The importance of Agilent's inertness for columns, consumables and hardware

Better Business Solutions for GC and GC/MS

Jan Willem Marinissen

Technical Specialist Consumables Products

MS Users Day Amstelveen, 2nd July 2014

What Does GC System Inertness Look Like?

Easier question: What does poor inertness look like?

Symptoms of poor GC system inertness:

- * Tailing peaks
- * Reduced peak response
- * No peak response
- * Extra peaks!
- * Poor linearity of a peak usually at low concentrations
- * Unstable detector baseline

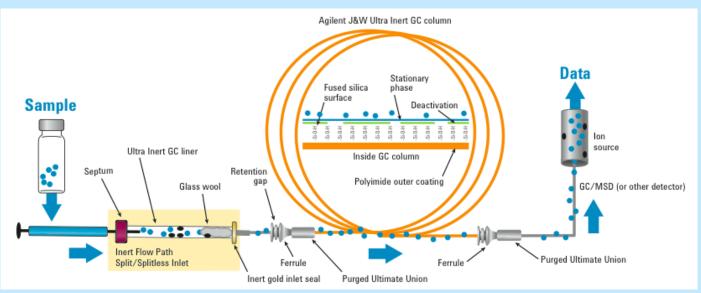
Protection from Injection to Detection

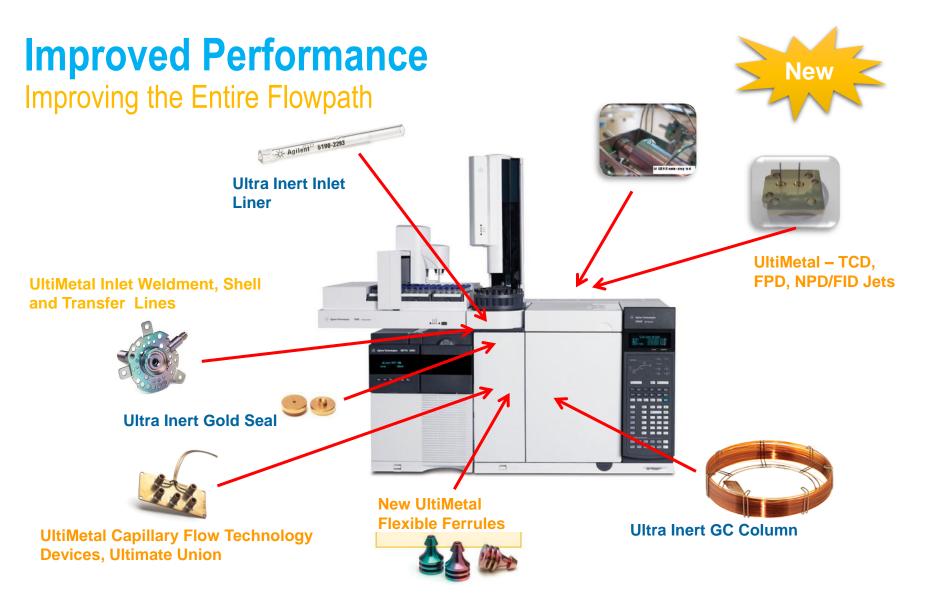
Agilent UltraInert and UltiMetal-Plus Technologies



Core Competency

Two Proprietary Deactivation Chemistrys for Glass, Steel, Gold, Fused Silica, Glass Wool are required to improve GC and GC/MS for trace analysis





...now from a single supplier

What is the Surface Area Contribution to Overall Flowpath Inertness?

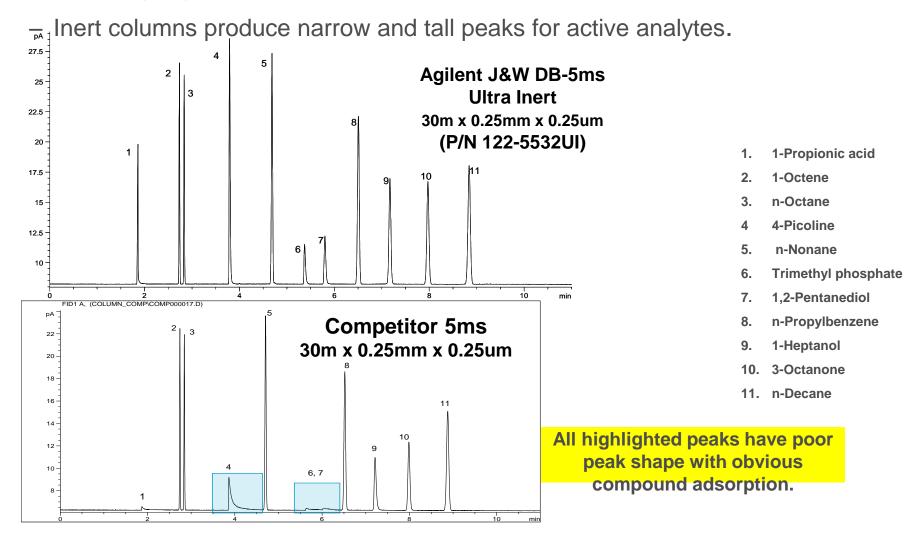
GC Flowpath Surface Areas

	L (cm)	d (cm)	π	Surface Area (cm²)
Liner (No wool)	7.85	0.4	3.142	9.86
Gold Seal		0.8	3.142	0.5
Column	3000	0.025	3.142	235.6



Column Inertness and Sensitivity

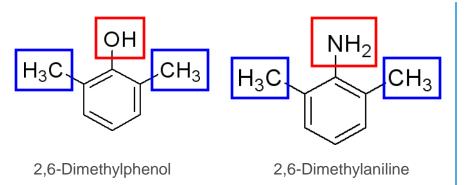
Increasing signal



Test Probes and Column Activity QC Testing

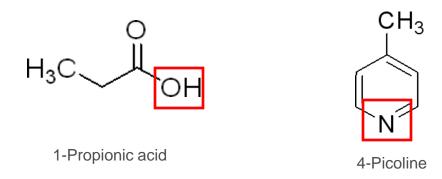
- Test probes are vital to ensure the quality and reproducibility of GC columns
 - Properly deactivated
 - Contain the correct amount of stationary phase
 - consistent column-to-column relative retention time
- Test probes can either highlight or mask the deficiencies of a column
 - An organic acid
 - A base
 - An alcohol
 - Non-active probes (e.g. alkanes)
- Good test probes allows the probative portion of the test module to penetrate and fully interact with the columns stationary phase and surface.
 - Low molecular weight
 - Low boiling points
 - No steric shielding of active group

Weak Probes vs. Strong Probes



Weak Probes

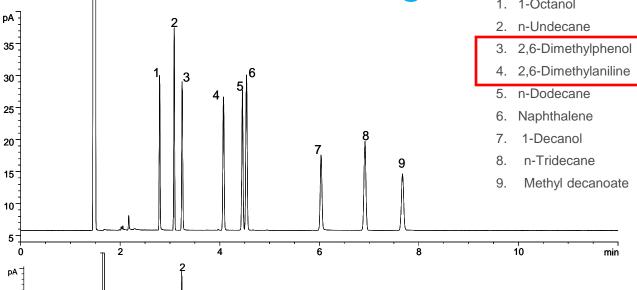
Acidic and basic portion of the molecules are shielded by the methyl groups of the 2,6-dimethyl substituted phenyl ring



Strong Probes

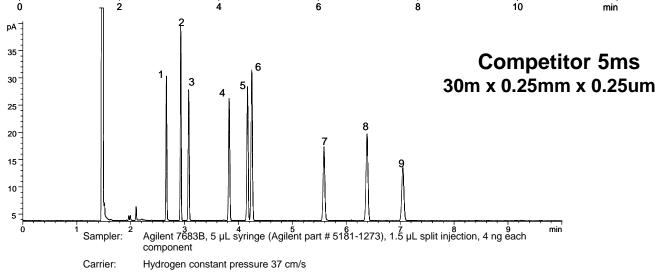
Active end of each compound is available to interact with any active sites on the columns

Grob-Type Test Mixture - QC Testing of the 80s



- Elevated oven temperature at 120°C
- Probes sweep past active sites and mask solute/column interactions.
- Least probative probes for column activity

NOT Probative



Inlet: Split/splitless; 250 °C, 1.4 ml/min. column flow, split flow 100 ml/min. Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)

Oven: 120 °C isothermal

Detection: FID at 325 °C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

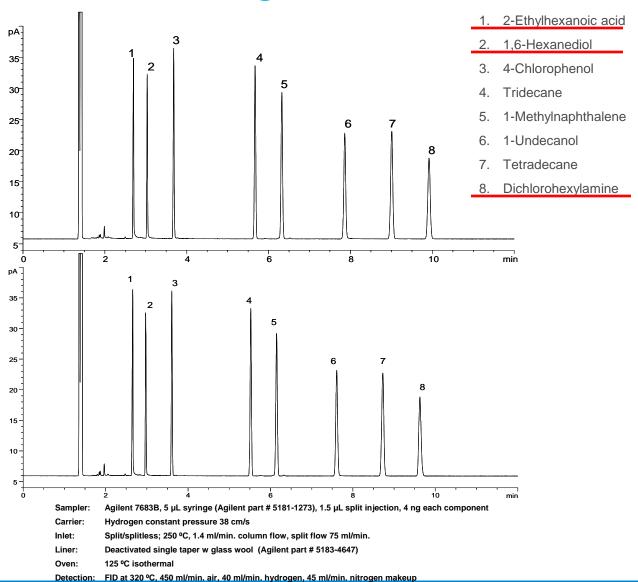


DB-5ms Test Mix – QC Testing of the 90s

Agilent J&W DB-5ms Ultra Inert 30m x 0.25mm x 0.25um (P/N 122-5532UI)

Competitor 5ms 30m x 0.25mm x 0.25um

More Probative





Ultra Inert Test Probe Mixture – QC Testing for Today's Demanding Applications

		Column	
Probe	(ng on	functional test	
	column)		
1. 1-Propionic acid	1.0	Basicity	
2 1-Octene	0.5	Polarity	
3. n-Octane	0.5	Hydrocarbon marker	
4. 4-Picoline	1.0	Acidity	
5. n-Nonane	1.0	Hydrocarbon marker	
Trimethyl phosphate	1.0	Acidity	
7. 1,2-Pentanediol	1.0	Silanol	
8. n-Propylbenzene	1.0	Hydrocarbon marker	
9. 1-Heptanol	1.0	Silanol	
10. 3-Octanone	1.0	Polarity	
11. n-Decane	1.0	Hydrocarbon marker	

Sampler: Agilent 7683B, 0.5 µL syringe (Agilent part # 5188-5246), 0.02 µL split injection

Carrier: Hydrogen constant pressure, 38 cm/s

Inlet: Split/splitless; 250 °C, 1.4 ml/min. column flow, split flow 900 ml/min., gas saver flow 75 ml/min. on at

2.0 min.

Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)

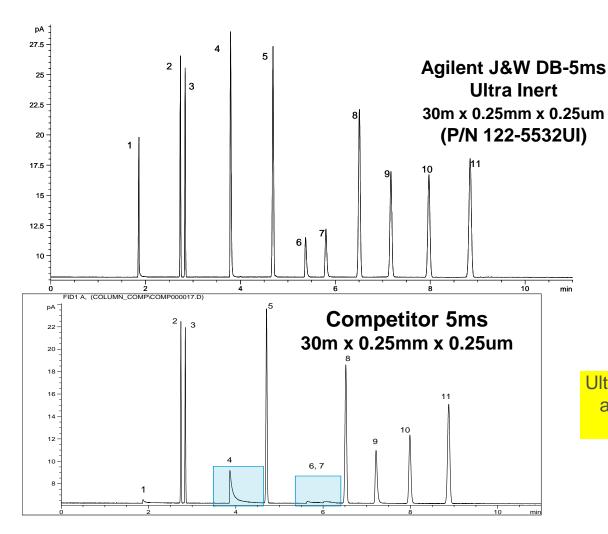
Oven: 65 °C isothermal

Detection: FID at 325 °C. 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min., nitrogen makeup



Column

Ultra Inert Test Probe Mixture shows the difference

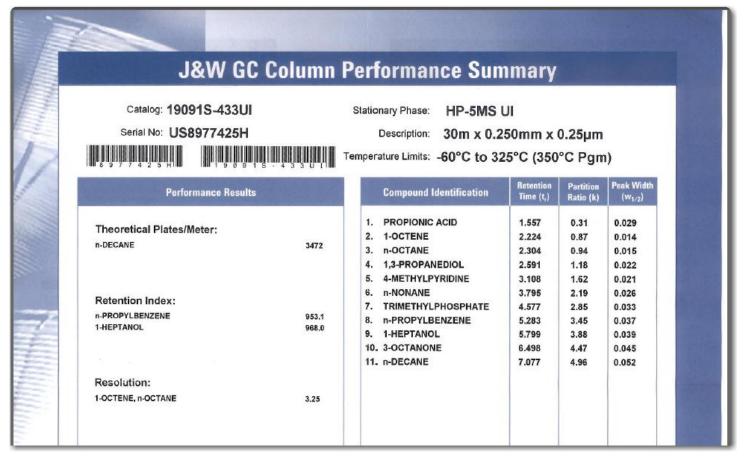


- 1. 1-Propionic acid
- 2. 1-Octene
- 3. n-Octane
- 4 4-Picoline
- 5. n-Nonane
- 6. Trimethyl phosphate
- 7. 1,2-Pentanediol
- 8. n-Propylbenzene
- 9. 1-Heptanol
- 10. 3-Octanone
- 11. n-Decane

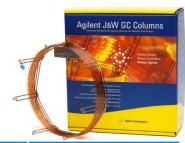
Ultra Inert test mix probes inertness and <u>differentiates an excellent</u> <u>column</u> from a mediocre one!

Ultra inert columns – Individually Tested

- Each column is individually tested against the Ultra Inert test probe mixture
- Test mixture optimized for column polarity / selectivity
- Performance Summary Sheet is shipped with each column



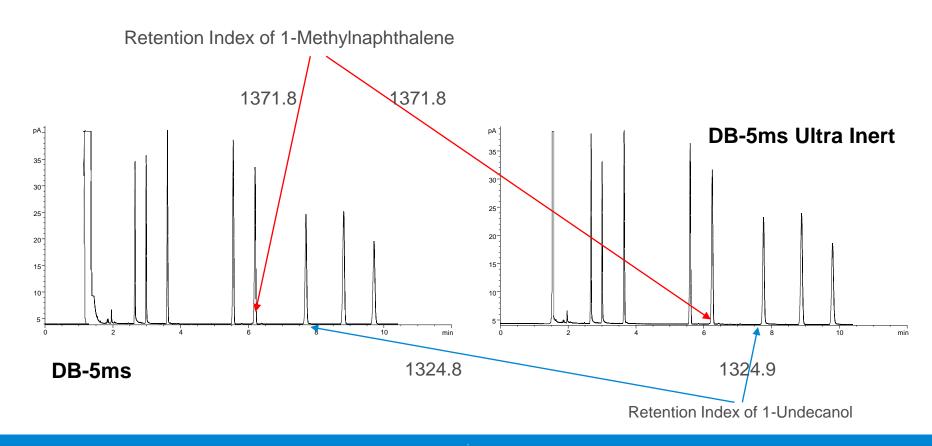
Agilent UI GC columns: chemistries



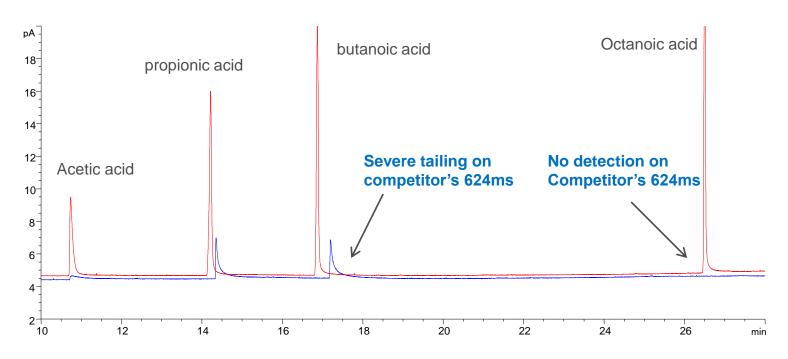
GC Column Phase	Characteristiques	Application	Configurations Fast GC
DB-1MS UI, HP-1MS UI	apolar	Classic separations, hydrocarbons	yes
DB-5MS UI, HP-5MS UI	apolar	First choice for trace analytes!	yes
DB-35MS UI	Medium polarity	Excellent for analysis of pesticides and drugs of abuse	yes
DB-624MS UI	Medium polarity	Analysis of solvents and volatiles	yes
DB-UI 8270D	Application specifique, apolar	Environmental analysis (semi-volatiles)	yes

Ultra inert columns vs. existing GC/MS columns

- Same selectivity without method re-development
- Same exceptionally low bleed
- Added inertness with additional testing procedure



Agilent J&W DB-624 UI vs. Other Guys 624ms Organic acid performance at 200 ppm



Column: Agilent J&W DB-624UI 30 m x 0.32 mm x 1.8 um vs . Alternative vendor 624ms

Oven: 35°C 7.45 min hold, 6.72°/min to100°C (2.23min hold),10.08°/min to 220°C(4.47 min hold),

16.79 °/min (4.17 min hold)

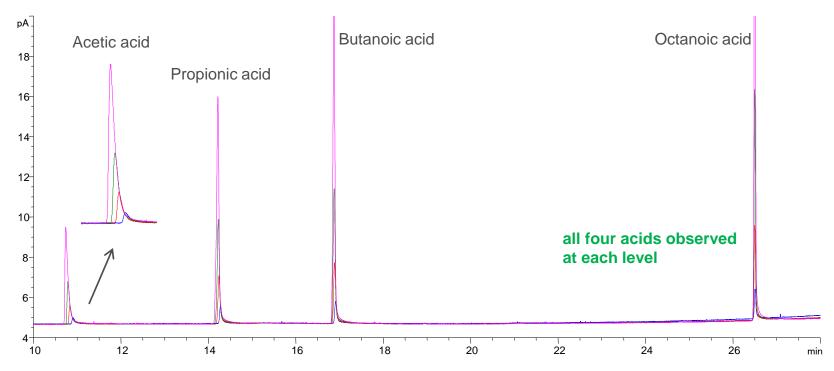
Carrier: Helium 39.6 cm/s (approx. 2.6 mL/min) set at 35°C, EPC-Constant Flow

Inlet: Split, 20:1 at 250°C (total flow approx 51 mL/min, and 11.2 psi)

Inlet liner: Ultra Inert with wool

Detector: FID at 280°C, H2 @ 40 mL/min, Air @ 400 mL/min, N2 makeup @ 30 mL/min

Agilent J&W DB-624 Ul-Organic Acid Proof of Performance 25 to 200 ppm



Column: Agilent J&W DB-624UI 30 m x 0.32 mm x 1.8 um (p/n 123-1334UI)

Oven: 35°C 7.45 min hold, 6.72°/min to100°C(2.23min hold),10.08°/min to 220°C(4.47 min hold),

16.79 °/min (4.17 min hold)

Carrier: Helium 39.6 cm/s (approx. 2.6 mL/min) set at 35°C, EPC-Constant Flow

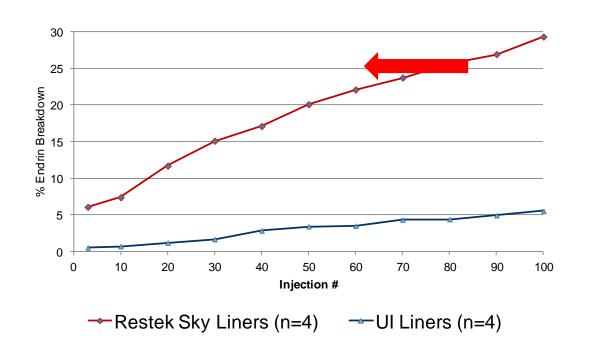
Inlet: Split, 20:1 at 250°C (total flow approx 51 mL/min, and 11.2 psi)

Inlet liner: Ultra Inert with wool

Detector: FID at 280°C, H2 @ 40 mL/min, Air @ 400 mL/min, N2 makeup @ 30 mL/min

Ultra Inert Inlet Liners

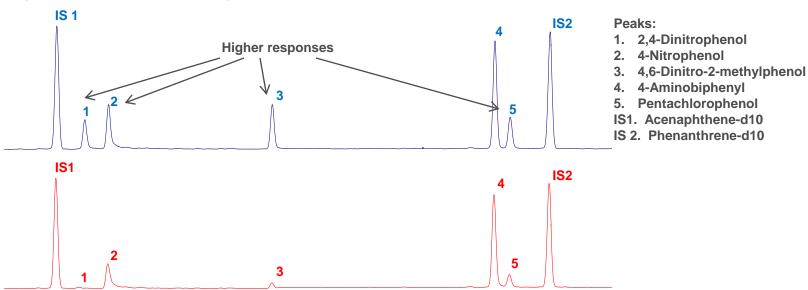
- Ultra Inert deactivated inlet liners provide higher response for sensitive compounds
- Ultra Inert Glass wool liners deliver benefits of glass wool w/o loss of active compounds
- 3. QC tested & certified for consistent performance



< 20% breakdown of Endrin after a sequence of 100 injections -- not just the first injection!

Semi-Volatiles Suitability - Ultra Inert





Competitor's deactivated gooseneck liner with deactivated wool

Even with glass wool, Agilent Ultra Inert deactivated liners provide high responses for sensitive semivolatile acidic compounds. Competitor's deactivated liners show activity and adsorption



Reliability / Quality Assurance : Ultra Inert Liner Certificate of Performance

Lot to Lot Liner Reproducibility assured:

Each deactivation lot is Certified to ensure consistent and efficient coverage using both acidic and basic probes at trace (2 ng) levels on column

Certificate with every liner is printed on a label ready to peel and stick into analysts' laboratory notebooks for easier compliance.

Traceability:

Deactivation Lot number is on Certificate Liner lot number (and part number) is permanently etched on glass

Certificate of Performance

5190-2293 Ultra Inert Liner Splitless, Sngl taper, Glass Wool

Liner Body Lot: 0023A

Deactivation Lot: B11002

Tested for: 2ng 4-Aminopyridine 2ng 2,4-Dinitrophenol

Ultra Inert liners and packaging – something for everyone

Ease of Use - exclusive "Touchless" packaging...

Plasma treated Non-Stick O-ring is preinstalled on the liner

Packaging is Pharmaceutical grade PTEG tubing approved by GCMS extraction testing for cleanliness

Install new liner with O-ring without touching – or risk contaminating – the new, clean Ultra Inert liner



Individual liner in Touchless packaging



Convenient 5 pack in Touchless package



25 pack of liners in Touchless Dispenser

Ultra Inert Liners Available for non-Agilent GC's

Cross Lab

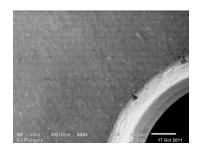
We currently support:

- Bruker, Varian*
- CTC
- PerkinElmer
- Shimadzu
- Thermo Scientific
- And more coming soon



Agilent UI Inlet Seal: Deactivated gold surface

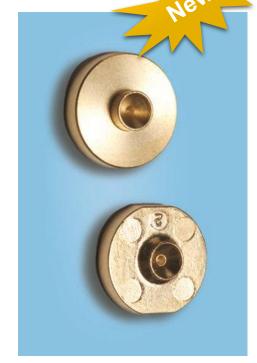
- Soft gold plating is essential for proper sealing
- Ultra Inert chemistry blocks active sites (gold is NOT inert)
- Smooth surface doesn't leak
- Advantage Agilent



Agilent MIM seal

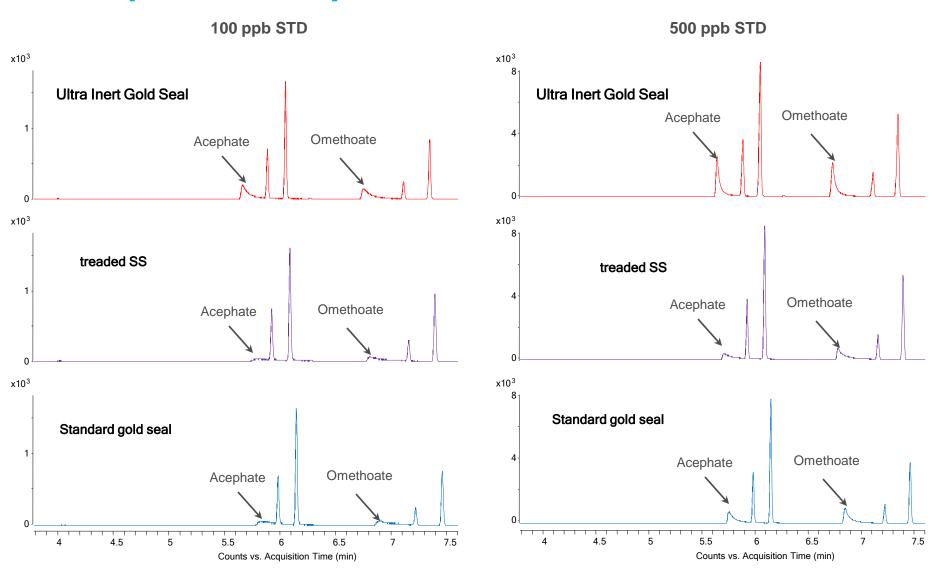


Competitor's machined seal



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

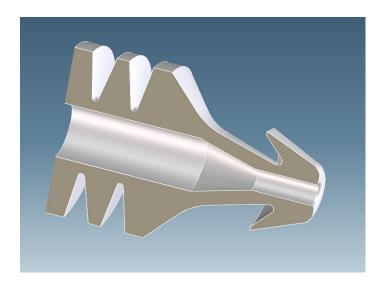
Response Comparison for Sensitive Pesticides



Ultimetal Plus Flexible Metal Ferrule

At ppb Levels, the Small Things Matter







Proprietary design offers improved sealing, easy installation (over tightening) and deactivation



Packaging promotes ease of use and system cleanliness....

- Lower detection limits
 - Faster maintenance

Intermezzo: capillary column nuts

Column nuts are determined by the instrument fitting

Basic mechanical fittings with little enhancements

- Brass for the MS
- Some finger tight designs for ease of use

New design addresses ease of use and productivity issues of leaking and over tightening



Better Connections: Capillary Flow Technology Devices

UltiMetal Plus Ultimate Union/UltiMetal Plus Tee

- Stainless Steel Micro Fluidic plates technology
- Deactivation essential to block active sites
- Column connection easy to assemble Release hole for stuck ferrules

Using Flexible Metal ferrules to overcome issues

- UltiMetal Plus surface chemistry prevents activity
- Flexible design reduces risk of over tightening or column breaks
- Leak free seal remains after repeated temperature cycles

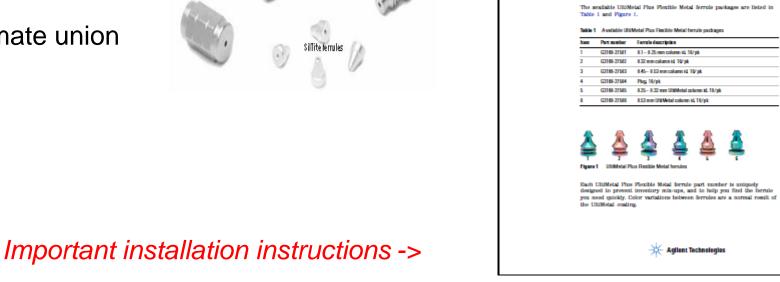


Application of Flexible Metal Ferrules

Replace stainless steel SilTite ferrules in:

- Capillary flow technology devices
 - QuickSwap
 - Backflush
- Ultimate union
- LTM



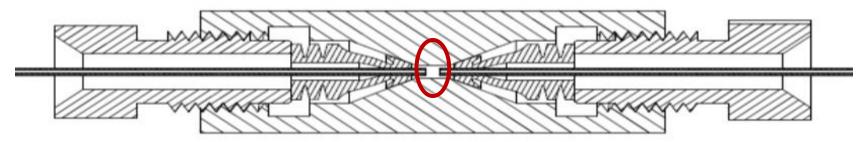


UltiMetal Plus Flexible Metal Ferrules

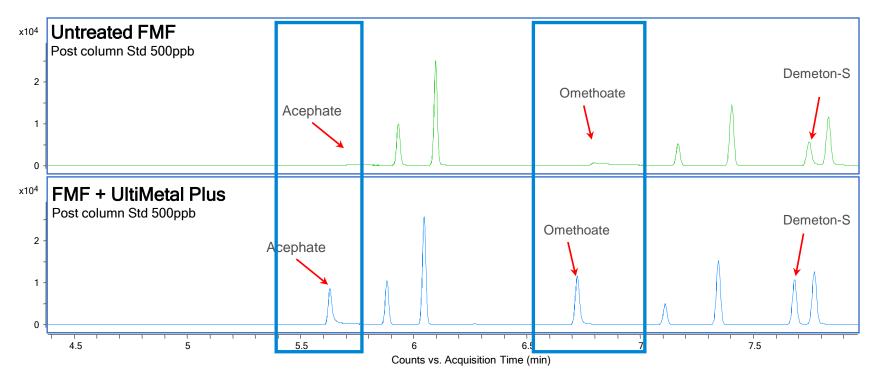
Swaqing Guide

Parts Supplied

Impact of ferrule surface on inertness



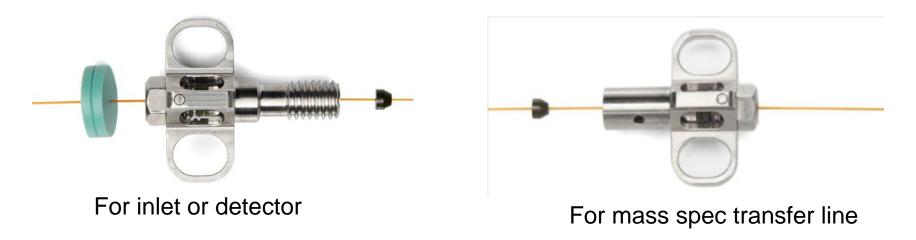
Very small amount of ferrule surface exposed to active pesticides



Better Connections: Agilent Self Tightening Column Nuts

Designed for use with short graphite/polyimide blend ferrules –

both at the inlet and the MS interface – so only one type of ferrule needed for both ends of the column!



Short ferrule exposes more thread of the fitting for better sealing

How do Self Tightening Column Nuts work?

- Ease of use install in dark, small space in GC oven without wrenches
- Wing design for finger tight installation with graphite/polyimide blend ferrules
- No tools dramatically reduces force preventing over tightening or damage
- Robust stainless steel construction

Plus....

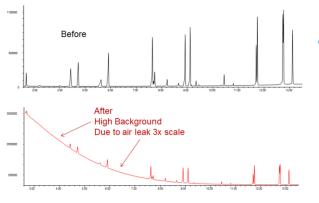
 Novel spring driven piston design that continuously presses against the ferrule

to maintain a leak-free fitting

even when the ferrule shrinks during temperature program!

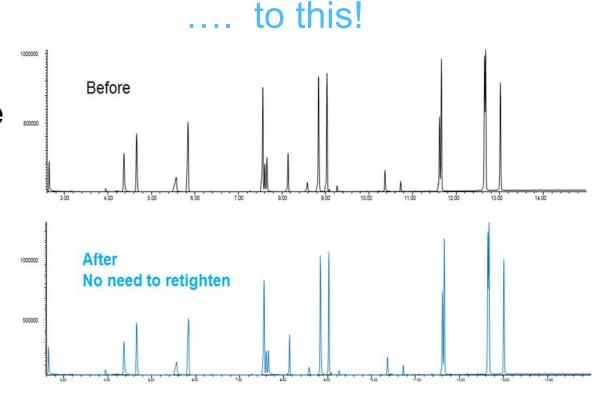


Benefit of Self Tightening Column Nuts



Take you from this....

Without retightening, the baseline remains flat after 400 runs with no indication of leaks when using the Self Tightening Column Nuts



Better Connections: Glass column connectors

Ultra Inert Press Fits

Join retention gap or guard column to analytical, or split effluent

Dependable inertness performance at a lower cost

Improved robustness, holding strength

Batch certified inertness

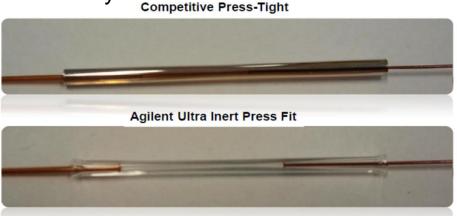
Improved packaging and installation instructions

Easier to use - transparent deactivation gives visibility of the

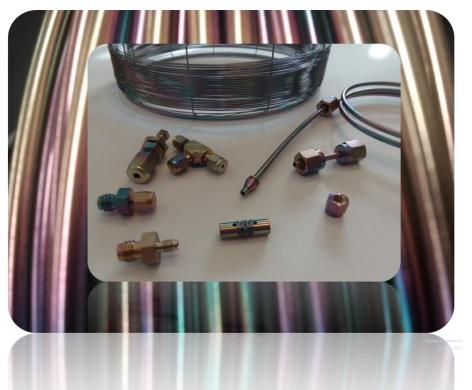
column connection







Better connections: UltiMetal Plus Tubing and Fittings



- UltiMetal Plus Deactivated metal tubing and valves
- 0.53 and 0.25 mm id guards and transfer lines
- Metal fittings (unions, tees and nuts)
- Steel tubing (1/16", 1/8", 1/4")



Ensure the entire chromatographic solution is inert and corrosion resistant to provide superior performance with improved peak shapes even for active compounds

Agilent Inert Inlet

UltiMetal Plus treatment for inert surface 7890 inlet shell & top weldments

- 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

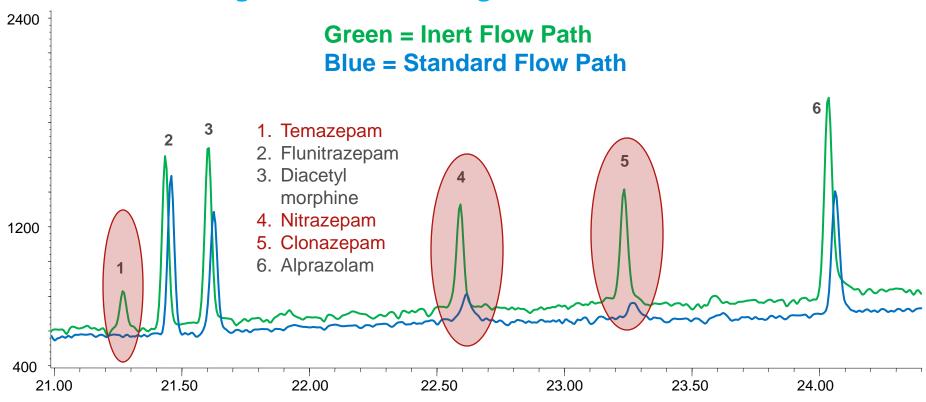


Agilent's proven proprietary Ultimetal Plus surface treatment

Putting It All Together — Agilent Inert Flowpath

Dramatic Improvement at Low Levels

Drugs of Abuse 0.25 ng Column SIM Mode

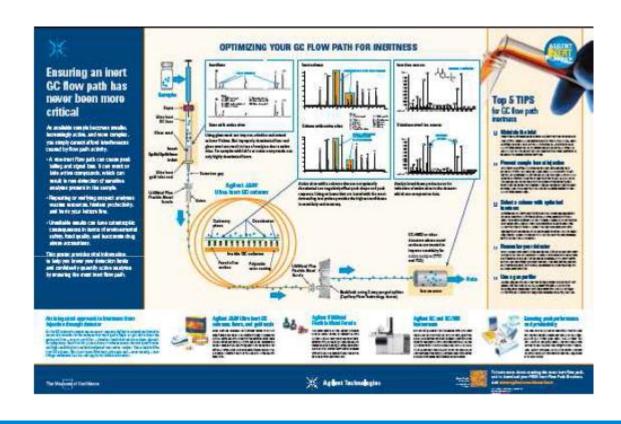


Summary: inert and clean flow components improve results in today's chromatography laboratory.

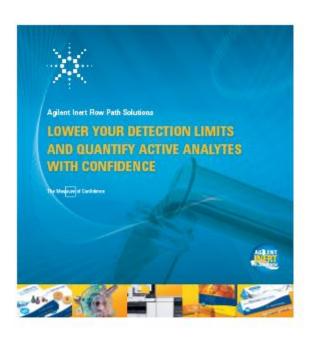
- Less adsorption of analytes results in improved detection limits from increased response (s/n) and better precision
- Improved peak shape at low concentration improves resolution and peak integration
- Higher response per unit concentration results in more injections before required system maintenance
- Less chemical noise from clean packaging, touchless packaging and pre-conditioned consumables means less "conditioning" time and increased productivity

Literature and ordering guides: Inert Flowpath

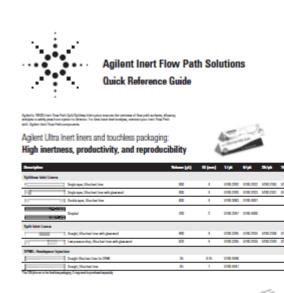
www.agilent.com/chem/inert



Literature and ordering guides: Inert Flowpath







Agilent Ultra Inert gold seals and washers:

A smooth, leak-free surface for active analytes





Learn more at www.agillert.com/chem/altrainert





Literature and ordering guides: GC connections

www.agilent.com/chem/betterGCconnections

Order the poster...

View the video...





Instrumentation



Inert Flow Path



Questions?

