



Introducing the P&EP 3.0

GC/MS/MS Pesticides and Environmental Pollutants Analyzer

Confirming Pesticide
Residues with Confidence

May 2014

[illegible]

A cartoon illustration of a man wearing a yellow hard hat and a green shirt, looking up at a chemical structure. The structure shows a carbon atom bonded to a chlorine atom (Cl) and a nitrogen atom (N).

Pesticide Multi-Residue Analysis

Motivation

Food safety and regulations to protect public

Need for analysis of a large number of analytes

Unknown “history” of the analyzed sample (field treatment, post-harvest application, contamination)

Economic aspects (costs, time, and labor)



Pesticide Residue Analysis

Drivers – Governance of Pesticide Use

Maximum Residue Limits – Amount On or In Food

Tolerance – Level that Triggers Enforcement Action

Safety – Toxicity of pesticide and breakdown product

Use – Reasonable Certainty of “No Harm”



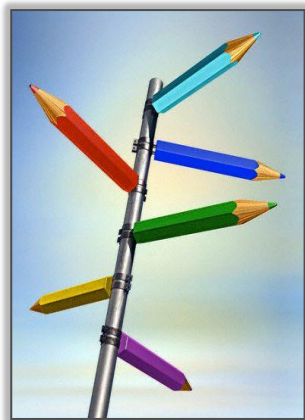


Pesticides Residue Analysis



The
Analytical
Iceberg

Pesticide Multi-Residue Analysis



Classical approach:

Multiple methods and techniques to cover different analytes/classes of interest

e.g.: GC-FPD, NPD, ECD, ELCD
LC-UV or LC-FLD

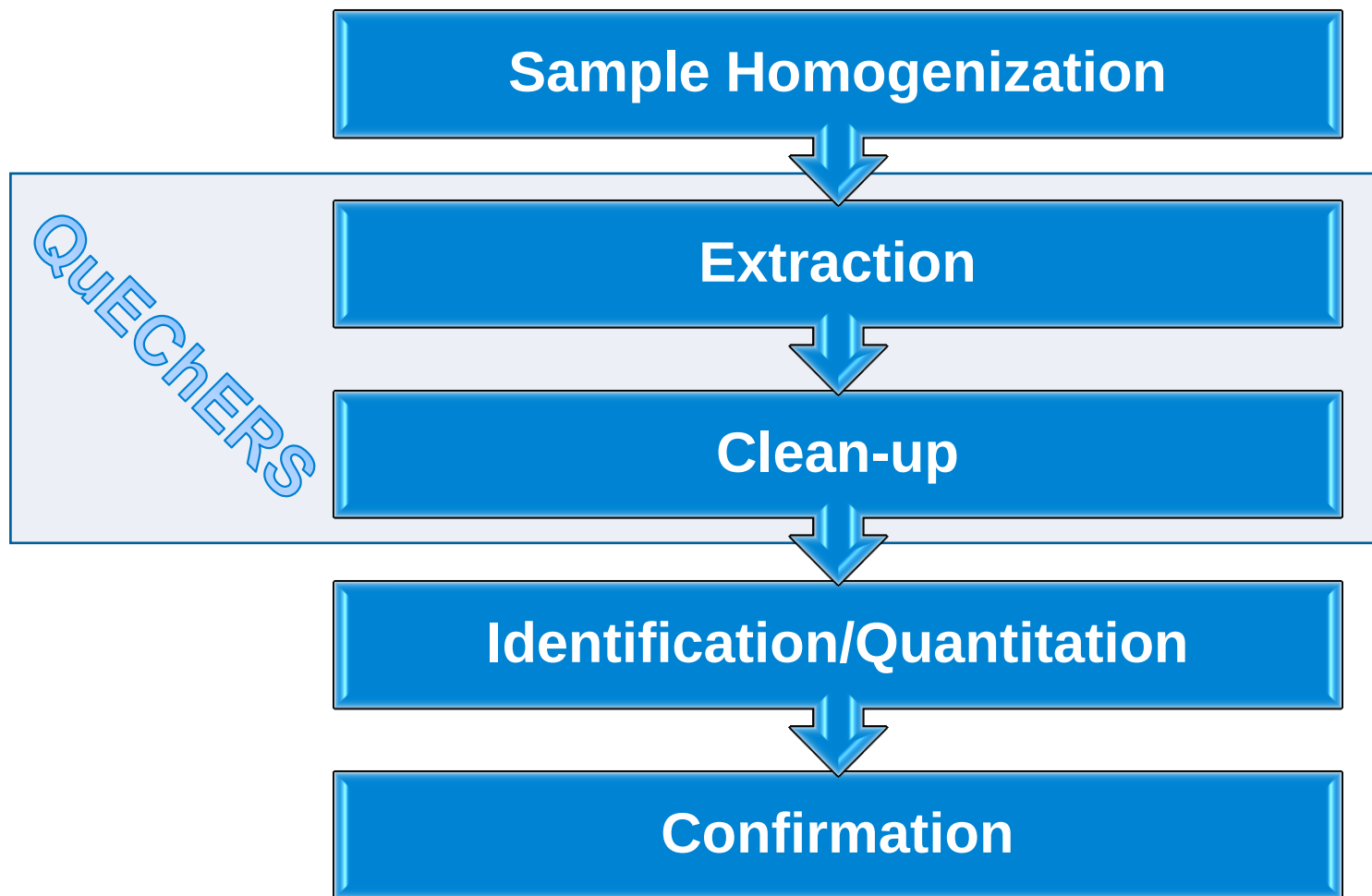
Modern approach:

Multi-analyte methods with simultaneous quantification and identification using

GC-MS and **LC-MS**, especially with **MS/MS** capabilities for increased selectivity



Pesticide Multi-Residue Analysis



QuEChERS = Quick, Easy, Cheap, Effective, Rugged, and Safe



The Bond Elut QuEChERS Selection Guide

Home BACK FORWARD GET YOUR SAMPLE PREP CATALOG GET HELP

**Agilent
Bond Elut QuEChERS
Selection Tool**



Let us help you find the best Agilent QuEChERS products for your application.

BEGIN

[Agilent Food Safety Applications Notebook: Volume 2 – Bond Elut QuEChERS](#)

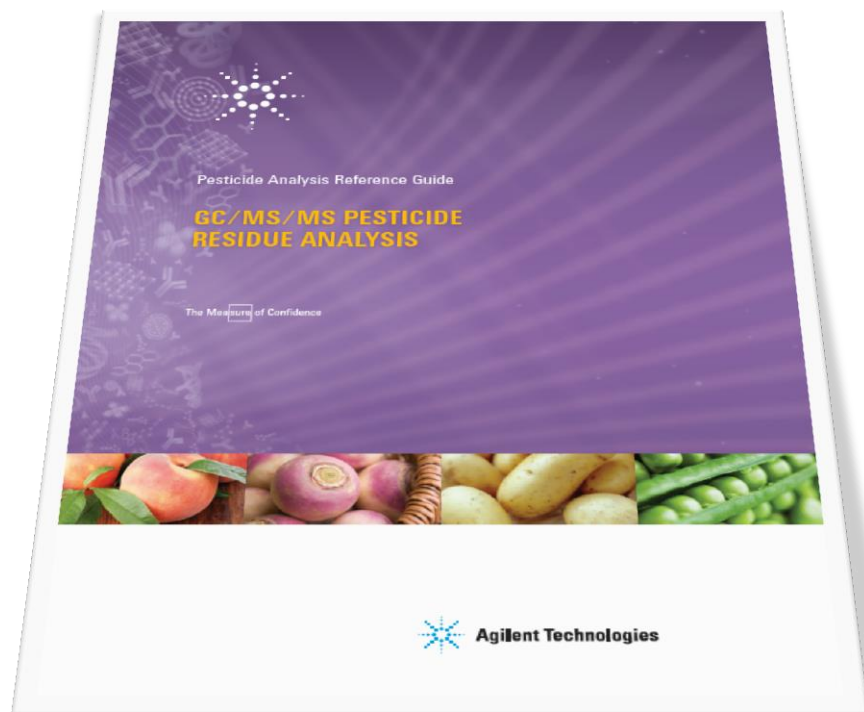
[What is QuEChERS?](#)

[QuEChERS video](#)

www.agilent.com/chem/selectQuEChERS

The Agilent Pesticide Analysis Reference Guide

Available from Your Local Agilent Representative



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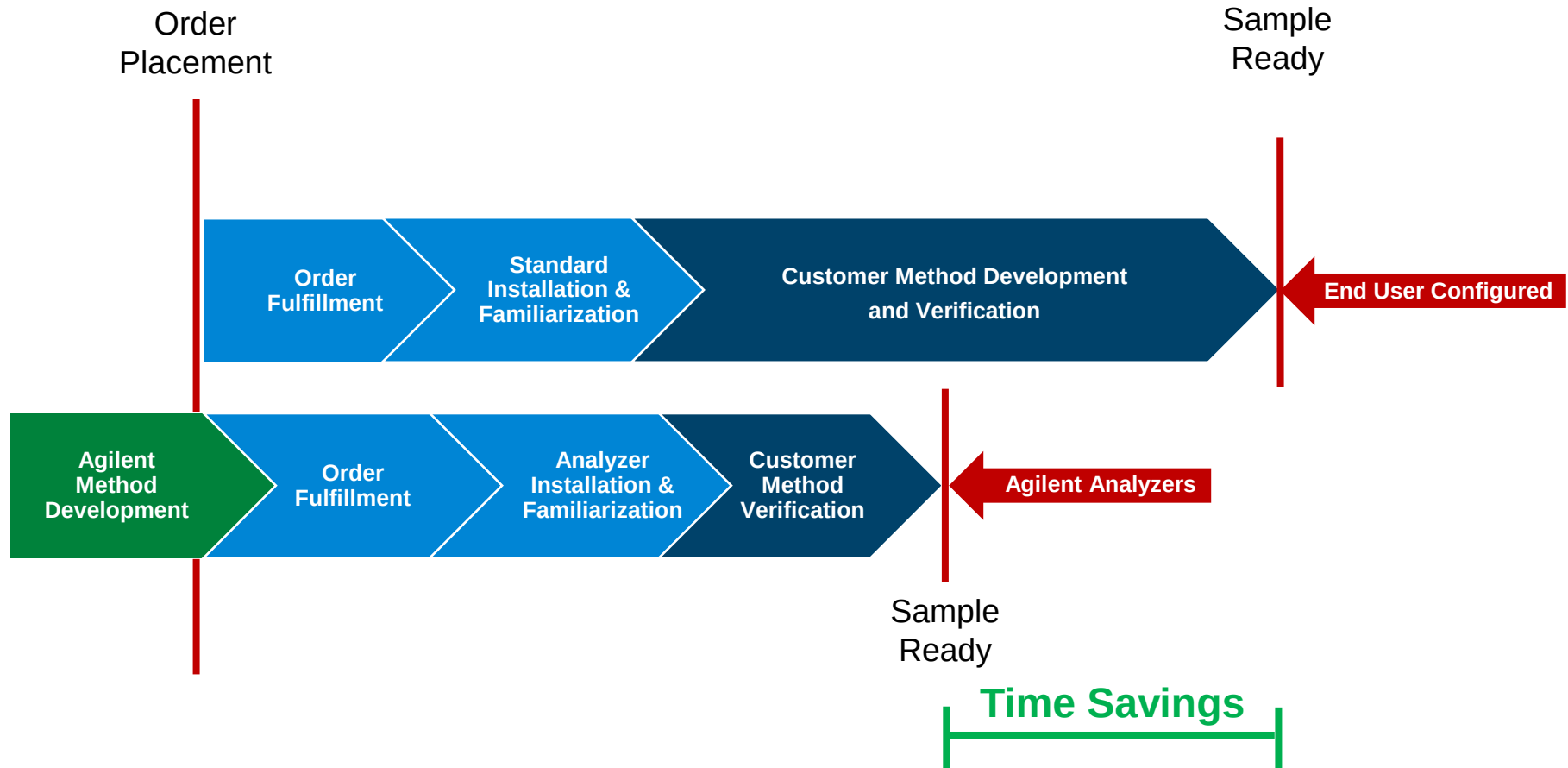
P&EP 3.0 GC/MS/MS Pesticides Analyzer



Get Customers on the
Analytical ***FAST TRACK***

Customer Concerns

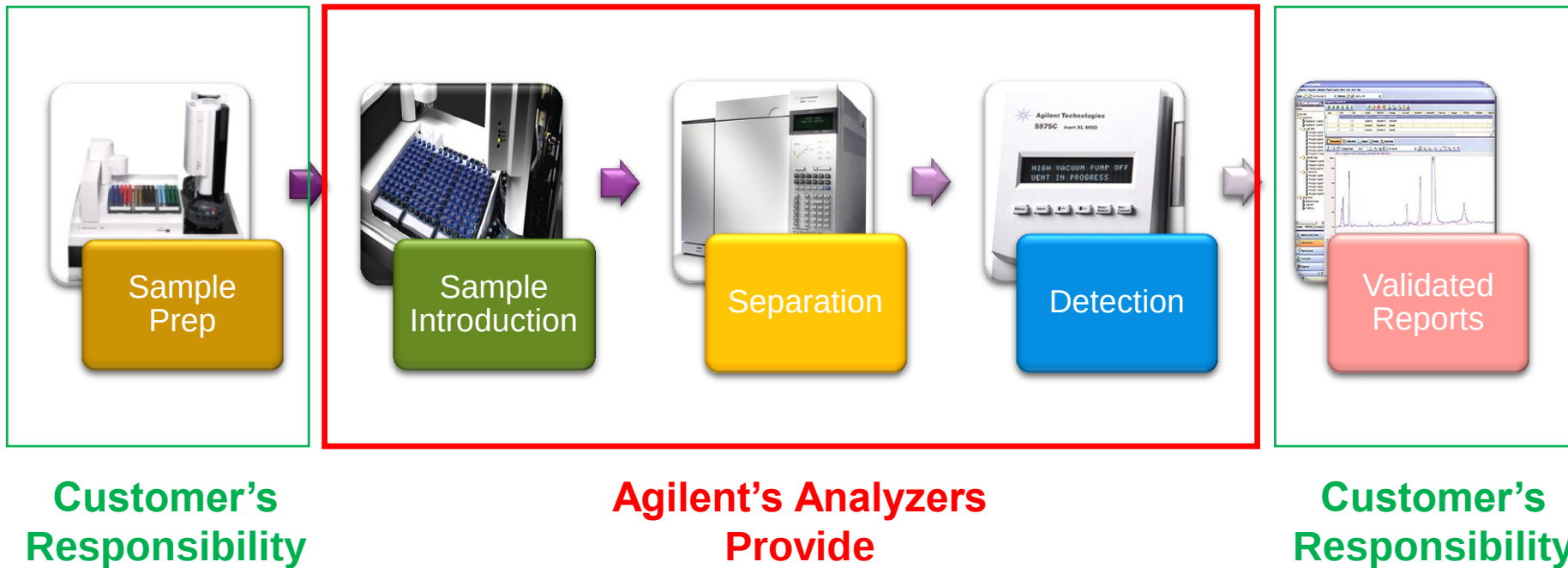
Confident Identification of Pesticide Residues



...Faster Application Startup with a Guaranteed Method

The Pesticide Analyst Needs

Agilent Analyzer Solutions



Feature Elements for P&EP 3.0 Analyzer

GC/MS/MS Pesticides and Environmental Pollutants Analyzer

- **MRM database** with **~1100** compounds and **~8500** optimized transitions including 710+ pesticides and breakdown products
- **GUI** that facilitates building MRM acquisition and quantitation methods based on *customer's compound list*
- **Retention Time Locked** separation for reliable peak identification and quantitation for CF and CP BF methods
- **Multimode inlet** for large volume injection helps optimize detection limit performance
- **Opt 114 UI S/SL** for when there is no requirement for optimal LOD performance
- **Capillary Flow Technology Backflush** for faster cycle time and reduced system maintenance



Pesticide & Environmental Pollutant Analyzer 3.0



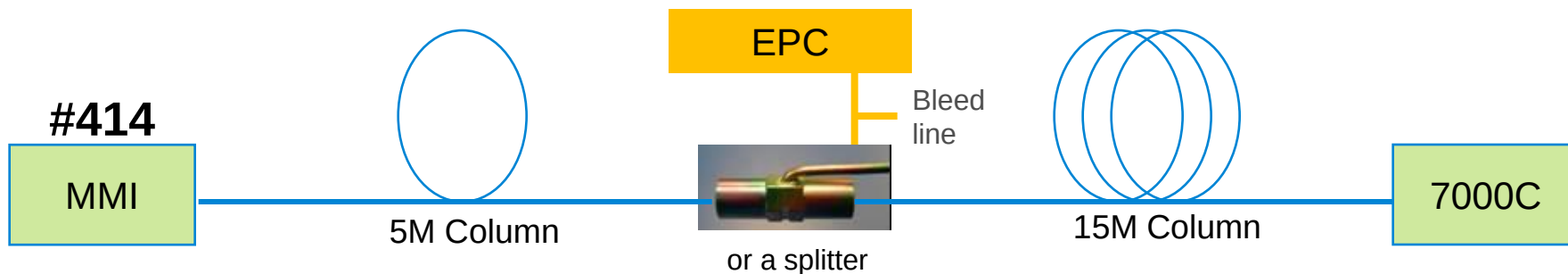
P&EP 3.0 New Method Configurations

– Constant Pressure and Constant Flow Modes

— post-column backflush components



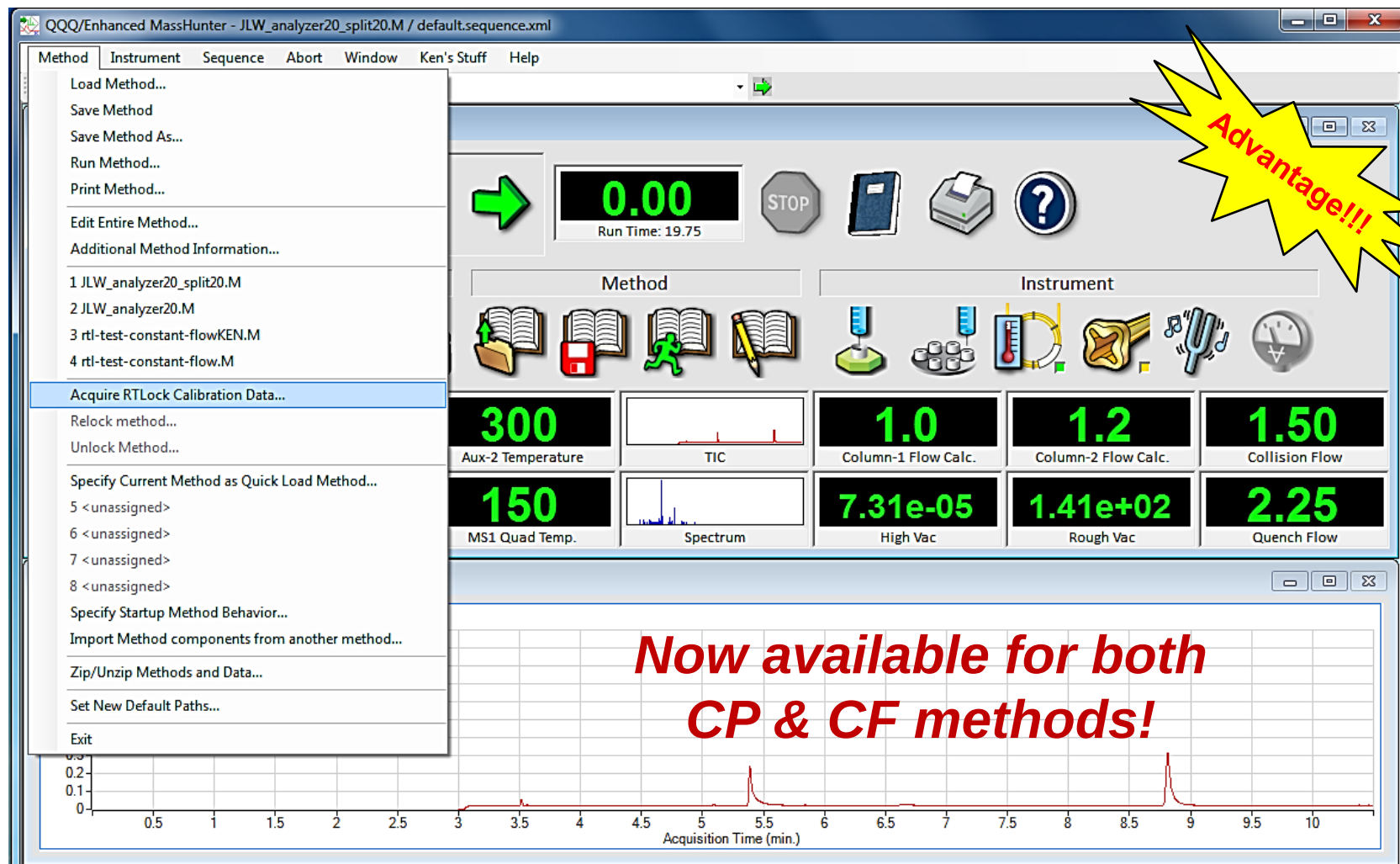
Flexibility to add GC detectors and easily scaled for shorter runtime



Provides ultimate performance and shortest cycle time



Acquire Retention Time Locking (RTL) Calibration Data

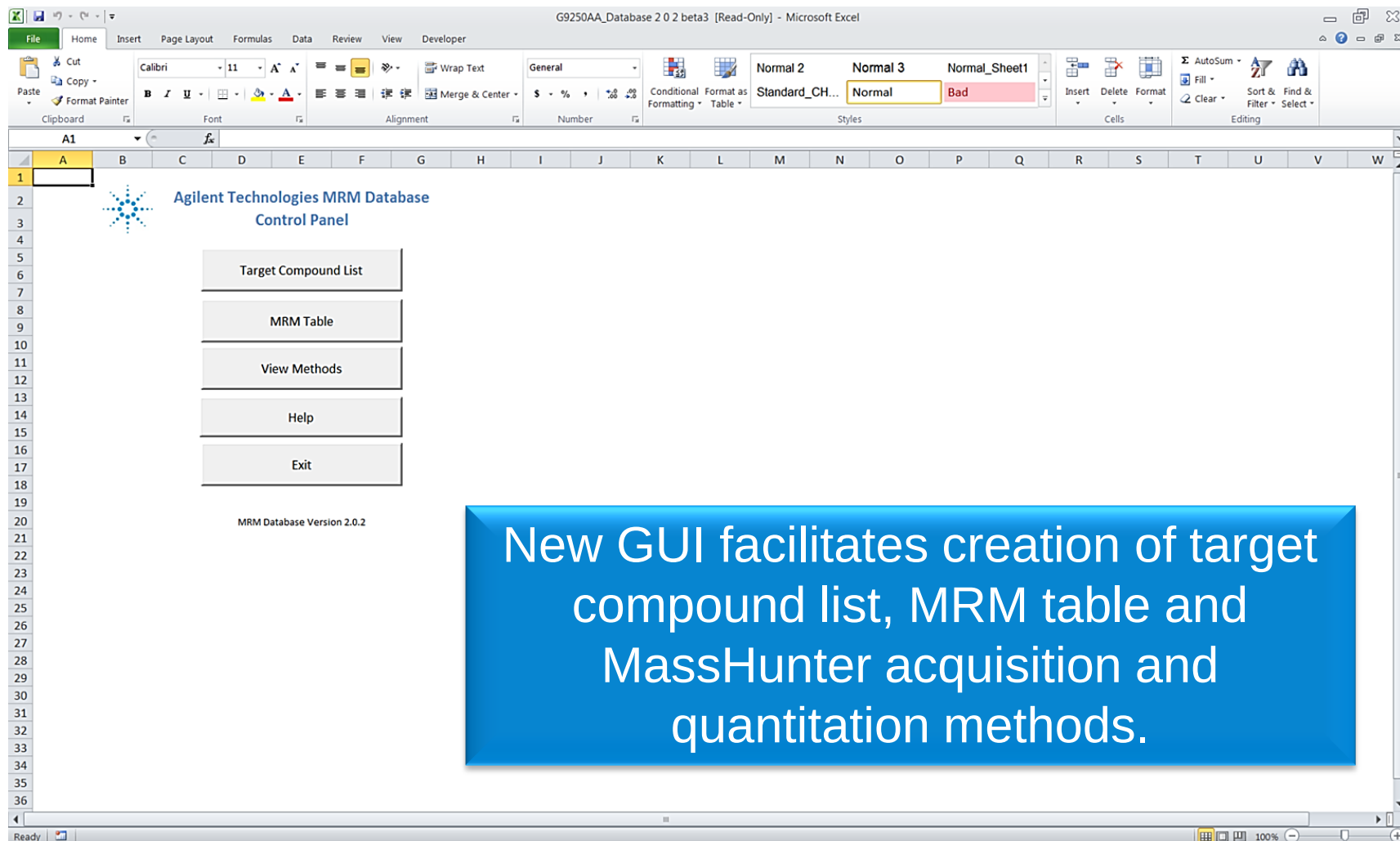


Note: In this example, the acquisition is in full-scan mode.



Home Screen of the MRM Database

Sure it's an Excel file, but...



View of "Target Compound List"

The image shows two overlapping screenshots from the Agilent Technologies MRM Database software. The background screenshot displays the 'Control Panel' with buttons for 'Target Compound List' (circled in red), 'MRM Table', 'View Methods', 'Help', and 'Exit'. A red arrow points from the 'Target Compound List' button to the foreground screenshot.

The foreground screenshot shows the 'My Target Compound List' view, which includes a table of compounds and a sidebar with action buttons.

	A	B	C	D	E
1		Compound Name	CAS #	Target	My Target Compound List
2	1	Dichlorvos	62-73-7	Target	
3	2	Mevinphos	7786-34-7	Target	
4	3	Ethalfuralin	55283-68-6	Target	
5	4	Trifluralin	1582-09-8	Target	
6	5	Atrazine	1912-24-9	Target	
7	6	BHC-gamma (Lindane, gamma HCH)	58-89-9	Target	
8	7	Chlorpyrifos-methyl	5598-13-0	Target	
9	8	Heptachlor	76-44-8	Target	
10	9	Malathion	121-75-5	Target	
11	10	Chlorpyrifos	2921-88-2	Target	
12	11	Dieldrin	60-57-1	Target	
13	12	DDE-p,p'	72-55-9	Target	
14	13	Hexazinone	51235-04-2	Target	
15	14	Propargite	2312-35-8	Target	
16	15	Leptophos	21609-90-5	Target	
17	16	Mirex	2385-85-5	Target	
18	17	Fenarimol	60168-88-9	Target	
19	18	Coumaphos	56-72-4	Target	
20	19	Etofenprox (Ethofenprox)	80844-07-1	Target	
21	20	Deltamethrin	52918-63-5	Target	

On the right side of the foreground screenshot, there is a sidebar with the following buttons: 'Create New Target List', 'Save Current Target List', 'Manage Target Lists', 'Add Compounds', 'Import CAS Numbers', 'Build MRM Table', and 'Home'.



Develop MassHunter Methods

Easy interface to MassHunter Method Development Tools

MRM Acquisition Method via MH Compound List Assistant (CLA)

The screenshot shows the MassHunter software interface with the MRM Acquisition Method development tool. A red arrow points to the 'Export For CLA Optimizer' button. A dialog box is open, asking to export the current MRM Table to a CSV file for import into the CLA Optimizer.

CAS	Compound name	Retention Time	ISTD?	Precurser ion	MS1 resolution	Product ion	MS2 resolution	Dwell	Collision energy	Left RT Delta	Right RT Delta	RT Window	Rel. Intensity
62-73-7	Dichlorvos	4.002491583	false	109 LowRes	79 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.457692308
62-73-7	Dichlorvos	4.002491583	false	184.9 LowRes	93 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
7786-34-7	Mevinphos	4.844	false	127 LowRes	109 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
7786-34-7	Mevinphos	4.844	false	95 LowRes	127 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.385714286
55283-68-6	Ethalfuralin	6.317643893	false	275.9 LowRes	202.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.530434783
55283-68-6	Ethalfuralin	6.317643893	false	315.9 LowRes	275.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1582-09-8	Trifluralin	6.43037533	false	305.9 LowRes	264 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1582-09-8	Trifluralin	6.43037533	false	160.1 LowRes	160.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.594594595
1912-24-9	Atrazine	7.168	false	214.9 LowRes	58.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1912-24-9	Atrazine	7.168	false	214.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.657534247
58-89-9	BHC-gamma (Lindane, gamma)	7.410931839	false	216.9 LowRes	181 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
58-89-9	BHC-gamma (Lindane, gamma)	7.410931839	false	181 LowRes	181 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.952380952
5598-13-0	Chlorpyrifos-methyl	8.528	false	124.9 LowRes	8.528	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
5598-13-0	Chlorpyrifos-methyl	8.528	false	124.9 LowRes	124.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.71
76-44-8	Heptachlor	8.769848226	false	271.7 LowRes	271.7 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
76-44-8	Heptachlor	8.769848226	false	273.7 LowRes	273.7 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.63
121-75-5	Malathion	9.227719109	false	126.9 LowRes	126.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
121-75-5	Malathion	9.227719109	false	172.9 LowRes	99 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.4
2021-88-2	Chlorpyrifos	9.493231875	false	196.9 LowRes	169 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2021-88-2	Chlorpyrifos	9.493231875	false	198.9 LowRes	171 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.95
72-55-9	DDE-p,p'	11.431	false	246.1 LowRes	176.2 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
72-55-9	DDE-p,p'	11.431	false	315.8 LowRes	246 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.62
60-57-1	Dieldrin	11.58695951	false	277 LowRes	241 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
60-57-1	Dieldrin	11.58695951	false	262.9 LowRes	193 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
51235-04-2	Hexazinone	13.355	false	171 LowRes	71.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
51235-04-2	Hexazinone	13.355	false	171 LowRes	85.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.46
2312-35-8	Propargite	13.579	false	135 LowRes	107.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2312-35-8	Propargite	13.579	false	149.9 LowRes	135.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.27
21609-90-5	Leptophos	14.55095856	false	171 LowRes	171 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
21609-90-5	Leptophos	14.55095856	false	154.9 LowRes	77.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.45
2385-85-5	Mirex	14.631	false	271.8 LowRes	236.8 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2385-85-5	Mirex	14.631	false	273.8 LowRes	238.8 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.63
60168-88-9	Fenarimol	14.8448856	false	219 LowRes	107.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1

MH Quantitation Method via the MRM database

The screenshot shows the MassHunter software interface with the MRM Quantitation Method development tool. A red arrow points to the 'Create Quant Method' button. A dialog box is open, asking to create a QQ Quant Method from the current MRM Master Table.

CAS	Compound name	Retention Time	ISTD?	Precurser ion	MS1 resolution	Product ion	MS2 resolution	Dwell	Collision energy	Left RT Delta	Right RT Delta	RT Window	Rel. Intensity
62-73-7	Dichlorvos	4.002491583	false	109 LowRes	79 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.457692308
62-73-7	Dichlorvos	4.002491583	false	184.9 LowRes	93 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
7786-34-7	Mevinphos	4.844	false	127 LowRes	109 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
7786-34-7	Mevinphos	4.844	false	95 LowRes	127 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.385714286
55283-68-6	Ethalfuralin	6.317643893	false	275.9 LowRes	202.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.530434783
55283-68-6	Ethalfuralin	6.317643893	false	315.9 LowRes	275.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1582-09-8	Trifluralin	6.43037533	false	305.9 LowRes	264 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1582-09-8	Trifluralin	6.43037533	false	160.1 LowRes	160.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.594594595
1912-24-9	Atrazine	7.168	false	214.9 LowRes	58.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
1912-24-9	Atrazine	7.168	false	214.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.657534247
58-89-9	BHC-gamma (Lindane, gamma)	7.410931839	false	216.9 LowRes	181 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
58-89-9	BHC-gamma (Lindane, gamma)	7.410931839	false	181 LowRes	181 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.952380952
5598-13-0	Chlorpyrifos-methyl	8.528	false	124.9 LowRes	8.528	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
5598-13-0	Chlorpyrifos-methyl	8.528	false	124.9 LowRes	124.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.714285714
76-44-8	Heptachlor	8.769848226	false	271.7 LowRes	271.7 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
76-44-8	Heptachlor	8.769848226	false	273.7 LowRes	273.7 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.637254902
121-75-5	Malathion	9.227719109	false	126.9 LowRes	126.9 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
121-75-5	Malathion	9.227719109	false	172.9 LowRes	99 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.43697479
2021-88-2	Chlorpyrifos	9.493231875	false	196.9 LowRes	169 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2021-88-2	Chlorpyrifos	9.493231875	false	198.9 LowRes	171 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.956175299
72-55-9	DDE-p,p'	11.431	false	246.1 LowRes	176.2 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
72-55-9	DDE-p,p'	11.431	false	315.8 LowRes	246 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.626794258
60-57-1	Dieldrin	11.58695951	false	277 LowRes	241 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
60-57-1	Dieldrin	11.58695951	false	262.9 LowRes	193 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
51235-04-2	Hexazinone	13.355	false	171 LowRes	71.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
51235-04-2	Hexazinone	13.355	false	171 LowRes	85.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.460606061
2312-35-8	Propargite	13.579	false	135 LowRes	107.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2312-35-8	Propargite	13.579	false	149.9 LowRes	135.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.273809524
21609-90-5	Leptophos	14.55095856	false	171 LowRes	171 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
21609-90-5	Leptophos	14.55095856	false	154.9 LowRes	77.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.455645161
2385-85-5	Mirex	14.631	false	271.8 LowRes	236.8 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1
2385-85-5	Mirex	14.631	false	273.8 LowRes	238.8 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.632911392
60168-88-9	Fenarimol	14.8448856	false	219 LowRes	107.1 LowRes	10	0.1	0.1	0.1	0.1	0.1	0.1	0.1





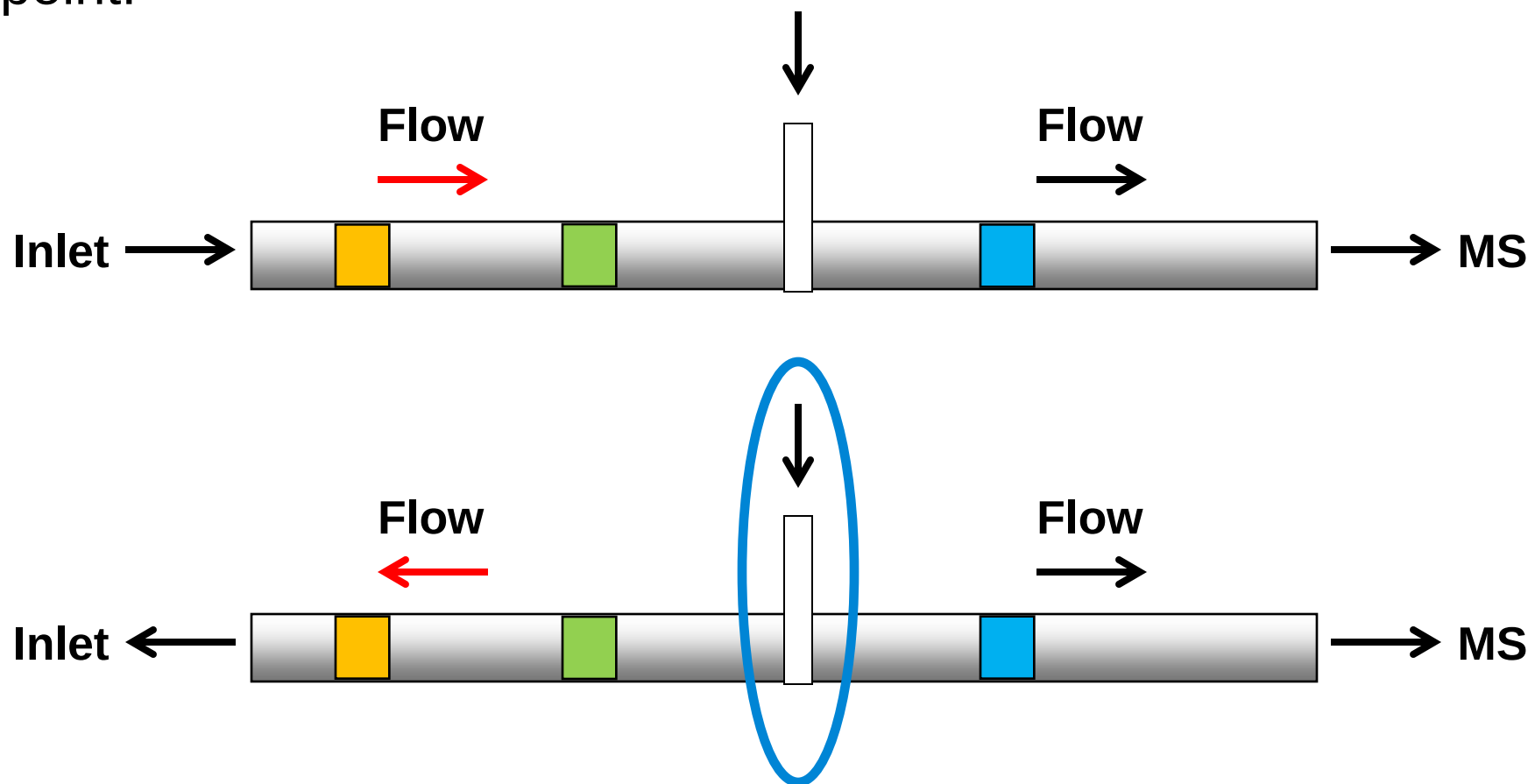
P&EP 3.0

GC/MS/MS Pesticides Analyzer

Value Overview and
Summary

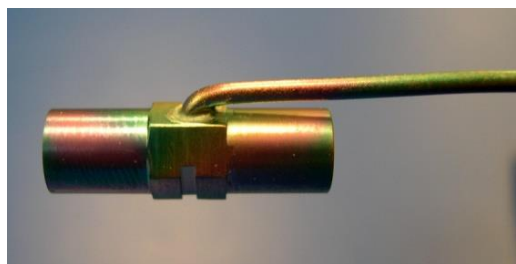
Column Backflush

Eliminates less volatile matrix components from the GC column by reversing the column flow at a pressure junction point:

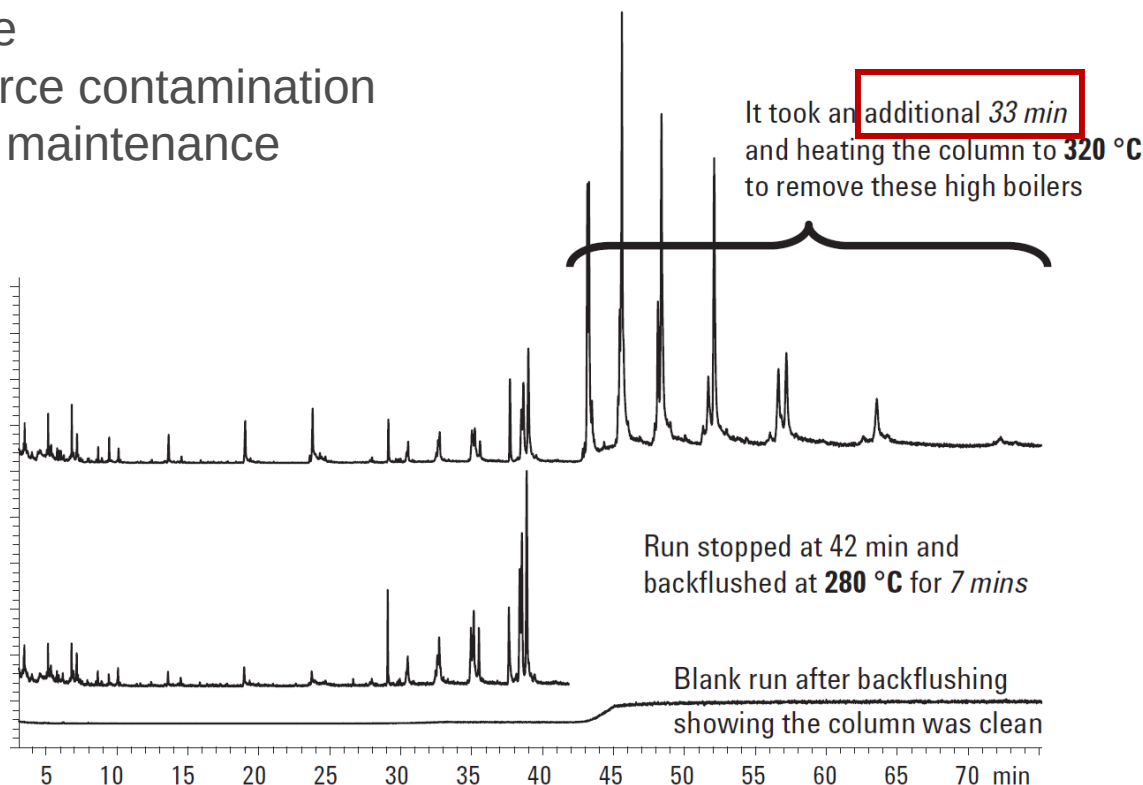


Column Backflush - Benefits

- Elimination of long “baked out” at a high temperature to remove less volatile, late eluting matrix components
- Reduced analysis time
- Increased column life time
- Prevention of the MS source contamination
- Less frequent MS source maintenance



Purged Ultimate Union



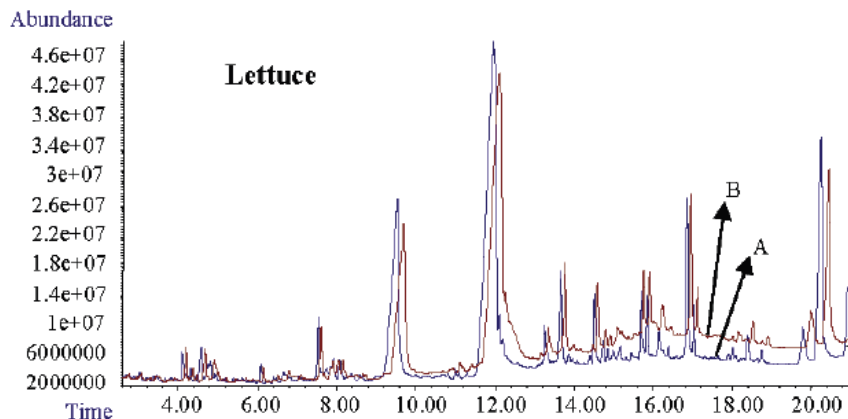
C.-K. Meng, Agilent Application Brief 5989-6018EN



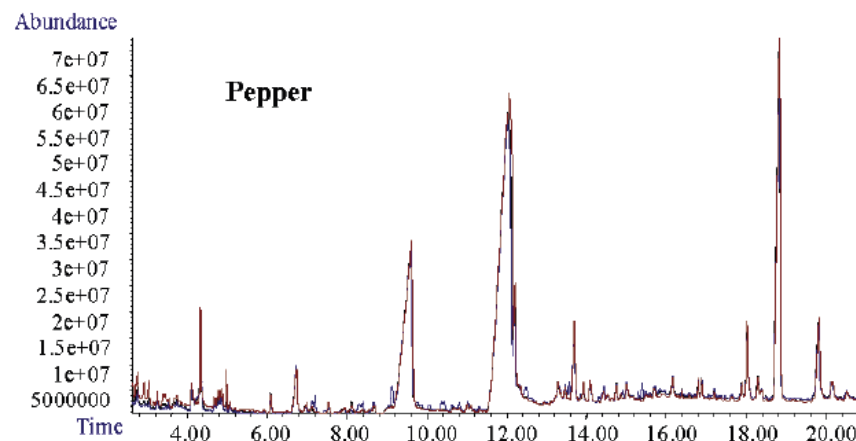
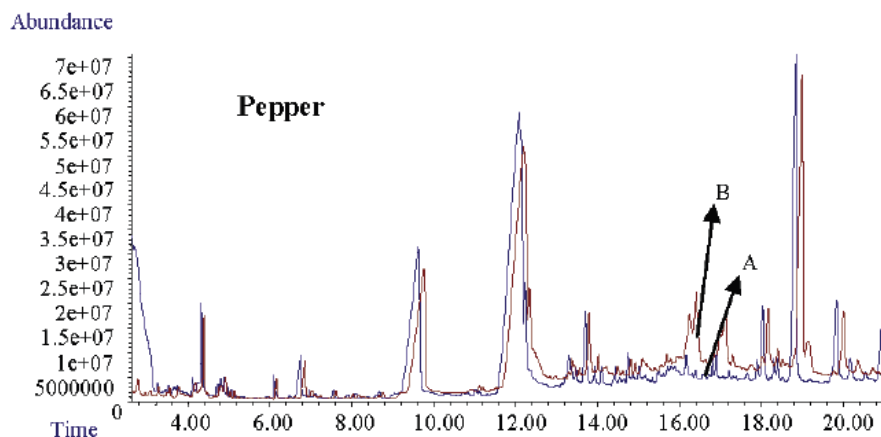
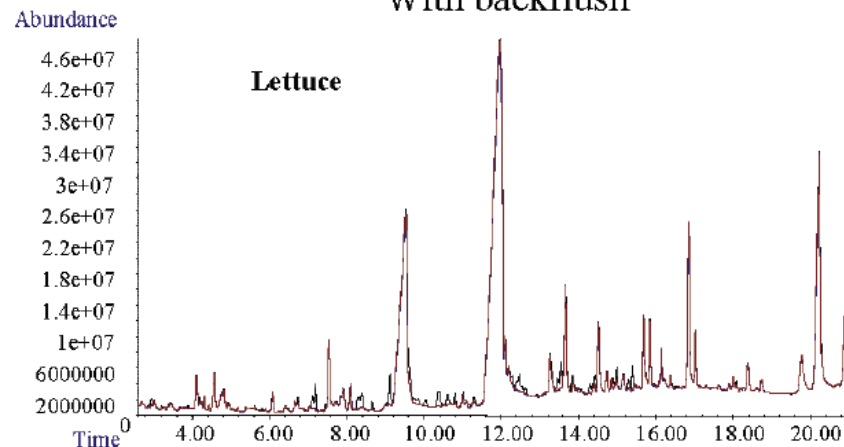
Agilent Technologies

Column Backflush - Benefits

Without backflush



With backflush

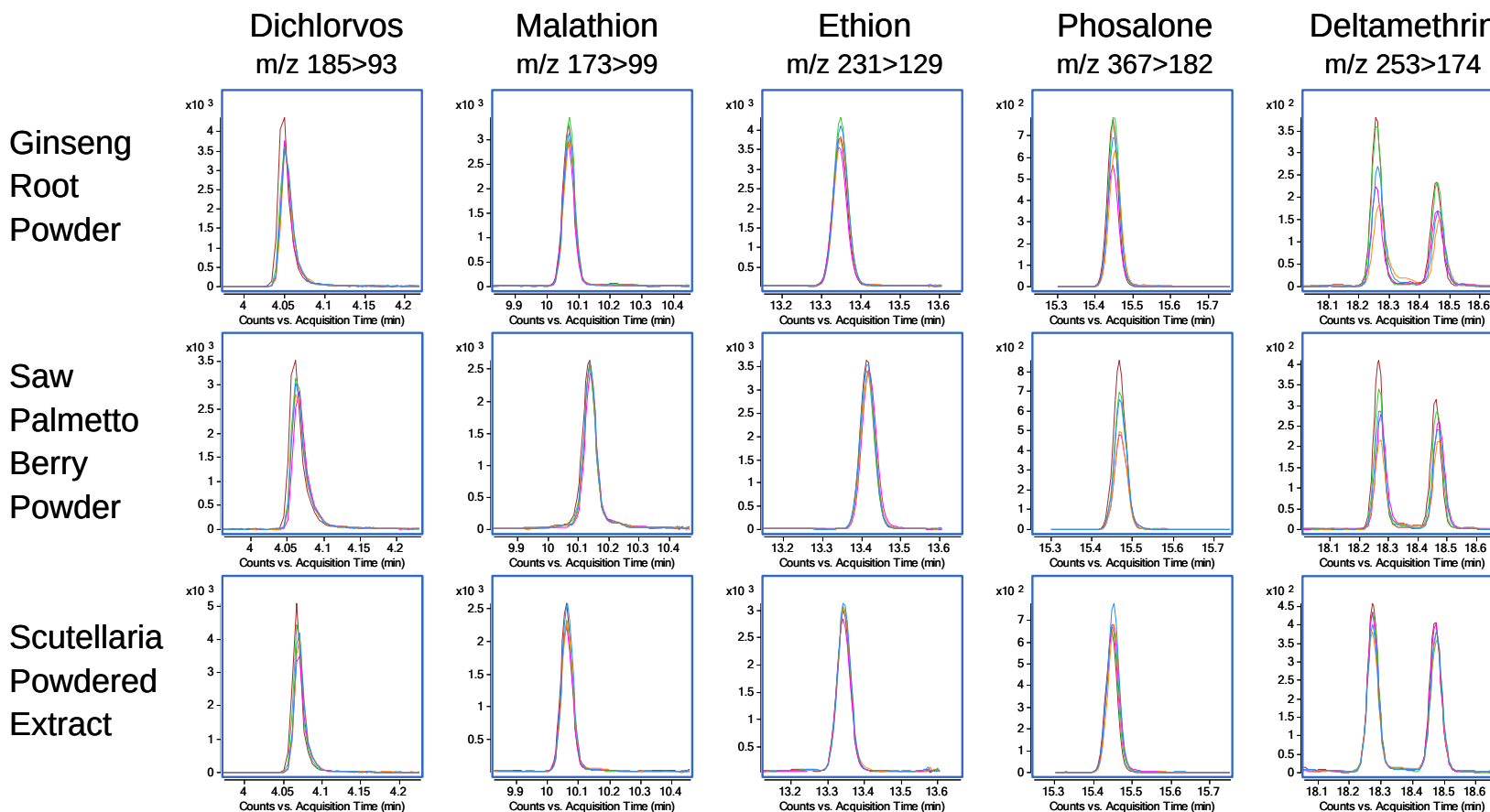


M. Mezcua, M.A. Martinez-Uroz, P.L. Wylie, A.R. Fernandez-Alba,
J. AOAC Int. **92** (2009) 1790-1806.



Column Backflushing - Benefits

Overlays of GC-MS/MS chromatograms for selected analytes (at 50 ng/g)
obtained within a 2.5-day sequence of 125 dietary supplement sample injections



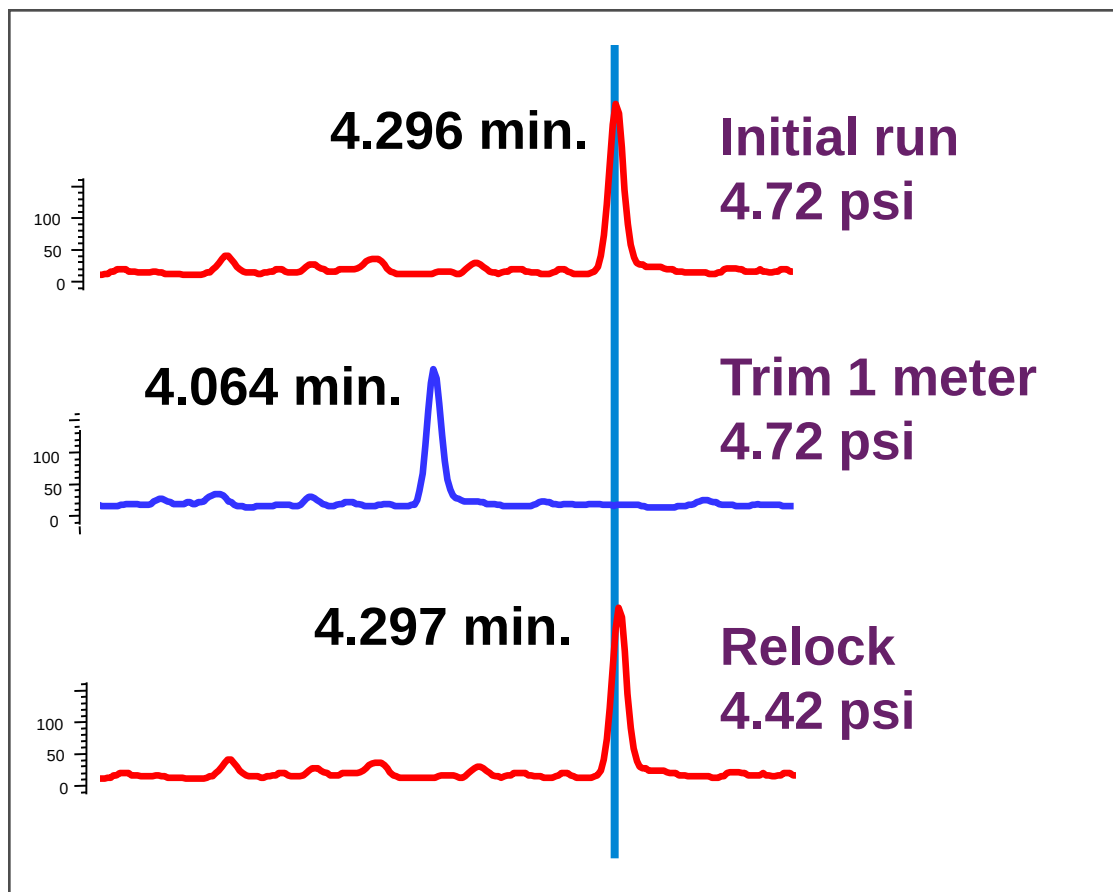
K. Mastovska and P.L. Wylie,
J. Chromatogr. A **1265** (2012) 155-164



Retention Time Locking (RTL)

Improve Confidence with Retention Time Locking

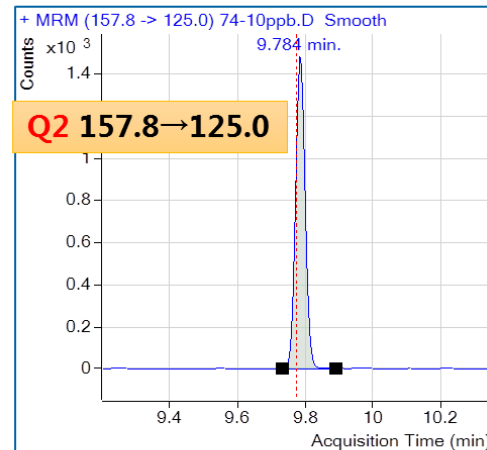
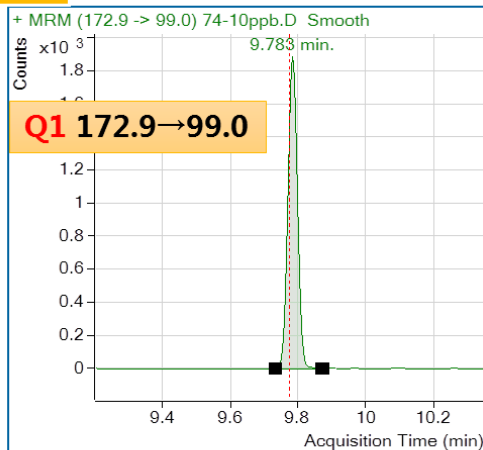
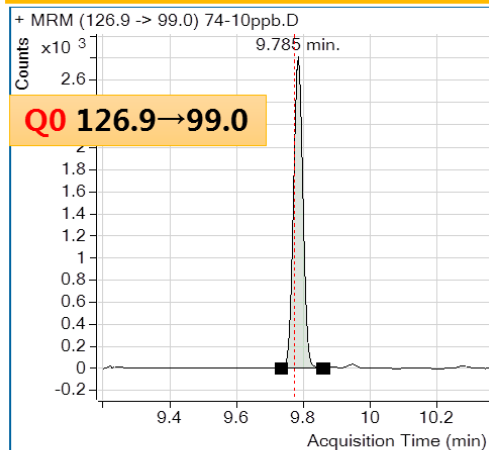
- **MRM Spectrum Unchanged**
- **Troubleshooting**
- **Repeatability**
 - Run-to-run
 - Operator-to-operator
 - Instrument-to-instrument



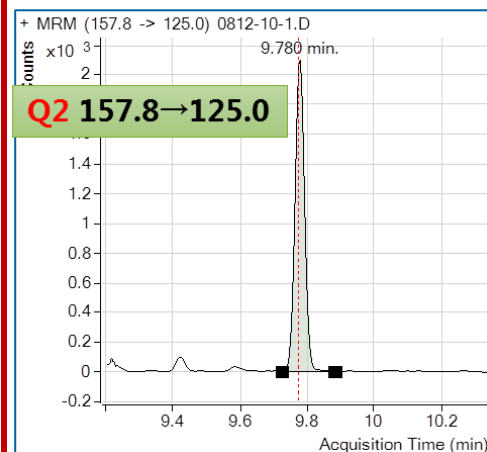
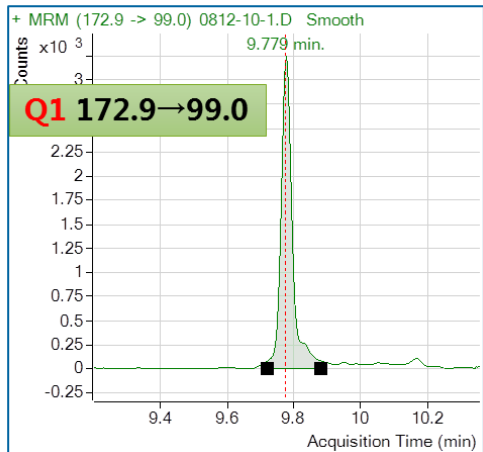
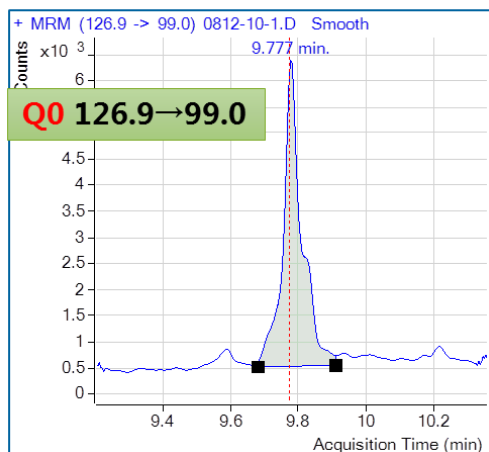
Importance of MRM Transitions

Malathion Identification

Malathion 10ppb standard MRM



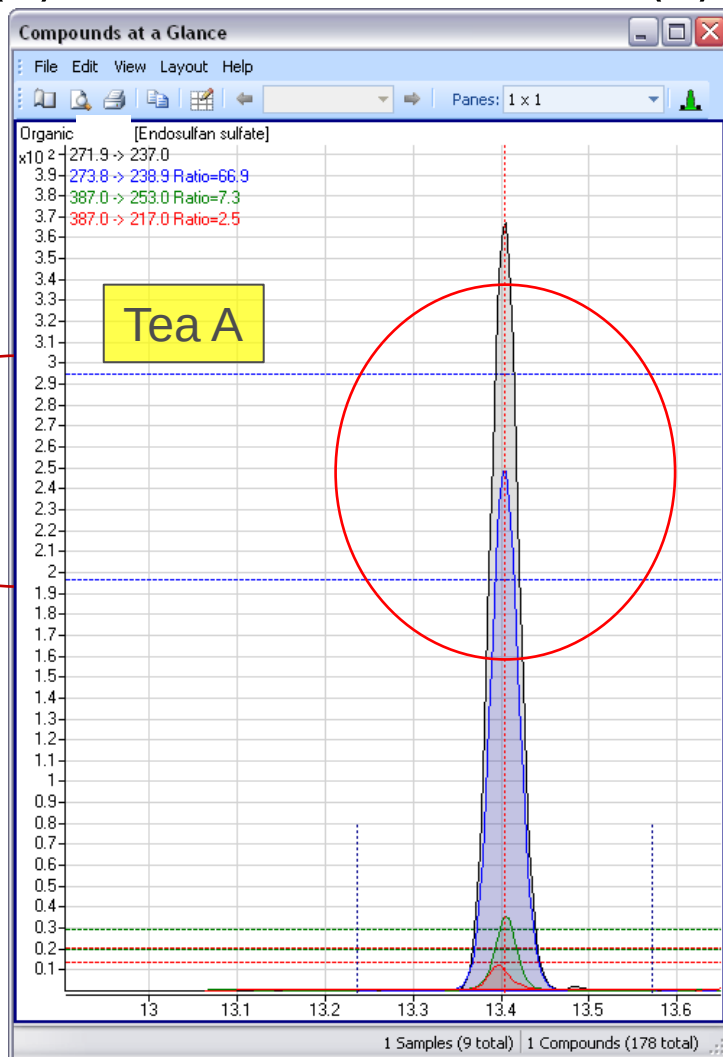
Malathion 10ppb MRM spiked in sample



Why are MRM Transitions Important?

Endosulfan Sulfate (?) in Tea Extract – Two (2) Transitions

- Rerun of data
- Ion Ratio within 80-120% confidence band
- CONFIRMED!!

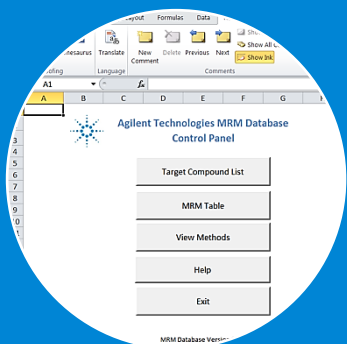


- Demonstrates the value of multiple optimized transitions in the MRM Database.
- Not just to avoid matrix interferences but also for additional confirmation!



GC/MS/MS Pesticide Analyzer

Value Overview



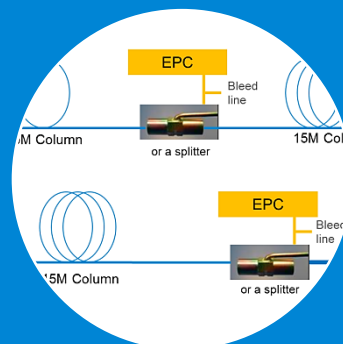
Powerful MRM Database

- The most Comprehensive and Flexible MRM database!
- New User Interface that streamlines the creation of custom MRM acquisition and quant methods.



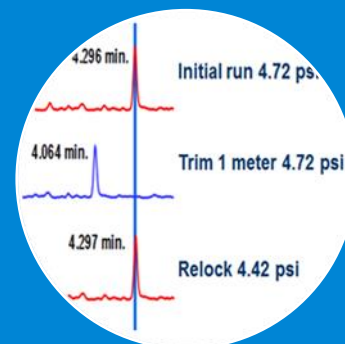
MultiMode Inlet (MMI)

- Large volume injection helps optimize LOD performance
- Opt 114 UI S/SL for customers that do not require for optimal LOD performance



Includes New Analytical Methods

- Expands analysis options:
 - Constant Pressure methods: #411 & #415
 - Constant Flow methods: #412 & #414



Auto RTL for CFT BF Methods

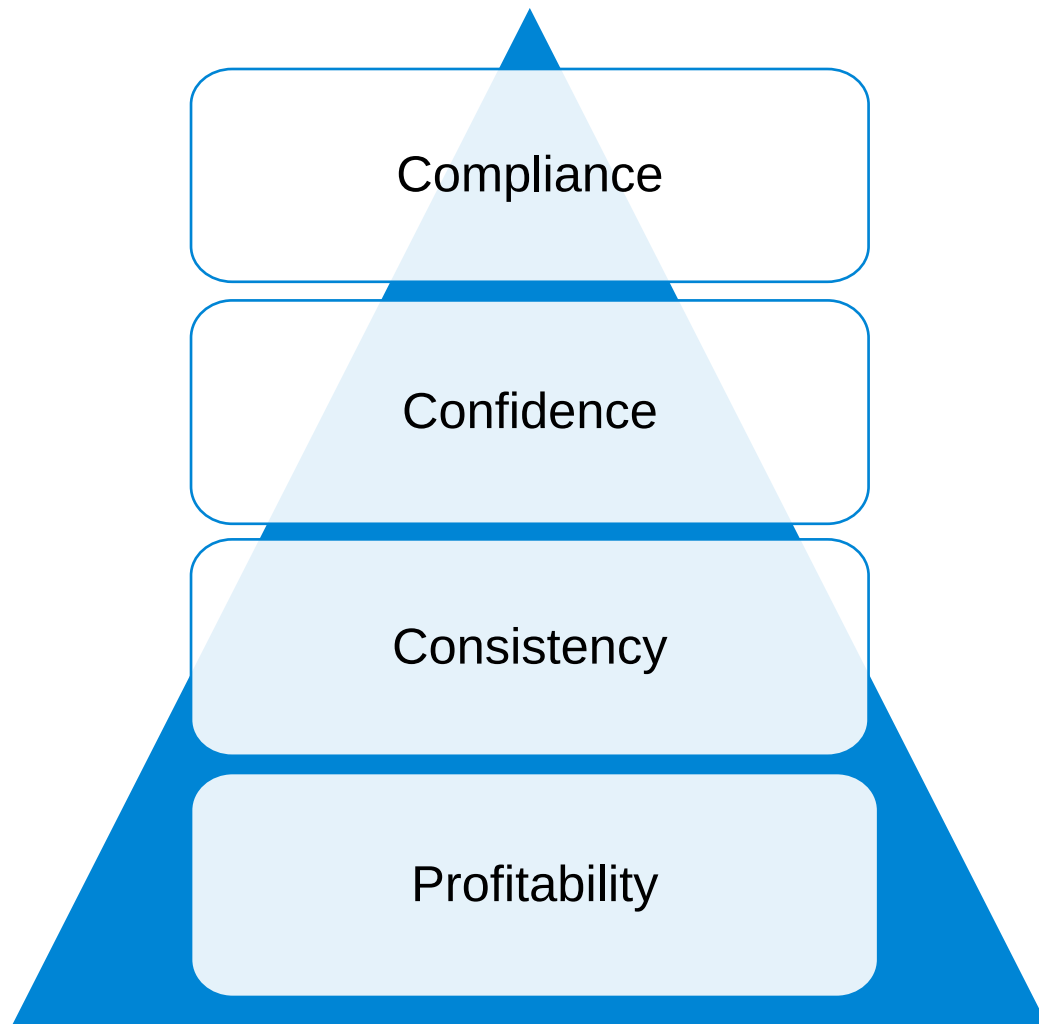
- Auto RTL for CF and CP BF method
- Facilitates relocking methods

Accurately confirm target pesticides while reducing the time required from start-up to results



Agilent GC/MS/MS Pesticide Analyzer

Confident Identification of Pesticide Residues



Questions...

Thank you for your attention

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