New Specialized GC Columns for the Petroleum Industry

-Integrated Particle Trap PLOTs
-DB-Sulfur SCD

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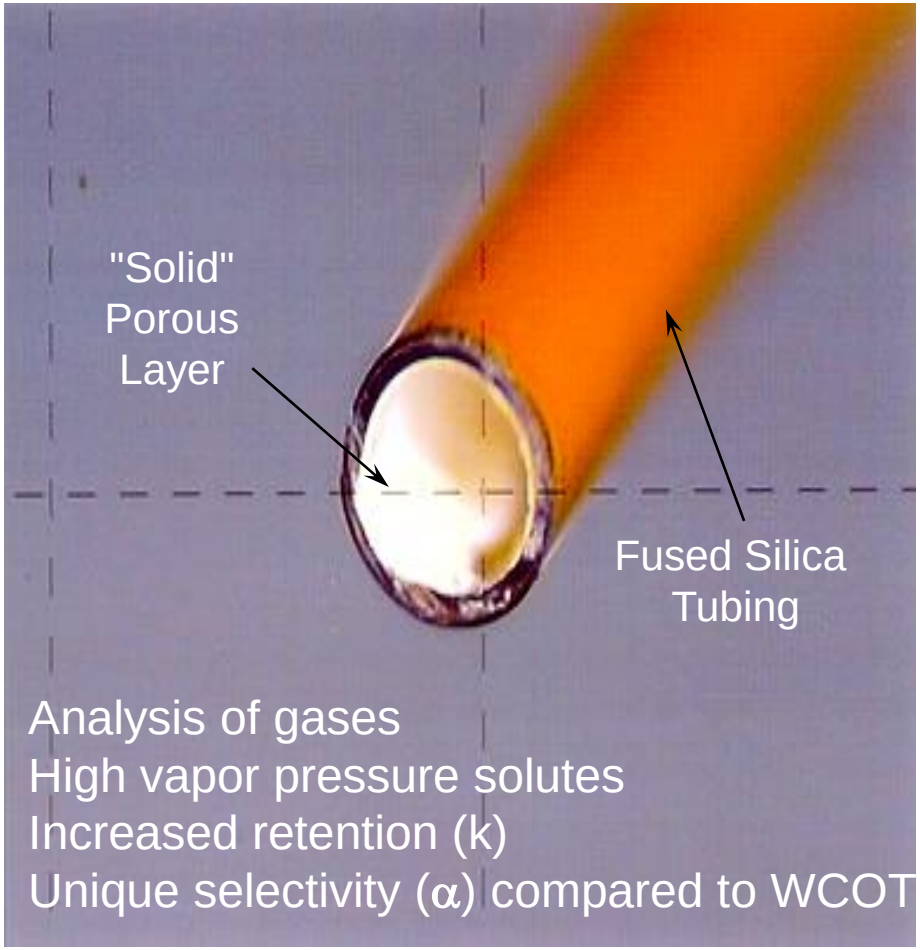
Porous Layer Open Tubular (PLOT) Columns



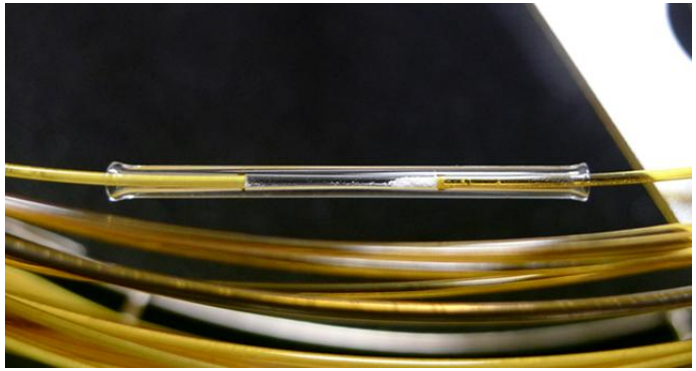
Challenges:

- ❖Stationary phase particle shedding
- ❖ Detector spikes impacts results
- ❖ Changes restriction interferes with instrument control/tuning
- ❖ Risks switching valves, CFT devices & connectors
- ❖ Can not be combined with GCMS

Solutions for PLOT Column issue--- stationary phase particle shedding

1. Install a particle trap on the end of the column

- Drawbacks: set-up time, prone to leaks, clog, add labor cost…



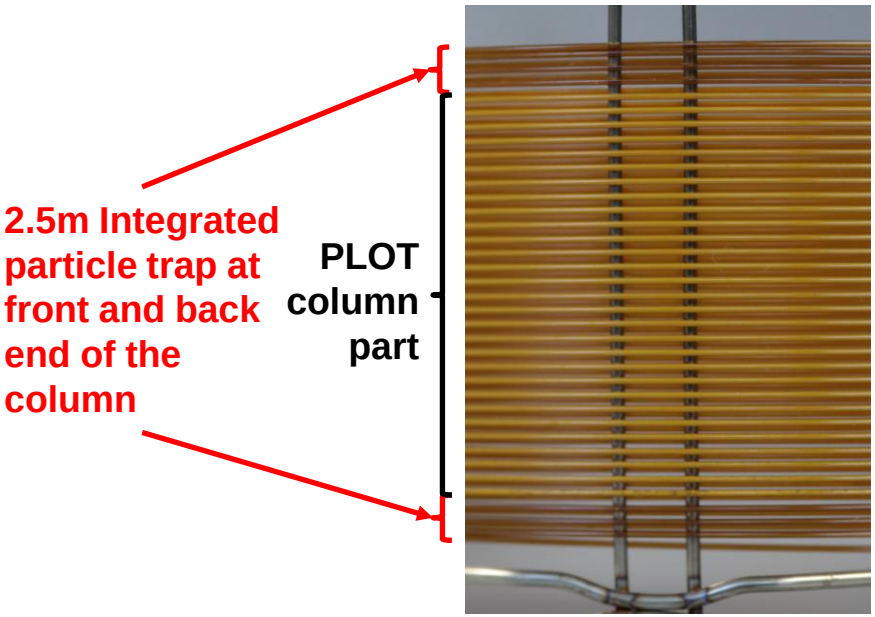
2. Install inline filters

- Drawbacks: eventually clog and cause flow restriction over time

3. Just live with it - ***majority of analysts***

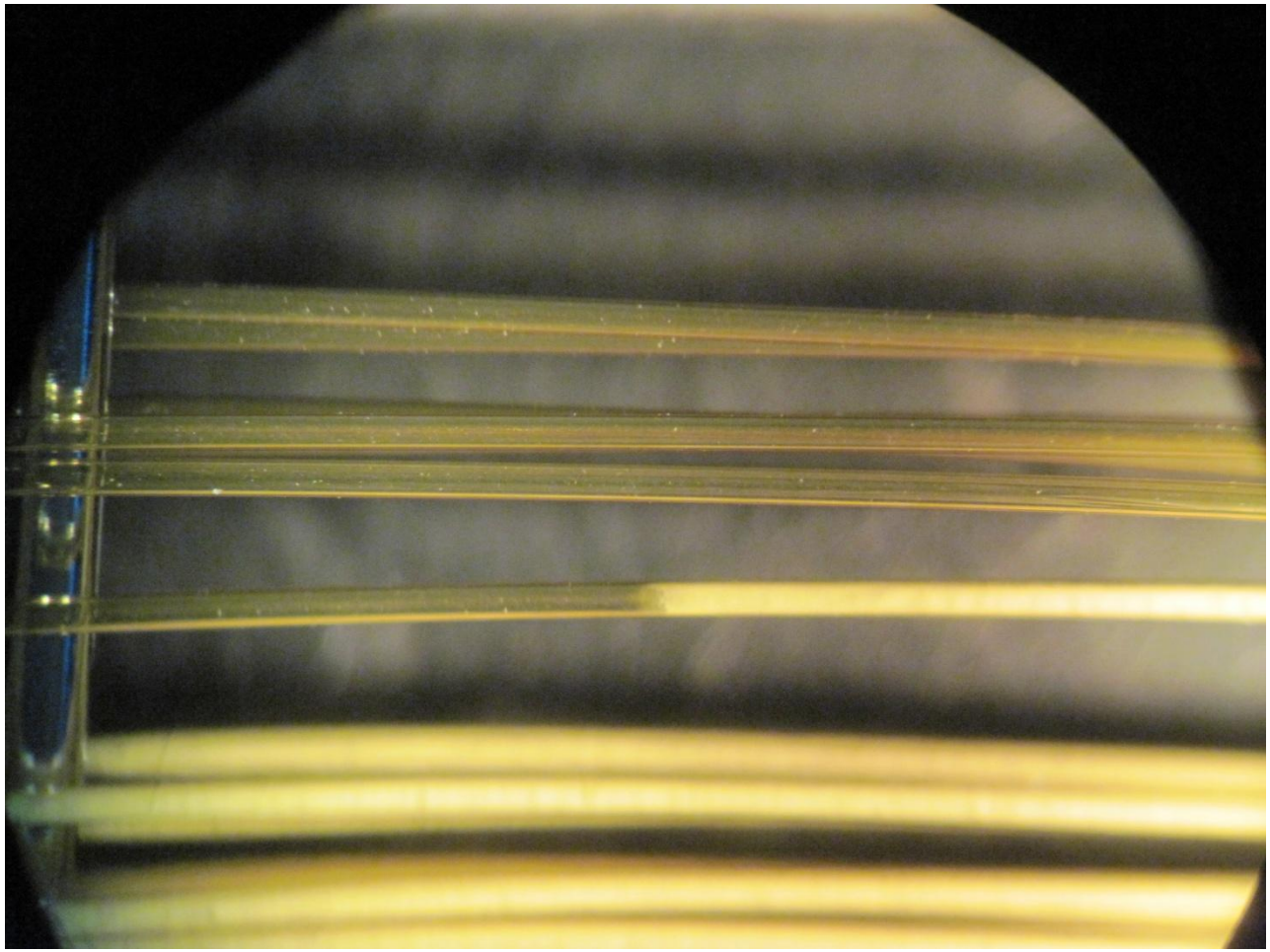
What is an Integrated Particle Trap PLOT Column?

PLOT columns with 2.5 meter integrated particle traps on both ends virtually eliminates the classic particle shedding problem

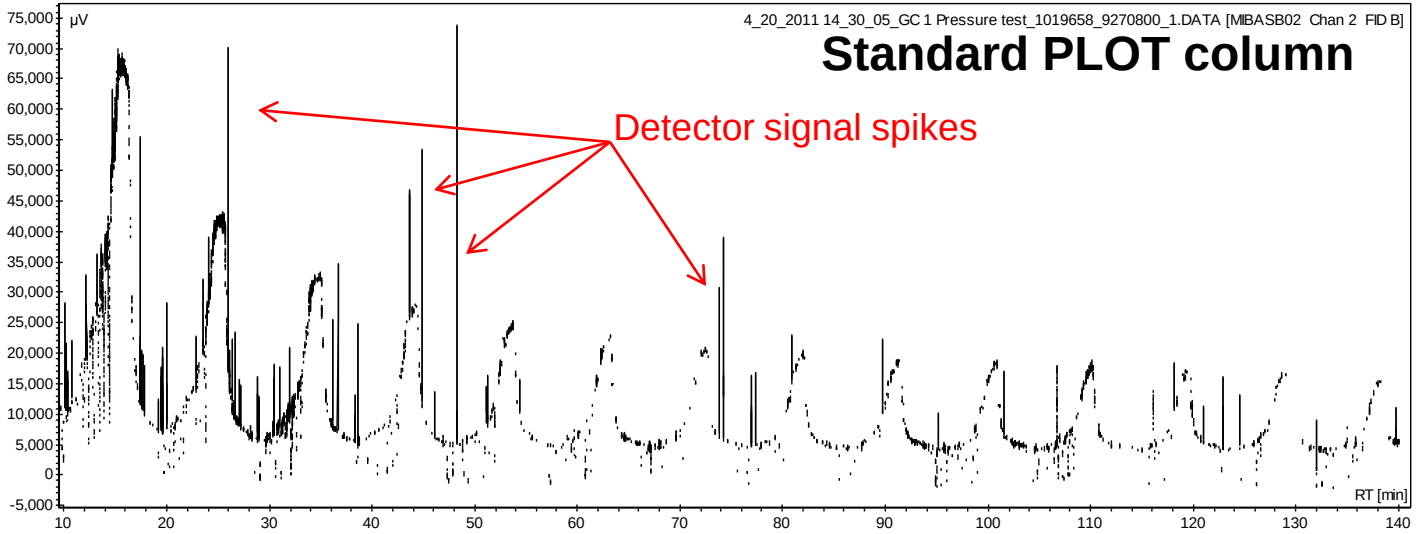


- Particle traps are integrated – no unions and/or fittings
- Compatible with capillary GC, GC/MS and valve switching GC systems including Capillary Flow Technology (CFT)
- Similar selectivity, plates and peak shape performance to existing Agilent J&W PLOT columns

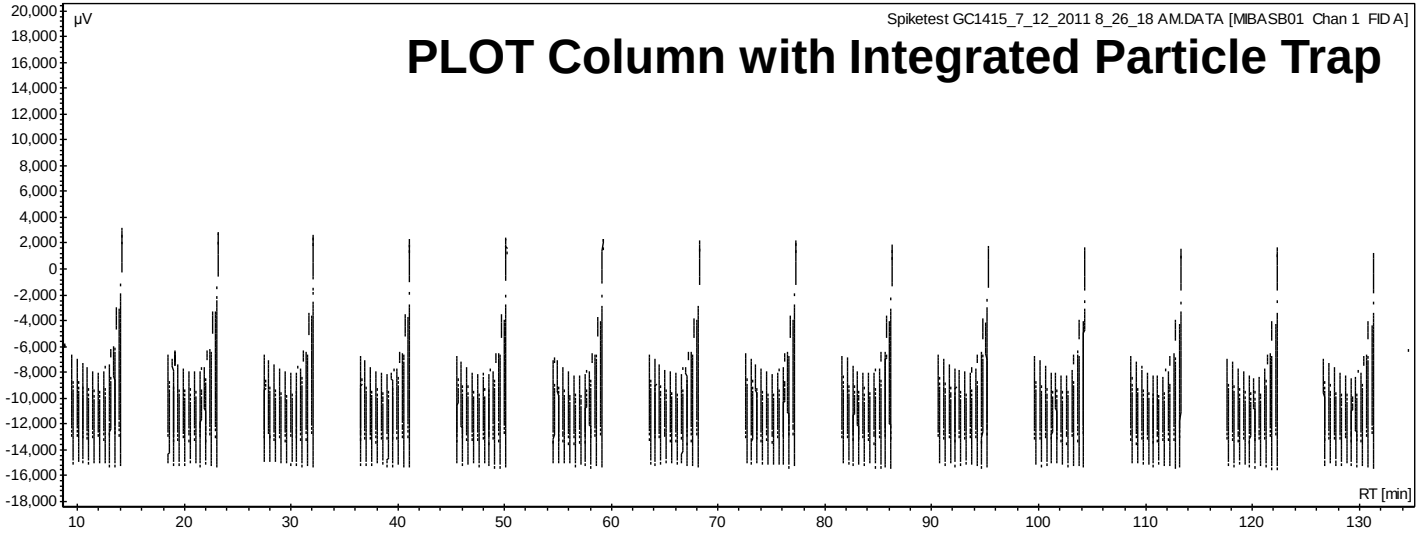
The Column and Integrated Particle Trap



No Detector Spikes Observed on PT Columns with Repeated Temperature and Pressure Cycling



- Temperature: 150°C + 20°C/min → 250°C;15 times
- Pressure 3x optimum
- Each run switch off/on carrier gas 10 times



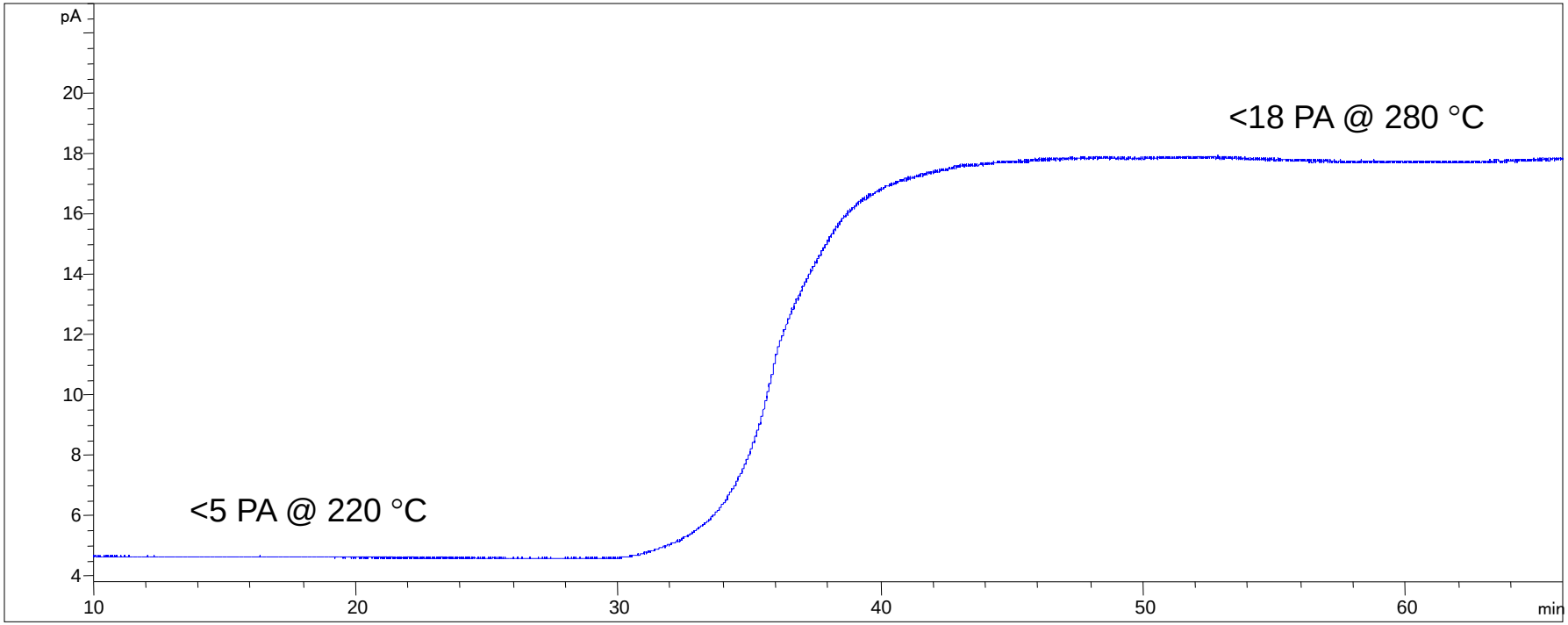
The unusual "chromatogram" shows the detector signal profile of the temp and pressure cycling

FID Baseline Testing from 220°C to 280°C

Column: PoraBond Q PT, 25m×0.25mm,3um
(30 meter total length)

Carrier : Helium, 36cm/s @220 ℃
Oven: : 220 °C for 30 min
220 °C - 280 °C at 10°C/min
280 °C for 30 min
Detector: FID, 300 ℃

No spikes

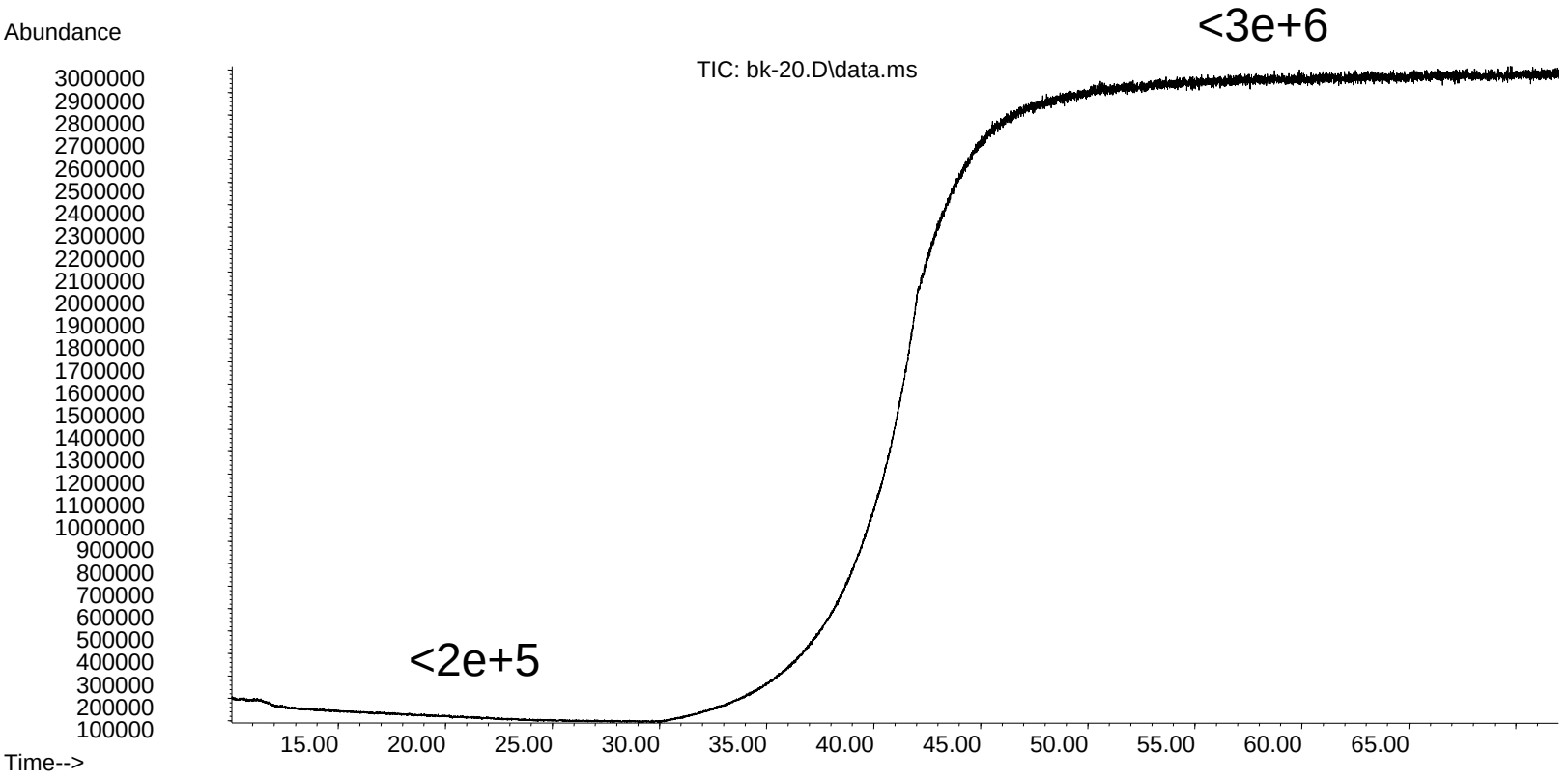


MSD! Baseline Testing from 220°C to 280°C

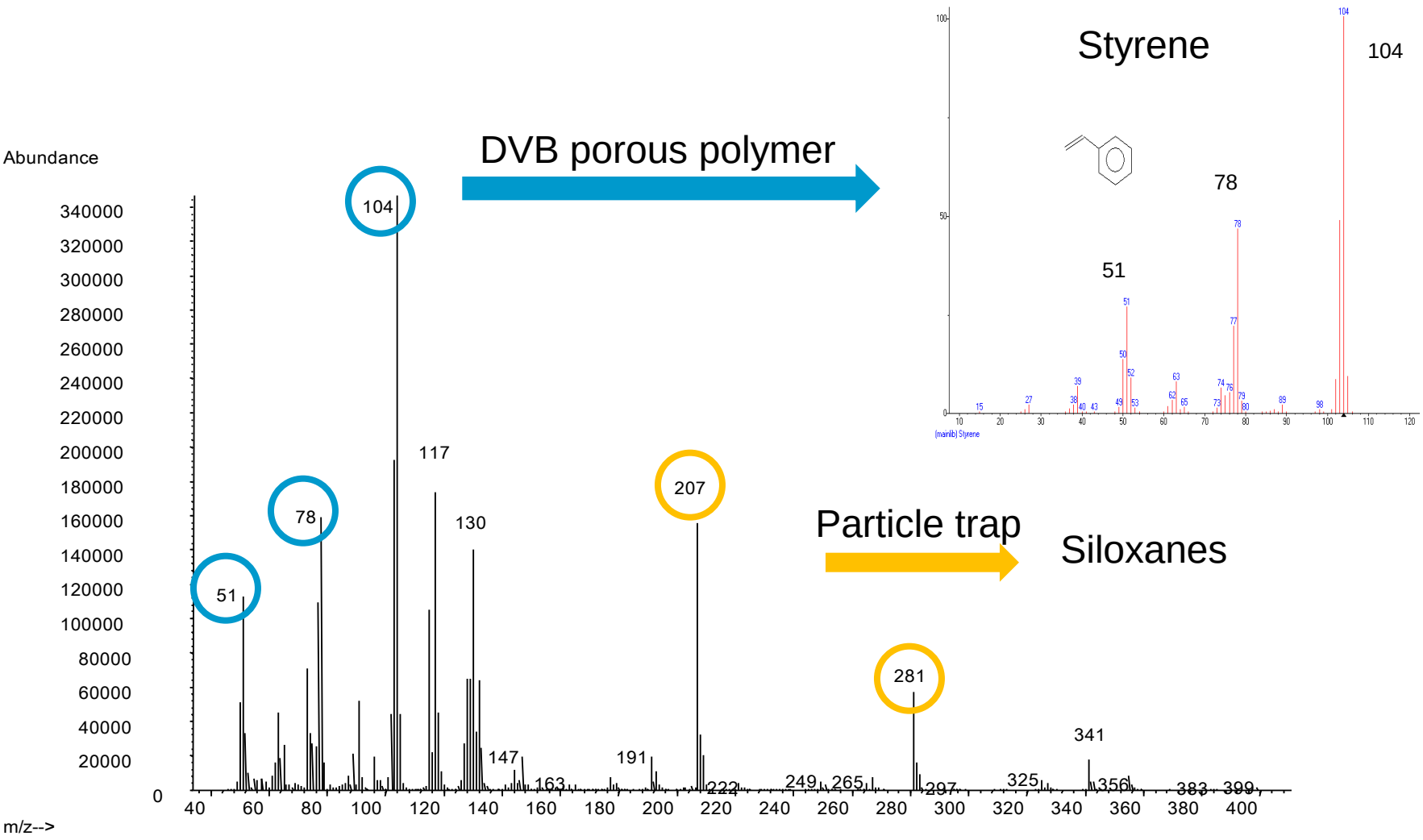
Column: PoraBond Q PT, 25m×0.25mm,3um
(30 meter total length)

Carrier : Helium, 34cm/s @220 ℃
Oven: : 220 °C for 30 min
220 °C - 280 °C at 5°C/min
280 °C for 30 min
Detector: **MSD**, 280 ℃ Transfer line, full Scan at m/z 40-300

No spikes



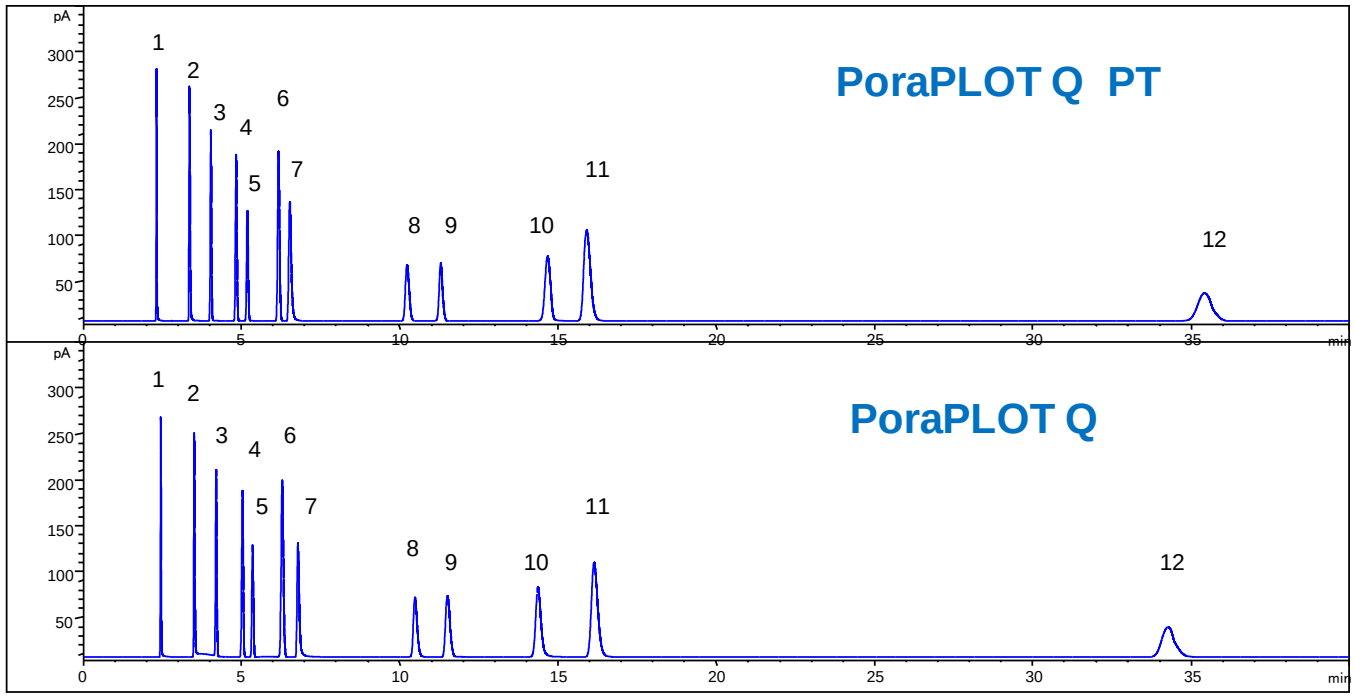
“Clean” Mass Spectrum, PoraBond Q PT at 280°C



Similar Selectivity

Solvent Analysis

Carrier : Helium, 5.25 mL/min
Oven: 150℃
Inlet: 200 ℃, split ratio=60:1
Detector: FID 250℃
Inj. Vol: 0.2uL



1. Methanol
2. Ethanol
3. Acetonitrile
4. Acetone
5. Methylene Chloride
6. Diethyl ether
7. 1-propanol
8. Trichloromethane
9. Ethyl acetate
10. Hexane
11. Benzene
12. Heptane

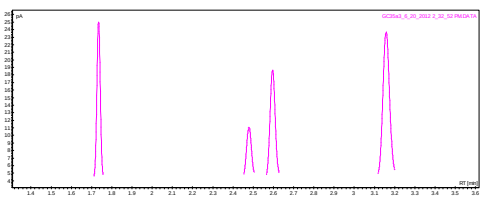
Differences observed between our standard PLOT columns and their PLOT PT counterparts shows that the variability in results is generally within the column to column reproducibility range for PLOT column manufacturing

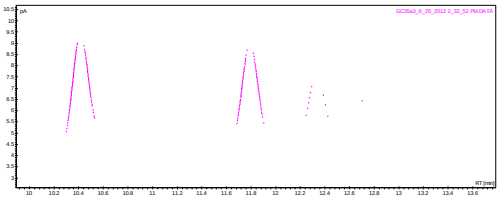
Similar Selectivity

Important Application C2 – C3

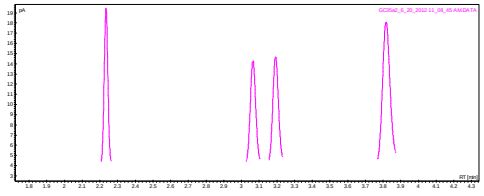
PoraBOND Q :C1-C2 PoraBOND Q: C3

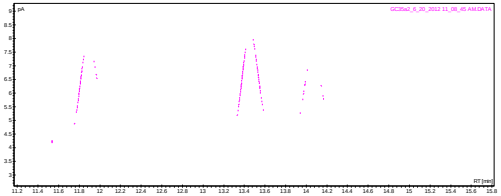
With PT





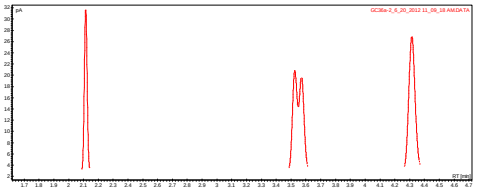
Without PT

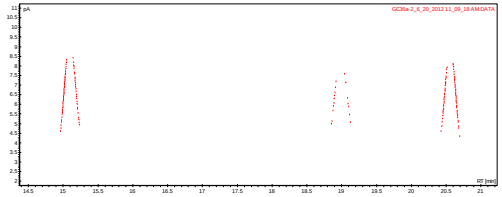




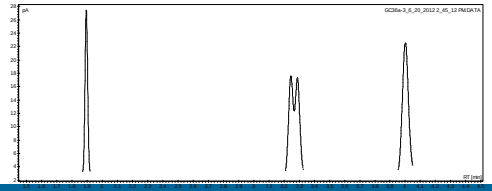
PoraPLOT Q :C1-C2 PoraPLOT Q: C3

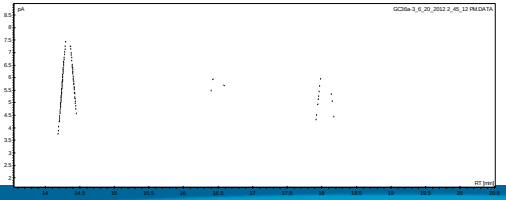
With PT





Without PT



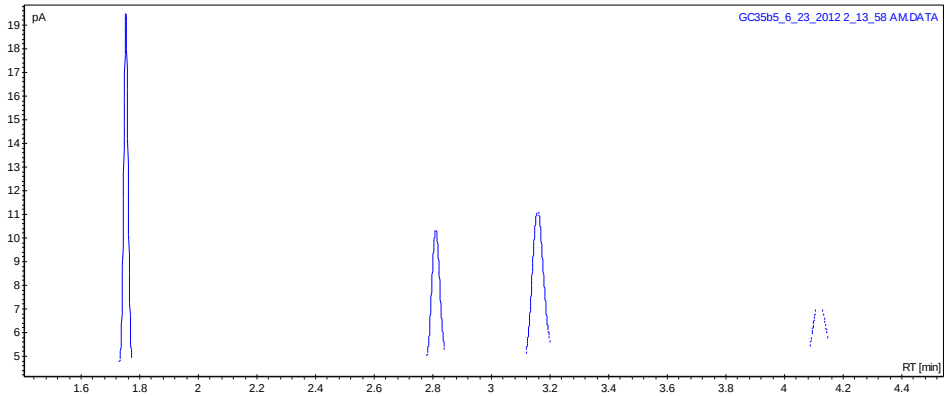


Similar Selectivity

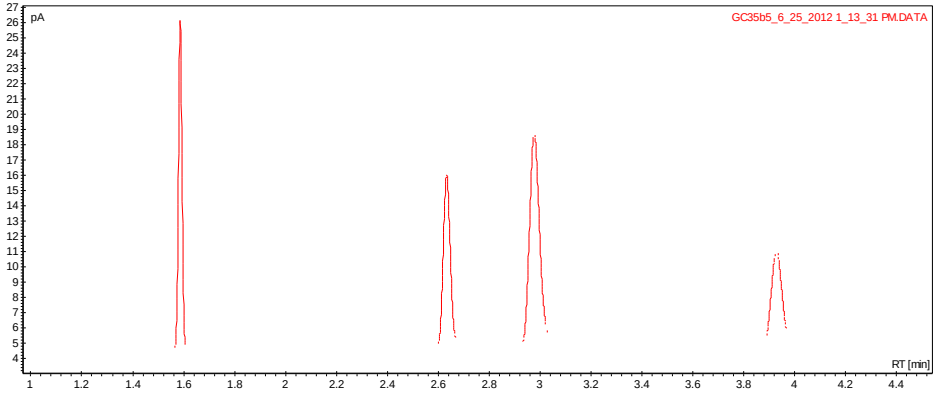
Important Application C1 – C2

PoraPLOT U: C1-C2

With PT



Without PT



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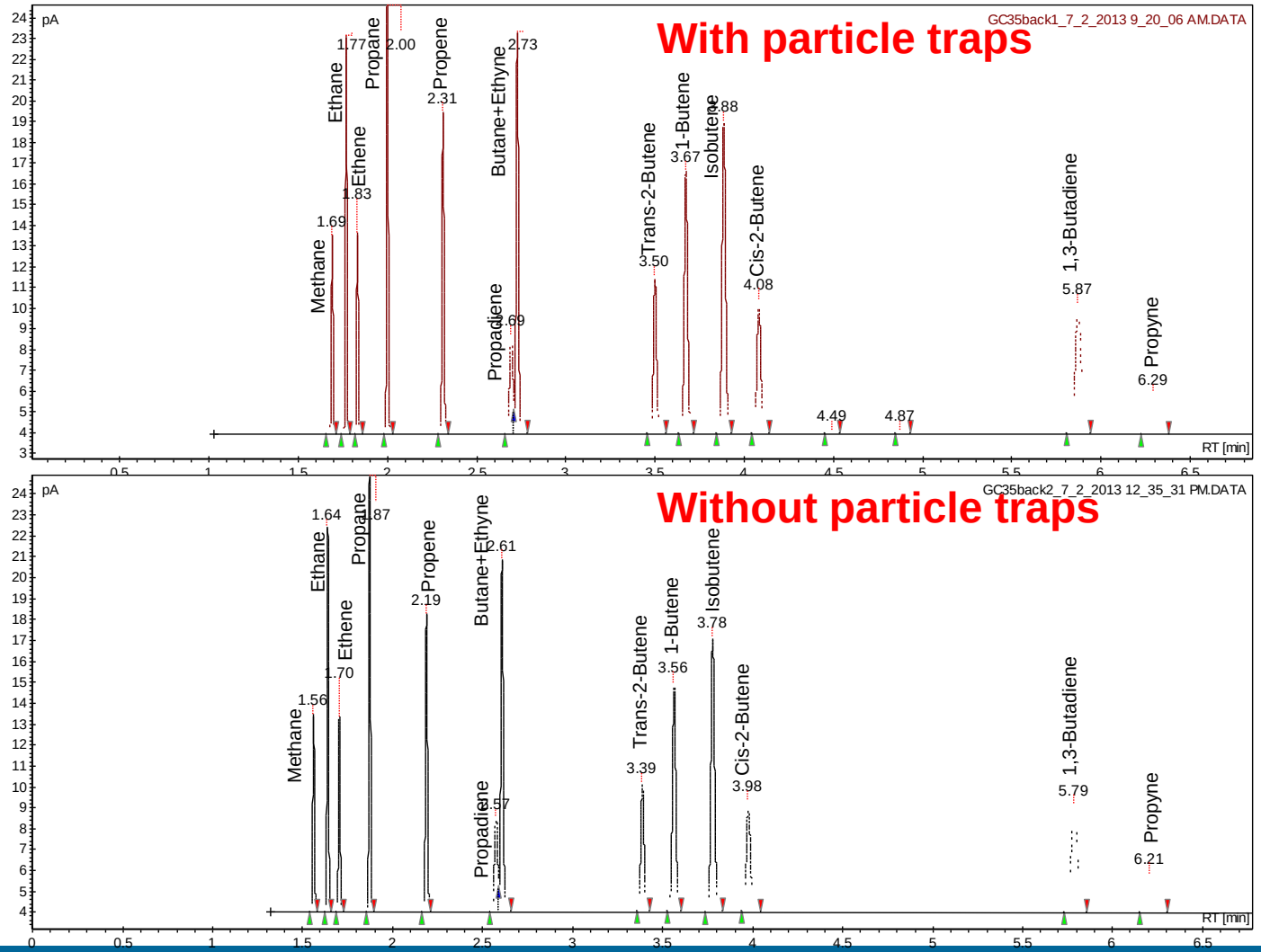
Influence of integrated particle traps on selectivity (Aluminum oxide PLOT)

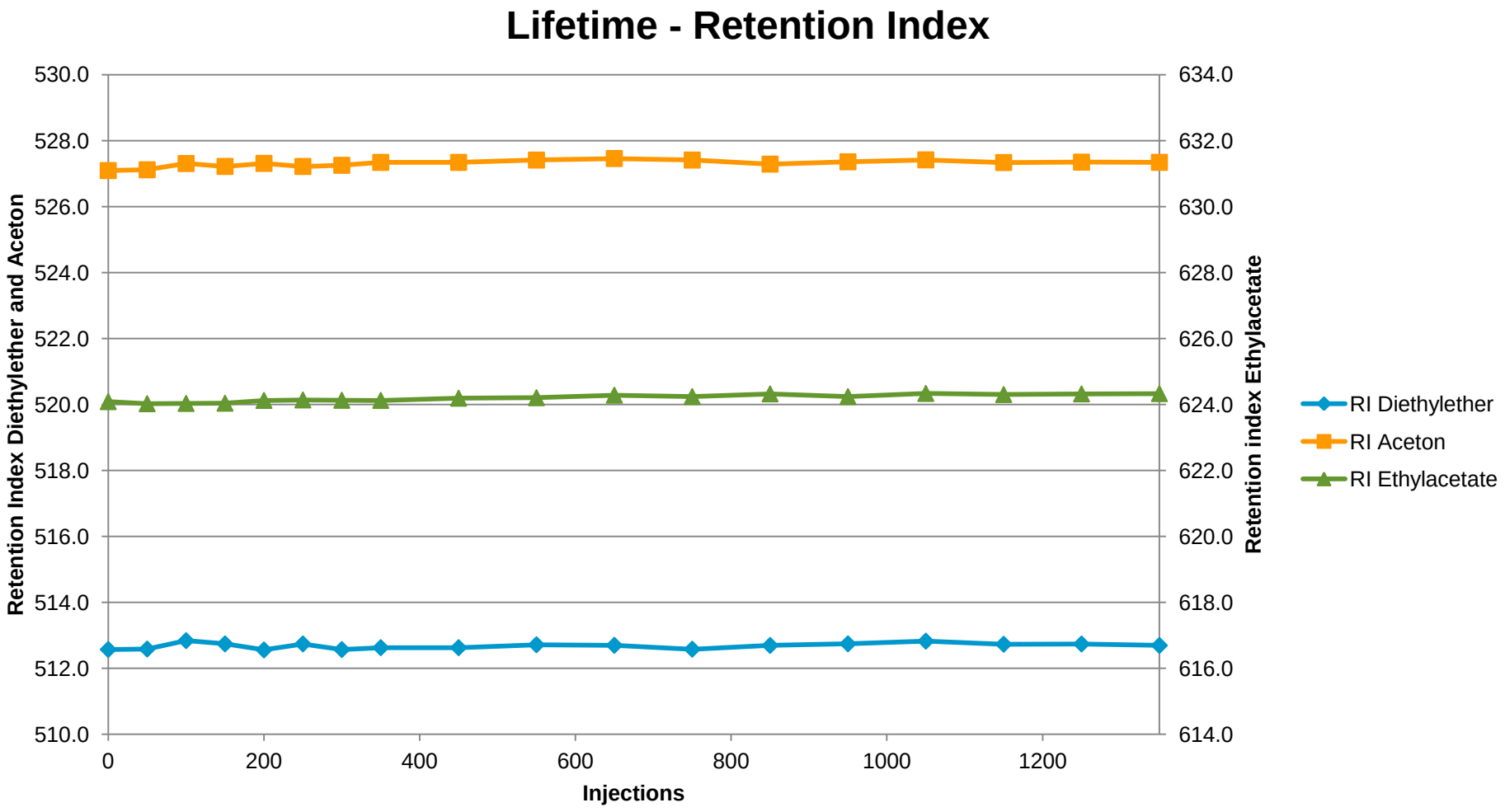


- First test with integrated particle traps
- Second test after removal of particle traps (pressure adjusted to correct for length difference)

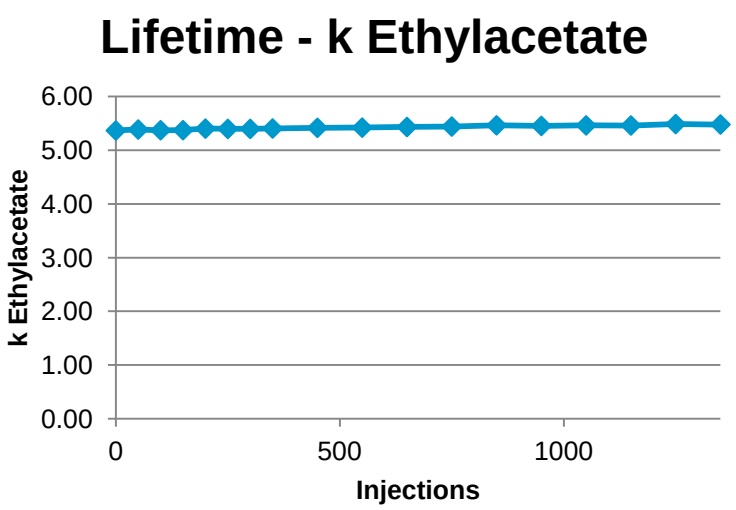
Lifetime test: Porous Polymer PLOT PT columns

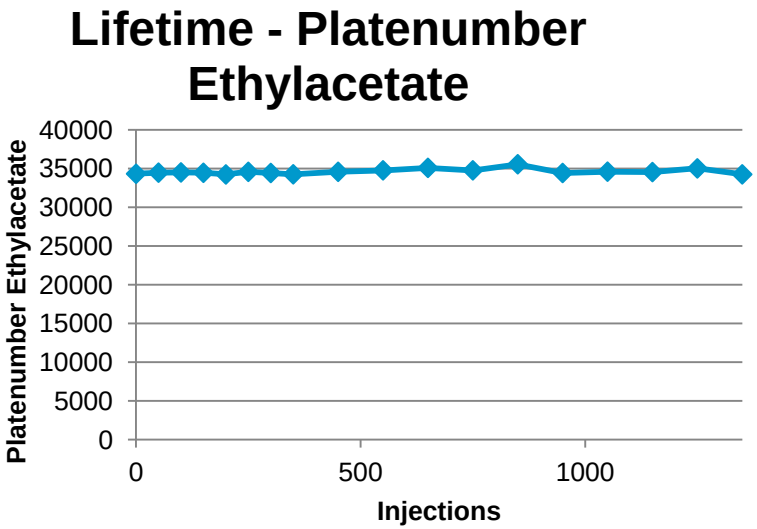
- Test done with PPQ, PPU,PBQ and HP-PLOT-Q
- Lifetime test performed by a high number of injections of methanol with 10% water
- 350 to 1350 injections are done
- After these injections, the columns are tested again
- No change in performance is observed

Lifetime test PoraPLOT U PT
1350 injections Methanol / 10% water



Lifetime test PoraPLOT U PT
1350 injections Methanol / 10% water





Conclusion: No change in performance of the column after multiple injections
(Similar results for all porous polymer PLOT PT columns)

Lifetime Test Al_2O_3 – PT 50m x 0.32mm

Temperature program: 40°C + 10°C/min --> 200°C (40 min.)
Carrier gas N2, 50 kPa. FID
In 3 weeks 450 runs, 300 hours at the maximum T of 200°C.

KCl	Before lifetime test		After lifetime test		
6509506	total	st dev	total	st dev	Difference
RI ethene	234.8	0.1	234.9	0.0	0.1
RI propene	347.7	0.1	347.7	0.0	0.0
RI ethyne	362.1	0.0	361.7	0.1	-0.4
RI propadiene	385.2	0.0	385.0	0.0	-0.2
RI t-2-butene	439.2	0.0	439.1	0.0	-0.1
RI 1-butene	443.5	0.0	443.5	0.0	0.0
RI isobutene	450.6	0.0	450.6	0.0	0.0
RI c-2-butene	458.7	0.0	458.7	0.0	0.0
RI propyne	485.9	0.0	485.4	0.1	-0.4
RI 1,3-butadiene	499.3	0.0	499.3	0.0	0.0
N 1,3-butadiene	153264	836	155972	1105	2707
k 1,3 butadiene	1.5	0.0	1.5	0.0	0.0
N/m 1,3-butadiene	2787	15	2836	20	49
u (cm/sec)	51.8	0.0	51.8	0.0	0.0
CFR	1.0	0.0	1.0	0.0	0.0

Na2SO4	Before lifetime test		After lifetime test		
6509513	total	st dev	total	st dev	Difference
RI ethene	242.9	0.1	243.3	0.1	0.4
RI propene	357.4	0.0	357.8	0.0	0.4
RI propadiene	396.6	0.2	396.7	0.1	0.1
RI ethyne	400.0	0.0	400.0	0.0	0.0
RI t-2-butene	445.8	0.0	446.1	0.0	0.3
RI 1-butene	453.4	0.0	453.7	0.0	0.4
RI isobutene	461.8	0.0	462.2	0.0	0.5
RI c-2-butene	468.9	0.0	469.2	0.0	0.4
RI 1,3-butadiene	514.9	0.0	515.2	0.0	0.4
RI propyne	522.6	0.1	523.1	0.1	0.5
N 1,3-butadiene	150900	1001	148253	209	-2647
k 1,3 butadiene	2.4	0.0	2.5	0.0	0.0
N/m 1,3-butadiene	2744	18	2696	4	-48
u (cm/sec)	54.2	0.0	54.2	0.0	0.0
CFR	1.1	0.0	1.1	0.0	0.0

Please note: No PDMS degradation at 200°C!

Lifetime Test Molsieve 5A
30m x 0.53 mm 50µm Molsieve 5A PT

Temperature program: 40°C + 10°C/min --> 200°C (23 min) + 10°C/min --> 300°C (5min)
Carrier gas N2, 25 kPa. TCD

In 3 weeks 531 runs, with 204 hours at 200°C and 44 hours at the maximum temperature of 300°C.

	Before lifetime test		After lifetime test		
9278538	Average	StDev	Average	StDev	Difference
Asym Carbonmonoxide	1.49	0.00	1.55	0.04	0.05
N methane	32177	5	31037	8	-1140
K methane	2.35	0.00	2.35	0.00	0.00
Res He/Ne	1.10	0.00	1.09	0.00	-0.02
Res Ar/O2	1.17	0.00	1.16	0.00	-0.01
CFR	1.04	0.00	1.05	0.00	0.01

	Before lifetime test		After lifetime test		
9257038	Average	StDev	Average	StDev	Difference
Asym Carbonmonoxide	1.52	0.02	1.57	0.11	0.05
N methane	36336	13	35603	472	-733
K methane	2.41	0.00	2.41	0.00	0.00
Res He/Ne	0.97	0.00	1.11	0.01	0.14
Res Ar/O2	1.23	0.01	1.22	0.01	-0.01
CFR	1.11	0.00	1.11	0.00	0.01

Lifetime Test Conclusions

After prolonged exposure at high operating temperature there is no significant change of the chromatographic performances of the Molsieve 5A and Al_2O_3 columns.

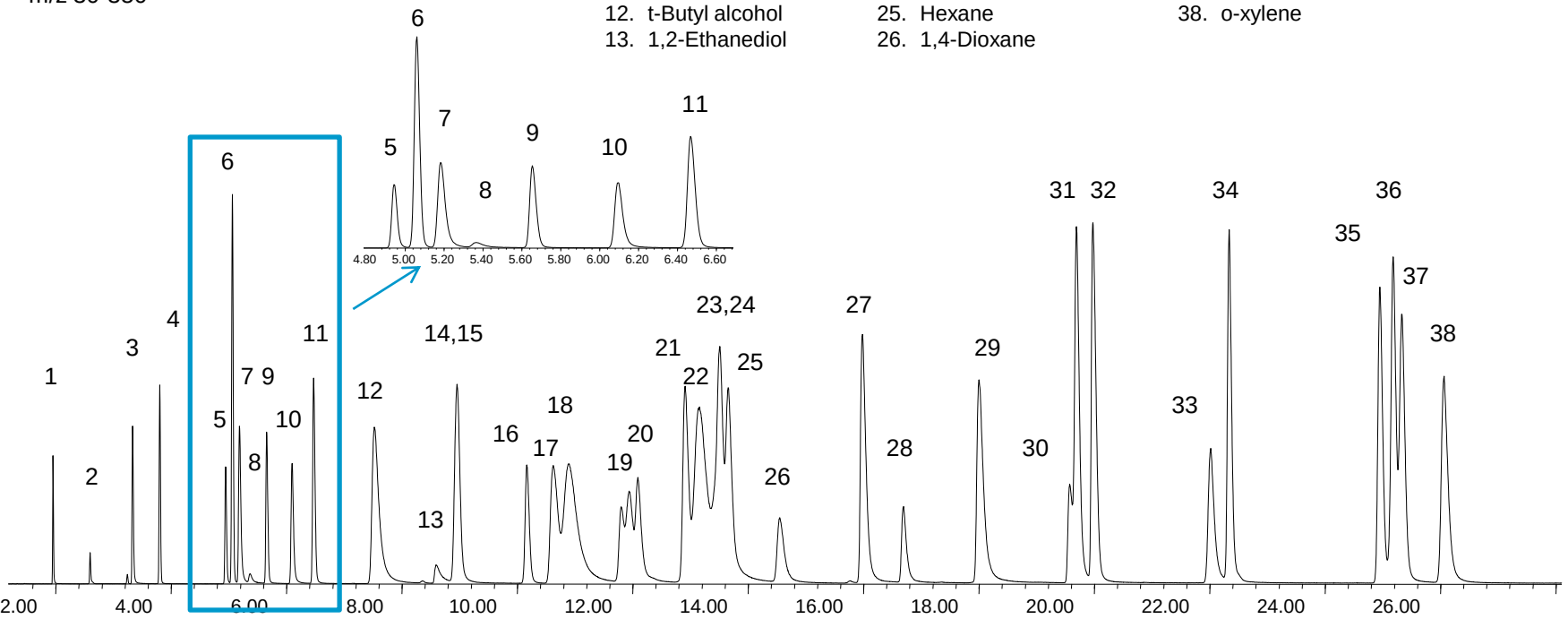
The bleed of the particle traps (front end) has no negative effect on the PLOT phases.

Ideal for Solvent Analysis by GCMS

Column: PoraBond Q PT, 25m×0.25mm,3um (30m total length)

Carrier : Helium, 43cm/s @90 ℃
Oven: : 90 °C- 140 °C at 10°C/min
140 °C for 6min
140 °C - 200 °C at 5°C/min
200 °C for 10 min
Injection: Split, 250 ℃, split ratio1:160
Detector: MSD, 280 ℃ Transfer line, full Scan at m/z 30-350

1. Methyl Alcohol
2. **Acetaldehyde**
3. Ethanol
4. Acetonitrile
5. Acetone
6. Methylene Chloride
7. Isopropyl Alcohol
8. **2-Propanamine**
9. Ethyl Formate
10. 1-Propanol
11. Ethyl ether
12. t-Butyl alcohol
13. 1,2-Ethanediol
14. Trichloromethane
15. 2-Butanone (MEK)
16. Ethyl Acetate
17. sec-Butyl alcohol
18. MTBE
19. 2-chlorobutane
20. 1-Butanol
21. Benzene
22. 1,1,1-Trichloroethane
23. 1-chlorobutane
24. Carbon Tetrachloride
25. Hexane
26. 1,4-Dioxane
27. Pyridine
28. Dimethyl Formamide (DMF)
29. Isoamyl Alcohol
30. Dimethyl Sulfoxide (DMSO)
31. Toluene
32. Heptane
33. Paraldehyde
34. Chlorobenzene
35. Ethylbenzene
36. m-Xylene
37. p-Xylene
38. o-xylene



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Excellent Peak Shape for Alcohols by GCMS

Column: PoraBond Q PT, 25m×0.25mm,3um (30 m total length)

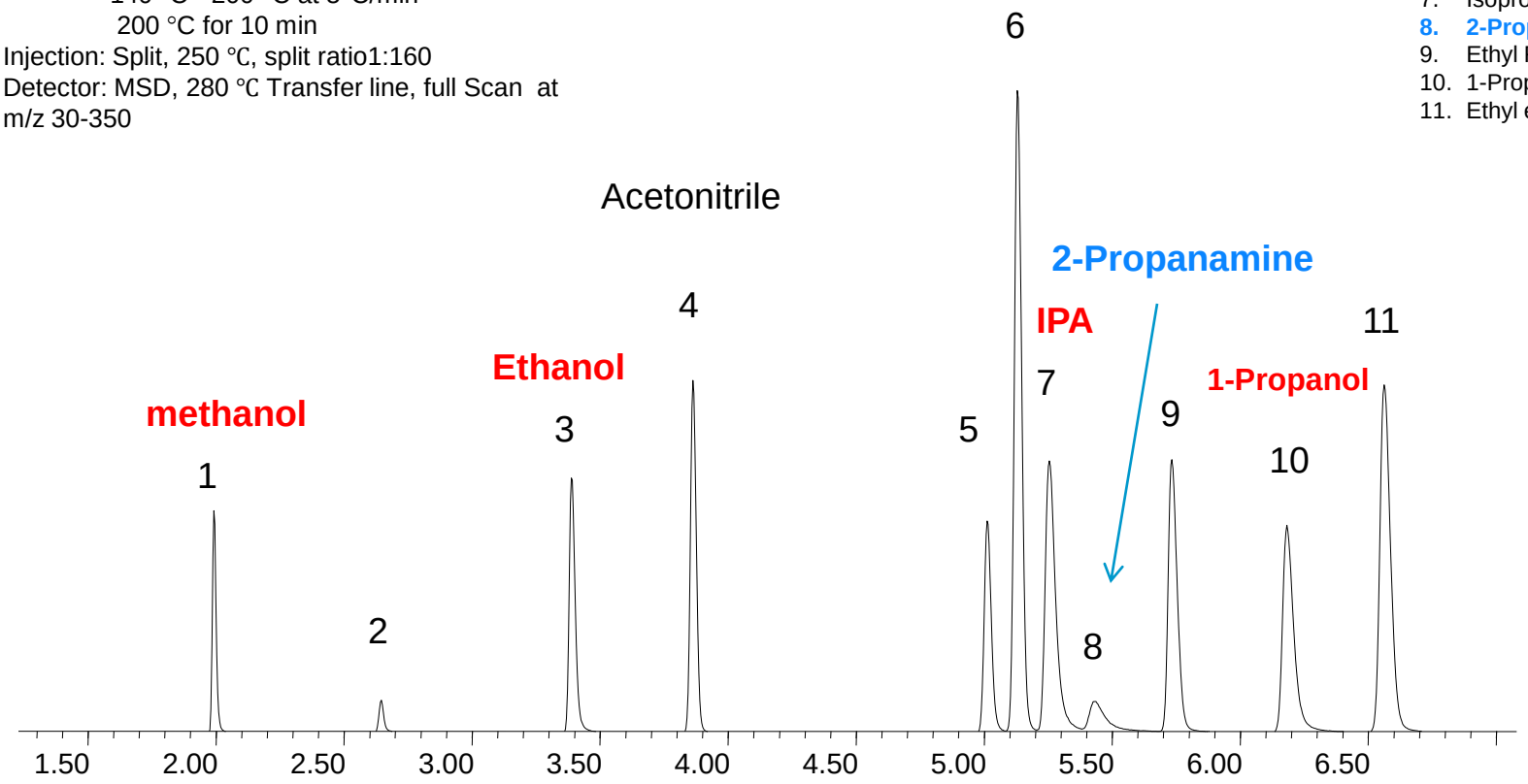
Carrier : Helium, 43cm/s @90 ℃

Oven: : 90 °C- 140 °C at 10°C/min
140 °C for 6min
140 °C - 200 °C at 5°C/min
200 °C for 10 min

Injection: Split, 250 ℃, split ratio1:160

Detector: MSD, 280 ℃ Transfer line, full Scan at m/z 30-350

1. Methyl Alcohol
2. **Acetaldehyde**
3. Ethanol
4. Acetonitrile
5. Acetone
6. Methylene Chloride
7. Isopropyl Alcohol
8. **2-Propanamine**
9. Ethyl Formate
10. 1-Propanol
11. Ethyl ether



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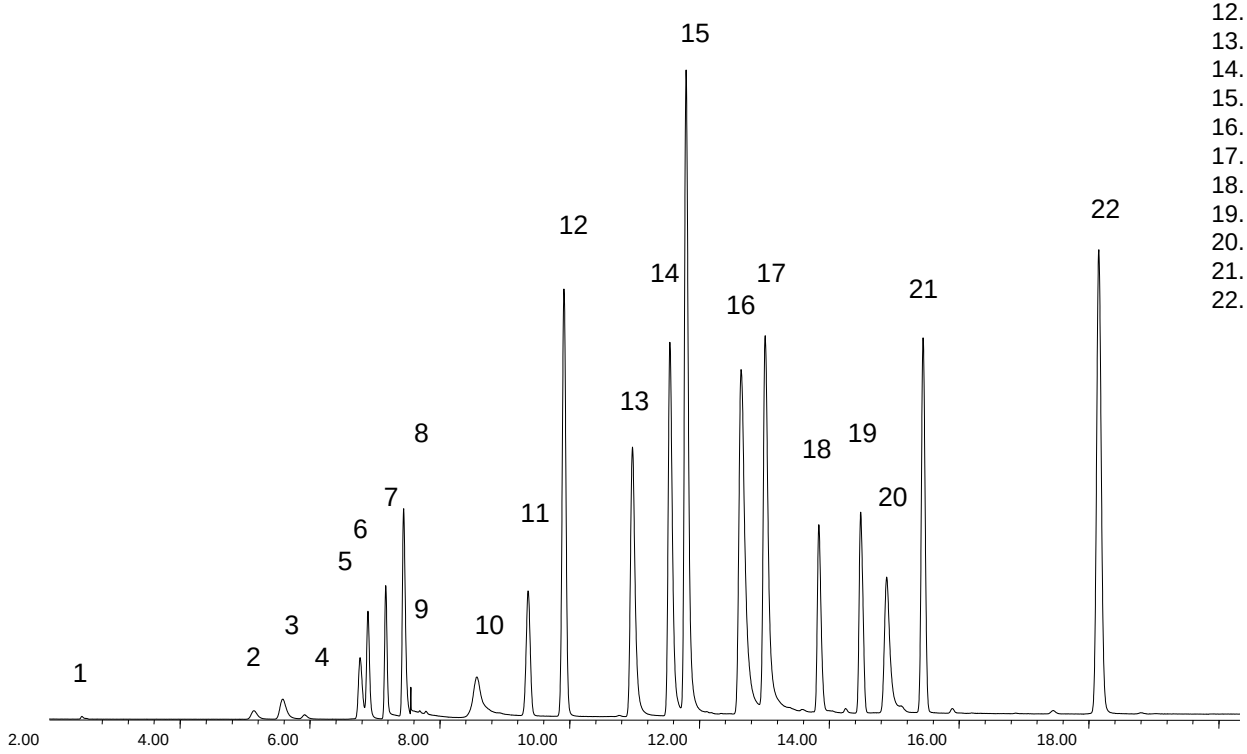
Halocarbons by GCMS

Column: PoraPLOT Q PT, 25m×0.32mm,10um (P/N CP7551PT)
(30m total length)

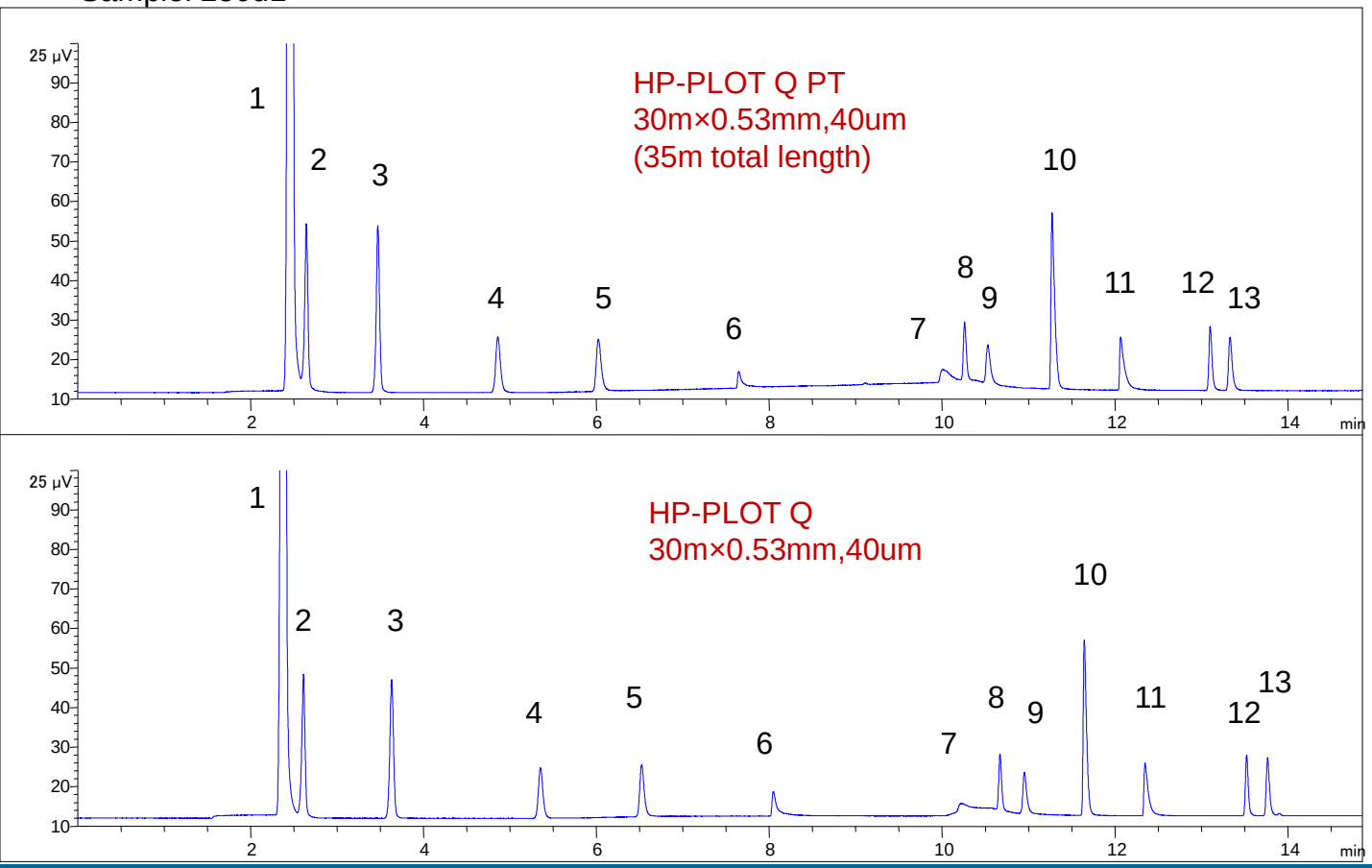
Carrier : Helium, 42cm/s @55 ℃
Oven: : 55 °C for 5min
55 °C - 200 °C at 12°C/min
200 °C for 10min
Injection: 250 ℃, splitless, 0.2min purge activation time
Detector: MSD, 280 ℃ Transfer line, full Scan at m/z 45-185
Sample: 1uL



1. Fluoroform (Freon-23)
2. 1,1,1-trifluoroethane (Freon-143a)
3. Pentafluoroethane (Freon-125)
4. Bromotrifluoromethane (Freon-13b1)
5. 1,1,1,2-Tetrafluoroethane (Freon-134a)
6. 1,1-difluoroethane (Freon-152a)
7. Difluorochloromethane (Freon-22)
8. 1,1,2,2-tetrafluoroethane (Freon-134)
9. 1-chloro-1,1-difluoroethane (Freon-142)
10. Bromochlorodifluoromethane (Freon-12b1)
11. Ethyl Chloride (Freon-160)
12. Fluorodichloromethane (Freon-21)
13. Trichloromonofluoromethane (Freon-11)
14. 1,1-Dichloro-1-fluoroethane (Freon-141)
15. 2,2-dichloro-1,1,1-trifluoroethane (Freon-123)
16. 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)
17. 1,2-dibromo-1,1,2,2-tetrafluoroethane (Freon-114b2)
18. Trichloromethane (Freon-20)
19. 1,2-dichloroethane
20. 1,1,1-trichloro-ethane
21. Trichloroethylene
22. 1,1,2-trichloroethane

Coal to Chemical Process Gas Analysis

Carrier : H2, 36cm/s @32 ℃
Oven: : 32 °C for 5 min
32 °C - 180 °C at 15°C/min
Injection: 170 ℃ , split ratio 5:1
Detector: TCD, 250 ℃
Sample: 250uL



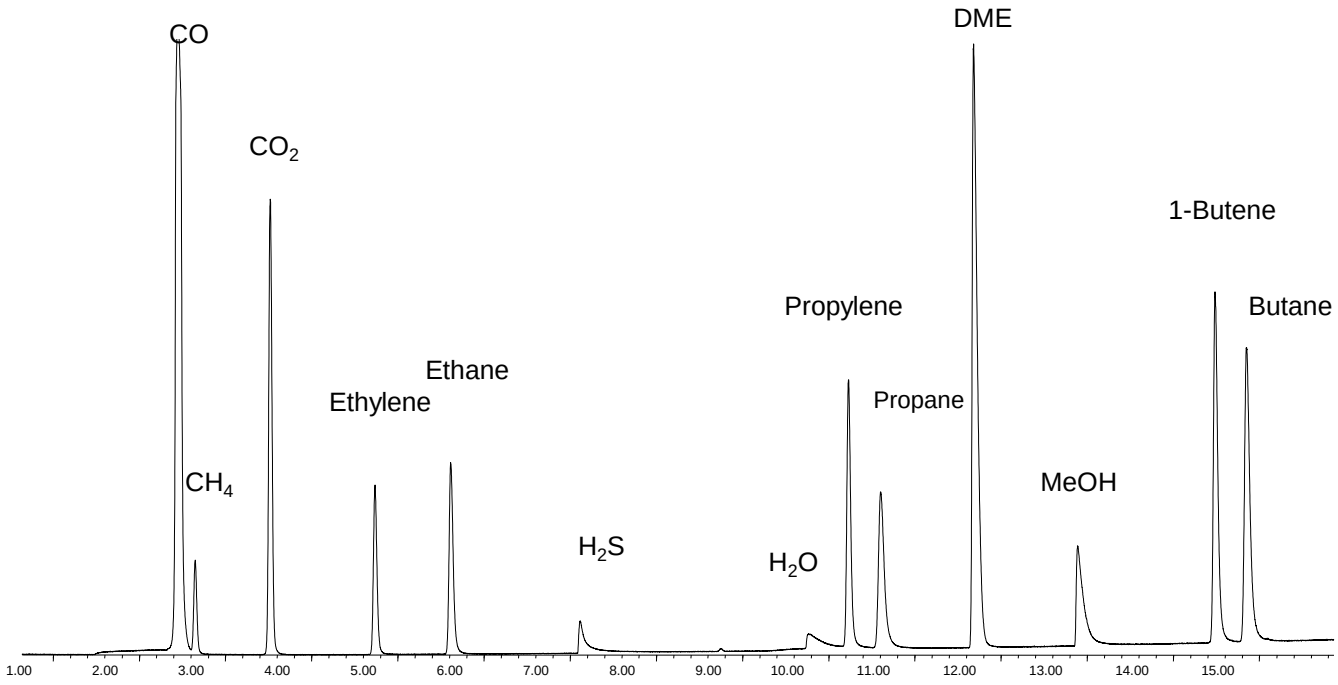
1. Carbon monoxide
2. Methane
3. Carbon dioxide
4. Ethylene
5. Ethane
6. Hydrogen sulfide
7. Water
8. Propylene
9. Propane
10. Dimethyl ether
11. Methanol
12. 1-Butene
13. Butane

Coal to Chemical Process Gas Analysis

Identify compounds by MSD

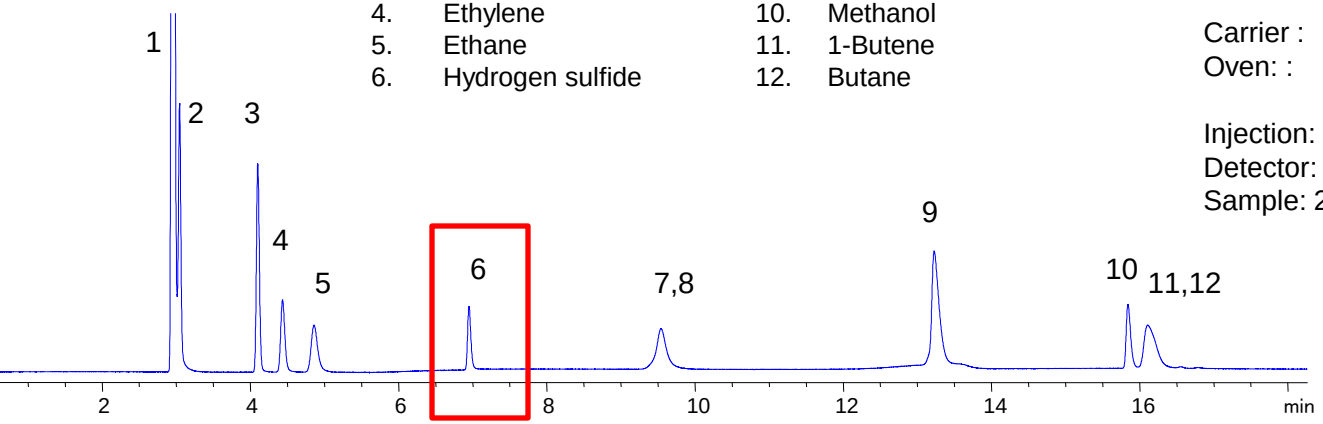
Column: HP-PLOT Q PT, 30m×0.32mm,20um (P/N 19091P-Q04PT)
(35m total length)

Carrier : Helium, 1mL/min
Oven: : 32 °C for 3 min
32 °C - 180 °C at 15°C/min
Injection: 170 ℃ , split 5:1
Detector: MSD, 280 ℃ Transfer line, full Scan at m/z 10-100
Sample: 250uL

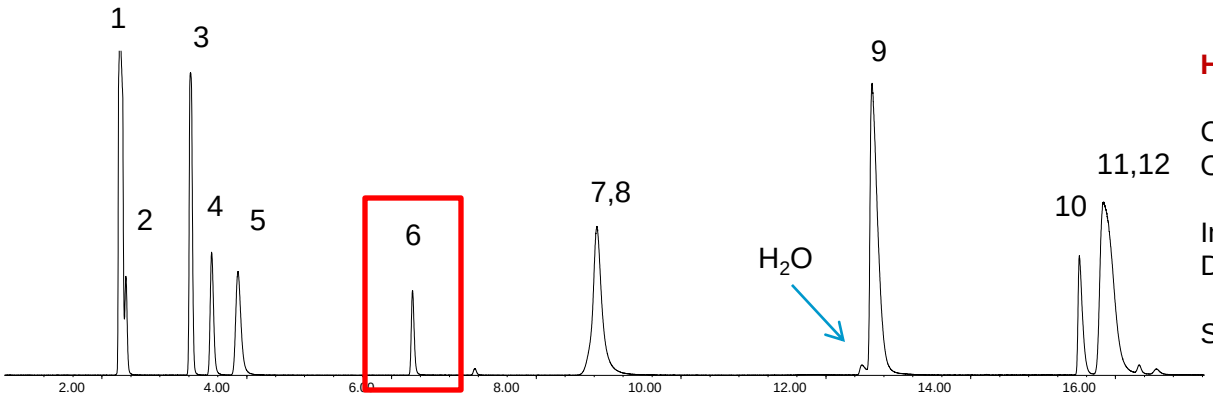


Excellent Peak Shape of Hydrogen Sulfide on HP-PLOT U PT Column

1. Carbon monoxide
2. Methane
3. Carbon dioxide
4. Ethylene
5. Ethane
6. Hydrogen sulfide
7. Propylene
8. Propane
9. Dimethyl ether
10. Methanol
11. 1-Butene
12. Butane

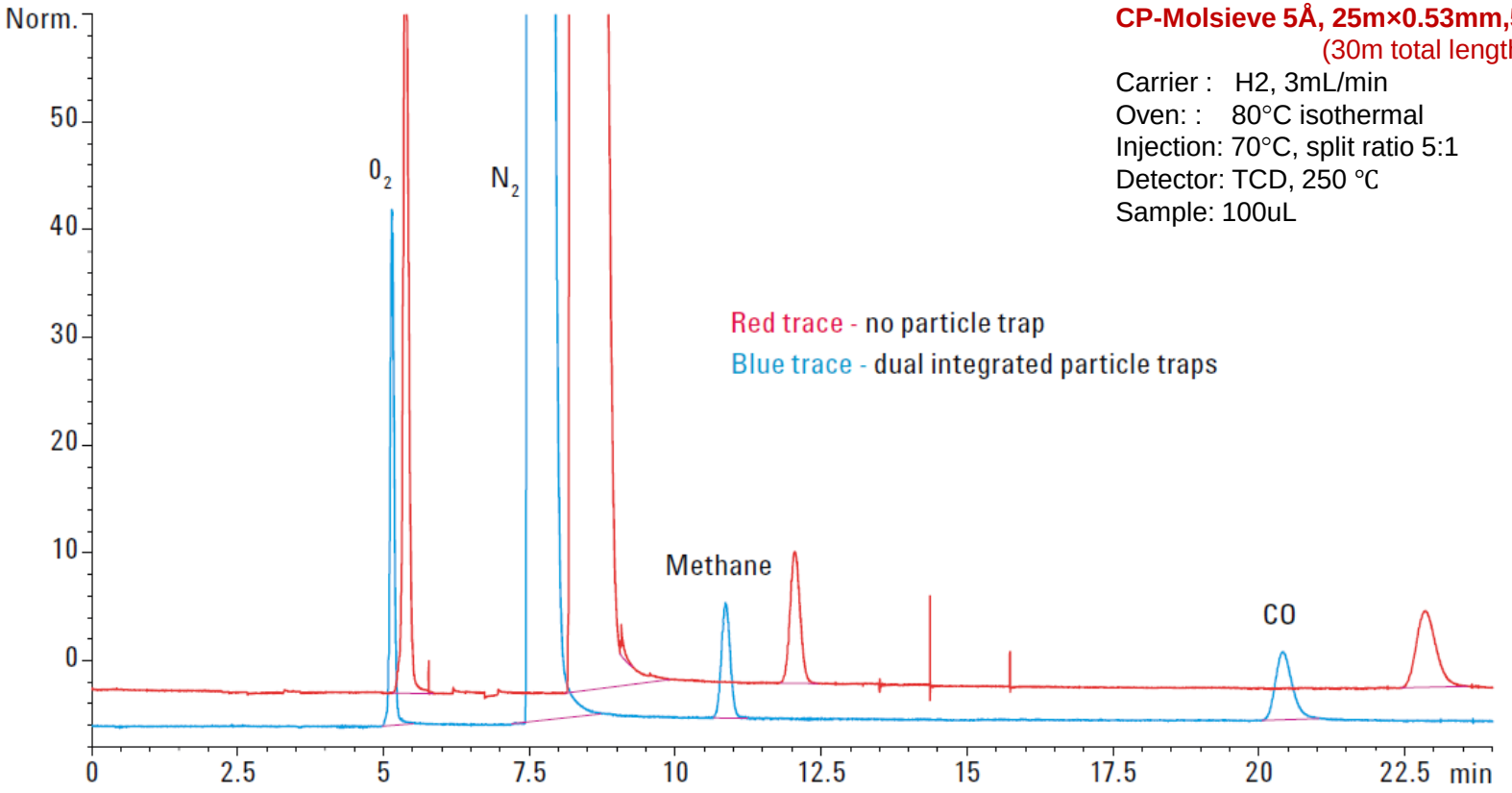


HP-PLOT U PT, 30m×0.53mm,20um
(35m total length)
Carrier : H2, 40cm/s @32 ℃
Oven: : 32 °C for 5 min,32 °C - 70 °C at 30°C/min
70 °C for 5 min,70 °C - 140 °C at 10°C/min
Injection: 170 ℃ , split ratio 5:1
Detector: **TCD**, 250 ℃
Sample: 250uL



HP-PLOT U PT, 30m×0.32mm,10um
(35m total length)
Carrier : H2, 35cm/s @32 ℃
Oven: : 32 °C for 5 min,32 °C - 70 °C at 30°C/min
70 °C for 5 min,70 °C - 140 °C at 10°C/min
Injection: 170 ℃ , split ratio 5:1
Detector: **MSD**, 280 ℃ Transfer line,
full Scan at m/z 10-100
Sample: 250uL

No spikes at Fixed Gases Analysis on CP-Molsieve 5Å PLOT PT column



CP-Molsieve 5Å, 25m×0.53mm,50um
(30m total length)
Carrier : H2, 3mL/min
Oven: : 80°C isothermal
Injection: 70°C, split ratio 5:1
Detector: TCD, 250 ℃
Sample: 100uL

CP-Molsieve 5Å showing spikes when particle traps are removed (Red trace).
No spikes with manufacturer integrated particle trap (Blue Trace).

Agilent J&W PLOT PT Columns

Available PLOT PT columns:

- Porous polymers:
  - PoraPLOT Q
  - PoraBOND Q
  - PoraBOND Q HT
  - HP-PLOT Q
  - GS-Q
  - PoraPLOT U
  - HP-PLOT U
- *NEW!* Aluminum oxide
  - HP-PLOT Al2O3 S
  - HP-PLOT Al2O3 M
  - HP-PLOT Al2O3 KCl
  - GS-Alumina
  - GS-Alumina/KCl
  - CP-Al2O3 KCl
  - CP-Al2O3 Na2SO4
- *NEW!* Molsieve
  - CP-Molsieve 5A

Custom PLOT PT columns are available for these phases

Phase type	Part number	Description	Dimensions
PLOT Q	CP7348PT	PoraBOND Q PT	25m x 0.25mm x 3µm
	CP7351PT	PoraBOND Q PT	25m x 0.32mm x 5µm
	CP7352PT	PoraBOND Q PT	50m x 0.32mm x 5µm
	CP7353PT	PoraBOND Q PT	10m x 0.53mm x 10µm
	CP7354PT	PoraBOND Q PT	25m x 0.53mm x 10µm
	CP7550PT	PoraPLOT Q PT	10m x 0.32mm x 10µm
	CP7551PT	PoraPLOT Q PT	25m x 0.32mm x 10µm
	CP7554PT	PoraPLOT Q PT	25m x 0.53mm x 20µm
	CP7557PT	PoraPLOT Q-HT PT	25m x 0.32mm x 10µm
	115-3432PT	GS-Q PT	30m x 0.53mm
	19091P-QO3PT	HP-PLOT Q PT	15m x 0.32mm x 20µm
	19091P-QO4PT	HP-PLOT Q PT	30m x 0.32mm x 20µm
	19095P-QO3PT	HP-PLOT Q PT	15m x 0.53mm x 40µm
	19095P-QO4PT	HP-PLOT Q PT	30m x 0.53mm x 40µm
PLOT U	CP7584PT	PoraPLOT U PT	25m x 0.53mm x 20µm
	19095P-UO4PT	HP-PLOT U PT	30m x 0.53mm x 20µm
Al2O3 KCl deactivated	CP7515PT	CP-Al2O3/KCl PT	50m x 0.32mm x 5µm
	CP7517PT	CP-Al2O3/KCl PT	25m x 0.53mm x 10µm
	CP7518PT	CP-Al2O3/KCl PT	50m x 0.53mm x 10µm
	19091P-K15PT	HP-PLOT Al2O3 KCl PT	50m x 0.32mm x 8µm
	19095P-K23PT	HP-PLOT Al2O3 KCl PT	30m x 0.53mm x 15µm
	19095P-K25PT	HP-PLOT Al2O3 KCl PT	50m x 0.53mm x 15µm
	115-3352PT	GS-Alumina/KCl PT	50m x 0.53mm
Al2O3 Na2SO4 deactivated	CP7565PT	CP-Al2O3/Na2SO4 PT	50m x 0.32mm x 5µm
	CP7568PT	CP-Al2O3/Na2SO4 PT	50m x 0.53mm x 10µm
	19091P-S12PT	HP-PLOT Al2O3 S PT	25m x 0.32mm x 8µm
	19091P-S15PT	HP-PLOT Al2O3 S PT	50m x 0.32mm x 8µm
	19095P-S23PT	HP-PLOT Al2O3 S PT	30m x 0.53mm x 15µm
	19095P-S25PT	HP-PLOT Al2O3 S PT	50m x 0.53mm x 15µm
Al2O3 with proprietary deactivation	115-3532PT	GS-Alumina PT	30m x 0.53mm
	115-3552PT	GS-Alumina PT	50m x 0.53mm
	19095P-M25PT	HP-PLOT Al2O3 M PT	50m x 0.53mm x 15µm
Molsieve	CP7534PT	CP-Molsieve 5A PT	30m x 0.32mm x 10µm
	CP7536PT	CP-Molsieve 5A PT	25m x 0.32mm x 30µm
	CP7538PT	CP-Molsieve 5A PT	25m x 0.53mm x 50µm
	CP7539PT	CP-Molsieve 5A PT	50m x 0.53mm x 50µm

Conclusions

Agilent's integrated particle trap technology for PLOTs–

- Similar selectivity to non-PT PLOT columns

- Virtually eliminates problems due to particle shedding

- Possible to use MS detection, valves and CFT worry-free

Now Let's Switch Gears…

New DB-Sulfur SCD for GC-SCD Analysis of Sulfur Compounds

Why so much focus on Sulfur?

Sulfur Compounds

-can be corrosive to equipment, pipe lines, reactors

-can inhibit or destroy catalysts employed in downstream processing

-can impart undesirable odors to products

-in fuel pollutes the air (Environmental regulations require lower levels)

Challenges for Sulfur Analysis

Low levels often require maximum sensitivity

Matrix interference from the hydrocarbons present

Highly reactive and polar molecules

Detectors for Sulfur Analysis

Why not use an FID or MSD?

Sulfur Detection

Detector	GC-FPD	GC-PFPD	GC-SCD
Supplier	Agilent	OI	Agilent
MDL Sulfur	3.6 pg/sec	1 pg/sec	<0.5 pg/sec
Selectivity	10^6	10^6	10^7
Dynamic Range	10^3	10^3	10^5
Quenching	yes	yes	no
Equimolar response	No	No	yes
Packed Col Compatible	yes	No, < 1ml/min	yes
Other Elements	P, Sn	P	N
Cost	$	$$	$$$

SCD for Sulfur Analysis

Basis for several ASTM methods

Very sensitive but….

- Slow to stabilize
- “tricky” to operate
- Prone to “coking” in the ceramic reaction tubes with resulting costly maintenance and slow recovery

Method	Description
ASTM D6228	Volatile sulfur in C1, C2, C3 and C4 monomers and LPG
ASTM D6628	Volatile Sulfur in NGA, fuel gas
ASTM D5504	Sulfur in gas fuels by SCD
ASTM D5623	Sulfur in light petroleum liquids by SCD

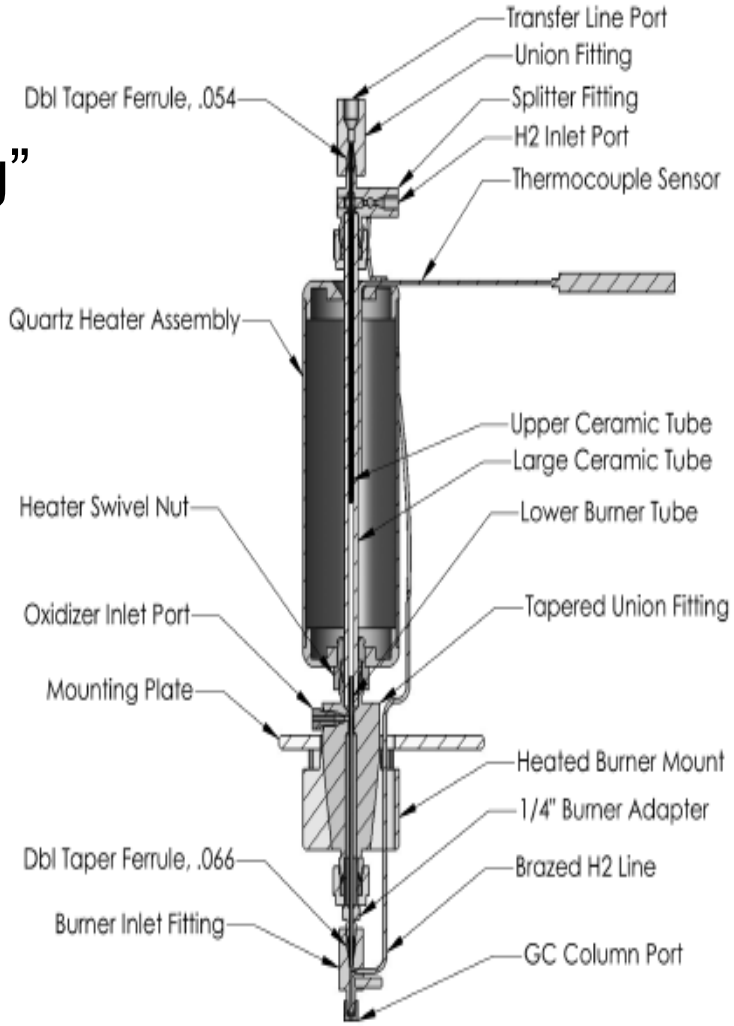
About SCD Maintenance….

Ceramic reaction tube fouling/ “coking”

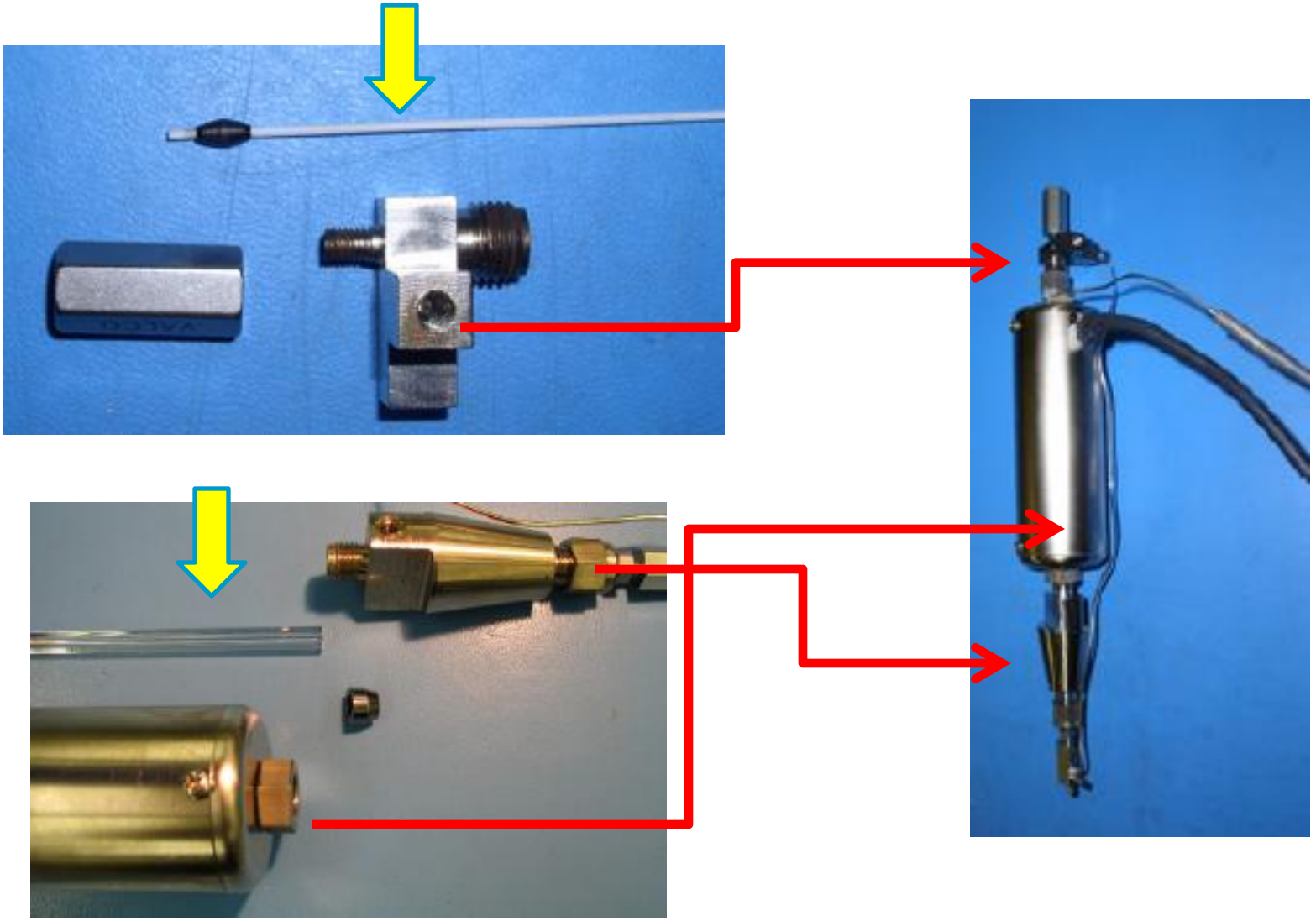
Typical costs in the US:

- Price per incident preventative maintenance service call for GC-SCD: $1755 USD
- Cost of SCD ceramics: G6602-67000 $356 USD
- Dual plasma burner kit: G6602-60037 $459 USD.

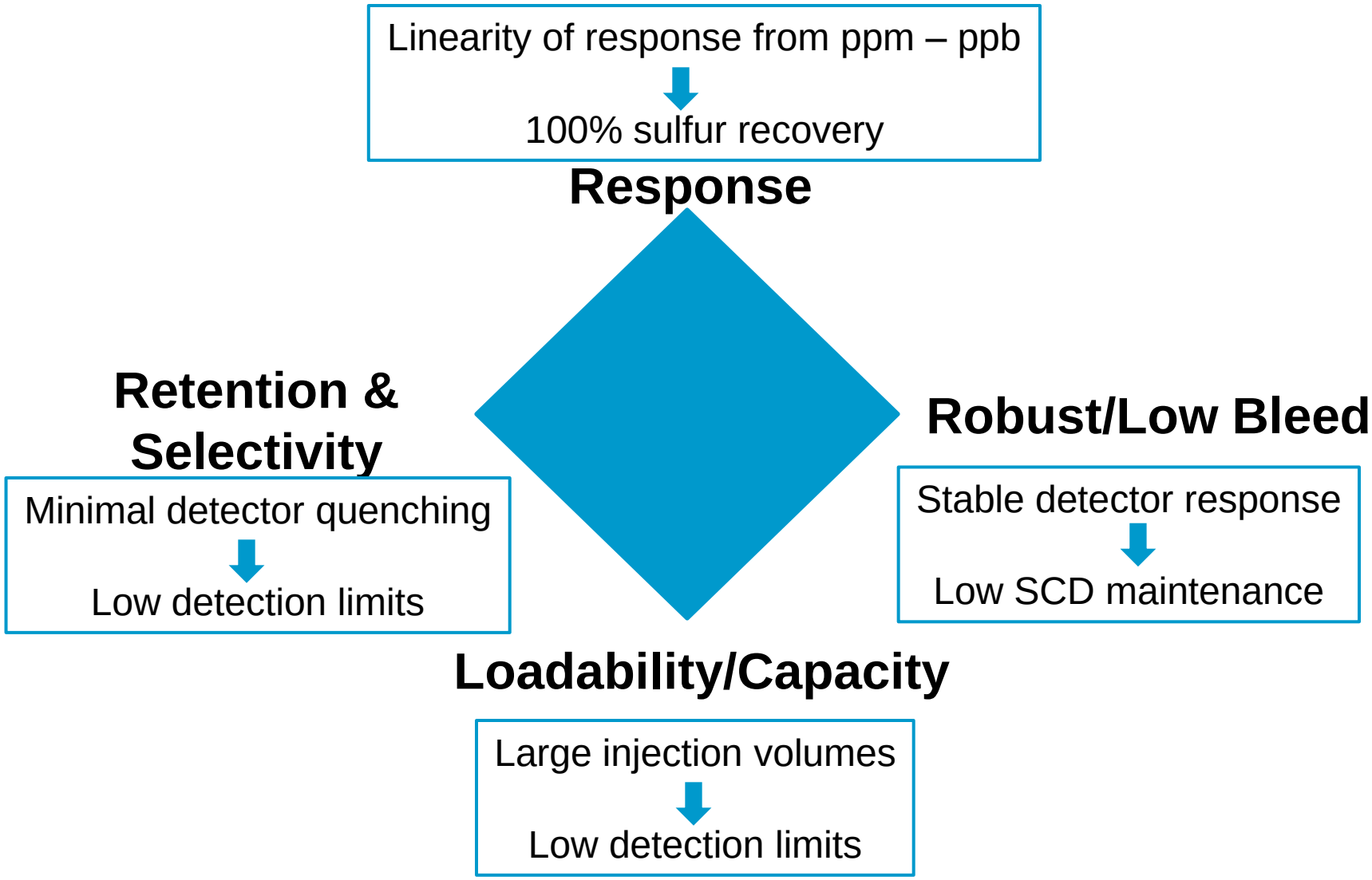
Plus the self repair time of 4 hours and several days to stabilize.



SCD Ceramic Combustion Tubes & Burner



What is required of GC column for Sulfur Analysis?



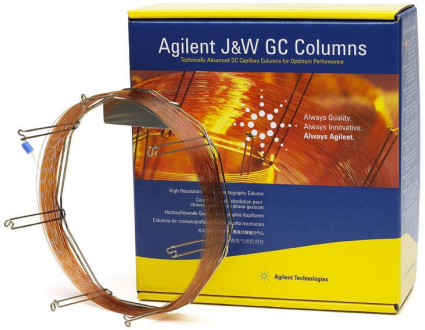
Introducing DB-Sulfur SCD

New optimized low polarity column with low bleed and exceptional inertness to sulfur even at trace levels

Developed with Dow Chemical and other leading companies

Excellent for a broad range of sulfur compounds from light sulfur gasses to sulfur containing hydrocarbons out to C24

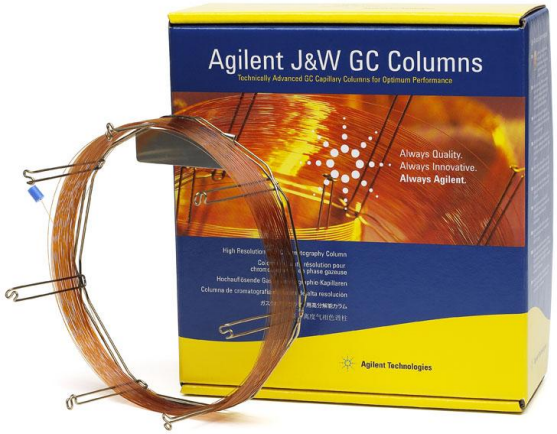
Optimized for the lowest possible contribution to SCD reaction tube fouling.



DB-Sulfur SCD

Easy to change from existing columns but with:

- Greatly improved SCD performance
- Increased stability
- Less frequent burner tube maintenance



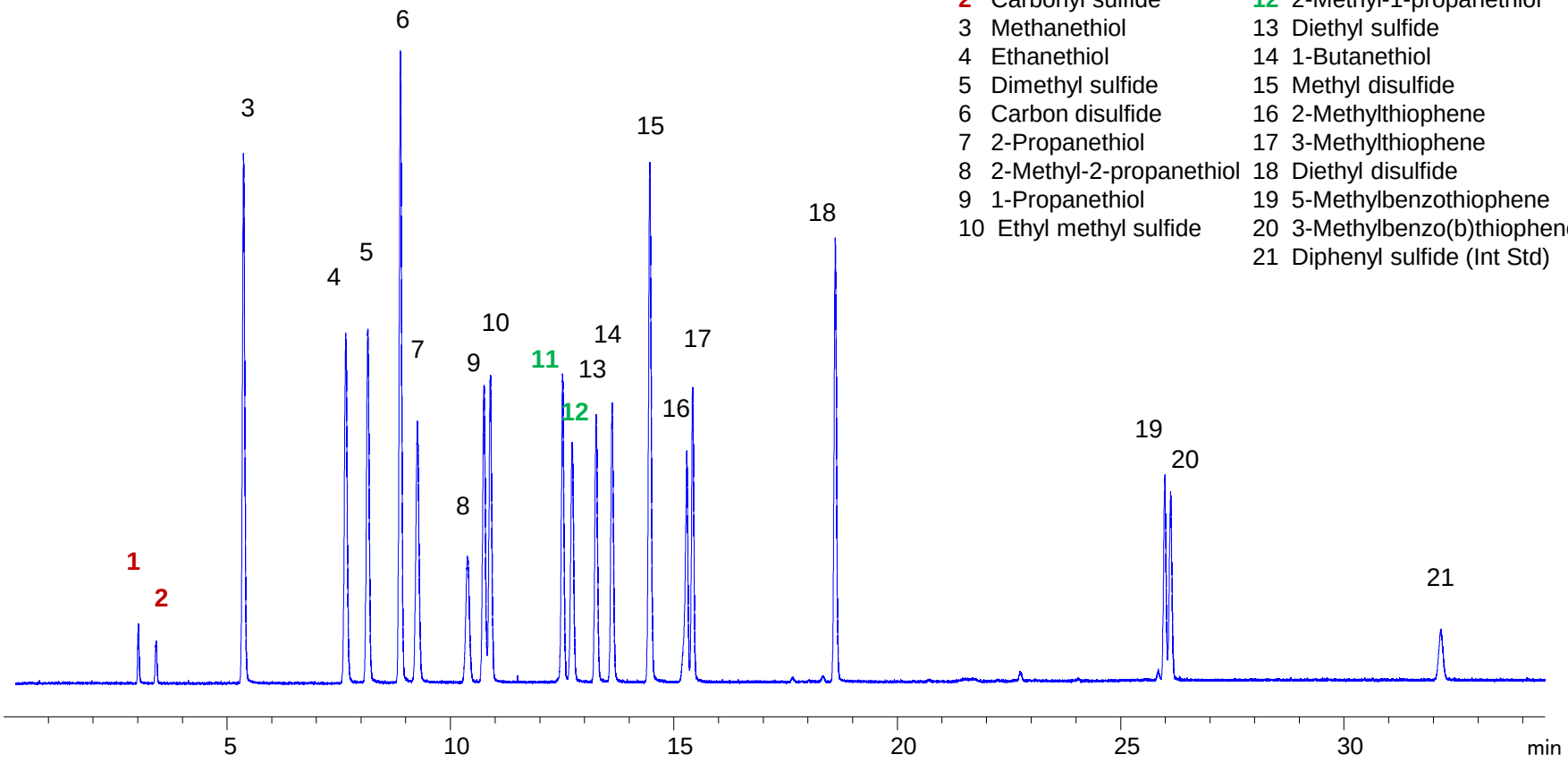
Part Number	Description	Temperature limits
G3903-63001	DB-Sulfur SCD 60m, 0.32mm, 4.2um	250°/270°C
G3903-63002	DB-Sulfur SCD 40m, 0.32mm, 0.75um	270°/290°C
G3903-63003	DB-Sulfur SCD 70m, 0.53mm, 4.3um	250°/270°C
G3903-63004	DB-Sulfur SCD 40m, 0.32mm, 3um	250°/270°C

DB-Sulfur SCD:

sulfur standards in Toluene

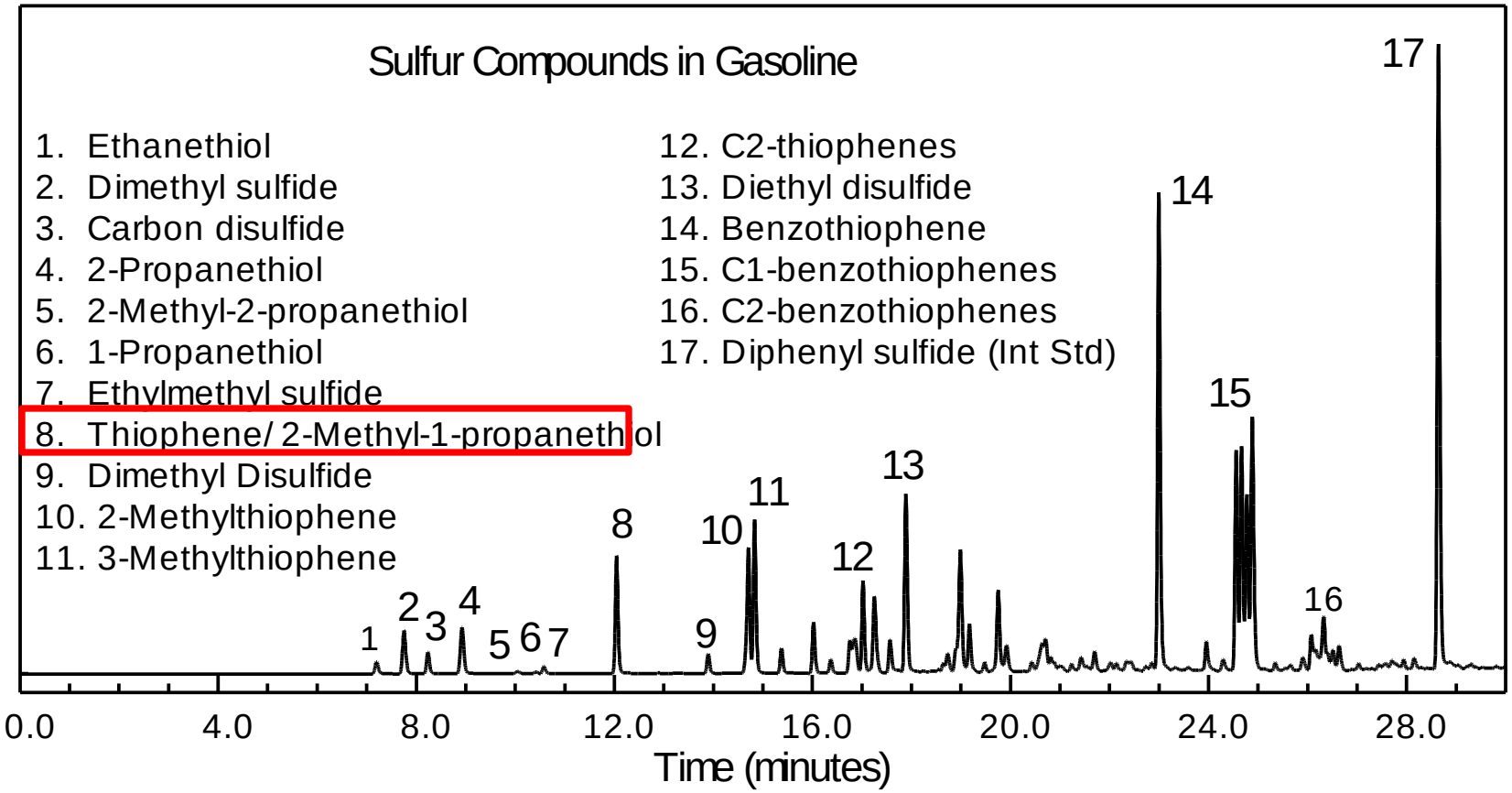
Good resolution of H2S and COS at room temperature

Thiophene and 2-Methyl-1-propanethiol can be baseline separated



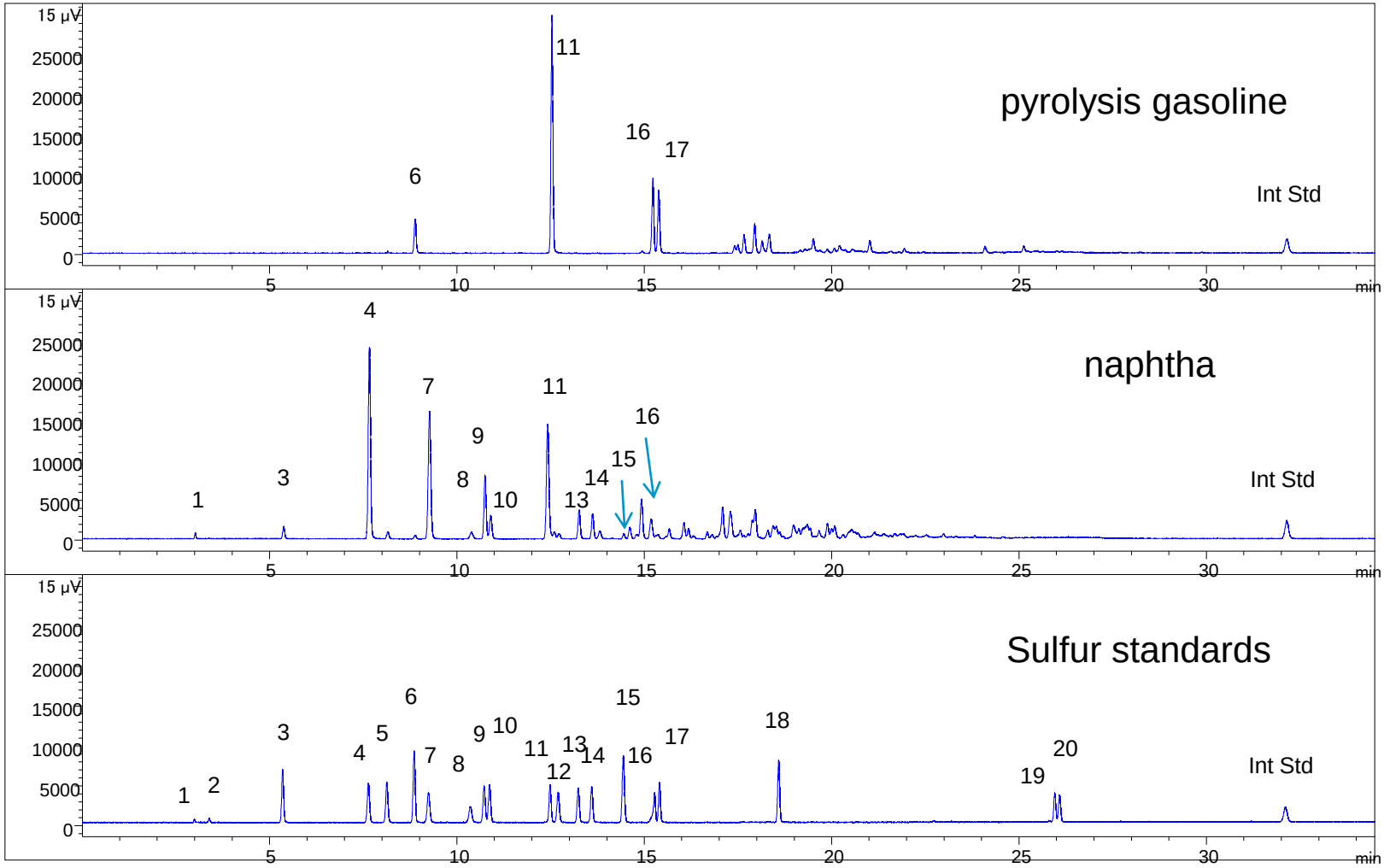
Column: Agilent J&W DB-Sulfur SCD, 60 m x 0.32 mm, 4.2 µm (p/n G3903-63001)

Typical chromatogram of Sulfur compounds in Light Petroleum Liquids by ASTM D5623



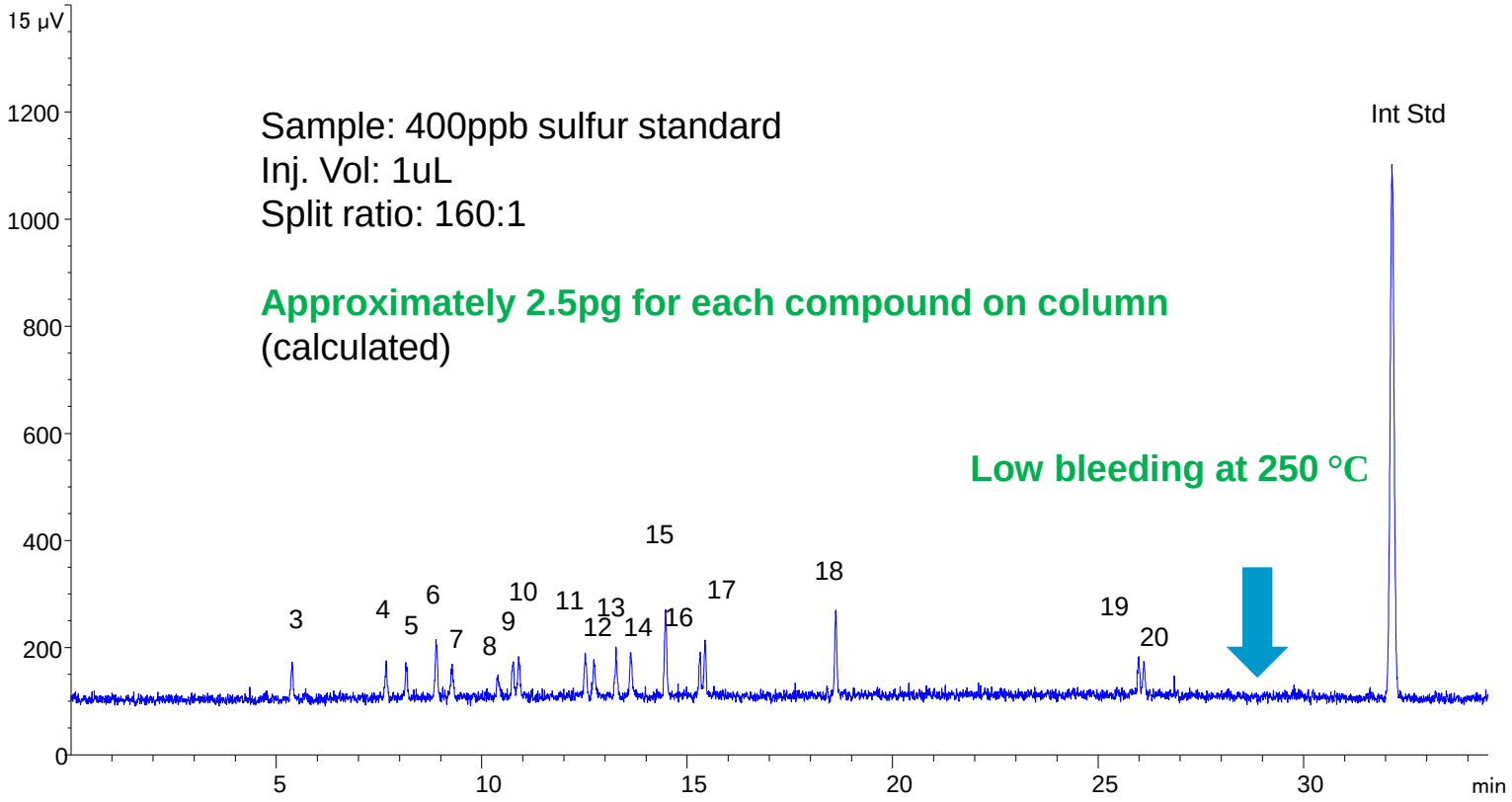
… the industry standard

DB-Sulfur SCD: Real Samples



DB-Sulfur SCD: Sulfur Sensitivity

Peak No.	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
S/N	3.0	3.1	2.7	4.4	2.8	2.1	2.9	3.3	3.5	3.0	3.8	3.5	6.3	3.2	4.4	6.3	3.1	2.9



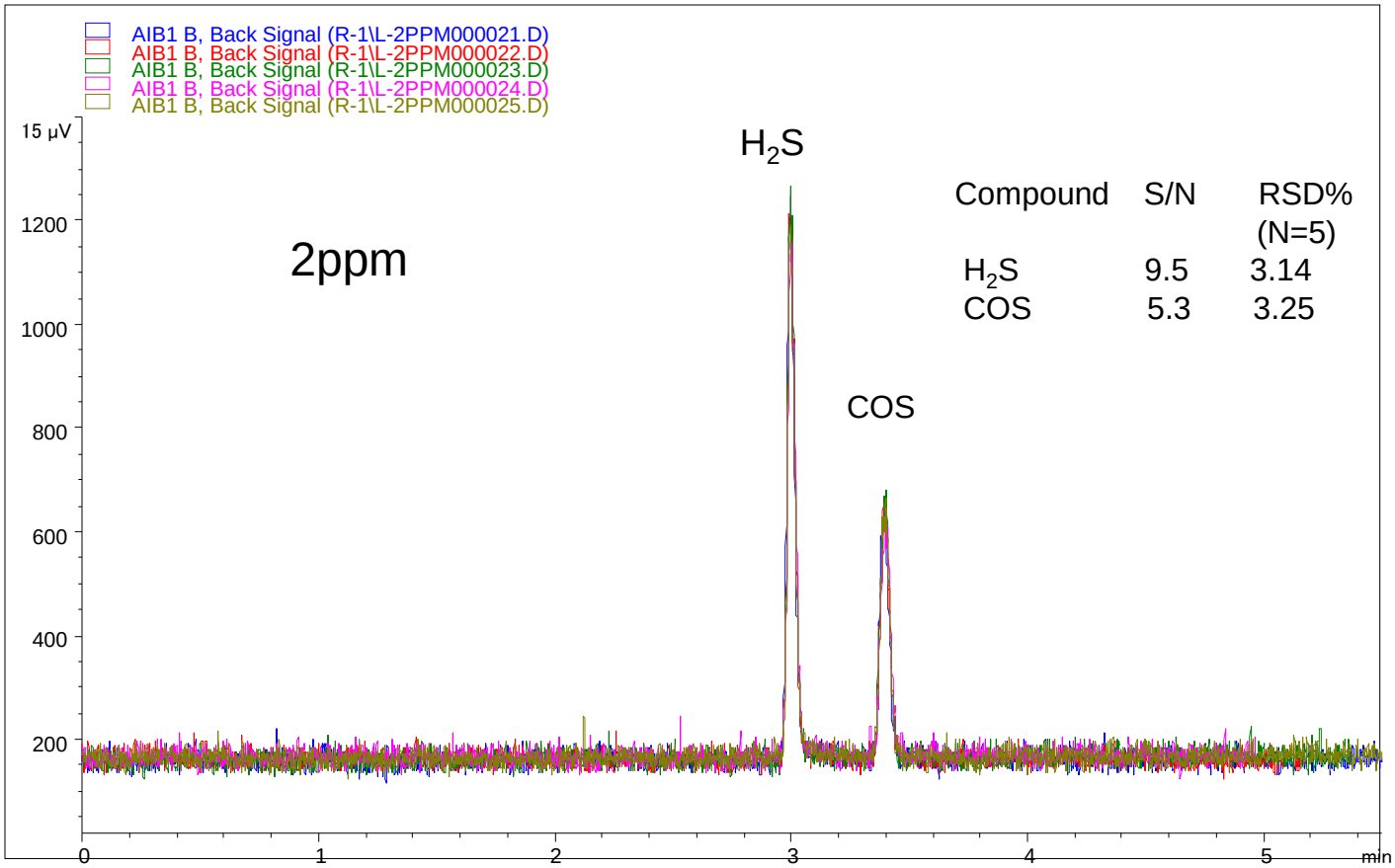
Agilent Technologies

Repeatability

N=6

No	Compound	10 ppm RSD%	1ppm RSD%	0.1ppm RSD%
1	Methyl mercaptan	2.94	4.46	5.12
2	Ethyl mercaptan	2.53	3.00	4.38
3	Methyl sulfide	2.53	2.79	4.79
4	Carbon disulfide	2.13	3.29	5.43
5	2-Propanethiol	2.49	3.98	4.85
6	2-Methyl-2-propanethiol	2.89	4.47	4.41
7	1-Propanethiol	2.81	3.88	4.91
8	Ethyl methyl sulfide	2.34	4.17	5.24
9	Thiophene	2.24	3.06	3.49
10	2-Methyl-1-propanethiol	1.87	2.31	5.86
11	Diethyl sulfide	2.00	2.97	4.80
12	1-Butanethiol	2.46	3.36	6.47
13	Methyl disulfide	3.62	4.15	4.23
14	2-Methylthiophene	3.59	4.62	5.95
15	3-Methylthiophene	2.85	3.90	4.90
16	Diethyl disulfide	2.74	3.16	6.34
17	3-Methylbenzothiophene	2.48	4.87	5.29
18	5-Methylbenzo(b)thiophene	2.42	4.25	7.37

DB-Sulfur SCD: Sulfur Sensitivity



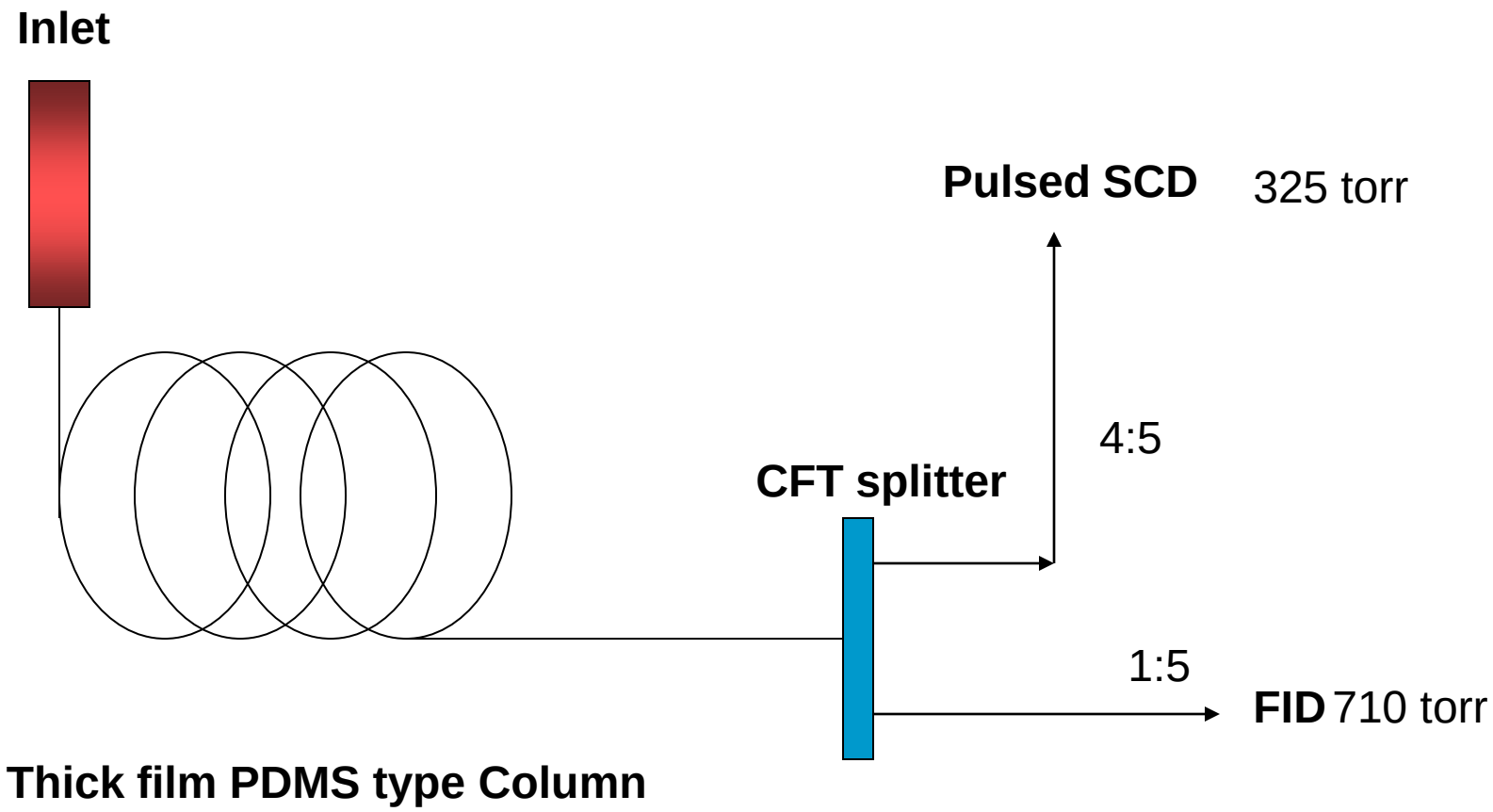
Agilent Technologies

Linearity

Compound	Concentration Range	Linearity (R^2)
Hydrogen sulfide	2ppm-25ppm	0.9976
Carbonyl sulfide	2ppm-25ppm	0.9990
Methanethiol	0.1ppm-10ppm	0.9987
Ethanethiol	0.1ppm-50ppm	0.9998
Dimethyl sulfide	0.1ppm-10ppm	0.9991
Carbon disulfide	0.1ppm-10ppm	0.9990
2-Propanethiol	0.1ppm-50ppm	0.9999
2-Methyl-2-propanethio	0.1ppm-10ppm	0.9989
1-Propanethiol	0.1ppm-10ppm	0.9990
Ethyl methyl sulfide	0.1ppm-50ppm	0.9998

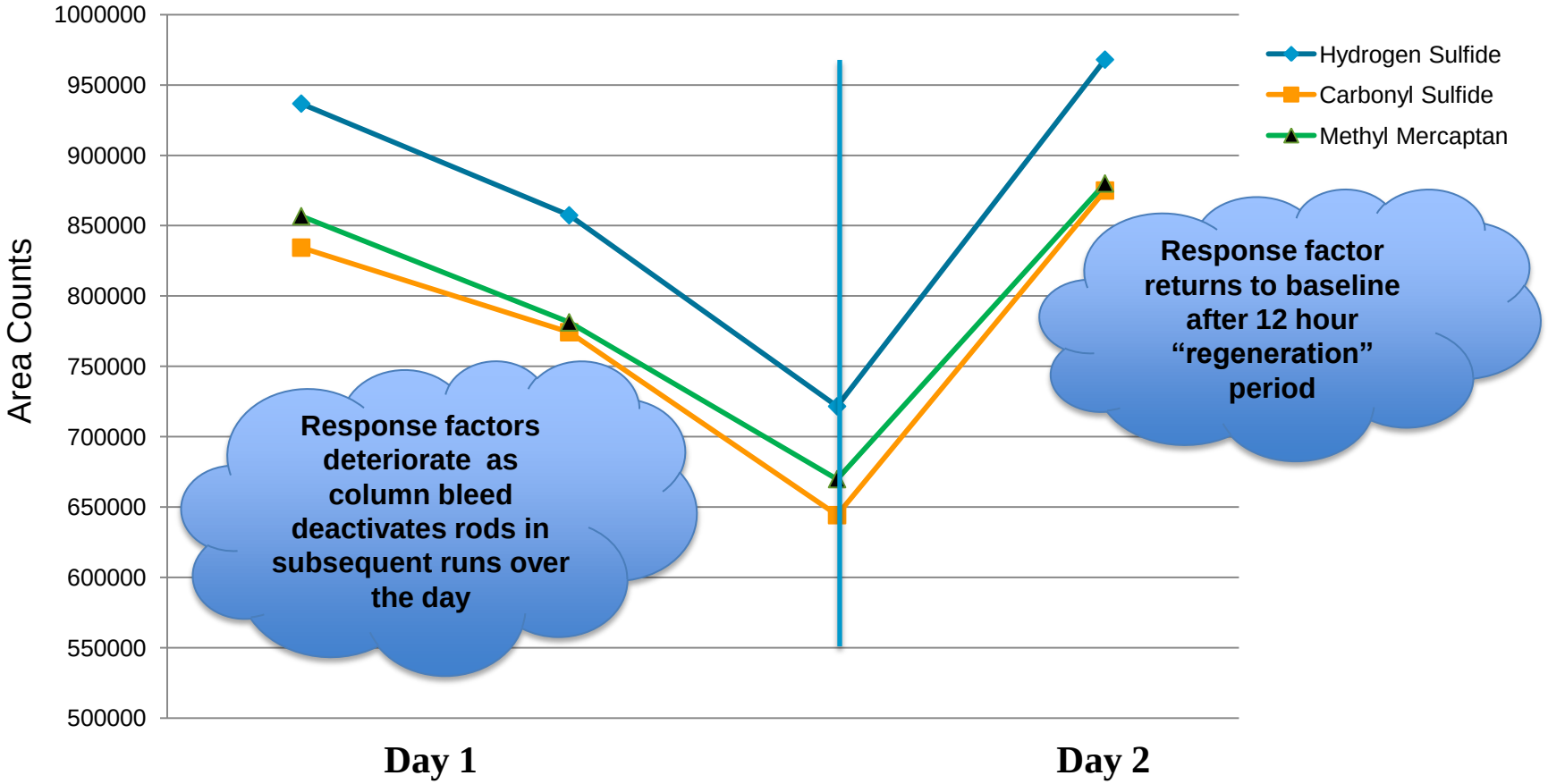
Compound	Concentration Range	Linearity (R^2)
Thiophene	0.1ppm-50ppm	0.9997
2-Methyl-1-propanethiol	0.1ppm-10ppm	0.9991
Diethyl sulfide	0.1ppm-10ppm	0.9992
1-Butanethiol	0.1ppm-10ppm	0.9990
Methyl disulfide	0.1ppm-10ppm	0.9987
2-Methylthiophene	0.1ppm-50ppm	0.9991
3-Methylthiophene	0.1ppm-50ppm	0.9996
Diethyl disulfide	0.1ppm-10ppm	0.9990
5-Methylbenzothiophene	0.1ppm-10ppm	0.9984
3-Methylbenzothiophene	0.1ppm-50ppm	0.9988

Configuration to test SCD Quenching Issue



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

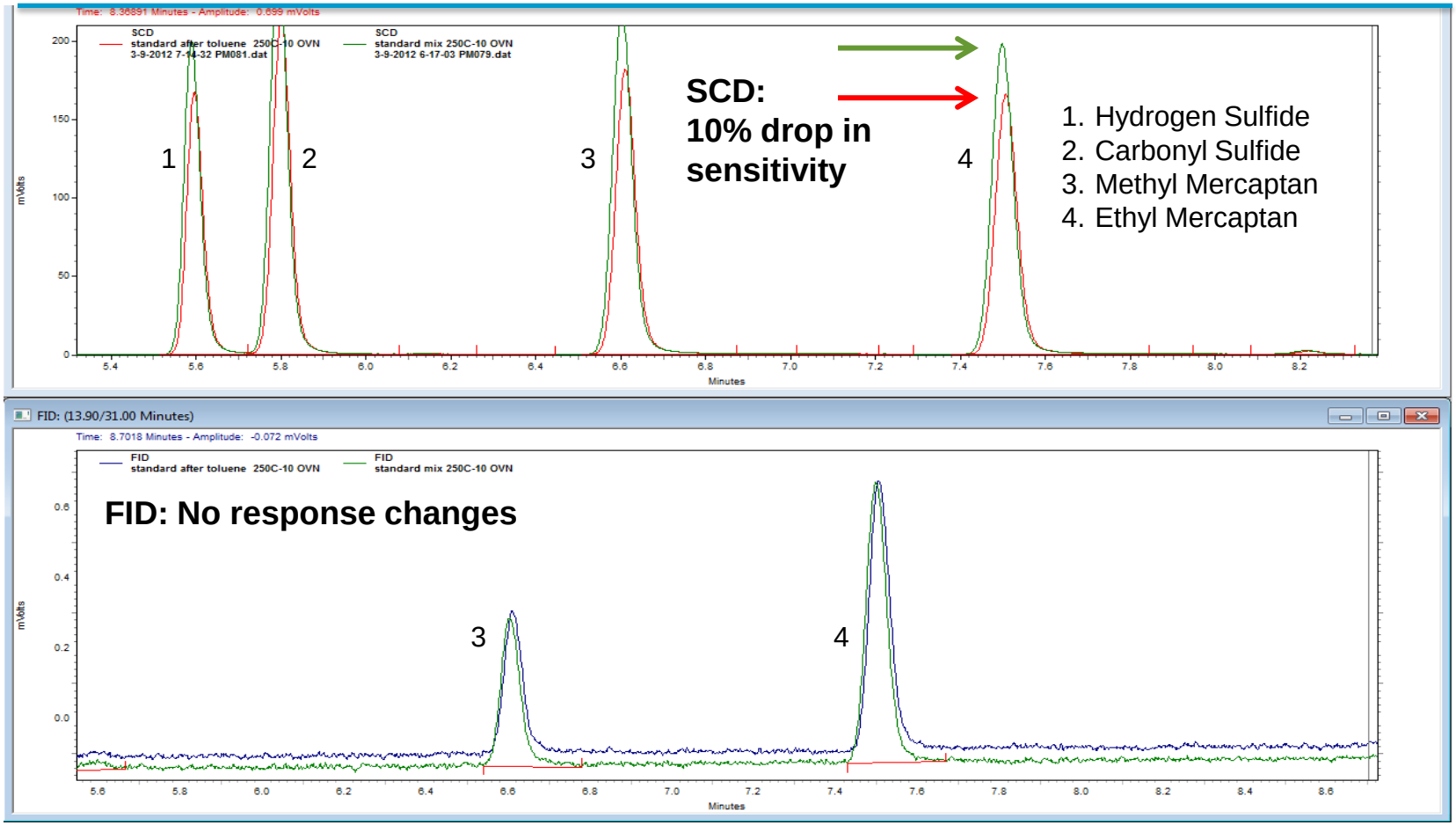
Traditional PDMS column– SCD Ceramic Reaction Tube Deactivation



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Traditional PDMS column- Coking (desensitization) of Reactor Tubes

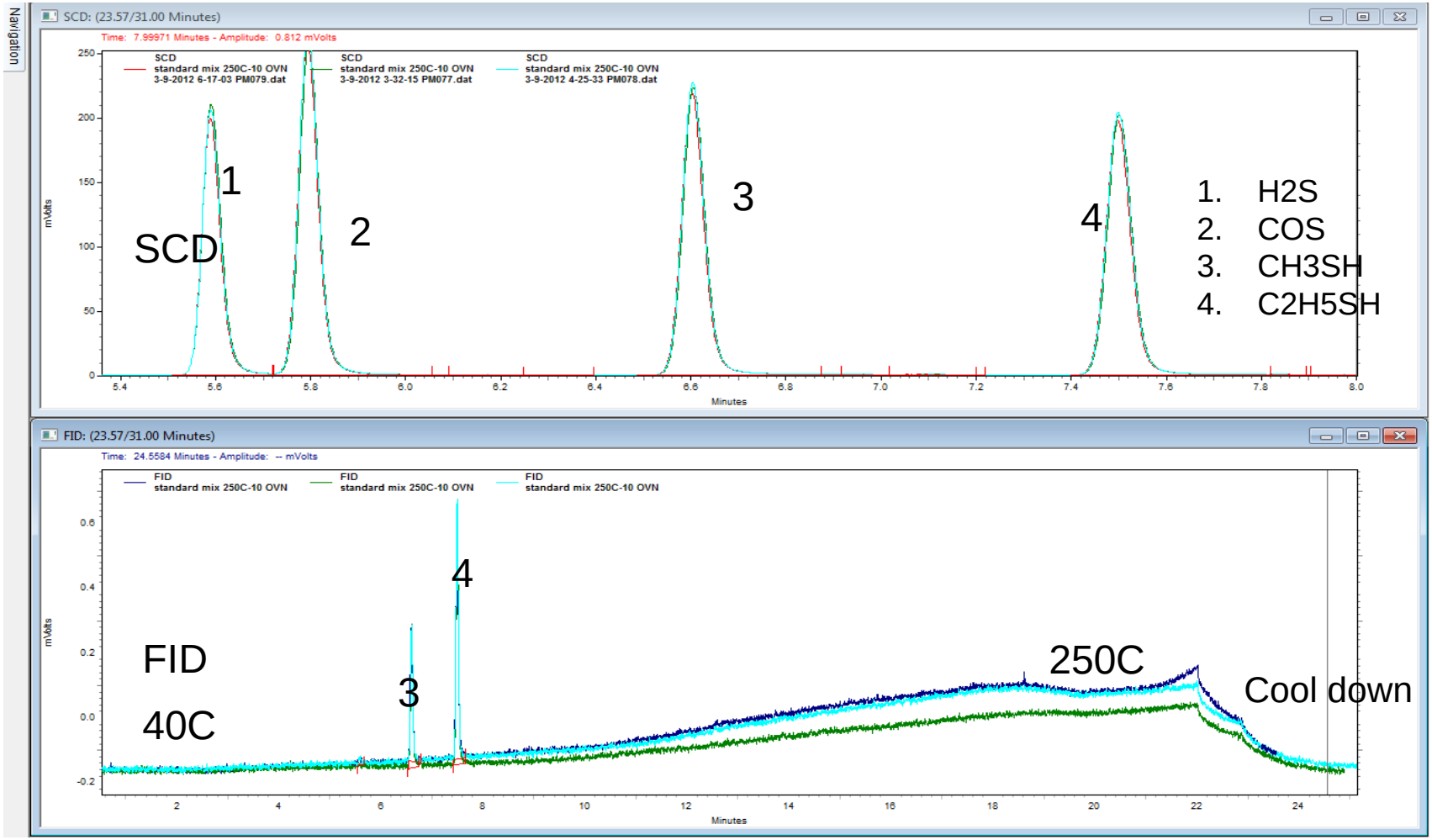
Overlay of before (green) and after 2 x 2 uL neat toluene Injection (red)



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

New DB-Sulfur SCD column

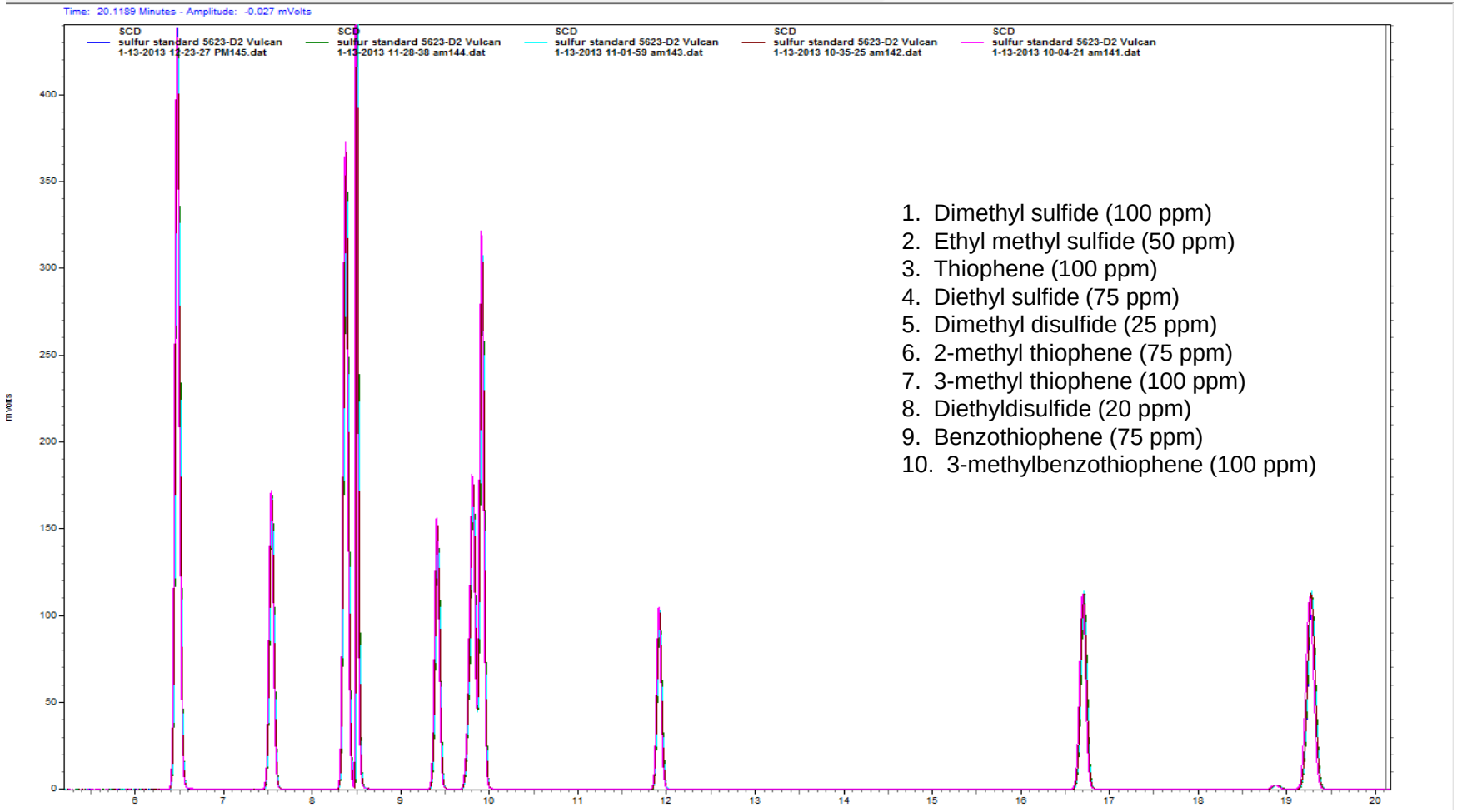
Last Three Runs of the day (n=20, 100 ppmv std)



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

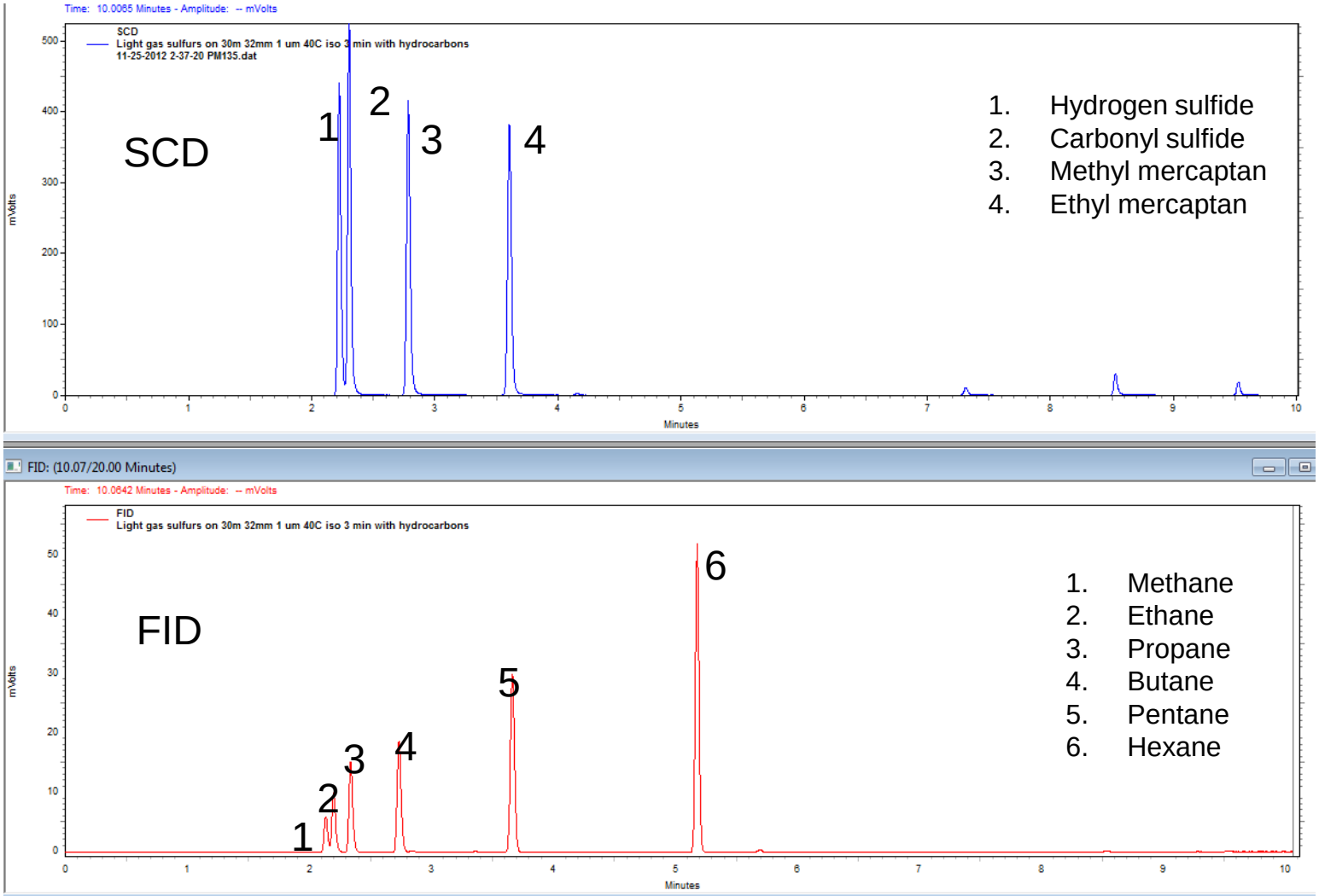

Agilent Technologies

Sulfides and Thiophenes (n=5)



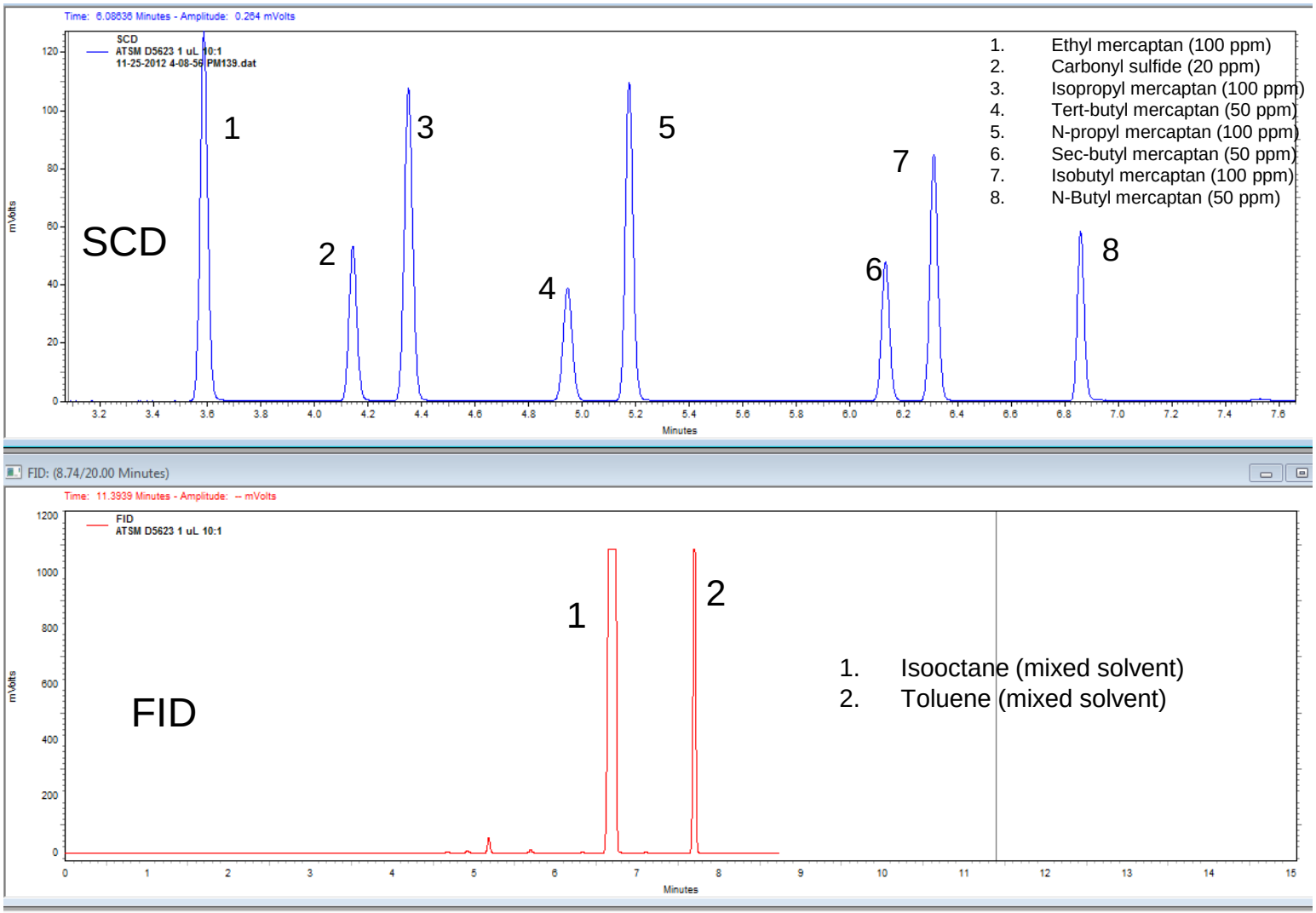
Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Chromatograms of 50 ppm_v each of sulfides and mercaptans and 500 ppm_v each of hydrocarbons in nitrogen



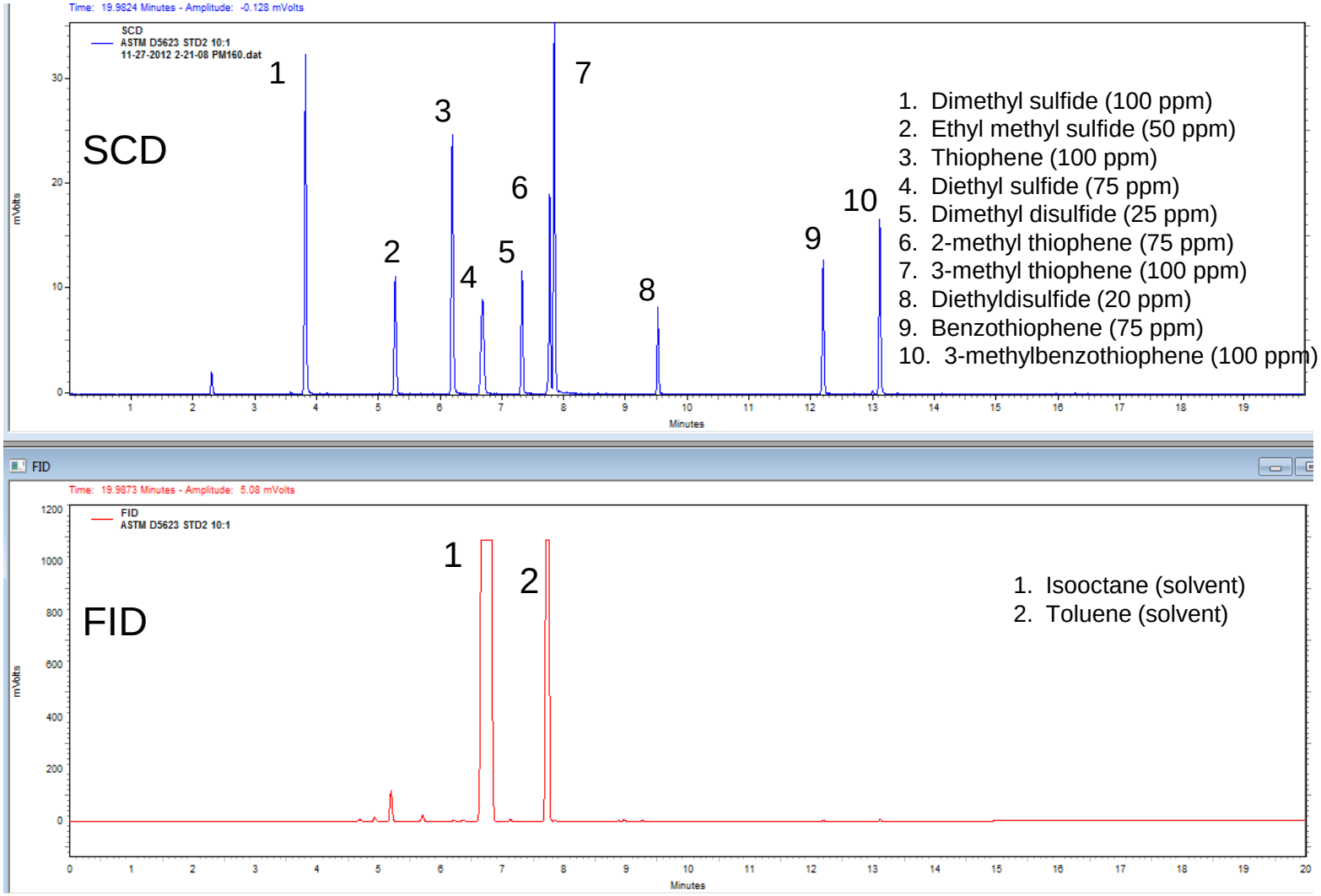
Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Chromatogram of carbon disulfide and alkyl mercaptans



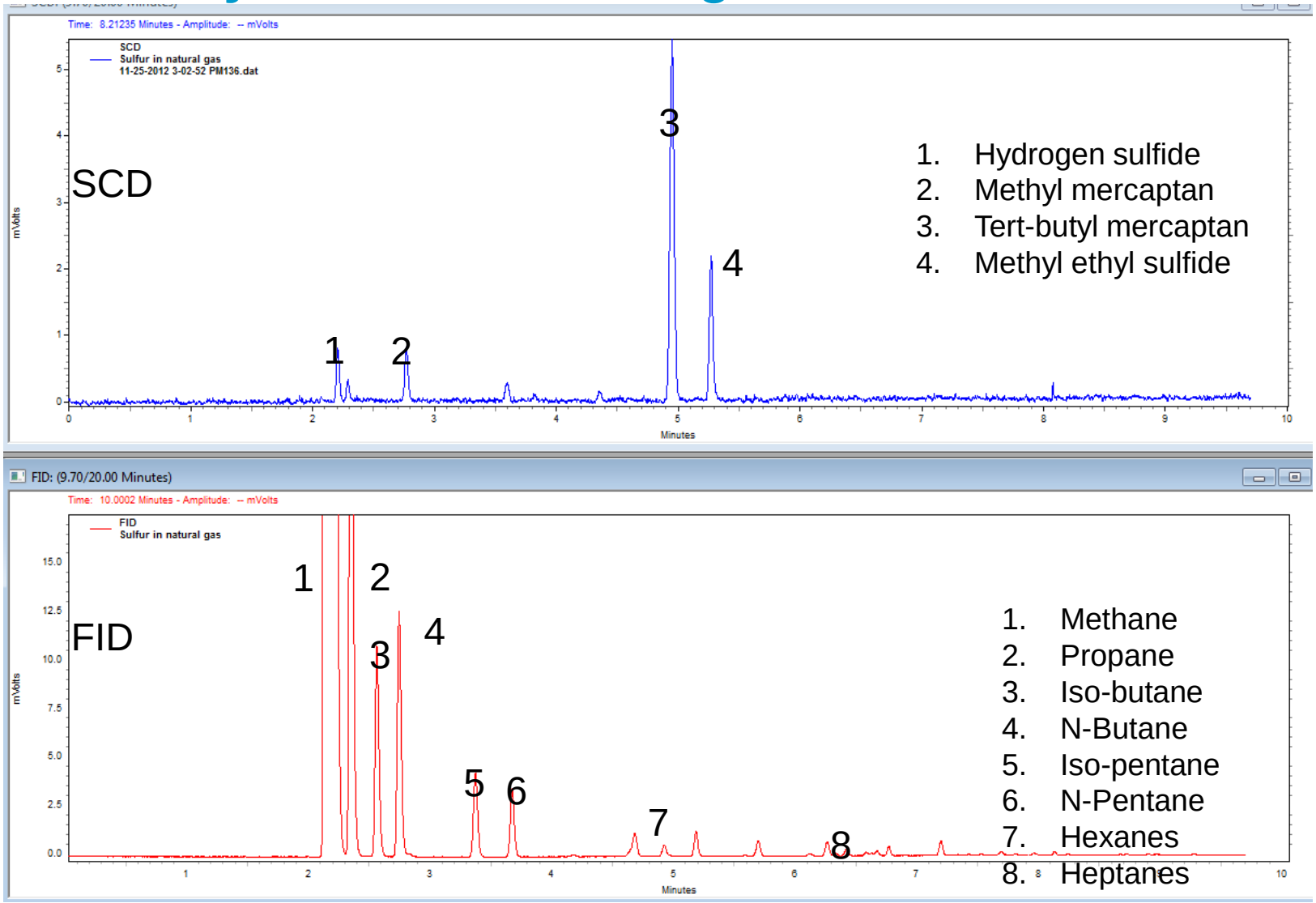
Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Chromatogram of sulfides, disulfides, thiophene, alkyl thiophenes, benzothiophene, and alkyl benzothiophenes



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Chromatogram volatile sulfur odorants in commercially available natural gas



Data courtesy of Jim Luong, Ronda Gras, Myron Hawryluk of Dow Chemical Canada

Conclusions

The new Agilent J&W DB-Sulfur SCD with low bleed and excellent inertness can provide:

- Excellent resolution and peak shape
- Excellent linearity at ppm to ppb levels
- Excellent repeatability
- Less ceramic tube fouling/less detector maintenance

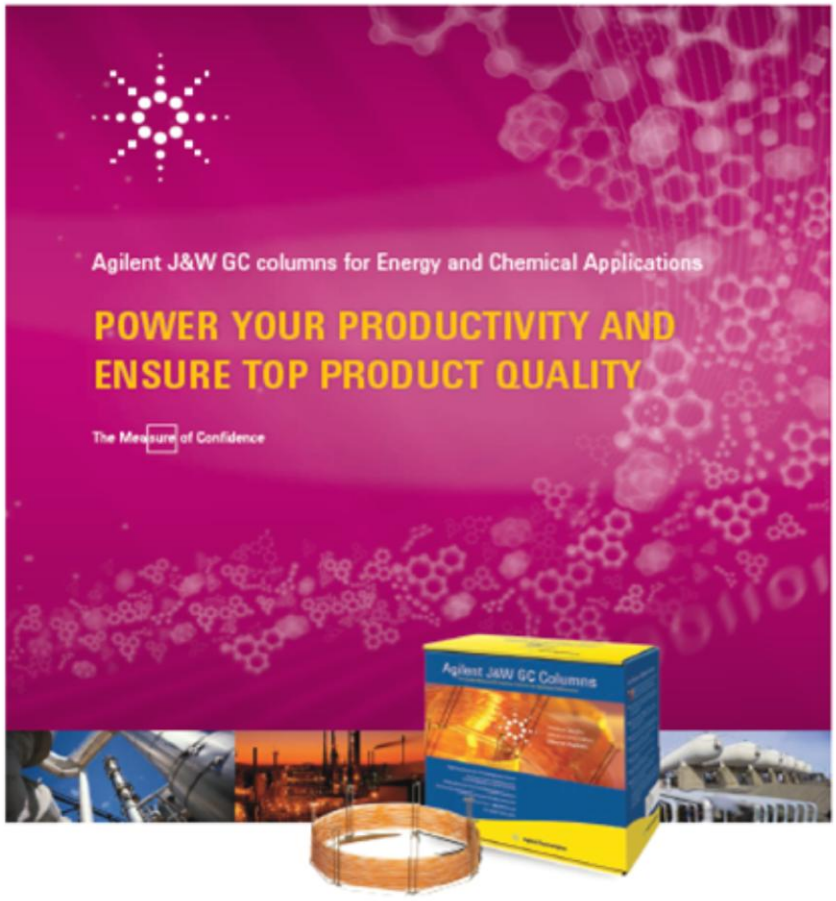
  Before – detector maintenance every 3 weeks

  Now – over 6 months, no SCD maintenance!

Application Notes and Literature:

ASTM D5623 and ASTM D5504

Brochure number 5991-2977EN



New Column Summary

PLOT PT columns

- Similar selectivity to non-PT columns
- Virtually eliminates problems due to particle shedding
- Possible to use MS detection, valves and CFT worry-free

DB-Sulfur SCD columns

- Perfect for dependable volatile sulfur compound analysis utilizing the SCD