

# Hocheffiziente und hochsensitive GC-Säulen

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Säulen und Verbrauchsmaterialien



# Chromatographische Grundgleichung

## Auflösung

$$R_s = 1/4 \sqrt{N} \left[ \frac{(\alpha-1)}{\alpha} \right] \left( \frac{k}{1+k} \right)$$

$\alpha$  = Selektivität ← stationäre Phase

$N$  = Effizienz (Trennleistung) - Säulendurchmesser, Säulenlänge, Gastyp

$k'$  = Retention (Retentionsfaktor) - Filmdicke, Temperatur

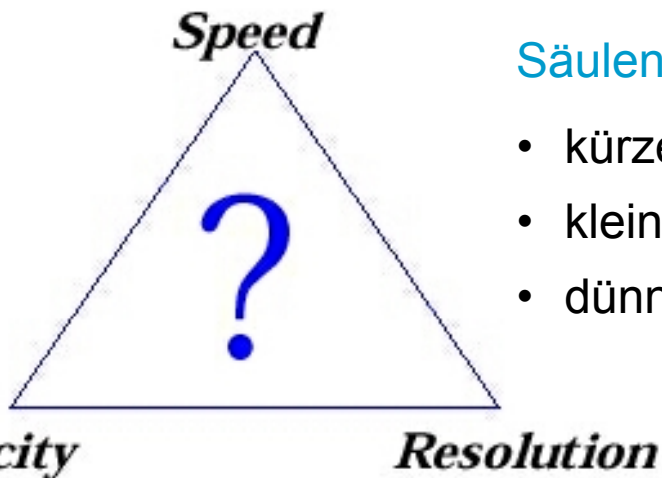
# Agilent GC Phasen

## Großer Selektivitätsbereich

- **Unpolare Phasen** - reine Siloxan- und substituierte Siloxanphasen
  - DB-1, HP-1, DB-5, HP-5, V4-1, VF-5, DB-1ht, DB-5ht, **ms-Phasen**, **HE-Phasen**, **UI Phasen**
- **Phasen mit mittlerer Polarität** - substituierte Siloxanphasen
  - DB-35, DB-35ms, DB-17, DB-200 DB-210, DB-624, DB-1301, DB-1701 **ms-Phasen**, **HE-Phasen**
- **Polare Phasen** - hoch substituierte Siloxanphasen und Polyethylenphasen
  - DB-23, DB-225, **HE-Phasen**
  - Polyethylen Glycol: HP-Innowax, DB-Wax, VF-Wax ms, DB-Wax etr, HP-FFAP, DB-FFAP, **HE-Phasen**
- **Säulendimensionen**
  - Id: 0.05 - 0.53 mm
  - Längen: 10 – 60 m bzw. bis 150 m

# Parameter für Geschwindigkeit und Auflösung

	Speed	Resolution	Capacity
Column length	Decrease	Increase	Increase
Internal diameter	Increase	Decrease	Increase
Film thickness	Decrease	Increase	Increase
Carrier gas	Helium	Nitrogen	-
	Hydrogen		
Velocity	Increase	Optimum	-
Temperature	Increase	Decrease	





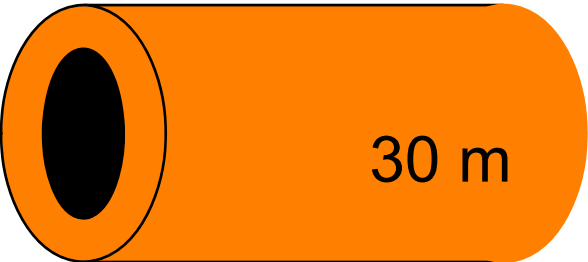
## Säulenparameter

- kürzere Säulen
- kleinerer Säulen ID
- dünnere Schicht

## Trennbedingungen

- anderes Trägergas
- höhere Trägergasgeschw.
- schnellere T-programmraten

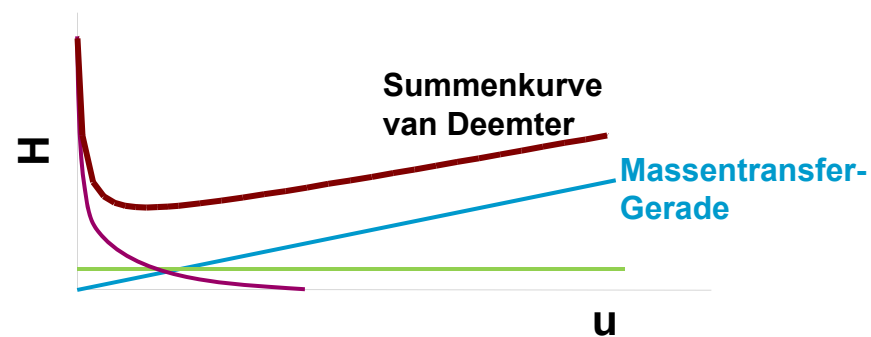
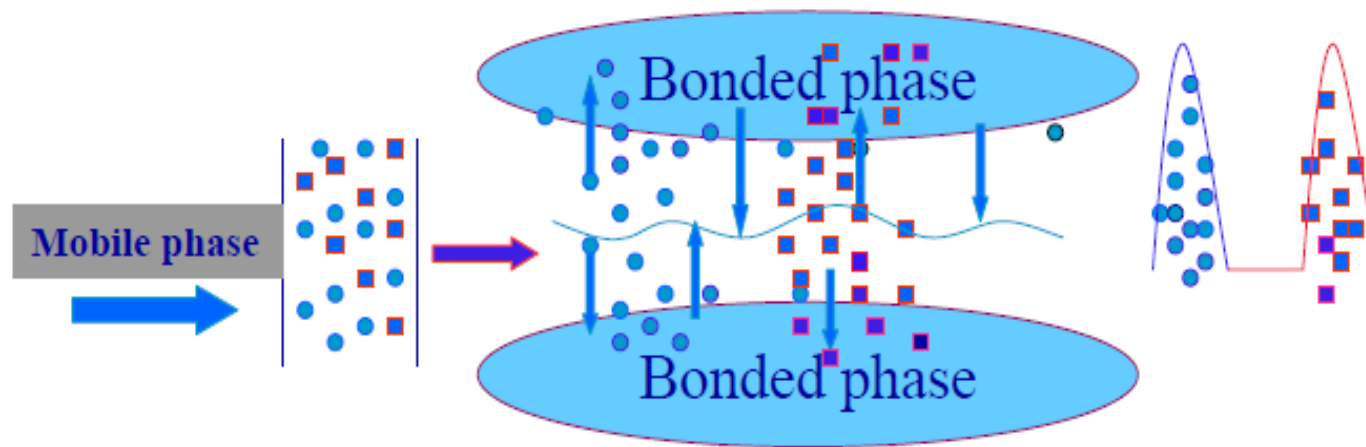
# Säulendurchmesser – Theoretische Trenneffizienz

	Total Plates	I.D. (mm)	n/m
 5 m	N ~ 112,000	0.05	23,160
 10 m	N ~ 112,000	0.10	11,580
<hr style="border-top: 1px dashed #e91e63;"/>			
	<b>High-efficiency GC</b>	0.18	6,660
 20 m	N ~ 112,000	0.20	5830
		0.25	4630
 30 m	N ~ 112,000	0.32	3660
		0.45	2840
		0.53	2060

k = 5

# Optimierung des Massentransfers

Verkürzung des Diffusionspfads zur Wand



# Probenkapazität

I.D. (mm)	Kapazität (ng)	Filmdicke (um)	Kapazität (ng)
0.05	1-2	0.10	50-100
0.10	6-13	0.25	125-250
<b>0.18</b>	<b>25-55</b>	1.0	500-1000
0.20	35-70	3.0	1500-3000
0.25	80-160	5.0	2500-5000
0.32	110-220	0.32 mm I.D.	
0.45	600-800		
0.53	1000-2000		

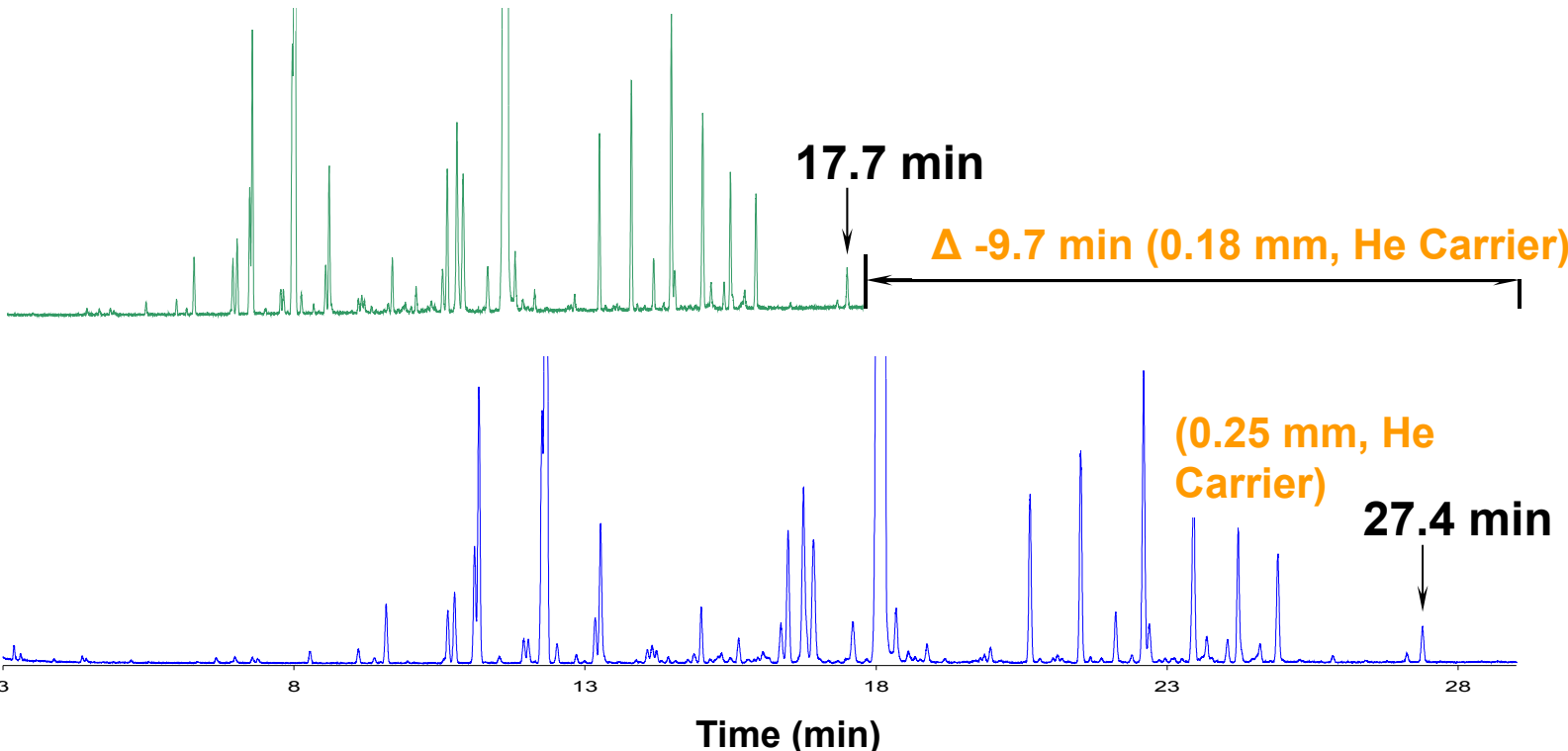
# Methodentransfer - Freeware

GC Method Translation - 530320.mxd

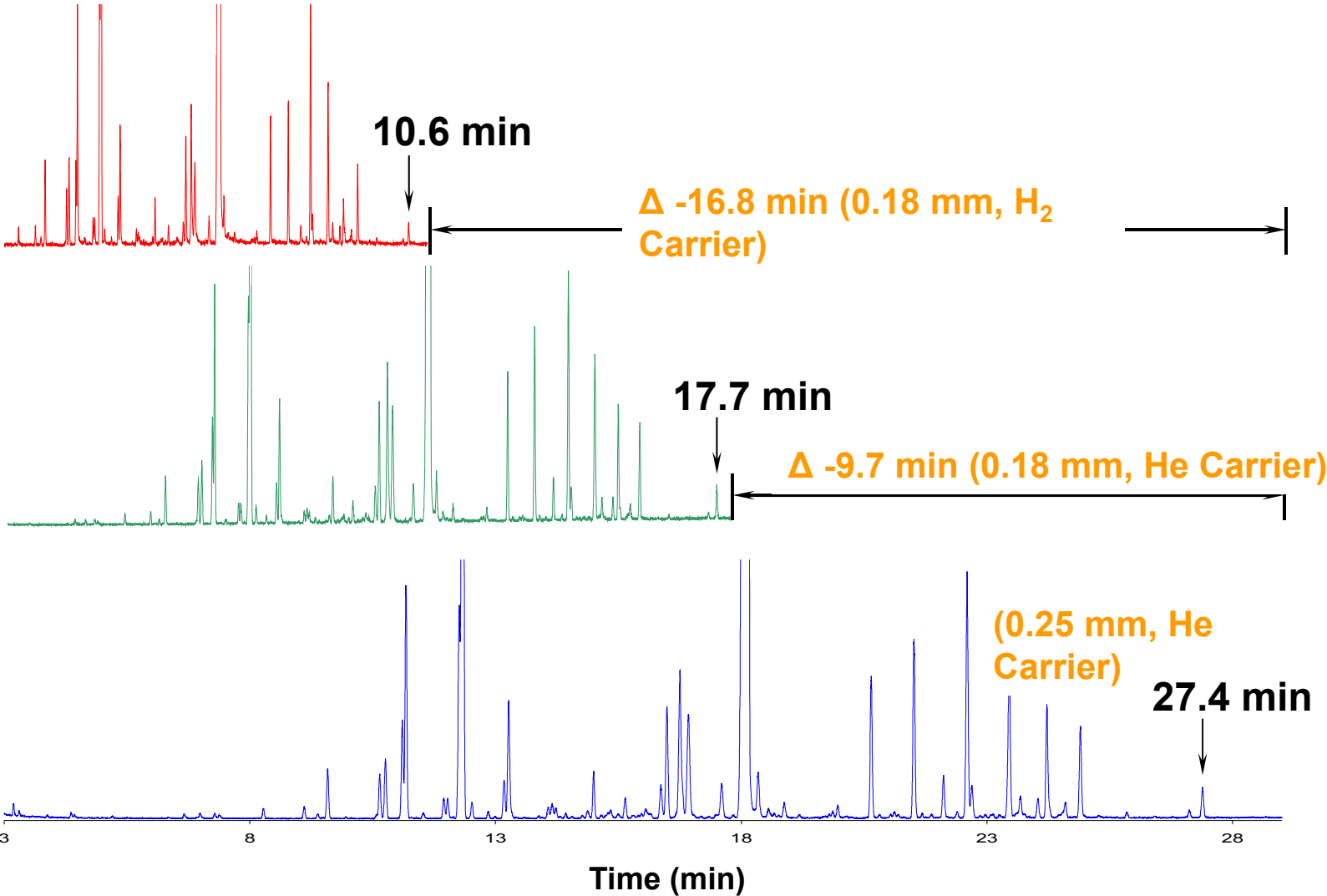
Criterion:  Translate Only  Best Efficiency  Fast Analysis  None **Speed gain: 2.27176**

	Original Method	Translated Method																								
<b>Column</b>																										
Length, m	30	<input type="checkbox"/> 18.2																								
Internal Diameter, $\mu\text{m}$	530	<input type="checkbox"/> 320																								
<b>Film</b>		<input checked="" type="radio"/> Unlock																								
Thickness, $\mu\text{m}$	0.88	<input checked="" type="radio"/> 0.52																								
Phase Ratio	150.6	<input type="radio"/> 153.8																								
<b>Carrier Gas</b>	Helium	<input type="checkbox"/> Helium																								
<b>Enter one Setpoint</b>		<input checked="" type="radio"/> Unlock																								
Head Pressure, psi	4.989	<input type="radio"/> 11.409																								
Flow Rate, mLn/min	5.3217	<input type="radio"/> 3.1646																								
Outlet Velocity, cm/sec	50.65	<input type="radio"/> 82.61																								
Average Velocity, cm/sec	43	<input type="radio"/> 58																								
Hold-up Time, min	1.16279	<input type="radio"/> 0.522989																								
Outlet Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																								
Ambient Pressure (absolute), psi	14.696	<input type="checkbox"/> 14.696																								
<b>Oven Temperature</b> 1-ramp Program																										
	<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th><math>^{\circ}\text{C}/\text{min}</math></th> <th><math>^{\circ}\text{C}</math></th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>100</td> <td>1</td> </tr> <tr> <td>4.5</td> <td>325</td> <td>15.000</td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		100	1	4.5	325	15.000	<table border="1"> <thead> <tr> <th>Ramp Rate</th> <th>Final Temp.</th> <th>Final Time</th> </tr> <tr> <th><math>^{\circ}\text{C}/\text{min}</math></th> <th><math>^{\circ}\text{C}</math></th> <th>min</th> </tr> </thead> <tbody> <tr> <td></td> <td>100</td> <td>0.440</td> </tr> <tr> <td>10.223</td> <td>325</td> <td>6.603</td> </tr> </tbody> </table>	Ramp Rate	Final Temp.	Final Time	$^{\circ}\text{C}/\text{min}$	$^{\circ}\text{C}$	min		100	0.440	10.223	325	6.603
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10.223	325	6.603																								
Initial																										
Ramp 1																										
<b>Sample Information</b> None																										

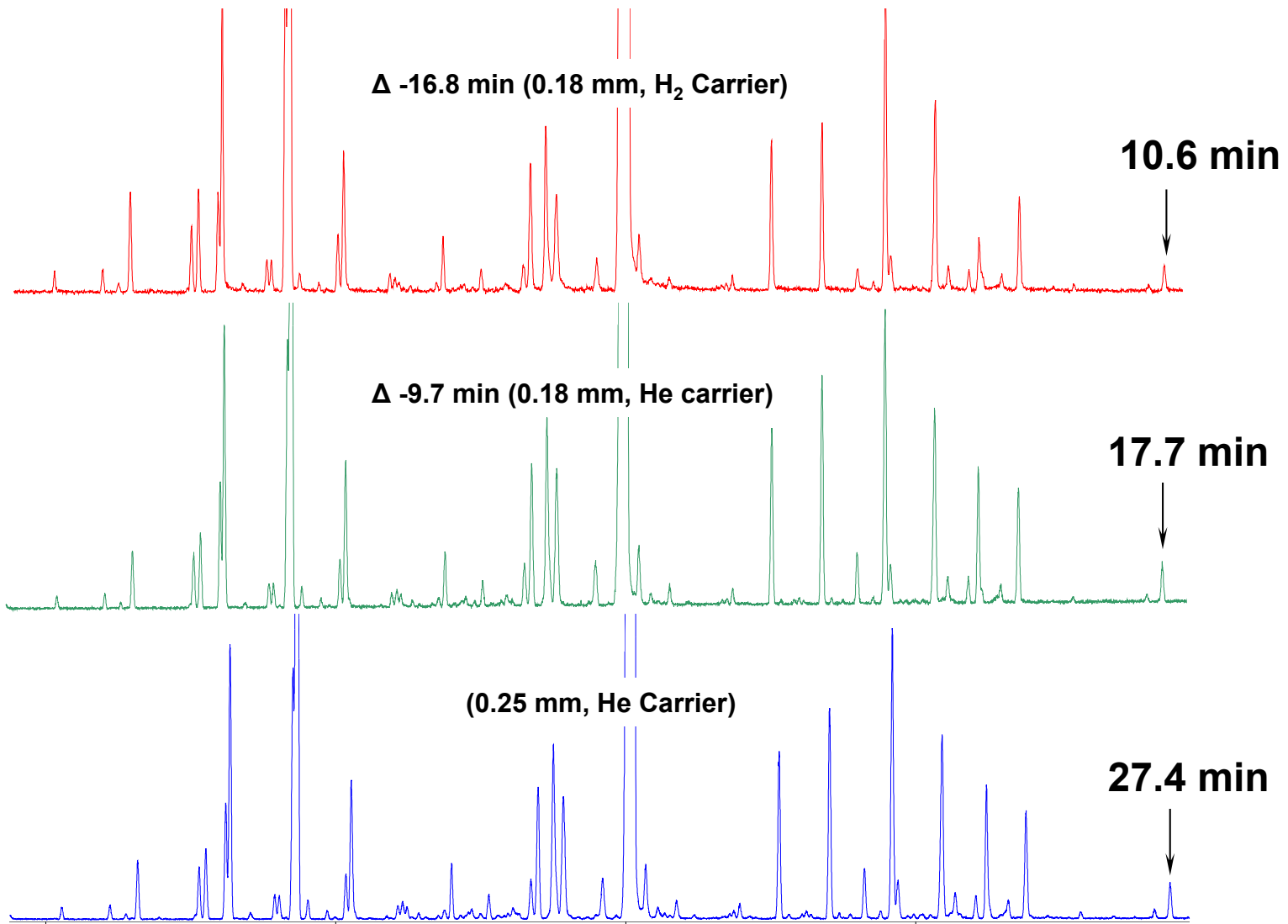
# Pefferminzöl



# Pefferminzöl



# Pefferminzöl - Auflösungsüberprüfung



# Optimierung unter Beibehaltung der Auflösung

Compounds	Compound Resolution		
	0.25 mm	0.18 mm	0.18 mm
	Helium	Helium	Hydrogen
Sabinene	1.52	1.59	1.56
$\beta$ -Pinene			
$\alpha$ -Terpinene	1.61	1.73	1.86
p-Cymene			
<b>Speed Gain</b>	<b>N/A</b>	<b>35%</b>	<b>61%</b>



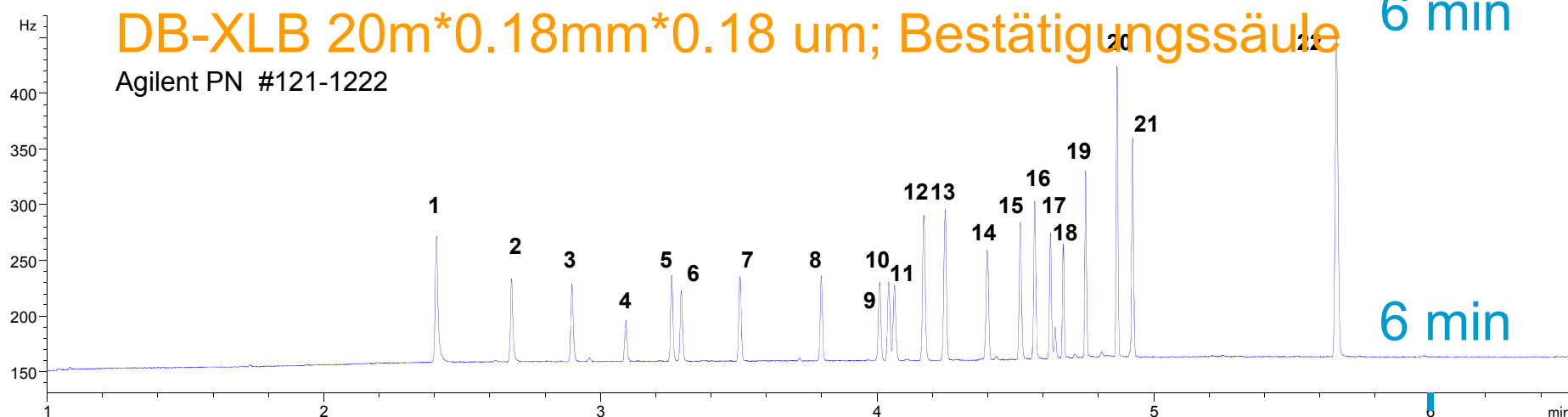
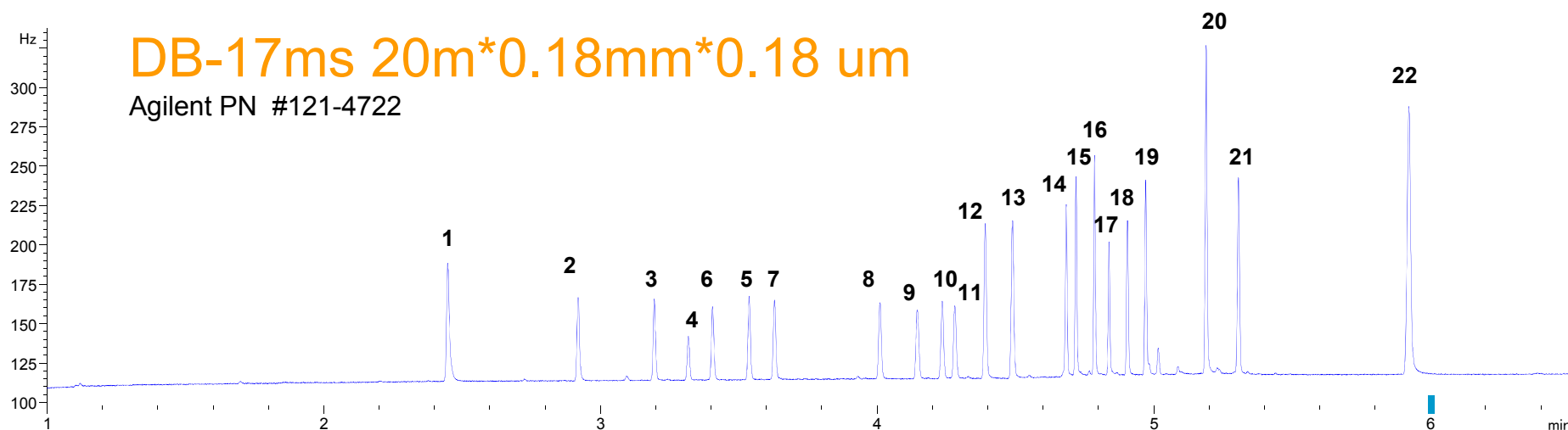
# Chromatographische Bedingungen

## Contract Laboratory Program CLP Pestizide

übersetzt via MT von 0.53 mm ID Säule mit Analysenzeit von 40 min

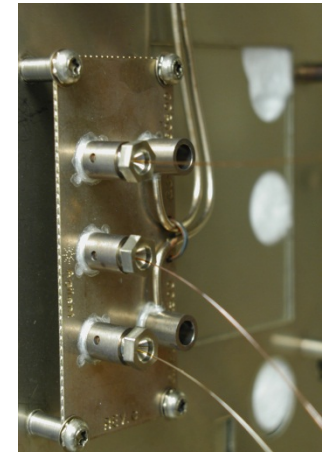
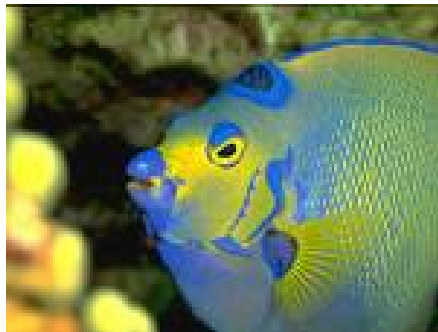
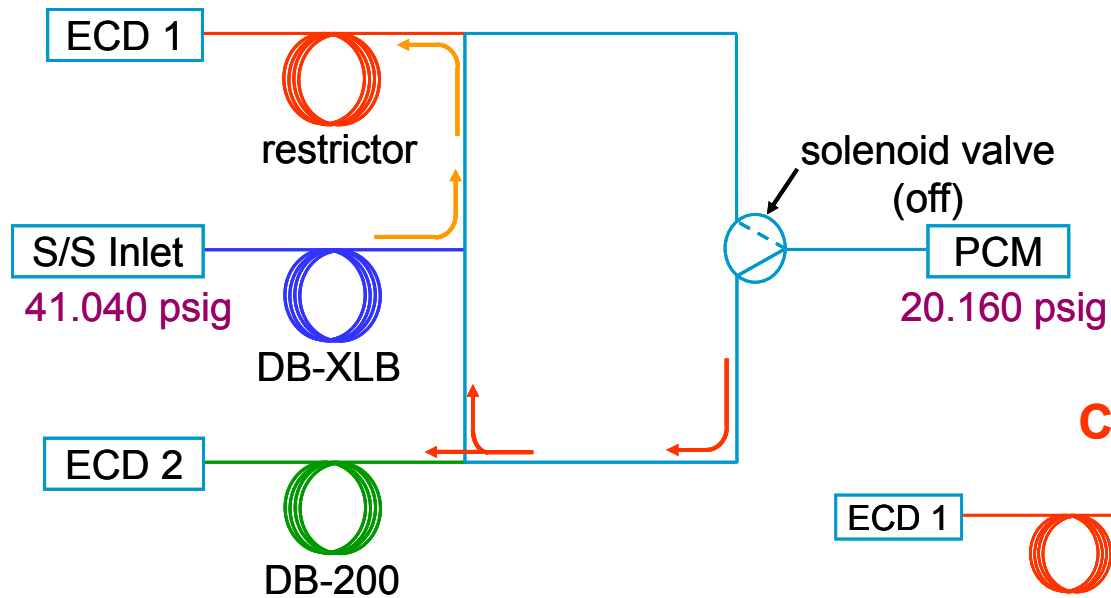
GC :	Agilent 6890N
Sampler:	Agilent 7683B, 5 µL syringe (Agilent part # 5181-1273), 0.5 µL injection
Carrier:	Hydrogen ( flow programmed , 69 cm/sec at 120° C, ramped at 99ml/min to 106 cm/sec at 4.4 minutes, purified through a Big Universal Trap Agilent part # RMSH-2)
Inlet:	Split/splitless; 220° C, pulsed splitless (35 psi for 0.5 min, purge flow of 40 ml/min on at 1 minute, gas saver flow 20 ml/min on 3 minutes
Inlet Liner:	Deactivated single taper direct connect (Agilent part # 1544-80730)
Retention Gap:	5m x 0.25 mm ID deactivated (Agilent part # 160-2255-5)
Y-splitter :	Quartz deactivated (Agilent part # 5181-3398)
Columns:	
1	20m x 0.18mm x 0.18µm DB-17ms (Agilent part # 121-4722)
2	20m x 0.18mm x 0.18µm DB-XLB (Agilent part # 121-1222)
Oven:	120° C (0.32 min); 120 ° C/min to 160° C; 30 ° C/min to 258°C (0.18 min); 38.81° C/min to 300° C (1.5 min)
Detection:	µECD 320° C; nitrogen makeup; constant column + makeup flow 60 (ml/min)
Probe:	CLP Standard; organochlorierte Pestizide in Elutionsreihenfolge: Tetrachloro-m-Xylol, α-BHC, γ-BHC, β-BHC, D-BHC, Heptachlor, Aldrin, Heptachlorepoxyd, γ Chlordan, αChlordan, Endosulfan1, 4,4 DDE, Dieldrin, Endrin, 4,4DDD, Endosulfan2, 4,4DDT, Endrinaldehyd, Endosulfansulfat, Methoxychlor, Endrinkeeton, Decachlorobiphenyl

# Analyse: CLP Pestizide - 0.4 pg/Komponente



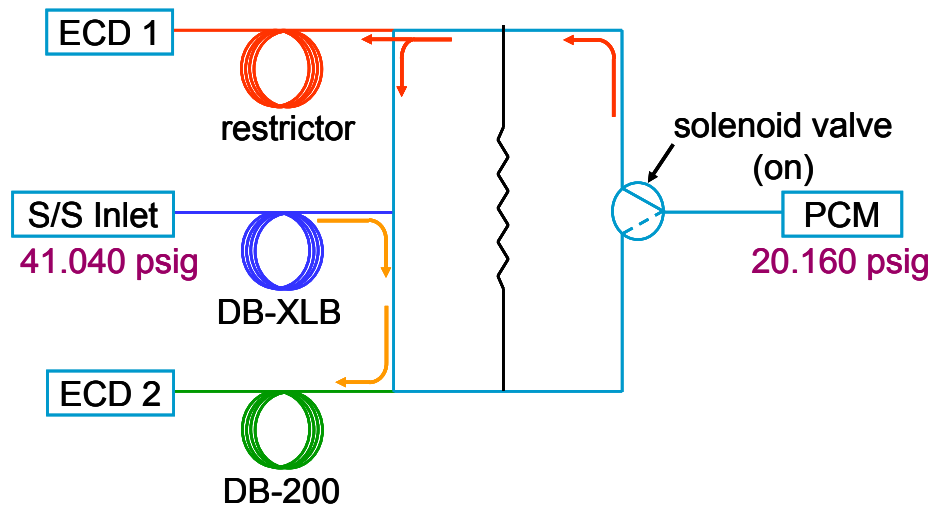
# Agilent 7890A: Heart Cutting und Backflushing Analyse: PCDDs, PCDFs, PCBs in Fischöl

No Cut



Deans Switch  
CFT

Cut to Column 2



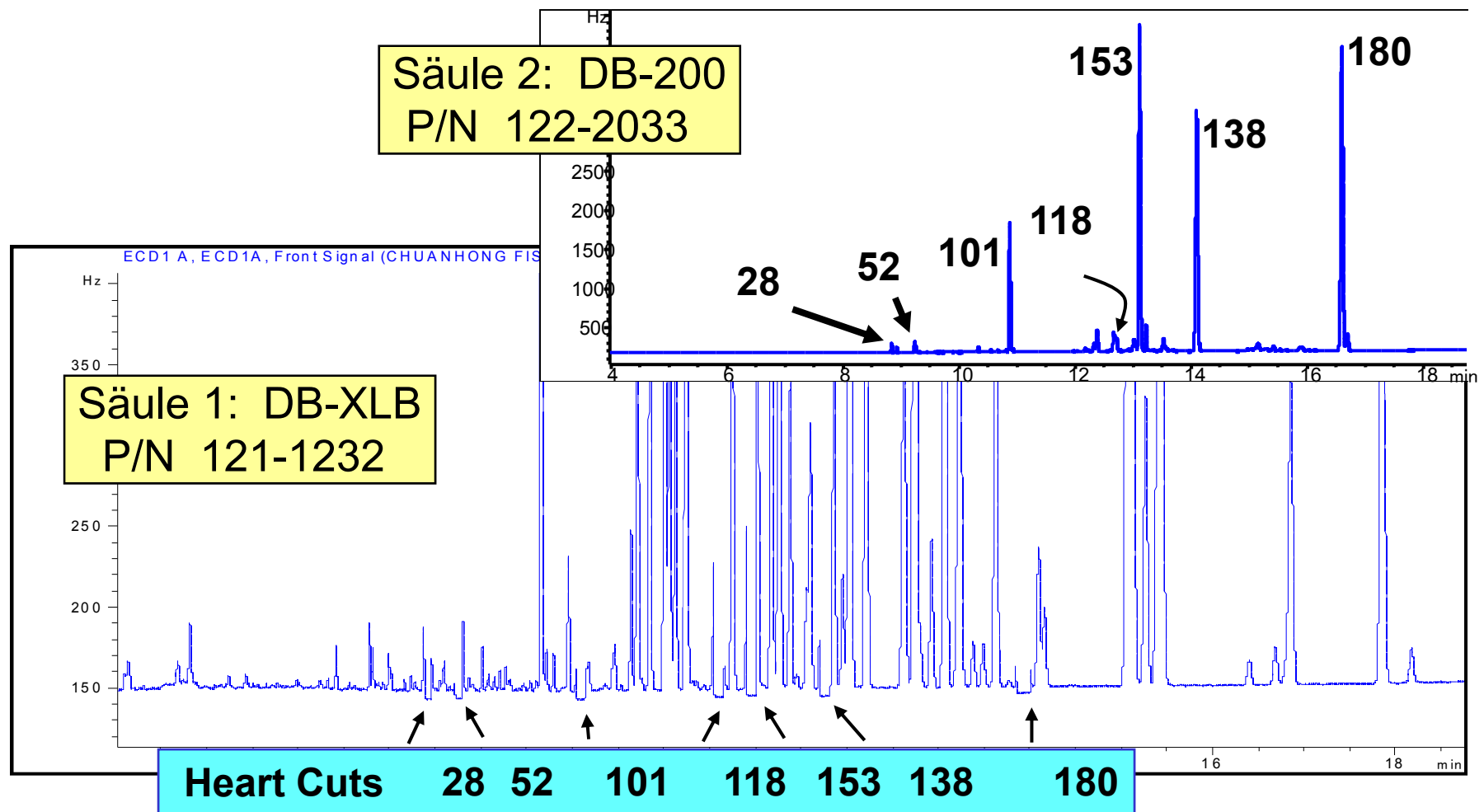
# Analysenbedingungen

- 1. Säule: J&W DB-XLB, 30m\*0.18mm\*0.18 um, PN 121-1232
- 2. Säule: J&W DB-200, 30m\*0.25mm\*0.5 um, PN 121-2033
- Liner: Agilent deactivated, single taper PN5062-3587
- Inlet: Split/splitless bei 330C
- T-Programm: 1 min - 80C, 50C/min bis 200C(0min), 10c/minbis290C (5min)
- Detektoren: ECD bei 340C
- ECD make up: N2 mit 60 l/min
- Trägergas: H2
- T für Backflush:290C für 2.4 min

**Optimal durchführbar jetzt mit den neuen flexiblen Metallferrulen**

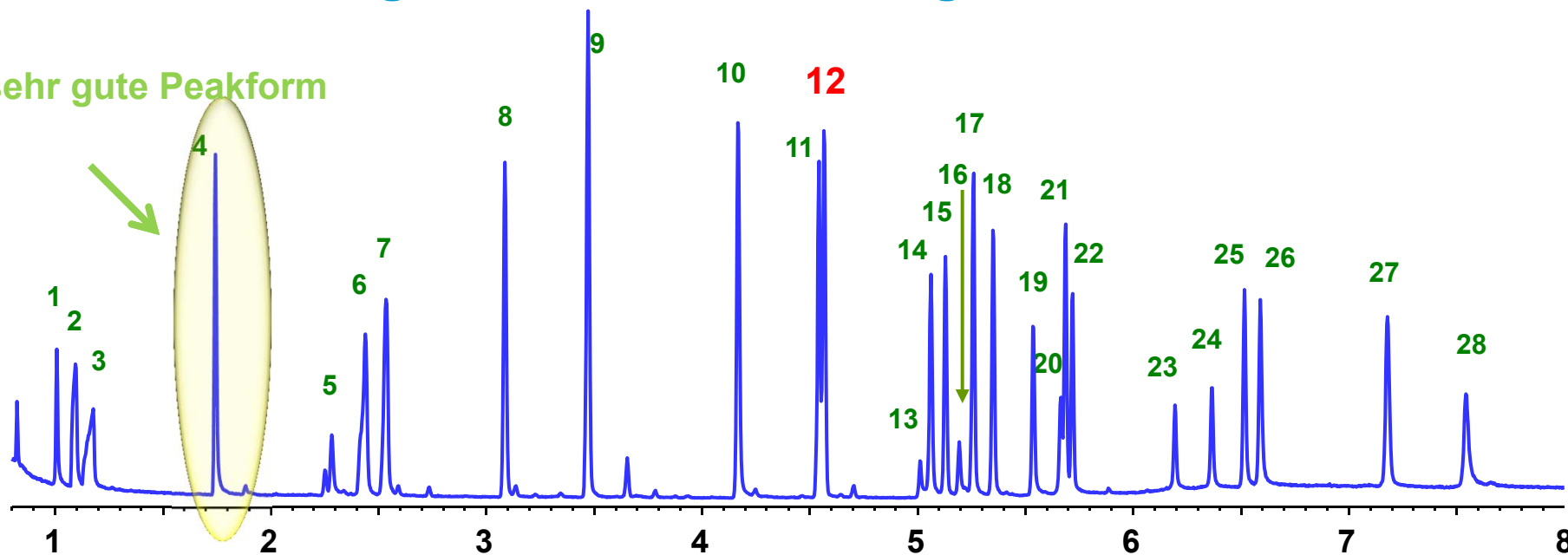
**Appl.Note: 5989-6095**

# Heart Cutting zur Isolierung von PCBs von Fischöl gespiked mit Aroclor 1260



# 28 Verbindungen aus der Toxikologiedatenbank

sehr gute Peakform



- |                                       |                     |
|---------------------------------------|---------------------|
| 1 Amphetamine                         | 15 Codeine          |
| 2 Phentermine                         | 16 Lorazepam        |
| 3 Methamphetamine                     | 17 Diazepam         |
| <b>4 Nicotine</b>                     | 18 Hydrocodone      |
| 5 Methylenedioxyamphetamine(MDA)      | 19 Oxycodone        |
| 6 Methylenedioxymethamphetamine(MDMA) | 20 Temazepam        |
| 7 Methylenedioxyethylamphetamine      | 21 Diacetylmorphine |
| 8 Meperidine                          | 22 Flunitrazepam    |
| 9 Phencyclidine                       | 23 Nitrazepam       |
| 10 Methadone                          | 24 Clonazepam       |
| 11 Cocaine                            | 25 Alprazolam       |
| 12 SKF-525a (RTL Compound)            | 26 Verapamil        |
| 13 Oxazepam                           | 27 Strychnine       |
| 14 Tetrahydrocannabinol               | 28 Trazodone        |

## Instrument Conditions

Carrier: Helium fixed pressure 35.0 PSI  
 Inlet: splitless 1 µl 280 °C, total flow 56.4 ml/min, 3 ml/min switched septum purge, gas saver off, 50 ml/min after 0.4 minutes  
 Inlet Liner: dual taper deactivated (Agilent p/n 5181-3315)  
 Column: Agilent J&W **DB-35msUI 15m x 0.18mm x 0.18µm** (Agilent p/n 122-3832UI) 35 PSI constant pressure mode  
 Back-flush: post run: 1 min. 1 psi inlet, 75 psi aux EPC  
 Oven: 100 °C (0.25 min) to 345 °C (40 °C/min, 2.25 min hold)  
 MSD: transfer line 300 °C, source 300 °C, quadrapole 180 °C scan mode  
 NPD: Bloss bead 300 °C H<sub>2</sub> 3 ml/min, 60 ml/min air, 11 ml/min makeup + col flow  
 CFT Device: 2-Way splitter with solvent venting between MSD and NPD

# Applikationsbeispiel – Aromatische Solventien

- ASTM Unified aromatic solvent methods D16
  - Combines 10 ASTM aromatic methods into one
  - One method can be used for up to 16 different sample types
  - One capillary GC column replaces up to 6 different columns.
- Due to demands for increased productivity, many QC / QA laboratories need to analyze large number of samples every day. Faster analysis is highly desirable for increased sample throughput and therefore lower cost per sample.

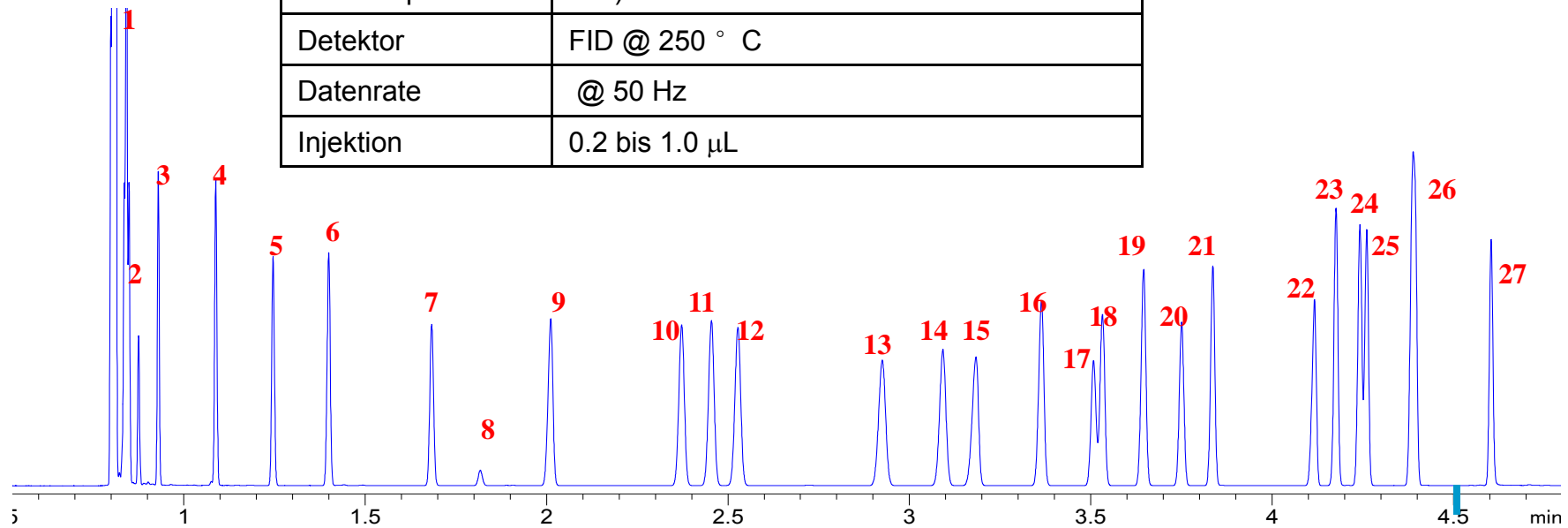
<b>1</b> heptane	<b>8</b> 1,4-dioxane	<b>15</b> o-xylene	<b>22</b> tridecane
<b>2</b> cyclohexane	<b>9</b> undecane	<b>16</b> propylbenzene	<b>23</b> diethylbenzene isomer
<b>3</b> octane	<b>10</b> ethylbenzene	<b>17</b> p-ethyltoluene	<b>24</b> diethylbenzene isomer
<b>4</b> nonane	<b>11</b> p-xylene	<b>18</b> m-ethyltoluene	<b>25</b> n-butylbenzene
<b>5</b> benzene	<b>12</b> m-xylene	<b>19</b> t-butylbenzene	<b>26</b> a-methylstyrene
<b>6</b> decane	<b>13</b> cumene	<b>20</b> s-butylbenzene	<b>27</b> phenylacetylene
<b>7</b> toluene	<b>14</b> dodecane	<b>21</b> styrene	

Die Klassische Methode mit einer 60 m HP Innowax-Säule dauert 25 Minuten

# Aromatische Solventien

geschwindigkeitsoptimierte Methode

Säule	HP-Innowax, 20 m x 0.18 mm x 0.18 $\mu$ m
Trärgas	Helium @ 33.00 psi constant pressure mode
Inlet	Split/Splitless @ 250 ° C
	100:1 to 600:1 split Verhältniss
Ofentemp	70 ° C ( 3 min); 45 ° C/min to 145 ° C (1 min)
Detektor	FID @ 250 ° C
Datenrate	@ 50 Hz
Injektion	0.2 bis 1.0 $\mu$ L



4.5 min

HE

# Lebensdauerbetrachtung: Applikationsbeispiel

## Umwelt- und Lebensmittelanalytik– CLP Pestizide

5989-7616EN

Agilent Appl. Note

EPA Methode: 22 CLP Pestizide

- Die Bedingungen für schnelle GC wurden routinemässig mit 0.18 mm I.D. Säule über 9 Monate hinweg eingesetzt. Die Säulen wiesen die gleiche hohe Lebensdauer auf wie die zuvor eingesetzten 0.32 mm I.D. Säulen.

	Stationary Phase	Column Description	Carrier Gas	Run Time (min)	Method Translation Mode	Comments	Productivity Gain
Method 1	DB-XLB	30m x 0.32mm x 0.25um	He	16		Coelution of one compound pair A, and 90% coelution for another compound pair B	
Method 2	DB-XLB	20m x 0.18mm x 0.18um	He	13	Translate only	Compound pair A baseline resolved and compound pair B 80% based line resolved	<b>19% faster</b> than Method 1 and a <b>vast improvement in resolution</b>
Method 3	DB-XLB	20m x 0.18mm x 0.18um	H2	8.5	Tranlate only	Compound pairs A and B still resolved with no change in selectivity	<b>47% faster</b> than Method 1
Method 4	DB-XLB	20m x 0.18mm x 0.18um	H2	7	Fast Analysis	Slight loss of resolution	<b>56% faster</b> than Method 1
Method 5	DB-XLB	20m x 0.18mm x 0.18um	H2	6.5	Optimized method	Maintained similar resolution	<b>60% faster</b> than Method 1 with <b>more than twice as many samples</b> analyzed

# Ultra-Inert Säulen

Agilent J&W DB-5ms, HP-5ms, HP-1ms, DB-1ms,  
DB-35 ms **Ultra Inert**

Minimiertes Säulenbluten ist nur die halbe Miete für die Spurenanalytik

Ultra-hohe Inertheit ist für aktive Verbindungen ebenfalls unerlässlich für die Spurenanalytik

Der gesamte Probenpfad, nicht nur die Säule, muss sich durch ultra-Inertheit auszeichnen: UI Liner, UI flexible Metallferrules (UltiMetal Plus), etc....



UI

# Gründe für ultra-inerte Säulen und einen ultra-inerten Probenpfad

## Bessere Sensitivität

### Reduktion des Rauschens

Injektionssystem (Septa, **Liner**,  
**Ferrules....**)

Träger-und Detektorgase  
Leaks Temperatur  
**Säulenbluten**

### Erhöhung des Signals

Probenkonzentration und -grösse  
**inert Injektions/Detektionsport**  
Gasgeschwindigkeit  
Temperaturrate  
**Säuleninertheit**

## Bessere Richtigkeit und Genauigkeit

Reduktion des **Peaktailings** erhöht die  
Reproduzierbarkeit der Integration

**Geringere Fehlerbreiten - kleinere RSD**

**Bessere Auflösung für komplexe Analysen**

**Höhere Peakkapazität**

# Herausforderung: neue Testbedingungen

1. **1-Propionsäure**
2. 1-Octen
3. n-Octan
4. **4-Picolin**
5. n-Nonan
6. **Trimethyl phosphat**
7. 1,2-Pentanediol
8. n-Propylbenzol
9. 1-Heptanol
10. 3-Octanon
11. n-Decan

**GC:** Agilent 6890N

**Sampler:** Agilent 7683B, 0.5 µL Spritze (Agilent PN # 5188-5246), 0.02 µL Splitinjektion

**Carrier:** Wasserstoff 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 PN # 5183-4647)

**Säule A:** 30m x 0.25mm x 0.25µm 5%-Phenylsäule

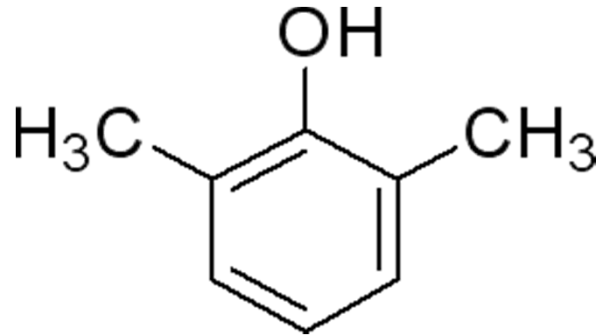
**Säule B:** Agilent J&W DB-5ms Ultra Inert 30m x 0.25mm x 0.25µm (Agilent part # 122-5532UI)

**Ofen:** **65 Grad C isotherm**

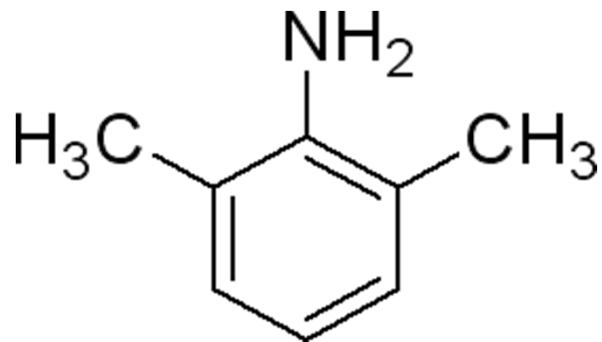
**(ursprünglicher Grobtest 120 Grad isotherm)**

**Detektion:** FID at 325% C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min., nitrogen makeup

## “schwache” Proben



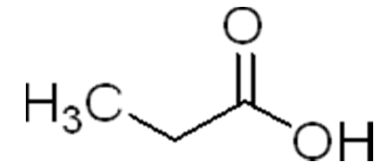
2,6-Dimethylphenol



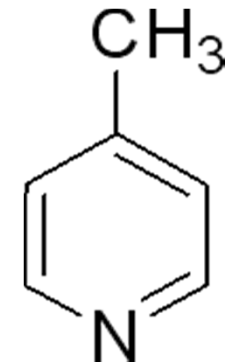
2,6-Dimethylanilin

ursprünglicher Grobtest

## aussagekräftige Proben



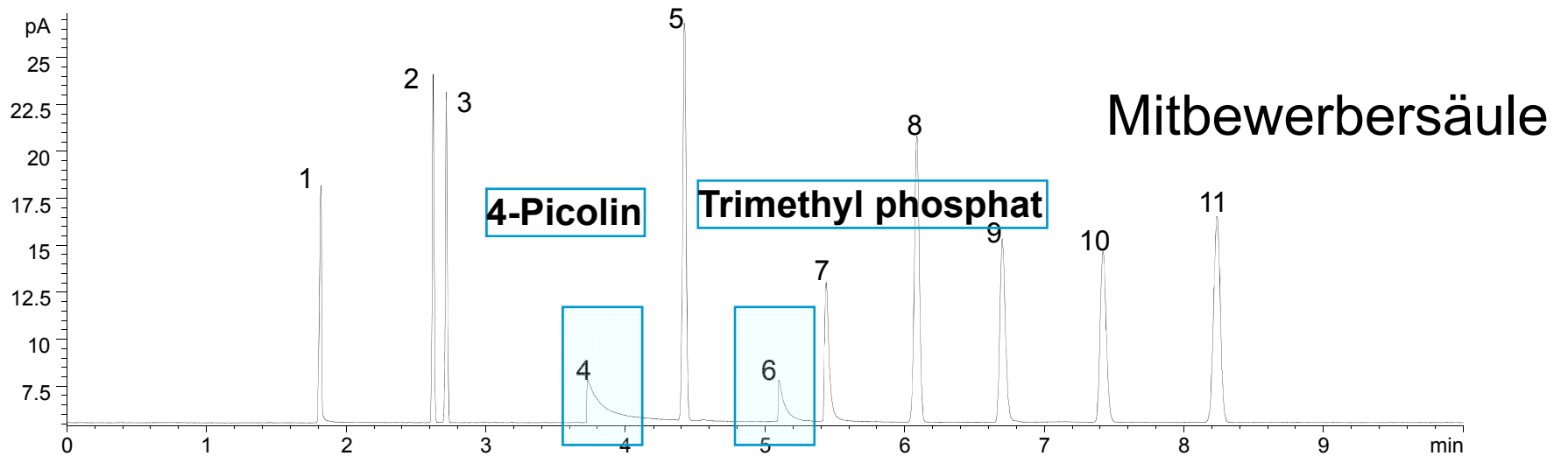
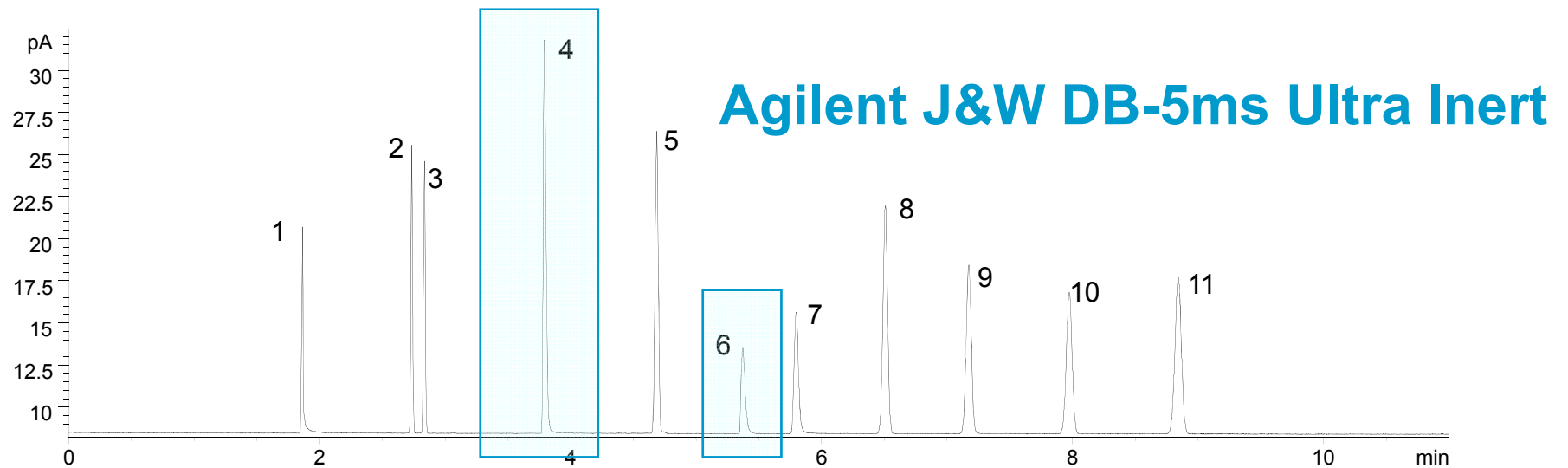
Propionsäure



4-Picolin

verfeinerter Grobtest

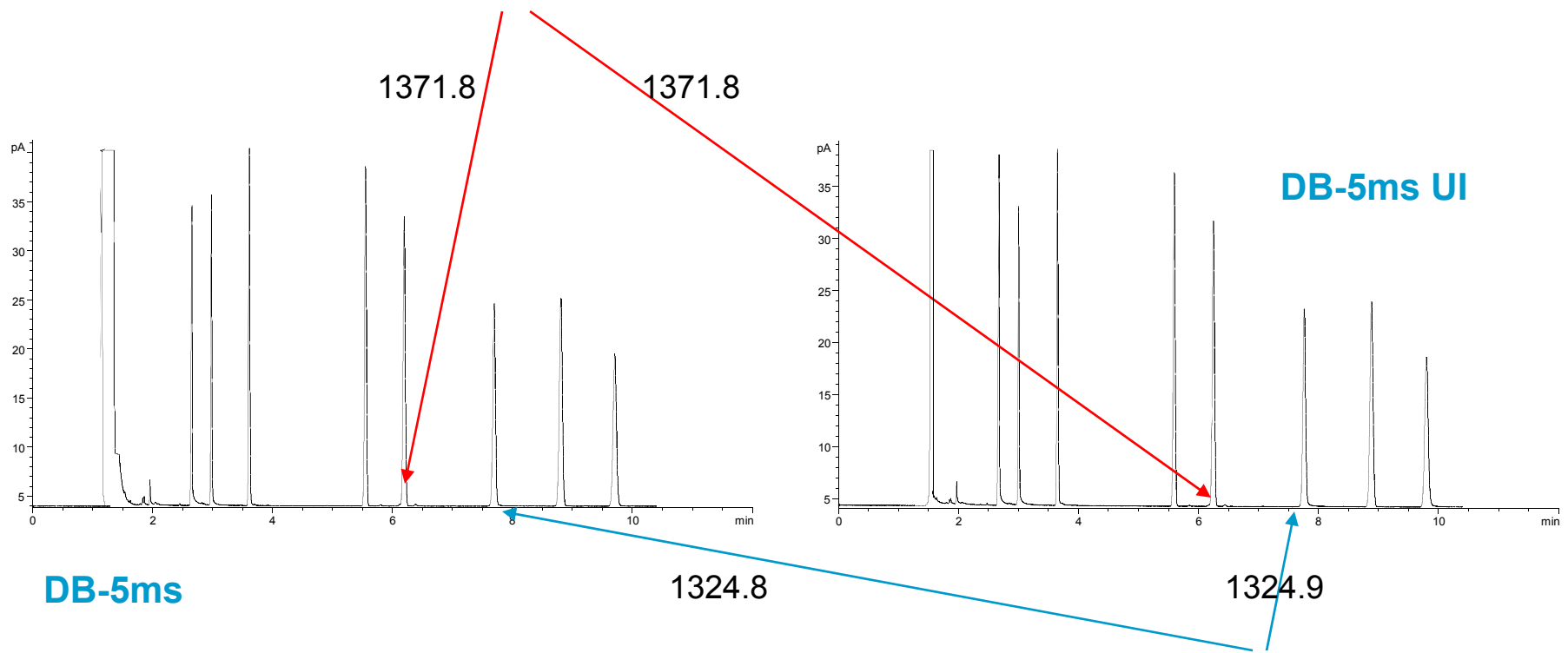
# Herausforderung: neue Testmischung



# DB-5ms Ultra-Inert und klassische DB-5ms

## Gleiche Selektivität gilt auch für andere UI Säulen.

Retention Index von 1-Methylnaphthalen



Retention Index von 1-Undecanol

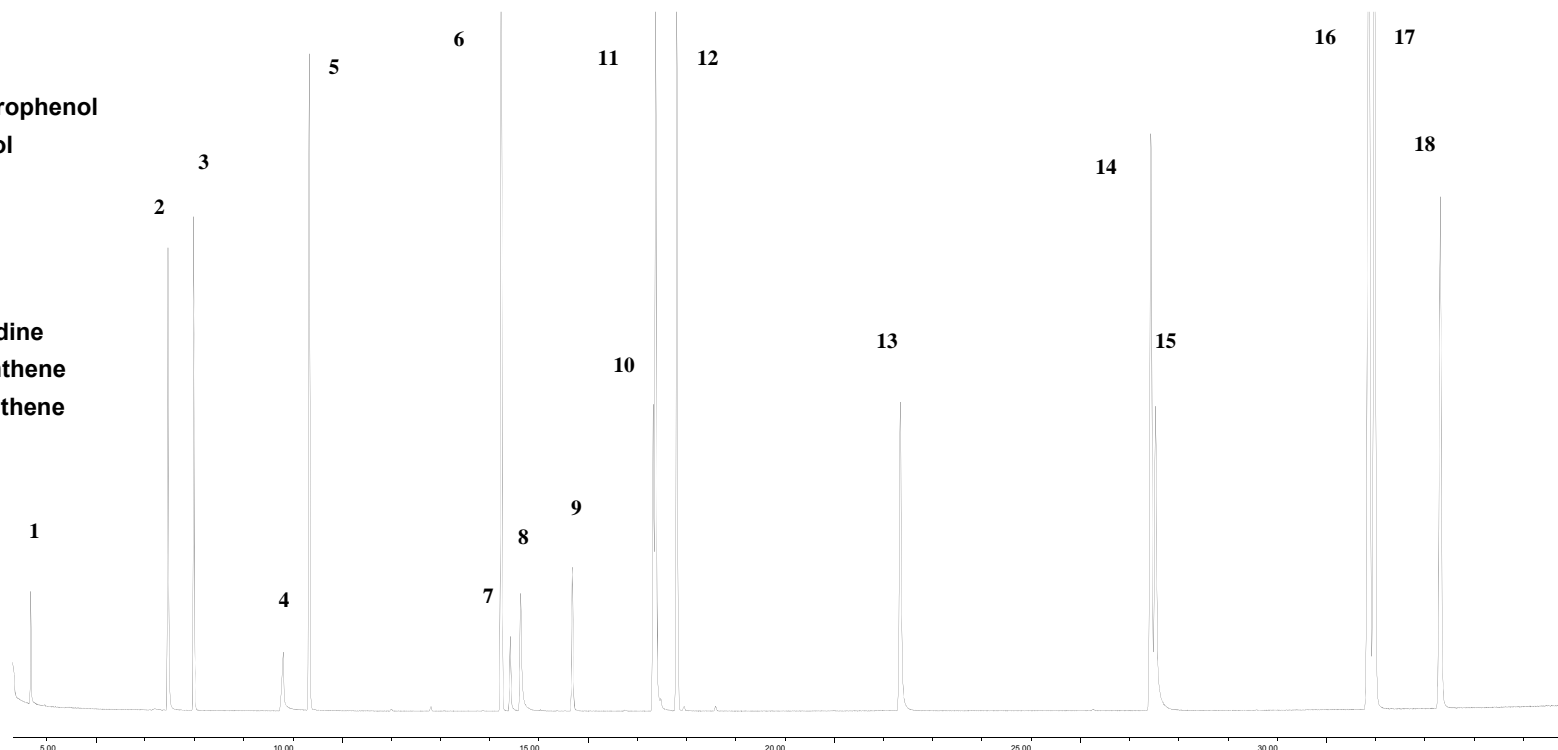
UI

# Ultra Inert Applikation - Semi Volatile Analysis

## EPA 8270

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 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

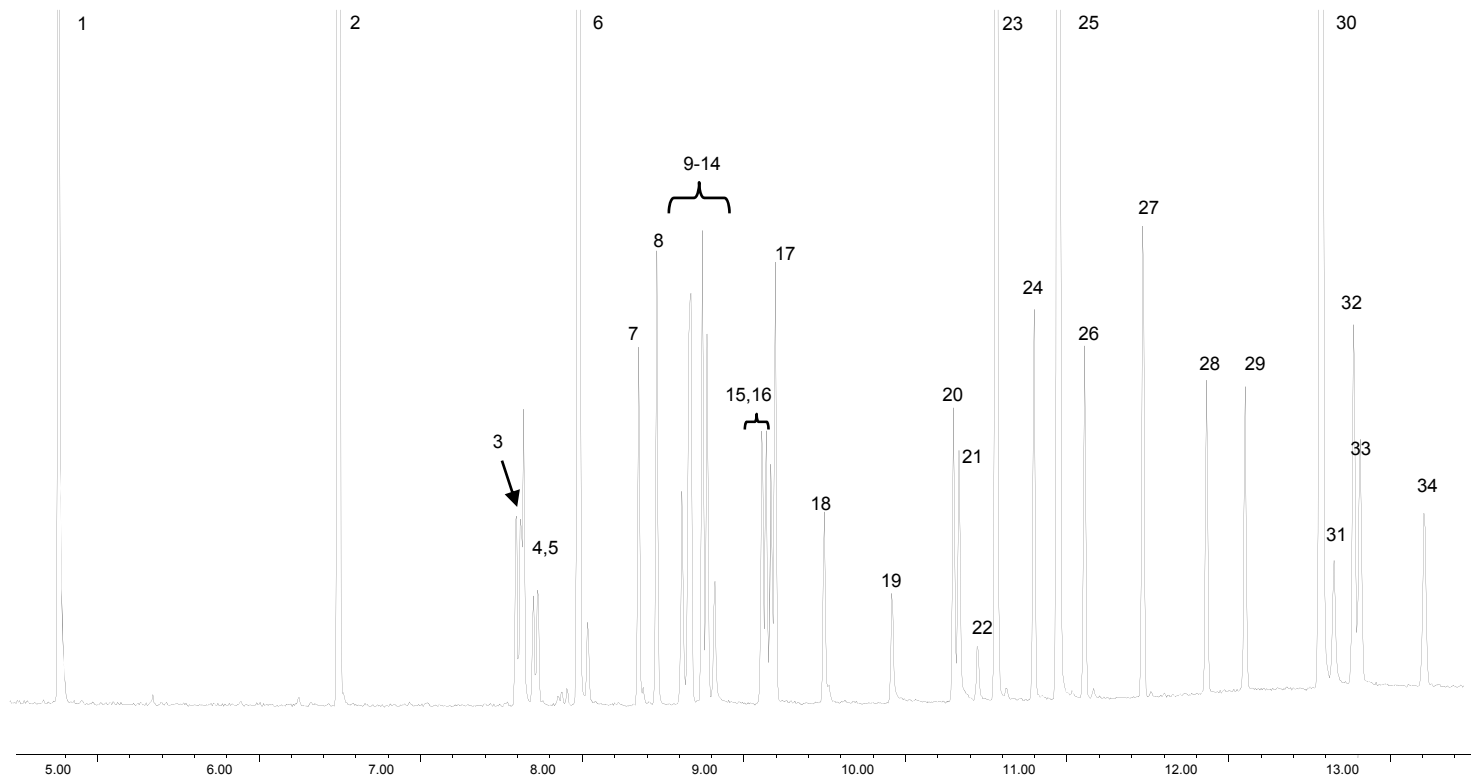


# Pestizide und Flammenschutzmittel (US EPA 527)

- 1,2-Dimethyl-2-nitrobenzene
- Acenaphthalene-D10
- Dimethoate
- Atrazine
- Propazine
- Anthracene-D10
- Vinclozoline
- Prometryne
- Bromacil
- Malathion
- Thiazopyr
- Dursban
- Benthiocarb
- Parathion
- Terbus sulfone
- Bioallethrin
- Oxychlorane
- Fenamiphos
- Nitrophen
- Norflurazone
- Kepone
- Hexazinone
- Triphenyl phosphate
- Bifenthrin
- Chrysene-D12
- BDE-47
- Mirex
- BDE-100
- BDE-99
- Perylene-D12
- Fenvalerate
- Esfenvalerate
- Hexabromobiphenyl
- BDE-153

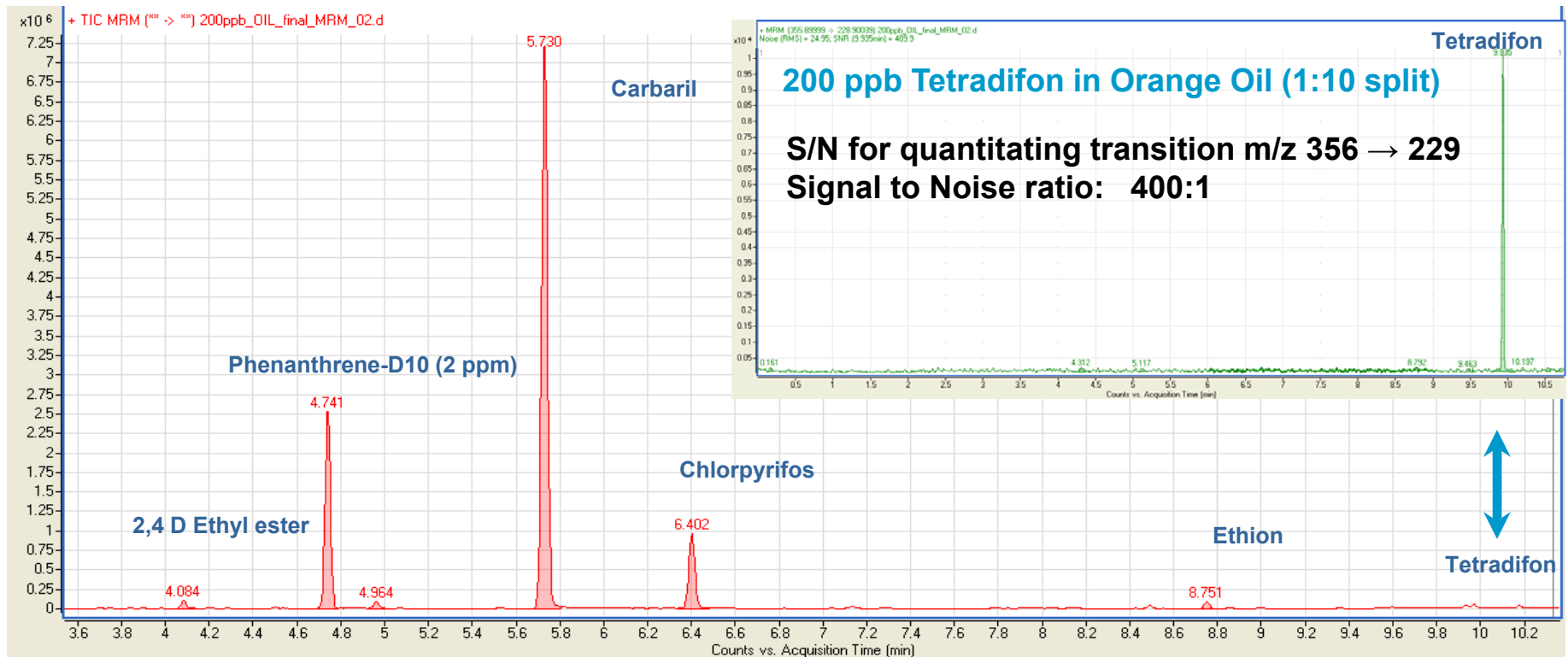
## GC/MSD Bedingungen

**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



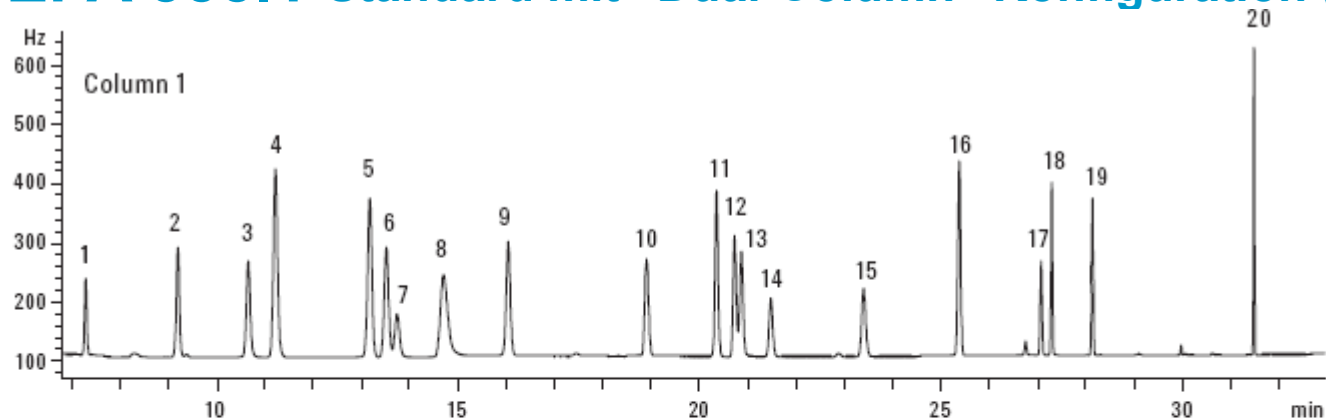
# Pestizide in Orangenöl

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  $\mu$ 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 $\mu$ 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.

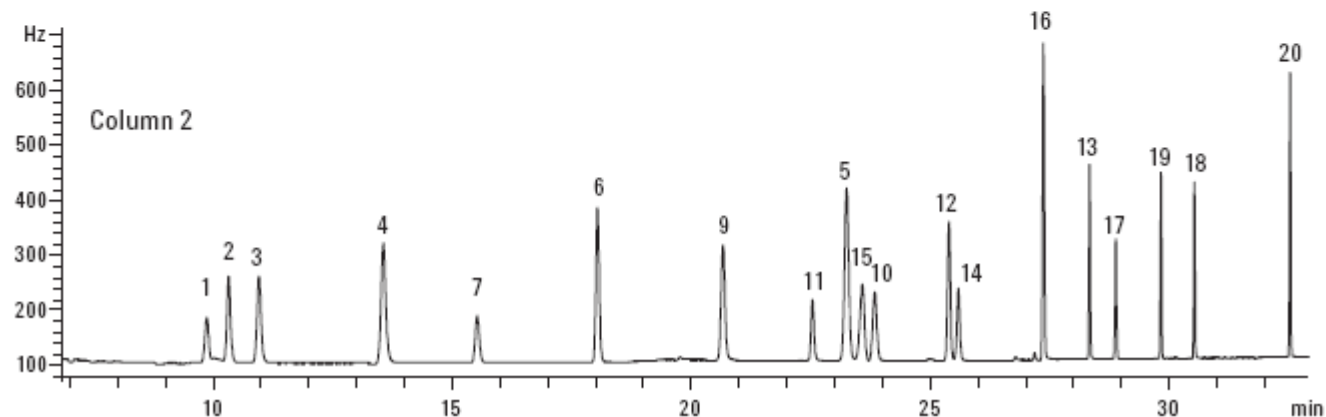


# Chlorierte Lösungsmittel / Desinfektionsprodukte

## EPA 555.1 Standard mit "Dual-Column" Konfiguration (5pg on column)



1. Chloroform
2. 1,1,1-Trichloroethane
3. Carbon Tetrachloride
4. Trichloroacetonitrile
5. Dichloroacetonitrile
6. Bromodichloromethane
7. Trichloroethylene
8. Chloral hydrate
9. 1,1-Dichloro-2-propanone
10. 1,1,2-Trichloroethane
11. Chloropicrin
12. Dibromochloromethane
13. Bromochloroacetonitrile
14. 1,2-Dibromoethane
15. Tetrachloroethylene
16. 1,1,1-Trichloro-2-propanone
17. Bromoform
18. Dibromoacetonitrile
19. 1,2,3-Trichloropropane
20. 1,2-Dibromo-3-chloropropane



GC/Dual ECD: 7890A/G2397A equipped with QuickSwap column flow technology

Sample: 0.5ul injection of 0.02ug/ml EPA 551.1 Standard

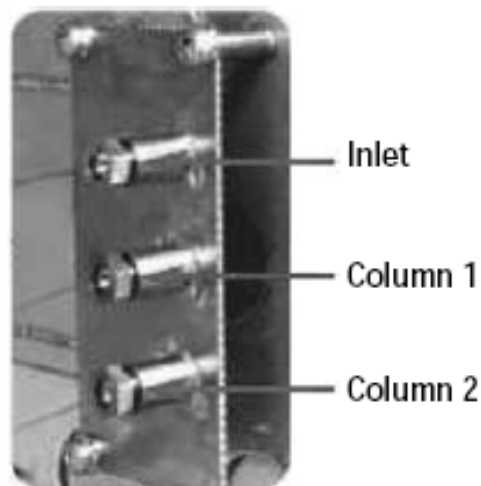
Säule 1: HP-1ms Ultra Inert 30 m x 0.25 mm x 1.0 µm (Agilent P/N 19091S-933UI)

Säule 2: DB-1301 30 m x 0.25 mm x 1.0 µm (Agilent P/N 122-1333)

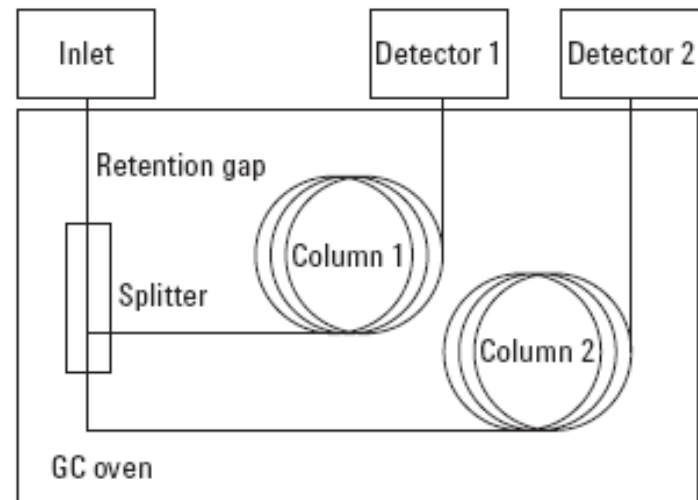
Carrier: He 25.057 cm/sec constant flow; Oven: 33°C (14 min) to 60°C (5°C/min), 15°C/min to 275°C (7 min)

Inlet: splitless, 200°C, purge flow 20ml/min at 0.25 min; ECDs:300°C, Makeup (N<sub>2</sub>) = constant col + makeup = 30ml/min

# “Dual-Column” Konfiguration (5pg on column) EPA 551.1 Standard



Two-way splitter without makeup gas connections  
CFT = Capillary Flow Technology



Plumbing diagram for the two-way splitter without  
makeup gas

Optimal durchführbar jetzt mit den neuen flexiblen Metallferrulen

# EPA 551.1 Standard

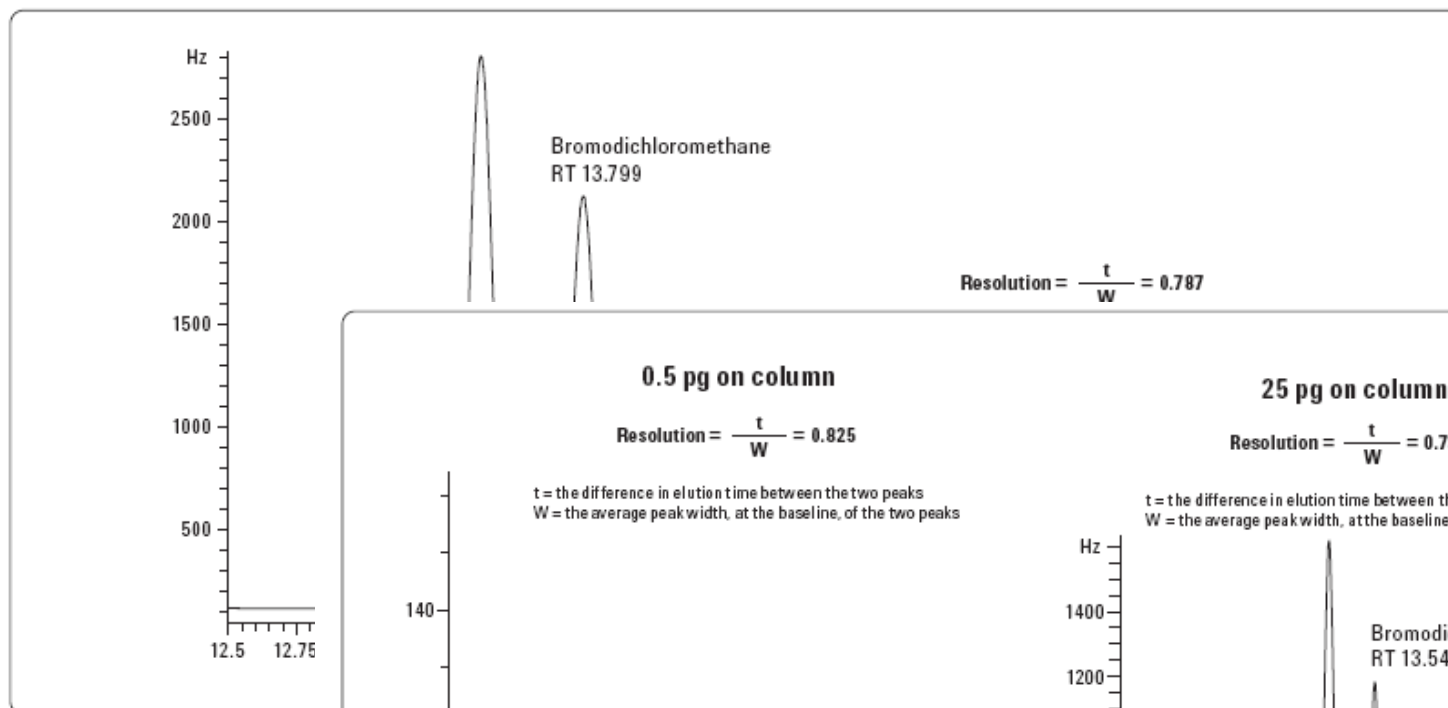


Figure 6. Enlarged chromatogram  
Method criteria for column

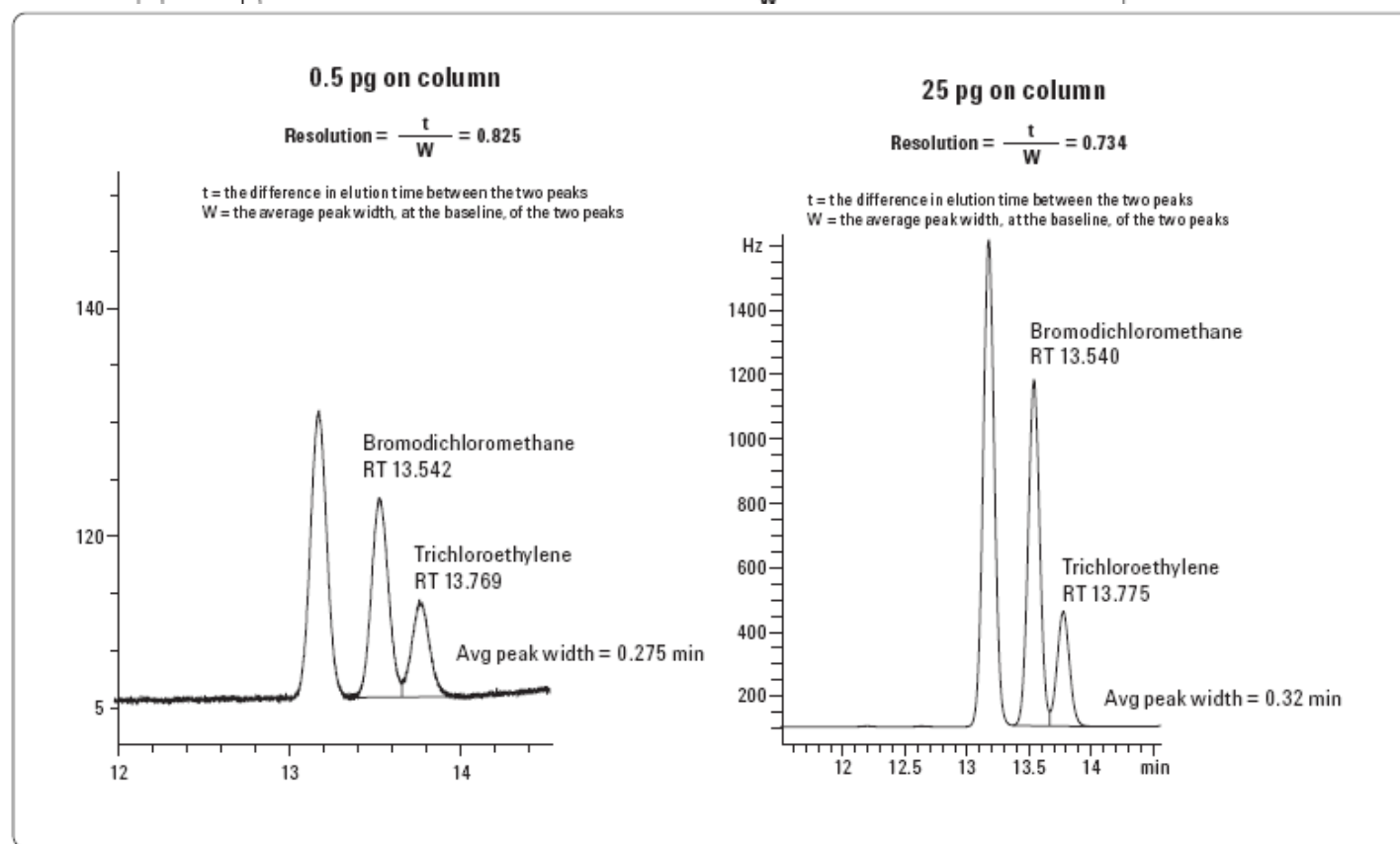


Figure 7. Enlarged chromatograms of the low and high range EPA551.1 standards on the Agilent J&W HP-1ms Ultra Inert 30 m × 0.25 mm × 1.0 μm capillary GC column. Method criteria for column performance is a resolution greater than 0.50 between bromodichloromethane and trichloroethylene.

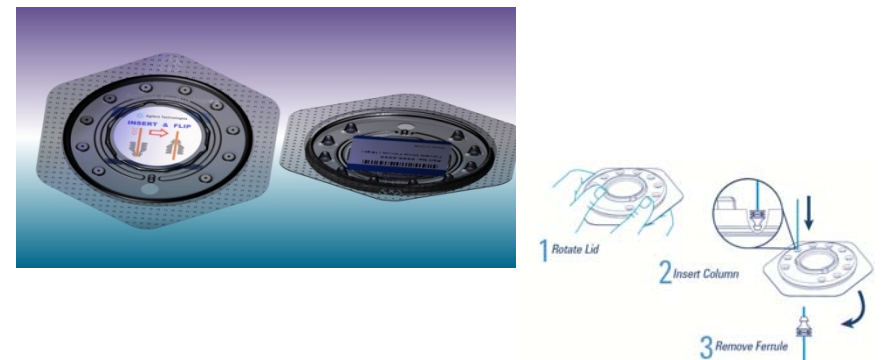
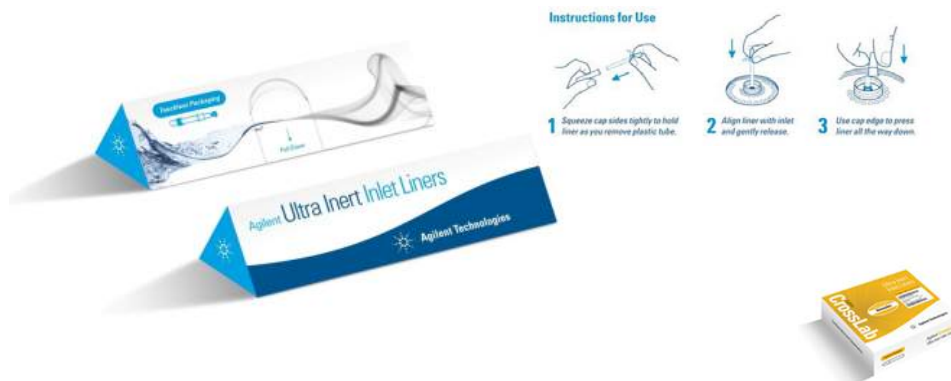
# Ultra Inert Liner & Ultra Inert Flexible Metall Ferrule

## Eigenschaften: UI Liner

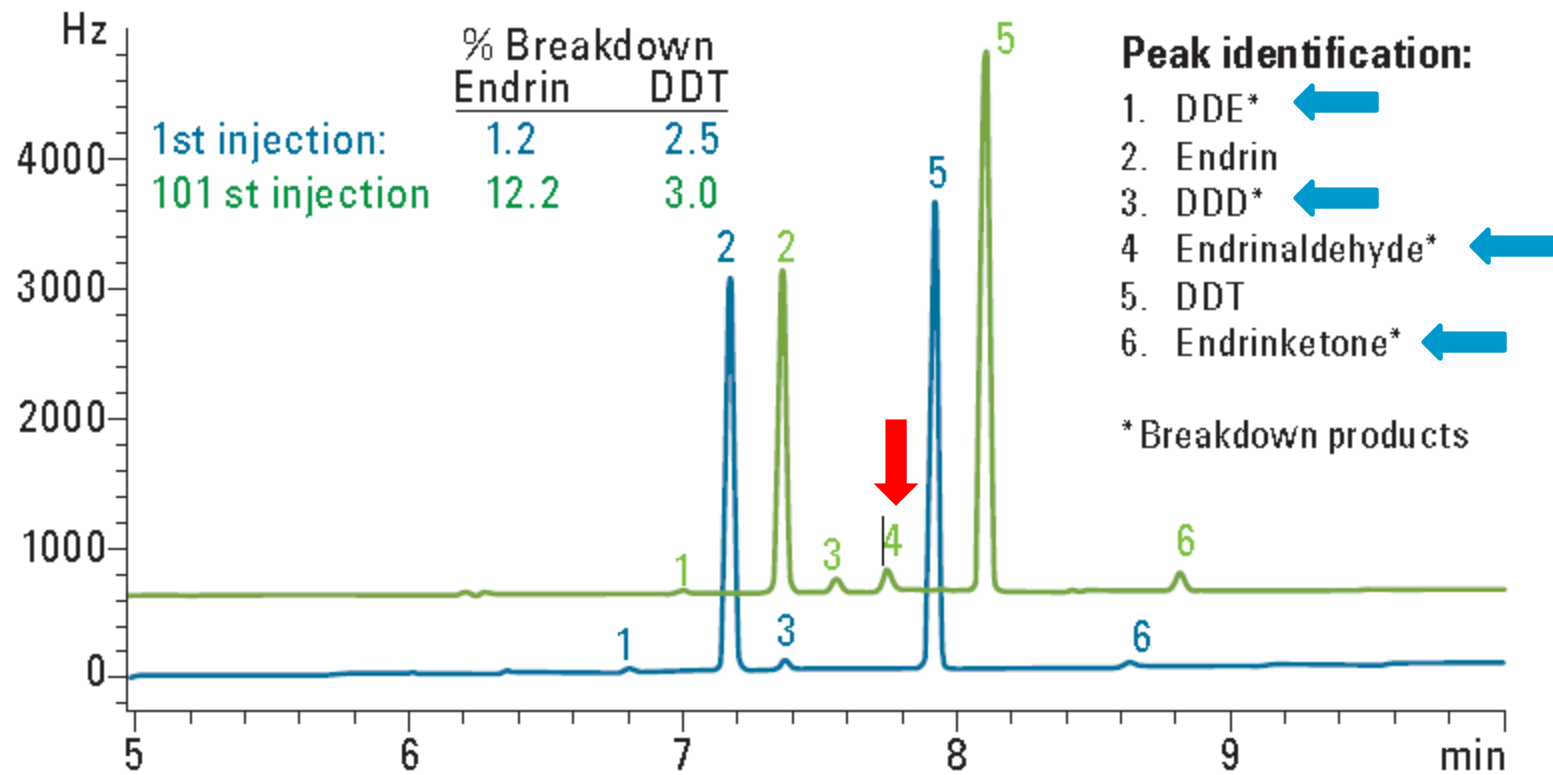
1. Agilent Ultra Inert “vapor deposition process”
2. Permanente Deaktivierung
3. Ultra Inert **deaktivierte Glaswolle**
4. QC: Lottest
5. berührungsfreie Handhabung

## Eigenschaften: UI flexible Metall Ferrule

1. Neues Design mit optimalen Kompressionseigenschaften zur Minimierung von Säulenbruch und Fittingzerstörung
2. Permanente UltiMetal Deaktivierung für eine ultra-inerte Oberfläche - Spurenanalytik
3. berührungsfreie Handhabung



# Ultra Inert Liner- Endrin & DDT Degradation Pestizidanalytik



**5990-7380EN**

UI

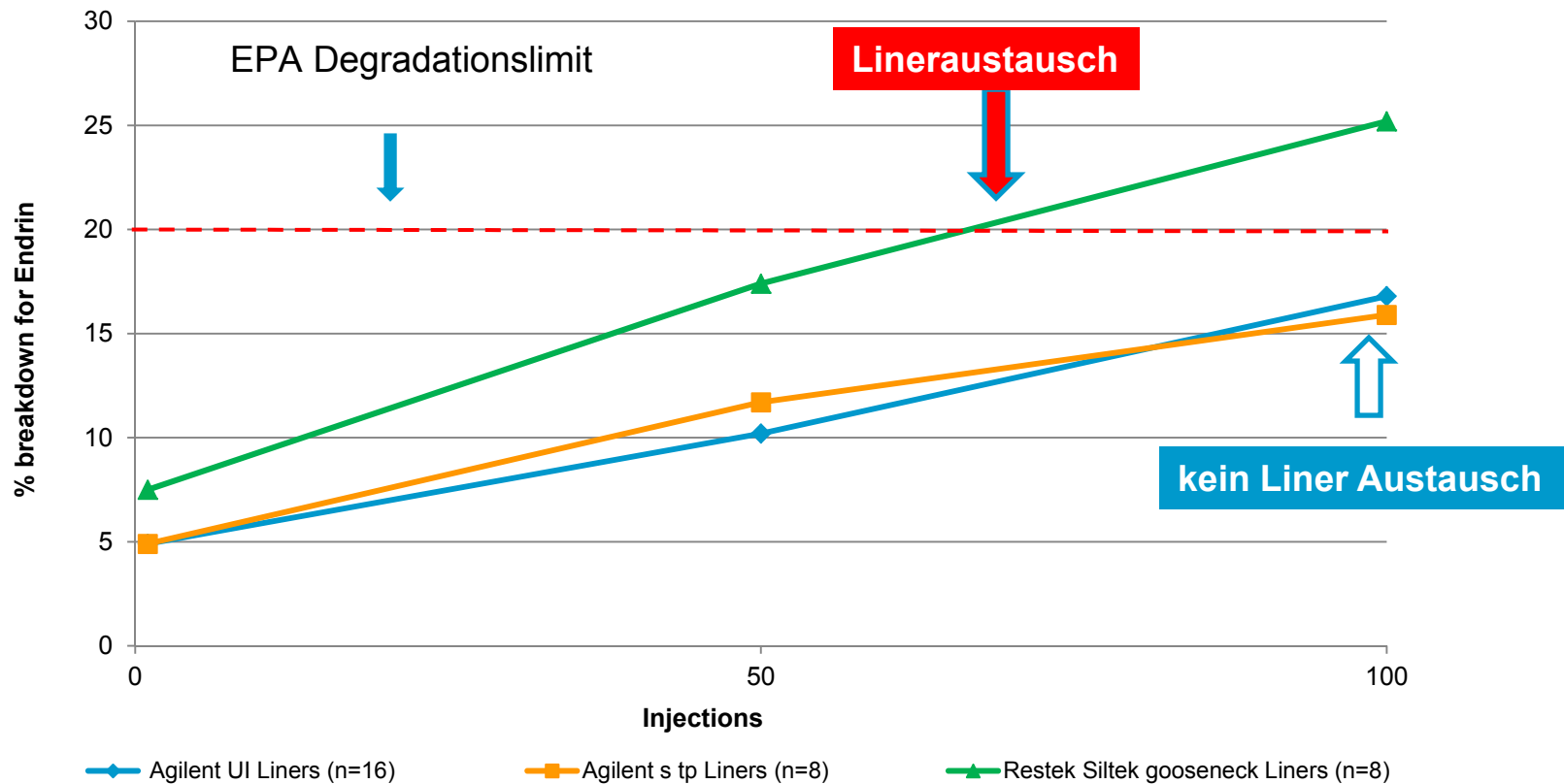
# Vergleich: Ultra Inert Liner - Endrin Degradation

100 Injektionen Endrin Degradation:

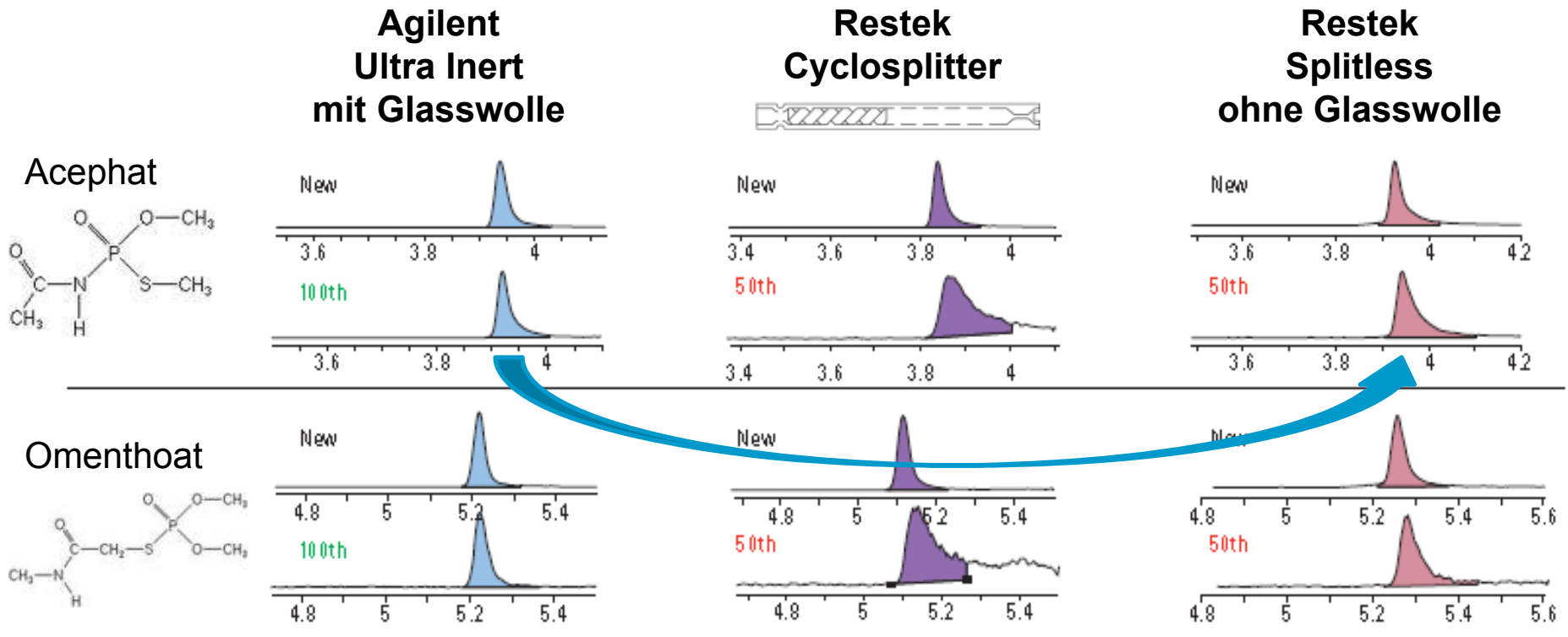
Liner Konfiguration: Splitless, single taper liners OHNE Glasswolle

Deaktivierungsvergleich: **Ultra Inert**, **Agilent Proprietary** und Restek **Siltek**

59990-7380EN



# Vergleich: Degradation von empfindlichen Organophospho Pestiziden



50 ng/ml

**Ultra Inert Liners mit Glaswolle liefern gleiche oder bessere Peakformen als die Mitbewerber Produkte:**

- UI liner: widerstandsfähiger gegen Matrixkontamination
- UI liner: bessere Datenqualität mit kleinerer Anzahl an Linerwechseln

**5990-7706EN**

UI

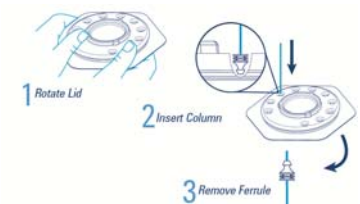
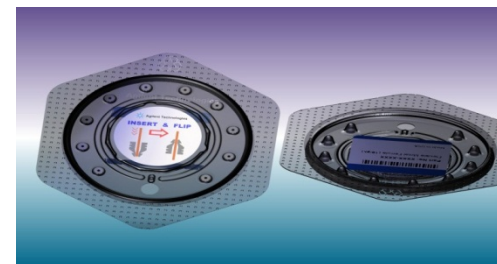
# Ultra Inert Liner & Ultra Inert Flexible Metall Ferrule

## Eigenschaften: UI Liner

1. Agilent Ultra Inert "vapor deposition process"
2. Permanente Deaktivierung
3. Ultra Inert **deaktivierte Glaswolle**
4. QC: Lottest
5. berührungsfreie Handhabung: O-Ring

## Eigenschaften: UI flexible Metall Ferrule

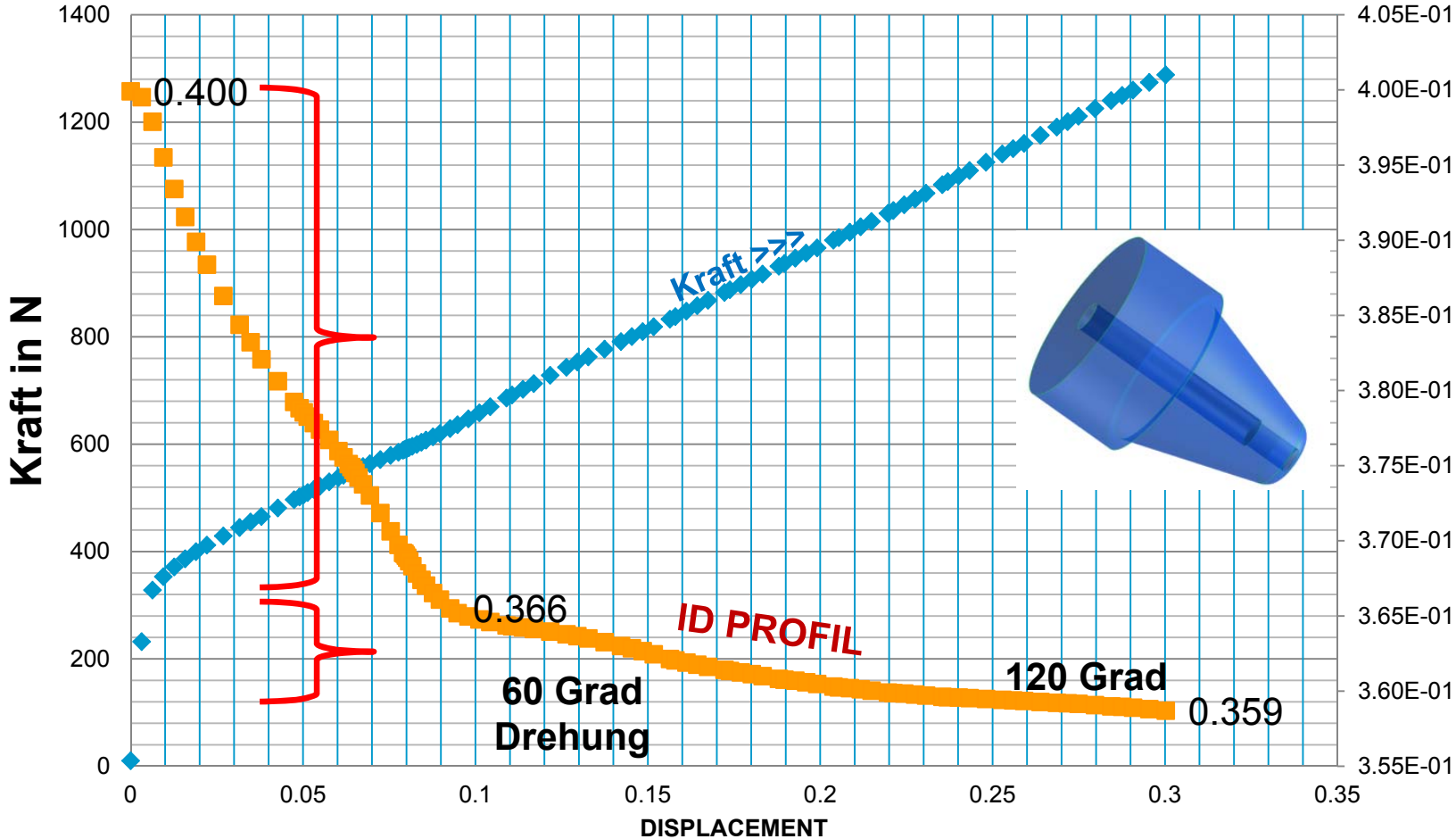
1. Neues Design mit optimalen Kompressionseigenschaften zur Minimierung von Säulenbruch und Deformation des Fittingsitzes
2. Permanente UltiMetal Deaktivierung für eine ultra-inerte Oberfläche - Spurenanalytik
3. berührungsfreie Handhabung



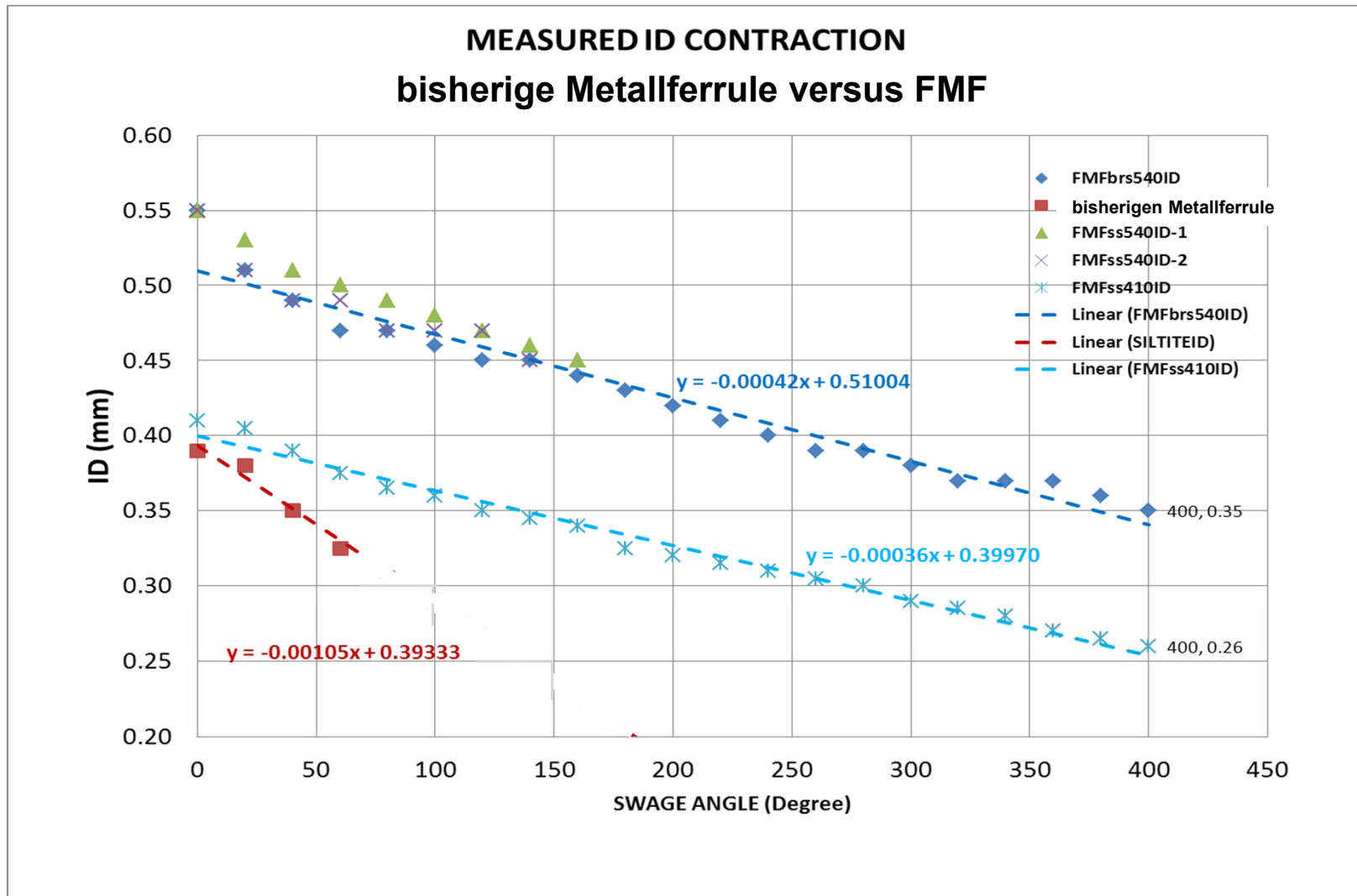
UI

# Bisherige Metallferrule

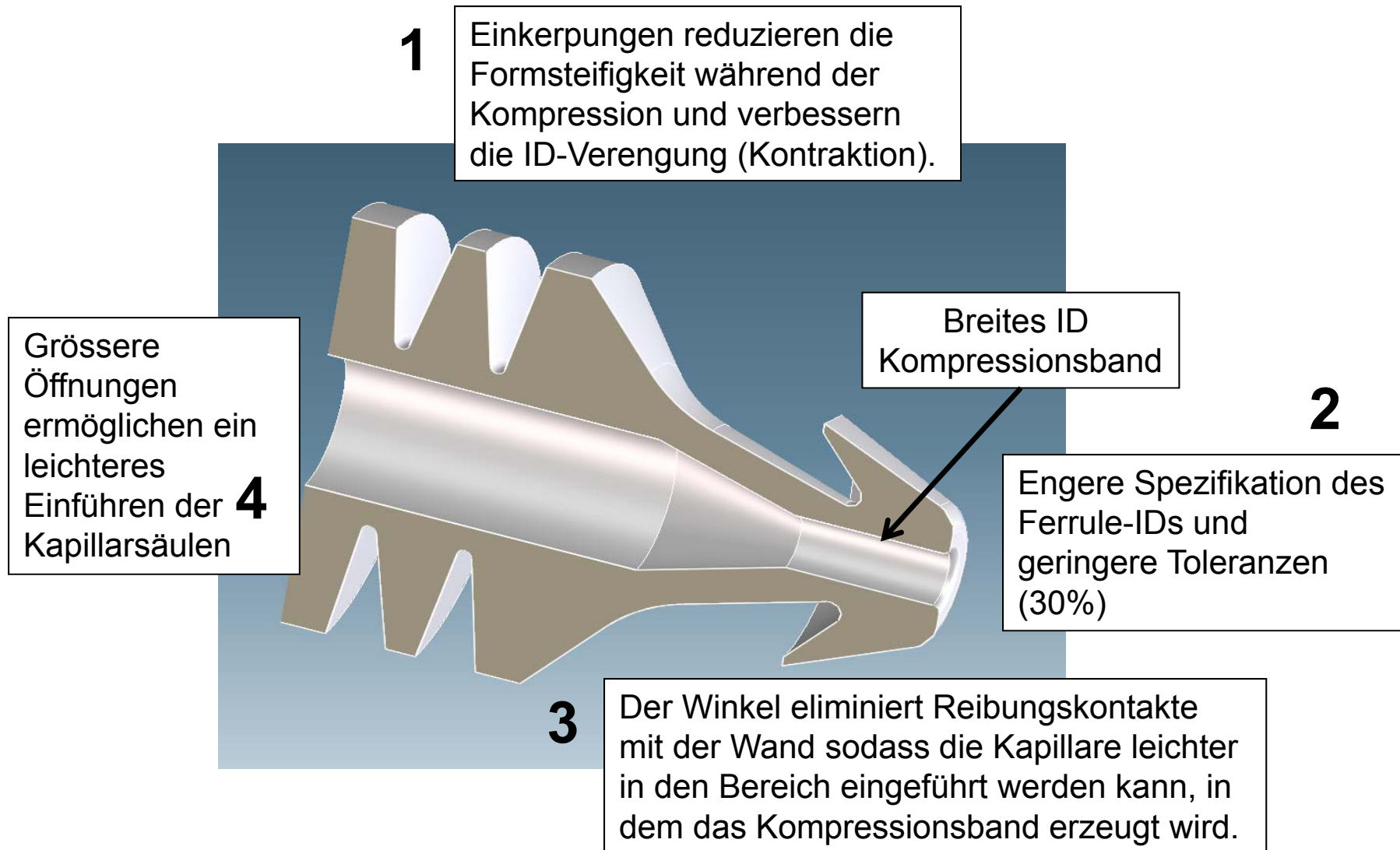
## Kompression des bisherigen Metallferrule



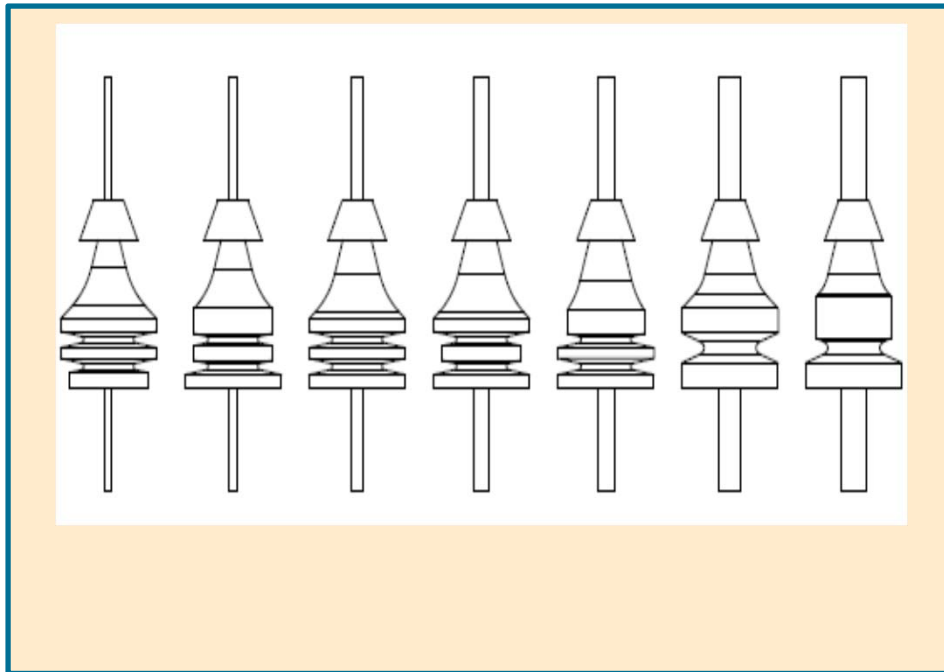
# Flexible Metallferrule (FMF) vs bisherige Metallferrule



# Designmerkmale des Flexibles Metallferrules



# Praktische Vorteile des neuen Designs



Die neuen Ferrule (Familie) können für alle Agilent GC-Säulen inkl. UltiMetal-Säulen eingesetzt werden

Die neue UltiMetal Plus Oberflächenbehandlung erzeugt ein ultra-inertes Ferrule; eine Inertheit, die über die der bisherigen Metallferrule hinausgeht

Jeder ID hat sein eigenes Design, ist also optimiert auf den jeweiligen Säulentyp

Die macht das Einordnen leicht, Verwechslungen sind nicht möglich.

Agilent Intellectual Property: Gebrauchs- & Designpatent angemeldet

# Endrin/DDT Degradationstest

## Method

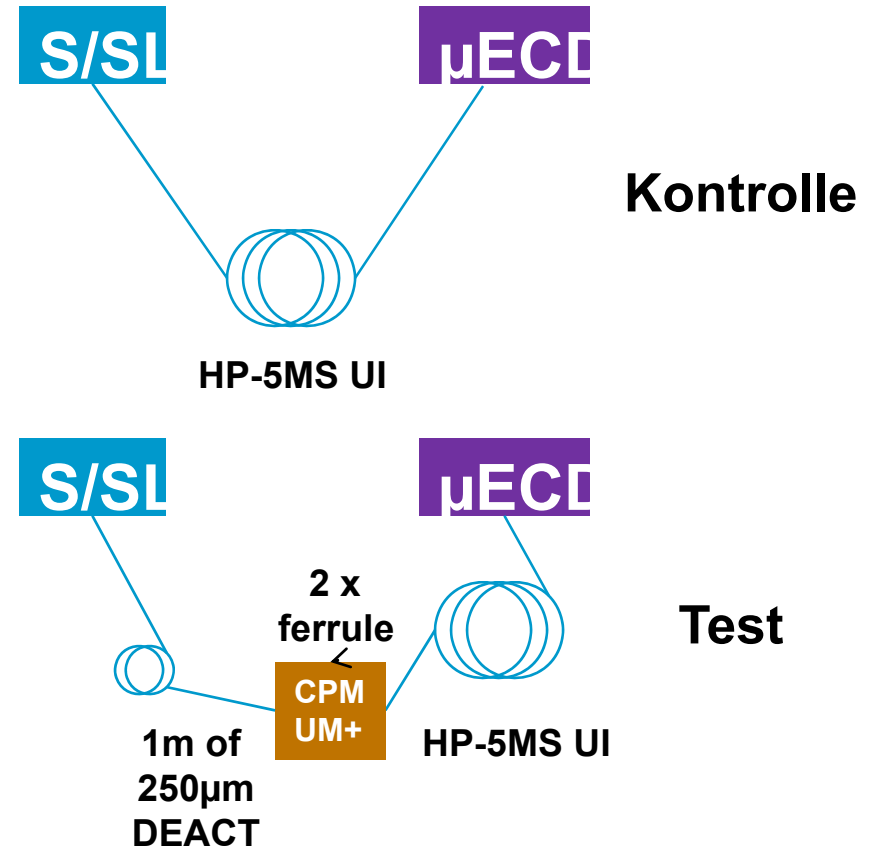
Inlet: Splitless mode. 250°C.  
1µL injection volume

Column: HP-5MS UI, 15m x 250µm x 0.25µm,  
CF= 1mL/min, Helium

Oven 120C, hold 1.0min  
30°C/min to 220C, no hold  
8C/min to 280C, hold 1min

µECD: 280C, Makeup (N<sub>2</sub>) = 60mL/min

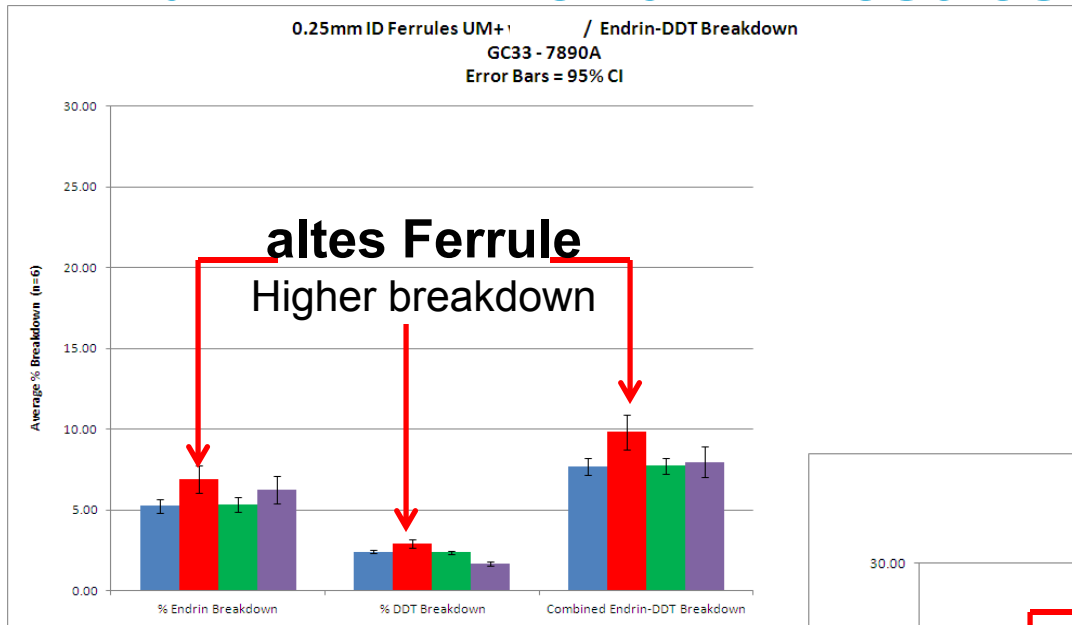
Sample: 50ppb Endrin / 100ppb DDT in iso-C8



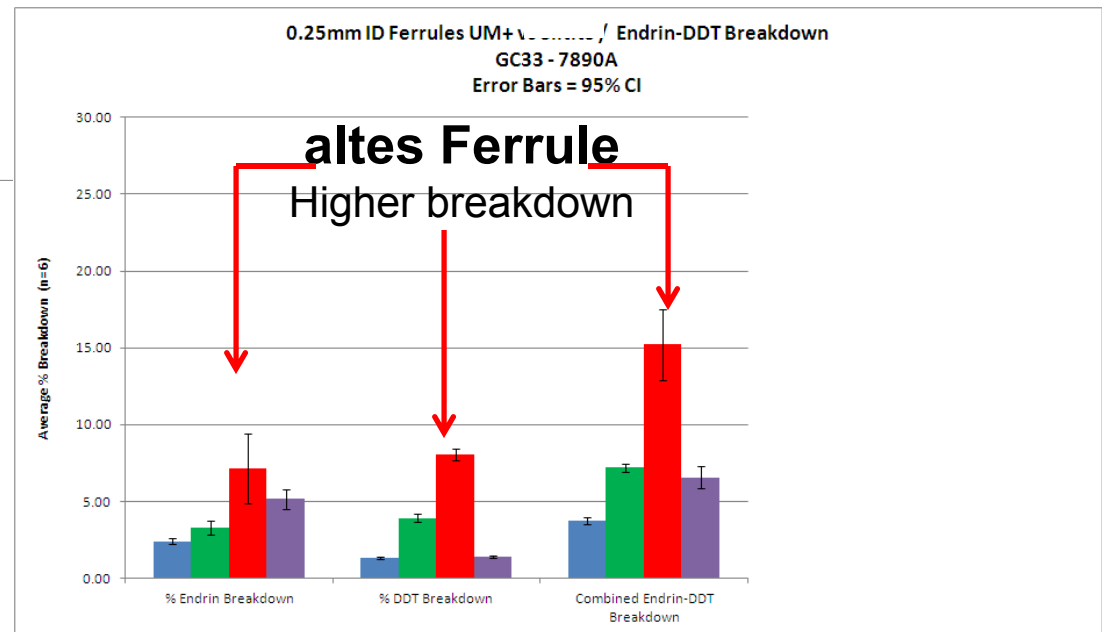
## Performance Metrics

1. % Breakdown of Endrin and DDT individually
2. % Breakdown of Endrin and DDT combined

# Flexible Metal Ferrule vs bisherigem Metallferrule Endrin-DDT Zerfall - Testresultate



- Vorkontrolle
- bisheriges Ferrule
- FMF + UM
- Nachkontrolle



**Flexible Metal Ferrule**  
Low breakdown



**Vielen Dank für Ihre Aufmerksamkeit!**



Agilent Technologies

# Compare current vs new Agilent ferrule

## Current Ferrule

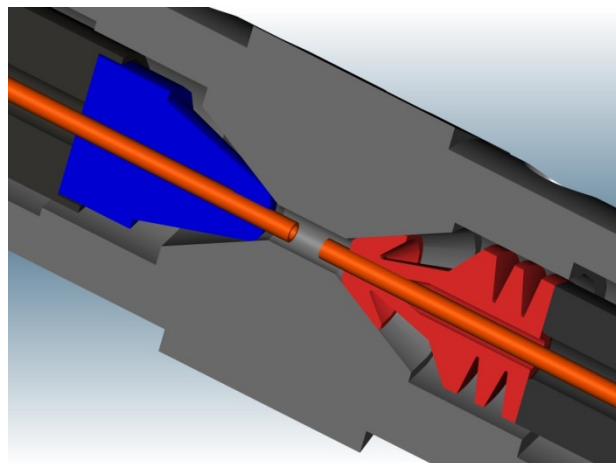
Stiff, prone to cause  
**damage to column**

Small ID compression (40  
to 60um) – **leaks**

Tolerances poorly  
controlled

Quality issues

- mixed inventory
- not-fitting
- column crushing
- leaking



## Agilent

### Flexi-Metal Ferrule

Computer aided Design  
Finite Element Analysis

More flexible

Less column damage

Large range of  
ID compression  
(100 to 120um) – less leaks

Agilent design alleviates current  
quality issues



# Metal (Siltite) Ferrules in Agilent Systems

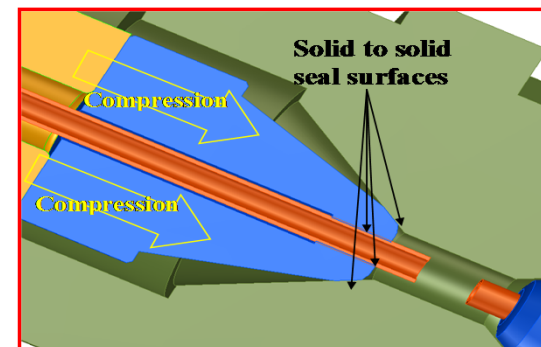


- Fastest growing family of GC ferrules
- Connections with Capillary Flow Technique devices
- Secure leak free connections LTM columns
- GC/MS transfer lines

**We experience high scrap rate due to non-fitting ferrules for LTM (Low Thermal Mass modules)**

***Computer aided theoretical modeling of Siltite design***

- ***Design flaw identified***
- ***ID spec and tolerance recommendations made to supplier but was considered too costly***



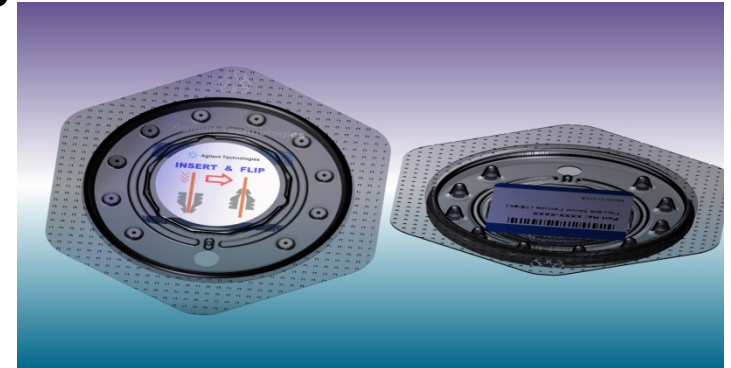
# New Dial Packaging

**Agilent**  
**Confidential**

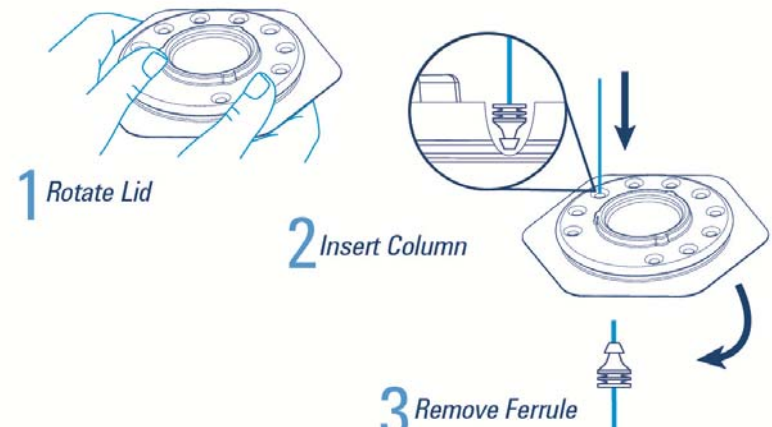


## Touchless dial package

- Easy column insertion
- No lost ferrules
- No contamination risks



ra Inert Flowpath branding  
and Agilent website details



Clear installation instructions  
on box & Agilent website

# High-Efficiency GC Säulen – Bestellnummern

65 Säulen sind verfügbar: 43 7" cage und 22 5" cage

Stationary Phase	Part Number (7" cage)	I.D. (mm)	Length (m)	Film Thickness (µm)	Part Number (5" cage)
DB-1	121-1012	0.18	10	0.18	121-1012E
	121-1013	0.18	10	0.40	121-1013E
	121-101A	0.18	10	0.20	
	121-1022	0.18	20	0.18	121-1022E
	121-1023	0.18	20	0.40	
	121-1043	0.18	40	0.40	121-1043E
HP-1	19091Z-577	0.18	20	0.18	19091Z-577E
DB-1ms	121-0122	0.18	20	0.18	121-0122E
HP-1ms	19091S-677	0.18	20	0.18	19091S-677E
DB-5	121-5012	0.18	10	0.18	121-5012E
	121-5013	0.18	10	0.40	
	121-5022	0.18	20	0.18	121-5022E
	121-5023	0.18	20	0.40	121-5023E
	121-5042	0.18	40	0.18	
HP-5	19091J-577	0.18	20	0.18	19091J-577E
DB-5ms	121-5522	0.18	20	0.18	121-5522E
	121-5542	0.18	40	0.18	
	121-5523	0.18	20	0.36	
HP-5ms	19091S-577	0.18	20	0.18	19091S-577E
DB-XLB	121-1222	0.18	20	0.18	121-1222E
	121-1232	0.18	30	0.18	

Stationary Phase	Part Number (7" cage)	I.D. (mm)	Length (m)	Film Thickness (µm)	Part Number (5" cage)
DB-35ms	121-3822	0.18	20	0.18	121-3822E
DB-17	121-1722	0.18	20	0.18	
	121-1723	0.18	20	0.30	
DB-17ms	121-4722	0.18	20	0.18	121-4722E
HP-50+	19091L-577	0.18	20	0.18	
DB-23	121-2323	0.18	20	0.20	
DB-225	121-2223	0.18	20	0.20	
DB-624	121-1324	0.18	20	1.00	121-1224E
DB-1301	121-1313	0.18	10	0.40	
DB-1701	121-0713	0.18	10	0.40	
	121-0722	0.18	20	0.18	
DB-WAX	121-7012	0.18	10	0.18	
	121-7022	0.18	20	0.18	121-7022E
	121-7023	0.18	20	0.30	121-7023E
	121-7042	0.18	40	0.18	121-7042E
	121-7043	0.18	40	0.30	
HP-INNOWax	19091N-577	0.18	20	0.18	19091N-577E
DB-5.625	121-5621	0.18	20	0.18	
	121-5622	0.18	20	0.36	
DB-VRX	121-1524	0.18	20	1.00	
	121-1544	0.18	40	1.00	121-1544E
DB-608	121-6822	0.18	20	0.18	

# Agilent J&W 5ms Ultra Inert Säulen

122-5522UI	DB-5MS Ultra Inert 25m, 0.25mm, 0.25um
122-5532UI	DB-5MS Ultra Inert 30m, 0.25mm, 0.25um
122-5533UI	DB-5MS Ultra Inert 30m, 0.25mm, 1.0um
122-5536UI	DB-5MS Ultra Inert 30m, 0.25mm, 0.50um
122-5552UI	DB-5MS Ultra Inert 50m, 0.25mm, 0.25um
122-5562UI	DB-5MS Ultra Inert 60m, 0.25mm, 0.25um
122-5563UI	DB-5MS Ultra Inert 60m, 0.25mm, 1.0um
123-5532UI	DB-5MS Ultra Inert 30m, 0.32mm, 0.25um
123-5533UI	DB-5MS Ultra Inert 30m, 0.32mm, 1.0um
123-5536UI	DB-5MS Ultra Inert 30m, 0.32mm, 0.50um
123-5563UI	DB-5MS Ultra Inert 60m, 0.32mm, 1.0um

19091S-577UI	HP-5MS Ultra Inert 20m, 0.18mm, 0.18um
19091S-133UI	HP-5MS Ultra Inert 30m, 0.25mm, 0.50um
19091S-213UI	HP-5MS Ultra Inert 30m, 0.32mm, 1.0um
19091S-233UI	HP-5MS Ultra Inert 30m, 0.25mm, 1.0um
19091S-413UI	HP-5MS Ultra Inert 30m, 0.32mm, 0.25um
19091S-431UI	HP-5MS Ultra Inert 15m, 0.25mm, 0.25um
19091S-433UI	HP-5MS Ultra Inert 30m, 0.25mm, 0.25um

# Kundenaussage

**Customer Testimonial – Lisa Wool at EPA in her article published in Journal of Chromatographic Science:**

**Fast analysis conditions were used routinely in the laboratory over a 9-month period with surprisingly good durability... They (0.18 mm I.D. columns) typically lasted approximately as long as what was typically seen in the past with the 0.32 mm I.D. columns... Twice as many samples were analyzed in that same time frame.**



# Detektorvoraussetzungen für HE Säulen

**Seit:**

**6890 FID, NPD, FPD, uECD, TCD und für alle späteren**

**Sampling rate – 1 Kanal:**

**200 Hz**

**Minimale Peakbreite:**

**0.001min = 0.06 Sekunden**

**Sampling rate – 2 Kanäle:**

**100 Hz**

**Minimale Peakbreite:**

**0.002 min = 0.12 Sekunden**

**Seit 6890 MSD**

**Sampling rate – Scan Geschwindigkeit:**

**$2^n$  (n=0-7)**

**für 0.25 um ID  $2^2$  oder  $2^3$  =**

**4 Wiederholungen pro SCAN**

**Datenpunkt für den Massenbereich von 35-450 sind das 3 Datenpunkte pro Sekunde**

**Für 0.18 mm ID:  $2^1$  =**

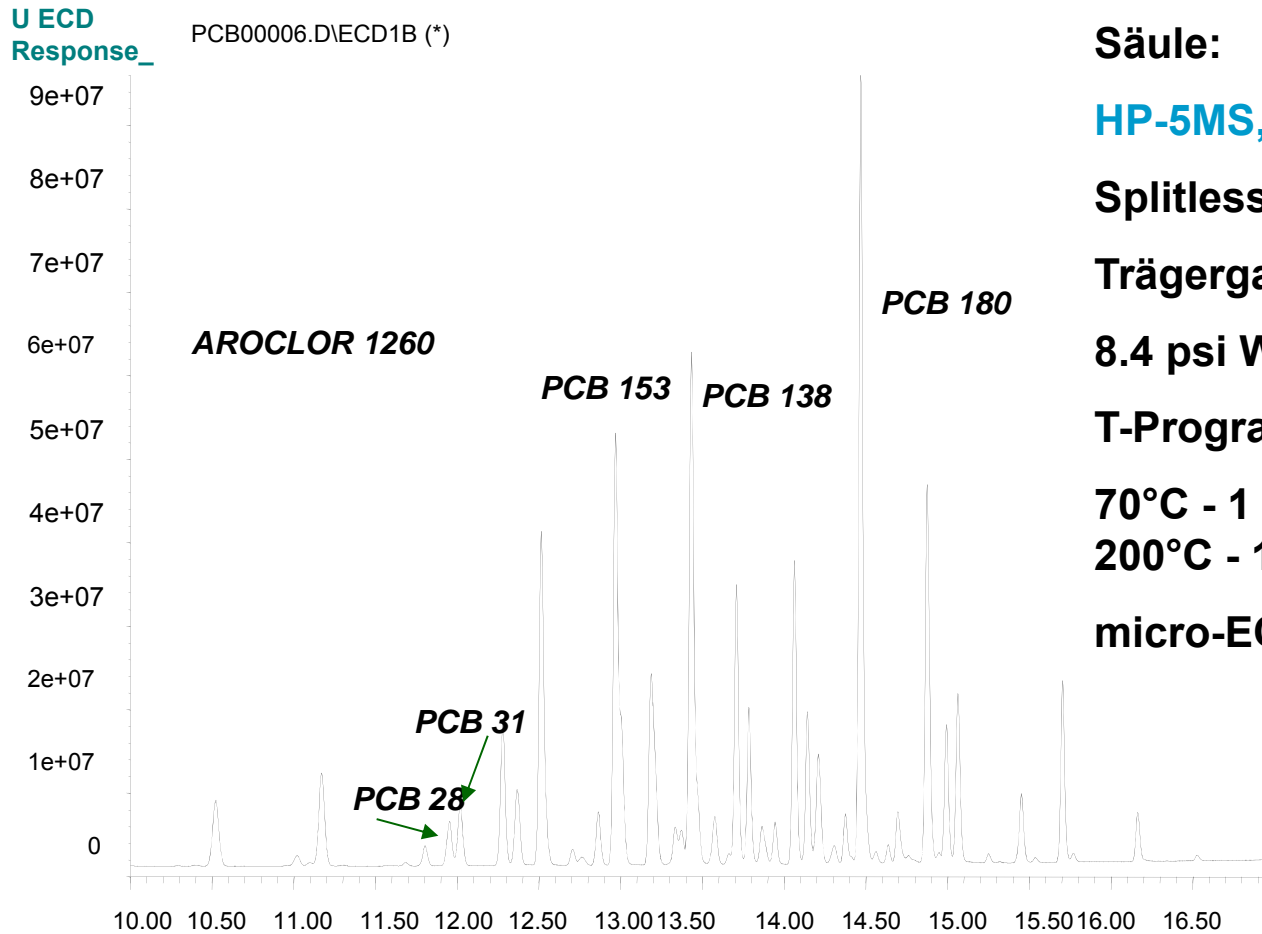
**2 Wiederholungen pro SCAN-**

**Datenpunkt, für den Massenbereich von 35 - 450 sind das 6 Datenpunkte pro Sekunde, liefert 12 Punkte für den 2 Sek-Peak und 24 Punkte für den 4 Sek-Peak**

**Mittlere Peakbreite HE Säulen: 2-4 Sek / Min.: 13 Datenpunkte pro Peak**



# Schnelle PCB Analyse



**Säule:**

**HP-5MS, 12 m x 0.20 mm i.d. x 0.33 µm**

**Splitless Injektion**

**Trägergas:**

**8.4 psi Wasserstoff (angepasst via RTL).**

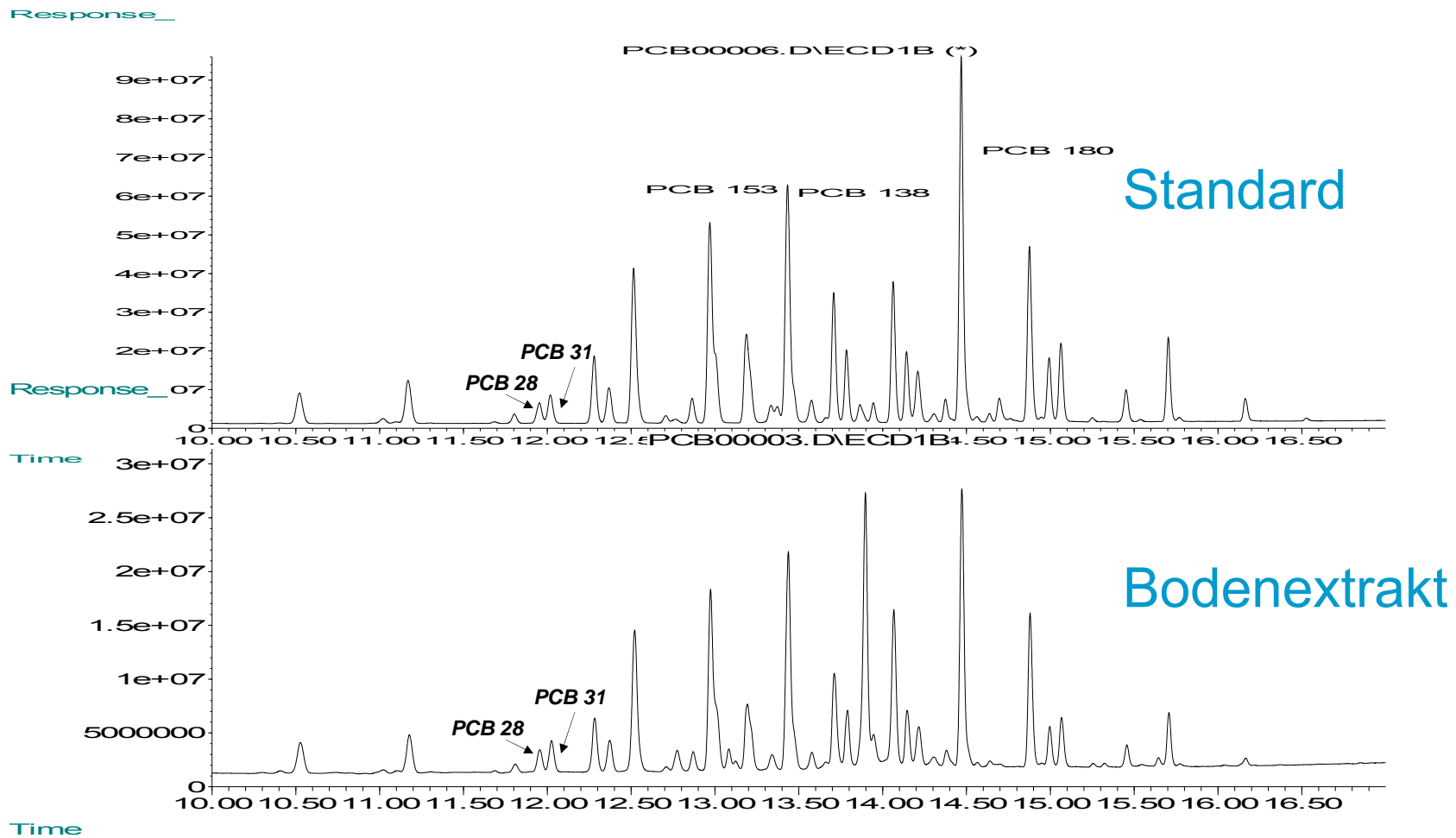
**T-Programm:**

**70°C - 1 min - 50°C/min - 150°C - 6°C/min**

**200°C - 16°C/min - 320°C**

**micro-ECD Detektion**

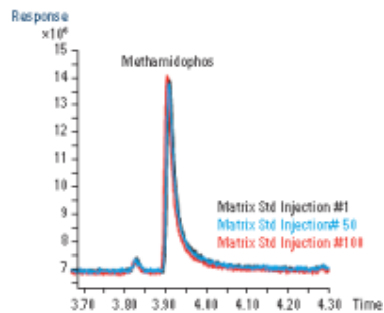
# Schnelle Analyse PCB aus Bodenextrakt



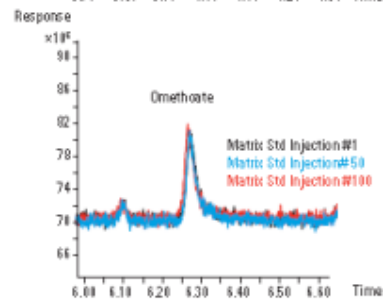
# Organophospho Pestizide mit & ohne “Analyte Protectant” - Ultra Inert Liner mit Glaswolle

Matrix: Olivenöl, Quechers  
“Analyte Protectant”: Gulonolacton  
Säule: DB-35ms UI

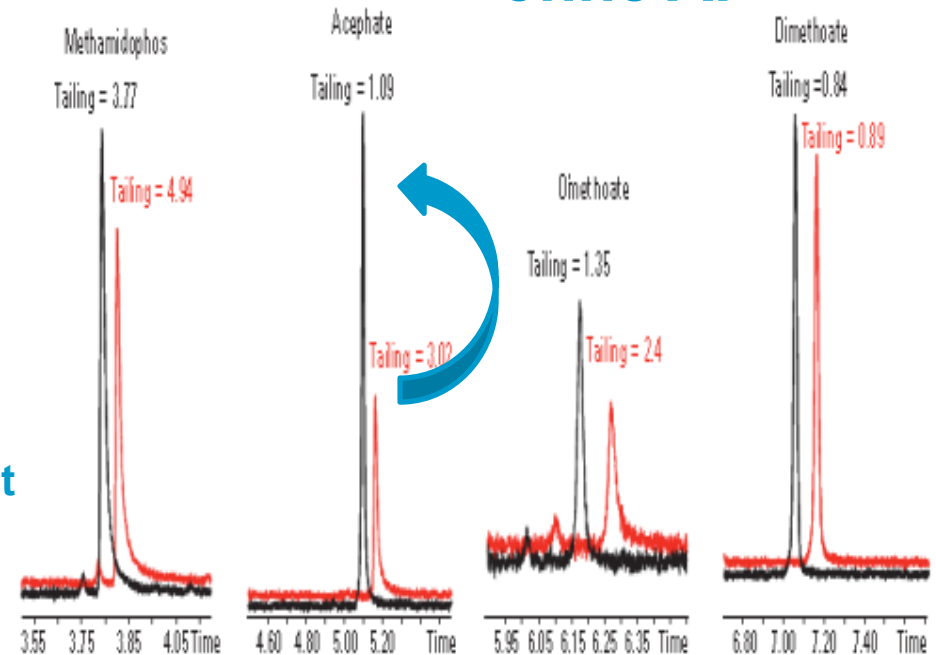
■ mit AP  
■ ohne AP



**Overlay:**  
1<sup>st</sup>, 50<sup>th</sup>, 100<sup>th</sup> Injektion  
ohne AP mit UI liner



**exzellente  
Reproduzierbarkeit  
verbessert die Robustheit  
der Methode  
Weniger Linertausch  
Geringere Kosten**



Für die Pestizidrückstandsanalytik erzielt man bessere Signalhöhen mit “Analyte Protectant” (100· Injektion)

**5990-8235EN**

UI

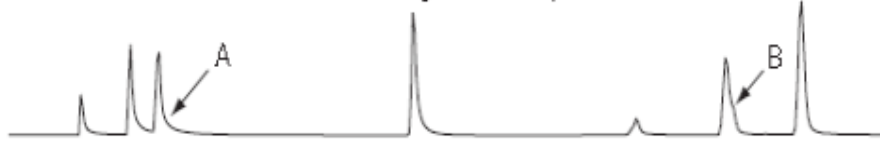
# Forensische Drogenanalytik

## Peakformen

Restek Base gooseneck splitless liner with wool



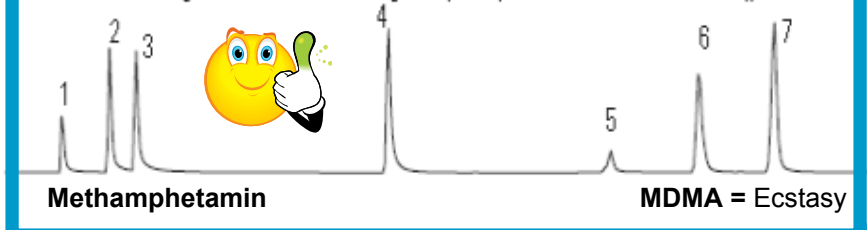
Restek IP SemiVolatile gooseneck splitless liner with wool



Restek Sky gooseneck splitless liner with wool



Agilent Ultra Inert single taper splitless liner with wool (p/n 5190-2)



**5990-7596EN**

**Ultra Inert Liner  
liefern  
Lot-to-Lot  
Reproduzierbarkeit**

Compounds	Liner 1 (Lot 1)	Liner 2 (Lot 1)	Liner 3 (Lot 1)	Liner 4 (Lot 2)	Liner 5 (Lot 3)	Liner 6 (Lot 4)	Mean RF	RSD
Methamphetamine (3)	0.875	0.876	0.882	0.940	0.955	0.904	0.905	3.8
MDMA (6)	0.807	0.789	0.783	0.848	0.874	0.841	0.824	4.4
Phencyclidine (9)	0.494	0.510	0.494	0.488	0.509	0.521	0.503	2.5
Cocaine (11)	0.636	0.645	0.647	0.637	0.660	0.668	0.649	2.0
Oxazepam (13)	0.050	0.055	0.052	0.055	0.062	0.057	0.055	7.6
Codeine (14)	0.096	0.098	0.095	0.090	0.099	0.102	0.097	4.2
Oxycodone (19)	0.073	0.071	0.070	0.076	0.082	0.080	0.075	6.5
Temazepam (20)	0.101	0.121	0.115	0.088	0.096	0.104	0.104	11.7
Heroin (22)	0.097	0.099	0.096	0.095	0.100	0.102	0.098	2.7
Nitrazepam (23)	0.038	0.032	0.037	0.034	0.037	0.036	0.036	6.3
Clonazepam (24)	0.035	0.035	0.034	0.032	0.034	0.033	0.034	3.5
Trazodone (28)	0.061	0.065	0.064	0.058	0.060	0.064	0.062	4.4

**Ultra Inerte Liner für DoA:  
Bessere Peakform, keine Degradation  
Reproduzierbarkeit heisst bessere Datenqualität**

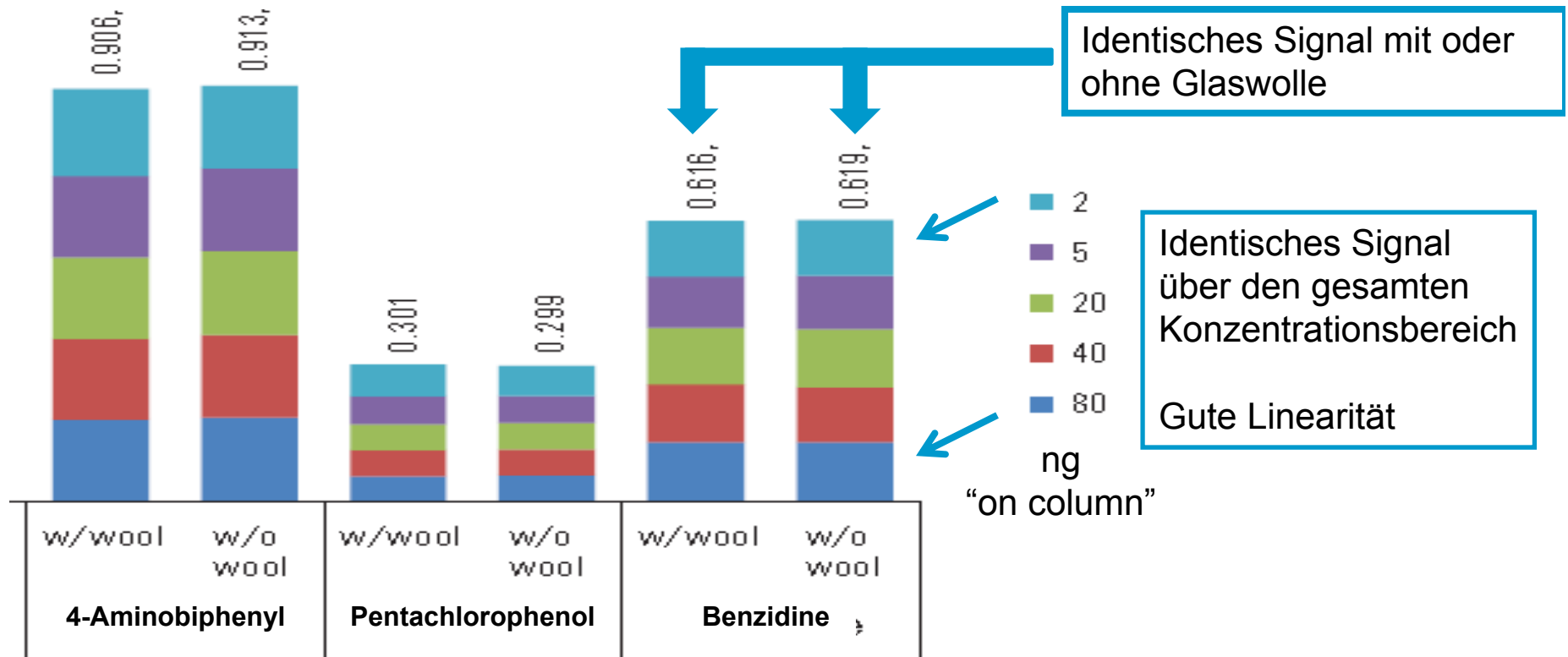
UI

# Umwelt: Semivolatiles mit GC/FID, EPA 8270

**5990-7381EN**

Für die EPA 8270 Applikation schützt Glaswolle Säule und Detektor aber empfindliche Verbindungen könnten mit Glaswolle leicht degradieren.

Glaswolle der Ultra Inert Liner heisst bewiesene Linearität für schwierige EPA 8270 Verbindungen und optimaler Systemschutz. Keine Signalunterschiede im Vergleich zu Linern ohne Glaswolle!



# Ultra Inert Applikationsbeispiel - PAH Analyse

## GC/MSD Bedingungen

Sample: 10ug/ml PAH Standard PN # 8500-6035

Column: **DB-5MS Ultra Inert**

30m x 0.25mm x 0.25um (**Agilent PN # 122-5532UI**)

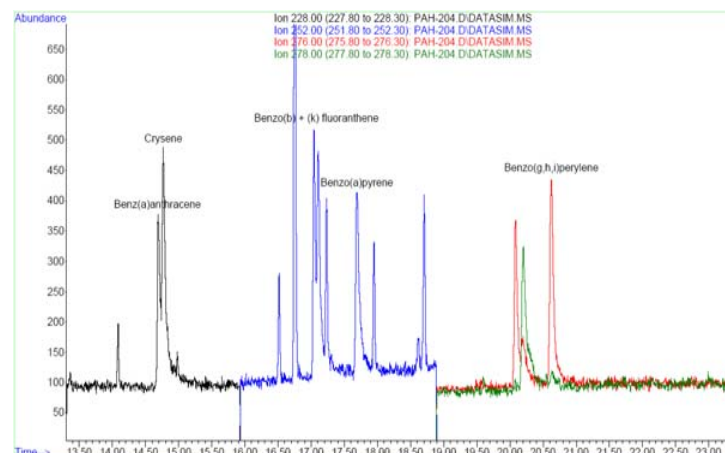
Carrier: Helium 45cm/sec, constant flow

Oven: 55°C (1min) to 320°C (25°/min), hold 3 min

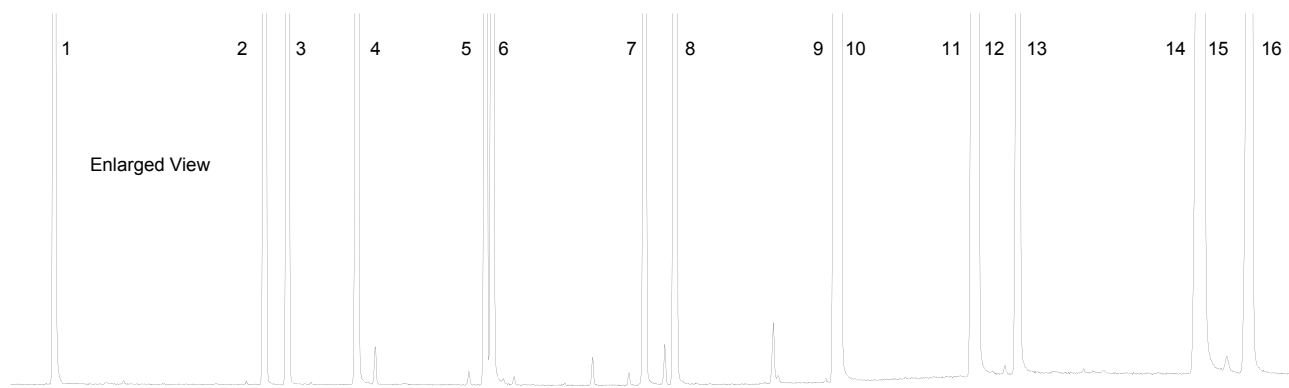
Injection: Pulsed splitless, 300°C, 40psi until 0.2 min, purge flow  
30ml/min at 0.75 min

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



# Agilent J&W Ultra Inert - PAH Analyse

## GC/MSD Bedingungen

Sample: 10ug/ml PAH Standard PN # 8500-6035

Column: **DB-5MS Ultra Inert**

30m x 0.25mm x 0.25um (**Agilent PN # 122-5532UI**)

Carrier: Helium 45cm/sec, constant flow

Oven: 55°C (1min) to 320°C (25°/min), hold 3 min

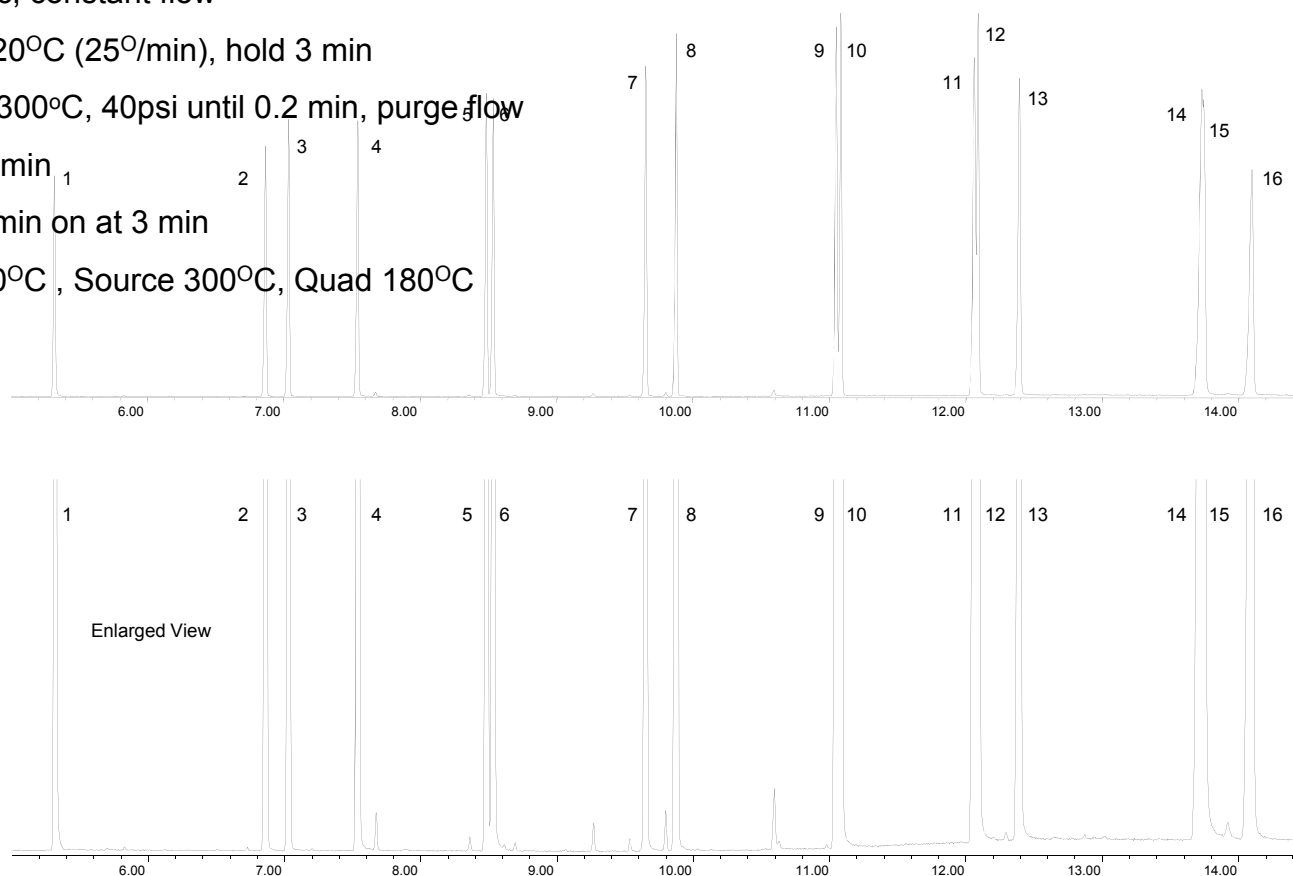
Injection: Pulsed splitless, 300°C, 40psi until 0.2 min, purge flow

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
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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



# Lebensdauerbetrachtung: Applikationsbeispiel aus der Umweltanalytik – EPA 8270 - Semivolatiles

- Schnellere GC Methode mit **DB-5** 20m x 0.18 mm I.D.
- Reduktion der Analysendauer von 25 Min. auf 17 Min. => **30% schneller**

Column Diameter (mm)	Length (m)	Film (um)	# Column Changes/ Month	Column Cost/ Month	Column Cost/ Sample
0.25	30	0.25	1.3	642	\$ 1.53
0.18	20	0.36	2.0	820	\$ 1.41
0.18	20	0.36	2.6	1075	\$ 1.41

- Dieses Testmodel wurde mit unseren Kunden ermittelt.
- In diesem speziellen Fall, Vergleich von 0.18 mm vs. 0.25 mm, musste die Säule mit dem kleineren Durchmesser aufgrund der Matrixbelastung **1.5x bis 2x Mal öfter gewechselt werden** und trotzdem konnte eine **9% ige Reduktion der Kosten pro Probe erzielt werden.**

# Grob Testmischung und Analysenbedingungen

	<b>GC:</b> Agilent 6890N
1. 1-Octanol	<b>Sampler:</b> Agilent 7683B, 5 $\mu$ L syringe (Agilent part # 5181-1273), 1.5 $\mu$ L split injection, 4 ng each component on column
2. n-Undecane	<b>Carrier:</b> Hydrogen constant pressure 38 cm/s
3. 2,6-Dimethylphenol	<b>Inlet:</b> Split/splitless; 250% C, 1.4 ml/min. column flow, split flow 75 ml/min.
4. 2,6-Dimethylaniline	<b>Liner:</b> Deactivated single taper w glass wool (Agilent part # 5183-4647)
5. n-Dodecane	<b>Column A:</b> 30m x 0.25mm x 0.25 $\mu$ m 5%-Phenyl Säule
6. Naphthalene	<b>Column B:</b> Agilent J&W DB-5ms Ultra Inert 30m x 0.25mm x 0.25 $\mu$ m (Agilent part # 122-5532UI)
7. 1-Decanol	<b>Oven:</b> <b>120% C isotherm</b>
8. n-Tridecane	<b>Detection:</b> FID at 325% C, 450 ml/min. air, 40 ml/min. hydrogen, 45 ml/min. nitrogen makeup
9. Methyl decanoate	

# Grob Test Mischung

