Hydrogen can be harsh on the filament so the JetClean **Clean Only** method uses filament number two, leaving the analytical filament to be filament number one. (In CI operation there is only one filament available.)

To obtain the best possible sample throughput with minimal source impairment, during the development of your JetClean **Clean Only** method, there are several parameters you can adjust, test, and readjust to obtain optimal results. Some of these parameters include:

- Time
- Hydrogen flow
- Emission
- Source temperature
- Number of samples between JetClean applications
- Type of samples
- Etc.

It is important to obtain the lowest possible hydrogen flow, while still observing good results. To begin, start by using the lowest setting you feel will be effective. If the results are not satisfactory, increase the flow and retest.

- **Too little hydrogen** will fail to clean the source sufficiently.
- **Too much hydrogen** will “over condition” the source – a topic that will be discussed later.

Applying the **Clean Only** process, either manually or as a method run in a sequence, creates a scan datafile of the conditioning process. This datafile contains valuable information that provides insight into the degree of conditioning.

NOTE

The method should be configured to collect this scan data over a range suitable to the instrument – this is described below.

Figure 6 on page 19 shows an example of the EIC for such a collected JetClean **Clean Only** datafile on a 5977B with HES source. The extracted ions of 55, 57, 91 are examples of ions indicating the background contamination; 55 and 57 reflect the hydrocarbons and 91 aromatics, etc.

2 Clean Only Mode

Optimize Parameters (Setpoints)

Notice the ions indicate not all species have exactly the same kinetics nor initial or final abundancies. You can determine which degree of treatment is suitable for your compounds and your analysis.

Note the possibility of a rapid light treatment for ~1.5 minutes, more extensive for ~3.5 minutes, or an even longer duration. By manipulating the parameters, short periods of treatment can be used to remove varying degrees of contamination.

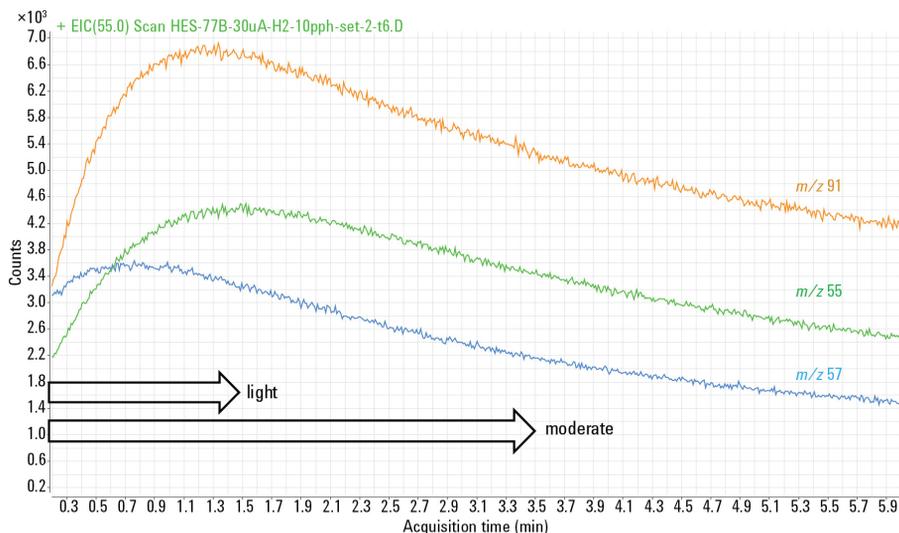


Figure 6. EICs for JetClean Clean Only mode for 5977B SQ-HES over the range of 0 to 6-mins

The approach to exploring this is sequential in the following logic.

Starting with the default parameters, treat a contaminated source and then continue operating. If it is judged insufficient, first extend the time of the treatment and continue to increment the time to about a 10-minute run time. At that stage treatments are becoming too long in duration so shorten the time back to 1 or 2 minutes and increase the hydrogen flow by doubling or tripling the amount. Continue this until the hydrogen flow setpoint nears half the recommended maximum flow for that source type. Then reduce the time and flow back and increase the emission.

The last parameter to be implemented is increasing source temperature. This is helpful in removing very recalcitrant deposits but adds additional time to the process as the source must cool to the operating temperature, so typically it is a last resort.

2 Clean Only Mode

Optimize Parameters (Setpoints)

Like standard manual cleaning, there is a period where the analyzer must stabilize. After manual cleaning, the process is baking and retuning, but for the JetClean **Clean Only** mode, there are other additional approaches, the most successful of which is to “shoot the analyzer in”. Instead of the alternative of a long stabilization time setting, a short setting there is made up for by several injections of standards or matrix or analyte protectants (as in pesticide applications) which stabilize the system. Retuning should be applied again at the end of this process and before sample batches are submitted.

It is possible to “over-condition” a source which results in peak tailing for compounds that are not associated with GC issues. Note these circumstances and JetClean **Clean Only** parameters, and avoid them in the future.

Table 1 on page 20, **Table 2** on page 21, and **Table 3** on page 21 give defaults and ranges of the important parameters. Refer to these tables while you develop your own JetClean “Clean Only” method.

Table 1 Range of 5975, 5977A/B, and 7000A/B/C/D JetClean parameters

Parameter / Starting Setpoint	Lower end	Upper end	Comment
Hydrogen Flow: Clean Only mode 0.67 mL/min	0.13 mL/min	3.52	Flow steps are in 0.069 sccm units. The standard 3 mm Drawout lens configuration should not require more than ~3.5 mL/min in any Clean Only mode setpoint.
Emission (µA) 10 µA	10	35	Emission and flow increases have the greatest effects in accelerating source cleaning. To keep times short, increment these two parameters.
Duration (min)	1 min	120	Although the upper limit is high, an advantage in JetClean is time savings, so more aggressive parameters should be explored.
Source temperature Use operating Tune file setting.	150 °C	350 °C	Start with your acquisition method's Tune File source temperature to save time. Same for the Quadrupole temperature.
Hydrogen Flow: Acquire & Clean mode 0.13	0.13 mL/min	0.49 mL/min	Most applications will use very low settings for the Acquire & Clean mode (<0.5 mL/min) and step up this parameter in small increments.

2 Clean Only Mode

Optimize Parameters (Setpoints)

Table 2 Range of 5977B HES and 7010 HES JetClean parameters

Parameter / Starting Setpoint	Lower end	Upper end	Comment
Hydrogen Flow: Clean Only mode 0.67 mL/min	0.13 mL/min	3.52	Flow steps are in 0.069 sccm units. No setpoint should exceed ~3.5 mL/min in any Clean Only method setpoint.
Emission (µA) 10 µA	10	100	Recommended maximum is 50 µA; low setpoints will take some time to stabilize.
Duration (min) 1 min	1 min	120	Although the upper limit is high, an advantage in JetClean is time savings, so more aggressive parameters should be explored.
Source temperature. Use operating Tunefile settings.	150 °C	350 °C	Start with your acquisition method's Tune File source temperature to save time. Same for the Quadrupole temperature.
Hydrogen Flow: Acquire & Clean mode	0.13 mL/min	0.53 mL/min	

Table 3 JetClean Clean Only mode scan parameters per instrument and source type

Parameter	5975 5977A/B	5977B HES 7000A/B/C/D	7010A HES 7010B HES
• eV	70 eV	70 eV	70 eV
• Gain Factor*	1	0.2	0.2
• Mode	Scan	Scan / MS1 Scan	MS1 Scan
• Starting mass [†]	29	29 / 45	29 / 45
• Ending mass	300	300	300
• Time / samples	2^5	2^5 / 250 msec (5)	250 msec (5)
• Threshold	25	25	25

* The Gain Factor should be adjusted based on the parameters to keep total counts < 10⁵ for any one ion current (an EM saver should be on). As the current and H₂ flow are increased, ion counts will increase.

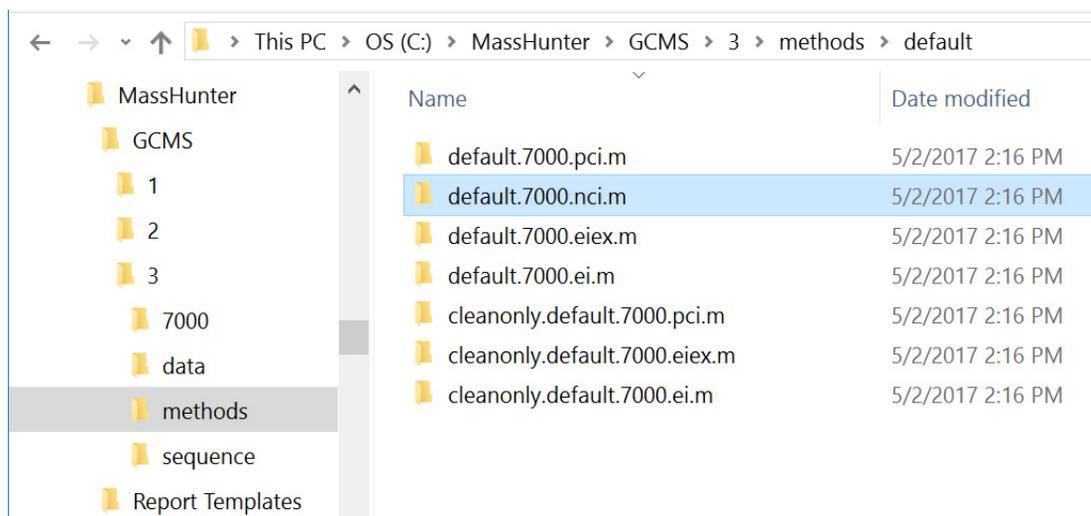
† Starting mass at 29 will show the presence of N₂H⁺ which will indicate H₂ is on and the process is operating. After that, the lower mass should be raised to encompass the interesting range, perhaps 50 and above, but here the lower edge of 45 (above CO₂) is cited.

Default Methods

Included with MassHunter are methods that may be used as starting points for developing your own JetClean Clean Only methods. Once MassHunter is installed, you will find these methods in the locations shown below.

See **Table 1** on page 20, **Table 2** on page 21, and **Table 3** on page 21 for more information and ranges of the important parameters for each method.

7000 “Clean Only” default methods



2 Clean Only Mode

7010 “Clean Only” default methods

7010 “Clean Only” default methods

Name	Date modified
cleanonly.default.7010.eihs.m	5/2/2017 2:10 PM
cleanonly.default.7010.pci.m	5/2/2017 2:10 PM
default.7010.eihs.m	5/2/2017 2:10 PM
default.7010.nci.m	5/2/2017 2:10 PM
default.7010.pci.m	5/2/2017 2:10 PM

597X “Clean Only” default methods

Name	Date modified
default.597x.m	5/2/2017 2:08 PM
CleanOnly.HES_atune.m	5/2/2017 2:08 PM
CleanOnly.etune.m	5/2/2017 2:08 PM
CleanOnly.EI-Clsource.m	5/2/2017 2:08 PM
CleanOnly.atune.m	5/2/2017 2:08 PM

2 Clean Only Mode

597X “Clean Only” default methods

These methods refer to the tune file which in turn dictates what source is supported.

- For a SS, inert, or extractor sources, use the **CleanOnly.atune.m** method.
- For a CI source, use the **CleanOnly.El-CIsource.m** method.

Table 4 Clean Only mode - Default method parameters

Method	Type	H2 Flow	Emission	Filament	Source	Analyzer	Time	Stabilization
CleanOnly.atune.m	Clean Only mode	0.7	20	2	230	150	1.3	10
CleanOnly.etune.m	Clean Only mode	0.7	20	2	230	150	1.3	10
CleanOnly.HES_Atune.m	Clean Only mode	0.7	10	2	230	150	1.3	10
CleanOnly.El-CIsource.m	Clean Only mode	0.7	20	1	230	150	1.3	10

You may change the tune file specified in the method.

All of the methods have six MS monitors. The monitors are:

- MS Source
- MS Quad
- Cleaning Operation
- H₂ Flow (mL/min)
- Emission (uA)
- Filament

All of the GC monitors are turned off because the GC does not participate in the method.

The GC method is configured under Inlet and Injection Parameters as “Other/None” for the Sample Inlet and “Valve/Immediate Start” as the Injection Source. The supplied default methods already have this configuration but any other (existing) acquisition method being converted to Clean Only must be altered to this configuration to prevent sample injections during Clean Only mode. The column flow rate and other GC parameters (e.g., transfer-line, inlet temperature, etc.) can remain unchanged. To create a Clean Only method from existing GC-MS methods, be sure to make the changes to the Inlet and Injection

2 Clean Only Mode

597X "Clean Only" default methods

Parameters as stated above, confirm the changes, then save the method. If the GC attempts to inject a sample during execution of a Clean Only method, these configuration parameters need to be checked.

When a method is loaded, the monitors reflect the current state of the instrument. When the method is run, the parameters in **Table 4** will prevail. The **Cleaning and Stabilization** times (post run time) may be monitored by viewing the **Retention Time Clock**.

2 Clean Only Mode

Example Method for the Clean Only Mode

Example Method for the Clean Only Mode

Figure 7 on page 26 and **Figure 8** on page 27, and **Figure 9** on page 28 are JetClean **Clean Only** method default parameters for a 7000/7010 Triple Quadrupole instrument and a 5977 MSD instrument.

Each of the default JetClean **Clean Only** methods will have settings specific to the instrument and ion source combination.

See **Table 1** on page 20, **Table 2** on page 21, and **Table 3** on page 21 for more information and ranges of the important parameters for each method.

Triple Quadrupole

Triple Quadrupole MS Method Editor

Method
Acquisition
Chromatograms
Timed Events
Instrument Curves
Tune File Parameters
JetClean
Convert to dMRM

Operation: Clean Only

Cleaning

Hydrogen Flow (mL/min): 0.67
(Limited to steps of 0.0666 mL/min)

Filament: 2

Emission (μA): 10

Source Temperature (°C): 230

Quadrupole Temperature (°C): 150

Duration (min): 1.3

Post Cleaning

For Post Cleaning, the Source and Quadrupole temperatures are derived from the temperature settings in the Tune File associated with this method.

Stabilization Duration (min): 10

Buttons: Apply, Ok, Reset, Cancel, Help

MSD

JetClean

Operation: Clean Only

Cleaning

Hydrogen flow: 0.13 mL/min
(limited to steps of 0.0666 mL/min)

Filament: Filament 2

Emission: 10 μA

Source Temperature: 230 °C

Quadrupole Temperature: 150 °C

Duration: 1.3 min

Post Cleaning

For Post Cleaning, the Source and Quadrupole temperatures are derived from the temperature settings in the Tune File associated with this method.

Stabilization Duration: 10.0 min

Figure 7. Example - JetClean Clean Only method – Treatment parameters

2 Clean Only Mode

Example Method for the Clean Only Mode

Triple Quadrupole MS Method Editor

Method

- Acquisition
- Chromatograms
- Timed Events
- Instrument Curves
- Tune File Parameters
- JetClean
- Convert to dMRM

Tune File

atunes.ehls Browse...

Source Parameters

Ion Source: EI

Source Temperature (°C): 230

Electron Energy Mode: Use Tune Setting

Electron Energy (eV): 70

Detector Setting

Use Gain Factor

Use Delta EMV

EM Saver Limit: 1E+08

Time Segments

Time (min)	Scan Type	Electron Energy (eV)	Delta EMV (V)	Calculated EMV (V)	Gain	Data Saved	# of Ions	CanEditStartTime
1	25	MS1 Scan	70	1045.1	2	<input checked="" type="checkbox"/>		<input type="checkbox"/>

4 cycle/sec 247.6 ms/cycle

Run Time

Run time (min): 1

Solvent Delay (min): 25

Time Filter

Off

On

Variable*

Time (min) Peak Width (sec)

Automatically Subtract Baseline

Advanced MRM/SIM filtering

* The feature is instrument dependent

Scan Segments

Segment Name	MS1 Start Mass	MS1 End Mass	Scan Time (ms)	Data Samples	Expected ScanTime (ms)
1 Background	45	300	250	6	248

Scan Parameters

Step size (amu): 0.1

Threshold: 25

Profile Data

Apply OK Reset Cancel Help

Figure 8. Example -Triple Quadrupole method editor – Acquisition parameters

2 Clean Only Mode

Example Method for the Clean Only Mode

The screenshot displays the 'Single Quadrupole MS Method Editor' window. The interface is divided into several sections for configuring the method.

Tune File: HES_atune.u

Tune Type: EI

Tune EMV: 1200

MS Source: Current is 230, MS Quad download 150

Acquisition Type: SIM

Detector Setting: Trace Ion Detection (unchecked), EM Setting: Gain Factor, Gain Factor: 0.20, Applied EM Voltage (V): 1185, EM Saver (checked), Limit: Sum Limit 1e8 (Default)

Scan Time Segments Table:

Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
0.25	50.00	550.00	150	1,562 (N=2)	2.9	342.63	0.1

SIM Time Segments Table:

Time	Group Name	Number of Ions	Total Dwell Time (ms)	Cycle Time (Hz)	Resolution	Gain Factor	Calculated EMV
0.25		1	1	100	8.3333	Low	1185

Real-Time Plot Window: Shows a table with columns m/z, Dwell Time, Plot Ion, and Label. The first row has m/z 74.10, Dwell Time 100, and Plot Ion checked.

Method Last Saved: 8/22/2016 7:23:26 AM

Buttons: OK, Cancel, Help

Figure 9. Example - MSD method editor – Acquisition parameters

3

Acquire & Clean Mode

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Running JetClean in the Acquire & Clean Mode 31

Set the JetClean Parameters for the Acquire & Clean Mode 32

Concept

Without JetClean, during normal operations, each time a sample is processed some amount of contamination will be deposited on the ion source. Over time, this builds up to the point at which the system must be shut down and the ion source must be manually cleaned.

When you use JetClean in the **Acquire & Clean** mode, each time a sample is analyzed a small amount of hydrogen is introduced into the system along with it. Once the hydrogen reaches the ion source, a chemical reaction takes place that will remove some, but not all, sample contamination from the source. Over time you will still need to manually clean the ion source. However, by removing a small amount of contamination from the source each time a sample is processed, you will greatly increase the amount of samples you may process before you need to perform a full manual cleaning of the ion source.

Because this process requires a modified sample processing method, there are some things to consider before proceeding.

- **If you are testing Non-polar compounds**, i.e., not containing oxygen, nitrogen, sulfur, or other highly polar groups, which show little or no reactivity with hydrogen, the JetClean **Acquire & Clean** mode may be able to be incorporated into your workflow.
- **If you are testing polar compounds**, i.e., containing oxygen, nitrogen, sulfur, or other highly polar groups, which may show reactivity with hydrogen, verify that the introduction of hydrogen still allows you to meet your qualitative and quantitative requirements. If not, it may be better to work with the JetClean **Clean Only** mode.
- Because your sample processing method will be modified, it may need to be revalidated. The Clean Only mode, on the other hand, does not modify your existing sample processing method.

WARNING

If chlorinated solvents are used with this process you must insure the solvent delay is sufficient. Not eliminating solvent, and operating JetClean concurrently, could void the warranty of the GC/MS system.

Carefully check that all solvent has eluted or been eliminated before the filament is engaged. Some examples of solvents that are most critical to eliminate prior to the filament being lit are:

- Dichloromethane (DCIM)
- Chloroform
- Carbon tetrachloride
- Carbon disulphide

Running JetClean in the Acquire & Clean Mode

Generally speaking, the JetClean online processing workflow will be similar to the following.

- 1 Create your JetClean **Acquire & Clean** analytical method.
 - a Start with a clean ion source.
 - b Modify your sample processing method to include the smallest amount of hydrogen.
 - c Run standards as usual and observe the results. It may be that the Gain Factor(s) or even ion ratios may need to be re-adjusted.
 - If the compound results are satisfactory (i.e., the compound ion signals are not degraded or compromised by new ion interferences, and there is no compound tailing or other chromatographic problems for example), consider increasing the hydrogen and again review the results. The objective is to use the highest hydrogen flow that does not compromise the results as this will provide the most robust source conditioning.
 - If the results have gotten worse, and no longer meet the analytical criteria, and no hydrogen flow can be added during analysis, consider the Clean Only mode.
 - d When the **Acquire & Clean** method seems to be running smoothly, begin analyzing samples as usual.
- 2 Acquire the samples using the JetClean Acquire & Clean method. As long as your results are acceptable, continue running samples as usual.
- 3 When you observe signal loss, or background noise, perform your standard troubleshooting routine (GC inlet and column maintenance, leak test, Gain Factor Update, Bakeout, GF update, Autotune). (See "Troubleshooting" on page 35.)
- 4 If the troubleshooting did not improve the results, it may be time to apply the Clean Only mode to the source or perform manual cleaning.

The hydrogen flow can be incremented until adverse effects such as degraded spectra, peak tailing, compromised detection limits, etc., affect the analysis. Even a low flow setting, near the lowest values allowed (0.15, 0.21, 0.28 mL/min) should be found to be effective at extending the time between manual cleaning without substantial analytical degradation.

3 Acquire & Clean Mode

Set the JetClean Parameters for the Acquire & Clean Mode

Set the JetClean Parameters for the Acquire & Clean Mode

The Hydrogen Flow limit parameters associated with the **Acquire & Clean** mode are located in the screen shown below.

- 1 Click the **MS Parameters** icon in the **Instrument Control** view.
- 2 Load the method with its tune file that was developed for this **Acquire & Clean** mode. The hydrogen flow in this tune file must match the hydrogen flow in this JetClean method.
- 3 Select **JetClean**. (See Figure 2 on page 10.)
- 4 Select **Acquire & Clean** from the **Operation** dropdown menu.
- 5 Enter the **Hydrogen Flow** provided by your method developer. (See **Figure 10** on page 33.) This flow must match the hydrogen flow used for the tune file.
- 6 Save the method.

3 Acquire & Clean Mode

Set the JetClean Parameters for the Acquire & Clean Mode

The screenshot displays the 'Triple Quadrupole MS Method Editor' interface. On the left, a navigation pane lists 'Method' sub-items: Acquisition, Chromatograms, Timed Events, Instrument Curves, Tune File Parameters, JetClean (highlighted), and Convert to dMRM. The main area is titled 'Triple Quadrupole' and shows 'Operation' set to 'Acquire & Clean'. Under the 'Cleaning' section, 'Hydrogen Flow (mL/min)' is set to '0.13' with a note '(Limited to steps of 0.0666 mL/min)'. A note below states: 'Note: The Acquire & Clean mode of operation introduces Hydrogen into the flow path during acquisition which will affect the characteristics of the resulting spectra. As such, any use of Acquire & Clean mode methods must be fully validated before use.' Below this is a tabbed interface with 'SIM', 'Real-Time Plot', 'Timed Events', and 'JetClean' (selected). The 'MSD' section shows 'Operation' set to 'Acquire & Clean' and 'Hydrogen flow' set to '0.13 mL/min' with the same limitation note. A second note is present: 'Note: The Acquire & Clean mode of operation introduces Hydrogen into the flow path during acquisition which may affect the characteristics of the resulting spectra. As such, any use of Acquire & Clean mode methods must be fully validated before use.'

Figure 10. Hydrogen Flow limit screen in the Acquire & Clean mode

3 Acquire & Clean Mode

Set the JetClean Parameters for the Acquire & Clean Mode

4

Troubleshooting

General Troubleshooting 36

Troubleshooting JetClean 36

General Troubleshooting

When monitoring of the system background noise and signal strength or consistency indicates a need for troubleshooting, run these troubleshooting routines in the order shown here. After each step, run a sample to see if the problem is fixed.

- 1 Perform GC inlet and column maintenance.
- 2 Perform a leak test and correct any leaks. Always perform an air and water leak test before running any hydrogen through your system. In the presence of a leak, hydrogen can cause extreme damage.
- 3 Update the gain factor.
- 4 Bake the system and then check the gain factor again.
- 5 Retune and recalibrate the instrument.

If all of the above steps fail to resolve the problems, manually clean the ion source. Refer to your system's Operating Manual for details on how to manually clean the ion source.

If your system results are now satisfactory, and you are running in the JetClean **Clean Only** mode, run the JetClean **Clean Only** method, then resume your normal sample processing.

Troubleshooting JetClean

When setting up JetClean, make sure that all lines are well purged.

When invoking JetClean in either mode (**Clean Only** or **Acquire & Clean**), observe the ion gauge setting when the flow is on.

A scan acquisition at a low gain factor can also be acquired to make sure that the hydrogen is entering the ion source.

Unique ions will indicate the presence of the hydrogen beyond the H_2^+ itself: m/z 3 (H_3^+), 5 (HeH^+), 19 (H_3O^+) 29 (N_2H^+), etc. The intense presence of the m/z 29 ion is a good indicator of the hydrogen being present inside the source.

5

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Intended Use

Agilent products must only be used in the manner described in the Agilent product user guides. Any other use may result in damage to the product or personal injury. Agilent is not responsible for any damages caused, in whole or in part, by improper use of the products, unauthorized alterations, adjustments or modifications to the products, failure to comply with procedures in Agilent product user guides, or use of the products in violation of applicable laws, rules or regulations.

Supported Systems

The JetClean system accessory may be field installed by an Agilent Field Engineer or Service Representative. Agilent 7890 and 9000 GCs with the following MS configurations are supported:

- An Agilent 5975 series or 5977 series MSD with a high performance turbo pump.
- An Agilent 7000 and 7010 Series Triple Quadrupole MS

Currently only the 7890 GC supports JetClean and CI operating in EI mode.

System Operation and Maintenance

General warnings

WARNING

Before running an operation that requires the opening of the hydrogen supply shutoff valve to the JetClean system, all tubing, fittings, vacuum pump discharging plumbing, and controls must be thoroughly leak tested with an electronic hydrogen leak tester.

WARNING

Always check for leaks with an electronic hydrogen leak tester after changing a tank or servicing the gas lines. Never use soap to test a system using hydrogen for leaks.

WARNING

All compressed gas cylinders should be securely fastened to an immovable structure or permanent wall. Compressed gases should be stored and handled in accordance with the relevant safety codes. Gas cylinders should not be located in the path of heated oven exhaust.

WARNING

To avoid possible eye injury, wear eye protection when using compressed gas.

Hydrogen supply plumbing

Figure 11 illustrates the plumbing for the hydrogen filter used with JetClean.

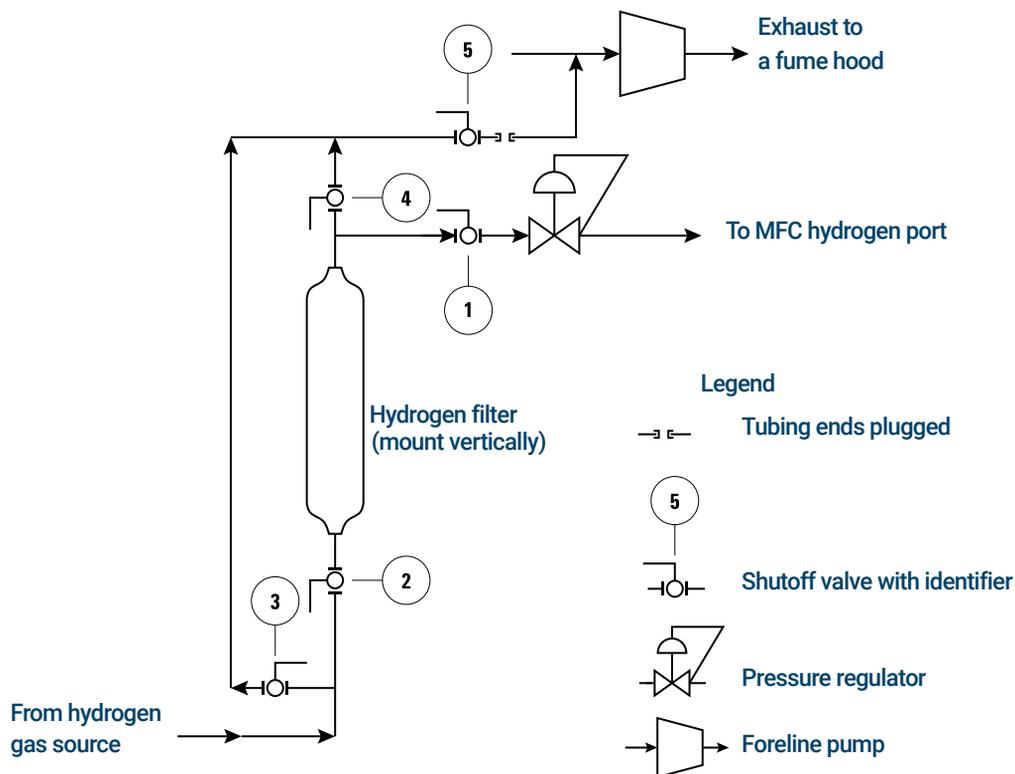


Figure 11. Filtered hydrogen supply to the MS

- 1 Connect the H₂ gas to the hydrogen port of the CI/JetClean MFC.
- 2 Configure the MS gases.
- 3 Tune the appropriate tune file.
- 4 Set the **Injection Source** to **valve\immediate start**. The inlet should not be set as **GC**.

Equipment Precautions

Take the following precautions when operating a GC/MS system with the JetClean option that supplies hydrogen to the MS from a flow controller located on the analyzer.

WARNING

You MUST make sure the top thumbscrew on the analyzer side plate(s) are fastened finger-tight. Do not over tighten the thumbscrews; this can cause air leaks.

You MUST remove the plastic cover over the glass window on the front of the analyzer. In the unlikely event of an explosion, this cover may dislodge.

Failure to follow the warning steps listed above greatly increases the chance of personal injury in the event of an explosion.

Operating Precautions

When using hydrogen gas, check the system for leaks to prevent possible fire and explosion hazards based on local Environmental Health and Safety (EHS) requirements. Always check for leaks after changing a tank or servicing the gas lines. Always make sure the vent lines from the foreline pump or purge lines are vented into a fume hood.

- Turn off the hydrogen at its source every time you shut down the GC or MS.
- Do not use hydrogen as a collision cell gas.
- Turn off the hydrogen at its source every time you vent the MS.
- Turn off the hydrogen at its source every time shutoff valves in the MS are closed.
- Turn off the hydrogen at its source if a power failure occurs.
- If a power failure occurs while the GC/MS system is unattended, even if the system has restarted by itself:
 - a** Immediately turn off the hydrogen at its source.
 - b** Turn off the GC.
 - c** Turn off the MS and allow it to cool for 1 hour.
 - d** Eliminate all potential sources of ignition in the room.
 - e** Open the vacuum manifold of the MS to atmosphere.
 - f** Wait at least 10 minutes to allow any hydrogen to dissipate.
 - g** Start up the GC and MS as normal.

In addition to the information provided in this document, you should read and understand the warnings in the Hydrogen Safety Manual supplied with your system, and the Hydrogen Safety section of your operating manual.

Hydrogen Plumbing

WARNING

All compressed gas cylinders should be securely fastened to an immovable structure or permanent wall. Compressed gases should be stored and handled in accordance with the relevant safety codes.

Gas cylinders should not be located in the path of heated oven exhaust.

To avoid possible eye injury, wear eye protection when using compressed gas.

General recommendations

- You must supply pre-cleaned, 1/8-inch stainless steel (SS) tubing and a variety of 1/8-inch SS Swagelok fittings to connect the JetClean system to the hydrogen gas supply source.
- Agilent strongly recommends two-stage regulators to eliminate pressure surges. High-quality, low flow, stainless steel diaphragm-type regulators are especially recommended.
- On/off valves mounted on the outlet fitting of the two-stage regulator are not essential but are very useful. Be sure the valves have stainless-steel, packless diaphragms.
- Agilent strongly recommends installation of shut-off valves at each MS inlet supply fitting to allow the MS to be isolated for maintenance and troubleshooting.
- Flow and pressure controlling devices require at least 10 psi (138 kPa) pressure differential across them to operate properly.
- Set the gas pressure regulator to deliver 20 to 25 psig to the MFC connector.
- Situate auxiliary pressure regulators close to the MS inlet fittings. This ensures that the supply pressure is measured at the instrument (rather than at the source); pressure at the source may be different if the gas supply lines are long or narrow.
- Never use liquid thread sealer to connect fittings.
- Never use chlorinated solvents to clean tubing or fittings.

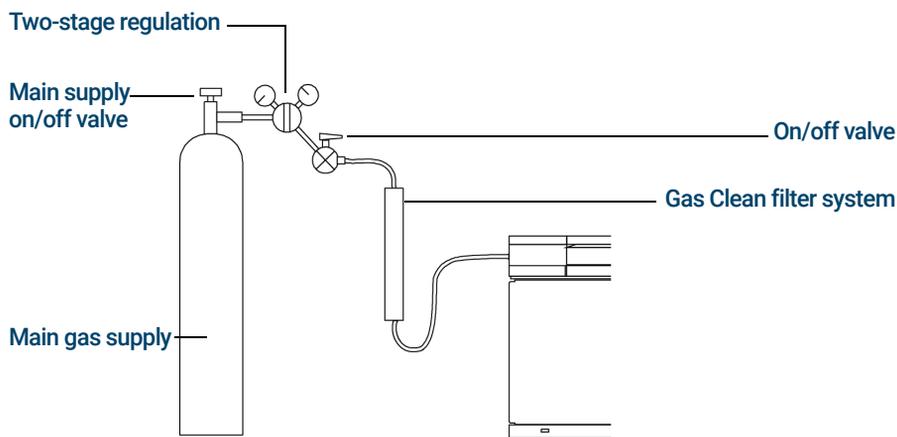
See Installation Kits for more information.

Supply tubing for hydrogen gas

- Agilent recommends using new chromatographic quality stainless steel tubing and fittings when using hydrogen.
- Do not reuse old tubing when installing or switching to hydrogen gas. Hydrogen gas tends to remove contaminants left on old tubing by previous gases (by helium, for example). These contaminants can appear in output as high background noise or hydrocarbon contamination for several weeks.
- Especially do not use old copper tubing, which can become brittle.

Hydrogen supply systems

To eliminate pressure surges, use a two-stage pressure regulator with each hydrogen gas cylinder. Stainless steel, diaphragm type regulators are recommended. The pressure flow to the back of the JetClean or CI flow module is approximately 25 psi. (See **Figure 12.**)



Gas Clean filter configuration will vary depending on the application.

Figure 12. Hydrogen gas cylinder supply tubing

The type of regulator you use depends on the gas type and supplier. The Agilent catalog for consumables and supplies contains information to help you identify the correct regulator, as determined by the Compressed Gas Association (CGA). Agilent Technologies offers pressure-regulator kits that contain all the materials needed to install regulators properly.

5 Hardware

Pressure regulator gas supply tubing connections

Pressure regulator gas supply tubing connections

Use PTFE tape to seal the pipe-thread connection between the pressure regulator outlet and the fitting to which you connect the gas tubing.

Instrument grade PTFE tape (part number 0460-1266), from which volatiles have been removed, is recommended for all fittings. Do not use pipe dope to seal the threads; it contains volatile materials that will contaminate the tubing.

Changing the Hydrogen Supply Filter

Periodically change the filter on the hydrogen supply to the JetClean system. If the system is pressurized with hydrogen you must evacuate hydrogen from the filter before removing the filter.

- 1 Close the hydrogen supply manual shutoff valve ① labeled hydrogen, to the mass flow controller (MFC). (See Figure 11 on page 42.)
- 2 Close the manual shutoff valve ② on the hydrogen supply filter inlet.
- 3 In the Tune view, open the hydrogen gas selection valve. This evacuates the hydrogen between the external manual shutoff valve through the pressure regulator, MFC, analyzer, turbo and foreline pumps.
- 4 Vent the MS.
- 5 Verify the filter bypass line exit shutoff valve ③ is closed and remove the threaded plug from this valve.
- 6 Remove the plug on the tubing line to the foreline pump inlet so it can attach to the filter bypass line in the next step.
- 7 Attach the filter bypass line exit valve ③ to the tubing line attached to the foreline pump inlet.
- 8 If the MS is off, turn it on and wait until the turbo pump starts. The JetClean system is now under vacuum.
- 9 In your Acquisition software program, go to the Tune view and set the MFC to maximum flow so that the pressure regulator and MFC both open fully.
- 10 Open the shutoff valve ④ on the filter outlet line going to the foreline pump inlet. This evacuates hydrogen from the filter through the foreline pump to a lab hood exhaust.
- 11 After 10 minutes, close the shutoff valve ④ on the filter outlet line going to the foreline pump inlet.
- 12 Remove the old filter and replace it with a new one.
- 13 With the vent line valve ⑤ closed, remove the tubing from this valve that is going to the foreline pump and plug the end of this tubing and also plug this valve. This prevents someone from mistakenly opening this valve and allowing hydrogen to flow directly into the laboratory.
- 14 Ensure that the vent shutoff valves on the filter inlet ③ and filter outlet ④ are closed.

5 Hardware

Changing the Hydrogen Supply Filter

- 15 In your Agilent MassHunter GC/MS Acquisition software program, go to the Tune view and close the hydrogen gas selection valve.
- 16 With the vacuum pump still running, open the hydrogen shutoff valves on the filter inlet ② and filter outlet ① and use an electronic hydrogen leak tester to test the system for leaks.

Tune the 5975/5977 series MS for an Acquire & Clean Mode Method

- 1 In the Instrument Control view, select **MS Tune File** from the **Instrument** menu to load a tune file for the JetClean method.
- 2 Select **Edit Tune Parameters** from the **Instrument** menu to display the Manual Tune dialog.
- 3 Click the **JetClean** tab to view the operations that can be done on the mass flow controller (MFC).
- 4 Verify that the analyzer is under vacuum and then open the manual shutoff valve to the JetClean hydrogen supply.
- 5 Click **Purge Gas**, set the purge time to 300 seconds, click **Apply** to start the purge, and wait for the purge to finish.
- 6 Select **Hydrogen flow**, enter the hydrogen flow for your method, and click **Apply** to set this flow for the MFC.
- 7 Perform an autotune or manual tune with this hydrogen flow active.
- 8 Save the tune file.

5 Hardware

Tune the 7000/7010 Series Triple Quad MS for an Acquire & Clean Mode Method

Tune the 7000/7010 Series Triple Quad MS for an Acquire & Clean Mode Method

- 1 In the Instrument Control view, select **MS Tune** from the **Instrument** menu to display the Manual Tune dialog.
- 2 Click **Files and Reports** then click **Load** to load a tune file for JetClean.
- 3 Click the **Manual Tune Ion Source** tab and go to the **JetClean** area to view the Operations that can be done on the JetClean mass flow controller (MFC).
- 4 Verify that the analyzer is under vacuum and then open the manual shutoff valve to the JetClean hydrogen supply.
- 5 Click **Hydrogen** to enable hydrogen gas flow to the MFC for the JetClean system.
- 6 Click **Purge** to start a purge with hydrogen gas and wait for the purge to finish.
- 7 Enter the **Hydrogen Flow** value for your method, and click **Set** to set this flow for the MFC.
- 8 Perform an autotune or manual tune with this hydrogen flow active.
- 9 Save the tune file.

Manually Cleaning the Ion Source

See your system's Operating Manual for details on how to manually clean the ion source.

General Laboratory Precautions

The use of hydrogen as a GC carrier gas, or as a reagent gas for the JetClean system, is potentially dangerous. Hydrogen presents a number of dangers. Some are general, others are unique to GC or GC/MS operation.

Be sure to read the following information and the section on Hydrogen Safety in your instruments Operating manual before operating the JetClean system.

- 1 Clearly identify the JetClean system hydrogen tubing runs with local code required hydrogen labels.
- 2 Use leak-checking equipment to periodically monitor for leaks in the JetClean system. This includes the hydrogen supply source system (tank or generator), hydrogen supply lines to the JetClean gas inlet on the MS, the mass flow controller (MFC) system plumbing, the reagent system plumbing including the CI calibration valve/vial, and transfer line to the analyzer chamber. Agilent highly recommends the G3388B Leak Detector to safely check for hydrogen leaks.
- 3 Eliminate from your laboratory as many ignition sources as possible (for example, open flames, devices that can spark and sources of static electricity).
- 4 Do not allow hydrogen from a high pressure cylinder to vent directly to atmosphere due to danger of self-ignition.
- 5 If you have a pin hole in your line, you can have a flame, and hydrogen flames are invisible. Additionally, the lower explosive limit for hydrogen is 4%.
- 6 Include hydrogen sensors in your laboratory in the locations as recommended by the sensor manufacture.

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