Application of Agilent Ultra Inert Liners with Wool for the Analysis of Active Analytes by GC/MS and GC/MS/MS

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Introduction

GC inlet liners with wool provide many benefits during sample introduction to the GC column
- Aid the vaporization of liquid sample
- Promote homogeneous sample mixing and better quantitation
- Trap the non-volatile residues in sample matrix thus protect column and MS source from contamination
- Prevent sample from hitting the bottom of the inlet before vaporization.

However, the use of liners with wool has been limited for the analysis of active compounds such as pesticides, drugs of abuse, due to
- Inefficient deactivation of the large glass wool surface area using traditional deactivation processes
- High variations of inertness performance as well as rapid degradation of inertness

Agilent's Ultra Inert liner deactivation process significantly improves the efficiency and robustness of glass wool deactivation, allowing the use of Ultra Inert liners with wool for
- Quantitative analysis of pesticides;
- Screening of drugs of abuse

The feasibility evaluation tests include
- Chromatographic quality: peak shape and intensity of active compounds
- Liner to liner reproducibility
- Calibration curve linearity for quantitative analysis
- Injections repeatability and performance stability
- Column lifetime and MS source contamination
- Comparison to other equivalent liners or popular used liners

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

Certificate of Performance

Experimental

Screening of drugs of abuse by GC/MS

Instrument conditions
- Agilent 7890A/5975C GC/MSD system, equipped with Agilent 7683B Autosampler
- S/SL inlet under splitless mode @ 280°C, 1 µL injection volume
- Inlet liner: Ultra Inert single taper splitless liner with wool (Agilent 5190-2293)
- He, constant pressure, RT locked with SKF-525a @ 8.569 min
- DB-5MS UI, 15m x 0.25mm, 0.25µm (Agilent p/n 122-5512UI)
- Purged Ultimate Union (Agilent p/n G3182-61580) for back flushing
- Restrictor: Inert Fused Silica tubing, 0.65m × 0.15mm (Agilent p/n 160-7625-5)
- Oven profile: 100 °C (0.5 min), 20 °C/min to 325 °C (2.5min)
- Postrun back flush: 2 min @ 325°C , back flush pressure @ 75psi, inlet pressure @ 1psi during back flush
- MSD: Transfer line @ 300°C, Source @ 300°C, Quad @ 150°C
- Data acquisition: Scan/SIM

Testing standards
- Agilent GC/MS Forensic/Toxicology analyzer checkout mixture standard (p/n 5190-0471)
- 28 popular and difficult drugs of abuse, 5 µg/mL and 500 ng/mL

Matrix sample
- Whole blood samples extracted by a single step liquid-liquid extraction. (supplied by NMS labs, Willow Grove, PA)

Analysis of pesticides by GC/MS/MS

Instrument conditions
- Agilent 7890A/7000 GC/MS/MS system, equipped with Agilent 7693 Autosampler
- MMI Multi Mode inlet under pulsed splitless mode @ 280°C, 1 µL injection
- Inlet liner: Ultra Inert single taper splitless liner with wool (Agilent p/n 5190-2293)
- He, constant pressure, RT locked with Chlorpyrifos methyl @ 8.298 min
- HP-5MS UI, 15m x 0.25mm, 0.25µm (Agilent p/n 19091-431UI)
- Purged Ultimate Union (Agilent p/n G3182-61580) for back flushing
- Restrictor: Inert Fused Silica tubing, 0.65m × 0.15mm (Agilent p/n 160-7625-5)
- Oven profile: 100 °C (2 min), 50 °C/min to 150 °C, 6°C/min to 200°C at, 16°C/min to 280°C (6 min)
- Postrun back flush: 2 min @ 280°C , back flush pressure @ 75psi, inlet pressure @ 1psi during back flush
- Transfer line/Source/Quad (Q1 &Q2) @ 280°C/300°C/ 150°C
- Data acquisition: MRM

Testing standards
- 33 representative pesticides, 5 ng/mL to 500 ng/mL six points calibration standards, 50 ng/mL QC
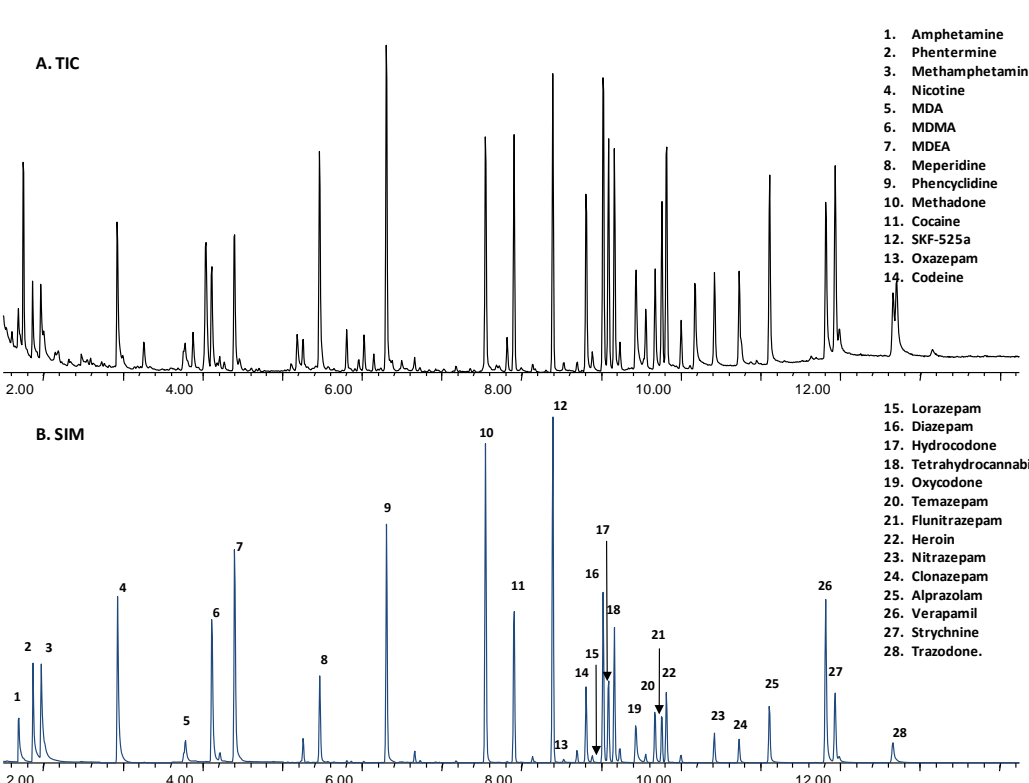
Matrix sample
- Five different fruits and vegetables matrix mixture: flower, banana, strawberry, pear, and lettuce
- Blank matrices sample were extracted following QuEChERS AOAC method
- Agilent BondElut QuEChERS AOAC extraction (p/n 5982-5755) and dispersive SPE kit for general fruits and vegetables (p/n 5982-5022) were used
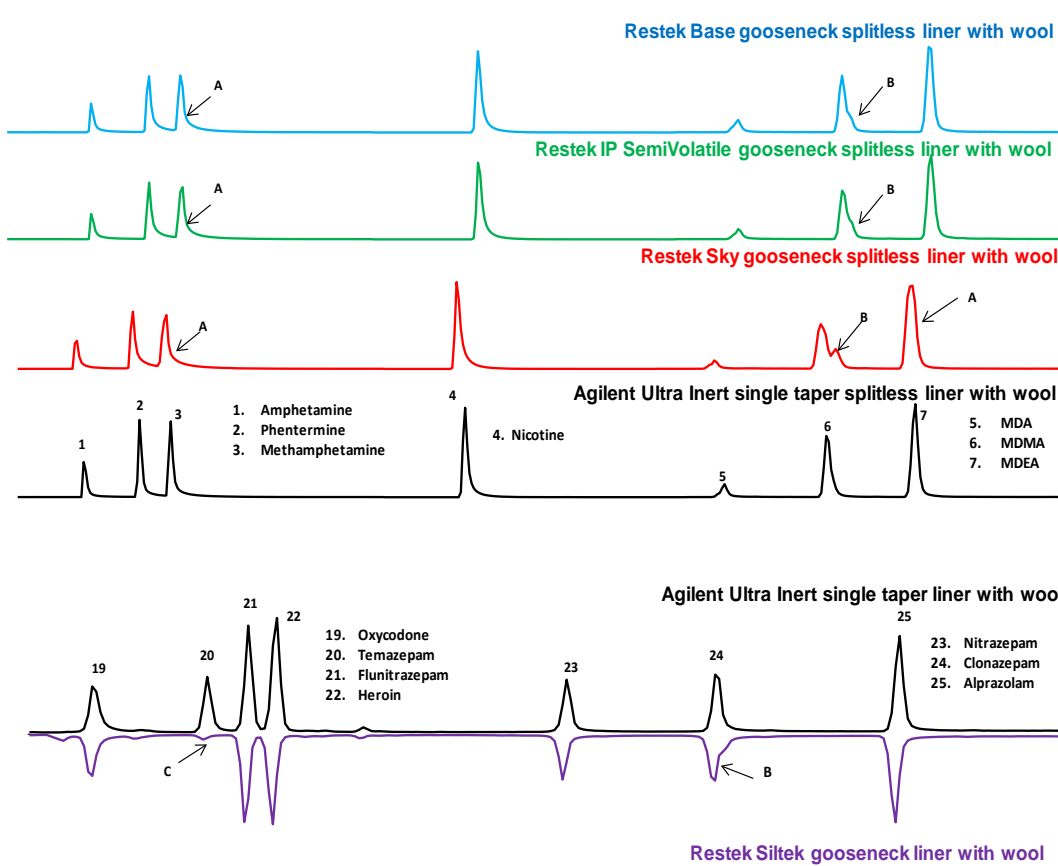
Results and Discussion

Agilent Ultra Inert liners with wool provide satisfactory GC chromatograms for active compounds in complicate matrices

GC/MS chromatograms of 2 µg/mL of drugs of abuse spiked in whole blood extract

1. Amphetamine
2. Phentermine
3. Methamphetamine
4. Nicotine
5. MDA
6. MDMA
7. MDEA
8. Meperidine
9. Phencyclidine
10. Methadone
11. Cocaine
12. SKF-525a
13. Oxazepam
14. Codeine
15. Lorazepam
16. Diazepam
17. Hydrocodone
18. Tetrahydrocannabinol
19. Oxycodone
20. Temazepam
21. Flunitrazepam
22. Heroin
23. Nitrazepam
24. Clonazepam
25. Alprazolam
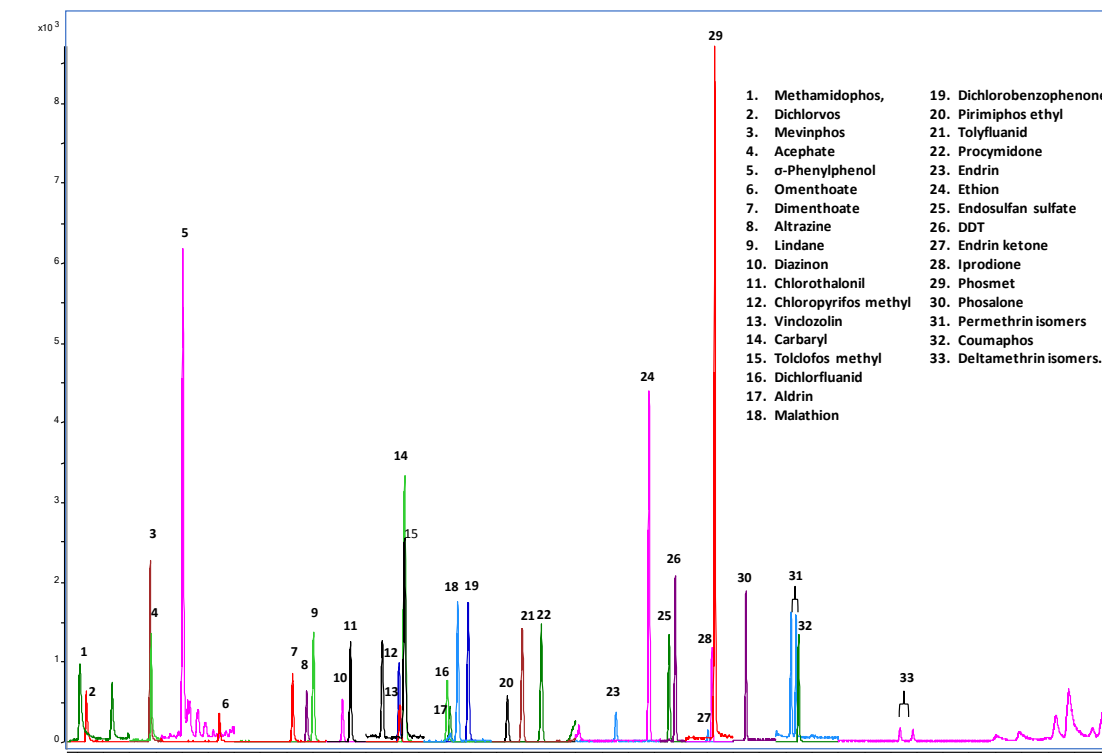26. Verapamil
27. Strychnine
28. Trazodone.

Active site on inlet liners may cause various chromatographic problems, as observed in liners tests using the checkout standard with other equivalent liners. Agilent Ultra Inert liners with wool show better peak shape and typically higher responses for all of tested analytes, especially for the most active compounds.

Restek Base gooseneck splitless liner with wool
Restek IP SemiVolatile gooseneck splitless liner with wool
Restek Sky gooseneck splitless liner with wool
Agilent Ultra Inert single taper splitless liner with wool

1. Amphetamine
2. Phentermine
3. Methamphetamine
4. Nicotine
5. MDA
6. MDMA
7. MDEA

Agilent Ultra Inert single taper liner with wool

19. Oxycodone
20. Temazepam
21. Flunitrazepam
22. Heroin
23. Nitrazepam
24. Clonazepam
25. Alprazolam

Restek Siltek gooseneck liner with wool

5ng checkout standards on column. A) Broad or distorted peak, B) ghosting shoulder, C) poor sensitivity.

GC/MS/MS chromatogram (MRM) of 10 ng/mL of pesticides in fruits and vegetables extract.

1. Methamidophos
2. Dichlorvos
3. Mevinphos
4. Acephate
5. o-Phenylphenol
6. Omenthoate
7. Dimenthoate
8. Altraine
9. Lindane
10. Diazinon
11. Chlorothalonil
12. Chlorpyrifos methyl
13. Vinclozolin
14. Carbaryl
15. Toldofos methyl
16. Dichlorfluanid
17. Aldrin
18. Malathion
19. Dichlorobenzaphenone
20. Primiphos ethyl
21. Tolylfluanid
22. Procymidone
23. Endrin
24. Ethion
25. Endosulfan sulfate
26. DDT
27. Endrin ketone
28. Iprodione
29. Phosmet
30. Phosalone
31. Permethrin isomers
32. Coumaphos
33. Deltamethrin isomers.

Excellent liner to liner reproducibility achieved by Agilent Ultra Inert liners with wool

The liner to liner performance reproducibility was tested with replicates of six Ultra Inert liners with wool from three different lots for both drugs of abuse and pesticides applications. Groups of 12 difficult active drugs of abuse and 12 pesticides were selected as representative analytes.

Selected Active Drugs of Abuse (Peak # for drugs)	RSD of average RF (Two concentrations: 5 µg/mL and 500 ng/mL) # UI liners = 6 (3 lots)	Selected Active Pesticides (Peak # for pesticides)	RSD of average RF (Calibration curve over 5 ng/mL to 500 ng/mL) # UI liners = 6 (3 lots)
Methamphetamine (3)	3.8	Methamidophos (1)	14.2
MDMA (6)	4.4	Acephate (4)	5.6
Phencyclidine (9)	2.5	Omenthoate (6)	6.7
Cocaine (11)	2.0	Dimenthoate (7)	2.4
Oxazepam (13)	7.6	Chlorothalonil (11)	8.4
Codeine (14)	4.2	Carbaryl (14)	5.1
Oxycodone (19)	6.5	Dichlorfluanid (16)	4.7
Temazepam (20)	11.7	Tolylfluanid (21)	4.9
Heroin (22)	2.7	Endrin (23)	5.7
Nitrazepam (23)	6.3	DDT (26)	17.4
Clonazepam (24)	3.5	Iprodione (28)	9.8
Trazodone (28)	4.4	Phosmet (29)	7.0

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

Ultra Inert liners with wool generate higher responses for active compounds

Temazepam is a very difficult compound and extremely sensitive to the liner inertness. When an inefficient deactivated liner was used, the response of Temazepam (5 ng on column) can almost disappear. Compared to other similar liners, Agilent Ultra Inert liner with wool generated highest RF for Tempazepam.
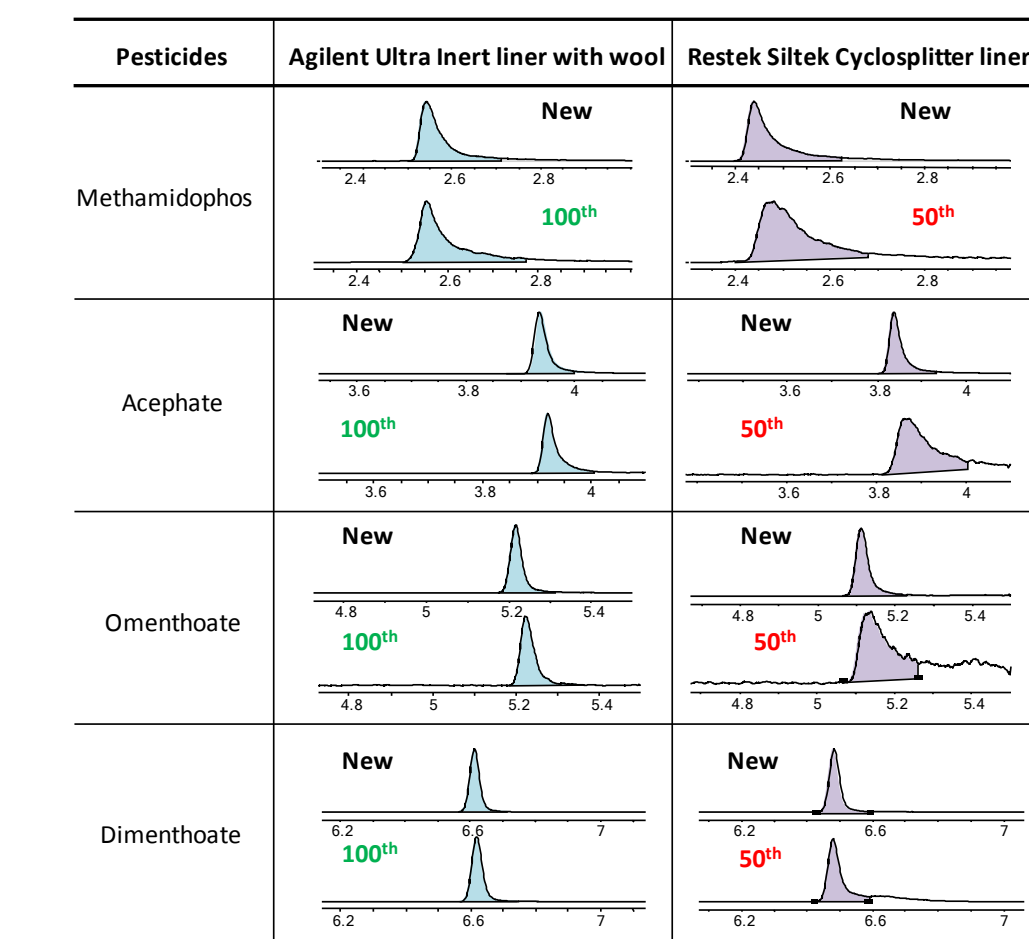
Temazepam Response Comparison between UI liners and other equivalent liners
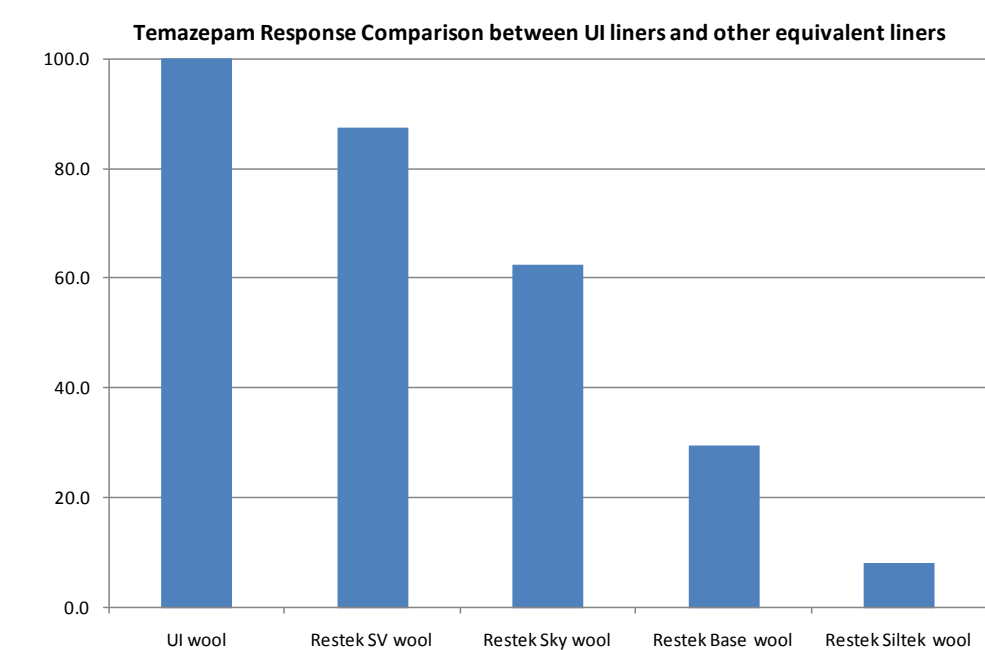
Better liner durability and injection repeatability obtained by Agilent Ultra Inert liners with wool

Better consistency on the active compounds responses

Pesticides (peak #)	Repeatability: RSD (%) for analytes' RF values of 50 ng/mL standard injections			
	In reagent solvent Ultra Inert liners w/ wool by 100 injections (# liners = 5)	In fruits and vegetables QuEChERS extract		
		Ultra Inert liners w/ wool by 50 injections (# liners = 7)	Ultra Inert liners w/ wool by 100 injections (# liners = 7)	Siltek Cyclosplitter liners by 50 injections (# liners = 3)
Methamidophos (1)	7.7	5.1	11.7	25.4
Acephate (4)	5.4	16.6	30.1	55.6
Omenthoate (6)	10.5	27.1	44.8	49.9
Dimenthoate (7)	4.8	7.3	13.4	14.9
Chlorothalonil (11)	8.6	9.3	15.2	11.7
Carbaryl (14)	8.7	11.7	19.9	13.4
Dichlorfluanid (16)	5.3	6.3	11.8	3.9
Tolylfluanid (21)	7.3	7.3	13.4	5.7
Endrin (23)	6.6	3.2	6.0	2.7
Endosulfan sulfate (25)	7.7	9.5	14.1	10.5
DDT (26)	6.3	23.1	36.4	16.8
Endrin ketone (27)	7.3	9.3	14.2	9.4
Iprodione (28)	5.1	5.0	8.1	20.3
Phosmet (29)	6.9	15.4	27.2	16.3

Better peak shape consistency for very active compounds

Pesticides	Agilent Ultra Inert liner with wool	Restek Siltek Cyclosplitter liner
Methamidophos		
Acephate		
Omenthoate		
Dimenthoate		

Tested samples were 50 ng/mL pesticides spiked in fruits and vegetables bank extract.

Conclusions

Ultra Inert liners with wool provide the following benefits for drugs of abuse screening and pesticides analysis applications in real matrices.

- Excellent inertness for the analysis of active compounds
- Better peak shape and higher peak intensity for active compounds
- Superior linearity for active pesticides over 5 – 500 ng/mL in QuEChERS extract
- Consistent liner to liner (lot to lot) reproducibility
- Acceptable liner durability and performance stability
- Extended analytical column life-time
- Decreased MS source maintenance frequency
- Superior to other popular liners used for the above applications.

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