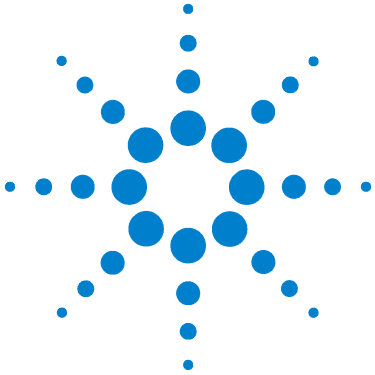


Agilent 926 Pesticide Analyzer

Quick Start Guide

Agilent Technologies

Notices

Manual Part Number

5973-1732

Edition

First Edition, July 2009

Printed in USA

Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19808-1610 USA

Achnowledgements

Microsoft ® is a U.S. registered trademark of Microsoft Corporation.

Software Revision

This guide is valid for A.01.xx revisions of the Agilent 926 Pesticide Analyzer software, where xx refers to minor revisions of the software that do not affect the technical accuracy of this guide.

Warranty

The material contained in this document is provided "as is," and is subject to being changed, without notice, in future editions. Further, to the maximum extent permitted by applicable law, Agilent disclaims all warranties, either express or implied, with regard to this manual and any information contained herein, including but not limited to the implied warranties of merchantability and fitness for a particular purpose. Agilent shall not be liable for errors or for incidental or consequential damages in connection with the furnishing, use, or performance of this document or of any information contained herein. Should Agilent and the user have a separate written agreement with warranty terms covering the material in this document that conflict with these terms, the warranty terms in the separate agreement shall control.

Technology Licenses

The hardware and/or software described in this document are furnished under a license and may be used or copied only in accordance with the terms of such license.

Restricted Rights Legend

U.S. Government Restricted Rights. Software and technical data rights granted to the federal government include only those rights customarily provided to end user customers. Agilent provides this customary commercial license in Software and technical data pursuant to FAR 12.211 (Technical Data) and 12.212 (Computer Software) and, for the Department of Defense, DFARS 252.227-7015 (Technical Data - Commercial Items) and DFARS 227.7202-3 (Rights in Commercial Computer Software or Computer Software Documentation).

Safety Notices

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.

WARNING

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.

Contents

1 Introduction

Your Agilent Pesticide Analyzer allows you to screen for 926 pesticides and endocrine disruptors in a single GC/MS analysis. The system uses Agilent's Deconvolution Reporting Software (DRS) to integrate the results obtained from the Agilent GC/MS ChemStation, Automated Mass Spectral Deconvolution and Identification System (AMDIS) software from NIST, and the NIST Mass Spectral Search Program.

Deconvolution is used to extract "cleaned," library-searchable mass spectra from the chromatographic background, allowing pesticide identification even in the presence of overlapping matrix peaks. Retention Time Locking (RTL) is used to duplicate analyte retention times stored with mass spectra in the Agilent Pesticide and Endocrine Disruptor mass spectral database. By comparing your retention times to those in the library, DRS can eliminate false positives and differentiate among isomers of pesticides with similar structure.

Your Pesticide Analyzer was pre-configured by Agilent and checked out at the factory to ensure that your system will be ready to run samples immediately after installation in your lab.

Where to find more information on using your Pesticide Analyzer

Application notes and publications

You can find a lot of information about using DRS for pesticide analysis in the application notes and publications included with your Pesticide Analyzer CD.

List of target pesticides and endocrine disruptors

The following application note contains a list of all 926 compounds in the Agilent Pesticide and Endocrine Disruptor Library:

"Screening for 926 Pesticides and Endocrine Disruptors by GC/MS with Deconvolution Reporting Software and a New Pesticide Library," Agilent Application Note 5989-5076EN

Training videos

You received a DVD entitled "DRS Familiarization Videos" (P/N 5973-1731) which contains seven videos that give step-by-step instructions for several key steps in running your system. If you are unfamiliar with any of these steps, please take a few minutes and watch the appropriate video(s).

- Video 1 "Software Installation"
- Video 2 "Initial 5-run RT Locking"
- Video 3 "Re-locking RTLocked Method"
- Video 4 "DRS Processes and Report"
- Video 5 "QEdit with DRS A04"
- Video 6 "Adding Compounds to DRS"
- Video 7 "AMDIS Basic Operations"

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.

DRS help files

The DRS software on your GC/MS ChemStation comes with extensive help files. You may reach the DRS Help Directory by clicking on **DRS > Help** on the ChemStation Data Analysis drop-down menu.

What's on the Pesticide Analyzer CD ROM

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

- The GC/MSD method used for retention time locking your system and for running the checkout standard (**Solvent_Vent.M**). This method was retention time locked at the factory.
- A chromatogram and DRS report obtained at the factory for the GC/MSD Pesticide Analyzer Checkout Sample (P/N 5190-0468) with phenanthrene-d_{10} added as the internal standard
- Agilent Application Notes that discuss RTL
- Agilent Application Notes that discuss pesticide analysis using DRS
- An AMDIS settings file called **Onsite.ini** that has appropriate settings for your Pesticide Analyzer
- A copy of this Quick Start Guide

1 Introduction

2 Running your First Sample

You may wish to spike some of the Agilent GC/MSD Pesticide Analyzer Checkout Sample (P/N 5190-0468) into one of your laboratory extracts (at 100-200 ppb initially) or to run a previously analyzed sample that you know to have pesticide residues. Add phenanthrene-d_{10} as your internal standard. The method called **Solvent_Vent.M** assumes that the phenanthrene-d_{10} concentration is 1 ppm, but you can change the method later if you wish.

Deciding which method to use

Load the method called **Solvent_Vent.M**. Check to be sure that you have a 50-µL syringe installed in the autosampler and that your method is still configured for a 50-µL syringe. Your method should be configured to make a 10-µL injection in the solvent vent mode.

You may need to optimize the GC injection parameters for your solvent since different solvents will evaporate at different rates under the same set of inlet parameters. The method called **Solvent_Vent.M** is optimized for acetone and may work well for your extraction solvent too. If you want to adjust your method for a different solvent, use the procedure described in "Creating a new PTV Solvent Vent Method optimized for a different solvent (or injection volume)".

Alternatively, you could inject 3-5 µL of your sample in the splitless mode using an inlet temperature program ("Cold Splitless Mode"). For example, you could inject 5 µL with the inlet at 50 °C and immediately ramp the temperature up to 280 °C.

Creating a new PTV Solvent Vent Method optimized for a different solvent (or injection volume)

Agilent's MSD ChemStation comes with a tool (Solvent Elimination Calculator) that helps you optimize the multimode inlet (MMI) and injection parameters for large volume injections. If your sample is in a solvent other than acetone, or you want to change your injection volume, you can modify the **Solvent_Vent.M** method using the following procedure:

1 In the **GC Edit Parameters** window, click on the **Inlets** icon and the **MMI** tab to bring up the following screen (Figure 1).

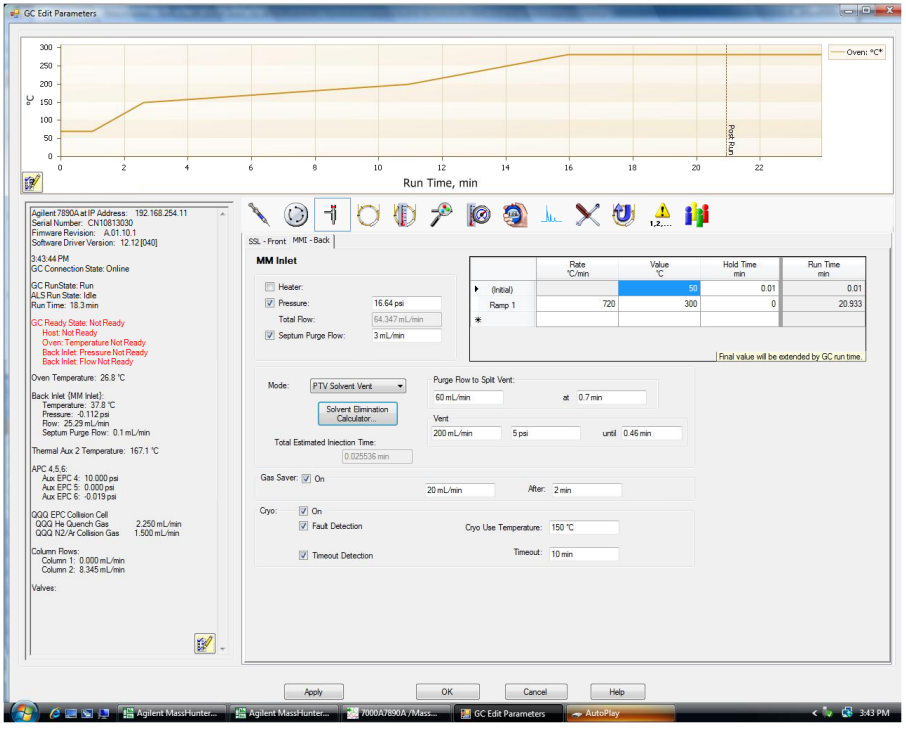


Figure 1 Multimode Inlet setup screen for the PTV Solvent Vent mode

2 Making sure that **PTV Solvent Vent** is chosen as the injection mode, click **Solvent Elimination Calculator**. This opens the first screen of this time-saving tool (Figure 2).

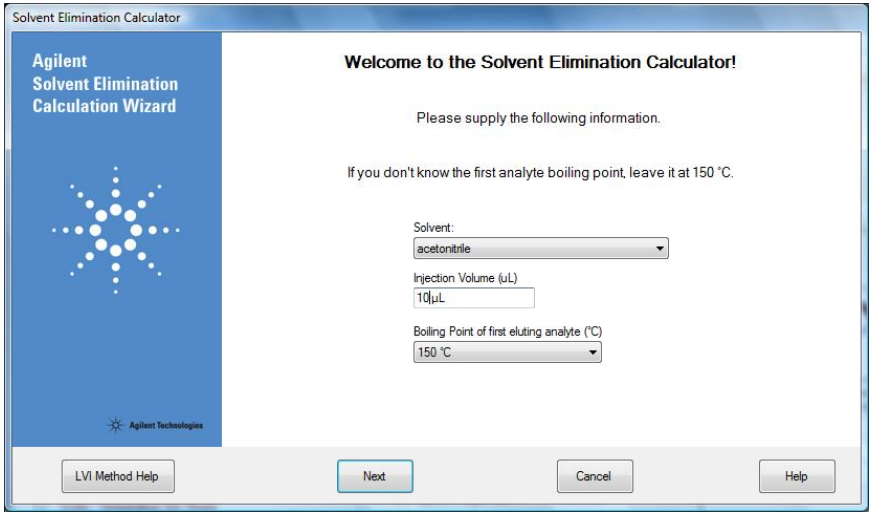


Figure 2 First screen of the Solvent Elimination Calculator

3 Choose your solvent, injection volume (10 µL), and the approximate boiling point of your first analyte (150 °C should be OK). When you click on **Next** several inlet parameters are suggested as shown in Figure 3.

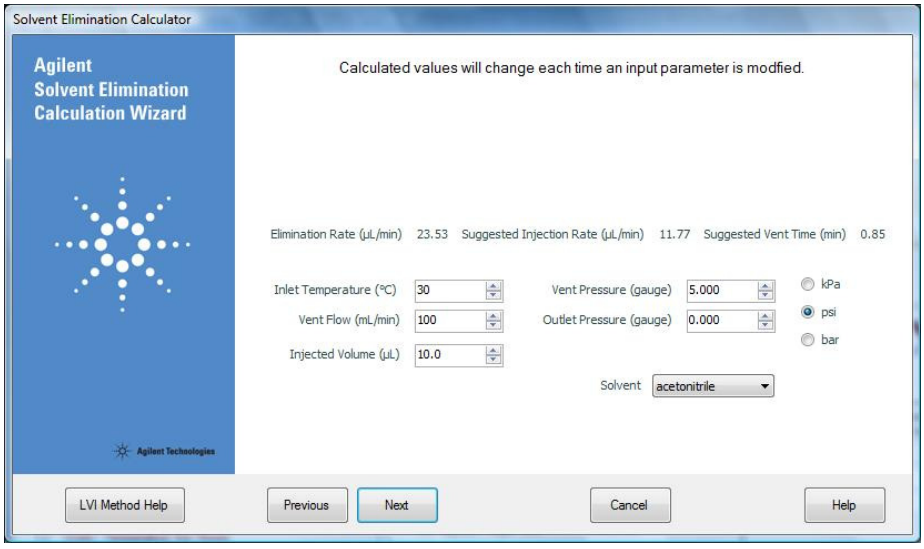


Figure 3 Suggested inlet parameters based on your choice of solvent, injection volume, and the boiling point of your first analyte

4 Note that the oven temperature program used for the pesticide method begins with a 1-minute hold at 70 °C. You should choose a set of conditions that result in a vent time of 1.0 min or less. If you want to change one or more of the suggested parameters enter your preferred value(s) and move your cursor to another field in the window. You will notice that the calculated values for the Elimination Rate, Suggested Injection Rate and Suggested Vent Time change accordingly. By comparing Figure 3 and Figure 4, you can see how these parameters changed when the inlet temperature was increased from 30 °C to 50 °C.

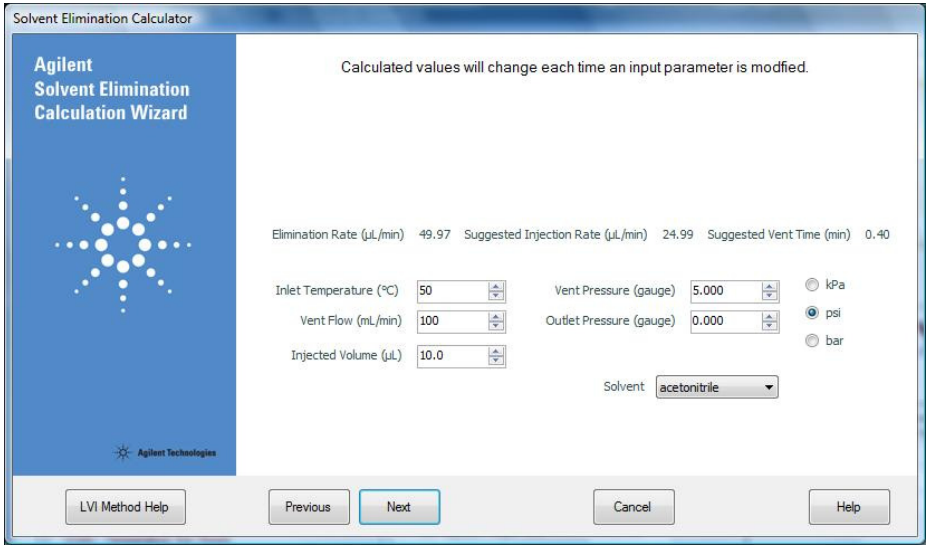


Figure 4 Suggested solvent elimination parameters for acetonitrile solvent when the inlet temperature is set to 50 °C

5 If you like these parameters, click **Next** to see all of the injection parameters (Figure 5).

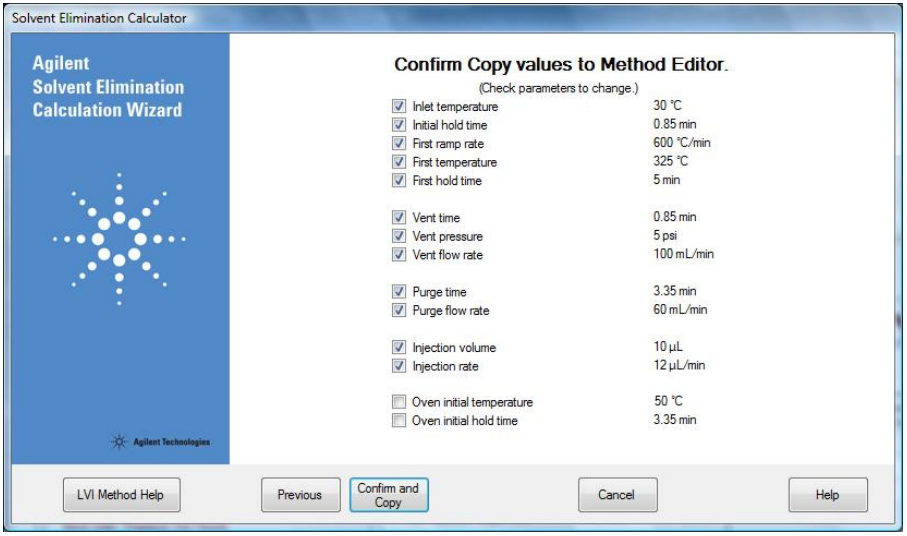


Figure 5 Inlet and injection parameters determined by the Solvent Elimination Calculator for a 10-µL injection of a sample in acetonitrile with the approximate boiling point of the first analyte set to 150 °C

6 Click **Confirm and Copy** to copy these values into your method. Save your method as a **Locked Method**, giving it a new file name (for example, **Solvent_Vent_Acetonitrile.M**). If you have any problems developing your method, click **LVI Method Help** for explanations and troubleshooting information.

Making a run

Create a one-line sequence or use **Run Method** to analyze your sample using **Solvent_Vent.M** or another method that you created for a different solvent.

3 Analyzing Sample Results with DRS

This chapter discusses DRS and how it is used to identify pesticides. After your sample run is complete, you can use DRS to analyze the results.

Running DRS on a sample

You can run DRS on a sample by using the following procedure:

1. At the end of the run, open Data Analysis, load the method called **Solvent_Vent.M,** and then load your chromatogram.
2. On the drop-down menu, choose **DRS > Quant + DRS single file**. After 2-4 minutes (depending on your AMDIS settings and the number of pesticide hits found), DRS will generate a report for your sample. The report shown in Figure 6 was obtained for a 5-µL injection of a carrot extract.

MSD Deconvolution Report
Sample Name: carrot1 from JW Data
File: C:\msdchem\1\data\carrot1_Boyle_60C_SL_scan_5µL.D
Date/Time: 9:57:43 AM Thursday, June 11, 2009

Adjacent Peak Subtraction = 2
Resolution = High
Sensitivity = High
Shape Requirements = Medium

The NIST library was searched for the components that were found in the AMDIS target library.

			Amount (ng/uL)		AMDIS		NIST	
R.T.	Cas #	Compound Name	Chem station	AMDIS	Match	R.T. Diff sec.	Reverse Match	Hit Num.
2.9796	89838	Thymol			93	-10.0	87	1
3.2909	3228033	Promecarb artifact [5-isopropyl-3-methylphenol]			61	14.2		
3.2909	294876601	Carbamic acid, N-[1,1-bis(trifluoromethyl)propyl]-, (2-isopropyl-5-methylphenyl) ester					62	1
4.4071	33704619	Cashmeran			60	11.6	74	13
6.9401	1517222	Phenanthrene-d10	35.96		93	9.2	79	2
8.0245	84695	Diisobutyl phthalate	16.19		85	9.2	83	8
9.270	84742	Di-n-butylphthalate	61.5		95	7.5	92	1
12.1357	72559	p,p'-DDE	6.15		86	15.0	82	1
12.9476	72548	p,p'-DDD	2.6		81	12.5	80	1
12.954	789026	o,p'-DDT	2.6					
13.5928	50293	p,p'-DDT	2.85		72	12.2	67	8
13.9902	115866	Triphenyl phosphate	15.11		96	15.3	85	1
14.585	120928098	Fenazaquin	73.95					
14.990	2385855	Mirex	1.38					
6.943		Phenanthrene-d10	10					

Figure 6 Initial DRS report obtained before review in QEdit

Reading the DRS report

The report in Figure 6 shows retention times, CAS Numbers, and the name of each target compound found by DRS and/or the ChemStation. There are two columns under **Amount** labeled **ChemStation** and **AMDIS**. The AMDIS column will be empty until you import your AMDIS results into the ChemStation in **QEdit** (see "Verifying your results in QEdit"). The ChemStation amount is an approximation based on a set of average response factors. In order to obtain true quantitative results for your samples, you must calibrate compounds in the usual way. DRS has the added capability of quantifying compounds using their deconvoluted target ion. Refer to Video 5 or the DRS Help file for more discussion.

Under the **AMDIS** heading the report shows a match factor (99 = best match) in one column and the difference in retention times between those listed in the Agilent Pesticide Database and those from your chromatogram in another column.

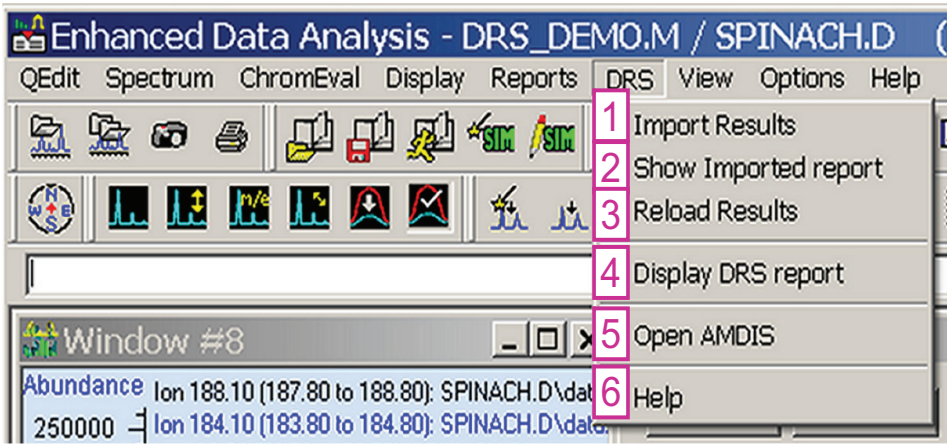
DRS sends the deconvoluted spectrum of each AMDIS hit to the NIST Library Search program where it searches the entire NIST mass spectral library. If the hit is also found in the NIST library, its match factor is reported along with the number of the hit within the list of possibilities. These values are listed in the columns under the **NIST** heading.

You can get a more thorough description of this report by watching Video 4 "DRS – Generating Your First DRS Report" and reading the DRS help file on this topic.

Verifying your results in QEdit

QEdit is a ChemStation tool for evaluating your quant results. Agilent's DRS software offers some features that are not available in normal ChemStation QEdit. Figure 7 describes the DRS Menu items in QEdit.

For more information about using QEdit to review your results, watch Video 5 "QEdit with DRS A04" and read about this topic in the DRS and ChemStation Help files.



1 - Import spectra and extracted ion from AMDIS

2 - Displays Text Report with MSD and AMDIS Results

3 - Reloads existing results from AMDIS results file

4 - Displays DRS report with updated amounts

5 - Opens AMDIS standalone

6 - Link to DRS Help

Figure 7 The DRS menu in QEdit

In ChemStation Data Analysis, choose **View > QEdit Quant Result** from the drop-down menu.

In the **Quick QEdit** window hits are marked with an x, an A, or xA, depending on whether they were found by the ChemStation or AMDIS or both. Review all of the hits, checking the integration and validity of the hit. Using QDelete, remove any hits that can't be verified.

Generating an updated DRS report

At any time during your QEdit review process, you can click **DRS > Display DRS Report** to generate a new report, incorporating your AMDIS Quant Results and any changes that you made in QEdit. Figure 8 shows the new report that now has the AMDIS Quant results. Two pesticides, o,p'-DDT and Mirex, that were originally found only by the ChemStation (Figure 6) were removed in QEdit and no longer appear in the report. Again, note that the quant values under both ChemStation and AMDIS headings are only approximations using an average response factor supplied with your Pesticide Analysis System. To get reportable values, you must calibrate your target compounds.

Close QEdit and save the changes you made to your quant results.

MSD Deconvolution Report
Sample Name: carrot1 from JW
Data File: C:\MSData\DRS 2X Test Files\GC_Q Data Pesticides Dec_08 Jan_09 S_SI and Boyle\Jan 02_09 Boyle cold 5µL SL_incurred\carrot1_Boyle_60C_SL_scan_5µL.D
Date/Time: 1:49:51 PM Thursday, June 11, 2009

Adjacent Peak Subtraction = 2
Resolution = High
Sensitivity = High
Shape Requirements = Medium

The NIST library was searched for the components that were found in the AMDIS target library.

			Amount (ng)		AMDIS		NIST	
R.T.	Cas #	Compound Name	Chem station	AMDIS	Match	R.T. Diff sec.	Reverse Match	Hit Num.
2.9796	89838	Thymol	171.56	158.08	93	-10.0	87	1
3.2909	3228033	Promecarb artifact [5-isopropyl-3-methylphenol]			61	14.2		
3.2909	294876601	Carbamic acid, N-[1,1-bis(trifluoromethyl)propyl]-, (2-isopropyl-5-methylphenyl) ester					62	1
4.4071	33704619	Cashmeran			60	11.6	74	13
6.9401	1517222	Phenanthrene-d10	35.96	22.7	93	9.2	79	2
8.0245	84695	Diisobutyl phthalate	16.19	12.14	85	9.2	83	8
9.270	84742	Di-n-butylphthalate	61.5	22.93	95	7.5	92	1
12.1357	72559	p,p'-DDE	6.15	3.49	86	15.0	82	1
12.9476	72548	p,p'-DDD	2.6	1.81	81	12.5	80	1
13.5928	50293	p,p'-DDT	2.85	1.36	72	12.2	67	8
13.9902	115866	Triphenyl phosphate	15.11	8.67	96	15.3	85	1
14.585	120928098	Fenazaquin	73.95					
6.943		Phenanthrene-d10	10					

Figure 8 Updated DRS report

Advanced DRS interpretation

The following discussion takes a closer look at the DRS reports in Figure 6 and Figure 8 and offers some suggestions on interpreting DRS results.

- In both figures, notice that most of the retention times are 9 to 15 sec away from their database values. This suggests that it is time to relock the method (refer to Video 3: “Retention Time Locking – Relocking an already Locked Method” and the ChemStation help files).
- Note that the retention time difference for Thymol is -10 sec while all of the other values are positive. While this may be correct, you should flag this hit for further review.
- In QEdit, there was sufficient evidence for the Fenazaquin identification to keep it in the Quant report even though it was not found by AMDIS. You might want to adjust some of the AMDIS parameters and rerun DRS to see if you can find it. If you are familiar with AMDIS, you can open this program, zoom in on the peak, which saves a lot of time, and try several different AMDIS settings.
- The AMDIS hit at 3.2909 min (Promecarb artifact [5-isopropyl-3-methylphenol]) was found by AMDIS but there are no NIST library search results on that line. This means that the compound was not in the NIST library (most likely) or was not among the top 100 hits. When this happens, DRS reports the best NIST library match on the next line, which in this case is Carbamic acid, N-[1,1-bis(trifluoromethyl)propyl].
- If a pesticide elutes under an unusually large matrix peak, it’s retention time may shift away from the database value, even when the method has been locked. If you suspect that there has been a shift of more than +/- 20 sec, you can widen the AMDIS window and rerun DRS.
- The database retention times were determined on instruments that did not incorporate backflushing. For this reason, very early and very late eluting compounds on your analyzer will show a somewhat larger deviation from the database values. The differences are small and the benefit of backflushing the column outweighs these small losses in retention time accuracy.
- Certain pesticide isomers (e.g., the four isomers of Cypermethrin) have nearly identical spectra and very similar retention times. DRS may identify them out of order, particularly if the AMDIS RI window is more than a few seconds wide (20 sec is the usual setting), or it may not identify all the isomers. Identifying the specific isomer is rarely necessary, but you can use a narrower retention time window if needed.

- For more information on the way DRS handles multiple pesticide isomers, see Application Note 5989-5076EN “Screening for 926 Pesticides and Endocrine Disruptors by GC/MS with Deconvolution Reporting Software and a New Pesticide Library.”

4 Consumables and Parts

Table 1 Consumables and parts

Description	Part number
Consumables	
Column, HP-5ms UI, 15 m × 0.25 mm × 0.25 µm	190910S-431UI
Column ferrule, MMI, graphite (10/pkg)	5080-8853
Column nut, GC capillary (2/pkg)	5181-8830
Inlet liner, helix double taper, deactivated	5188-5398
O-ring, inlet liner non-stick (10/pkg)	5188-5365
Septum, advanced green (50/pkg)	5183-4759
ALS syringe, 50 µL straight fixed needle, PTFE-tipped plunger	5183-0318
Restrictor, DB-5ms UI, 5 m × 0.15 mm × 0.15 µm	165-6626
Internal nut for capillary flow devices	G2855-20530
SilTite ferrule for 0.25 mm and smaller columns (10/pkg)	5288-5361
Column nut for MSD interface	05988-20066
MSD interface ferrule (10/pkg)	5062-3508
Big universal trap for helium purification	RMSH-2
External split vent trap	RDT-1020
Useful tools	
MSD column installation tool	G1099-20030
Ferrule pre-swage tool for capillary flow devices	G2855-60200

Table 1 Consumables and parts (continued)

Description	Part number
Chemical standards	
Pesticide retention time locking standard (3 × 1 mL ampoules containing 10 µg/mL each of dichlorvos, chloropyrifos-methyl and mirex in n-hexane)	5190-1441
Pesticide analyzer checkout sample (4 × 1 mL ampoules containing 20 pesticides at 10 µg/mL each in acetone	5190-0468
Internal standard solution (4 × 1 mL ampoules containing phenanthrene-d10 at 1000 µg/mL in methylene chloride)	5190-0472
QuEChERS extraction supplies (follow this web link: http://www.chem.agilent.com/en-US/products/consumables/samplepreparation/sampliqspe/sampliqquechers/Pages/default.aspx or go to: http://www.chem.agilent.com/ and type QuEChERS into the search field)	

Printed in USA 05/09

First Edition, May 2009

5973-1732