# Improving Your Analysis by Treating Your Flow Path with Agilent's NEW Ultra Inert Technology

### **Abby Folk GC Technical Support**



#### **GCMS Inert Flow Path**

Agilent's inert flowpath components improve the results, efficiency and throughout in GC/MS labs doing trace analysis in Environmental, Food pesticide and Forensic toxicology testing labs.



## **Agilent Inert Flow Path Technology**

- Aim for improved sensitivity for trace analyses
- Are dealing with troublesome, active compounds
- Battle with tailing peaks
- Want more reliable data at low concentration levels
- Higher productivity with less troubleshooting
- Want avoid wasting time with re-runs

## **Trace analysis in CAG labs**



Why are the method detection limits so low?

- · Our regulated world
- Scientific advancement
- Protecting our lives, environment and food supply

Why is it so challenging?

- Complex mixtures
- Active analytes
- System activity

What are the external challenges?

- Time
- Profit
- Skill level and training



## **GC/MS** Trace Analysis

Why do we focus on columns and supplies for improved trace analysis? Shouldn't a bigger, better, more sensitive GC/MS give you all the sensitivity you need?

It does! It just isn't enough anymore. For most analyses the gains from improving the chromatographic performance are also required to meet and maintain method requirements. Consider this:

What new 7890B features will have the greatest impact to your analyses? The Inert Flowpath, Improved GC detector specs, better GC/MS communications. All the above!



## **Laboratory challenges:**

"Critical" compounds?

Tailing peak shape? (specific compounds?)

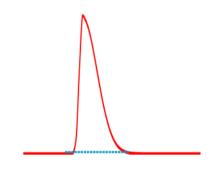
Difficulty detecting lower limits?

Breakdown?

Gradual loss of response?

Matrix influence?

Short liner lifetimes?





The Technical Argument is Very Compelling

### How can Agilent's Technology help?

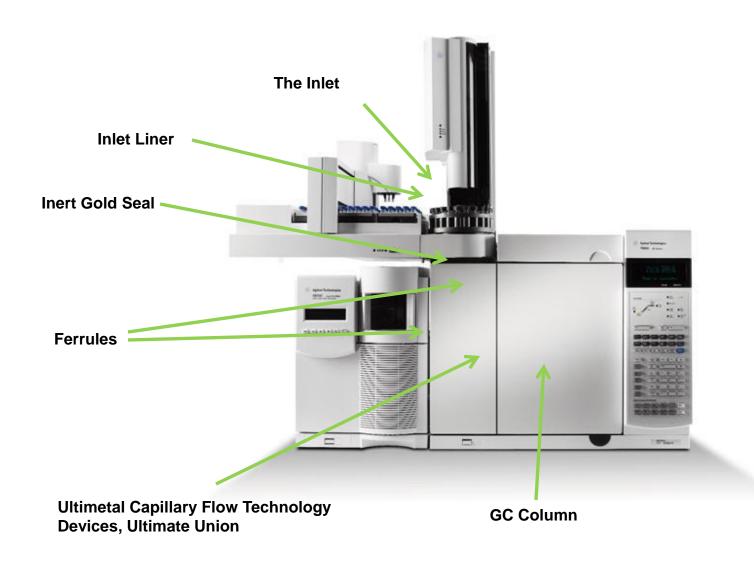
Agilent's approach to improving trace GC/MS lab performance?

- Failures from contamination
- Solving bleed problems
- "Active" solutes and "active" surfaces



Where are the active surfaces in the flowpath? Which are heated?

## GC surfaces that touch the sample



## Thirty years of R&D focused on surface deactivation

Two unique chemistries were developed to treat the surfaces in a GC flowpath. Agilent trade secrets!

#### **UltiMetal Plus**

- Inorganic vapor deposition in Middelburg
- CFT device
- Ultimate union
- Flexible Metal ferrule
- · Inlet welding
- Detector parts
- GC Tubing



#### **Ultra Inert**

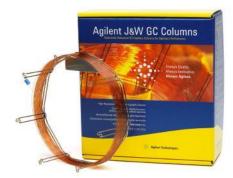
- Organic vapor deposition in Folsom
- UI GC columns:
   Arylene/phenyl
   Phenyl/cyanopropyl
- Ultra Inert liners
- Ultra Inert Gold seal

#### Which Products are the Focus of this Solution?

#### All supporting the GC/MS performance

#### **Columns**

•J&W Ultra Inert GC/MS columns



#### <u>Supplies</u>

- Ultra Inert Inlet Liners
- •Flexible Metal Ferrules
- Deactivated Inlet weldment
- Ultra Inert Gold Seal
- Gas Clean Filters
- GC/MS Quiet Cover









#### Sample Preparation Products

- Bond Elut SPE (Enviro, Forensic Tox)
- QuEChERS (Food Pesticides)







## Activity GC Flow Path Components that Influence Compound Response

1. GC column Largest surface area

2. Inlet liner Continuously high temperature

3. Ferrules Small surface area, important

4. Gold seal Gold is not inert

5. Unions, CFT devices Active metal base

6. Inlet weldment Metal activity (backflash)

7. Tubing Backflash, diffusion

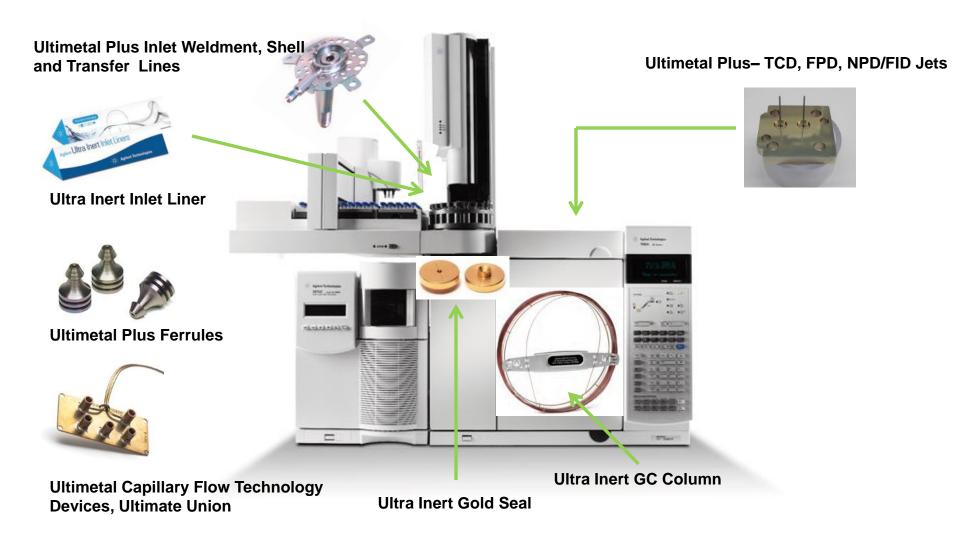
## **Benefits of Agilent Inert Flowpath**

An Inert Flowpath means			
Sharper peaks	More sensitivity Better resolution	Stays calibrated Higher quality data, easier analysis	Saves time Confidence in results
Staying calibrated longer	Less maintenance and less re-runs	Increased time analyzing real samples	Saves time and Improves throughput
Less false negatives	Less compounds adsorbed by the system	Higher data quality	Confidence in results
Cleaner components	Faster maintenance Less interference	Easier interpretation Improved data quality	Saves time Confidence in results

## **Inert Flowpath Story: What Agilent brings**

- Not one, but two proprietary Chemistries to deactivate surfaces and sustain ultimate inertness
- 2. Unique consumable products that work better and are designed to keep the GC and GC/MS delivering ultimate performance easier and longer.
- 3. Packaging that maintains cleanliness while in the drawer and during installation and supports fast error free installation.
- 4. Agilent brings products that make the GC/MS lab a nice place to work.
- 5. Agilent brings purifiers to keep O<sub>2</sub> out of the system and maintain inertness and performance longer
- 6. Finally Agilent brings sample prep technologies that keep the system clean and delivers better results.
- 7. Everything from Agilent

### **Agilent Inert Flow Solution**



### **Current IFP Product: Ultra Inert GC Columns**

Largest surface area in the flow path

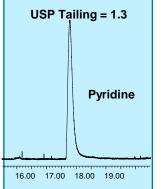
Improved peak shape at low levels

Reliable, verified inertness performance

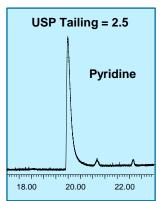
- DB, HP selectivity
- **Expanding offering**

Agilent Technologies

Agilent 624 UI **USP Tailing = 1.3** 

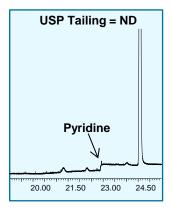


Competitor 624



Cheap 624

Agilent J&W GC Columns



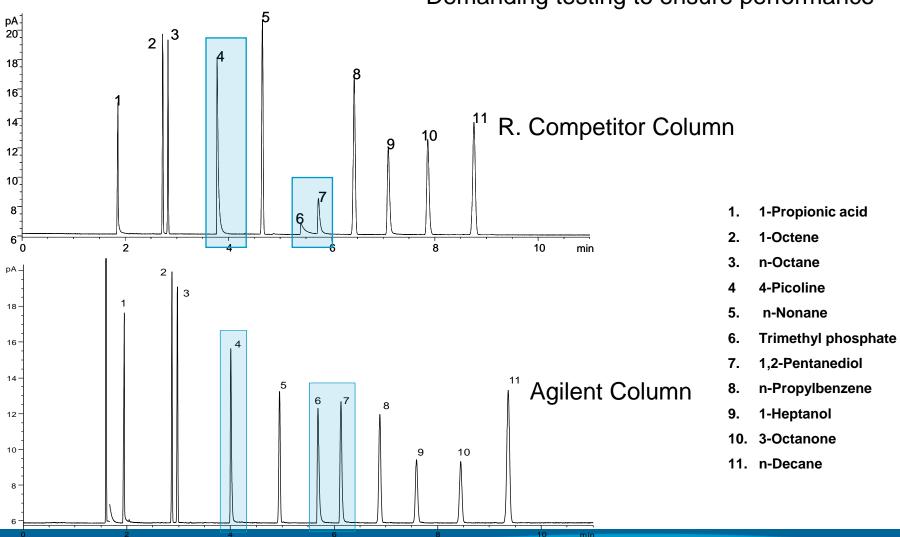
## **UI GC Columns: The Right Chemistry**



GC Column Phase	Characteristic	Application	Faster analysis configurations
DB-1MS UI, HP-1MS UI	General low polarity	General separations, hydrocarbons	yes
DB-5MS UI, HP-5MS UI	General low polarity	first choice for trace applications	Yes
DB-35MS UI	Mid polar	excellent for pesticide analysis, drug analysis	Yes
DB-624MS UI	Mid polar	choice for volatiles and solvents	Yes
DB-UI 8270D	Application specific low polarity	Specific for semi volatile environmental analysis	yes

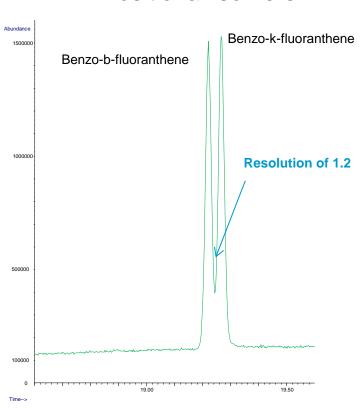
### **DB-5ms Ultra Inert**

- Ultra Inert deactivation technology
- Demanding testing to ensure performance

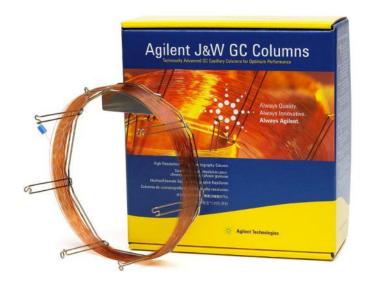


## DB-UI 8270D: Resolution of benzo-b & k fluoranthene isomers

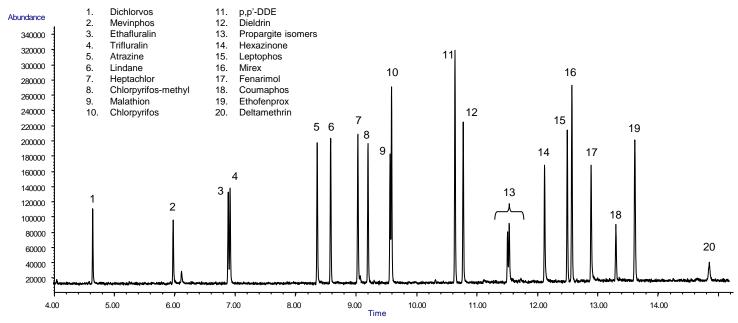
#### Positional isomers



- •Tested to ensure key environmental separations
- Excellent for "active" pesticide compounds



## **Separation of Pesticide Analyzer Checkout** Solution: DB-35ms UI



#### GC/MS Chromatogram of 1 ng on column loading of pesticides

Sample: 1 µg/mL Pesticide Analyzer Checkout solution (Agilent part

#5190-0468)

GC/MSD: 7890/5975B with purged ultimate union

Column: DB-35ms UI 20 m 0.18 mm 0.18 um (Agilent part #121-

3822UI)

MMInlet: 1μL, splitless, 50°C (0.02 min), 400°C/min to 250°C

purge flow 50mL/min at 1.5 min gas saver 30mL/min at 2.25 min

Carrier: Helium, 1.3 mL/min cnst flow

Oven: 50°C (1.3 min) to 135°C (50°C/min), 15°C/min to 200°C, 20°C/min to 310°C (2.35 min)

Restrictor: 0.7m x 0.15mm ID Deactivated capillary column tubing

3.8 psi constant pressure

Backflush: Post column, Postrun backflush 5 min @310°C

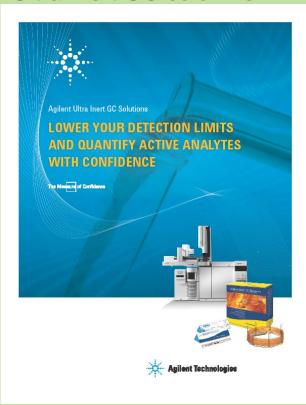
70 psi Backflush Pressure

2 psi Inlet Pressure during Backflush

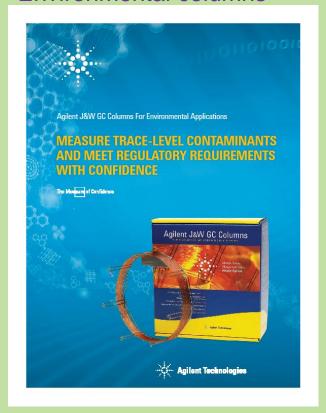
Transfer line 320°C Source 320°C Quad 150°C **Agilent Technologies**  Application note 5990-6595EN

## **Ultra Inert and Environmental GC Column Brochures**

#### Ultra Inert GC columns

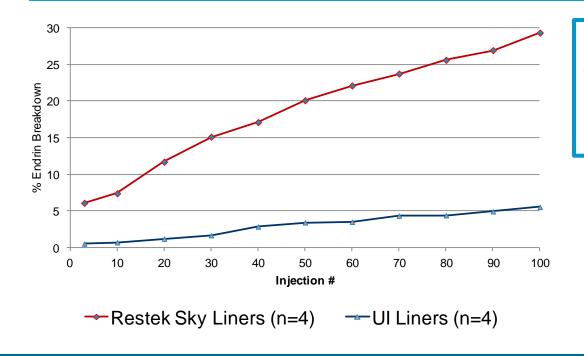


#### **Environmental columns**



#### **Ultra Inert Inlet Liners:**

- Ultra Inert deactivated inlet liners provide higher response for sensitive compounds
- 2. Ultra Inert **Glass wool liners** deliver benefits of glass wool w/o loss of active compounds
- 3. QC tested & certified for consistent performance



### **Productivity:**

Touchless packaging with preinstalled o-ring: quick & easy hassle free installation



## **Agilent Assurance**

UI liner Lot QC with demanding test probes

Assurance of consistent performance

Label for p/n, batch and lot testing

Traceability

Deactivation Lot number is on Certificate

Liner lot number (and part number) etched on glass

(dinitrophenol)

#### Certificate of Performance

5190-2293 Ultra Inert Liner

Splitless, Sngl taper, Glass Wool

Liner Body Lot:

0023A

Deactivation Lot:

B11002

Tested for:

2ng 4-Animopyridine 2ng 2,4-Dinitrophenol

## Packaging, Ease and Productivity



**Individual liner** 



Convenient 5 pack in Touchless package



CrossLab 5 pack in blister (no o-ring)



25 pack in Touchless Dispenser High throughput



100 bulk pack in blister (no o-ring)
High throughput



#### **Ferrules**









Graphite Siltite

Composition	Re-use	Max T	Use	Limitation
Vespel	yes	280	Easy seal	Leaks after T cycle, iso only
Vespel/graphite	limited	350	MS	Retighten after T cycle
Graphite	yes	450	Not MS	Contamination, leakages
Siltite	yes	450	MS, CFT	Column breakage



Dial packaging





"Short" ferrules for detector and inlet configurations on Agilent GC's, provide a robust seal.





"Long" ferrules for MS transfer lines and MS interface nut

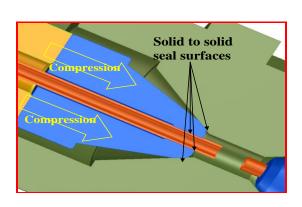
## Metal (Siltite) Ferrules in Agilent Systems

- Fastest growing family of GC ferrules
- Connections with Capillary Flow Technique devices
- Secure leak free connections LTM columns
- GC/MS transfer lines

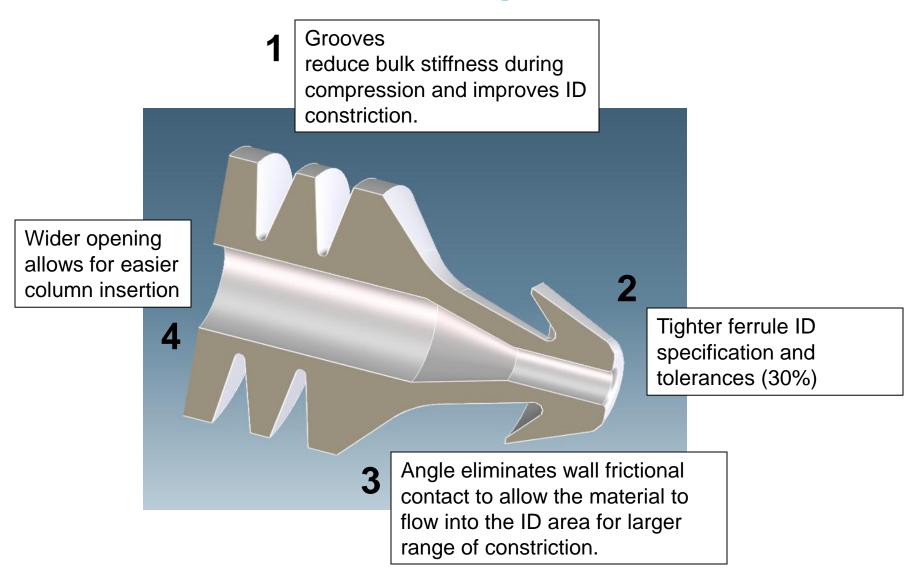
We experience high failure rate due to non-fitting ferrules for LTM (Low Thermal Mass modules)

### Computer aided theoretical modeling of Siltite design

- Design flaw identified
- ID spec and tolerance recommendations made to supplier but was considered too costly



## Flexible Metal Ferrule Design Features



### Compare current vs new Agilent ferrule

#### **Current Ferrule**

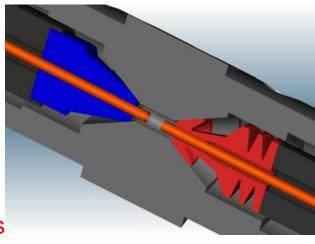
Stiff, prone to cause damage to column or fitting

Small ID compression (40 to 60um) – leaks

Tolerances poorly controlled

#### Quality issues

- mixed inventory
- not-fitting
- column crushing
- leaking



## Agilent Flexi-Metal Ferrule

Computer aided Design Finite Element Analysis

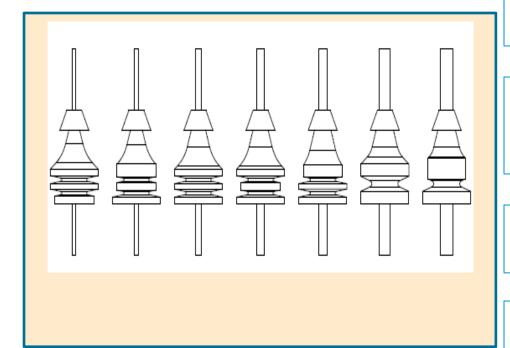
More flexible

Less column damage

Large range of ID compression (100 to 120um) – less leaks

Agilent design alleviates current quality issues

#### **More Added Value**



Family of ferrules for range columns Folsom and Middelburg including Ultimetal columns, and a no hole plug

New UltiMetal Plus surface treatment provides excellent ferrule inertness exceeding Siltite performance

Each ID has its own unique design Ease of recognition, no mix-ups

Agilent Intellectual Property: Utility patent & Design patent applications Exclusive Agilent product **New Touchless Dial Packaging** 

- Easy column insertion
- No lost ferrules
- No contamination risks



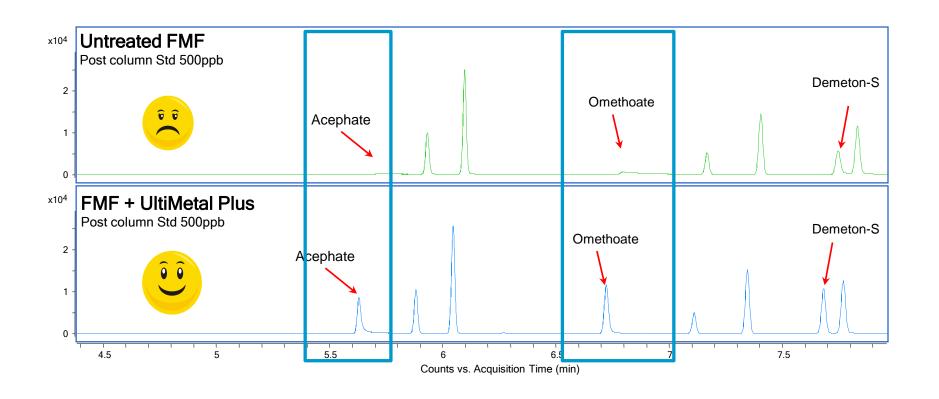


Clear installation instructions

Remove Ferrule

## UltiMetal Plus on Flexible Metal Ferrule Is deactivation needed for such small surface?

Enlarged chromatograms for sensitive pesticides



# Flexible Metal Ferrules Positioning

Substitute for Siltite ferrules in

- All CFT devices
  - QuickSwap Backflush
- Ultimate union
- □ LTM



"Long" ferrule applications

Not optimized for regular Agilent inlet / detector connections but possible! Graphite/vespel ferrules allow more "Short"

economical column trimming and re-tightning

"Short" ferrules

## Flexible Metal Ferrules: What are Customers Saying?

"We have tested them in our facilities. Can you also give me the part numbers for the 0.5 mm and 0.8 mm as well? We are planning to implement the technology swiftly to replace the Siltite ferrules."

Lab Director at a large chemical company





## **Summary Agilent Flexible Metal Ferrule**

Family of novel, patentable design capillary column metal ferrules
Capillary Flow Technology fittings
Agilent GC inlets and detectors
MS interface.

- Reduces risk of over tightening and breaking column
- Leak free seal after repeated temperature cycles
- UltiMetal Plus chemistry prevents loss of active analytes

#### Ease of Use

- Easy assembly and tighten to give a leak free seal ("forgiving")
- Ferrule ID identification by design
- Quick installation due to design & packaging

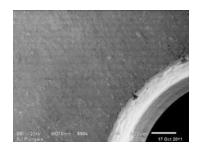
...and there's a video!



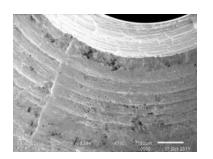
## Agilent UI Gold Seal: Deactivated gold surface

- Soft gold plating is essential for proper sealing
- •Ultra Inert chemistry blocks active sites (gold is NOT inert)
- Smooth surface doesn't leak (Injected molded)
- Advantage Agilent





Agilent MIM seal

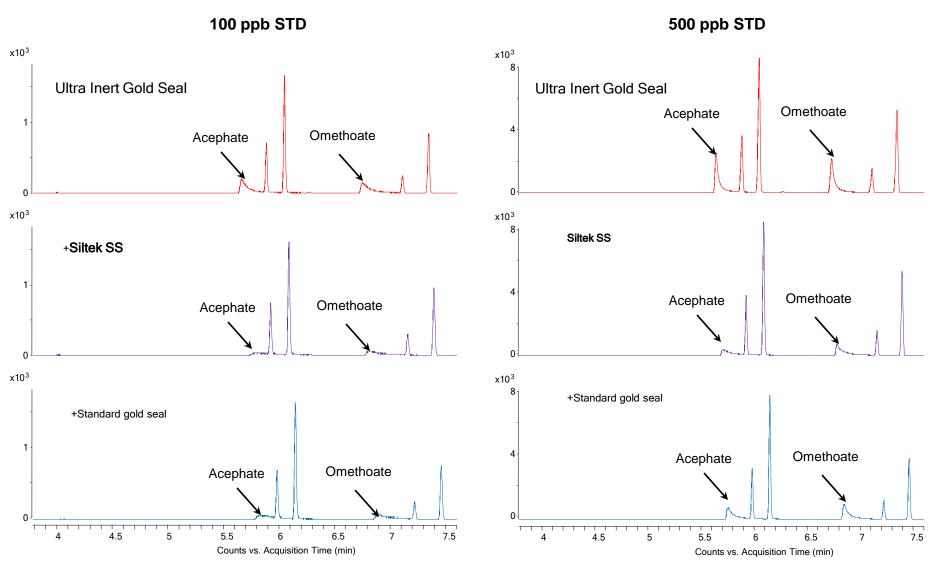


Competitor's machined seal

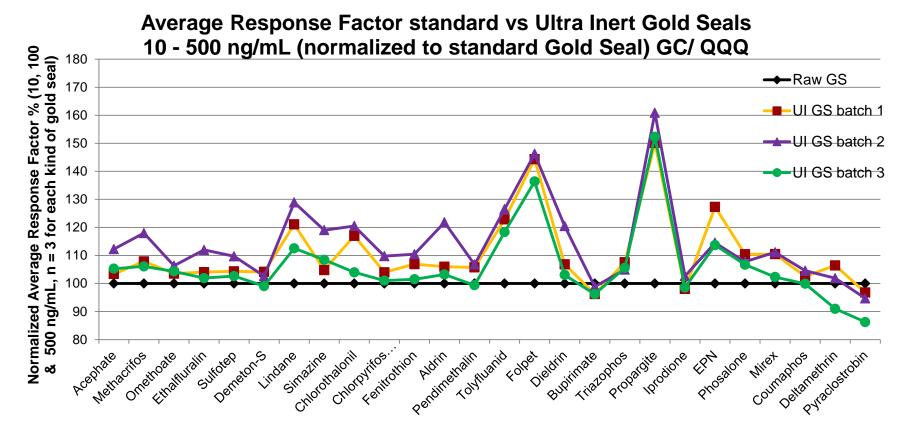
Reliable ppb and ppt measurements require attention to the little things!



## Results – Peak Shape Comparison for Sensitive OPs (Std GS vs New UIGS vs Siltek SS)



### **Ultra Inert Gold Seal - Pesticides Test (analytes responses)**



- •Pesticides responses (average response factor over 10 500 ng/mL) by using UI gold seal were higher (10-15%) than those by using Standard gold seal;
- 30-50% higher responses were achieved for several pesticides by using UI gold seal; and only two of 26 pesticides gave out a little bit lower responses, but the results may be linked to system performance

## **New Product: Agilent Inert Inlet**

## UltiMetal Plus treatment creates inert surface 7890 inlet weldment & shell

- Limit adsorption / degradation active analytes in contact hot metal parts.
- Target trace GC/MS and GC-ECD pesticide analysis
- Combine with Agilent's UI Liners, UI gold seals, UI GC columns
- Inert Flowpath option on the 7890B (#114) or upgrade existing 7890's in the field

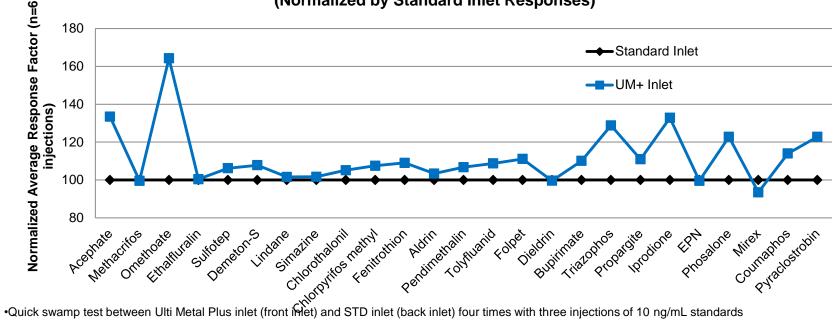


Agilent's proven proprietary Ultimetal Plus surface treatment

## Inlet Weldment – Analytes Response Comparison

### 10 ng/mL Average Response: UM+ Inlet vs STD Inlet

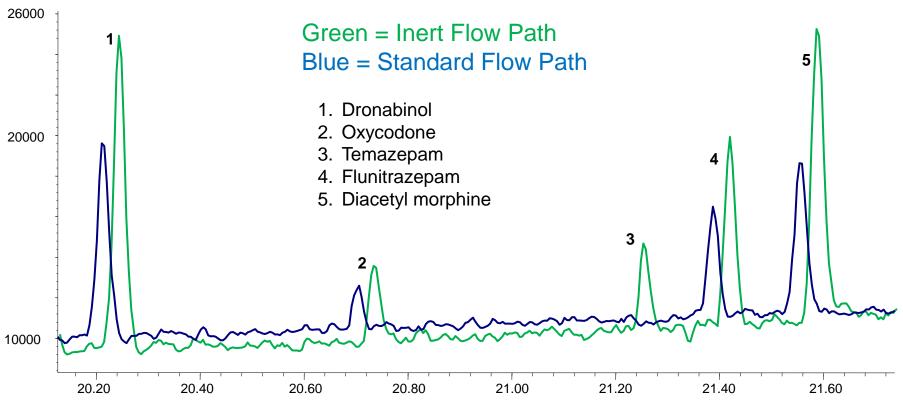
(Normalized by Standard Inlet Responses)



- •Column switched in between without trimming, and column was installed purposely with shorter protrusion to increase the contact.
- •UI single taper split/splitless liner without wool was used and switched in between.
- •Standard gold seals (from same batch) were used in both inlet.
- •Inlet weldments in general have very limited direct contact surface to liquid sample injection, therefore the inertness improvement impact is relatively hard to detect.
- •When column is installed correctly and a liner with wool is used, basically there's no difference founded between UM+ inlet and Stardard inlet weldments. Basically, UM+ inlet is equivalent to Standard inlet for pesticides analysis.

## Putting it all together – Inert Flow Path verses Standard Flow Path





Column: Agilent J&W HP-5ms UI 30 m x 0.25 mm x 0.25 um

Oven: 100°C 4 min hold, 10°/min to 280°C, 6 °/min to 300°C (4.67 min hold),

Carrier: Helium 52.7 cm/s (2 mL/min) set at 100°C, EPC-Constant Flow

Inlet: Pulsed Splitless 35 PSI pulse until 0.73 min, 0.75 min purge 50 ml/min, gas saver 20, ml/min at 2 min

Inlet liner: Ultra Inert with wool / Standard single taper liner with wool (p/n 5190-3165)

Gold Seal: UI Gold Seal / Standard gold seal

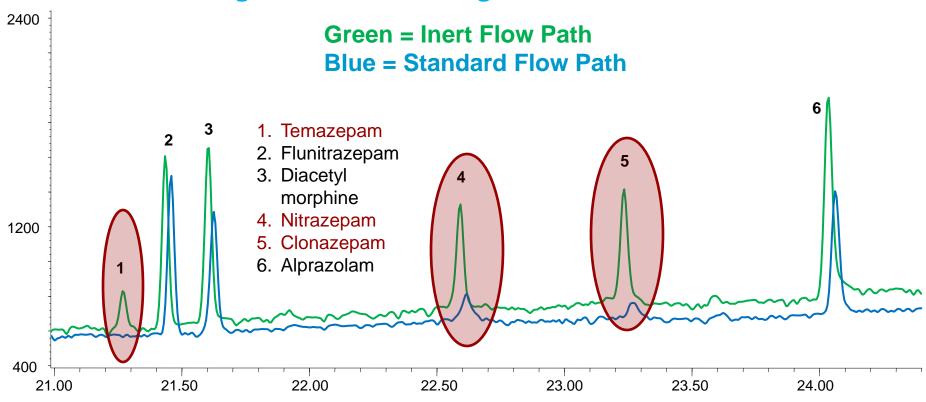
Detector: MSD Scan mode 40 t0 450 m/z, 230 °C source temp, 150 °C Quad temp, 310 °C transfer line



## Putting It All Together — Agilent Inert Flowpath

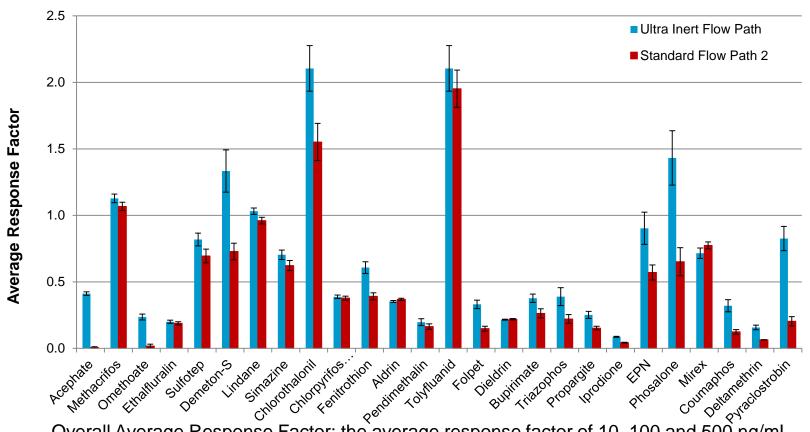
## **Dramatic Improvement at Low Levels**

### **Drugs of Abuse 0.25 ng Column SIM Mode**



## Putting it All Together: Pesticides Responses Improvement

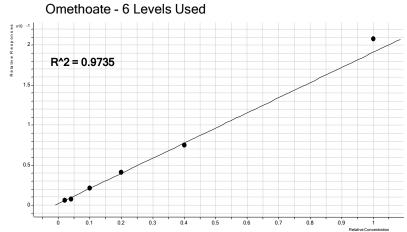
### **Overall Average Response Factor Comparison**

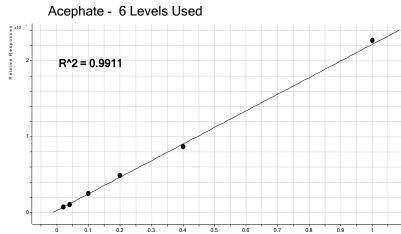


Overall Average Response Factor: the average response factor of 10, 100 and 500 ng/mL standards (n=4 for each level) for each analyte.

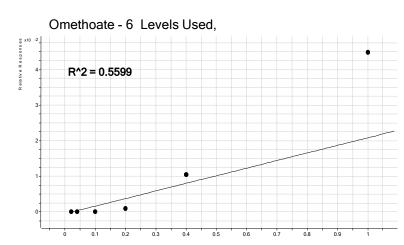
## **Putting it All Together: Linearity Improvement**

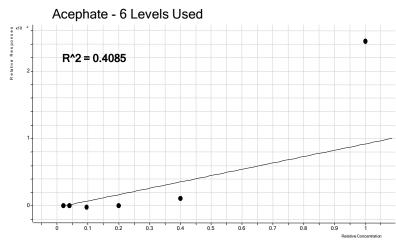
#### Ultra Inert Flow Path





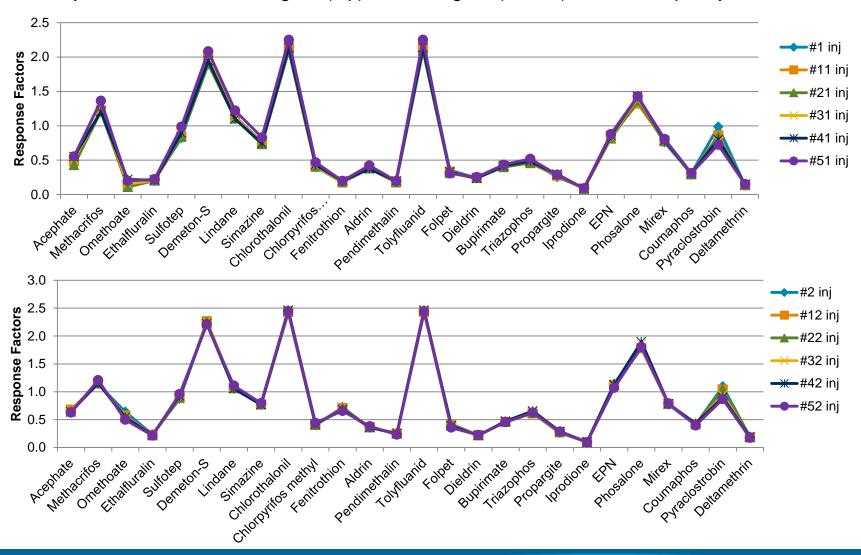
### Standard Flow Path





## Putting it Al Together: Flow Path Reproducibility

Six injections of Standard 50 ng/mL (top) and 500 ng/mL (bottom) over 50 sample injections

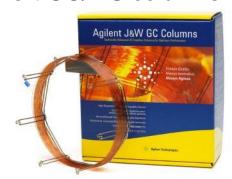


## So together this is a complete solution

### All supporting the GC/MS performance

### **Columns**

•J&W Ultra Inert GC/MS columns



### <u>Supplies</u>

- Ultra Inert Inlet Liners
- •Flexible Metal Ferrules
- Deactivated Inlet weldment
- Ultra Inert Gold Seal
- Gas Clean Filters
- GC/MS Quiet Cover











### Sample Preparation Products

- Bond Elut SPE (Enviro, Forensic Tox)
- QuEChERS (Food Pesticides)









## What about GC/MS/MS?

GC triple quads, such as the Agilent 7000, achieve incredible sensitivity for target analysis because the selectivity of the analyzer carefully removes the noise in the mass analyzer. The result is dramatically improved signal to noise because the noise contribution is diminished.

Some believe that because the spectrometer can't see the noise that it's not there.

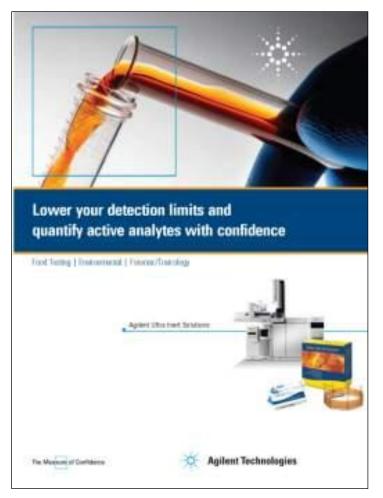
Nothing could be farther from the truth.

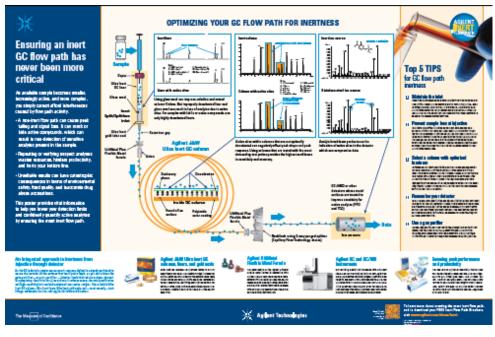
For GC triple quads, TOF's and other high resolutions systems the same rules apply:

- 1. Sharper peaks mean more sensitivity and better resolution.
- 2. Contaminates and non-volatile materials introduce activity that diminishes inertness and system performance.
- 3. The Agilent Inert Flowpath improves performance on *ALL* GC systems for trace analysis of active analytes



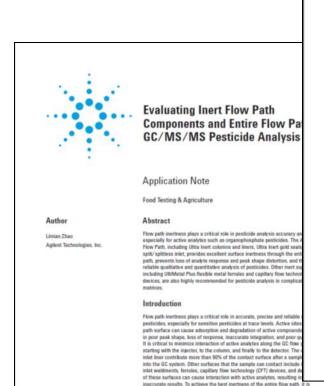
## **Agilent Ultra Inert Solutions Brochure and Poster**





http://www.chem.agilent.com/Library/brochures/Ultra%20Inert%20Portfolio%20Brochure.pdf

## **New CSD Application Notes**





Ken Lynam

Agilent Technologies, Inc.

#### Agilent Inert Flow Path Enhancements Improve Drugs of Abuse Testing

#### Application Note

Forensics & Drug Testing

#### Abstract

An evaluation of Sittle versus Agilent UttiMetal Plus Fixeible Metal terrules installed in an Agilent Uttimate Union showed improved bearcollarepine peak shape and response for UttiMetal Plus Fixeible Metal ferrules by FID. An Agilent 7869:76975. GC/MS Intert Row Path equipped with a spiti/spitiess inlet showed peak shape and response improvements for the benzodiszepine class of drugs whon

peak shape and response improvements for the benzodiazepine class of drugs when compared to a standard flow path split/splitless inlet in both SCAN and SIM modes. Example forensic/toxicology check-out mix chromatograms and overlays are shown.

#### Introduction

Drugs of abuse are chemically active substances that readily find and adordo not active aites anywhere in the flow past of a modern GC-VMS system. In page phase analysis, analyses must survive the passage from injection to detection. This is difficult to achieve when active analyses are compounds of interest are tracked at low level, and when the flow path has surface activity. As GC-VMS detection limits are pushed lower, the need for a suite of interflow path on components to like the flow path that analyses must travel through becomes ever more critical. Activity anywhere in the flow can lead to poor pask shape, poor quantifaction, or missed analyses.

Pravious efforts to eliminate active sites throughout the flow path have focused on column and lines deductation strategies (1.2). These strategies have been column and lines estantiages to the column and the strategies have been successful to the point where a focus on additional components in the next logical test proved achieving a totally invest lines with lines with lines gold reads. and ferralise used to make gas tight connections are all flow path. International components are successful and successful and successful and successful and components and successful and successful and successful and successful and components and successful a



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#### Application Note

Environmental

#### Abstrac

Flow path heretness plays a critical role in posticide analysis for accuracy and precision, especially for analyties subject to digradation in a hot inlet such as endrin and DDT. Catalytic endrin and DDT herealdown products were tracked as part of an inert flow path performance evaluation. The results indicate that the Agilient Inert Flow Platt Solution provides superior inertness and lower breakdown than standard flow posts split cypitaless index.

#### Introduction

The analysis of active compounds by gas chromatography (GC) continues to be challenging in areas such as pesticides, foods, environmental, and dray garalysis. To achieve reliable and solid results for active compounds, it is critical to minimize the interaction of active analysis along the GC five part, starting from the injector, to the column, and finally to the detector, All parts in this flow parts play a key role in enflowening the insertness of the flow parts. Active sizes in the flow part can lead to the degradation or advorption of active analysis, resulting its poor linearity of calibration curves and loss of sensitivity. Therefore, it is critical to decivate the entire sample flow path to make it fully insert. Minimizing surface activity throughout the flow path is essential to achieve consistent results.

Agilent Technologies

5991-1860EN

important to use an inert column and liner in combination with other inert supplies.

**Agilent Technologies** 

5991-1862EN



## A New GC and MS from Agilent

## Introducing the 7890B and the 5977

### 7890B

- Inert Flow Path
- New FPD
- Integrated Intelligence
- Parts Finder



### 5977

- Extractor Ion Source more sensitivity
- New vacuum systems
- Direct communication
- Quiet cover

Show video and Parts Finder



## **Inert Flowpath in Instrument Brochures**



**Inert Flow Path** 



## **Expanding the Opportunity: Maintaining GC/MS system performance**

## SPP and purifiers: for the rest of the lab!





Maintaining the advantages of an inert flowpath is important:

- Contamination from samples
- Oxygen contamination

Sample Preparation Products: Maintaining Performance



## Environmental Labs

- Role of LLE
- SPE for many applications

### Food Labs

- Trend for QuEChERS
- Still a role for SPE

### Forensics labs

High volume
 SPE labs

## Bond Elut QuEChERS Applications Notebook and Selection Guide





## Gas Clean GC/MS Filter

## **Optimizes your GC/MS performance**

- Single combination Filter, optimized to remove oxygen, moisture and hydrocarbons from GC/MS carrier gas
- Improved GC/MS filter design provides faster stabilization time to enhance productivity and reduce helium gas consumption
- Increased GC/MS sensitivity with more accurate data
- Prevents column damage, lowering column replacement costs
- Reduces instrument down-time



## Modular Design, Quick Installation





Remove plugs before installation





Efficient, fast, easy

## **Agilent Inert Flow Path Technology**

## Innovative and proprietary deactivation technologies improve GC and GC/MS performance

- Sensitivity
- Productivity
- Reliability

## Columns and supplies from Agilent add value to system performance

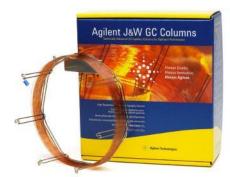
- Better product consistency and quality control
- Excellent availability and faster delivery
- Skilled and responsive technical support

## **A Complete Solution from Agilent**

### All supporting the GC/MS performance

### Columns

•J&W Ultra Inert GC/MS columns



### <u>Supplies</u>

- Ultra Inert Inlet Liners
- •Flexible Metal Ferrules
- Deactivated Inlet weldment
- Ultra Inert Gold Seal
- Gas Clean Filters
- GC/MS Quiet Cover









### Sample Preparation Products

- Bond Elut SPE (Enviro, Forensic Tox)
- QuEChERS (Food Pesticides)







## **Conclusion**

- •Flow path inertness is critical to the success of pesticides analysis, especially for the active pesticides at low ppb level.
- •Comparing to standard flow path, Agilent Ultra Inert flow path improves sensitive pesticides responses and peak shapes, thus improves the quantitation results.
- •Agilent Ultra Inert flow path is highly recommended for the accurate and precise pesticides analysis.

## TECHNICAL SUPPORT

1-800-227-9770, #3, #3, #1



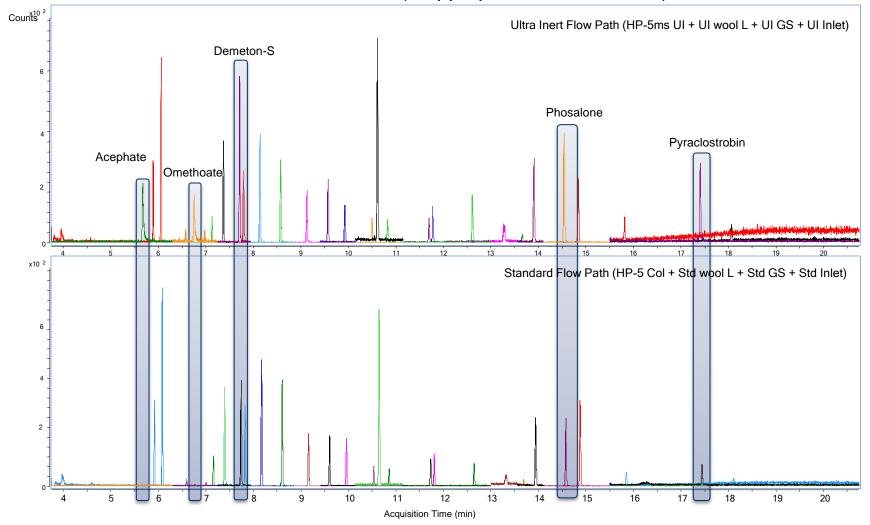
E-mail: gc-column-support@agilent.com

## **Additional Data**



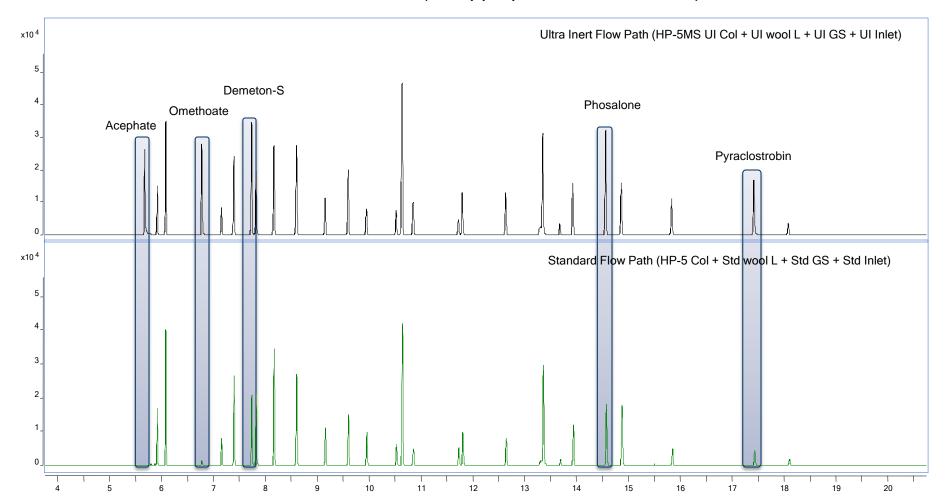
## Flow Path Test – Chromatographic Improvement

UI Flow Path vs Standard Flow Path (10 ppb pesticides standard)



## Flow Path Test – Chromatographic Improvement

UI Flow Path vs Standard Flow Path (500 ppb pesticides standard)

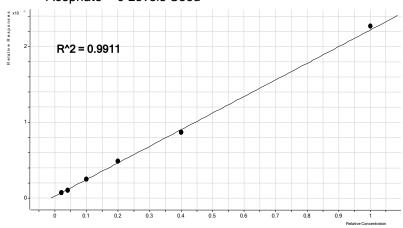


## Flow Path Test – Calibration Linearity Improvement

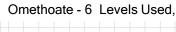
### Ultra Inert Flow Path

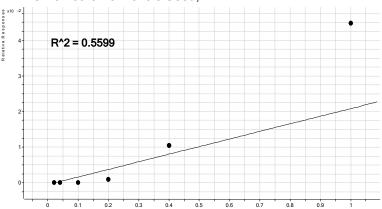
# Omethoate - 6 Levels Used $R^2 = 0.9735$

Acephate - 6 Levels Used

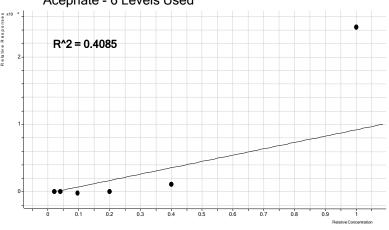


### Standard Flow Path



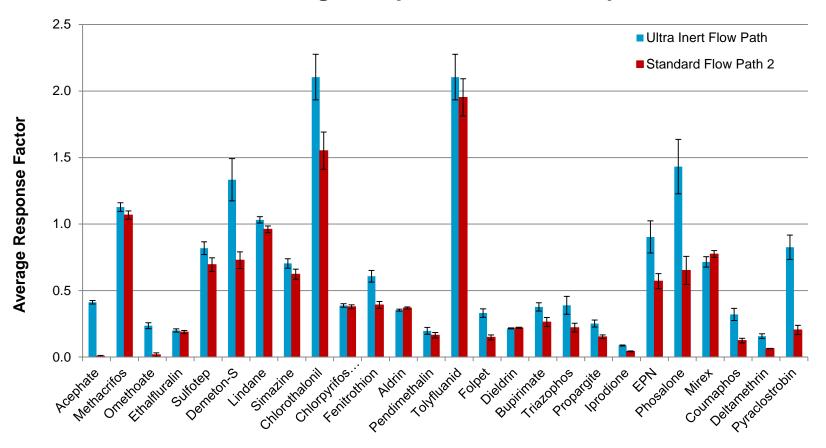


Acephate - 6 Levels Used



### Flow Path Test – Overall Responses Improvement

### **Overall Average Response Factor Comparison**



Overall Average Response Factor: the average response factor of 10, 100 and 500 ng/mL standards (n=4 for each level) for each analyte.

## Results and Discussion - Flow Path Reproducibility

Six injections of Standard 50 ng/mL (top) and 500 ng/mL (bottom) over 50 sample injections

