Agilent SP1 7890-0501 GC/MS/MS Pesticide Analyzer

Quick Start Guide

Agilent Technologies

Notices

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CAUTION

A **CAUTION** notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a **CAUTION** notice until the indicated conditions are fully understood and met.

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A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met.

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1 Introduction

Identifying and quantifying dozens (or hundreds) of pesticides in a wide range of fruit and vegetable samples is a complex task. The Agilent GC/MS/MS Pesticide Analyzer is a complete system that's factory configured and chemically tested to quickly identify a broad range of commonly analyzed pesticide residues with ultimate sensitivity. The pesticide analyzer comes with a Multiple Reaction Monitoring (MRM) database of several hundred pesticides.

The Analyzer is configured with Agilent's 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 clean.

Two configurations are available to meet different labs' needs:

- SP1 7890-0501 (# 0501): This method is based upon Agilent's Retention Time Locked (RTL) GC/MS/MS MRM database (500+ pesticides) running in the constant pressure mode with post-column backflushing. It provides flexibility to add GC detectors and can be easily scaled for shorter runtimes.

- SP1 7890-0502 (# 0502): This method is based upon a list of pesticides commonly found in food samples by US laboratories. The GC/MS/MS MRM database, with 200+ pesticides, runs in the constant flow mode with mid-column backflushing. This method provides ultimate performance and shorter cycle time with reduced carrier gas consumption.

Your system is configured as a # 0501 pesticide analyzer with post-column backflushing. Both configurations are easily interchangeable by changing the column(s) and adding/removing a capillary flow restrictor.

Your Pesticide 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 Pesticide 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 Pesticide Analyzer

Application notes and publications

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

Alternatively, go to: http://www.chem.agilent.com/ and select the **Literature Library** under the Quick Links.

List of the target pesticides

A list of target pesticides is included with your Specials Factory Information CD ROM in both pdf and Excel format.

QuEChERS extraction procedures and ready-to-use kits

The QuEChERS extraction procedure for pesticide residues in fruits and vegetables is being used by labs around the world. For a training video, references, and ready-to-use kits for performing QuEChERS extractions, go to the following Agilent web site:

http://www.chem.agilent.com/en-US/products/consumables/samplepreparation/sampliqspe/sampliqquechers/Pages/default.aspx

Alternatively, go to: http://www.chem.agilent.com/ and type QuEChERS into the search field.

What's on the Specials Factory Information CD ROM

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

- The GC/MS/MS method used for running the checkout sample (0501_checkout_PCSL.m). This method is a pulsed-cold-splitless MRM method with 17 analytes and is retention time locked at the factory.
- The GC/MS/MS method used for running the general pesticide analysis (Pulsed_cold_splitless.m) with several hundred analytes.
- The GC/MS/MS method used for running the general pesticide analysis (Hot_splitless.m) with several hundred analytes.
- A signal-to-noise data analysis method (0501_signal-to-noise.m).
- Checkout sample data file (0501_checkout_PCSL_1µL.d) and checkout report obtained at the factory for a 1-µL injection of the 100 ppb GC/MS/MS Pesticide Analyzer Checkout Sample (P/N 5190-0494)
- Agilent Application Notes that discuss pesticide analysis
- A copy of this Quick Start Guide
- Tutorials on Large Volume Injection and Swaging SilTite Ferrules
- A list of target pesticides in both pdf and Excel format
- An Excel database of RTs and MRM transitions for 500+ compounds

Copy Factory Methods and Test Results to the Hard Disk

Copy 0501_checkout_PCSL.m, Pulsed_cold_splitless.m, Hot_splitless.m, and 0501_signal-to-noise.m methods from the Specials Factory Information CD ROM to the “D:\MassHunter\GCMS\1\Method” folder.

NOTE If you do not have a MultiMode Inlet (MMI) installed, you should use the hot-splitless method (0501_checkout_HSL.m) instead of the pulsed-cold-splitless method (0501_checkout_PCSL.m).

NOTE Edit the MSDchem.ini file in the C:\GCMS\msexe folder to show _METHFILE$=0501_checkout_PCSL.m, so this method will be loaded as you bring up the 7000 acquisition software.

Copy 0501_checkout_PCSL_1µL.d from the Specials Factory Information CD ROM to the “D:\MassHunter\GCMS\1\Data” folder.

NOTE The qqqacqmethod.xml file in each method folder has all the MRM transitions in the method. The complete set of MRM transitions can be duplicated in another method by copying this xml file from one method to another. Make sure you back up the original qqqacqmethod.xml in the target method folder.

NOTE The GC parameters can also be found in the acqmeth.txt file within the method folder.

1 Introduction

2 Operations

Oveview of the Checkout Sample

At the factory, your system was checked out by making 1-µL injections of a 100 ppb checkout standard. We have provided you with the exact method used during factory checkout - 0501_checkout_PCSL.m. The method was retention time locked at the factory. At installation, Agilent's service engineer will check and relock (if necessary) the 0501_checkout_PCSL.m method.

The Pesticide Analyzer Checkout Sample contains 17 pesticides shown in Table 1. The concentration of each pesticide is 100 ppb (100 pg/µL) in acetone. Please note that some of the pesticides 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.

Table 1 Pesticides contained in the 100 pg/µL checkout sample (p/n 5190-0494)

Name	CAS #	RT
Dichlorvos	62-73-7	5.831
Mevinphos	7786-34-7	7.595
Ethalfluralin	55283-68-6	11.283
Trifluralin	1582-09-8	11.637
Atrazine	1912-24-9	13.159
Chlorpyrifos Methyl	5598-13-0	16.593
Heptachlor	76-44-8	16.796
Malathion	121-75-5	18.800
Dieldrin	60-57-1	23.870
p,p'-DDE	72-55-9	24.021
Hexazinone	51235-04-2	27.385
Propargite	2312-35-8	27.725
Mirex	2385-85-5	29.835

Table 1 Pesticides contained in the 100 pg/µL checkout sample (p/n 5190-0494) (continued)

Name	CAS #	RT
Fenarimol	60168-88-9	30.403
Coumaphos	56-72-4	31.667
Etofenprox (Ethofenprox)	80844-07-1	33.203
Deltamethrin	52918-63-5	36.001

Running the Checkout Sample

Procedure

1. Start the MassHunter acquisition software. The 0501_checkout_PCSL.m method is loaded if you edited the MSDchem.ini file _METHFILE$ before starting.
2. Load 0501_checkout_PCSL.m method if it is not automatically loaded. This is a pulsed-cold-splitless MRM method.
3. Make a 1-µL Injection of the 100-ppb Checkout Sample (p/n 5190-0494).
4. Name the Data File as 0501_checkout_PCSL.d in D:\MassHunter\GCMS\1\Data folder.

Reviewing Data and Generating a Report

1. Open MassHunter Qualitative Analysis (Qual)
2. Select **File > Open Data File...** to load data file 0501_checkout_PCSL.d
3. Select **Method > Open...** to load the 0501_signal-to-noise.m
4. Select **Actions > Run the Worklist Actions**
5. Open the pdf report in the **Reports** folder within the data file (0501_checkout_PCSL.d) folder

   If all signal-to-noise ratios (of Mirex) are greater than 2500, go to next step.
6. Compare your report with the factory report for the 0501_checkout_PCSL_1µL.d generated for this specific Analyzer. The results from the factory are on the Specials Factory Information CD ROM.

NOTE Relock the method if the Chlorpyrifos Methyl peak is more than ± 0.015 minute different than the method's locking compound retention time of 16.593 minute. See "Relocking the Data Acquisition Method" on page 16.

Relocking the Data Acquisition Method

Auto Retention Time Locking (RTL)

This Analyzer is retention time locked at the factory.

The retention time of the Analyzer can be automatically locked using either a full-scan method or a MRM method. The process of locking a method is selecting the **Acquire RTLock Calibration Data...** from the GC/MS/MS 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 MRM 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 acquired ion(s) from scan (125, 286) or MRM (208) in the RTL process if the locking compound is Chlorpyrifos Methyl.

Relocking

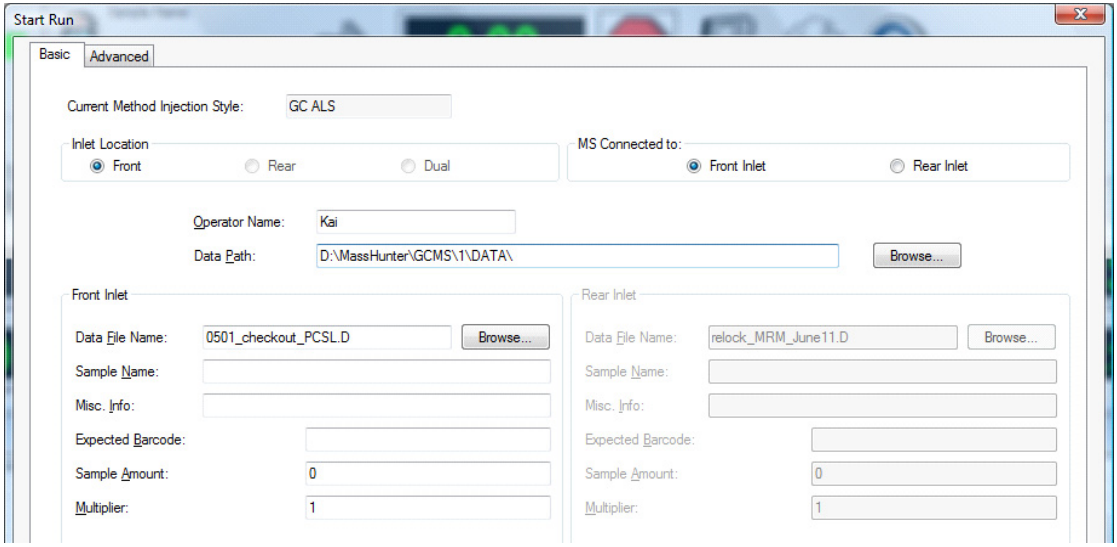
If you replaced or trimmed the GC column, you have to make an injection to relock the retention times so you don't have to adjust the time segments in your acquisition method.

First, load the 0501_checkout_PCSL.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/MS/MS data acquisition **Method** menu and entering the required information to start a run. The target retention time for the Chlorpyrifos Methyl peak is 16.593 minute.

- If a datafile is already collected, for example, 0501_checkout_PCSL.d, a quicker way to relock the system is by typing in and executing **acqlock 2,1** command in GC/MS/MS Acquisition command line. Before executing the command, make sure the method is loaded and data file name and data path are entered properly in the run method screen as shown below.

Save the method with the new locked pressure.



Enter the locked pressure from the above relocking process into your 300-analyte acquisition method (Pulsed_cold_splitless.m or Hot_splitless.m).

2 Operations

3 Columns and Supplies

Description	Part number	installed	extra
Columns and column accessories			
Agilent J&W HP-5ms Ultra Inert GC Column,	19091S-433 UI	1	0
30 m x 0.25 mm x 0.25 µm (# 0501 Analyzer)			
Agilent J&W HP-5ms Ultra Inert GC Column,	19091S-431 UI	2	0
15 m x 0.25 mm x 0.25 µm (# 0502 Analyzer)			
Column ferrule, MMI, graphite (10/pkg)	5080-8853	1	1 pkg
Column nut, GC capillary (2/pkg)	5181-8830	1	1
Swaging nut for MSD interface	G2855-20555	1	0
MSD interface ferrule (10/pkg) - SilTite ferrule (see below)	5188-5361	1	1 pkg
Siltek deactivated Fused Silica 5 m x 0.15 mm, flow restrictor (# 0501 Analyzer)	160-7625-5	0.7 m	4.3 m
GC inlet and instrument supplies			
Inlet liner (2-mm for cold splitless injections, deactivated)	5190-2296	1	0
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

Description	Part number	installed	extra
Backflush and capillary flow device supplies			
Internal nut for capillary flow device	G2855-20530	2	2 (plugs)
(installed on purged ultimate union)			
SilTite ferrule for 0.25 mm and smaller columns (10/pkg)	5188-5361	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, 316SS tubing	0100-2354	1	0
Bleed tee ferrule, 1/16-inch Vespel (10/pkg)	0100-1329	3	7 pieces
Useful tools and chemical standards			
MSD column installation tool	G1099-20030	0	1
SilTite Ferrule pre-swage wrench	G2855-60200	0	1
Capillary tubing cutter (4/pkg)	5181-8836	0	1
Pesticide analyzer checkout sample	5190-0494	0	1
(17 pesticides at 100 pg/µL each in acetone)			

For QuEChERS extraction supplies see www.agilent.com/chem/quechers.

4 Troubleshooting

Auto RTL Failed

Ion signals too weak - MassHunter cannot integrate them

Use MassHunter Qual to examine all 5 locking datafiles inside the method folder. Use > 1 ppm standards or set a higher electron multiplier Gain value if the ion signals are weak.

The r^2 value is less than 0.95

Use MassHunter Qual to examine all 5 locking datafiles inside the method folder. Make sure the locking compound exists in all 5 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 examine all 5 locking datafiles inside the method folder. Identify the ions acquired and then execute **acqlock 1,1** in GC/MS/MS Acquisition command line to lock. This command will reprocess the 5 previously collected datafiles.

NOTE Command **acqlock 2,1**, as described earlier, is used for processing a datafile to relock.

The GC Never Comes Ready or the Sequence Halted

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

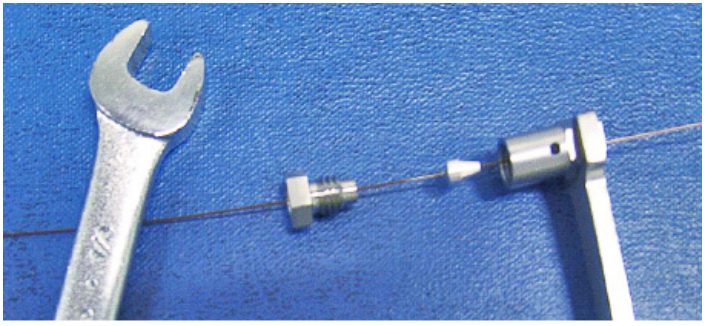
Fix all leaks, increase gas supply pressure, and configure the purged ultimate union to **Ignore Ready = True**.

Checking for Leaks

SilTite ferrule connection at the purged ultimate union (PUU)

Make sure the column is cut to proper length according to the *Swaging SilTite Ferrules Guide*, part number 5969-1573, 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.

Two updates to the Swaging SilTite Ferrules document are noteworthy. First, the Swaging Nut for tightening the ferrule on the column can be replaced by the Swaging Wrench (p/n, G2855-60200, supplied with your Analyzer) which is easier to use in making SilTite connections (see figure below). Second, it is important that the ceramic wafer which is used to make the column cuts have one side (rough) dedicated to only make contact with the column and the other side (smooth) dedicated to riding on the edge of the metallic SilTite ferrule.



Inlet

Blank off column 1, i.e., remove the restrictor from the PUU and place a SilTite plug instead. 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 connection

Spray the MSD transfer line nut with fluorinated aerosol duster, for example, Miller-Stephenson MS-222N Aero-Duster and look for ions 69 and 83 from 1,1,1,2-Tetrafluoroethane, CAS 811972.

4 Troubleshooting

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