



**Agilent GC/MSD  
Polycyclic Aromatic  
Hydrocarbons (PAH)  
Analyzer with JetClean**

**Quick Start Guide**



**Agilent Technologies**

## Notices

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### Software Revision

This guide is valid for A.01.xx revisions of the Agilent SP1 7890-0528 Agilent GC/MSD Polycyclic Aromatic Hydrocarbons (PAH) Analyzer with the Self-Cleaning Ion Source software, where xx refers to minor revisions of the software that do not affect the technical accuracy of this guide.

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# 1 Introduction

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Identifying and quantifying Polycyclic Aromatic Hydrocarbons (PAHs) at low levels in complex samples presents some challenges. PAHs have similar spectra and SIM ions which can complicate identification if retention times vary. They stick to cooler surfaces and will tail, making integration more difficult. The Agilent GC/MSD PAH Analyzer is a complete system that is factory configured and tested to quickly identify a range of commonly analyzed PAHs with ultimate sensitivity. The PAH analyzer comes with a Selected Ion Monitoring (SIM) database of 27 PAHs and 5 deuterated ISTDs.

The Analyzer is configured with the Agilent proprietary Capillary Flow Technology, enabling rugged, reliable GC column backflushing. Backflushing the GC column shortens run times, extends column life, reduces chemical background noise, provides consistent retention times and spectra, and keeps the MS ionization source cleaner. Also included with the analyzer is Agilent's proprietary JetClean feature. JetClean introduces a low flow of hydrogen directly into the source while samples are being analyzed. This low flow of hydrogen improves analytical performance and significantly reduces the need for manual source cleaning.

The analyzer is also now available with Agilent's High Efficiency Source (HES). Note that to be compatible with the HES, method adjustments are required, as described in Appendix 2.



## Instrument Configuration and Method

The complete method is on the CD and the instrument parameters can also be found in the acqmeth.txt file within the method folder. Below are some key highlights:

- Split/splitless inlet operating at 320 °C, pulsed at 50 psi for 0.5 min.
- Single-taper splitless liner with glass wool, p/n 5190-2293.
- Column 1, Agilent J&WDB-EUPAH, 30 m x 0.25 mm x 0.25 µm, p/n 122-9632. Retention Time Locked (RTL) to 1-methylphenanthrene at 12.500 min, constant flow mode, ~1.1 mL/min, initial pressure ~17 psi
- Column 2, Agilent Ultimate Plus deactivated tubing, 1.36m x 0.15mm, p/n CP801510, operating at 1.48 mL/min psi constant flow, initial pressure 4.0 psi. Column 2 is configured as a composite column. The Main segment, 1.19 m is heated by the oven. The Out segment, 0.17 m, is heated by the MS transfer line.
- Backflush for 5 min with Column 2 at 80 psi (Aux), flow = 18 mL/min and column 1 at -5.8 mL/min. Oven temperature = 335 °C during backflush.
- Data acquisition in 11 SIM groups, 5 ISTDs and 27 PAHs have 1 quant ion and 2-3 qualifier ions.
- JetClean feature set to deliver 0.33 mL/min hydrogen flow into the source while samples are being analyzed.

Your PAH Analyzer was configured and checked out at the factory to ensure that your system will be ready to run samples immediately after installation in your lab. The factory test results for your PAH Analyzer can be found on the Specials Factory Information CD ROM that was shipped with your instrument.

## Where to Find More Information on Using your PAH Analyzer

### Application notes and publications

You can find more of information about PAH analysis in the application notes and publications included with your Specials Factory Information CD ROM.

You can find more material at <http://www.agilent.com> and type **PAH analysis** into the search field.

### QuEChERS extraction procedures and ready-to-use kits

The QuEChERS extraction procedure for PAHs in sediments and seafood is being used by labs around the world. For more information about QuEChERS extractions, go to the following Agilent web site:

<http://www.agilent.com> and type QuEChERS PAH into the search field.



## What is on the Specials Factory Information CD ROM

Your PAH Analyzer comes with a CD that contains the following:

- The GC/MSD method used for running the checkout sample and for general sample use (**PAH\_checkout\_acq\_PHS�.m**). This method is a pulsed-hot-splitless SIM method with 27 analytes plus 5 ISTDs and is retention time locked at the factory.
- The GC/MSD method used for data analysis of the checkout sample (**PAH\_checkout\_DA.quantmethod.xml**)
- Checkout sample data file (**PAH\_checkout\_PHS�\_1.D**) and report (**QuantReport\_ISTD\_ResultsSummary\_FAST\_B\_04\_001.xlsx**) obtained at the factory for a 1 µL injection of the 100 ppb GC/MSD PAH Checkout Sample (p/n G3440-85009) [Note: the checkout sample is diluted to 10 ppb when checking out a system with the HES as described in appendix 2]
- Agilent Application Notes that discuss PAH analysis
- A copy of this Quick Start Guide
- A copy of the JetClean Module User Manual
- An Excel spreadsheet of RTs and SIM ions for the 32 compounds

## Copy Factory Methods and Test Results to the Hard Disk

- 1 If open, exit the MassHunter Data Acquisition and MassHunter Quantitative Data Analysis programs.
- 2 Copy **PAH\_checkout\_acq\_PHSL.m**, method from the Specials Factory Information CD ROM to the **D:\MassHunter\GCMS\1\Method** folder.
- 3 Copy **PAH\_checkout\_DA.quantmethod.xml**, method from the Specials Factory Information CD ROM to the **D:\MassHunter\GCMS\1\Data** folder. The quant method is also imbedded in the batch, **PAH\_checkout.batch.bin**, but was saved separately if needed for later use.
- 4 Copy the folder **QuantResults**, from the Specials Factory Information CD ROM to the **D:\MassHunter\GCMS\1\Data** folder. This QuantResults folder contains the batch file, **PAH\_checkout.batch.bin**, that you will use later for the checkout sample.
- 5 Copy **PAH\_checkout\_PHSL\_1.D** from the Specials Factory Information CD ROM to the **D:\MassHunter\GCMS\1\Data** folder. The available checkout datafiles may be higher numbered.
- 6 Edit the **Msdchem.ini** file in the **C:\GCMS\msexe** folder to show **\_METHFILES=PAH\_checkout\_acq\_PHSL.m**, so this method will be loaded as you bring up the acquisition software.

The **acq.ms** file located in each method folder contains all the SIM ions and groups for the method. The complete set of these SIM settings can be duplicated in another method by copying this file from one method to another. Make sure you back up the original **acq.ms** in the target method folder.





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## Overview of JetClean

The Agilent JetClean accessory is included in this PAH II Analyzer. Hardware, installation, and operation details can be found in the separate manual for the JetClean module (p/n G7077-90018).

JetClean should be used in Acquire & Clean (continuous cleaning) mode while analyzing all samples for PAHs, including standards, QC samples, real samples, etc. The small flow of hydrogen into the ion source will have little effect on PAH spectra or sensitivity. It will improve linearity, reproducibility and ISTD stability. The method flow setting for hydrogen should be 0.33 mL/min.

The hydrogen effect on non-PAH compounds can vary. If this Analyzer is used for compounds other than PAHs the user must determine the effect of the hydrogen.

Detailed instructions for setting up and using JetClean with this analyzer are provided in Appendix 1 of this manual titled "Setting Up JetClean."

## Overview of the Checkout Sample

At the factory, your system was checked out by making 1  $\mu\text{L}$  injections of a 100 ppb checkout standard (10 ppb if your MSD has the HES, see Appendix 2). We have provided you with the exact method used during factory checkout:

**PAH\_checkout\_acq\_PHSL.m.** The method was retention time locked at the factory. At installation, the Agilent service engineer will check and relock (if necessary) the **PAH\_checkout\_acq\_PHSL.m** method.

The checkout sample, G3440-85009, comes in two separate snap-open ampoules. Duplicates of each ampoule are provided. One of the ampoules contains 27 PAHs at 10 ppm each in acetone. The other ampoule contains 5 ISTDs at 50 ppm each in acetone. Acetone was used so that either standards or spikes could be made into various solvents. If using the HES, see Appendix 2.

To make the checkout sample proceed as follows:

- 1 Take a new 2 mL autosampler vial and fill it to the 1 mL mark with a suitable solvent. Isooctane is preferred but you can also use acetone, hexane, or toluene.
- 2 Snap open one of the two ampoules and transfer 10  $\mu\text{L}$  to the 2 mL autosampler vial (1  $\mu\text{L}$  if using the HES). Save the remaining volume in a separate crimp cap vial.
- 3 Snap open the second (non-duplicate) of the two ampoules and transfer 10  $\mu\text{L}$  to the same 2 mL autosampler vial (1  $\mu\text{L}$  if using the HES). Save the remaining volume in a separate crimp cap vial.

The remaining volumes saved in the above steps can be used to make additional calibration levels or spikes.

The above procedure results in a dilution of 1:100 of each of the ampoules into one vial. The PAH concentrations are 100 ppb each and the ISTD concentrations are 500 ppb each in the diluted mix. This is the ISTD level that is recommended by the NOAA method. [Note that if your analyzer uses the HES, the ISTD concentration needs to be much lower. See Appendix 2.]

The PAH Analyzer Checkout Sample contains the 27 PAHs and 5 ISTDs shown in [Table 1](#). The concentration of each PAH is 100 ppb (100  $\text{pg}/\mu\text{L}$ ) in the solvent you chose. Please note that some of the PAHs used to make up this sample were not 100%

pure, and that there may be some small impurities or isomers of the major components present. The listed R.T.s are approximate and are column dependent.

**Table 1** PAHs contained in the Checkout Sample (p/n G3440-85009)

Compound	CAS #	R.T.
Naphthalene-d8	1146652	5.797
Naphthalene	91203	5.820
1-methylnaphthalene	90120	6.458
2-methylnaphthalene	91576	6.648
Biphenyl	92524	7.060
2,6-dimethylnaphthalene	581420	7.107
Acenaphthylene	208968	7.892
Acenaphthene-d10	15067262	8.014
Acenaphthene	83329	8.073
2,3,5-trimethylnaphthalene	2245387	8.296
Fluorene	86737	8.858
Dibenzothiophene	132650	10.789
Phenanthrene-d10	1517222	11.007
Phenanthrene	85018	11.070
Anthracene	120127	11.138
1-methylphenanthrene	832699	12.500
Fluoranthene	206440	14.250
Pyrene	129000	15.096
Benz[a]anthracene	56553	18.617
Chrysene-d12	1719035	18.788
Chrysene	218019	18.882
Benzo[b]fluoranthene	205992	21.913

**Table 1** PAHs contained in the Checkout Sample (p/n G3440-85009)  
(continued)

<b>Compound</b>	<b>CAS #</b>	<b>R.T.</b>
Benzo[k]fluoranthene	207089	21.979
Benzo[j]fluoranthene	205823	22.081
Benzo[e]pyrene	192973	22.975
Benzo[a]pyrene	50328	23.127
Perylene-d12	1520963	23.400
Perylene	198550	23.491
Dibenz[a,c]anthracene	215587	26.503
Dibenz[a,h]anthracene	53703	26.668
Indeno[1,2,3-cd]pyrene	193395	26.732
Benzo[ghi]perylene	191242	28.187

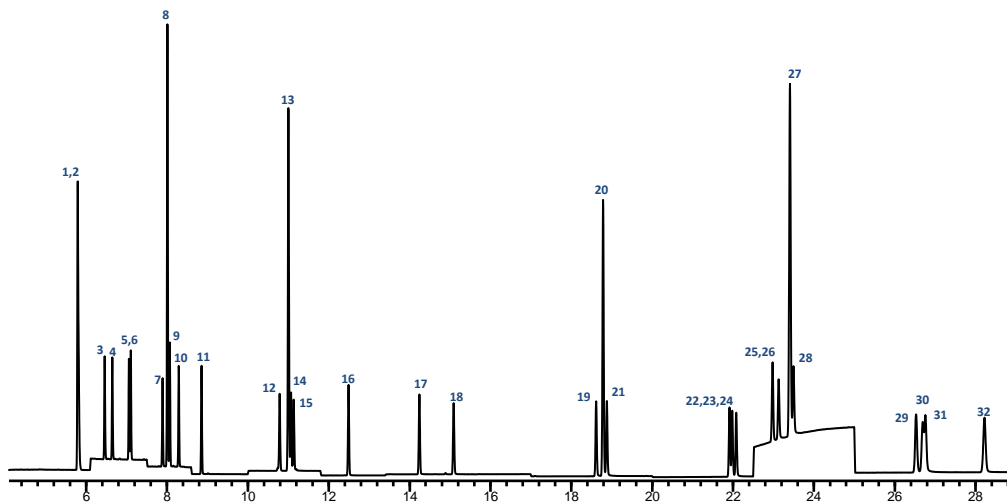


## Running the Checkout Sample

### Procedure

- 1 Start the MassHunter GC/MS Data Acquisition software. The **PAH\_checkout\_acq\_PHSL.m** method is loaded if you edited the **MSDchem.ini** file **\_METHFILE\$** before starting.
- 2 If it is not automatically loaded, load **PAH\_checkout\_acq\_PHSL.m** method.
- 3 Make a 1  $\mu\text{L}$  injection of the 100 ppb checkout sample (1 ppb with HES) you just co-diluted.
- 4 Name the Data File as **PAH\_onsite\_1.D** in **D:\MassHunter\GCMS\1\Data** folder.

1	Naphthalene-d8	12	Dibenzothiophene	23	Benzo[k]fluoranthene
2	Naphthalene	13	Phenanthrene-d10	24	Benzo[j]fluoranthene
3	1-methylnaphthalene	14	Phenanthrene	25	Benzo[e]pyrene
4	2-Methylnaphthalene	15	Anthracene	26	Benzo[a]pyrene
5	Biphenyl	16	1-methylphenanthrene	27	Perylene-d12
6	2,6-dimethylnaphthalene	17	Fluoranthene	28	Perylene
7	Acenaphthylene	18	Pyrene	29	Dibenz[a,c]anthracene
8	Acenaphthene-d10	19	Benz[a]anthracene	30	Dibenz[a,h]anthracene
9	Acenaphthene	20	Chrysene-d12	31	Indeno[1,2,3-cd]pyrene
10	2,3,5-trimethylnaphthalene	21	Chrysene	32	Benzo[ghi]perylene
11	Fluorene	22	Benzo[b]fluoranthene		

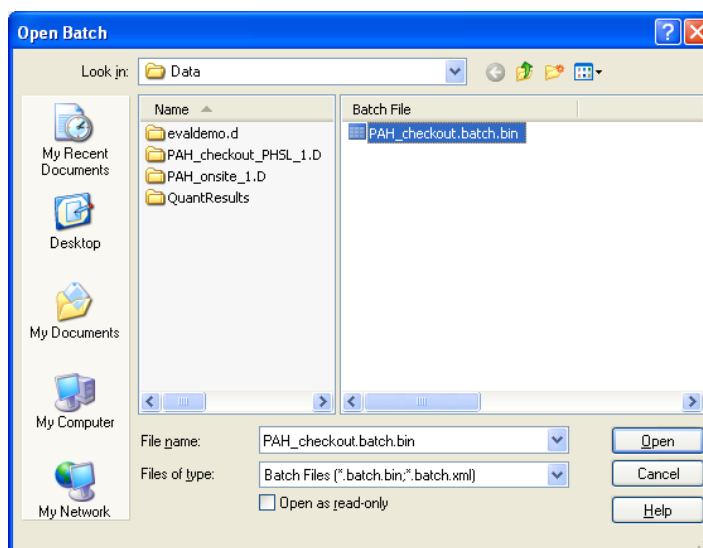


**Figure 1** SIM TIC of 100 pg/ $\mu\text{L}$  Checkout Standard with 5 ISTDs at 500 pg/ $\mu\text{L}$

## Reviewing Data and Generating a Report

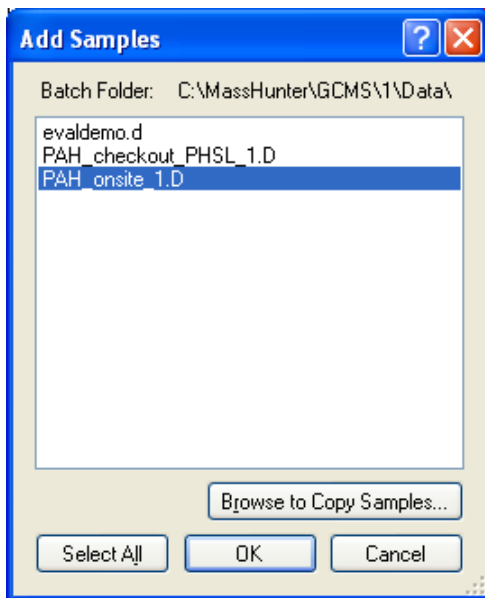
### Procedure

- 1 Open MassHunter Quantitative Data Analysis.
- 2 Select **File > Open Batch...** and select **PAH\_checkout.batch.bin**.

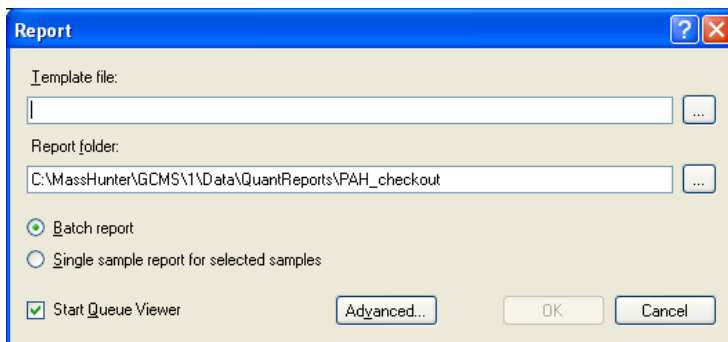


## 2 Operations

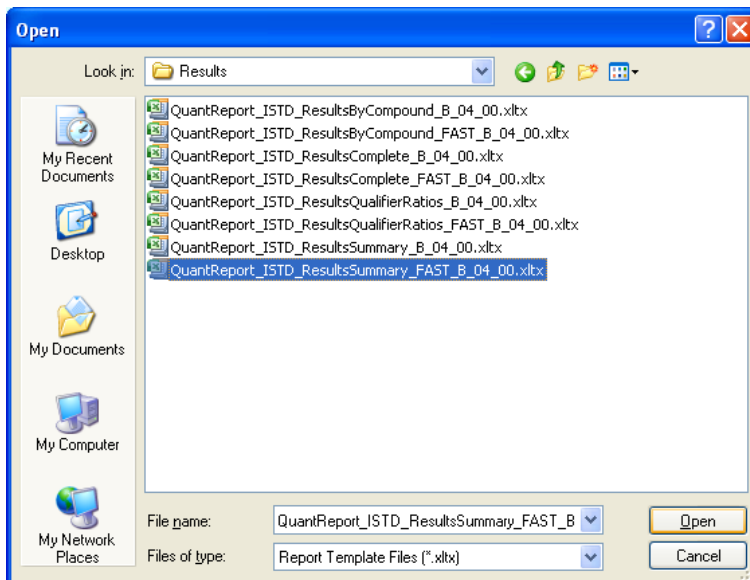
- 3 Select **File > Add Samples...** and select **PAH\_onsite\_1.D**, the file from the checkout sample you ran. Your data is probably on **D:\**.



- 4 Press **Analyze Batch**.
- 5 Save the batch.
- 6 Select **Report > Generate....** to display the **Report** dialog box.

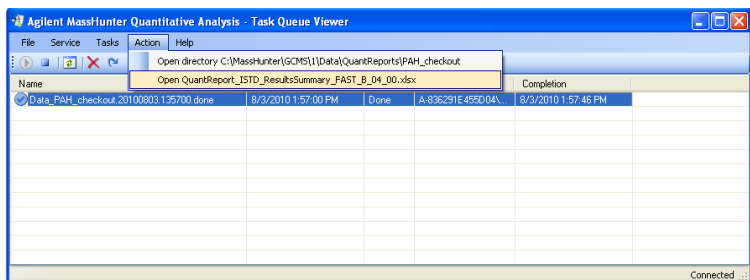


- 7 The **Template file** field is empty. Select the report template, as shown below, for the GC/MSD Analyzer.



This report will display all the analytes found and the respective responses.

- 8 After the **Task Queue Viewer** shows the completion time of the task, highlight the task. From the menu bar of the **Task Queue Viewer**, select **Action** and **Open** the .xlsx report.



- 9 Review your report and compare it with the factory .xlsx report for the **PAH\_checkout\_PHSL\_1.D** generated for this specific Analyzer. The results from the factory are on the Specials Factory Information CD ROM.
- 10 Relock the method if the 1-methylphenanthrene peak is more than  $\pm 0.015$  minutes different than the method's locking compound retention time of 12.500 minutes. See [Relocking the Data Acquisition Method](#) on page 21.

The **PAH\_checkout\_acq\_PHSL.m** method can now be used by MassHunter GC/MS Data Acquisition to acquire sample data. The MassHunter Quantitative Analysis quantitation method used in the PAH\_checkout batch can now be used for quantitative data analysis.

## Signal-to-Noise Check

There is no signal-to-noise (S/N) specification for the PAH Analyzer. In order to establish a guideline for S/N, multiple systems were run with the checkout mix diluted in isooctane. The Acquire and Clean mode of JetClean was used with the hydrogen flow set to 0.33 mL/min.. The S/N values were calculated using the Calculate Signal-Noise selection in MSD ChemStation Analysis. S/N values were determined for the 192 SIM ion for 1-methylphenanthrene at 12.500 minutes. The noise region specified was 11.9-12.1 minutes, as Peak-to-Peak noise. S/N values for the 192 ion ranged from 2240 to 2410. These values are a guideline only. Upon installation, the S/N values can be calculated on both the checkout run you make and the file provided on the factory CD, and compared.

## Relocking the Data Acquisition Method

### Auto Retention Time Locking (RTL)

This Analyzer is retention time locked at the factory to 1-methylphenanthrene at 12.500 minutes.

The Analyzer should only have to be relocked upon installation (see below). If the full initial locking process has to be done, then follow this procedure:

The retention time of the Analyzer can be automatically locked using either a full-scan method or a SIM method. The process of locking a method is selecting the **Acquire RTLock Calibration Data...** from the GC/MSD data acquisition **Method** menu. The system will automatically make 5 injections with varying inlet pressures to change the retention time of a locking compound.

If using a SIM method, make sure the locking compound transitions are in all the time segments where the peak will show up due to the adjusting of inlet pressure. Specify the ion from scan or SIM (192) in the RTL process if the locking compound is 1-methylphenanthrene.

### Relocking

If you replaced or trimmed the GC column, you have to make an injection to relock the retention times so you do not have to adjust the time segments in your acquisition method.

First, load the **PAH\_checkout\_acq\_PHSL.m** method. There are two ways to relock a method:

- The normal process of relocking a method is selecting the **Relock Method...** from the GC/MSD data acquisition **Method** menu and entering the required information to start a run. The target retention time for the 1-methylphenanthrene peak is 12.500 minutes.

- If a datafile is already collected, for example, **PAH\_checkout\_PHSL\_1.D**, a quicker way to relock the system is by typing in and executing an **acqlock 2,1** command in GC/MSD Acquisition command line. Before executing the command, make sure the method is loaded and the data file name and data path are entered properly in the run method screen as shown below.

The screenshot shows the 'Start Run' dialog box with the following configuration:

- Current Method Injection Style: GC ALS
- Inlet Location:  Front,  Rear,  Dual
- MS Connected to:  Front Inlet,  Rear Inlet
- Operator Name: Kai
- Data Path: D:\MassHunter\GCMS\1\DATA
- Front Inlet:
  - Data File Name: PAH\_checkout\_PHSL\_1.D
  - Sample Name: [empty]
  - Misc. Info: [empty]
  - Expected Barcode: [empty]
  - Sample Amount: 0
  - Multiplier: 1
- Rear Inlet:
  - Data File Name: relock\_MRM\_June11.D
  - Sample Name: [empty]
  - Misc. Info: [empty]
  - Expected Barcode: [empty]
  - Sample Amount: 0
  - Multiplier: 1

After relocking the **PAH\_checkout\_acq\_PHSL.m** acquisition method, save it with the new locked pressure.







## 3 Columns and Supplies

Description	Part number	Installed	Extra
<b>Columns and column accessories</b>			
Agilent J&W DB-EUPAH GC Column 30 m x 0.25 mm x 0.25 $\mu$ m	122-9632	1	0
Column ferrule, MMI, graphite (10/pkg)	5080-8853	1	1 pkg
Column nut, GC capillary (2/pkg)	5181-8830	1	1
Column Nut, Self Tightening MSD Nut	5190-5233	1	0
Ferrule, 0.4mm VG 0.1-0.25 col 10/PK	5181-3323	1	1 pkg
Ultimate Plus deactivated Fused Silica 5 m x 0.15 mm, flow restrictor	CP801505	1.36 m	3.6 m
<b>GC inlet and instrument supplies</b>			
Inlet liner, 4-mm for hot splitless injections, single taper, deactivated, with glass wool	5190-2293	1	2
O-ring, inlet liner, non-stick (10/pkg)	5188-5365	1	1 pkg
Septum, advanced green (50/pkg)	5183-4759	1	1 pkg
Big universal trap for helium purification	RMSH-2	2	0
Universal/External split vent trap (includes 3 cartridges)	RDT-1020	0	0
Split Vent Trap PM kit	5188-6495	0	0



### 3 Columns and Supplies

Description	Part number	Installed	Extra
<b>Backflush and capillary flow device supplies</b>			
Internal nut for capillary flow device (installed on purged ultimate union)	G2855-20530	2	2 (plugs)
Ultimetal Plus Flexible Metal ferrule for 0.25 mm and smaller columns (10/pkg)	G3188-27501	2	1 pkg
1/16-inch Brass Swagelok tee	0100-0680	1	0
Bleed line, 0.0625-in od x 0.010-in id x 100 cm, 316 SS tubing	0100-2354	1	0
Bleed tee ferrule, 1/16-inch Vespel (10/pkg)	0100-1329	3	7 pieces
MSD column installation tool	G1099-20030	0	1
Capillary Flow Ferrule Pre-Swage Tool	G2855-60200	0	1
Capillary tubing cutter (4/pkg)	5181-8836	0	1
<b>Chemical standards</b>			
PAH analyzer checkout sample (2 vials of 27 PAHs at 10 ppm each in acetone and 2 vials of 5 ISTDs at 50 ppm each in acetone)	G3440-85009	0	1



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## Auto RTL Failed

### **Ion signals too weak - MassHunter cannot integrate them**

Use MassHunter Qual to examine all five locking data files inside the method folder. Use > 1 ppm standards, or set a higher electron multiplier Gain value if the ion signals are weak.

### **The R<sup>2</sup> value is less than 0.95**

Use MassHunter Qual to examine all five locking data files inside the method folder. Make sure the locking compound exists in all five runs. If not, make sure the locking compound transitions exist in all relevant time segments.

### **The ions entered for MassHunter identification are incorrect**

Use MassHunter Qual to review the RTL data files and identify the ions acquired and then execute **acqlock 1,1** in GC/MSD Acquisition command line to **lock**. This command will reprocess the five previously collected data files.

#### **NOTE**

The command **acqlock 2,1**, as described earlier, is used for processing a data file to relock.

---

## The GC Never Comes Ready or the Sequence Halted

**Make sure all pressure setpoints can be reached or are properly ignored.**

Fix all leaks, increase gas supply pressure, and configure the purged ultimate union to **Ignore Ready = True**.

## Checking for Leaks

### **UltiMetal Plus Flexible Metal ferrule connection at the purged ultimate union (PUU)**

Make sure the column is cut to the proper length according to UltiMetal Plus Flexible Metal Ferrules Swaging Guide, part number G3188-90001, on the Specials Factory Information CD ROM. The end of the tubing need not be perfectly square, but should not have cracks that extend under the ferrule.

### **Inlet**

Remove the column from the inlet and plug the inlet with a column nut and blank ferrule. After reaching the 10 psi setpoint, set the inlet pressure to **OFF**. There should be less than 0.1 psi drop in one minute.

### **MSD transfer line and PUU connections**

Go to the **Manual Tune** view in the MS acquisition program. Go to the **Profile** tab and set ions 69 and 83 to be profiled. Note the abundance values. Spray the MSD transfer line nut and PUU connections with fluorinated aerosol duster, (for example, Miller-Stephenson MS-222N Aero-Duster: 1,1,1,2-Tetrafluoroethane, CAS 811972). Look for increases in abundances, which would indicate a leak.



## Appendix 1: Setting Up JetClean

The Agilent JetClean accessory is included with this analyzer. When your PAH analyzer is installed, the JetClean parameters are already incorporated in the supplied acquisition methods and are ready to be used. This section describes how to add JetClean to other methods, or to reset the JetClean parameters if necessary.

The JetClean module is designed to add a small flow of hydrogen gas directly into the ionization source when desired. When the source filaments are on, the presence of hydrogen produces a cleaning action inside the source. This results in a significant time saving for the laboratory, since the cleaning is done in place. Hydrogen cleaning can significantly extend the time between manual cleanings.

There are two modes of operation for JetClean. One is called **Clean Only** and the other **Acquire and Clean**. **Clean Only** mode is used to occasionally clean the source with hydrogen, much as you would with manual cleaning. **Clean Only** is run in off-line fashion when sample data is NOT being acquired. More information on this mode is available in the JetClean manual provided on the analyzer disk.

The **Acquire and Clean** mode is used with this analyzer. With this mode, a low flow of hydrogen (0.33 mL/min) is added continuously to the source while the analysis is taking place. This flow cleans the source during normal operation. For PAH analysis, this mode provides improved analytical performance and greatly reduces the need for manual source cleaning. The small flow of hydrogen into the ion source has little effect on the spectra or sensitivity of PAHs while improving linearity, reproducibility, and signal stability.

**Acquire and Clean** mode should be used when acquiring data for all PAH samples, including calibration standards, real samples, etc. The method flow for hydrogen is set to 0.33 mL/min. Use the following steps to set up JetClean for the PAH acquisition methods.

### Configure Gases

When starting MassHunter Data Acquisition for the first time (e.g. after new installation or start-up after a new revision is installed), a window should pop-up to select the type of installed system with choices of **None**, **JetClean -EI only system**, or **CI/JetClean -EI/CI system**.

Select **JetClean – EI only system** or **CI/JetClean -EI/CI system**, depending on what is installed on the mass spectrometer. The analyzer was developed with **JetClean -EI only system** and details relating to the **JetClean -EI only system** will be discussed. Selecting the **JetClean – EI only system** automatically enters the gas configuration details; the pop-up window for configuring gases is shown below.

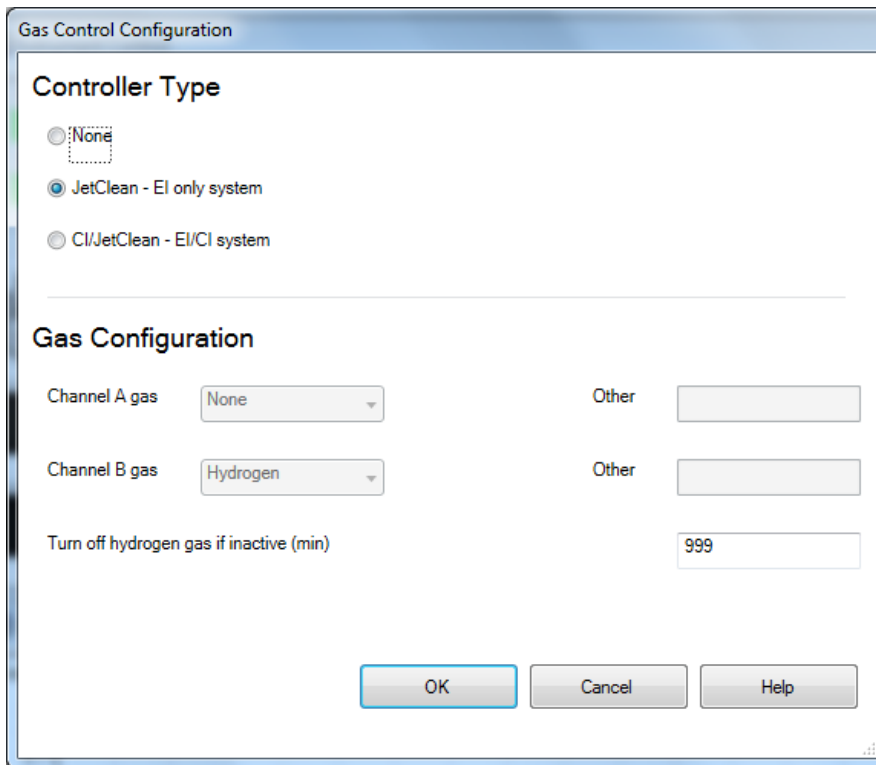


Figure A1. Configure MS gases window.

## Change the Inactive Time

The JetClean system will turn off hydrogen flow, based on the inactive time setting (minutes). The hydrogen flow will stop when the inactive time, which is set by the user, is reached. Inactive time is defined as when data is not being acquired and can be set between 10 minutes and 999 min. The inactive time is found in **Instrument control view**; select **Instrument > Configure MS Gases**. The inactive time can be altered in the pop-up window and it is best to select a value that is sufficiently longer than the method length. For the analyzer development, the maximum of 999 min was used (~16 hrs), which is shown in the above figure. Click **OK** and save the method.

## Adding JetClean Acquire and Clean mode to a method

There are 2 basic steps to adding JetClean Acquire & Clean mode to a method.

1. Add hydrogen cleaning to the MS tune file used with the method.
2. Add hydrogen cleaning to the acquisition method.

## Adding H<sub>2</sub> flow to the tune file used by the method

To run a method with hydrogen, the tune file associated with the method must have the hydrogen flow set as a parameter and saved.

If there is not a saved tune file with H<sub>2</sub> flow, an existing tune file will need to be modified or a new tune file will have to be created. In Instrument Control view, select **View > Tune and Vacuum Control**. In **Tune and Vacuum Control** view, select **File > Load Tune Parameters**. Select the tune file appropriate for your hardware and method. Load **etune.u** for the Inert Extractor source or **HES\_atune.u** for the HES source. The temperatures stored with these tunes are often different from what the method requires and the temperature setpoint box will appear. Set the temperature of the source to 320C and the quad to 180C and select **Apply**.

Next, select **Parameters > Manual Tune**. Click on the JetClean tab, then click on the bubble next to hydrogen flow, and enter 0.33 mL/min into the box. Then, click **Apply**. The system will start the hydrogen flow and once the flow is stable, the block box will show “**Gas B Done 0.33 mL/min**” in green lettering in the black box.

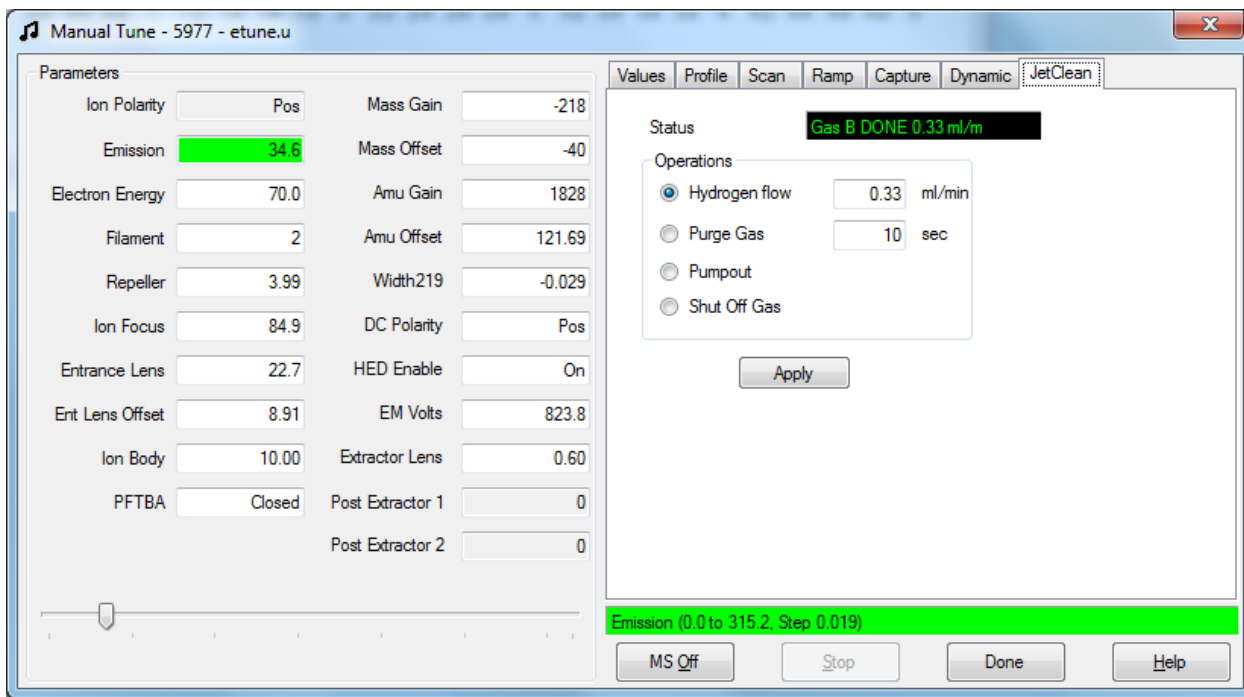


Figure A2. JetClean tab of the Manual Tune window.

Select the Values tab and verify that the MS temperatures are 320 °C for the source and 180 °C for the quad. Then, click **Done** and re-save the tune parameters under the

same name (**etune.u** or **HES\_atune.u**).

Next, select **Tune > Extraction Source Tune (or HES Autotune)**. The system will autotune with the hydrogen flowing at 0.33 mL/min and the correct temperatures. Review the report after the tune has completed to verify that the H<sub>2</sub> was on. Re-save the tune parameters as **etune.u** (or **HES\_atune.u**). Select **File > Save Tune Parameters** and save the file to a different name for your method. For the PAH Analyzer, use **PAH\_330H2\_etune.u** (or **PAH\_330H2\_HES\_atune.u**).

Note that once the tune file with the correct temperatures and hydrogen flow has been created and saved to the correct name for your method, you can easily retune that file directly by using the menu item in **Instrument Control** view. Make sure the correct method is loaded and then select **Instrument > Tune MSD**. This will re-tune using the tune file associated with the loaded method.

## **Adding H<sub>2</sub> flow to the acquisition method itself**

In **Instrument Control** view, load the method to which hydrogen is to be added, if it is not already loaded. Make sure the tune file with the correct temperatures and hydrogen flow created above is selected as the tune file for the method.

Next, click on the MS parameters icon to open the MS parameters method editor. Select the **JetClean** tab and select **Acquire & Clean** from the drop down menu labeled **Operation**. Then, type 0.33 mL/min into the box labeled Hydrogen flow.

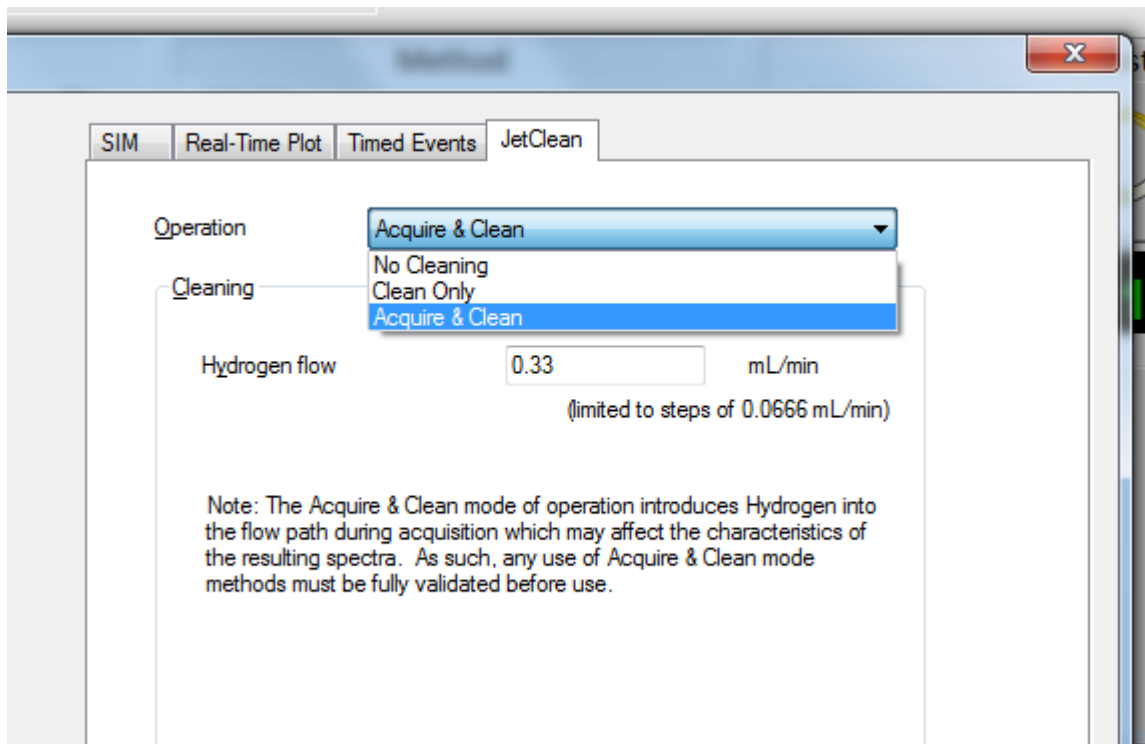


Figure A3. Set-up Acquire & Clean mode in MS parameters method editor in Instrument Control.

The tune file in this view must be the tune file with 0.33mL/min H<sub>2</sub> flow set up in the previous section. If the tune file with 0.33 mL/min has a different name than the one shown in the **Tune File** box, load the correct tune file by clicking the box next to the Tune File name and selecting the correct tune file. When the tune file has a matching H<sub>2</sub> flow to the value in the JetClean panel, select **OK**.

Note: If the H<sub>2</sub> flow does not match between the tune file and value entered into the JetClean panel, a pop-window will appear and indicate that these two values are different. The difference must be fixed before the program will close the MS parameters window.

Click on **Method > Save Method** to save the H<sub>2</sub> flow parameters to the overall method.

## Adding JetClean monitors to Instrument Control view

It is helpful (but not required) to have the JetClean monitors displayed in Instrument Control View. With the method set up above loaded, select the **Instrument > Edit Monitors** menu item. A list of monitor boxes to display is opened. Add the monitors **MS Cleaning Operation** and **MS H2 Flow** to the list of **Selected Monitors** if they are not already there. Select **OK**. Find the monitor boxes and drag them to where they will be visible. When the instrument is running the PAH method, they should display the

JetClean information.

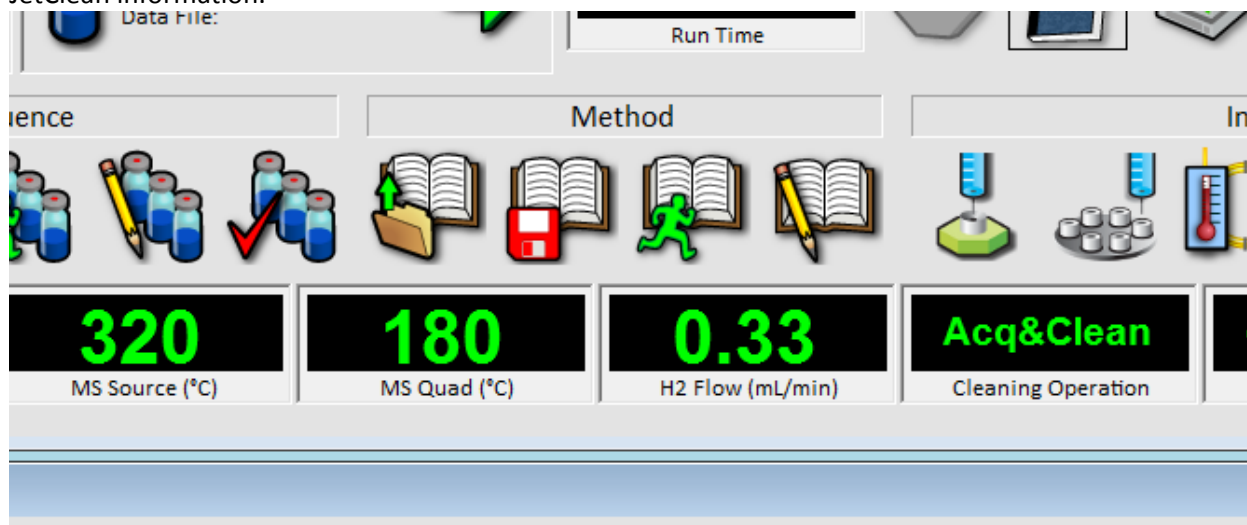


Figure A4. MS Cleaning Operation and MS H<sub>2</sub> Flow monitors displayed in Instrument Control view.

## Note on JetClean behavior

When a method that is configured to use JetClean is loaded with the **Load Method** item under the **Method** menu, the hydrogen flow will NOT be turned on, even though the method calls for it. The hydrogen flow will start when the method is run from either **Run Method** or in a **Sequence**. If you wish to turn on the hydrogen flow after loading a method configured with JetClean, follow the procedure below.

## Manually starting H<sub>2</sub> flow

Select the menu **Instrument >Tune Parameters**. The manual tune window will open with Instrument Control in the background. Select the **JetClean** tab. The hydrogen flow box should have a value of 0.33 mL/min; select **Apply**. The JetClean flow module will pump out and purge hydrogen for ~10-20 seconds before coming ready with a flow of 0.33 mL/min, which is identified by the green type in the black readout box.

After the hydrogen flow comes ready, close the manual tune box, select NO for Saving tune parameters. The manual tune windows will clear and show the Instrument Control window automatically.

## Appendix 2: Using the High Efficiency Source (HES) with the PAH Analyzer

The High Efficiency Source (HES) can be used with the PAH analyzer. The HES is a completely different type of source that generates many more ions per picogram of compound than the standard source. The higher ionization efficiency results lower detection limits for most compounds. When using the HES in a PAH analyzer, there are a few important changes required to the method parameters to obtain the best results.

The first changes required are the calibration range of the analysis. The standard analyzer with the Inert or Inert Extractor source works well from 1 pg to 1000 pg of analyte injected into the column. ISTDs are at 500pg. With the HES, this range should be changed to 0.2 pg to 200 pg of analyte injected into the column. The ISTD level with the HES should be lowered to at most 50 pg, For the earlier PAHs like naphthalene, it is better to lower the ISTD (naphthalene-d8) to 5 pg. At higher concentrations, the ISTD naphthalene-d8 can cause a negative response in the analyte baseline that interferes with integration at the lowest levels.

To make the HES checkout sample proceed as follows:

1. Take a new 2 mL autosampler vial and fill it to the 1 mL mark with a suitable solvent. Isooctane is preferred but you can also use acetone, hexane, or toluene.
2. Snap open one of the two ampoules and transfer 1  $\mu$ L to the 2 mL autosampler vial. Save the remaining volume in a separate crimp cap vial.
3. Snap open the second (non-duplicate) of the two ampoules and transfer 1  $\mu$ L to the same 2 mL autosampler vial. Save the remaining volume in a separate crimp cap vial.

The remaining volumes saved in the above steps can be used to make additional calibration levels or spikes.

The above procedure results in a dilution of 1:1000 of each of the ampoules into one vial. The PAH concentrations are 10 ppb each and the ISTD concentrations are 50 ppb each in the diluted mix. This is the ISTD level that is recommended when using the HES to keep responses in the nominal linear range of the instrument.

Note that when the sample is run and processed by the supplied data analysis method, the reported concentrations of the PAH analytes will be near 100 ppb instead of 10 ppb. This is because the calibration level in the method has the analytes at 100 ppb and the ISTDs at 500 ppb. This result can be used for checkout purposes, but should be changed before running further calibrations or samples.

If you wish to correct this, use the method edit function in MassHunter Quant to change the analytes to 10 ppb and ISTD amounts to 50 ppb.

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