



Highly Inert Capillary GC Columns: Less Activity, Better Peak Shape, and more Sample Signal



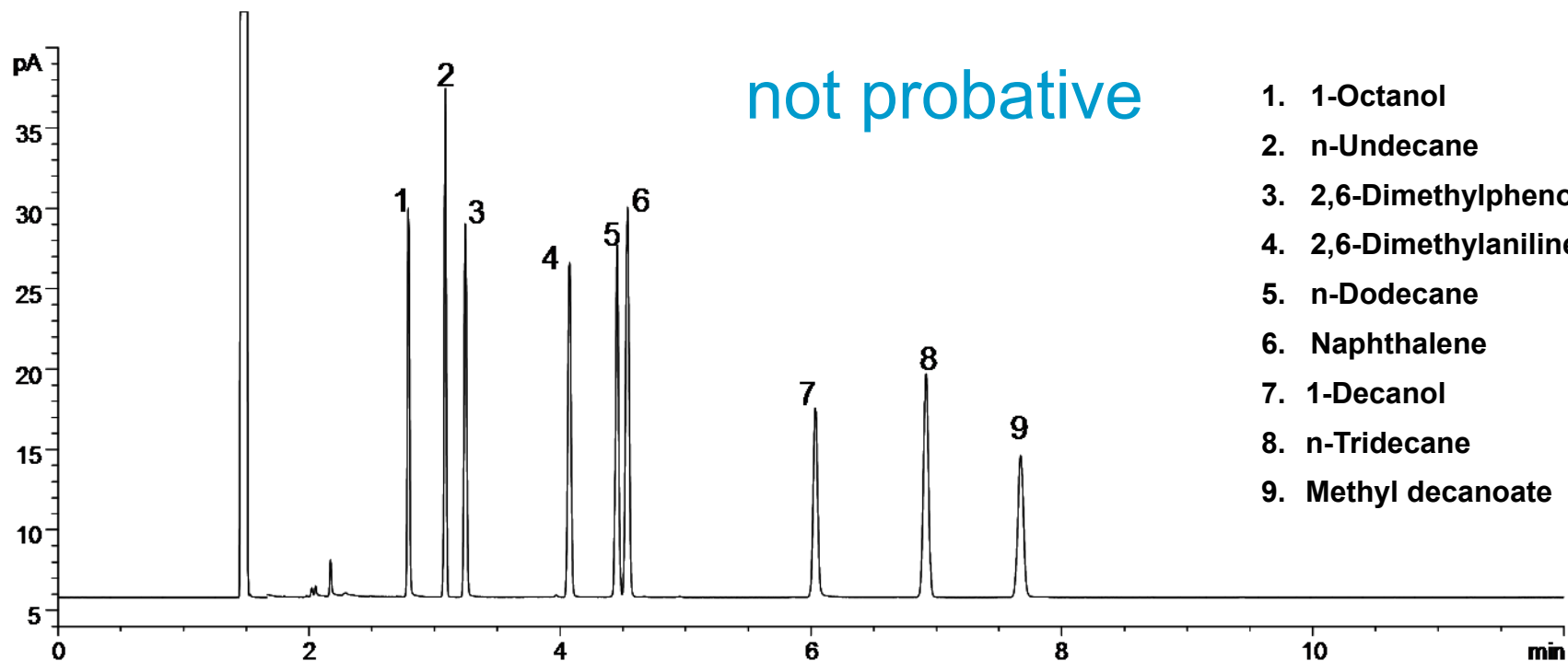
Agilent J&W Ultra Inert Capillary GC Columns



Presentation Outline

- Back ground, Evolution of capillary column QC
 - Grob's type mix, test for the 80
 - "DB-5ms mix", test for the 90s
 - "Ultra Inert" mix, test for today
- Best uses for Ultra Inert columns
- Application examples
- Take away messages

Grob-type Test Mix Results on a DB-5ms Ultra Inert



Sampler: Agilent 7683B, 5 μ L syringe (Agilent part # 5181-1273), 1.0 μ L split injection, 4 ng each component

Carrier: Hydrogen constant pressure 37 cm/s

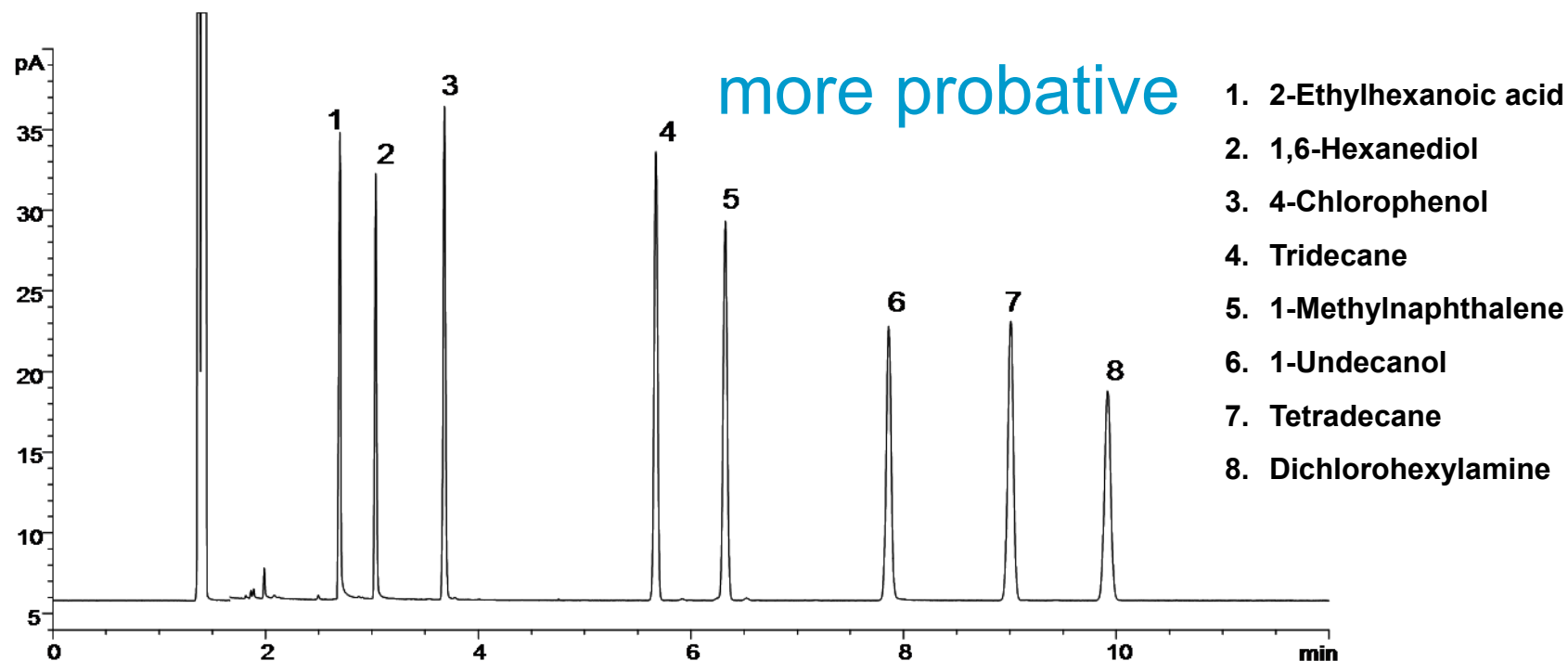
Inlet: Split/splitless; 250 $^{\circ}$ C, 1.4 ml/min. column flow, split flow 140 ml/min.

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

Oven: 120 $^{\circ}$ C isothermal

Detection: FID at 325 $^{\circ}$ C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

DB-5ms Mix on DB5-ms Ultra Inert



Sampler: Agilent 7683B, 5 μ L syringe (Agilent part # 5181-1273), 1.0 μ L split injection, 4 ng each component

Carrier: Hydrogen constant pressure 38 cm/s

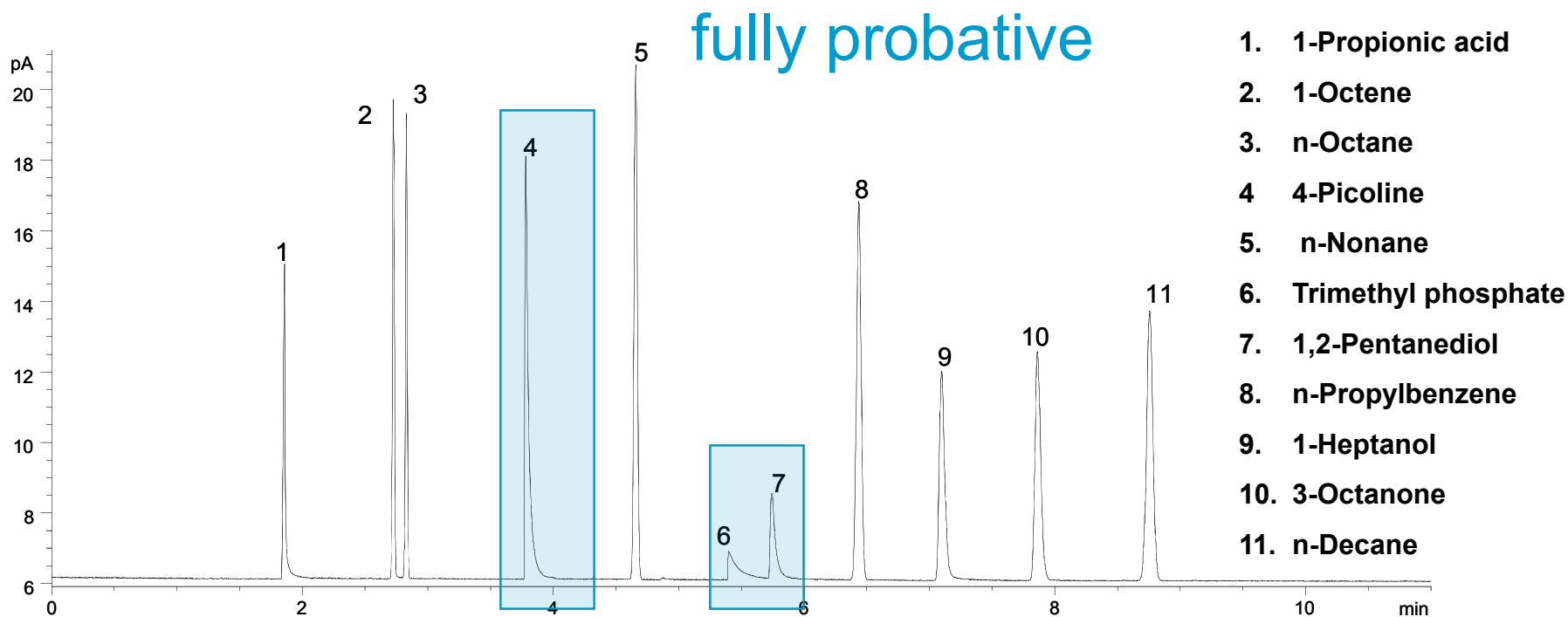
Inlet: Split/splitless; 250 $^{\circ}$ C, 1.4 ml/min. column flow, split flow 100 ml/min.

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

Oven: 125 $^{\circ}$ C isothermal

Detection: FID at 320 $^{\circ}$ C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup

UI Mix Results on a Brand X “Premium” Column



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

Carrier: Hydrogen constant pressure, 38 cm/s

Inlet: Split/splitless; 250 $^{\circ}$ C, 1.4 ml/min. column flow, split flow 900 ml/min., gas saver flow 75 ml/min. on at 2.0 min.

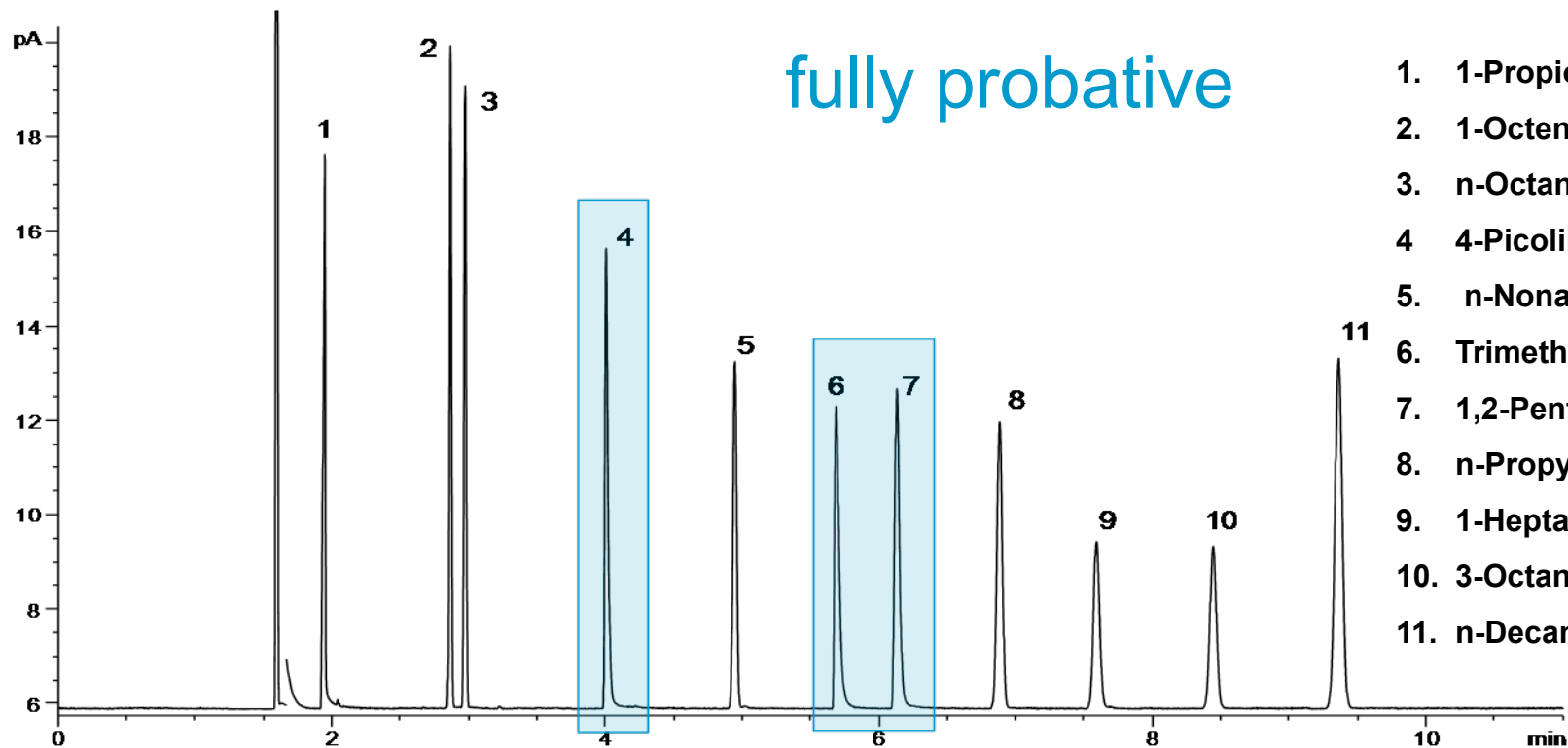
Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)

Oven: 65 $^{\circ}$ C isothermal

Detection: FID at 325 $^{\circ}$ C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min., nitrogen makeup



UI Mix Results on an 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

Sampler: Agilent 7683B, 0.5 μ L syringe (Agilent part # 5188-5246), 0.02 μ L split injection
Carrier: Hydrogen constant pressure, 38 cm/s
Inlet: Split/splitless; 250 $^{\circ}$ 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 $^{\circ}$ C isothermal
Detection: FID at 325 $^{\circ}$ C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min., nitrogen makeup



Test Mix Observations

- Grob's style mix not probative for inertness
- DB-5ms text mix good test for the 90s
- UI 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

Best use for Ultra Inert columns

- Active analyte analysis
- Trace and ultra trace analysis
- Critical samples
- Unknowns

Select Ultra Inert Application Examples

- Drugs of Abuse
- Semi Volatile Analysis
- PAHs
- Pesticides
- Miscellaneous

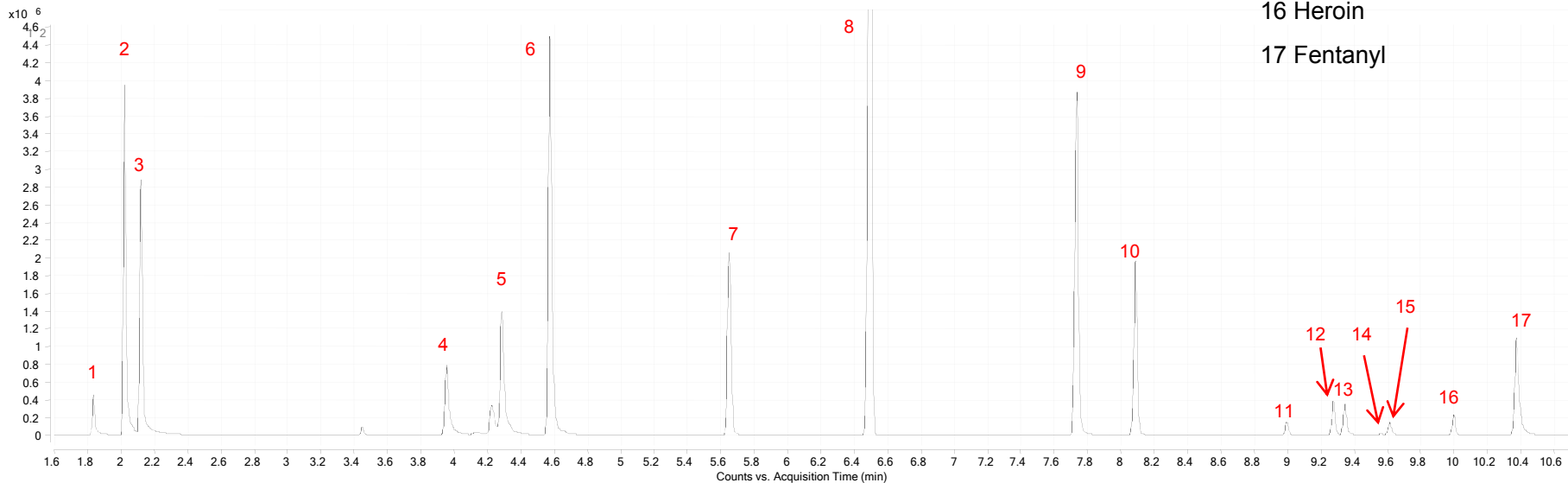
Drugs of Abuse Application:

DB-5ms Ultra Inert 15 m x 0.25 mm x 0.25 μm column

TIC of the Agilent 7000A Triple Quad
GC/MS system in MRM mode.
Standard solution of 1 ng/uL.

Peak identifications:

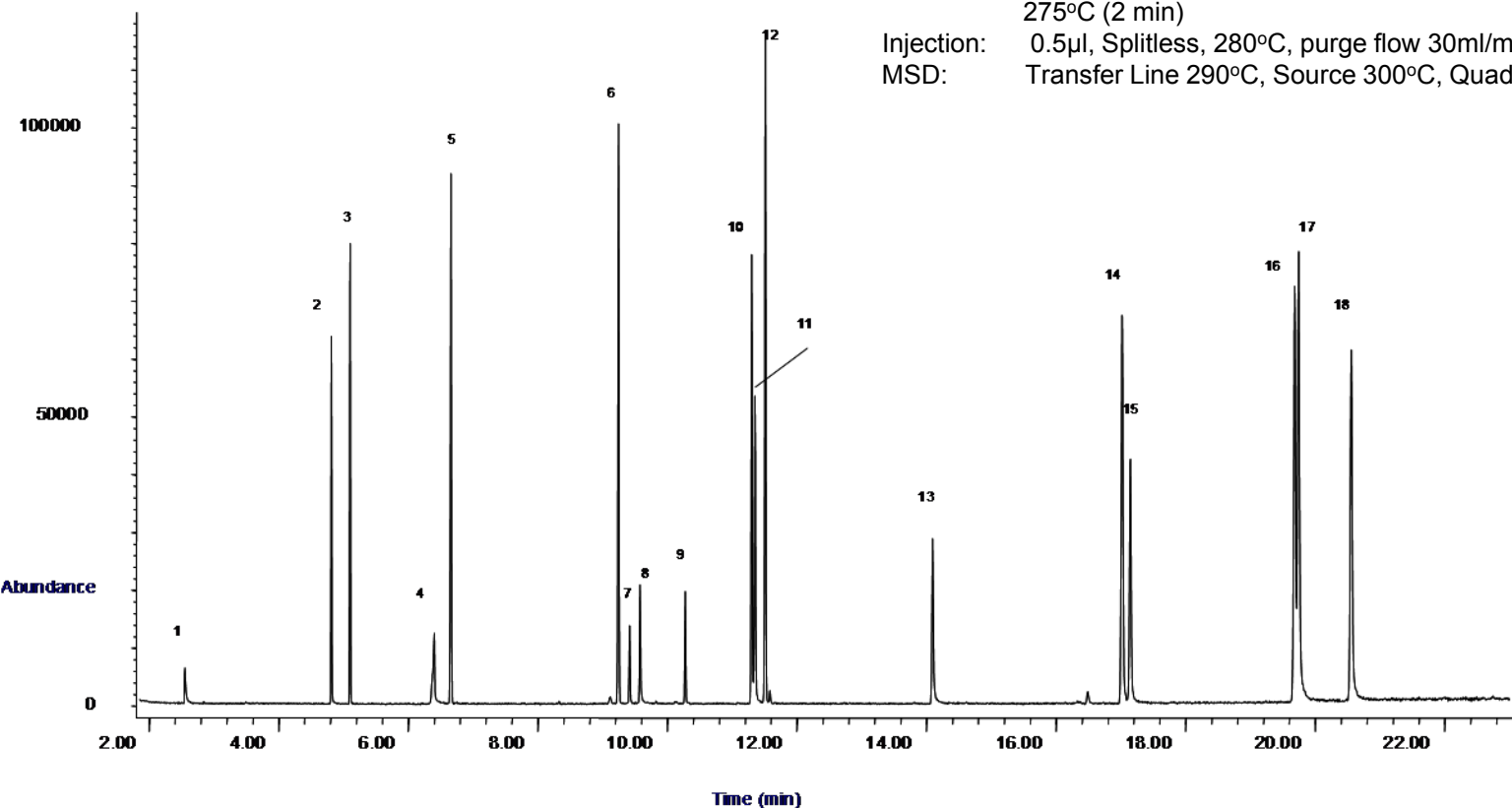
1 Amphetamine	6 MDEA	11 Codeine
2 Phentermine	7 Meperidine	12 Hydrocodone
3 Methamphetamine	8 PCP (phencyclidine)	13 THC
4 MDA	9 Methadone	14 6-Acetylmorphine
5 MDMA (ecstasy)	10 Cocaine	15 Oxycodone
		16 Heroin
		17 Fentanyl



Semi volatiles Application:

HP-5ms Ultra Inert 20m x 0.18 mm x 0.18 μm column

Sample: 0.5 ng on column loading of Short Mix components with ISTD
Column: Agilent J & W HP-5ms Ultra Inert 20m 0.18mm 0.18 μm
Carrier: Helium 37cm/sec, Ramped flow; 0.7ml/min (0.1min) to 1.3ml/min (15ml/min²)
Oven: 35°C (2.5 min) to 80°C (40°C/min), 15°C/min to 200°C, 8°C/min to 275°C (2 min)
Injection: 0.5 μl , Splitless, 280°C, purge flow 30ml/min at 0.75 min
MSD: Transfer Line 290°C, Source 300°C, Quad 180°C



1. n-Nitrosodimethylamine
2. Aniline
3. 1,4-Dichlorobenzene-d4
4. Benzoic Acid
5. Naphthalene-d8
6. Acenaphthene-d10
7. 2,4-Dinitrophenol
8. 4-Nitrophenol
9. 2-Me-4,6-dinitrophenol
10. 4-Aminobiphenyl
11. Pentachlorophenol
12. Phenanthrene-d10
13. Benzidine
14. Chrysene-d12
15. 3,3'-Dichlorobenzidine
16. Benzo[b]fluoranthene
17. Benzo[k]fluoranthene
18. Perylene-d12

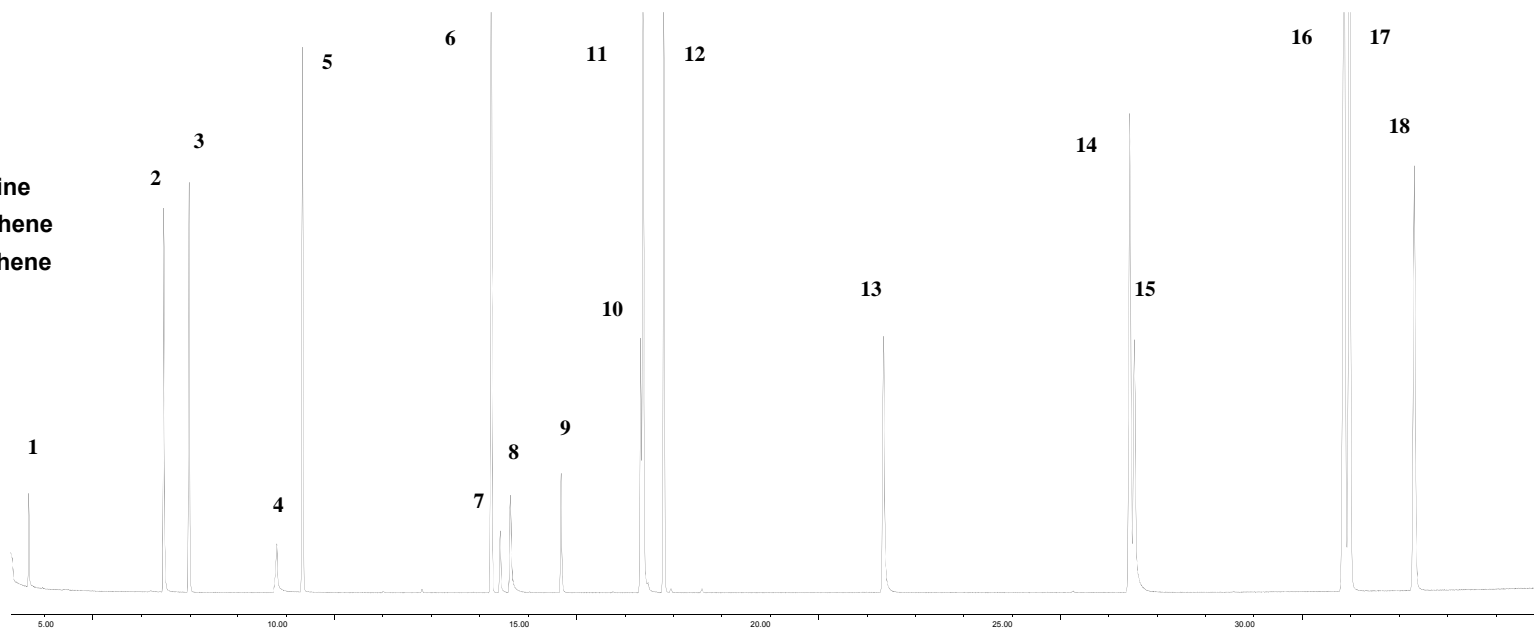


Semi Volatiles Application:

DB-5ms Ultra Inert 30m x 0.25mm x 0.25 μ m column

1. N-nitrosodimethylamine
2. Aniline
3. 1,4 dichlorobenzene-D4
4. Benzoic acid
5. Naphthalene- D8
6. Acenaphthene-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

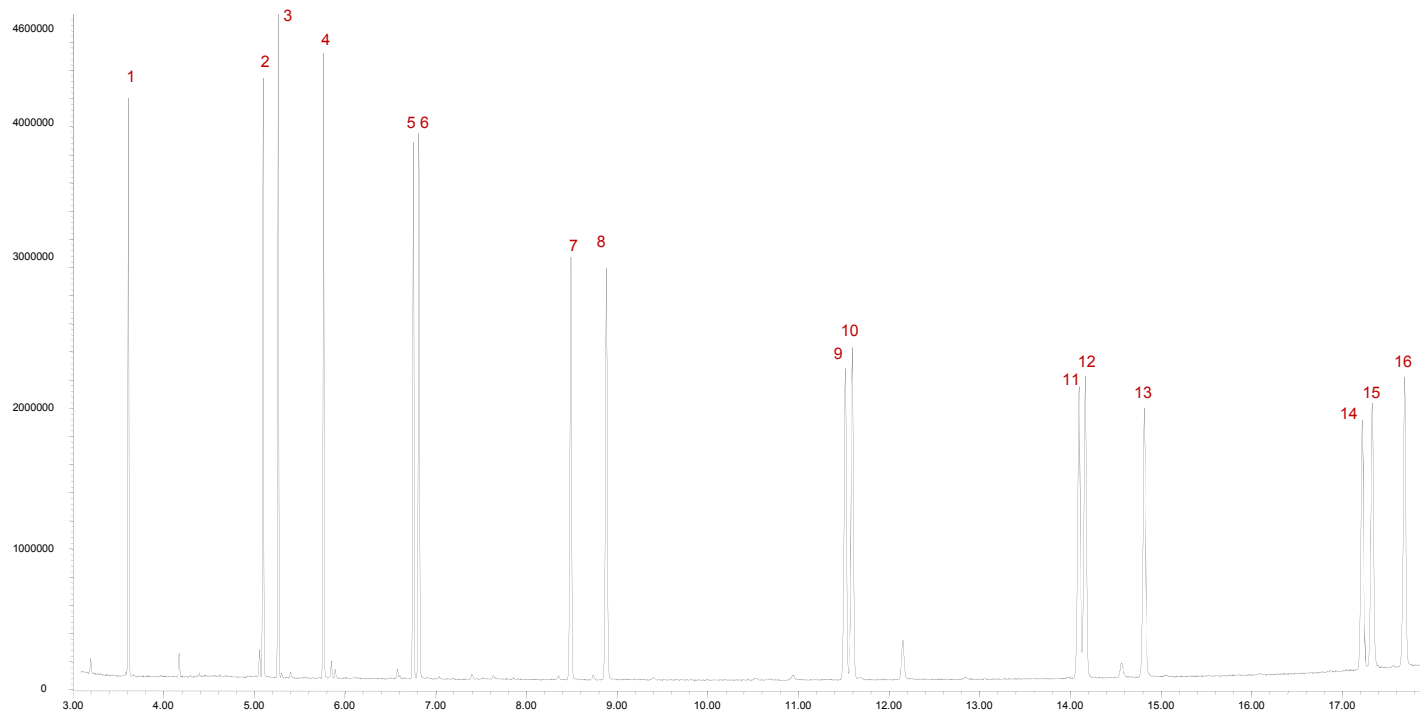
GC : Agilent 6890N/5975B MSD
Sampler : Agilent 7683B, 5.0 μ L syringe (Agilent part # 5188-5246), 1.0 μ L splitless injection, 5 ng on column
Carrier: Helium constant flow 30 cm/s
Inlet: Split/splitless; 260% C, 53.7 ml/min. total flow, purge flow 50 ml/min. on at 0.5 min., gas saver off
Inlet Liner: Deactivated single taper w glass wool (Agilent part # 5183-4647)
Column: Agilent J &W DB-5ms Ultra Inert 30m x 0.25mm x 0.25 μ m (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



PAH Application: DB-5ms Ultra Inert 20 m x 0.18 mm x 0.18 µm

GC/MSD Conditions

Sample: 2.5 µg/mL EPA PAHs
Column: DB-5ms UI 20 m x 0.18 mm x 0.18 µm (US8766313J)
Carrier: He 53.3 cm/sec constant flow
Oven: 55° C (0.25 min) to 200° C (25° C/min), 8° C/min to 280° C, 10° C/min to 320° C (2 min)
Inlet: splitless 300° C purge flow 60 ml/min at 0.2 min, switched septum purge 6 ml/min
MSD: transfer line 340° C, source 340° C, quad 180° C (TAD)

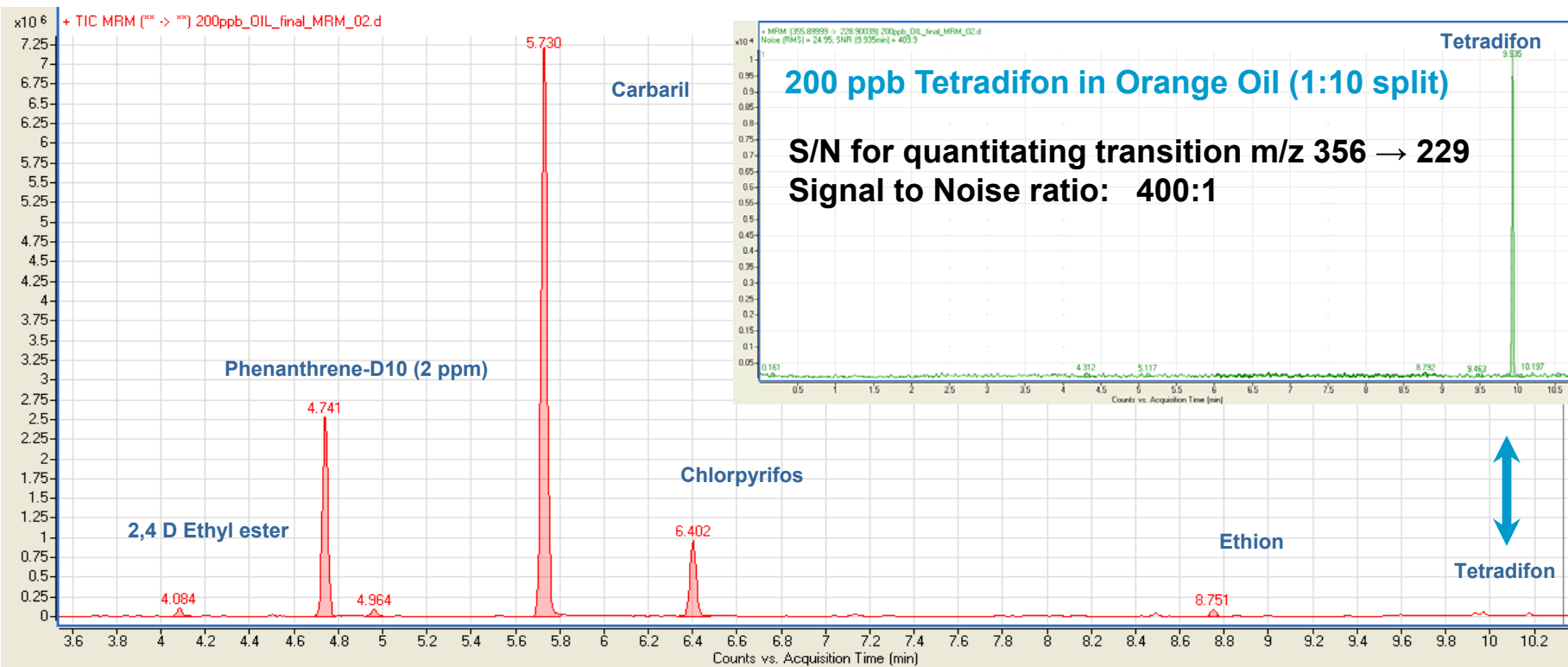


Peak #	Component
1	Naphthalene
2	Acenaphthylene
3	Acenaphthene
4	Fluorene
5	Anthracene
6	Phenanthrene
7	Fluoranthene
8	Pyrene
9	Benz[a]anthracene
10	Chrysene
11	Benzo[b]fluoranthene
12	Benzo[k]fluoranthene
13	Benzo[a]pyrene
14	Indeno[1,2,3-cd]pyrene
15	Dibenz[a,h]anthracene
16	Benzo[g,h,i]perylene

Pesticides in Orange Oil Application:

DB-5ms Ultra Inert 15 m x 0.25 mm x 0.25 μ m column

Analysis was carried out on the Agilent 7890A/5975 GC/MS or 7890A/7000 A (Prototype) 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 μ m 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 1 μ L 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.



Lavender Oil Application:

DB-1ms Ultra Inert 30 m x 0.25 mm x 0.25 μm column

Sample: Lavender Oil 1:20 in acetone

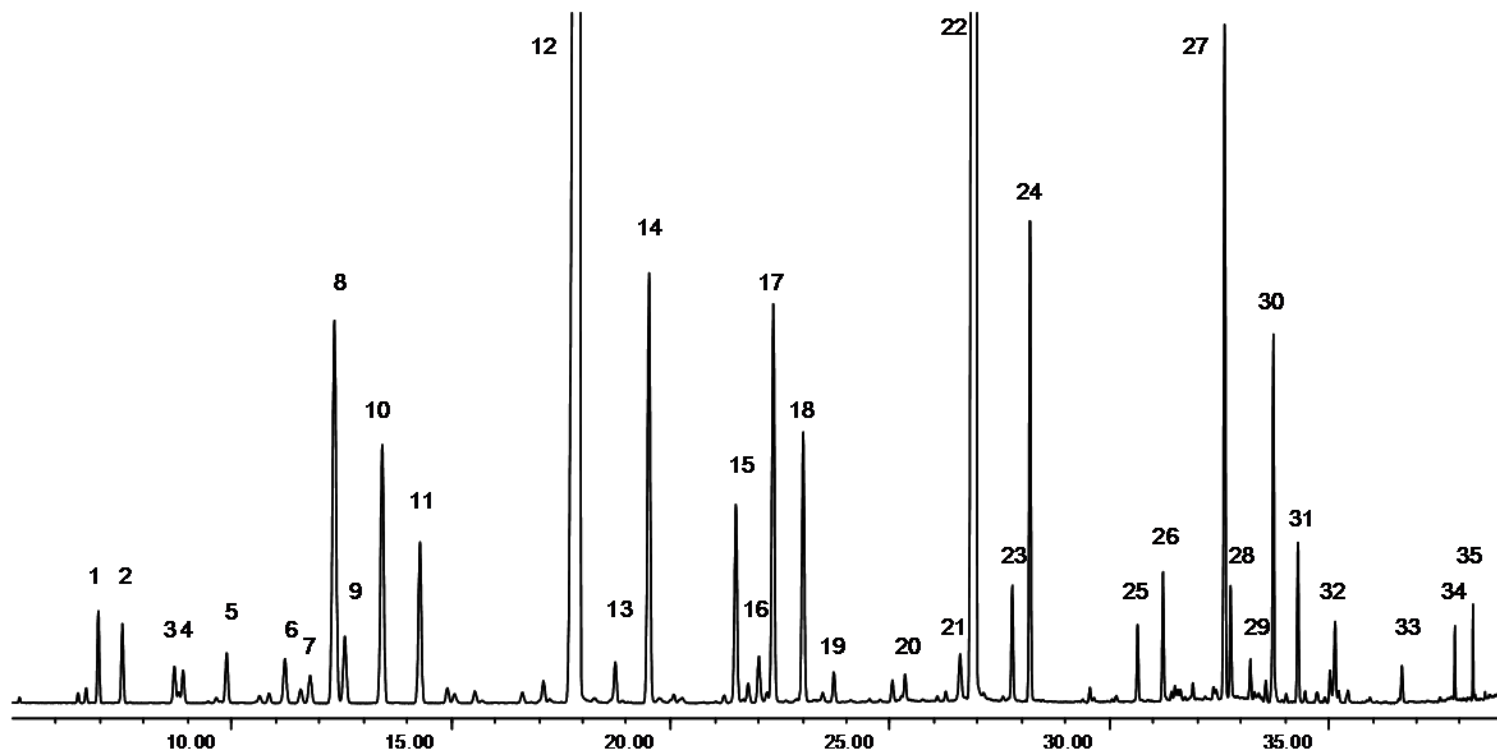
Column: DB-1ms UI 30 m x 0.25 mm x 0.25 μm

Carrier: He 40 cm/sec constant flow

Oven: 62°C (12.5min) to 95°C (3°C/min), 5°C/min to 165°C, 100°C/min to 310°C (2.5 min)

Inlet: 250°C 1 μl injection, split 200:1, gas saver 50 ml/min on at 2.0 minutes

MSD 300°C source, 180°C quad, 280°C transfer line temperatures scanning mode



- 1 α Pinene
- 2 Camphene
- 3 1-Octen-3-ol
- 4 3-Octanone
- 5 β Myrcene
- 6 3-Carene
- 7 o-Cymene
- 8 Eucalyptol
- 9 D Limonene
- 10 β Trans-Ocimene
- 11 β Cis-Ocimene
- 12 β Linalool
- 13 Octen-1-ol acetate
- 14 Camphor
- 15 Borneol
- 16 Lavandulol
- 17 Terpine-4-ol
- 18 α Terpinol
- 19 hexyl butyrate
- 20 cumic aldehyde
- 21 cis Geraniol
- 22 Linalool acetate
- 23 Borneol acetate
- 24 lavandulyl acetate
- 25 Nerol acetate
- 26 Geranyl Acetate
- 27 Caryophyllene
- 28 α -Santoloene
- 29 α -Bergamotene
- 30 β Farnesene
- 31 Germacrene D
- 32 γ Cardinene
- 33 Caryophyllene oxide
- 34 tau Cardinol
- 35 α Bisabolol

Phthalates in Toys Application:

DB-5ms Ultra Inert 30 m x 0.25 mm x 0.25 μm column

GC Conditions

Column	Agilent J&W DB-5ms Ultra Inert capillary column, 30 m x 0.25 mm, 0.25 μm (p/n 122-5532UI)
Inlet Temperature	290 $^{\circ}\text{C}$
Carrier Gas	Helium at 1 mL/min
Injection Mode	Splitless, pulse injection at 35 psi for 0.5 min, splitless injection liner (Agilent p/n 5188-3316).
Injection Volume	1 μL
Oven Program	50 $^{\circ}\text{C}$ for 1 min to 280 $^{\circ}\text{C}$ at 30 $^{\circ}\text{C}/\text{min}$ to 310 $^{\circ}\text{C}$ at 15 $^{\circ}\text{C}/\text{min}$ hold for 4 min

Compound Name

Dimethyl phthalate (DMP)
Diethyl phthalate (DEP)
Benzyl benzoate (BB)**
Dibutyl phthalate (DBP)
Dihexyl phthalate (DHP)
Benzyl butyl phthalate (BBP)
Bis(2-n-butoxyethyl)phthalate (DBEP)
Bis(2-ethylhexyl)phthalate (DEHP)
Di-n-octyl phthalate (DNOP)
Di-isononyl phthalate (DINP)
Di-isodecyl phthalate (DIDP)

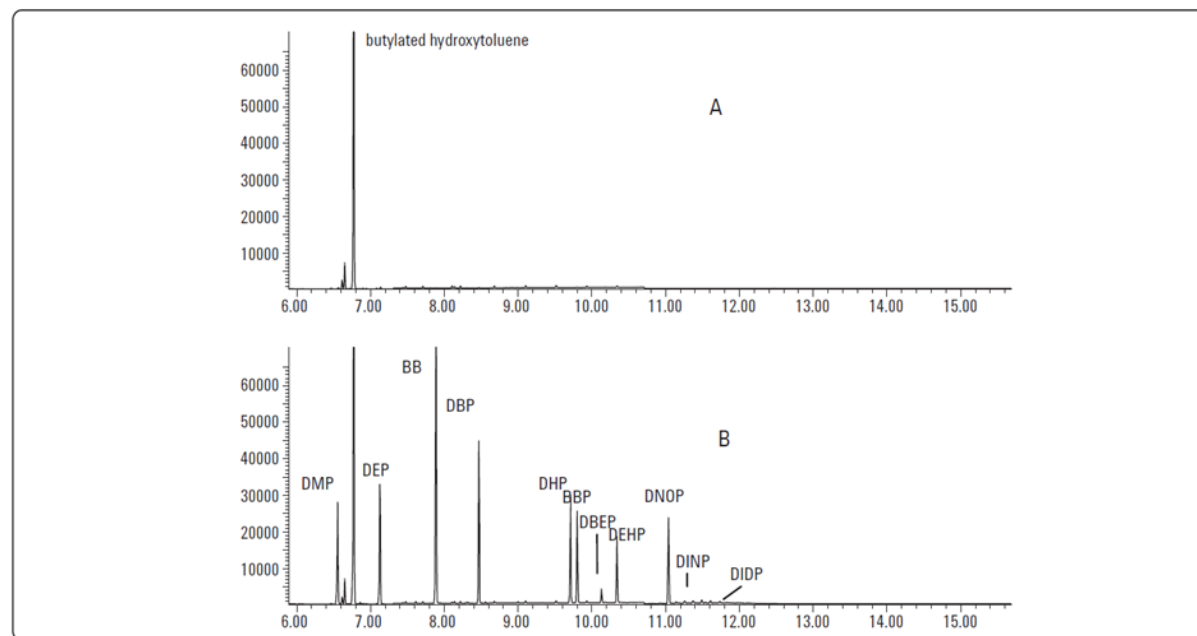


Figure 7. TIC of infant pacifier extract (A) and sample # 2 (pacifier) extract spiked with 2-ppm phthalate mixture (B).

Take Away Message

- Testing with aggressive probes is necessary for consistent inertness performance
- Use Ultra Inert columns for critical applications
- Excellent performance over a range of applications
- Best choice for trace level analysis
- **Ultra inert columns consistently deliver less activity, better Peak Shape, and more Sample Signal**



Thank You.

Questions?

