Improvement in the EPA 8270 Active Semivolatiles Analysis by GC/FID Using Agilent Ultra Inert Liners

Limian Zhao[1], David Mao[2], Mitch Hastings[2], Alan D Broske[1], Allen Vickers[2] and Lindy Miller[1]   Agilent Technologies Inc., [1] 2850 Centerville Road, Wilmington, DE 19808 USA; [2] 91 Blue Ravin Rd., Folsom, CA, 95630

Introduction

In order to achieve reliable and solid results for the active compounds, it is critical to minimize the interaction of active analytes along the GC flow path starting with the injector, to the column and finally to the detector. The inlet liner plays a key role in influencing the inertness of the GC flow path. The adsorption of sensitive analytes on active sites can cause bad peak shape, loss on sensitivity, and poor linearity of calibration curves. Therefore it is critical to deactivate the inlet liner completely to make it fully inert.

The analysis of active semi-volatile compounds in USEPA Method 8270 has always been a challenge for the GC flow path inertness. Samples contain a mix of acids, bases and neutrals, which can interact with the surface of sample flow path, and the inlet liner can be a significant contributor to system activity.

Agilent's Ultra Inert liner deactivation process significantly improves the efficiency and robustness of inlet liner, especially glass wool deactivation. The liner body together with large glass wool surface area can be deactivated thoroughly. The new deactivation technique enables the use of Ultra Inert deactivated liners with and without wool for the accurate and reliable analysis of EPA 8270 sensitive semi-volatile organic compounds.

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

Experimental

In order to ensure maximally that the evaluation focuses on the liner deactivation efficiency, we purposely used

- FID as the detector to eliminate any activity contributed from the mass spectrometer.
- Ultra Inert column to minimize the column activity.
- Splitless injection mode and single taper splitless liner configuration for its wide usage in practice .
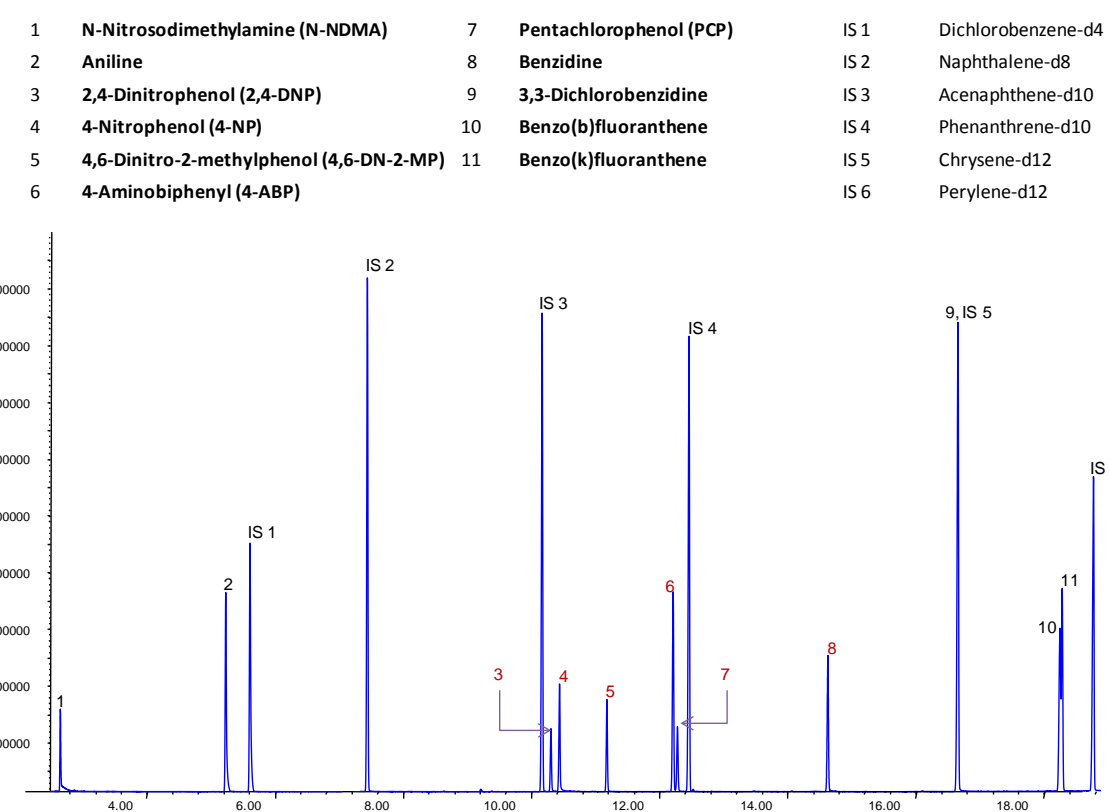- A new gold seal each liner test.

Instrument conditions

- Agilent GC/FID system, equipped with Agilent 7683B Autosampler
- S/SL inlet under splitless mode @ 250°C,
- purged flow 50 mL/min at 0.75 min for splitless liners w/o wool, and 1.5 min for splitless liners w/ wool
- 1 µL injection volume
- Inlet liner: Ultra Inert splitless liner w/o wool
  (Agilent 5190-2292)
  Ultra Inert splitless liner w/ wool
  (Agilent 5190-2293)
- He, constant flow @ 3 mL/min
- Analytical column: Ultra 2, 25m x 0.32mm, 0.52µm (Agilent p/n 19091B-112)
- Over profile: 40°C (1min), 15°C/min to 310°C (0min)
- Detector: FID, 250°C, H_2/Air/Makeup N_2: 40/450/45 mL/min

Testing standards

- EPA 8270 custom standard @ 2000 µg/mL mixture in methylene chloride, containing 11 active semi-volatile organic compounds.
- A series calibration standards at 2, 5, 20, 40 and 80 µg/mL in methylene chloride.
- The 8270 semi-volatile internal standard (IS) at 4000 µg/mL in methylene chloride. 40 µg/mL IS in calibration standards.

GC-FID of 11 Active Semi-volatiles Test Mix

1	N-Nitrosodimethylamine (N-NDMA)	7	Pentachlorophenol (PCP)	IS 1	Dichlorobenzene-d4
2	Aniline	8	Benzidine	IS 2	Naphthalene-d8
3	2,4-Dinitrophenol (2,4-DNP)	9	3,3-Dichlorobenzidine	IS 3	Acenaphthene-d10
4	4-Nitrophenol (4-NP)	10	Benzo(b)fluoranthene	IS 4	Phenanthrene-d10
5	4,6-Dinitro-2-methylphenol (4,6-DN-2-MP)	11	Benzo(k)fluoranthene	IS 5	Chrysene-d12
6	4-Aminobiphenyl (4-ABP)			IS 6	Perylene-d12

Results and Discussion

### Satisfactory linearity for active semi-volatiles by Agilent Ultra Inert liners w/ and w/o wool

Active semi-volatile analytes RFs & RSD over 2-80 ng on column
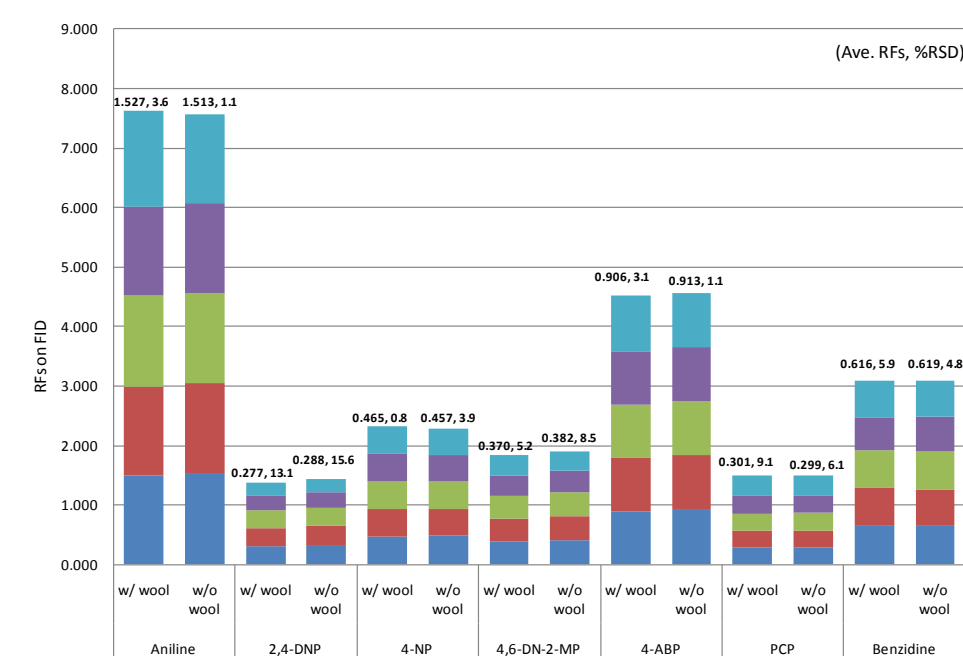
Agilent Ultra Inert liners w/ and w/o wool provide equivalent inertness for quantitative analysis of active semi-volatile analytes.
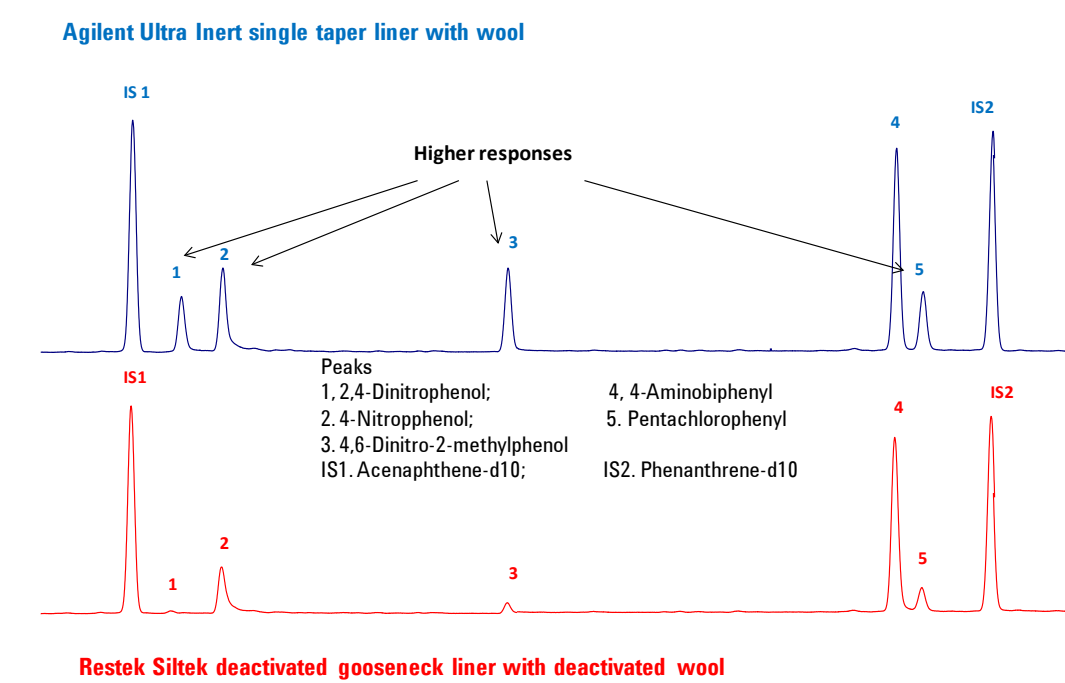
Agilent Ultra Inert liners provide higher response for critical semi-volatiles than other equivalent liners

2,4- DNP RF for single taper SL liner w/o wool
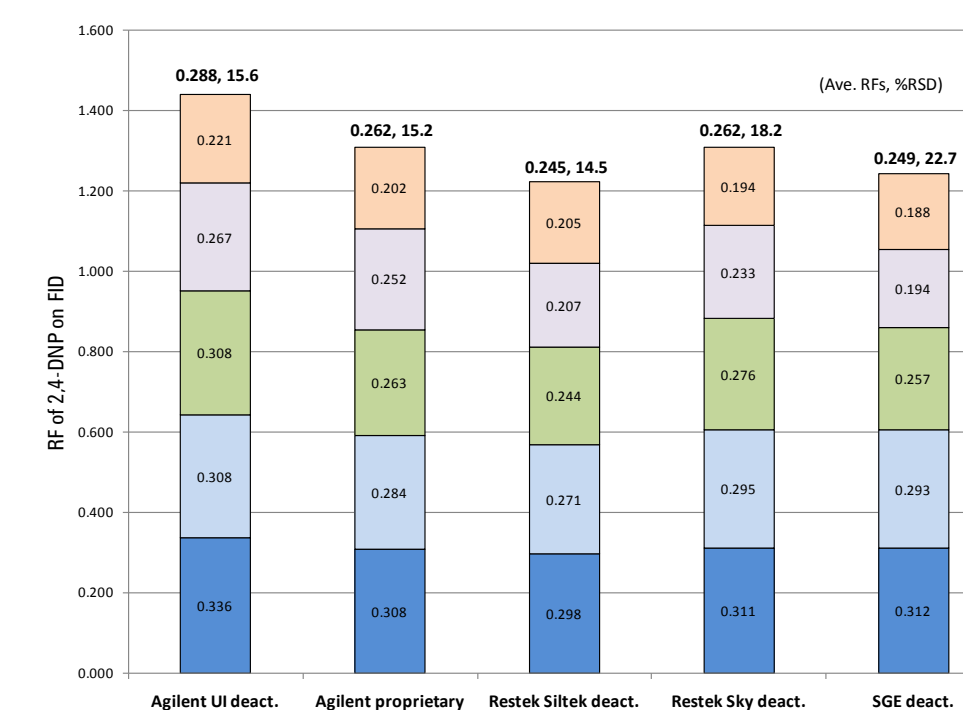
Agilent Ultra Inert liners w/o wool give higher average RF and better RSD for the most active analyte, 2,4-Dinitrophenol (2,4-DNP), over the calibration range of 2-80 ng on column

Active semi-volatile chromatograms using Ultra Inert liner with wool and Siltek liner with wool by GC/FID, 2 ng on column

Agilent Ultra Inert single taper liner with wool

Peaks
1,2,4-Dinitrophenol;   4, 4-Aminobiphenyl
2. 4-Nitrophenol;   5. Pentachlorophenyl
3. 4,6-Dinitro-2-methylphenol
IS1. Acenaphthene-d10;   IS2. Phenanthrene-d10

Restek Siltek deactivated gooseneck liner with deactivated wool

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

Excellent liner to liner reproducibility achieved by Agilent Ultra Inert liners

**Average RF and RSD over calibration range of 2 – 80 on column**

Active Semi-volatiles (Peak #)	Ultra Inert liners w/o wool (Agilent p/n 5190-2292)		Ultra Inert liners w/ wool (Agilent p/n 5190-2293)	
	Ave RF (# liners = 6)	RSD (%) (# liners =6)	Ave RF (# liners = 6)	RSD (%) (# liners =6)
N-NDMA (1)	0.559	3.1	0.569	1.3
Aniline (2)	1.539	1.9	1.527	1.3
2,4-DNP (3)	0.278	4.8	0.277	2.4
4-NP (4)	0.464	2.1	0.475	4.7
4,6-DN-2-MP (5)	0.377	1.8	0.370	3.4
4-ABP (6)	0.925	1.4	0.908	1.4
PCP (7)	0.307	3.0	0.306	5.3
Benzidine	0.617	2.3	0.617	4.0
Benzo[b]fluoranthene (10)	0.947	3.7	1.082	6.0
Benzo[k]fluoranthene (11)	0.912	3.7	0.973	1.8

Lot to Lot Liner Reproducibility assured: Each lot is Certified to ensure consistent and efficient deactivation using both acidic and basic probes at trace (2 ng) levels on column
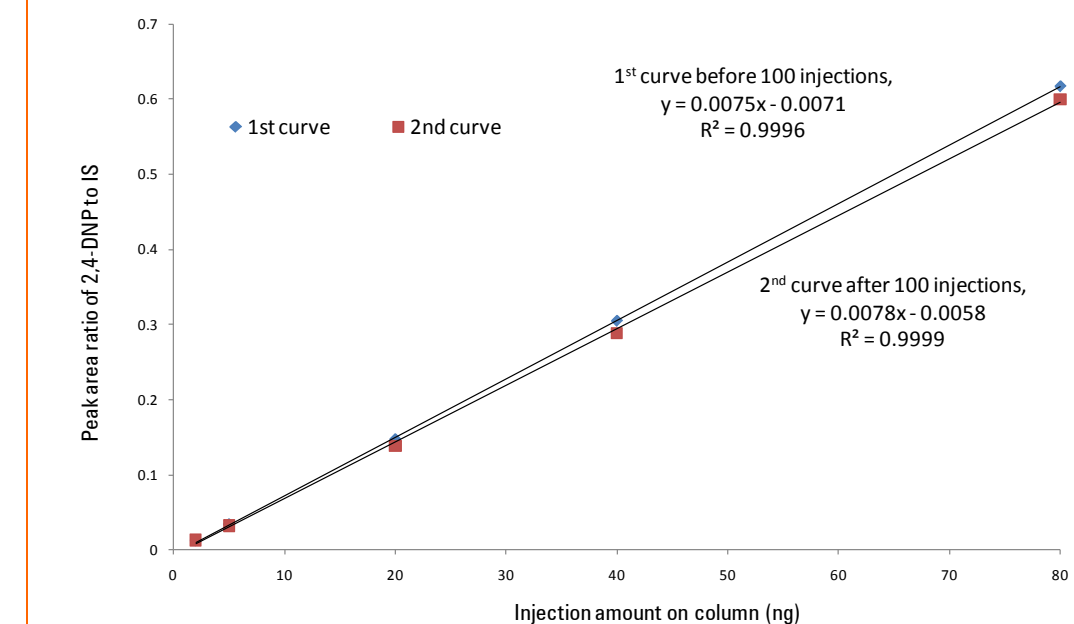
Results and Discussion

Agilent Ultra Inert liners provide superior liner durability and injection repeatability

Injections repeatability (n=100) using Ultra Inert liners w/wool by GC/FID, 2 ng on column
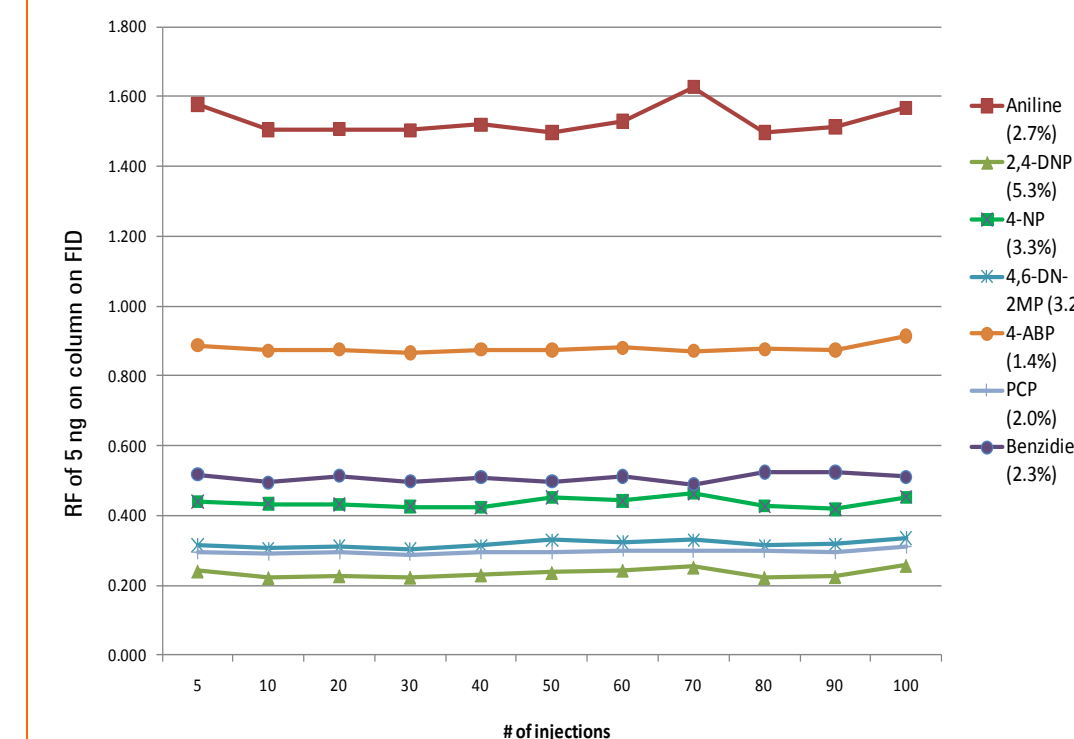
The injections repeatability test (n=100) was conducted using Agilent Ultra Inert deactivated liner w/ wool (P/N 5190-2293). Results show that less than 6% RSD achieved for all of active 8270 analytes over 100 injections with 5ng on column.

2, 4-DNP calibration curves before and after 100 injections using Agilent Ultra Inert liner w/ wool

1st curve before 100 injections,
y = 0.0075x - 0.0071
R^2 = 0.9996

2nd curve after 100 injections,
y = 0.0078x - 0.0058
R^2 = 0.9999

Calibration standards were run before and after 100 injections, and tight calibration curves were obtained, even for the most active analyte, 2,4-DNP. Excellent liner durability also assures the reliable calibration over multi-injections.

Excellent thermal stability obtained by Agilent Ultra Inert Liners

Thermal stability @ 330°C over 10 days using Ultra Inert liners w/wool by GC/FID, 5 ng on column
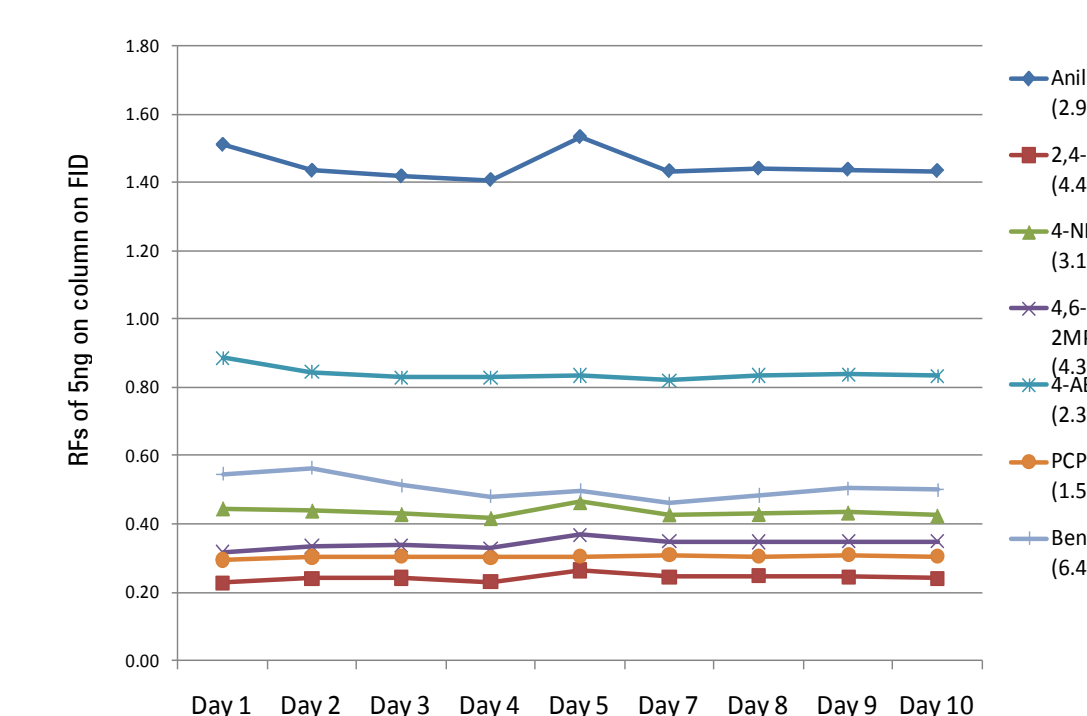
10-day thermal stability test @ 330°C using Agilent Ultra Inert liner w/ wool. Less than 7% RSD achieved for all of active semi-volatiles with 5 ng on column.

Conclusions

Agilent Ultra Inert splitless liners w/ and w/o wool provide following benefits for accurate and reliable analysis of active EPA 8270 semi-volatile analytes

- Excellent inertness for the analysis of active compounds
- Superior linearity for active semi-volatiles over 2 – 80 ng on column by GC/FID
- Consistent liner to liner (lot to lot) reproducibility
- Excellent liner durability, injections repeatability and thermal stability
- Equivalent or superior to other equivalent liners or popular used liners in the above applications

To learn more about Agilent Ultra Inert liners, visit us online at www.Agilent.com/chem/UltraInert

Please leave your business card to receive a reprint of this poster