New Agilent J&W Ultra Inert Capillary GC Columns

Raising the Bar for **CONSISTENT** Column Inertness Performance

Trace Level Analysis Made Routine

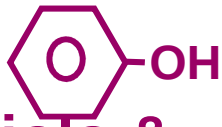
Outline

- What is inertness?
- Evolution of capillary column QC Testing
- Value of Consistent Column Inertness and Exceptionally Low Bleed
- Application examples
- Take away messages

GC System Inertness

What do we mean?

Problems with poor inertness usually limited to "active" solutes.

For example:
Alcohols & Diols (-OH), Phenols ( **),**
Amines (-NH3), Acids (COOH), Thiols &
Sulfur in general like to tail.

Thermally labile and structurally "strained" solutes will breakdown or rearrange, e.g., DDT, Endrin, Carbamates, Nitroglycerines.

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

GC System Inertness
What do we mean?

Problems with poor inertness usually limited to "active" solutes.

Tailing or breakdown of "benign" solutes is symptomatic of a more generalized system problem, usually related to gross contamination.

Possible Inertness Problem Areas

Inlet

- liner, liner packing, gold seal, stainless steel

Consumables

- septa, syringe, vial, caps, inserts, solvents

Column

GC Detector

- source geometry, material, column interface, acquisition rates

Temperatures

- inlet, transfer line, source, quads, oven

Other method factors i.e. samples and standards preparation

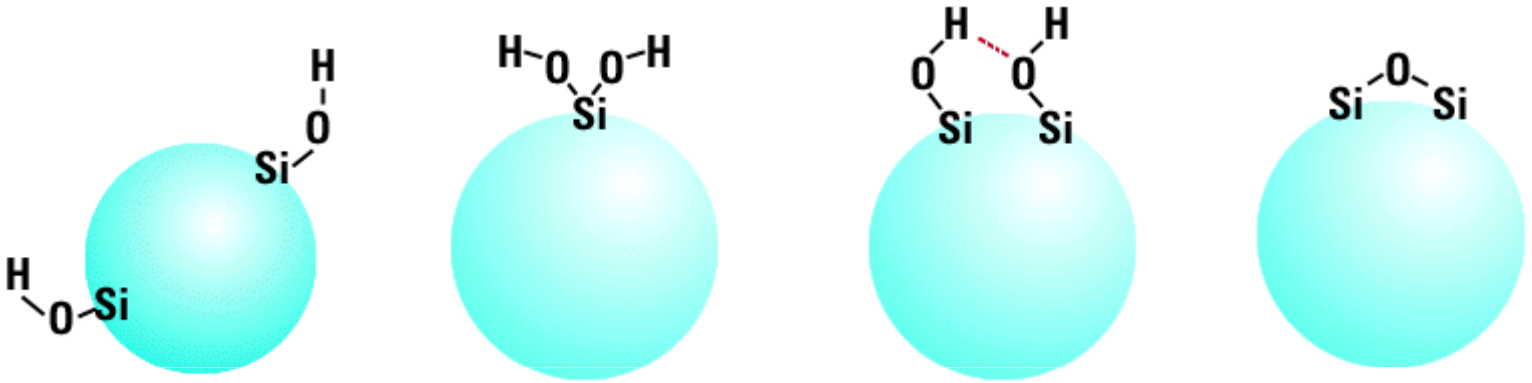
How important is Column inertness to overall Flowpath Inertness?

GC Flowpath Surface Areas

	l (cm)	d (cm)	pi	Surface Area (cm^2)
Liner	7	0.2	3.142	4.4
Seal	0.4	0.8	3.142	1.0
Column	3000	0.025	3.142	235.6

Where does column activity come from?

Isolated Geminal Vicinal Siloxane



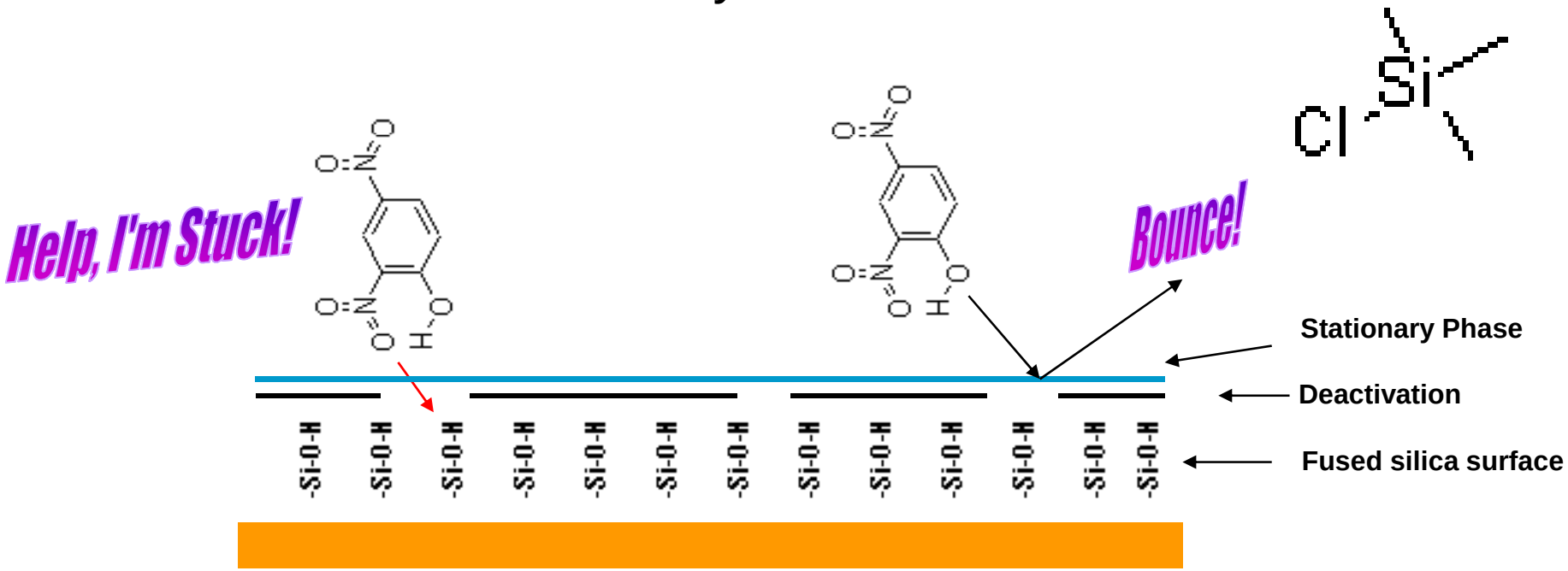
Sterically difficult to “cap” all of them—estimates 40-65% capped with traditional deactivation.

Non-traditional sources such as trace impurities in starting materials and manufacturing lines.

Traditional Deactivations

Dichlorodimethylsilane, various silizanes, etc… “endcaps”

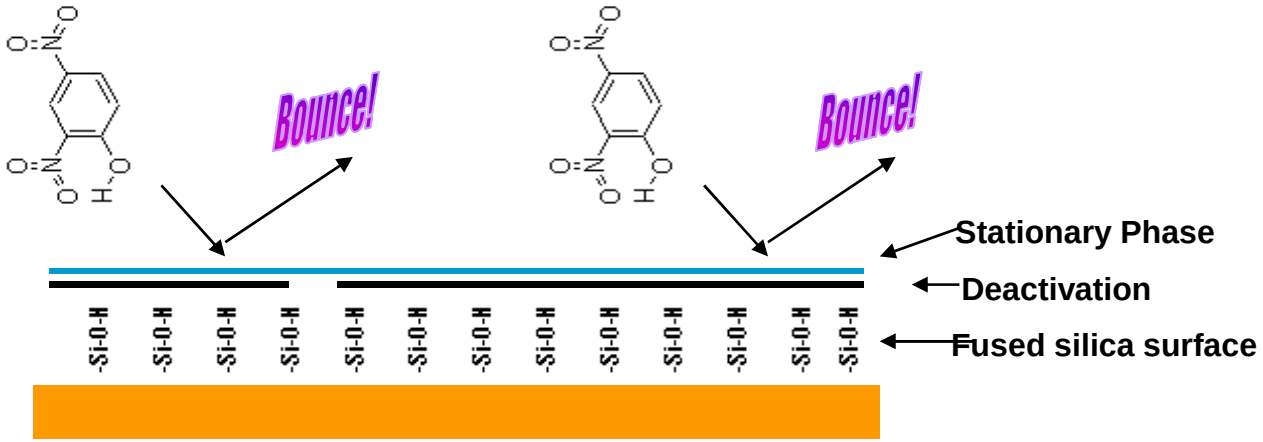
Traditional deactivation has gaps in surface coverage due to bulky TMS type moieties, and tight fused silica lattice, and is somewhat inert and chemically resistant.



DB-5ms and HP-5ms Engineered Deactivations

Polymeric Deactivation Technology

“Binds” at multiple points with many silanols



“Blankets” sterically hindered active silanols, fewer silanols

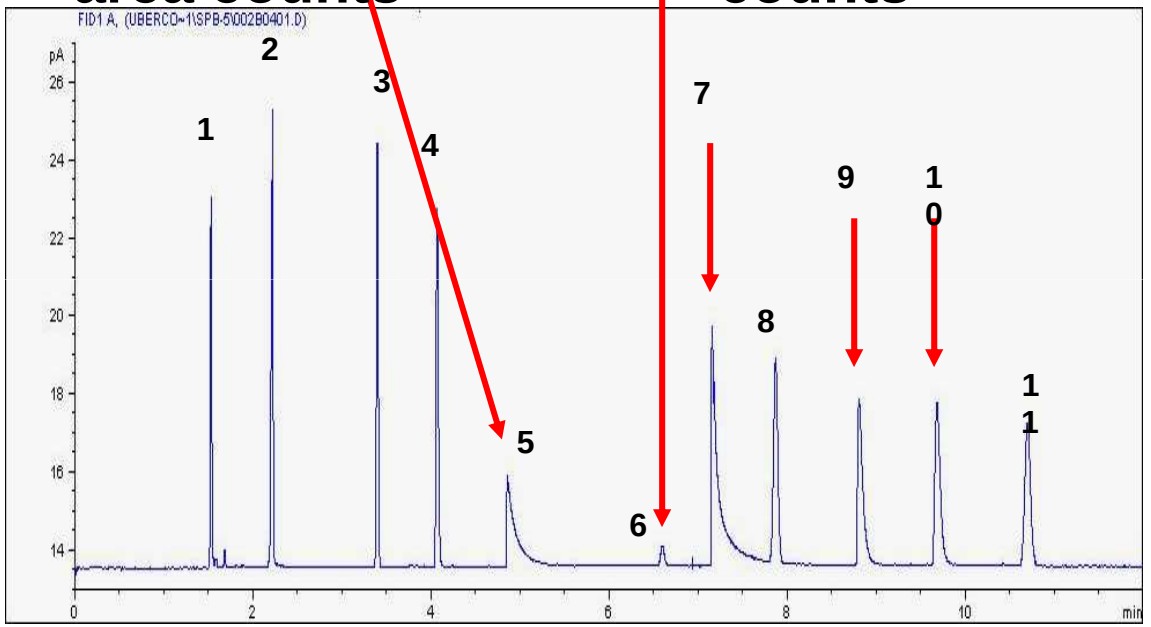
What does Column Activity look like?

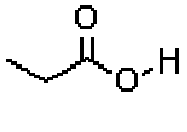
Tailing, and loss of response.

	Probe	0.5 ul inj, 1:50 split, (ng)
1	Methane	?
2	**Propionic Acid**	7.0
3	Octane	2.4
4	Nitrobutane	4.9
5	**4-Picoline**	2.7
6	**Trimethyl Phosphate**	12.7
7	**1,2-Pentanediol**	5.4
8	Propylbenzene	2.2
9	**1-Hetpanol**	3.3
10	3-Octanone	3.0
11	Decane	2.2

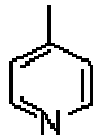
Loss of peak height, but same area counts

Loss of peak height and area counts

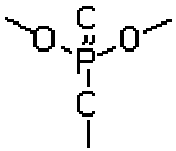




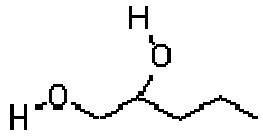
2



5



6



7

Agilent Technologies

What are the specific benefits of High Inertness?

Greater sensitivity for traditional trace active analytes

- **meet RRF requirements with greater ease**
- **more runs before maintenance**

Greater reliability for ultra-trace non-traditionally active analytes (<100 ppb PAHs, Chlorinated dioxins, etc…)

Who benefits from High Inertness Columns?

Anyone doing trace analysis of active analytes

Environmental semivolatile analysts

Pesticide residue analysts

Forensic/Drug analysts

Anyone in Industry, Government, or Academia interested in ultra-trace amounts of even modestly active analytes

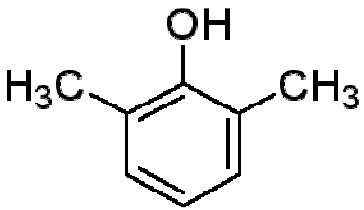
Test Probes and Column Activity QC Testing

- Test probes are vital to ensure the quality and reproducibility of GC columns
  - Properly deactivated
  - Contain the correct amount of stationary phase
  - consistent batch-to-batch relative retention time

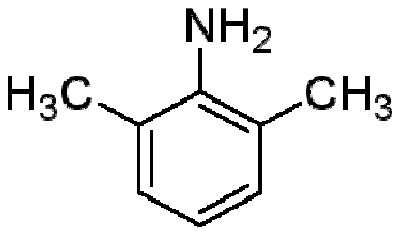
- Test probes can either highlight or mask the deficiencies of a column, normally include:
  - An organic acid (peak tailing or lost response of acid indicates the column is basic)
  - A base (peak tailing or lost response of base indicates the column is acidic)
  - An alcohol (gives indication of any oxygen damage or exposed silanols)
  - 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

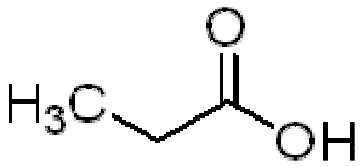
Weak Probes vs. Strong Probes



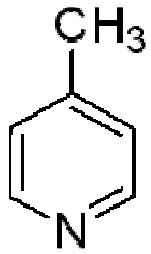
2,6-Dimethylphenol



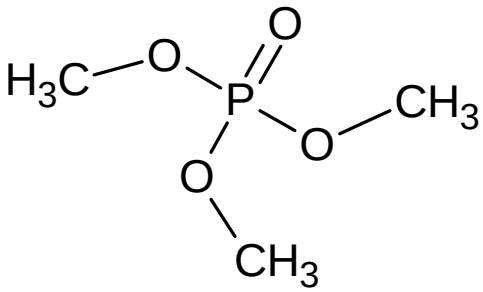
2,6-Dimethylaniline



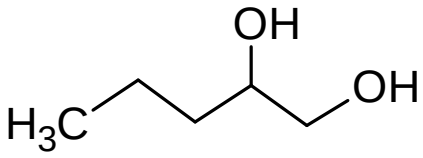
1-Propionic acid



4-Picoline



Trimethyl phosphate



1,2-Pentandiol

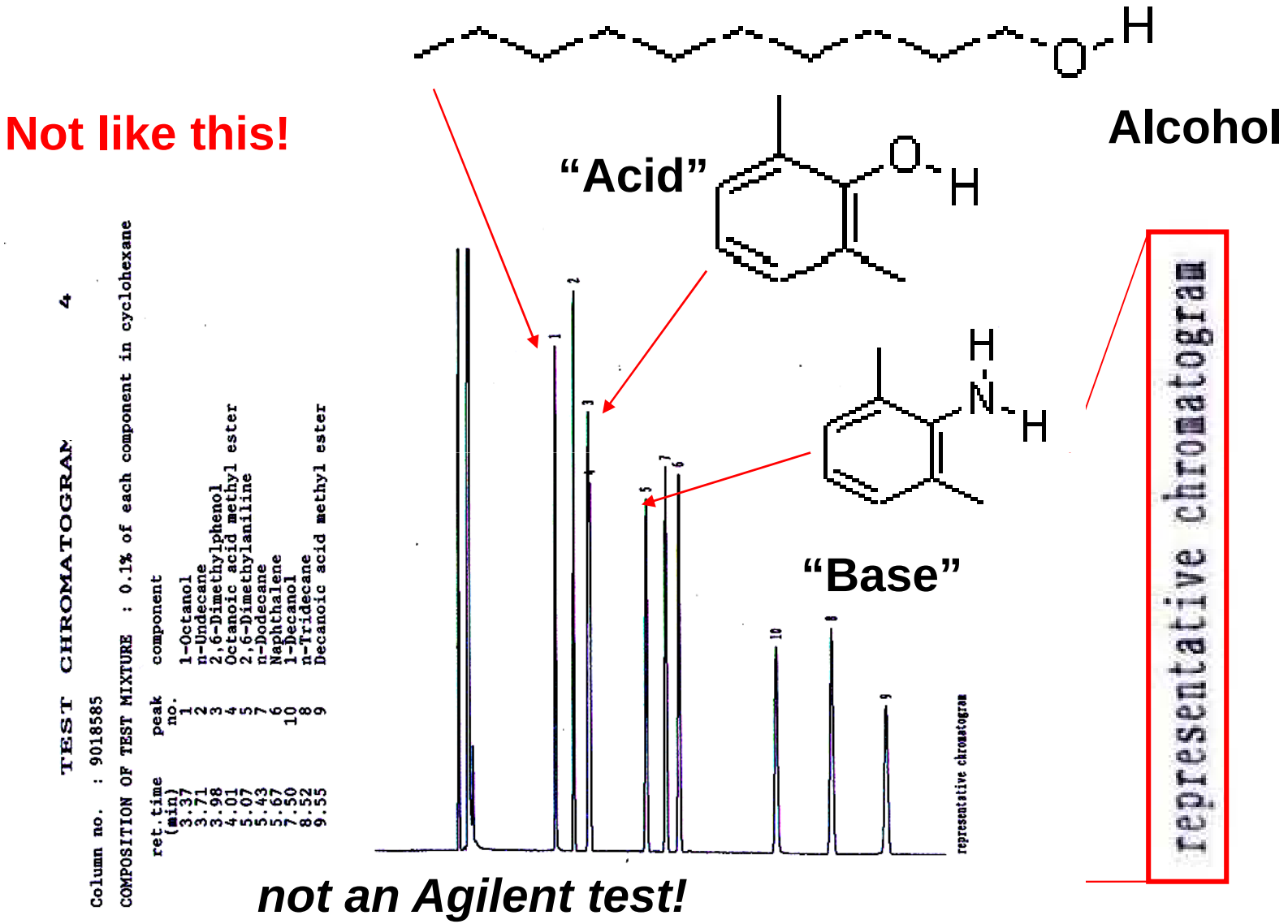
Weak Probes

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

Stronger Probes

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

How is High GC column Inertness Assured?

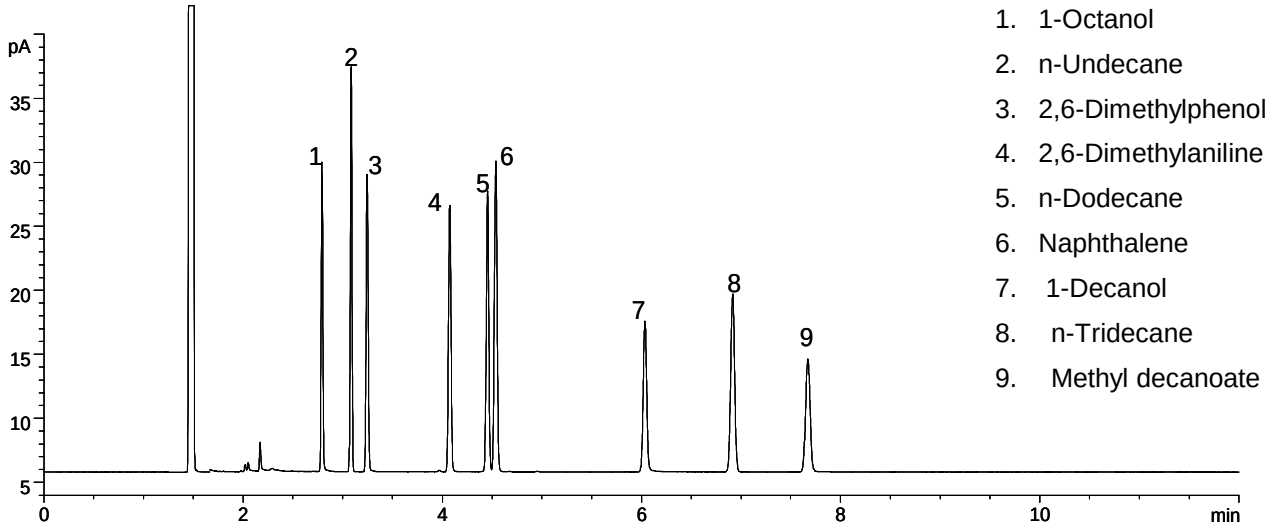


not an Agilent test!

Agilent Technologies

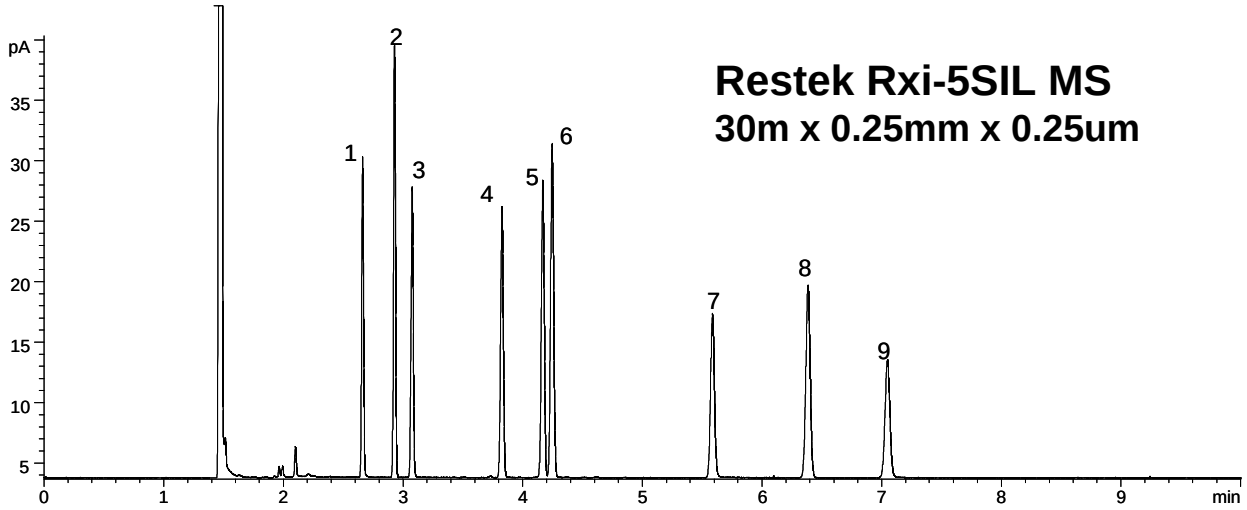
Grob-Type Mix - QC Testing of the 80s

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



1. 1-Octanol
2. n-Undecane
3. 2,6-Dimethylphenol
4. 2,6-Dimethylaniline
5. n-Dodecane
6. Naphthalene
7. 1-Decanol
8. n-Tridecane
9. Methyl decanoate

- Less demanding test mix
- Less probative probes for column activity
- Elevated oven temperature at 120℃ allows the molecules to sweep past active sites and mask solute/column interactions.

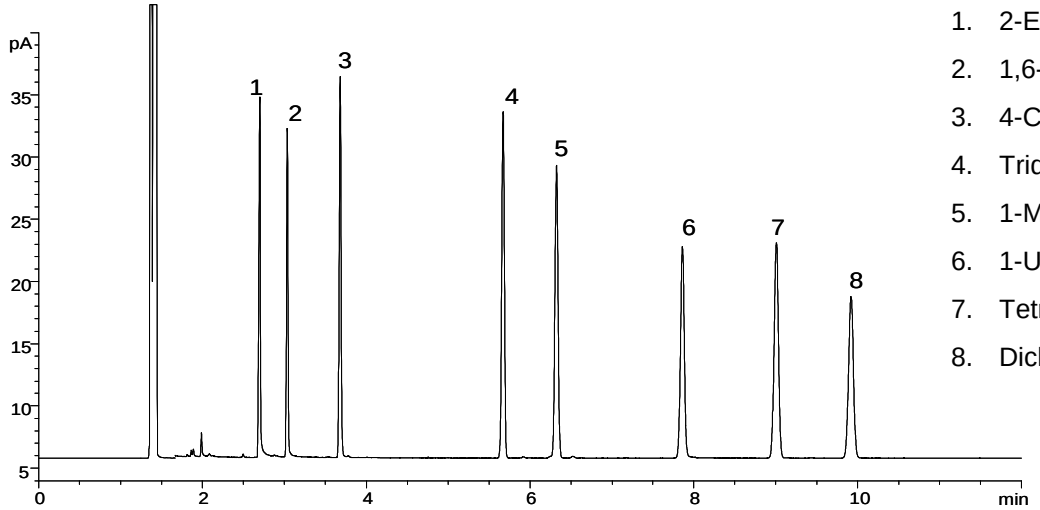


Restek Rxi-5SIL MS
30m x 0.25mm x 0.25um

Sampler:	Agilent 7683B, 5 µL syringe (Agilent part # 5181-1273), 1.5 µL split injection, 4 ng each component
Carrier:	Hydrogen constant pressure 37 cm/s
Inlet:	Split/splitless; 250 ºC, 1.4 ml/min. column flow, split flow 100 ml/min.
Liner:	Deactivated single taper w glass wool (Agilent part # 5183-4647)
Oven:	120 ºC isothermal
Detection:	FID at 325 ºC, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

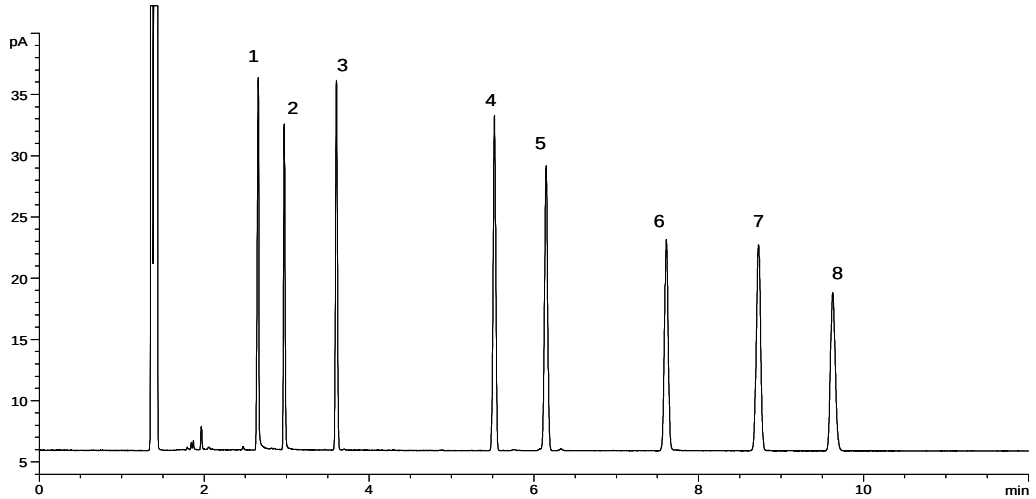
DB-5ms Test Mix – QC Testing of the 90s

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



1. 2-Ethylhexanic acid
2. 1,6-Hexanediol
3. 4-Chlorophenol
4. Tridecane
5. 1-Methylnaphthalene
6. 1-Undecanol
7. Tetradecane
8. Dichlorohexylamine

Restek Rxi-5SIL MS
30m x 0.25mm x 0.25um



Sampler:	**Agilent 7683B, 5 µL syringe (Agilent part # 5181-1273), 1.5 µL split injection, 4 ng each component**
Carrier:	**Hydrogen constant pressure 38 cm/s**
Inlet:	**Split/splitless; 250 ºC, 1.4 ml/min. column flow, split flow 75 ml/min.**
Liner:	**Deactivated single taper w glass wool (Agilent part # 5183-4647)**
Oven:	**125 ºC isothermal**
Detection:	**FID at 320 ºC, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup**

How Agilent Assures High Inertness on the HP-5ms columns, with Every Test

Performance Results

Theoretical Plates/Meter:

PENTADECANE	3945

Retention Index:

METHYL CAPRATE	1324.1
ACENAPHTHYLENE	1462.2
1-DODECANOL	1473.8

Peak Height Ratio:

1-DODECANOL, TETRADECANE	0.54
1-DECYLAMINE, TRIDECANE	1.43
4-CHLOROPHENOL, TRIDECANE	1.12

Compound Identification	Retent. Time	Part. Ratio	1/2-Width
1. UNDECANE	2.506	.70	.015
2. 4-CHLOROPHENOL	3.222	1.18	.023
3. 1-DECYLAMINE	3.813	1.58	.024
4. TRIDECANE	4.528	2.07	.028
5. METHYL CAPRATE	4.946	2.35	.031
6. TETRADECANE	6.677	3.52	.043
7. ACENAPHTHYLENE	8.700	4.89	.061
8. 1-DODECANOL	9.155	5.20	.062
9. PENTADECANE	10.293	5.97	.070

Test Conditions

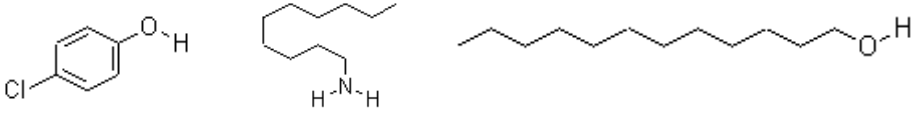
Inlet: Split (275°C) **Detector:** FID (325°C)

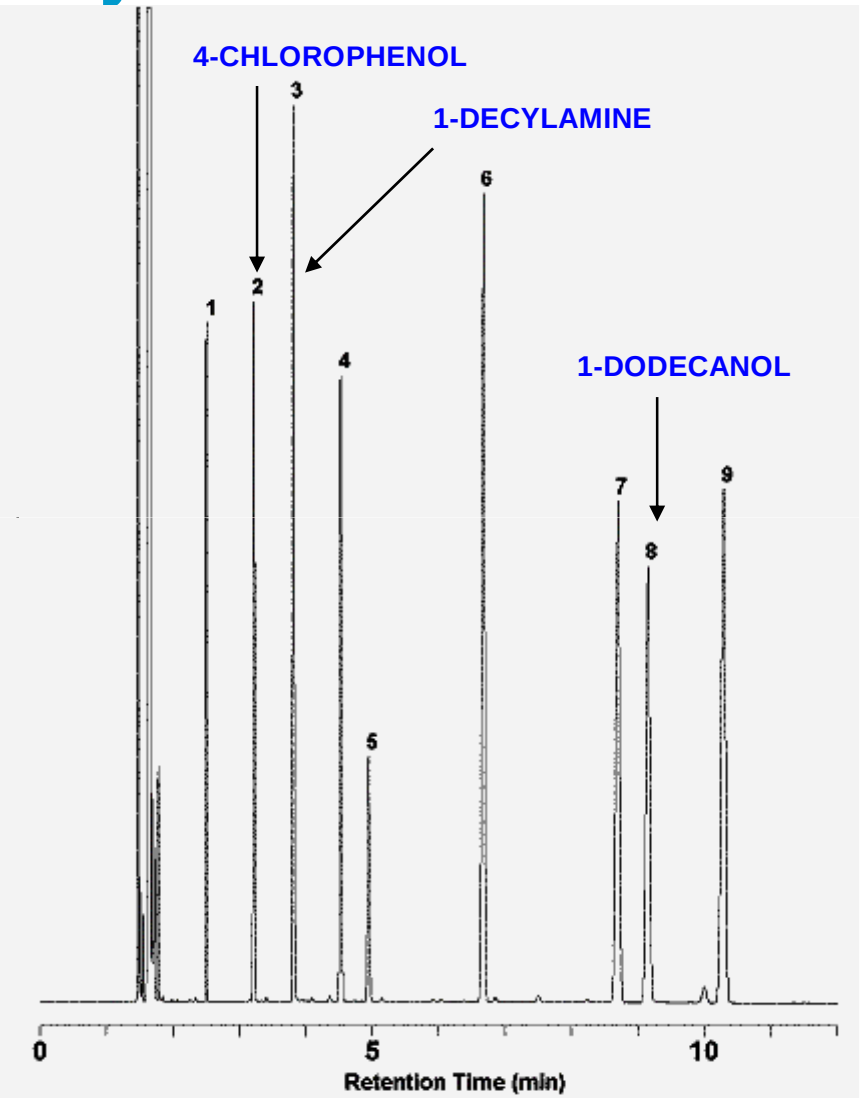
Carrier Gas: Hydrogen **Flow:** 33.9 cm/sec (1.0 ml/min)

Holdup Compound: Pentane (1.477-min)

Temperature Progra Isothermal at 135°C

Acid **Base** **Alcohol**





Agilent Technologies

How Agilent Assures High Inertness on the DB-5ms columns, with Every Test

Performance Results

Theoretical Plates/Meter:
TETRADECANE 4165

UTE%:
TETRADECANE 89.9%

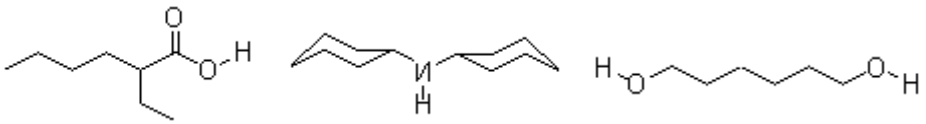
Retention Index:
1-METHYLNAPHTHALENE 1323.7
1-UNDECANOL 1370.7

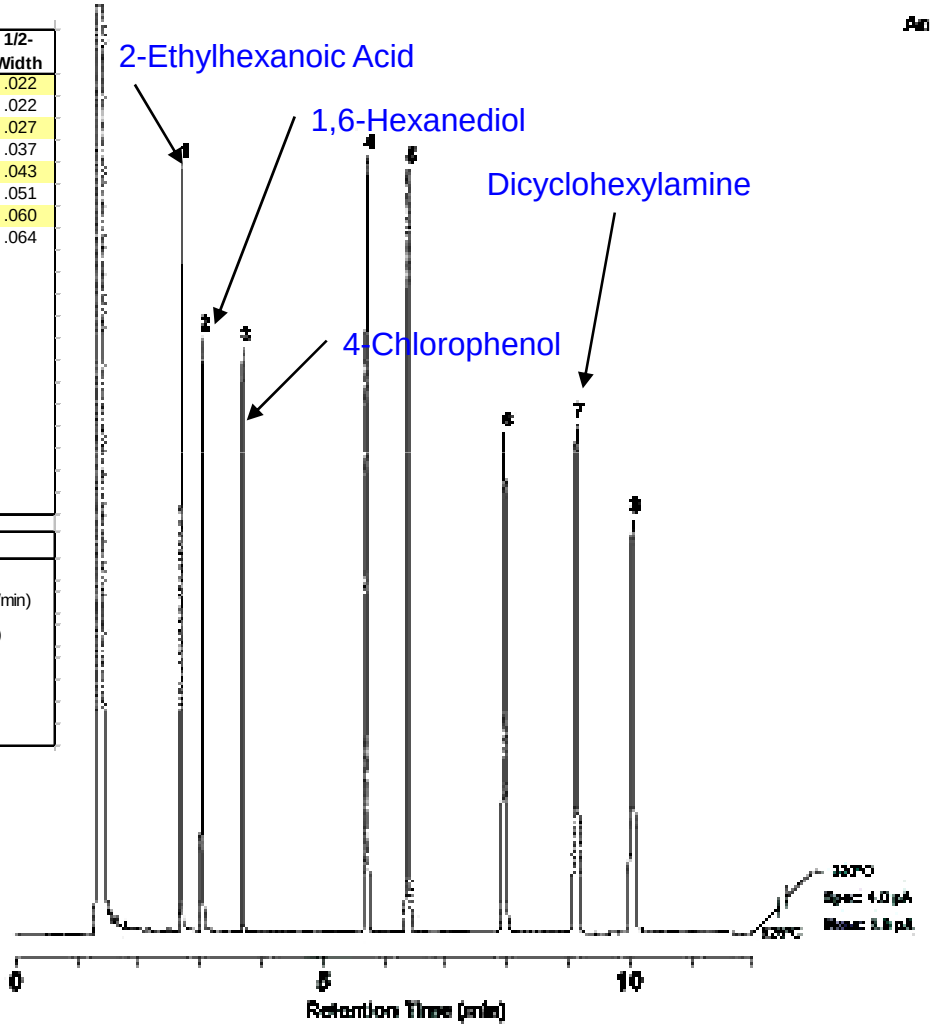
Compound Identification	Retent. Time	Part. Ratio	1/2-Width
1. 2-ETHYLHEXANOIC ACID	2.655	1.05	.022
2. 1,6-HEXANEDIOL	2.990	1.31	.022
3. 4-CHLOROPHENOL	3.635	1.80	.027
4. TRIDECANE	5.627	3.34	.037
5. 1-METHYLNAPHTHALENE	6.283	3.84	.043
6. 1-UNDECANOL	7.820	5.03	.051
7. TETRADECANE	8.963	5.91	.060
8. DICYCLOHEXYLAMINE	9.877	6.62	.064

Test Conditions

Inlet: Split (250°C) **Detector:** FID (320°C)
Carrier Gas: Hydrogen **Flow:** 38.6 cm/sec (1.2 ml/min)
Holdup Compound: Methane (1.297-min)
Temperature Progra Isothermal at 125°C

Acid **Base** **Diol**





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

Probe	(ng on column)	Column functional test
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
6. 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

- Carefully selected very demanding test probes for in-depth evaluation of column inertness
- Test temperature 65°C (isothermal), well below that normally used in conventional tests

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

Carrier: Hydrogen constant pressure, 38 cm/s

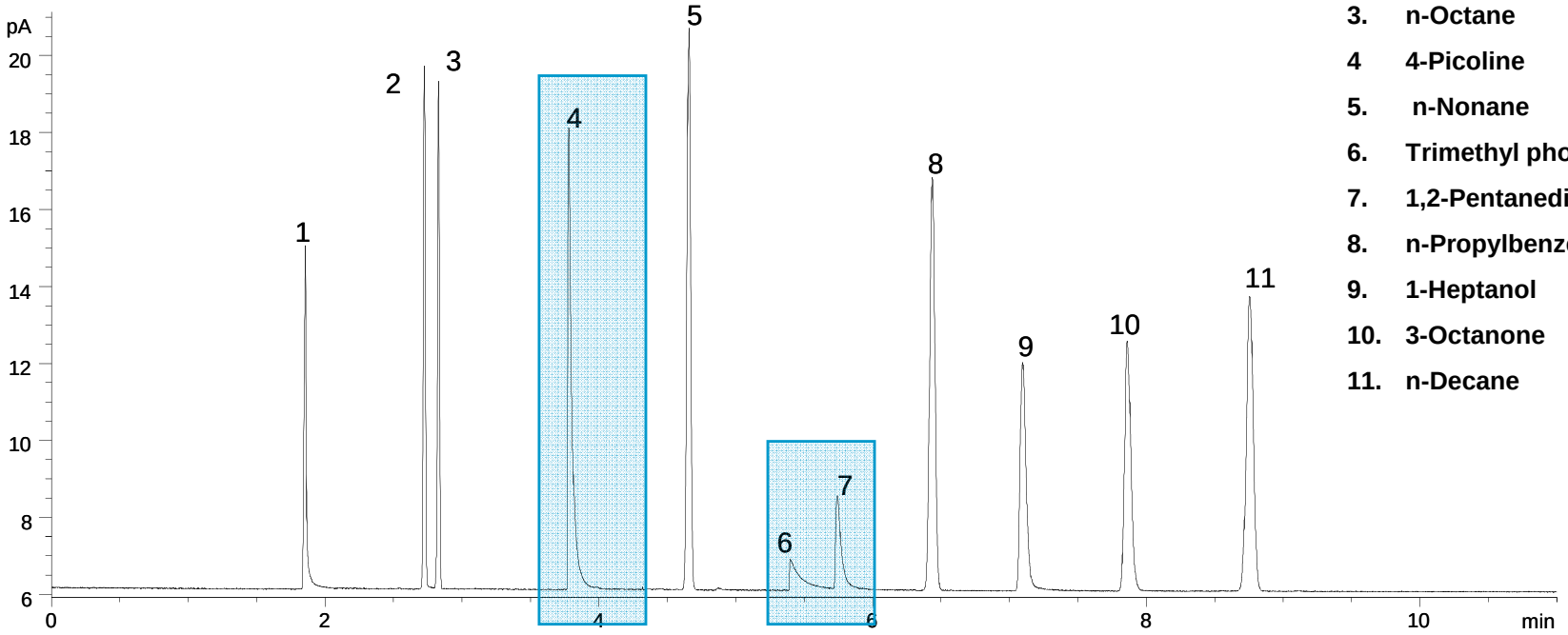
Inlet: Split/splitless; 250 ºC, 1.4 ml/min. column flow, split flow 900 ml/min., gas saver flow 75 ml/min. on at 2.0 min.

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

Oven: 65 ºC isothermal

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

Ultra Inert Test Mix on Restek Rxi-5Sil MS



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

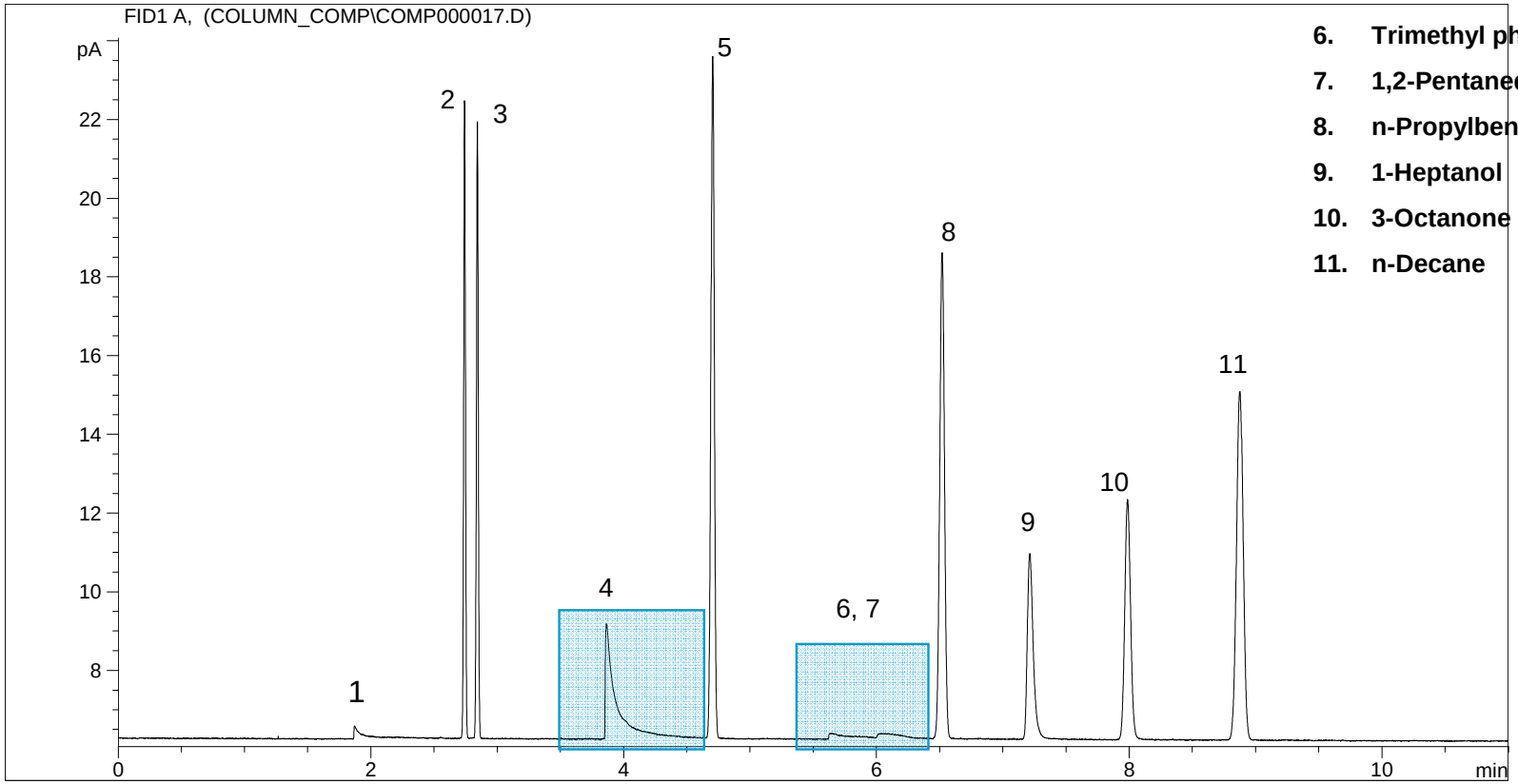
All highlighted peaks have poor peak shape – poor column deactivation

- The Restek column showed very poor performance when tested against the Über One test mix.
- Less demanding test probes masked the column activity for this Restek columns
  - The same column performed well with Grob-type test mix and DB-5ms test mix

Ultra Inert Test Mix on a Phenomenex Column

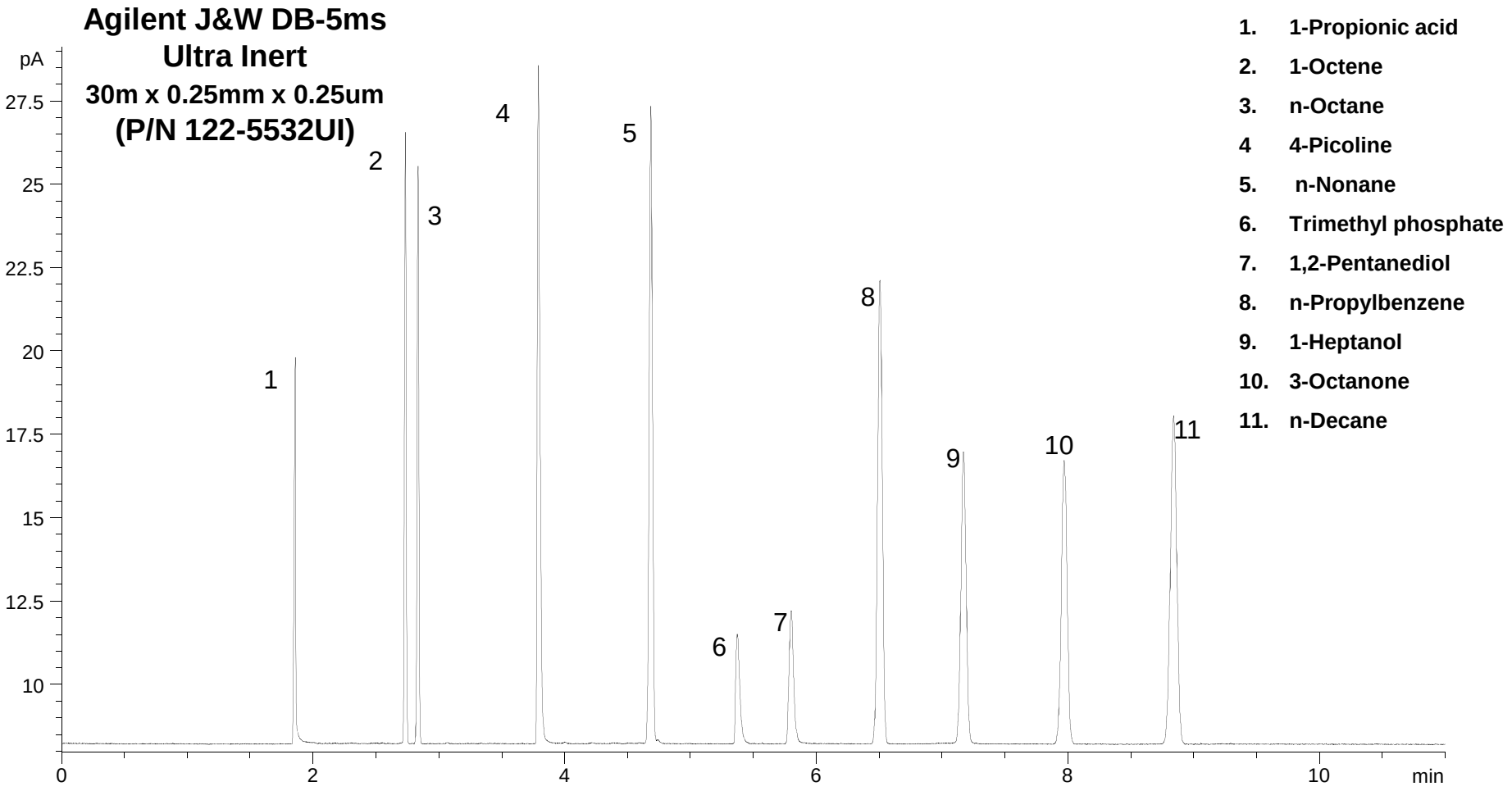
Phenomenex ZB-5ms
30m x 0.25mm x 0.25um

All highlighted peaks have poor peak shape with obvious lost of responses.



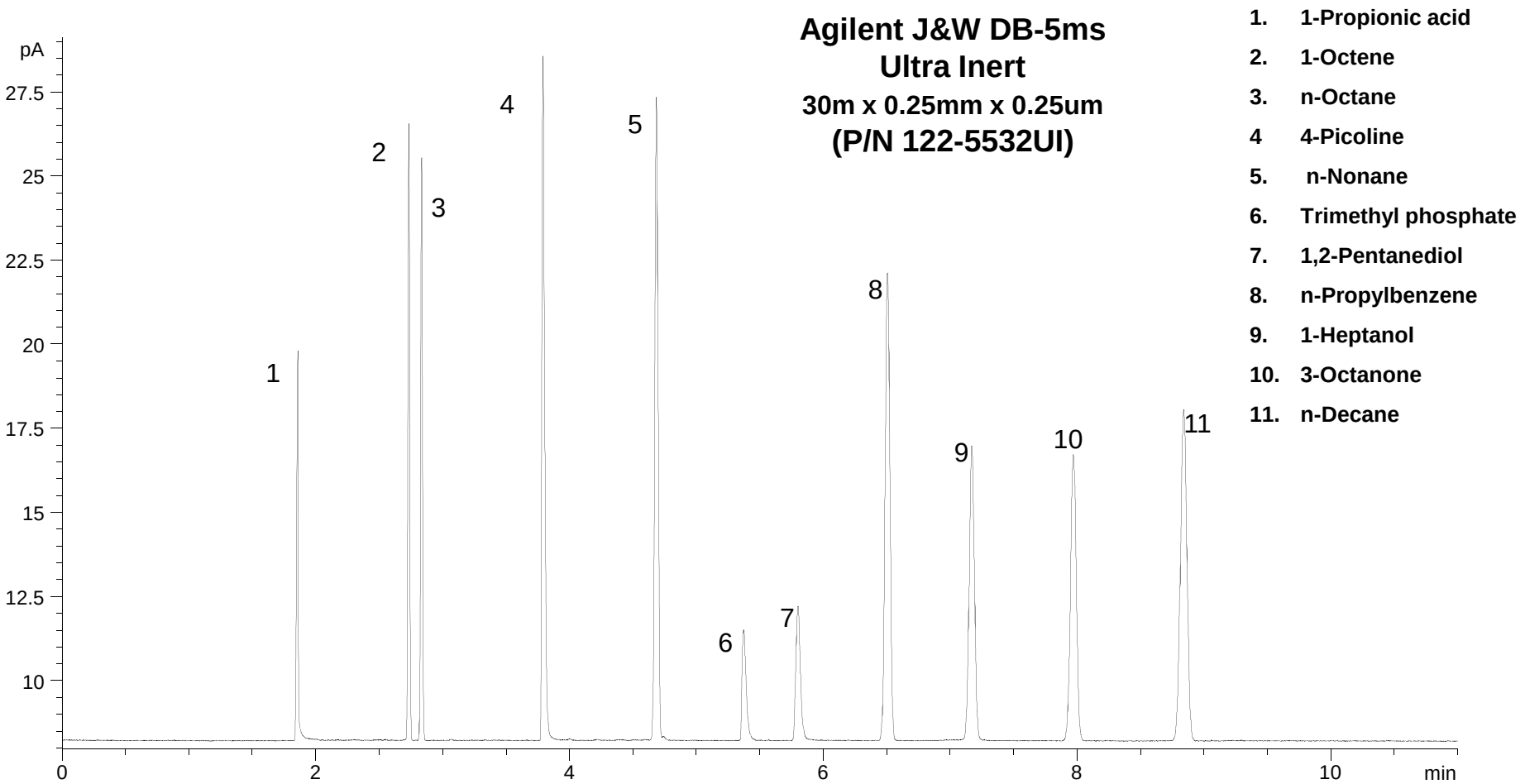
1. 1-Propionic acid
2. 1-Octene
3. n-Octane
4 4-Picoline
5. n-Nonane
6. Trimethyl phosphate
7. 1,2-Pentanediol
8. n-Propylbenzene
9. 1-Heptanol
10. 3-Octanone
11. n-Decane

Ultra Inert Test Mix on Agilent J&W DB-5ms Ultra Inert



- Nice Peak Shapes for challenging active compounds
- Increased peak heights for accurate integration and detection of trace samples
- Routine analysis of demanding analytes now feasible

Ultra Inert Test Mix on Agilent J&W DB-5ms Ultra Inert



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

- Nice Peak Shapes for challenging active compounds
- Increased peak heights for accurate integration and detection of trace samples
- Routine analysis of demanding analytes now feasible

Test Mix Summary

- Grob-type mix not probative for inertness
- DB-5ms text mix is a good test for the 90s
- Über One mix probes inertness and **differentiates an excellent column** from a mediocre one
- Well designed test mix uncovers potential adsorption of acid and base analytes and raises the bar in inertness QC

Challenges and Needs of Today's Laboratories

- Challenges
  - Qualification/quantification of trace samples
  - Keep instrument up and running
- Needs
  - Lower detection limits
  - Improved stability in GC or GC/MS system

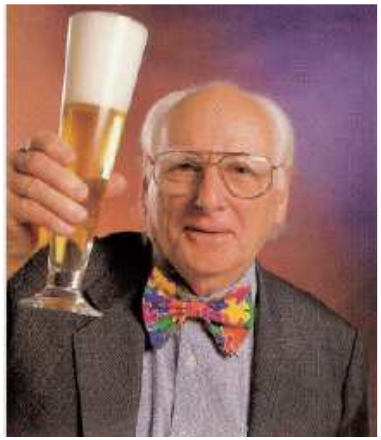
Lower Detection Limit	
Reduce noise	**Increase signal**
Injection system (septa, liners, connections)	Sample concentration
Carrier gas and detector gases	Sample size
Leaks	Inert injection and detection port sleeves/liner
Temperature setting	Gas velocity or temp program rate
Stationary phase and column bleed	**Column inertness**

- Only when a column exhibits **both low bleed** and **low activity** are results reliable.
  - Low bleed increases the signal-to-noise ratio, but if any of the analyte is adsorbed by active sites in the column, the results are flawed.
  - If the column is well deactivated but the bleed is high, some of the signal generated by the analytes is smothered by the bleed signal. Again, the results are flawed.

Agilent J&W Ultra Inert Columns – Innovation

- Only GC columns proven to deliver on the promise of column inertness and column bleed.
- Built on top of the existing Agilent J&W GC/MS columns with added column inertness.
- New QC testing for modern applications
  - A unique, very demanding test probe mixture to test columns individually
  - Worse case scenario testing to ensure column inertness performance
  - Raising the bar for GC column QC testing
- Critical for analysis of active compounds, trace level samples, and screening of unknown samples

Features	Advantages	Benefits
Unique QC testing procedure with demanding Ultra Inert test probe mixture	Consistent column inertness performance	More predictable and reliable results
Highest column inertness	Better peak shape	More accurate peak identification, more accurate quantification
	Greater S/N ratio	
	Minimum compound loss or degradation	
	More analysis without maintenance	Reduced costs and instrument downtime
Exceptionally low column bleed	Greater S/N ratio and increased sensitivity for all detectors	Lower detection limit, reduced detector contamination, and reliable compound identification
	Faster baseline stabalization	Minimized conditioning time and increased sample throughput
Support of 0.18mm ID configuration	Higher sample throughput	More sample analysis in less time



The Quality of the Ultra Inert Column Series Exceeds my Wildest Dream

*"[Agilent's] **breakthroughs** in surface pretreatments and **improvements** in surface deactivation came much more rapidly than I had anticipated. The quality of the new Ultra Inert series of columns exceed my wildest dreams."*

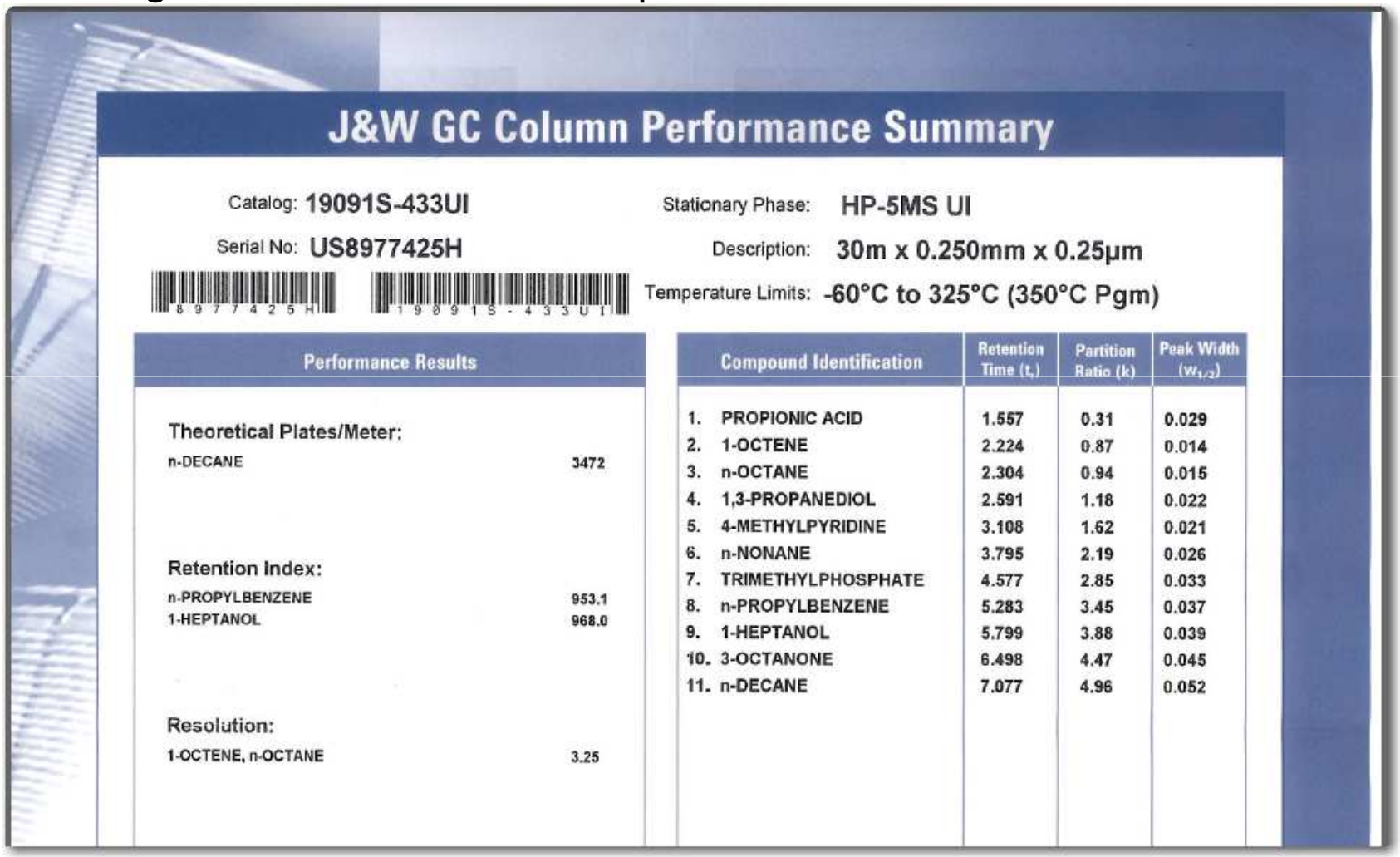
"I am satisfied that customers with the most demanding analyses of active analytes can have confidence that the DB-5ms and HP-5ms Ultra Inert Columns will provide the highest level of performance."

Walt Jennings

Professor Emeritus, University of California

Every Column Individually Test – Ensured Quality

- Performance summary sheet shipped with each column with QC testing results against the Ultra Inert test probe mixture.



J&W GC Column Performance Summary

Catalog: **19091S-433UI**

Serial No: **US8977425H**

Stationary Phase: **HP-5MS UI**

Description: **30m x 0.250mm x 0.25µm**

Temperature Limits: **-60°C to 325°C (350°C Pgm)**

Performance Results

Theoretical Plates/Meter:

n-DECANE	3472

Retention Index:

n-PROPYLBENZENE	953.1
1-HEPTANOL	968.0

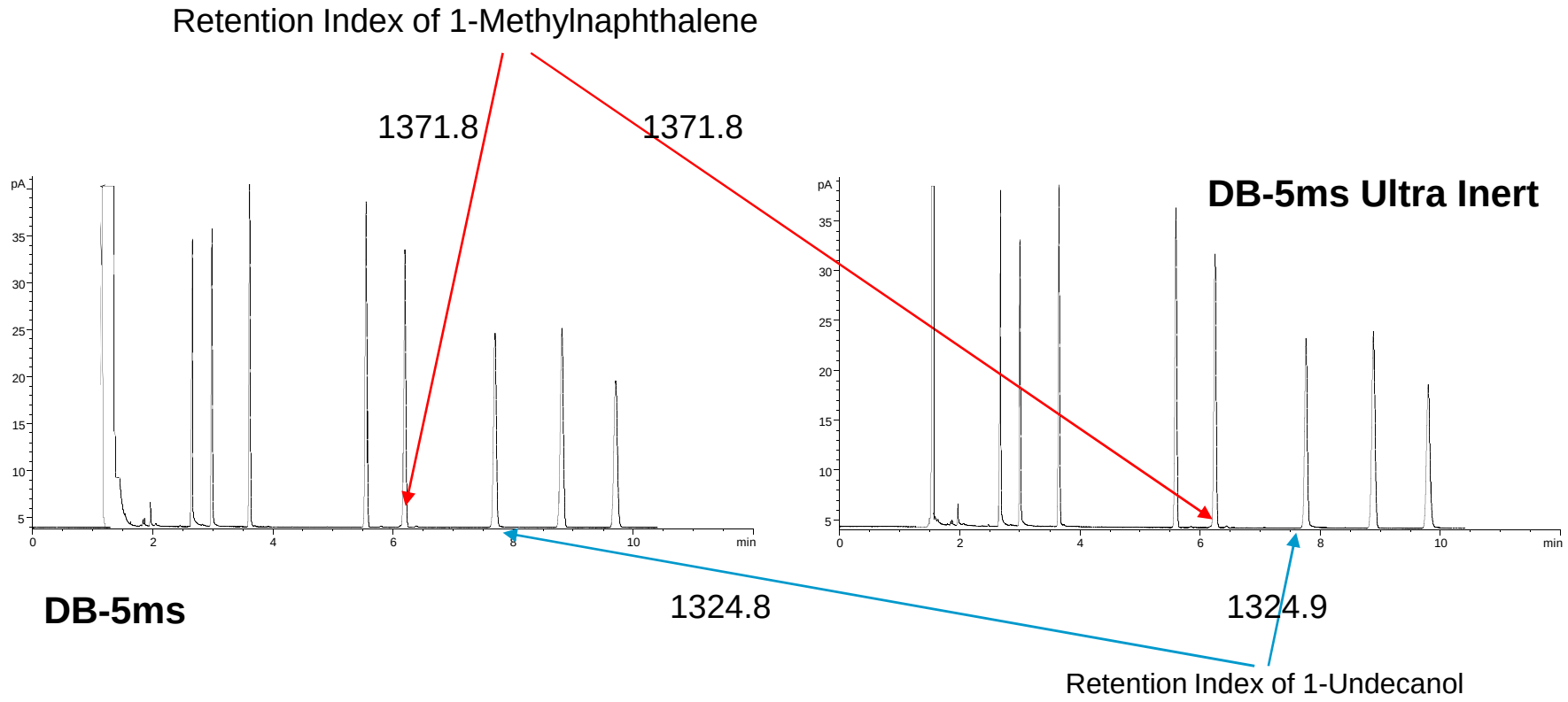
Resolution:

1-OCTENE, n-OCTANE	3.25

Compound Identification	Retention Time (t_r)	Partition Ratio (k)	Peak Width ($w_{1/2}$)
1. PROPIONIC ACID	1.557	0.31	0.029
2. 1-OCTENE	2.224	0.87	0.014
3. n-OCTANE	2.304	0.94	0.015
4. 1,3-PROPANEDIOL	2.591	1.18	0.022
5. 4-METHYLPYRIDINE	3.108	1.62	0.021
6. n-NONANE	3.795	2.19	0.026
7. TRIMETHYLPHOSPHATE	4.577	2.85	0.033
8. n-PROPYLBENZENE	5.283	3.45	0.037
9. 1-HEPTANOL	5.799	3.88	0.039
10. 3-OCTANONE	6.498	4.47	0.045
11. n-DECANE	7.077	4.96	0.052

Same Selectivity – No Method Re-Development

- DB-5ms Ultra Inert columns have the same selectivity as their DB-5ms counterparts
- HP-5ms Ultra Inert columns have the same selectivity as their HP-5ms counterparts



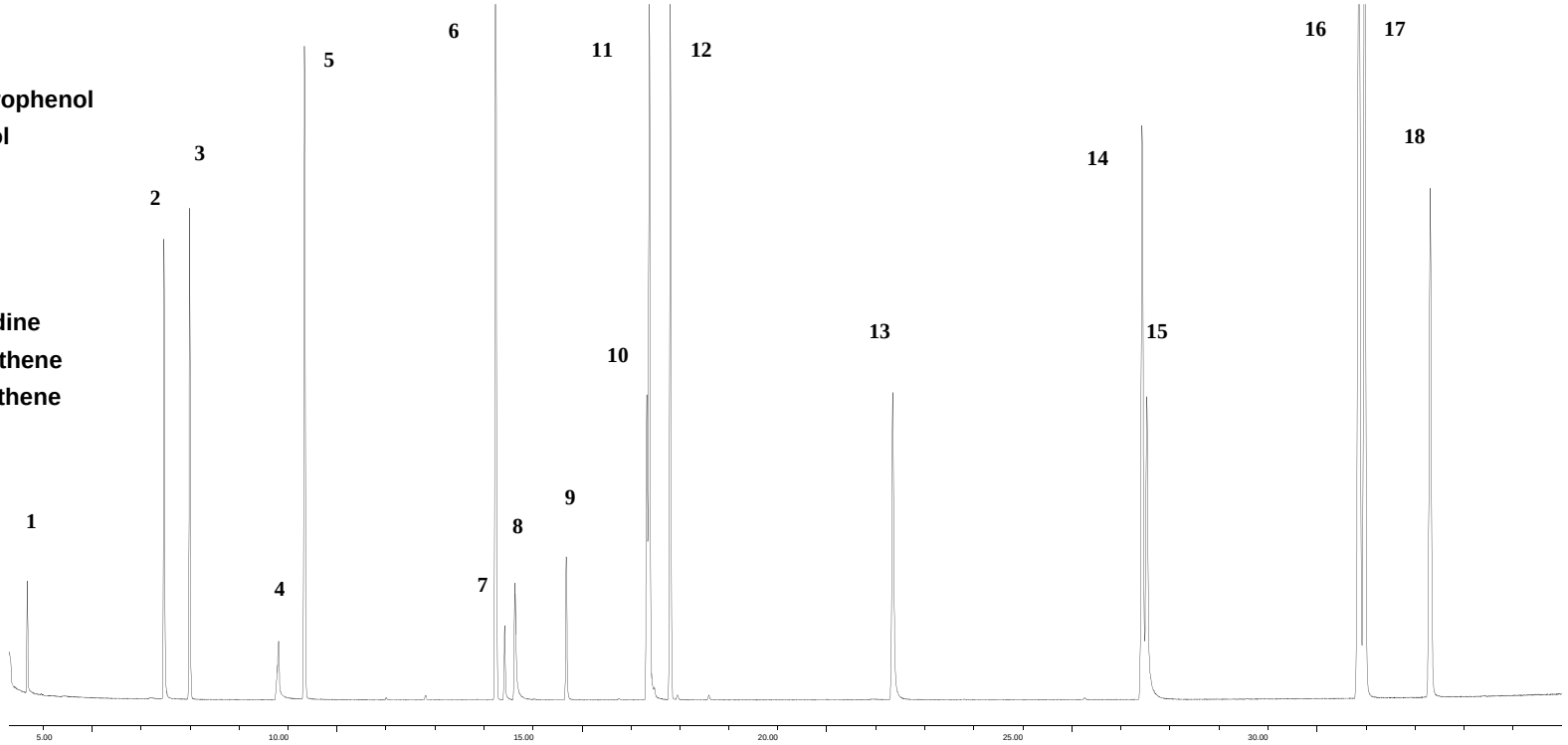
Application Examples

- Semi Volatile Analysis
- Brominated Fire Retardants
- Pesticides in Orange Oil
- PAHs
- PBDEs

Semi Volatile Analysis

GC :	**Agilent 6890N/5975B MSD**
Sampler :	**Agilent 7683B, 5.0 µL syringe (Agilent part # 5188-5246), 1.0 µL splitless injection, 5 ng on column**
Carrier:	**Helium constant flow 30 cm/s**
Inlet:	**Split/splitless; 260% C, 53.7 ml/min. total flow, purge flow 50 ml/min. on at 0.5 min., gas saver off**
Inlet Liner:	**Deactivated single taper w glass wool (Agilent part # 5183-4647)**
Column:	**DB-5ms Ultra Inert 30m x 0.25mm x 0.25µm (Agilent part # 122-5532UI)**
Oven:	**40% C (1 min) to 100%C (15% C/min), 10% C to 210% C (1 min), 5% C/min. to 310% C (8 min)**
Detection:	**MSD source at 300% C, quadrupole at 180% C, transfer line at 290% C, scan range 50-550 AMU**

1. **N-nitrosodimethylamine**
2. **Aniline**
3. **1,4 dichlorobenzene-D4**
4. **Benzoic acid**
5. **Naphthalene- D8**
6. **Acenapthene-D10**
7. **2,4-dinitrophenol**
8. **4-nitrophenol**
9. **2-methyl-4,6-dinitrophenol**
10. **pentachlorophenol**
11. **4-aminobiphenyl**
12. **Penanthrene-D10**
13. **Benzidine**
14. **Chrysene-D12**
15. **3,3'-dichlorobenzidine**
16. **Benzo [b] fluoroanthene**
17. **Benzo [k] fluoroanthene**
18. **Perylene-D12**



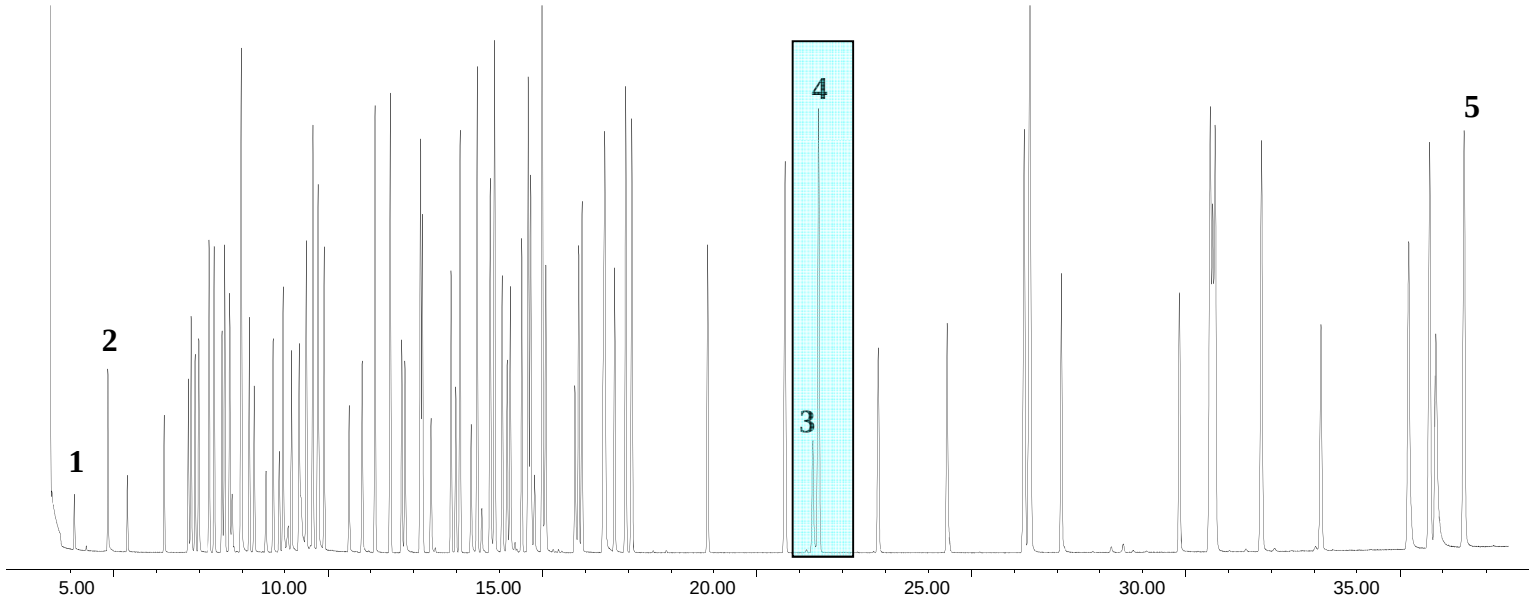
Agilent Technologies

“Large Mix” 5 ng on Column AccuStandard 8270 Mixes 1,2,3,4a,4b,5 &6 (93 Compounds) Select compound highlighted

1. **n-Nitrosodimethylamine**
2. **2-methyl pyridine**
3. **Benzidene**
4. **Flouranthene**
5. **Benzo (g,h,i) perylene**

GC/MSD Conditions

Column:	**DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm part # 122-5532UI**
Carrier:	**He 30 cm/sec constant flow**
Oven:	**40% C (1min) to 100 % C (15 % C/min), 10 % C /min to 210% C (1min), 5 % C/min to 310% C (8 min)**
Inlet:	**splitless 260 % C purge flow 50 % ml/min at 0.5 min, gas saver 80 ml/min on at 1 minute**
MSD:	**transfer line 290 % C, source 300 % C, quad 180 % C**



Pesticides and Fire Retardants (US EPA 527)

1. 1,2-Dimethyl-2-nitrobenzene
2. Acenaphthalene-D10
3. Dimethoate
4. Atrazine
5. Propazine
6. Anthracene-D10
7. Vinclozoline
8. Prometryne
9. Bromacil
10. Malathion
11. Thiazopyr
12. Dursban
13. Benthiocarb
14. Parathion
15. Terbus sulfone
16. Bioallethrin
17. Oxychlordane
18. Fenamiphos
19. Nitrophen
20. Norflurazone
21. Kepone
22. Hexazinone
23. Triphenyl phosphate
24. Bifenthrin
25. Chrysene-D12
26. BDE-47
27. Mirex
28. BDE-100
29. BDE-99
30. Perylene-D12
31. Fenvalerate
32. Esfenvalerate
33. Hexabromobiphenyl
34. BDE-153

GC/MSD Conditions

Sample: Pesticide/PBDE standards 1 ng with 5ng IS/SS on column

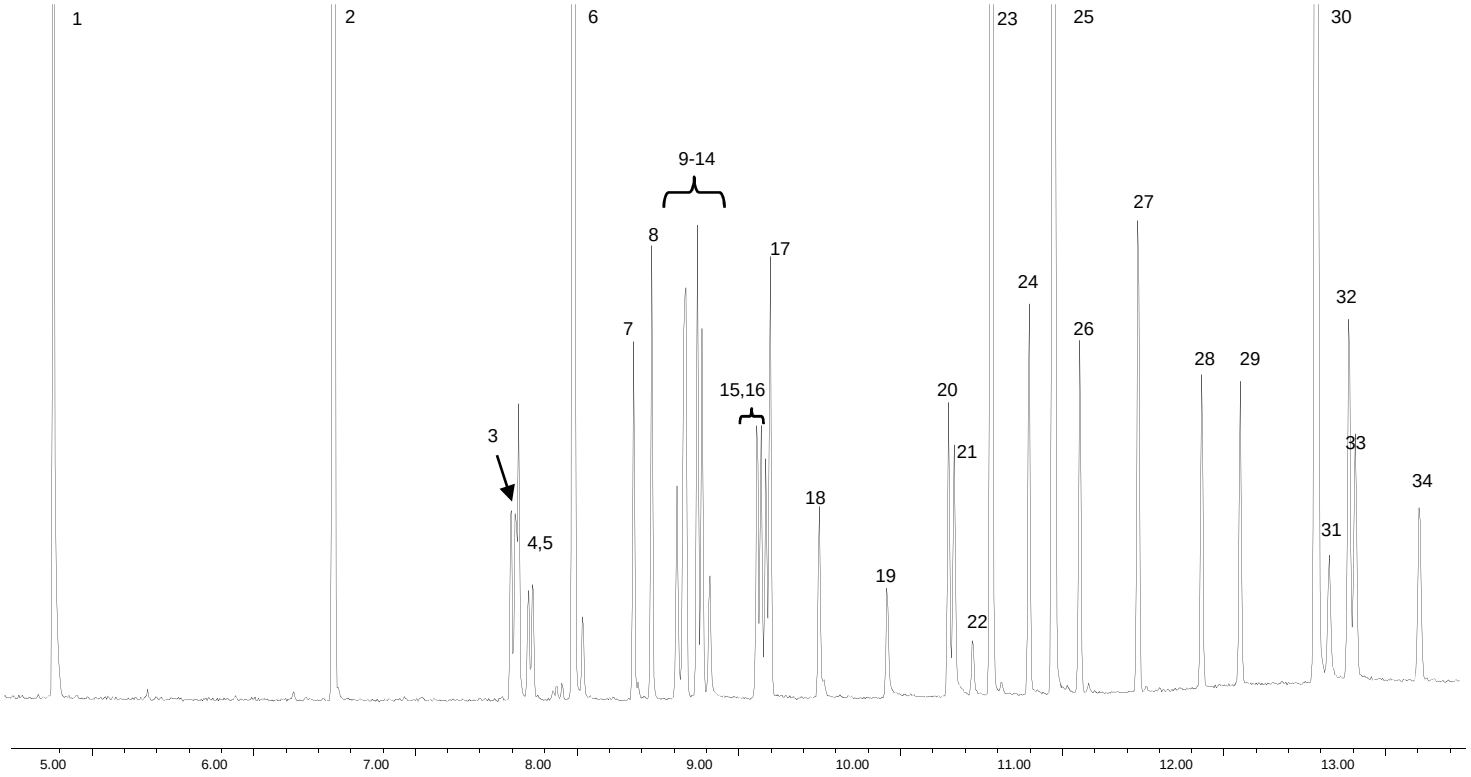
Column: DB-5MS Ultra Inert 30m x 0.25mm x 0.25um (Agilent part # 122-5532UI)

Carrier: Helium 52cm/sec, constant flow

Oven: 60°C (1min) to 210°C (25°/min), 20°C/min to 310°C (3 min)

Injection: Splitless, 250°C, purge flow 50ml/min at 1min, gas saver 80ml/min on at 3 min

MSD: Transfer Line 290°C , Source 300°C, Quad 180°C

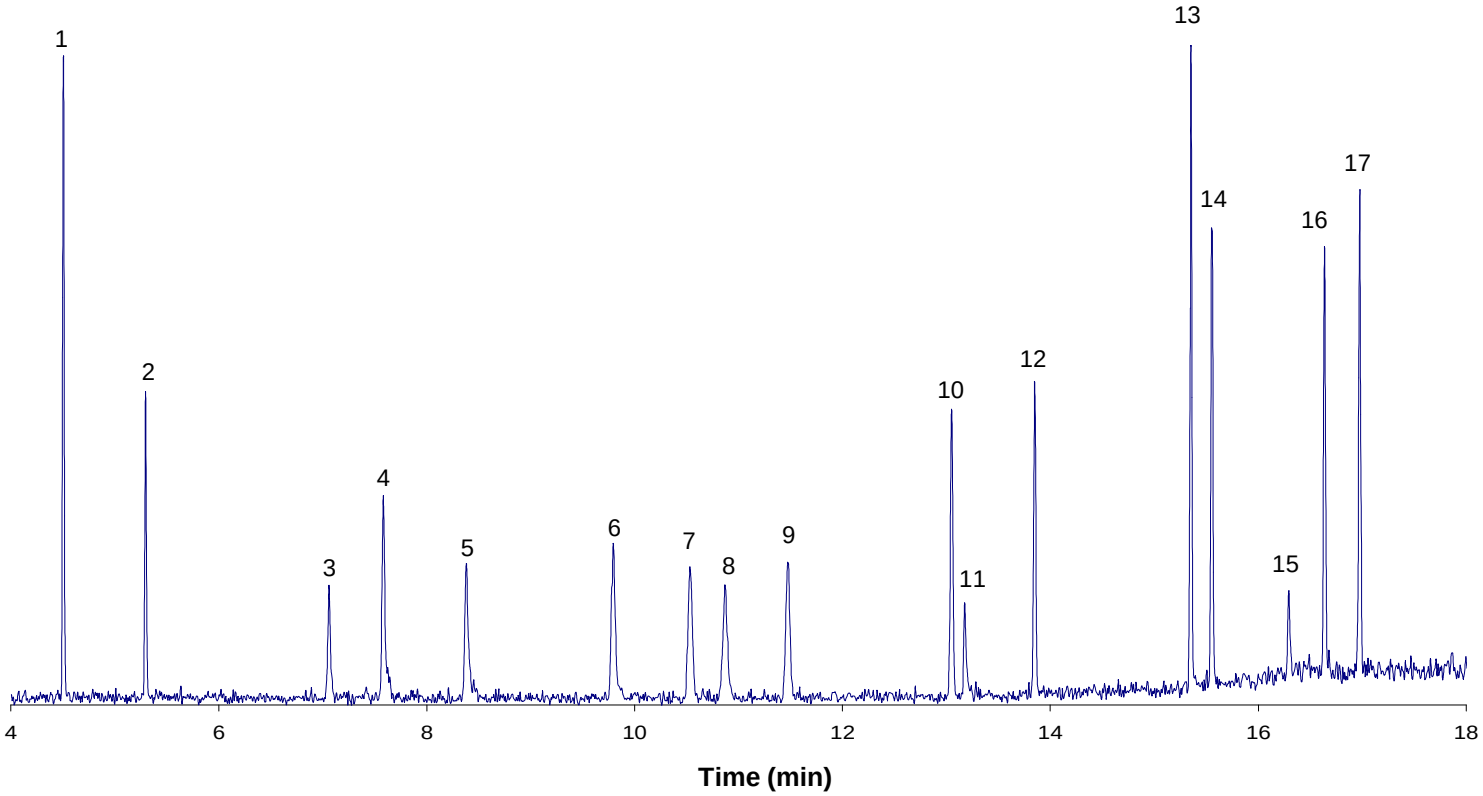


Agilent Technologies

Drugs of Abuse

Column:	**DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 µm (Agilent part # 122-5532UI)**
Carrier:	**Helium 43.8 cm/sec constant flow**
Oven:	**120% C (2min) 20 % C/min to 180 % C (6 min hold), 18 % C /min to 270% C (2min), 25 % C/min to 325% C (2 min)**
Inlet:	**split 30:1, ~ 1 ng on column 250 %C, single taper liner (Agilent # 5181-3316)**
MSD:	**transfer line 300 % C, source 280 % C, quad 200 % C, full scan m/z 50-450**

1. **Nicotine**
2. **Phenmetrazine**
3. **Ibupfrofen**
4. **Butabarbitol**
5. **Amobarbitol**
6. **Secobarbitol**
7. **Caffeine**
8. **Benzphetamine**
9. **Hexobarbitol**
10. **Tropacocaine**
11. **Phenbarbitol**
12. **Procaine**
13. **L-cocaine**
14. **Chlorcytizone**
15. **Codine**
16. **Diazpam**
17. **Oxymorphone**

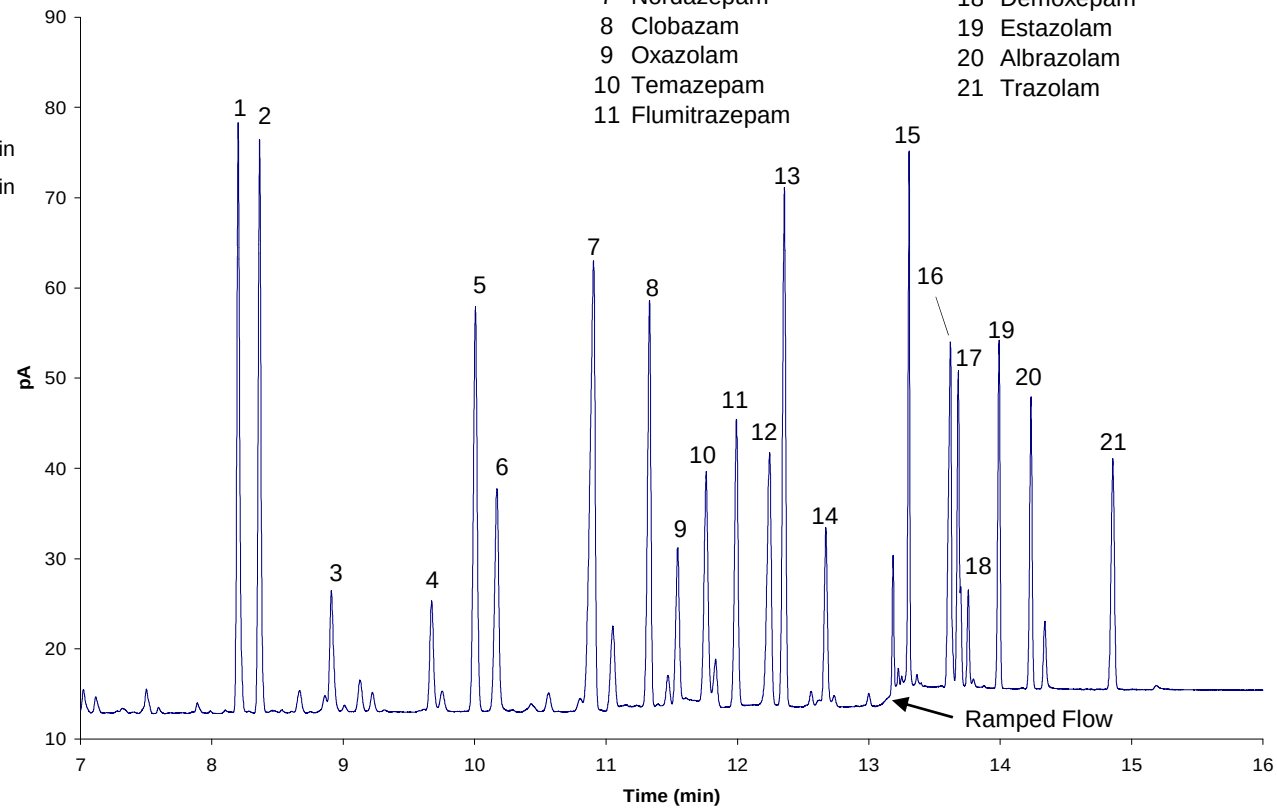


Agilent Technologies

Bezodiazepines

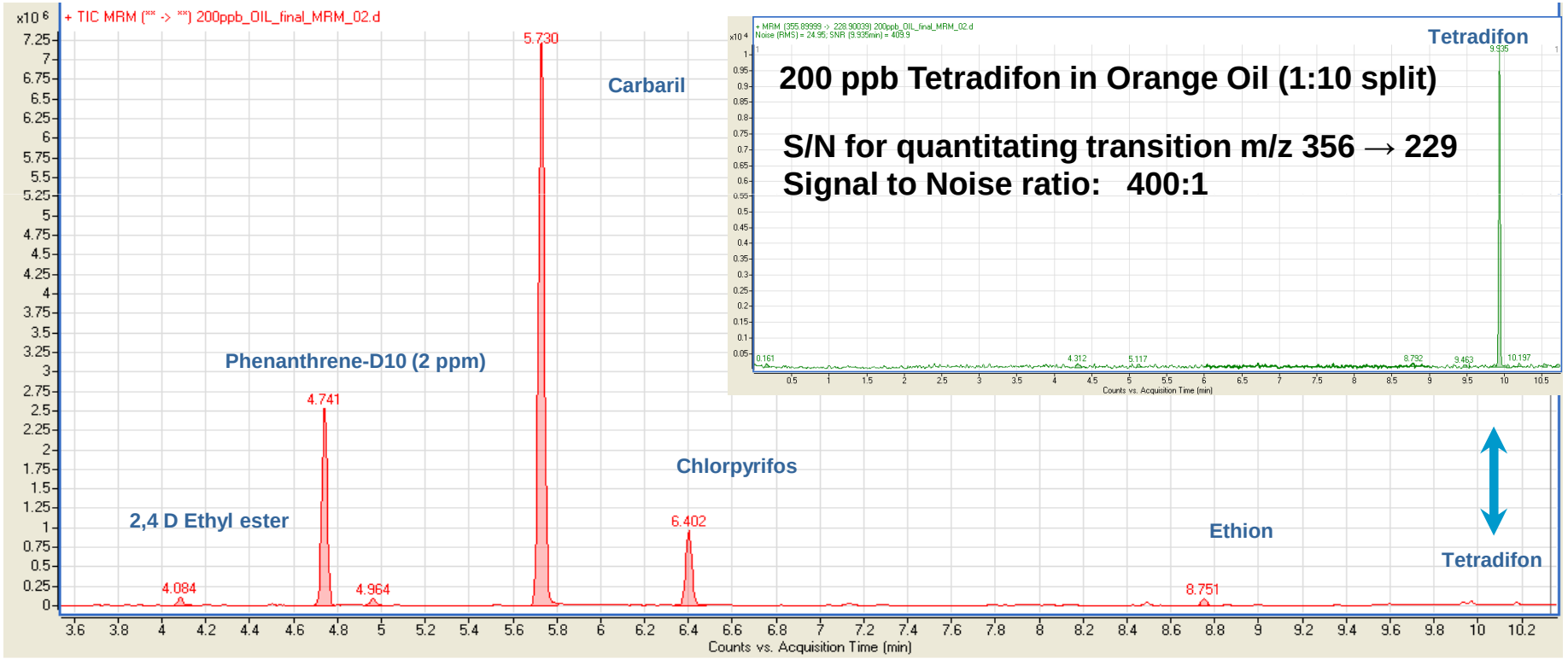
Column:	**DB-5ms Ultra Inert**
	122-5532UI
	30m x 0.25 mm x 0.25 µm
Carrier:	Hydrogen, 53 cm/sec, constant flow
Flow Program (mL/min):	1.6 for 11 min
	1.6 to 2.4 at 60 mL/min² hold 2 min
	2.4 to 5.0 at 50 mL/min² hold 9 min
Oven:	170 ℃ for 3.2 min
	170-250 ℃ at 24.7 ℃/min, hold 5.3 min
	250-280 ℃ at 18.6 ℃/min, hold 4.0 min
	280-325 ℃ at 50.0 ℃/min, hold 4 min
Injection:	Pulsed Splitless, 280 °C
	20 psi pulse pressure for 0.38 min
	50 mL/min purge at 0.40 min
Detector:	Direct Connect liner G1544-80730 FID, 350 ℃
Sample:	5-10 ng on column

1 Medazepam
2 Halazepam
3 Oxazepam
4 Lorazepam
5 Diazepam
6 Desalkyl Aurazepam
7 Nordazepam
8 Clobazam
9 Oxazolam
10 Temazepam
11 Flumitrazepam
12 Bromozepam
13 Prazam
14 Lormetazepam
15 Nitrazepam
16 Chlordiazepoxide
17 Clonazepam
18 Demoxepam
19 Estazolam
20 Albrazolam
21 Trazolam



Pesticides in Orange Oil

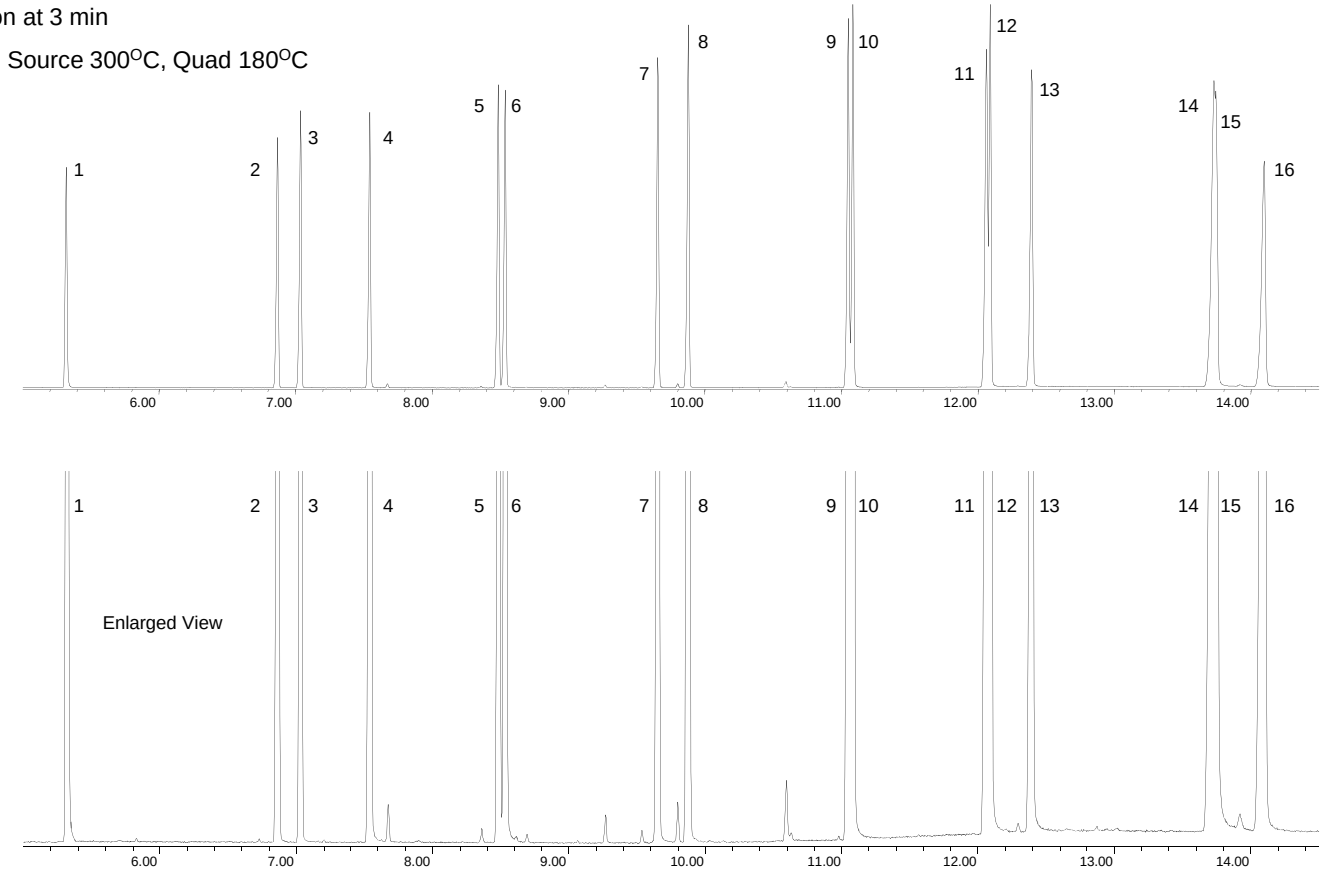
Analysis was carried out on the Agilent 7890A/5975 GC/MS or 7890A/7000 GC/MS/MS equipped with either a 7683 or 7683B Series ALS, split/splitless injection port and triple-axis detector. An Agilent J&W DB-5ms Ultra Inert 15 m x 0.25 mm x 0.25 um column (Agilent part # 122-5512UI) was used. The initial GC oven temperature was 70° C, which was held for 0.67 minutes. The oven was then ramped by 75° C/minute to 150° C, held for 0 minutes and ramped by 9° C/minute to 200° C and held for 0 minutes before ramping by 24° C/minute to 280° C and holding for 3 minutes. A six-minute post-run at 320° C was used. Pressure was held constant at 10 psi throughout the run and a split ratio of 10:1 for a 1uL injection. An open ended 4 mm helical liner was used (Agilent #5188-5396). The inlet temperature was 250° C and transfer line was set to 280° C. In the case of both detectors the source temperature was set to 300° C and the analyzer to 180° C.



PAH Analysis

GC/MSD Conditions

Sample:	10ug/ml PAH Standard
Column:	DB-5ms Ultra Inert 30m x 0.25mm x 0.25um **(Agilent part # 122-5532UI)**
Carrier:	Helium 45cm/sec, constant flow
Oven:	55°C (1min) to 320°C (25°/min), hold 3 min
Injection:	Pulsed splitless, 300°C, 40psi until 0.2 min, purge flow 30ml/min at 0.75 min
	Gas saver 80ml/min on at 3 min
MSD:	Transfer Line 280°C , Source 300°C, Quad 180°C

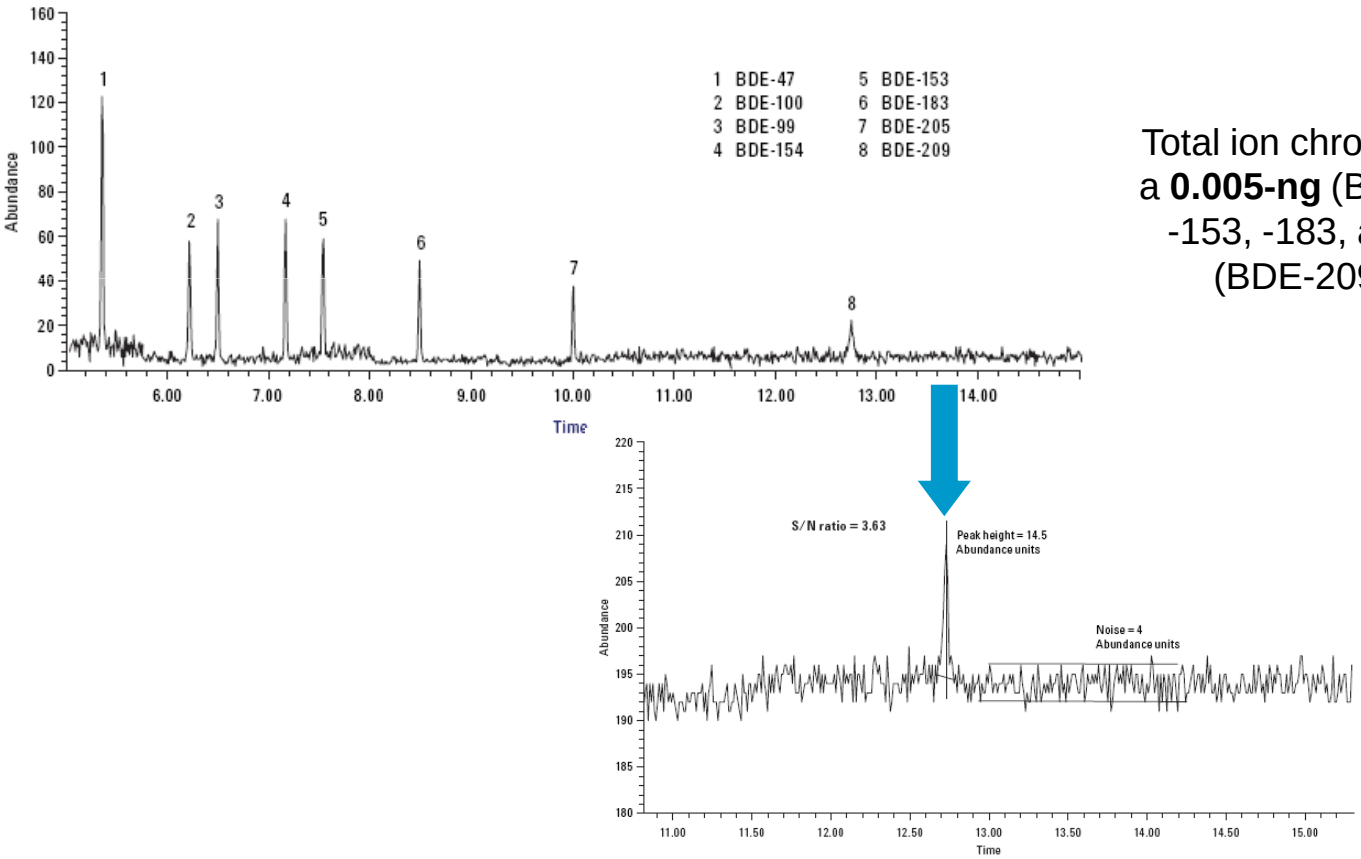


1. **Naphthalene**
2. **Acenaphthylene**
3. **Acenaphthene**
4. **Fluorene**
5. **Phenanthrene**
6. **Anthracene**
7. **Fluoranthene**
8. **Pyrene**
9. **Benz[a]anthracene**
10. **Chrysene**
11. **Benz[b]fluoranthene**
12. **Benz[k]fluoranthene**
13. **Benz[a]pyrene**
14. **Indeno[1,2,3-cd]pyrene**
15. **Dibenzo[a,h]anthracene**
16. **Benzo[g,h,i]perylene**

PBDE Analysis

GC/MS conditions

Column:	DB-5ms Ultra Inert 15 m × 0.25 mm × 0.25 µm (Agilent part # 122-5512UI)
Carrier:	Carrier Helium 72 cm/s, constant flow
Oven:	150 to 325 ℃ (17 ℃/min), hold 5 min
Injection:	Pulsed splitless; 325 ℃, 20 psi until 1 .5 min, purge flow 50 mL/min at 2.0 min
MSD:	Source at 300 ℃, Quadrupole at 150 ℃, transfe r line at 300 ℃, scan range 200–1000 amu



Total ion chromatogram (SIM mode) of a **0.005-ng** (BDEs -47, -100, -99, -154, -153, -183, and -205) and **0.025-ng** (BDE-209) on-column loading

Enlarged section of BDE-209 with excellent sensitivity for trace level analysis

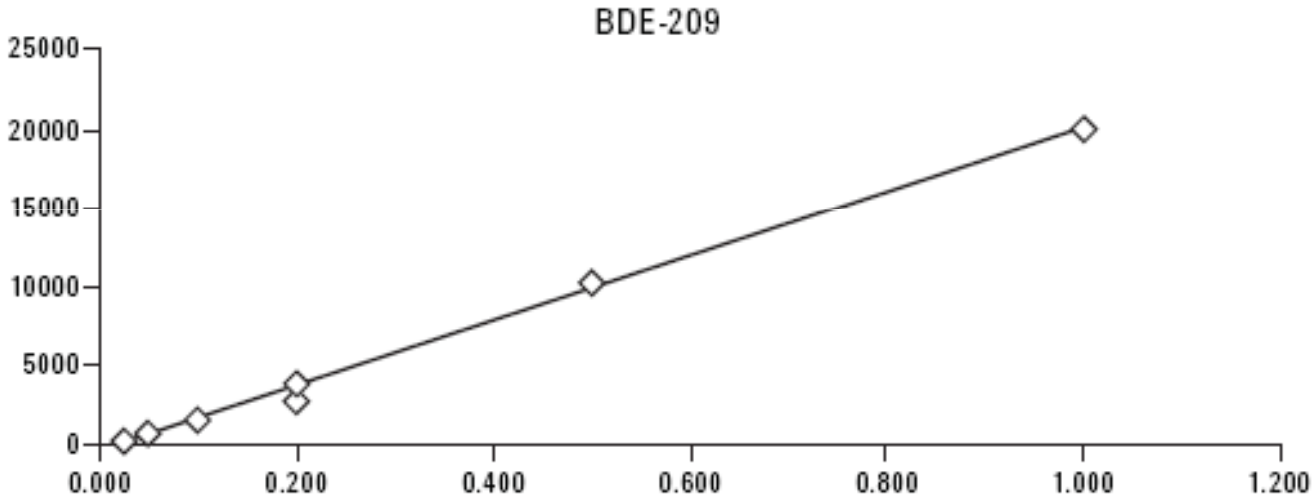
Agilent Technologies

PBDE Analysis

GC/MS conditions

Column:	DB-5ms Ultra Inert 15 m × 0.25 mm × 0.25 µm (Agilent part # 122-5512UI)
Carrier:	Carrier Helium 72 cm/s, constant flow
Oven:	150 to 325 ℃ (17 ℃/min), hold 5 min
Injection:	Pulsed splitless; 325 ℃, 20 psi until 1 .5 min, purge flow 50 mL/min at 2.0 min
MSD:	Source at 300 ℃, Quadrupole at 150 ℃, transfe r line at 300 ℃, scan range 200–1000 amu

BDR	R^2
47	0.999
100	0.999
99	0.999
154	0.999
153	1.000
183	0.999
205	0.999
209	0.997

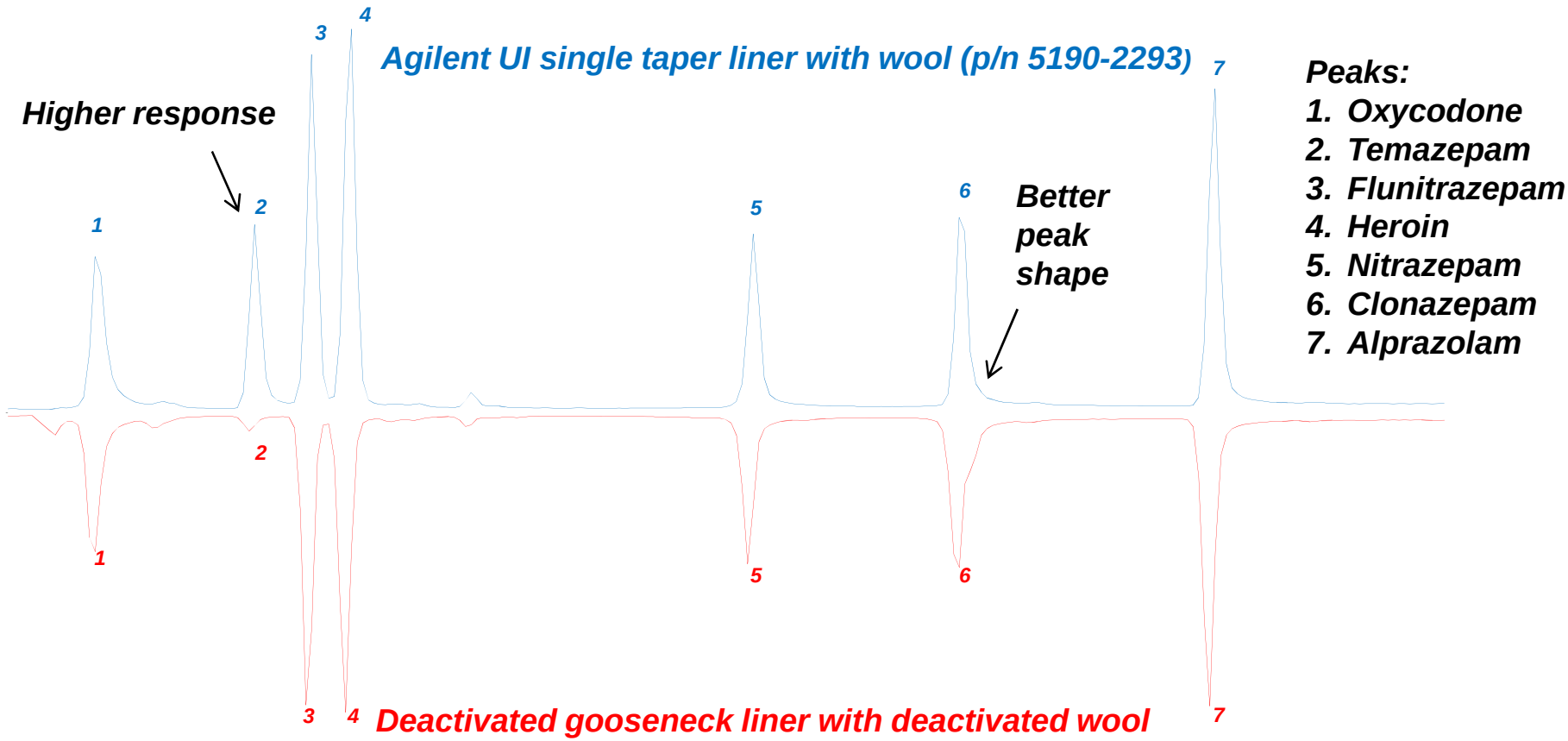


Linearity is excellent across the range studied (0.5 ng/mL to 1,000 ng/mL, except for BDE-209 at 2.5 to 1,000 ng/mL range), giving R^2 values of 0.997 or greater in all cases and demonstrating highly inert surface of the column.

We Have the Most Inert Column, What about the Rest of the Flow Path??

Ultra Inert Liners

Basic Drug Suitability



Drug of abuse are shown on GC/MS SIM chromatograms 5 ng of checkout standards on column

Ultra Inert GC Inlet Liners Objective:

Equivalent *or* *Better* performance for :

Response Levels: Highly inert surface for recovery at *trace levels*

Robustness: Stability of deactivation over time

Reliability: Reproducibility and linearity

Even when containing glass wool, Agilent ***Ultra Inert GC liners*** *provide a robust, reproducible and reliable inert flow path for the analysis of difficult, active compounds at trace levels.*

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 permenantly etched on glass

Take Home Message

- Raising the bar and setting a new industry standard for column inertness QC testing
- Best columns available for reactive analytes and trace analysis
  - Carefully selected Ultra Inert test mix for consistent column inertness
  - Excellent performance over a wide range of applications
  - Quality innovation to ensure the right answer the 1st time
  - Selectivity remains the same for consistent predicable separation
  - Low bleed profiles minimize interferences
- New Inert Liners to keep the entire flowpath ***INERT***
- **The bottom line**
  - **Highest and most consistent inertness performance**

Always Quality.
Always Innovative.
Always Agilent.

www.agilent.com/chem/ultrainert

References

1. US EPA Method 8270D Revision 4 February 2007 “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”

2. K. Grob Jr., G. Grob, and K. Grob “Comprehensive, Standardized Quality Test for Glass Capillary Columns,” *Journal of Chromatography A*, Vol. 156, Issue 1, 21 August 1978, Pages 1-20

3. Mitch Hastings, Allen K. Vickers, and Cameron George “Inertness Comparison of Sample of 5% Phenyldimethylpolysiloxane Columns,” Poster Presentation, 54th Annual Pittsburg Conference, Orlando, FL March 2003

4. Jim Luong, Ronda Gras, and Walter Jennings “An Advanced Solventless Column Test for Capillary GC Columns,” *J. Sep. Sci.*, 2007, 30, 2480-2492

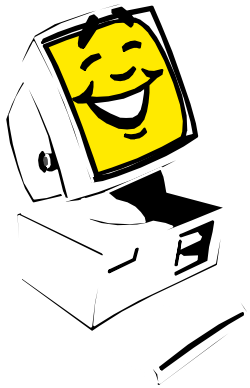
5. Mike Szelewski and Bill Wilson, “Improvements in the Agilent 6890/5973 GC/MSD System for Use with USEPA Method 8270,” Application Note 5988-3072EN, November 7, 2001

Agilent/J&W Technical Support

*800-227-9770 (phone: US & Canada)**

**** Select option 3..3..1***

866-422-5571 (fax)



email: gc-column-support@agilent.com

www.agilent.com/chem