# Advan 6 C 12.001 ed Troubleshoo 22 Ti 47.867 ng

**Daron Decker / Eric Pavlich**
**Chromatography Technical Specialist**

# Troubleshooting Tips

1. Isolate the problem.

(Blank Runs, Inject Un-retained Compound, Know what it is not)

2. Change only one variable at a time.

3. Compare before/after chromatograms.

(Peak shape, response, retention, baseline rise, background, look for trends, etc.)

# What Can Possibly Go Wrong?

INJECTOR – contamination, flow settings, flow path issues, overload, valve settings, faulty consumables

COLUMN – contamination, flow settings, flow path issues, damage (activity & bleed), breakage

DETECTOR – contamination, flow settings, flow path issues, electronics

# Logical Troubleshooting

- **Troubleshooting Starts with Isolating the problem**
  - There are 5 basic areas from where the problem arises

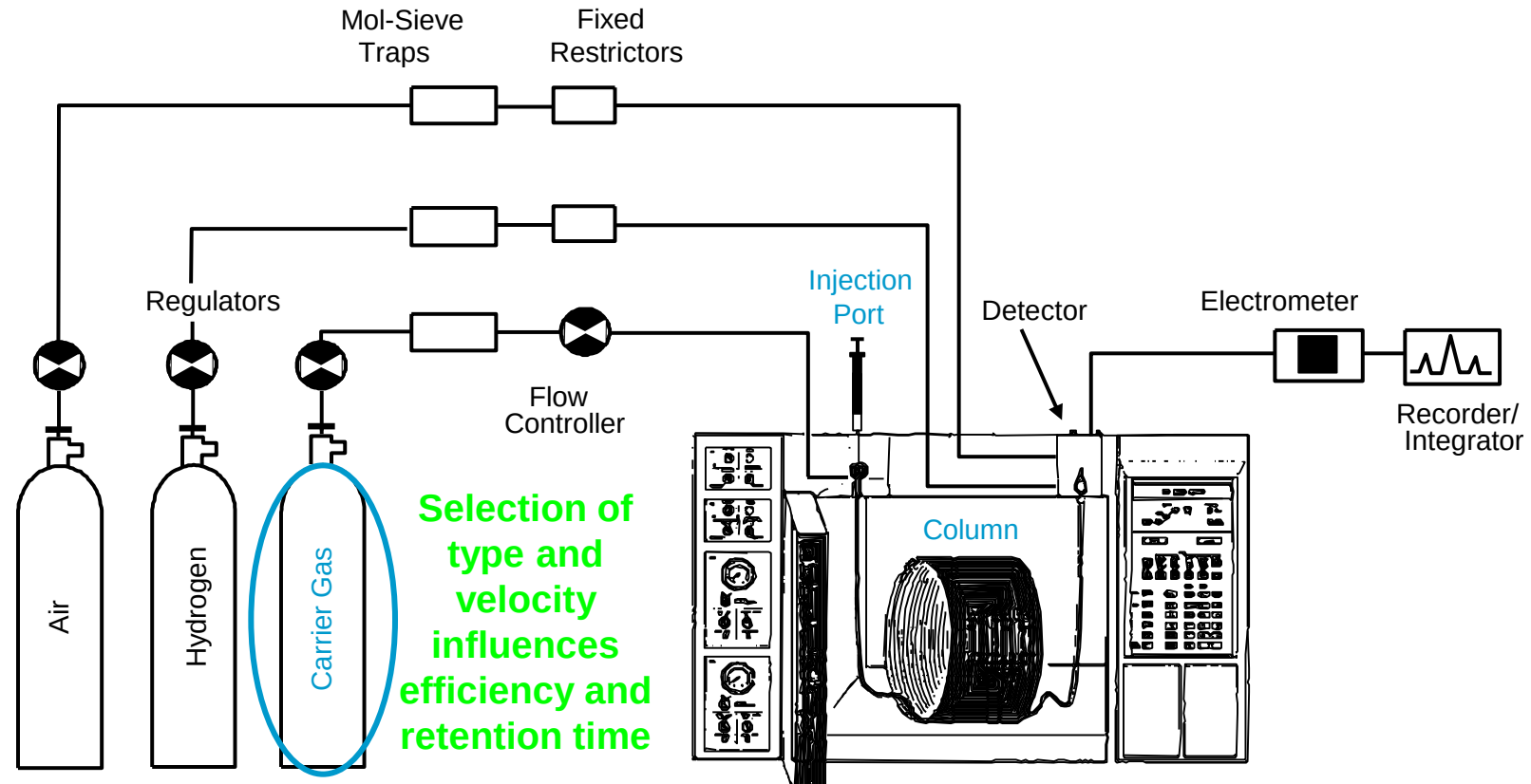
  - FLOW
  - INJECTOR
  - COLUMN
  - DETECTOR
  - ELECTRONICS

  (Temperature)

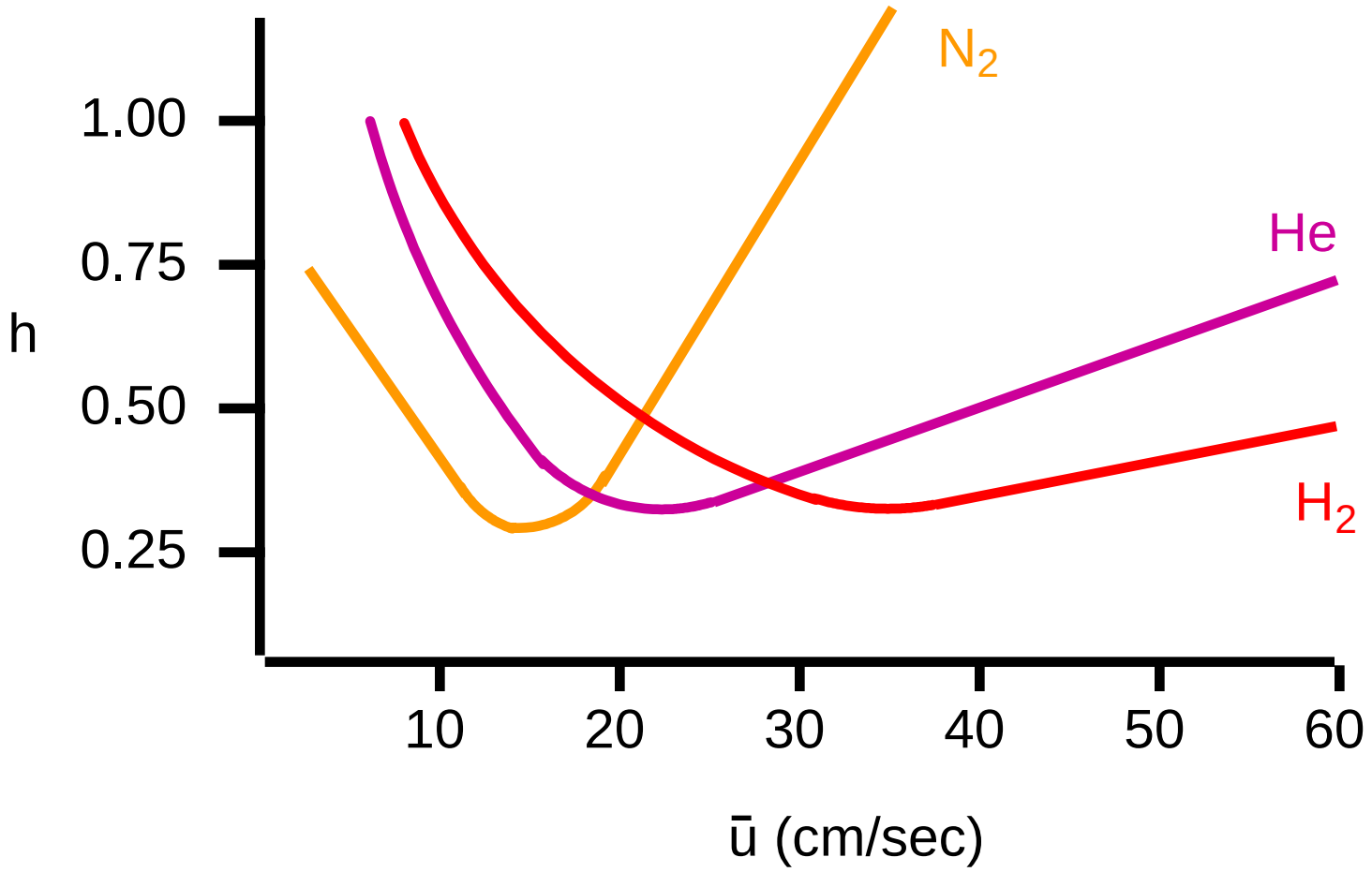
  - But of course it can always be some COMBINATION

- **Knowing what can & <u>can't</u> cause the symptom is the key**

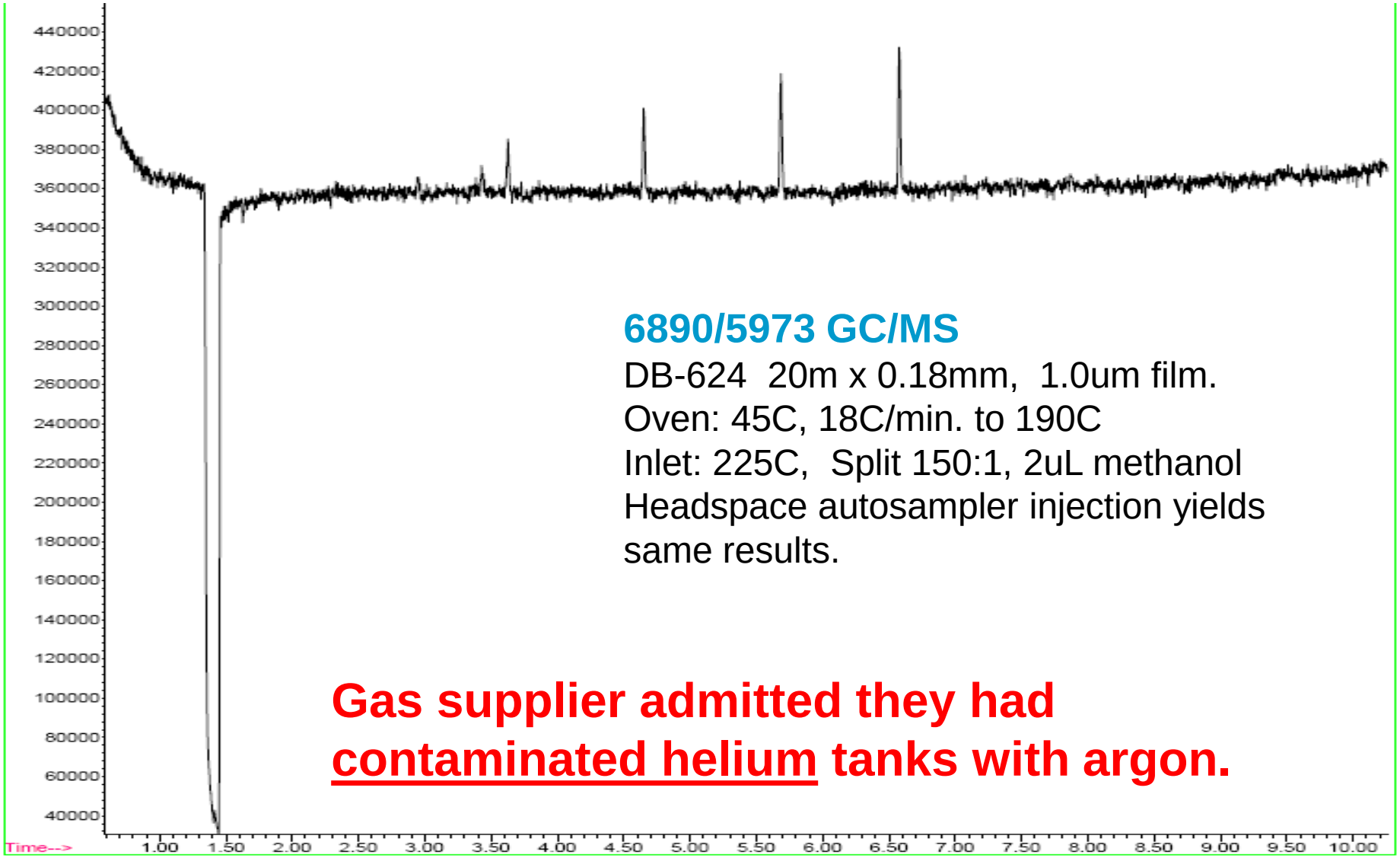
# Typical Gas Chromatographic System



# van Deemter Curves



# Noisy Baseline Even at Low Temperatures




**6890/5973 GC/MS**

DB-624 20m x 0.18mm, 1.0um film.
Oven: 45C, 18C/min. to 190C
Inlet: 225C, Split 150:1, 2uL methanol
Headspace autosampler injection yields same results.

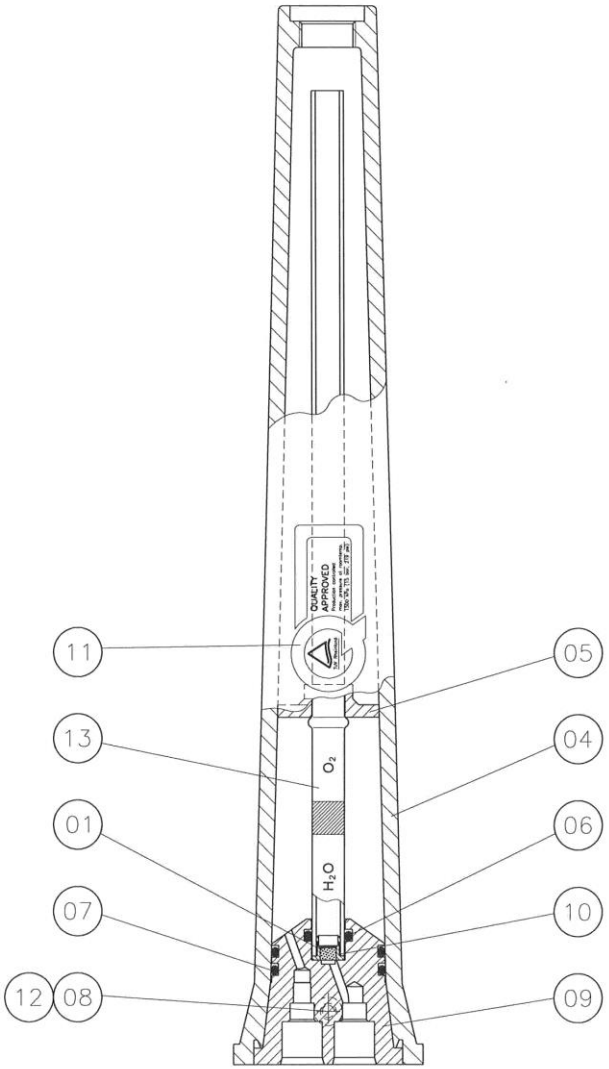
**Gas supplier admitted they had <u>contaminated helium</u> tanks with argon.**

# Gas Clean Filters




Agilent Technologies

# Gas-Clean Filters Design




Sensitive indicators for moisture and oxygen

Short filter equilibration time

Fast, leak-free filter replacement

- without turning off the gas
- NO tools needed

# Oxygen Damages GC Columns

Repair of the GC column is impossible

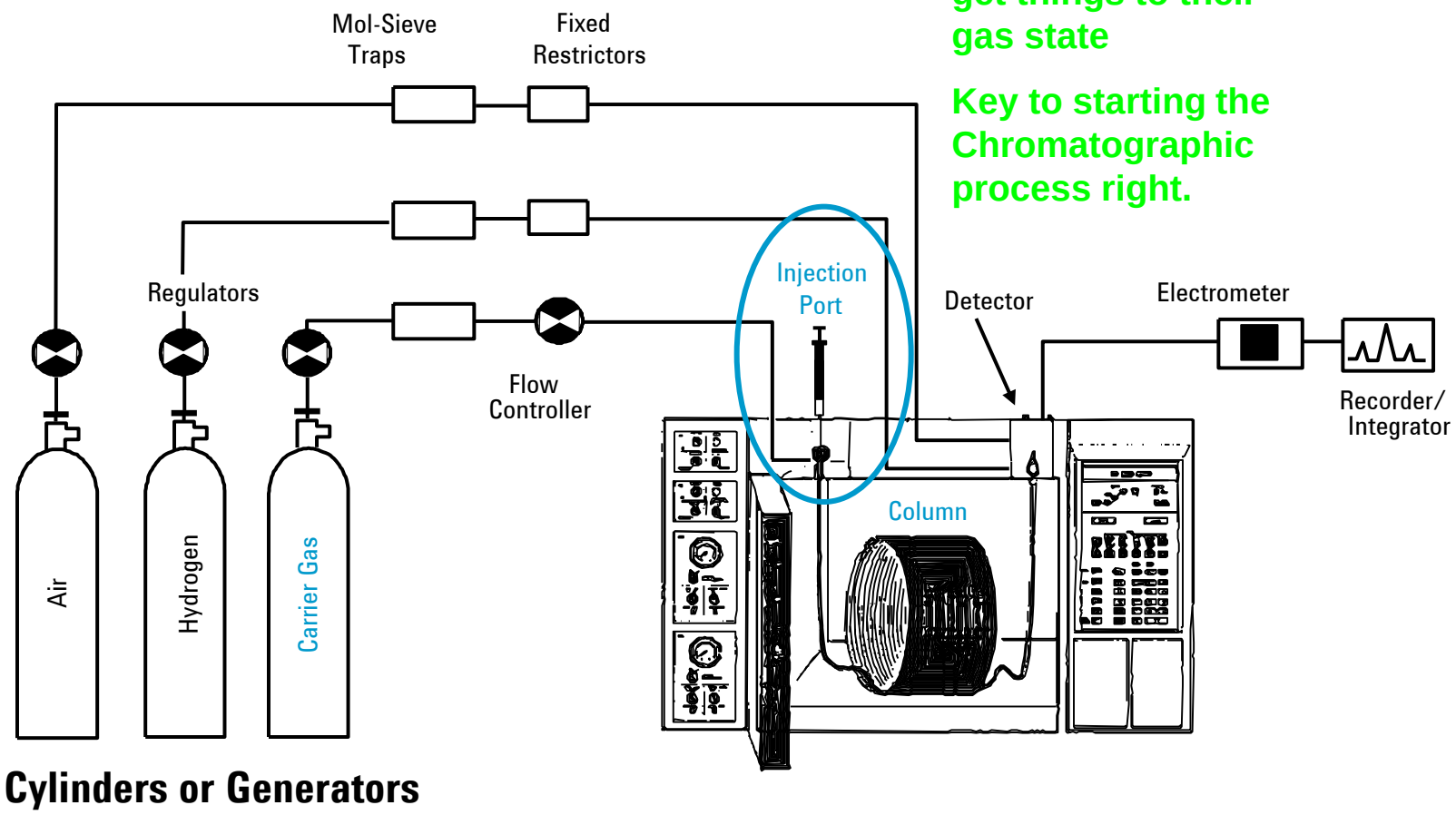
Replacements means re-calibration

Replacement means delay

Replacement column costs money

Solution = No Leaks!

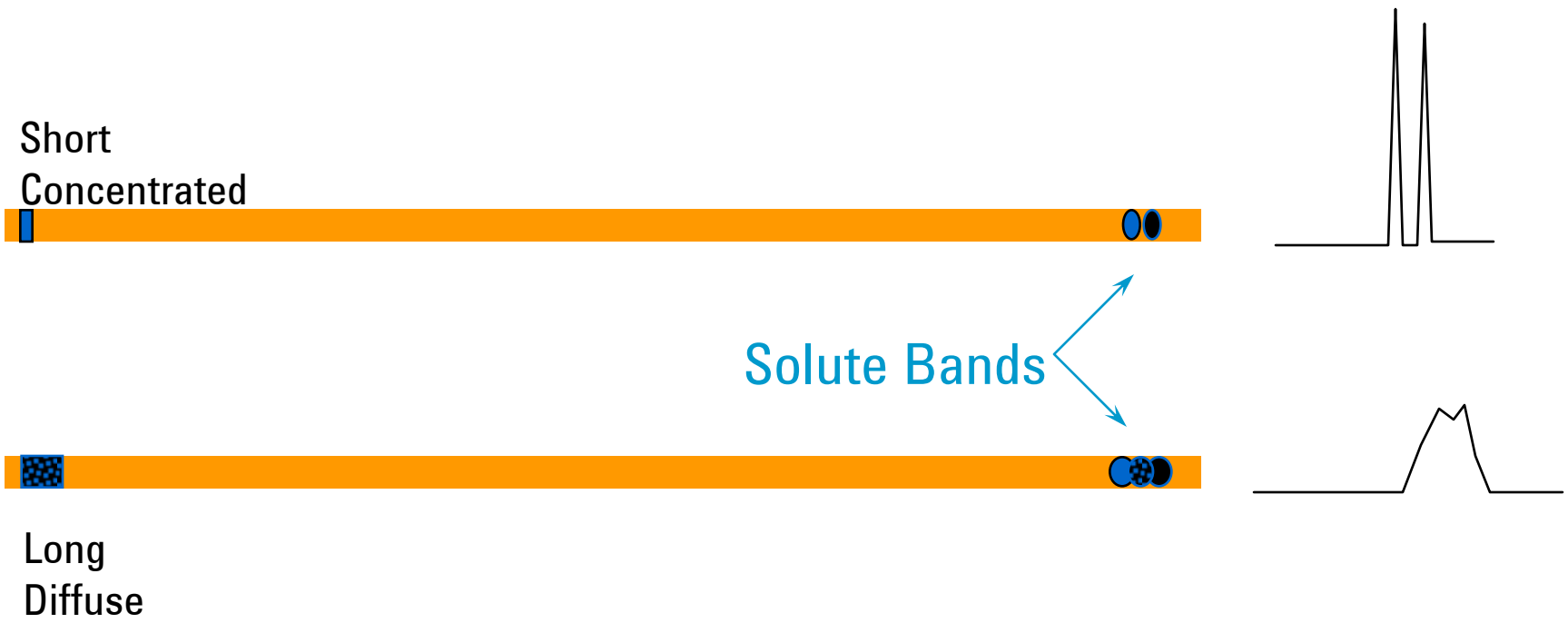
# Typical Gas Chromatographic System




Have to be able to get things to their gas state

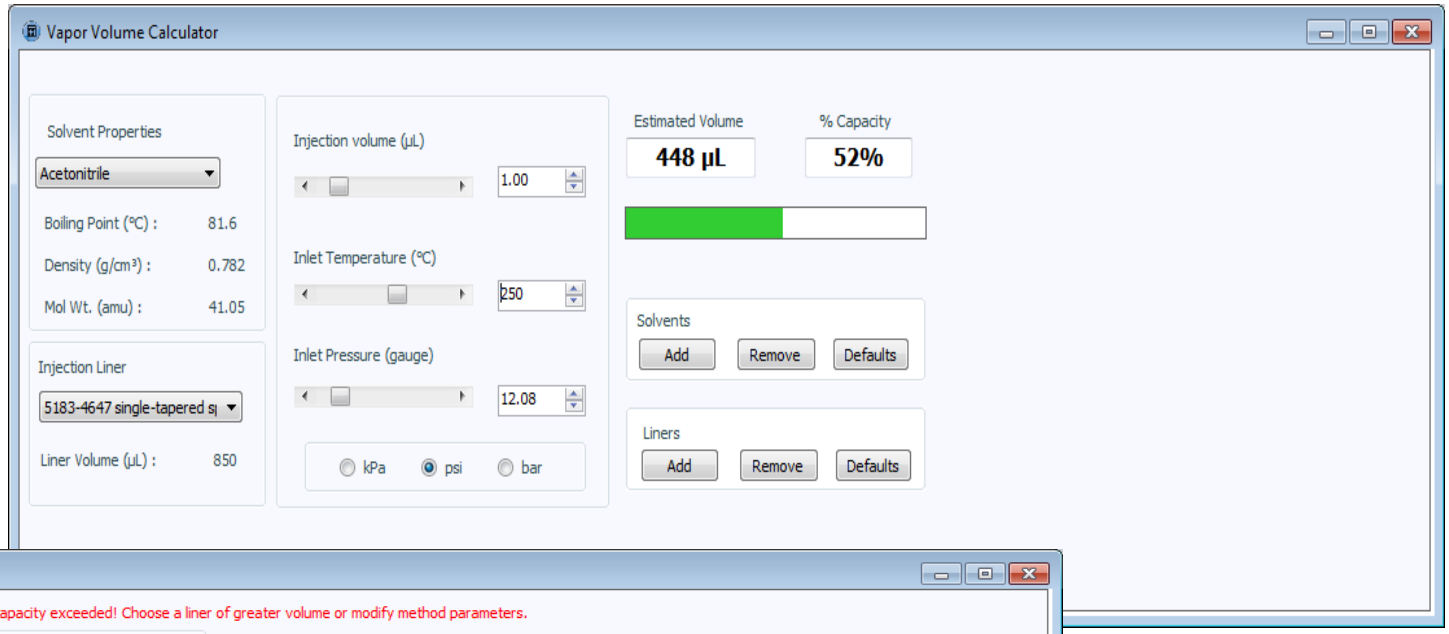
Key to starting the Chromatographic process right.

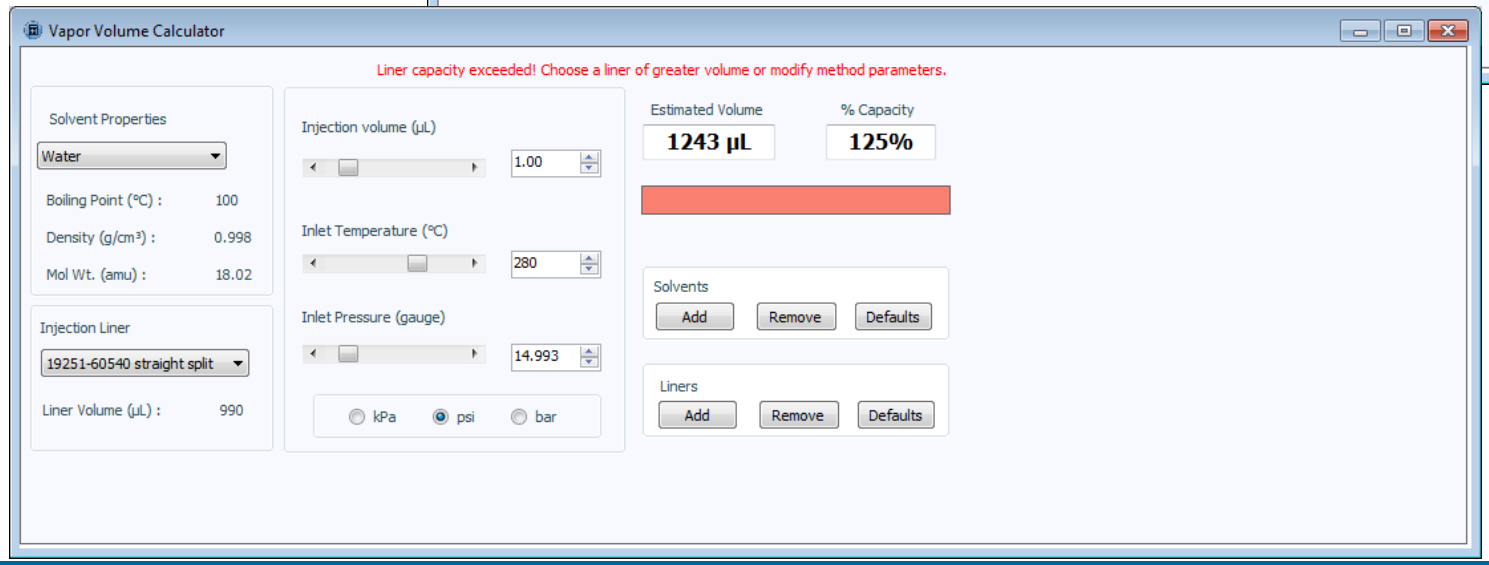
# Influence of Injection Efficiency




**Same column, same chromatographic conditions**

# Solvent Vapor Volume Calculator



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# The BIGGEST Problem in GC is…

There are more things that DON’T go through a GC than DO!

….therefore, don’t inject anything and you’ll never have problems.

OK, inject, but realize that everything just got dirty…deal with it!

# Where Does it Get Dirty?

Here

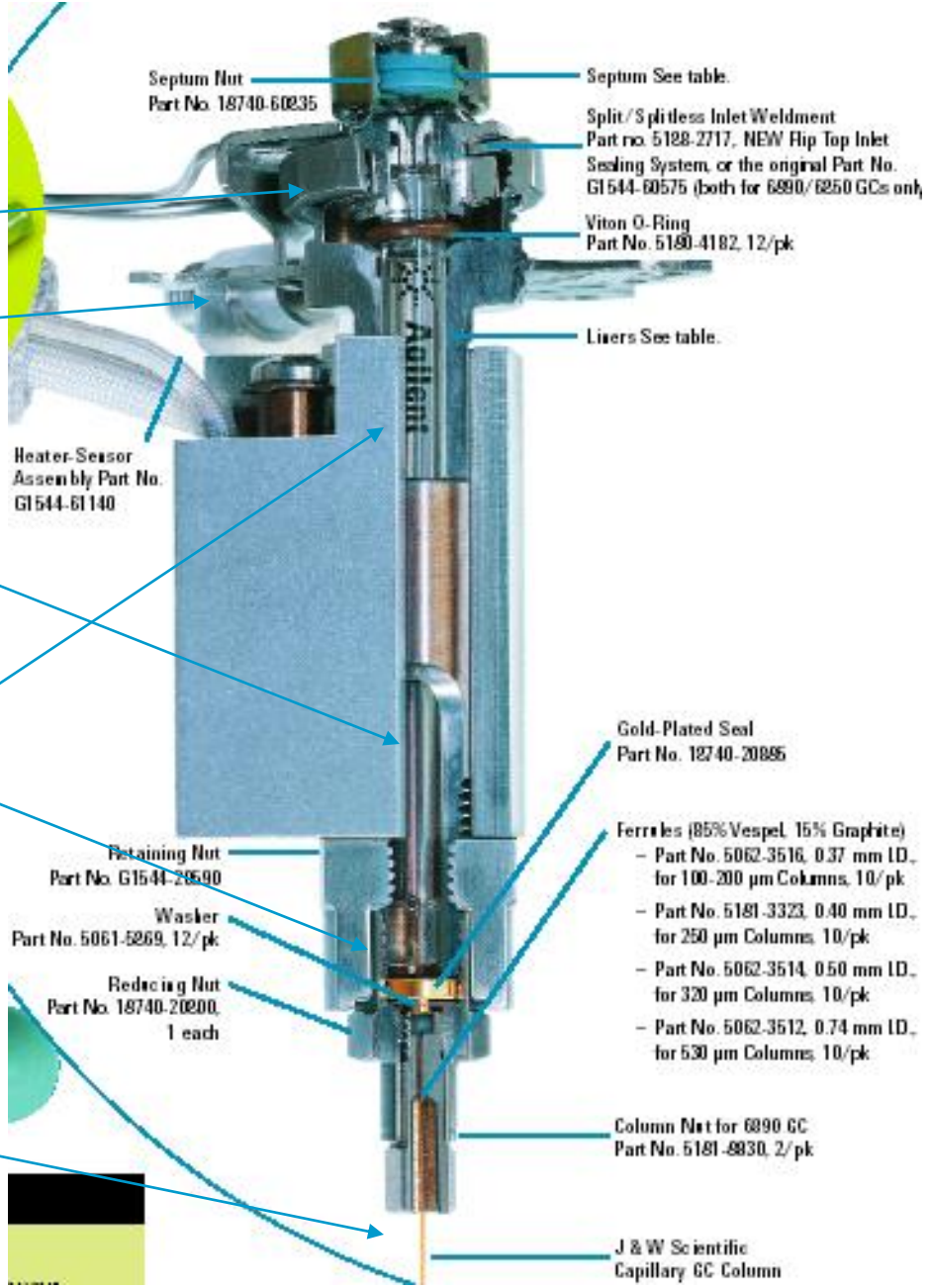
Here

Here

Here

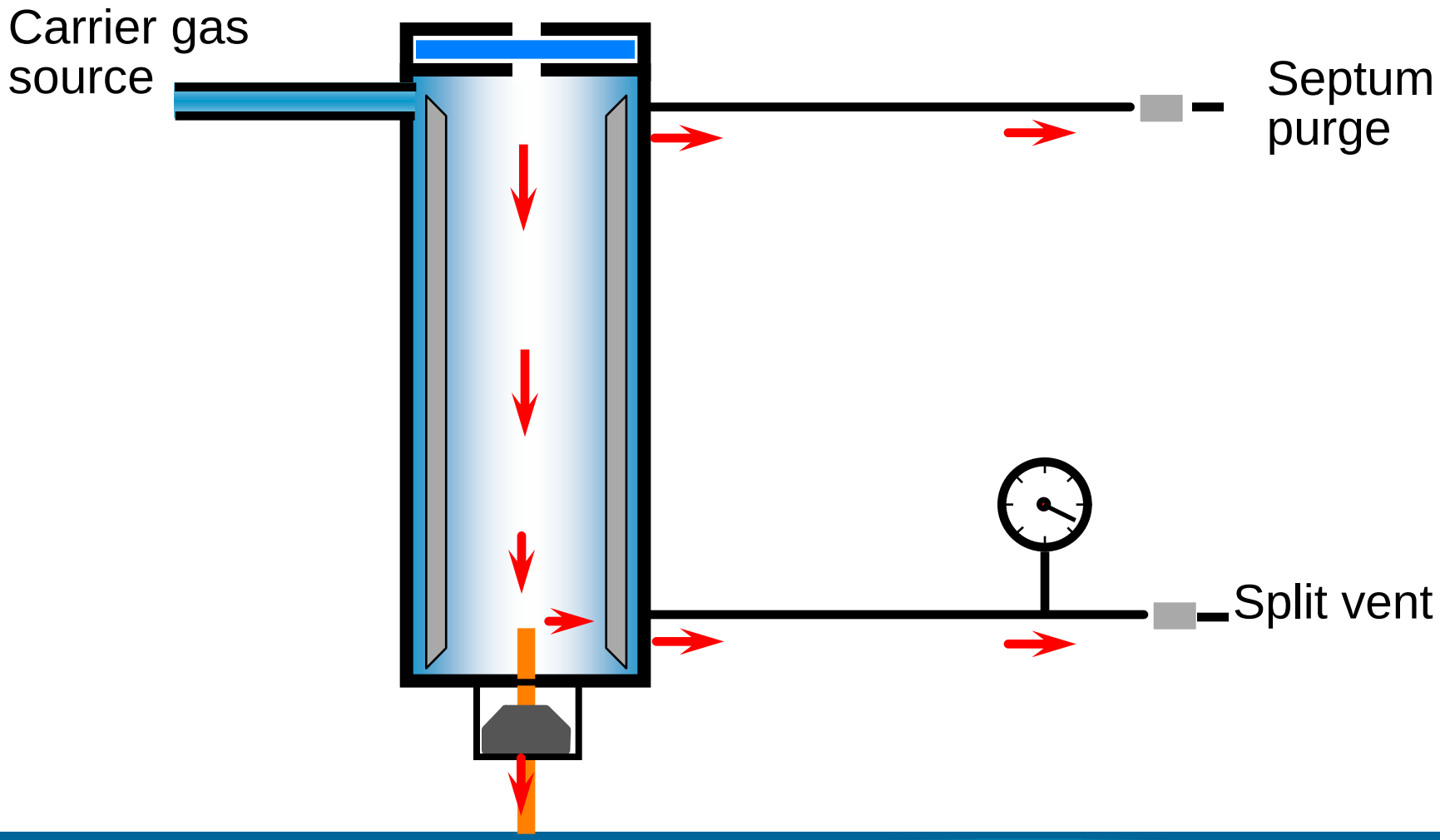
Here

Here

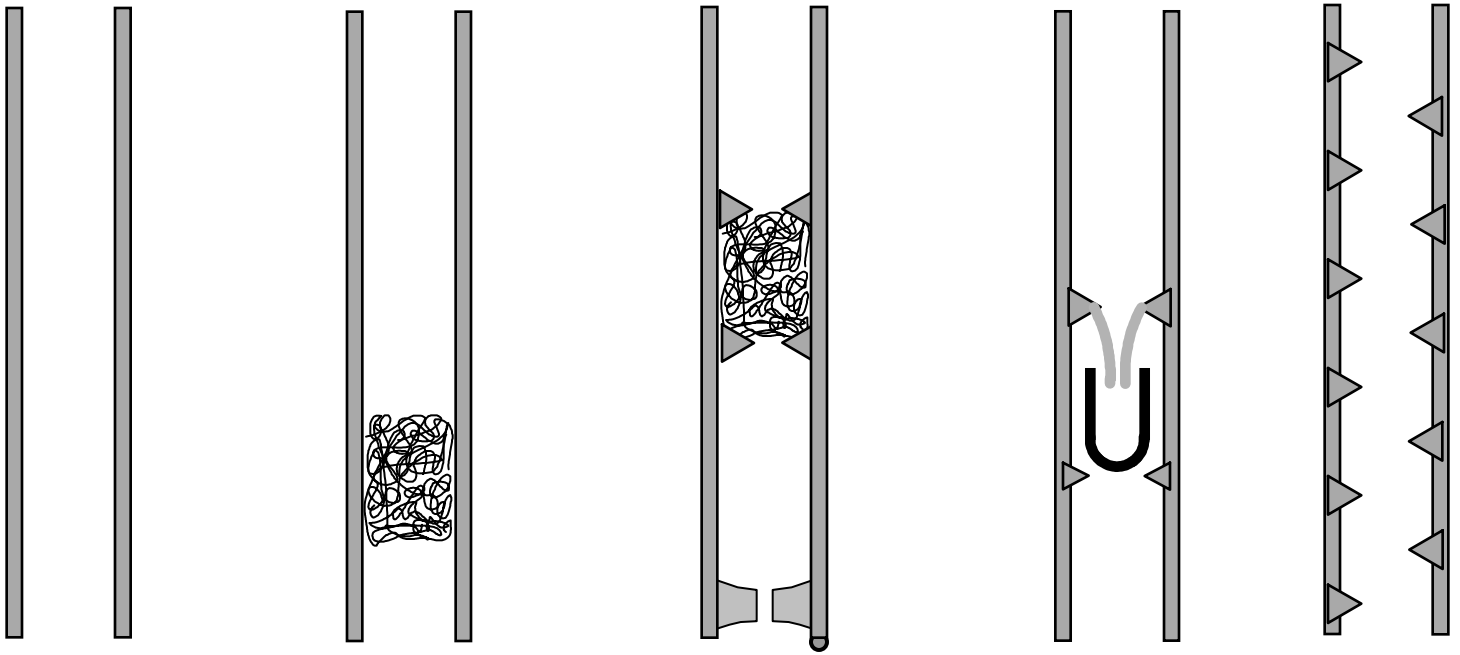


# What Are You Doing!?


# Split Injector
## Flow Path



# Split Liners – What's What?



Straight tube

Straight tube with glass wool

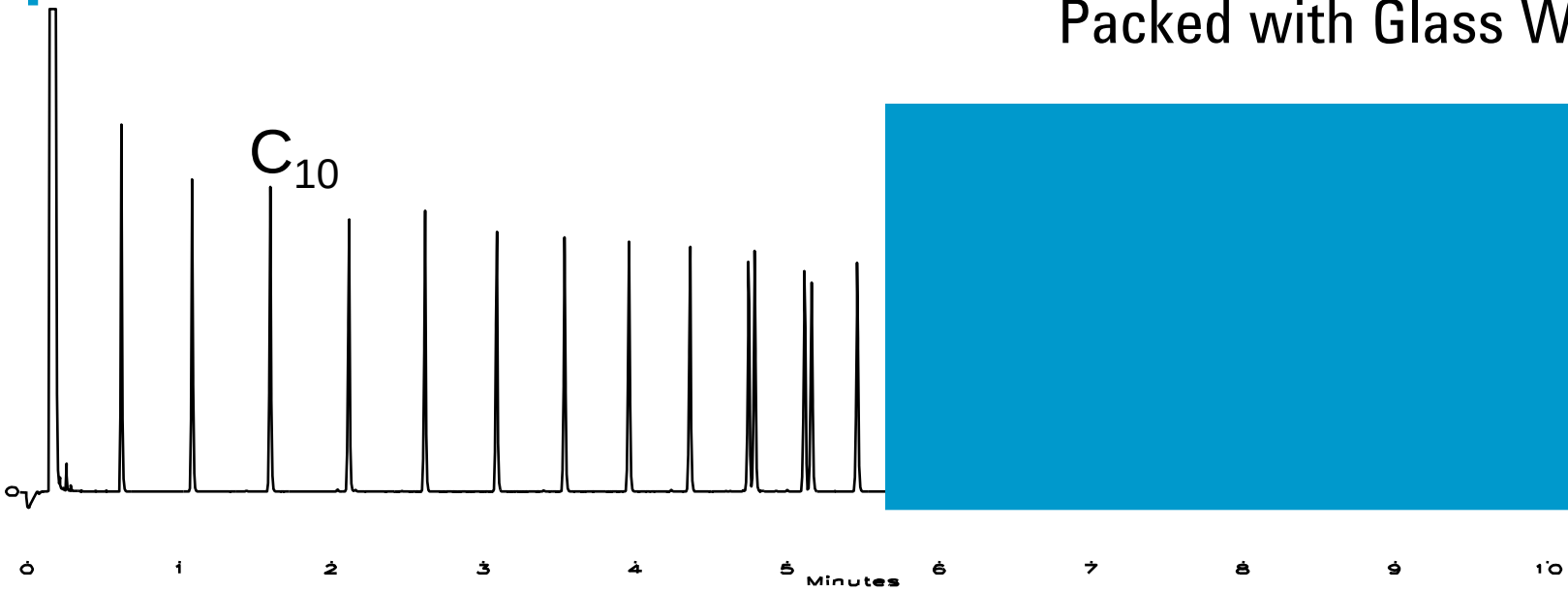
Fixed glass wool

Inverted cup

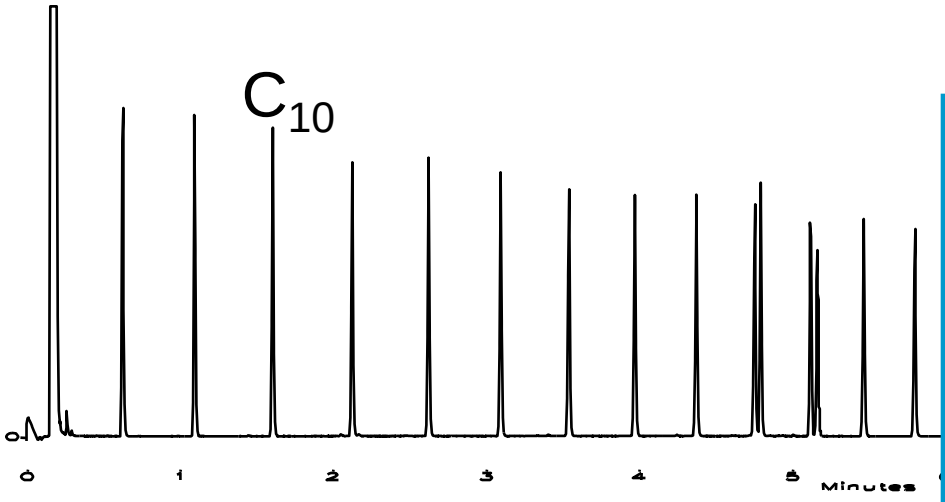
Baffle

# Split Liner

Packed with Glass Wool

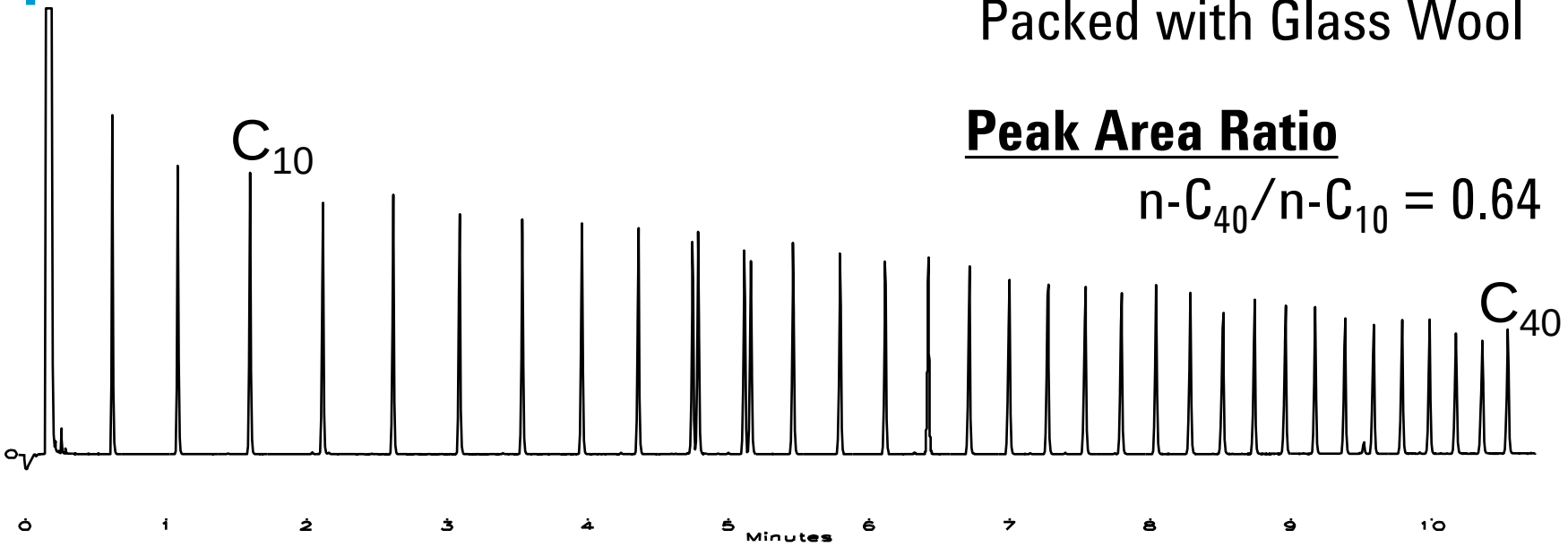



Without Glass Wool Packing



# Split Liner

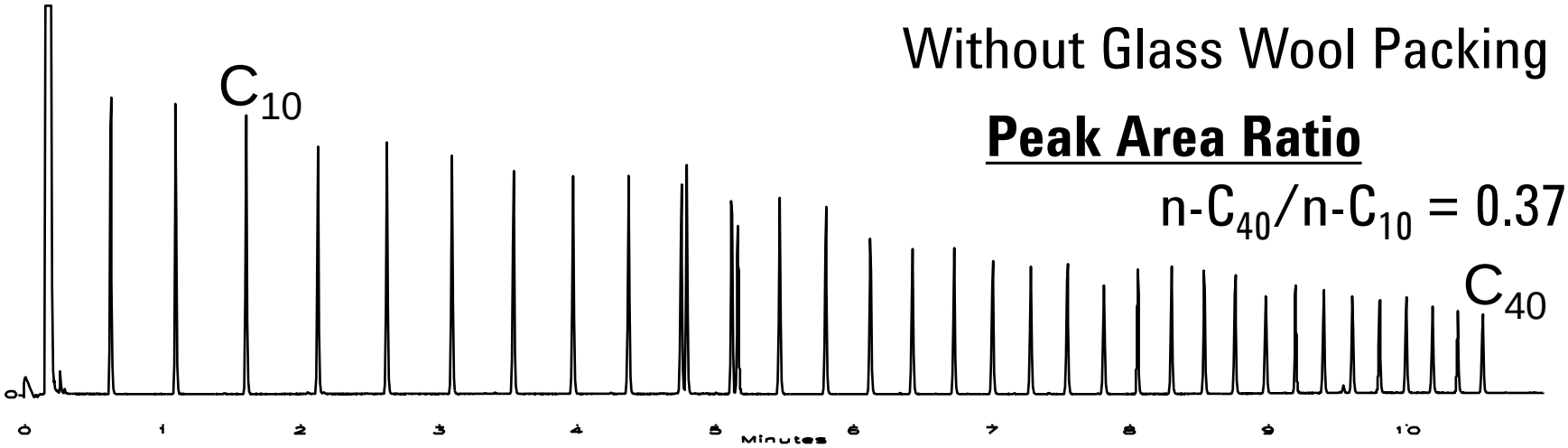
Packed with Glass Wool




**Peak Area Ratio**

$n\text{-}C_{40}/n\text{-}C_{10} = 0.64$

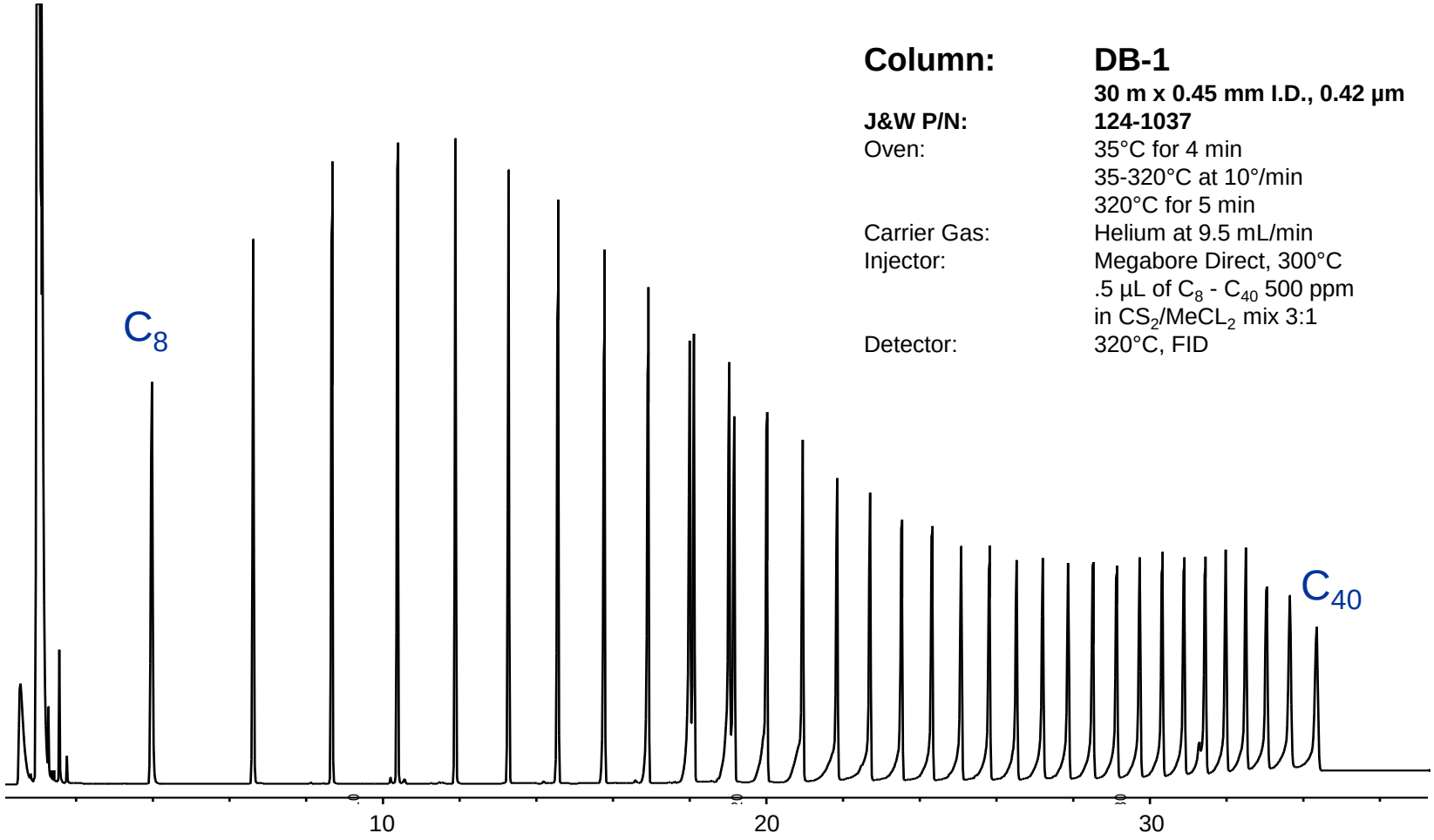
Without Glass Wool Packing




**Peak Area Ratio**

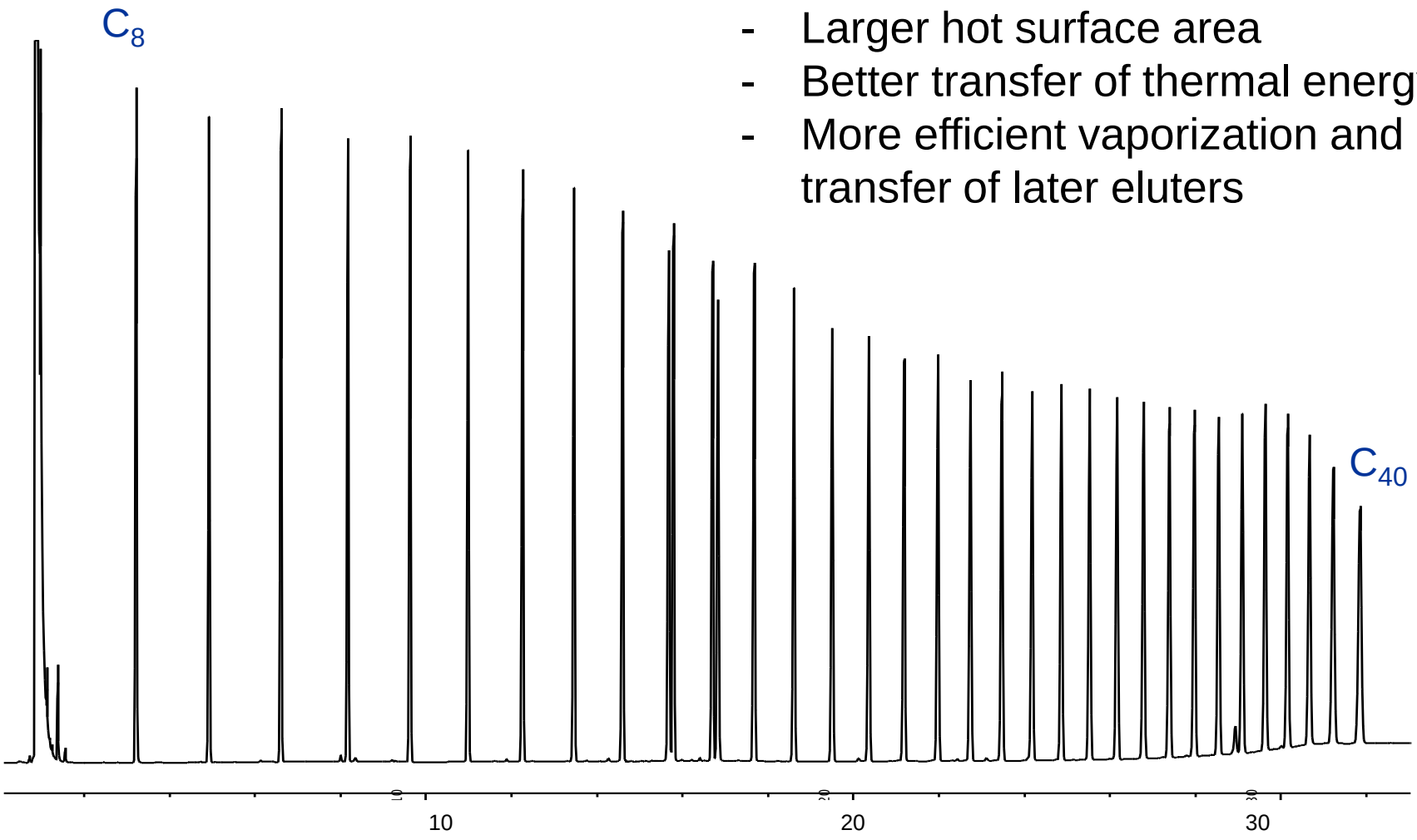
$n\text{-}C_{40}/n\text{-}C_{10} = 0.37$

# Fronting and Discrimination of Later Eluters




| | |
|---|---|
| **Column:** | **DB-1** |
| | **30 m x 0.45 mm I.D., 0.42 µm** |
| **J&W P/N:** | **124-1037** |
| Oven: | 35°C for 4 min |
| | 35-320°C at 10°/min |
| | 320°C for 5 min |
| Carrier Gas: | Helium at 9.5 mL/min |
| Injector: | Megabore Direct, 300°C |
| | .5 µL of $C_8$ - $C_{40}$ 500 ppm |
| | in $CS_2$/$MeCL_2$ mix 3:1 |
| Detector: | 320°C, FID |

# Large Glass Wool Plug Added to Liner




- Larger hot surface area
- Better transfer of thermal energy
- More efficient vaporization and transfer of later eluters

# Discrimination
## Considerations

Efficient heat transfer to injected sample

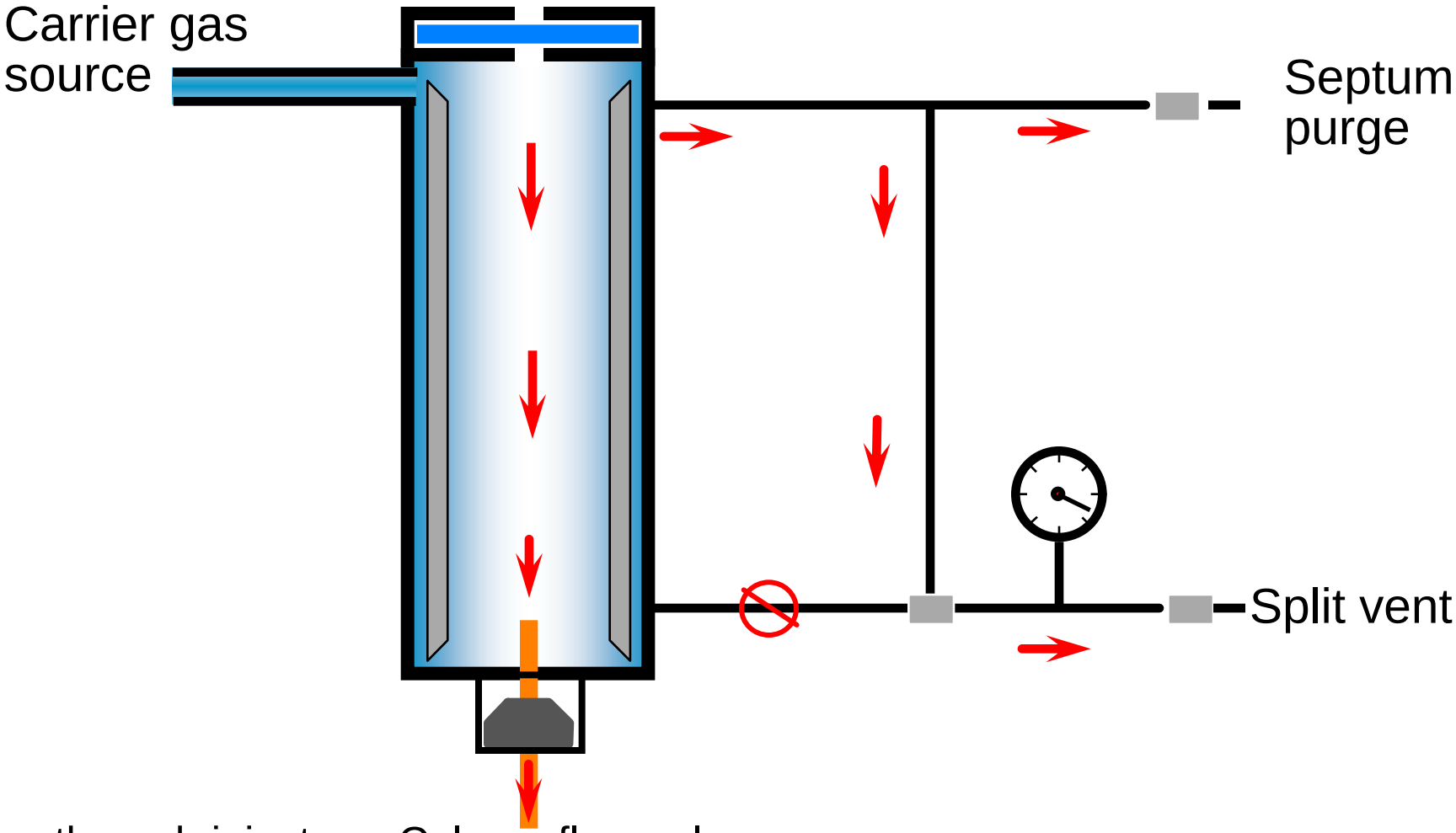
Efficient mixing of vaporized sample with carrier gas

Column position in inlet

Consistent conditions are important

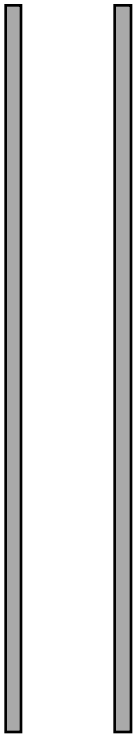
# Splitless Injector

## Purge Off At Injection

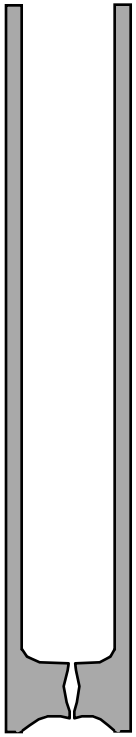



Flow through injector = Column flow only

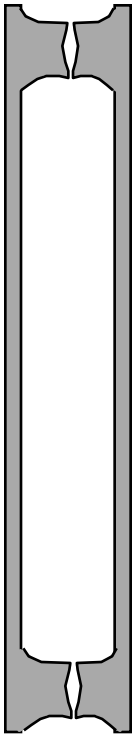
# Splitless Injector
## Liners



Straight tube



Bottom restriction

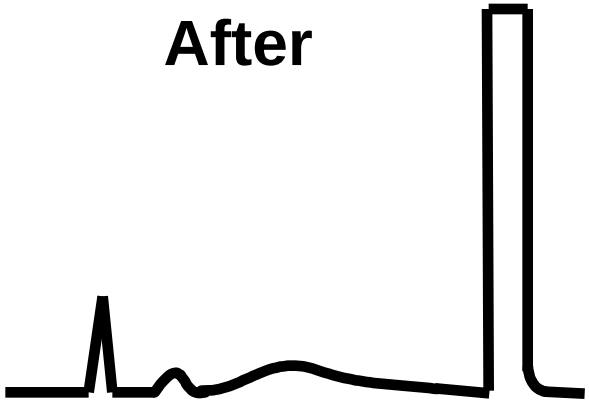


Dual restriction

# Bonus Peaks or Ghost Peaks

**Before**

**After**



Contamination in INJECTOR or FLOW (carrier gas)

-Contaminated consumables

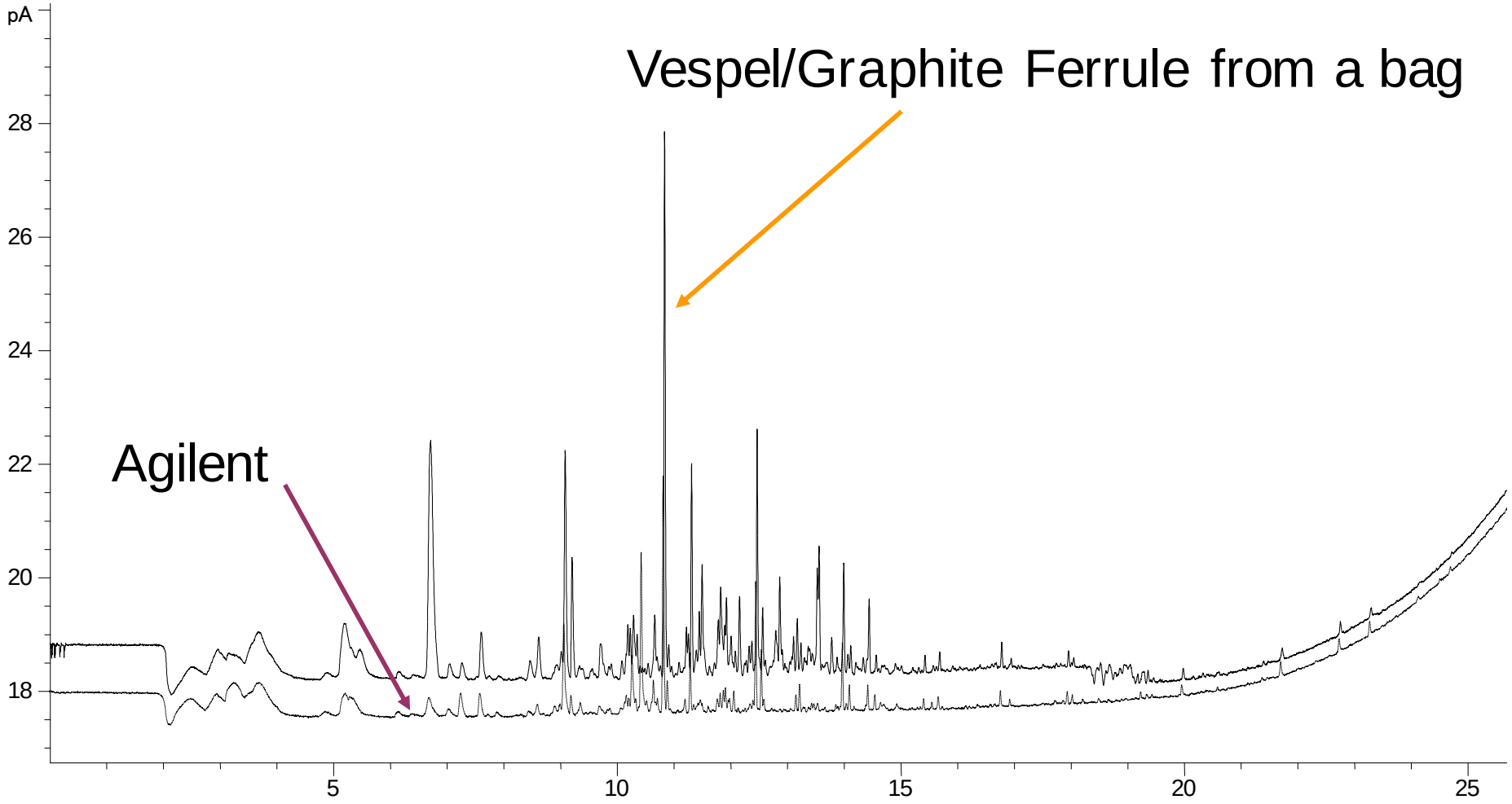
-Carryover from a backflash or previous sample

-Bad tank of gas or traps have expired

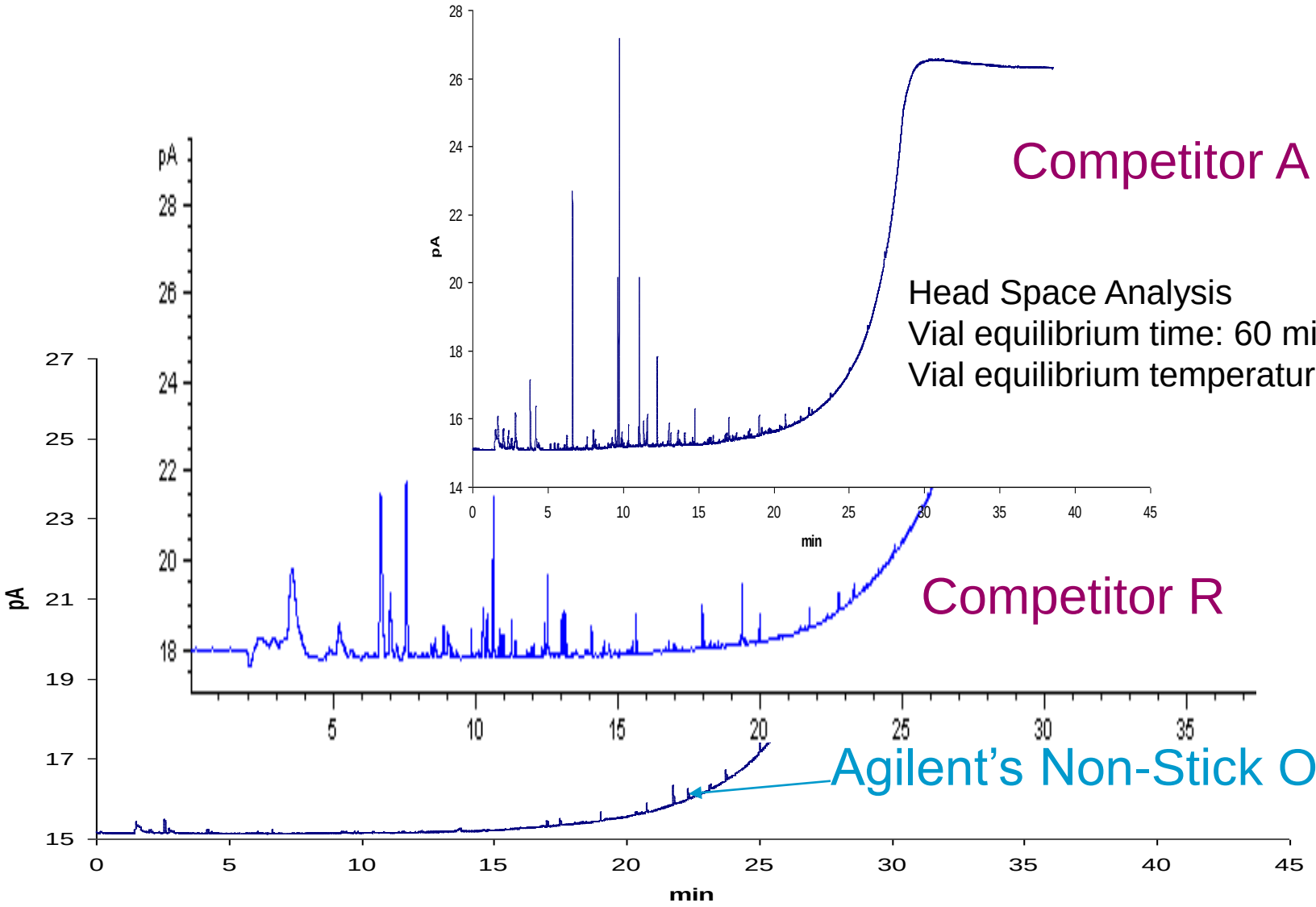
-Septum bleed

*TIP = Run a blank run…it should be blank!

# Bonus Peaks - Ferrule Contamination

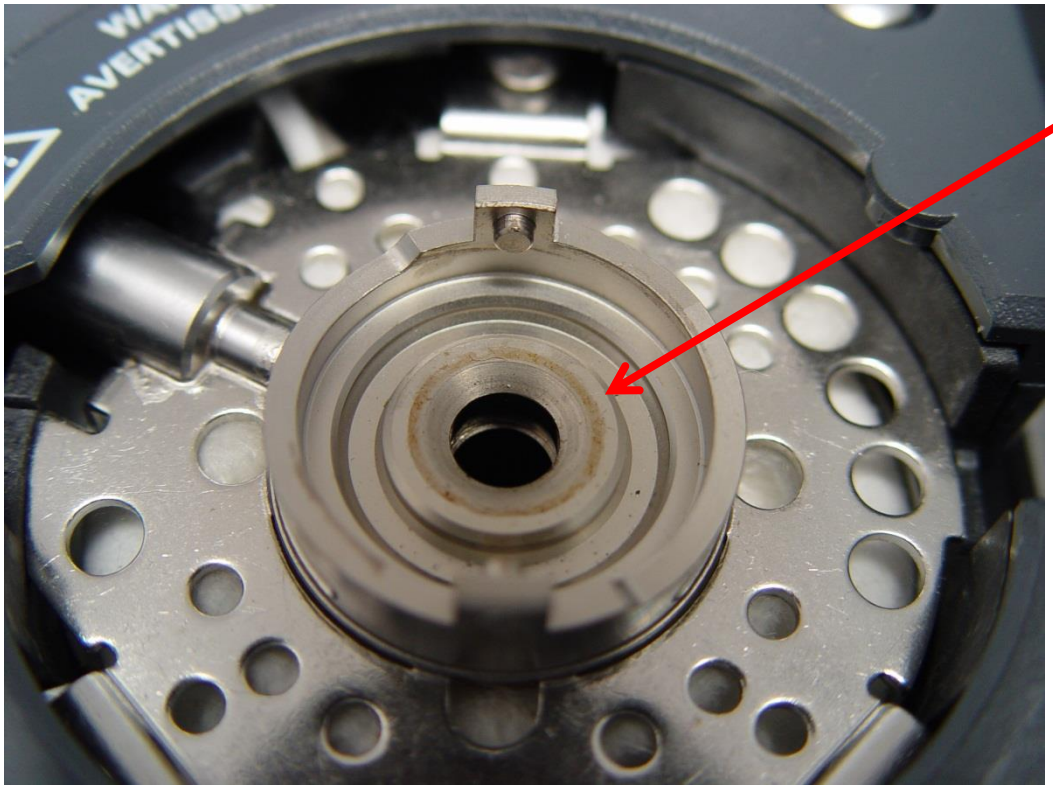


# Bonus Peaks - O-Ring Contamination



# More “Off-Brand” O-Ring Issues

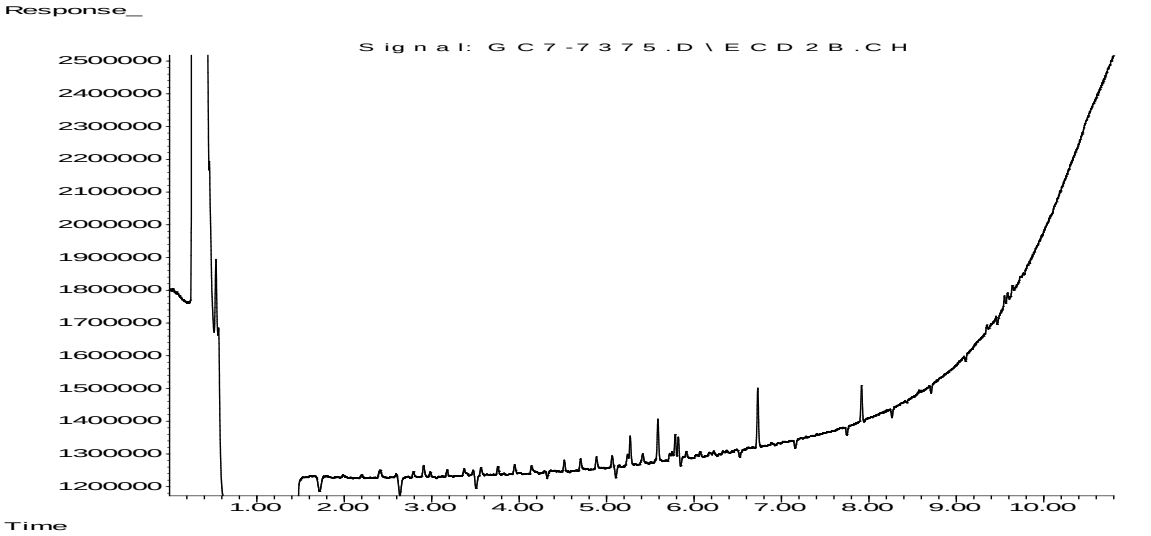
## Controlled Substances Analysis, H2 Carrier

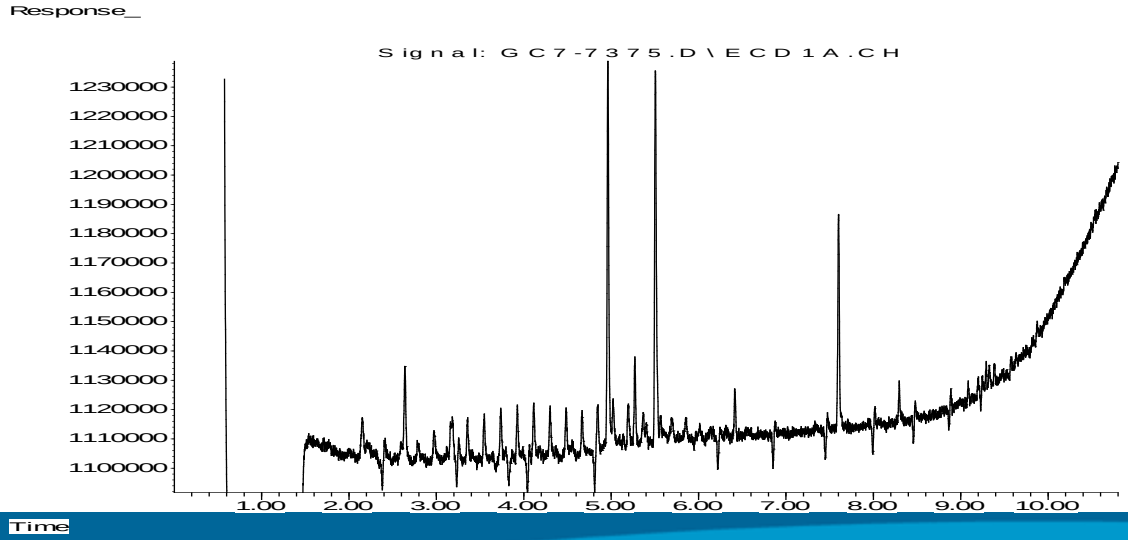


Residue on top of inlet weldment

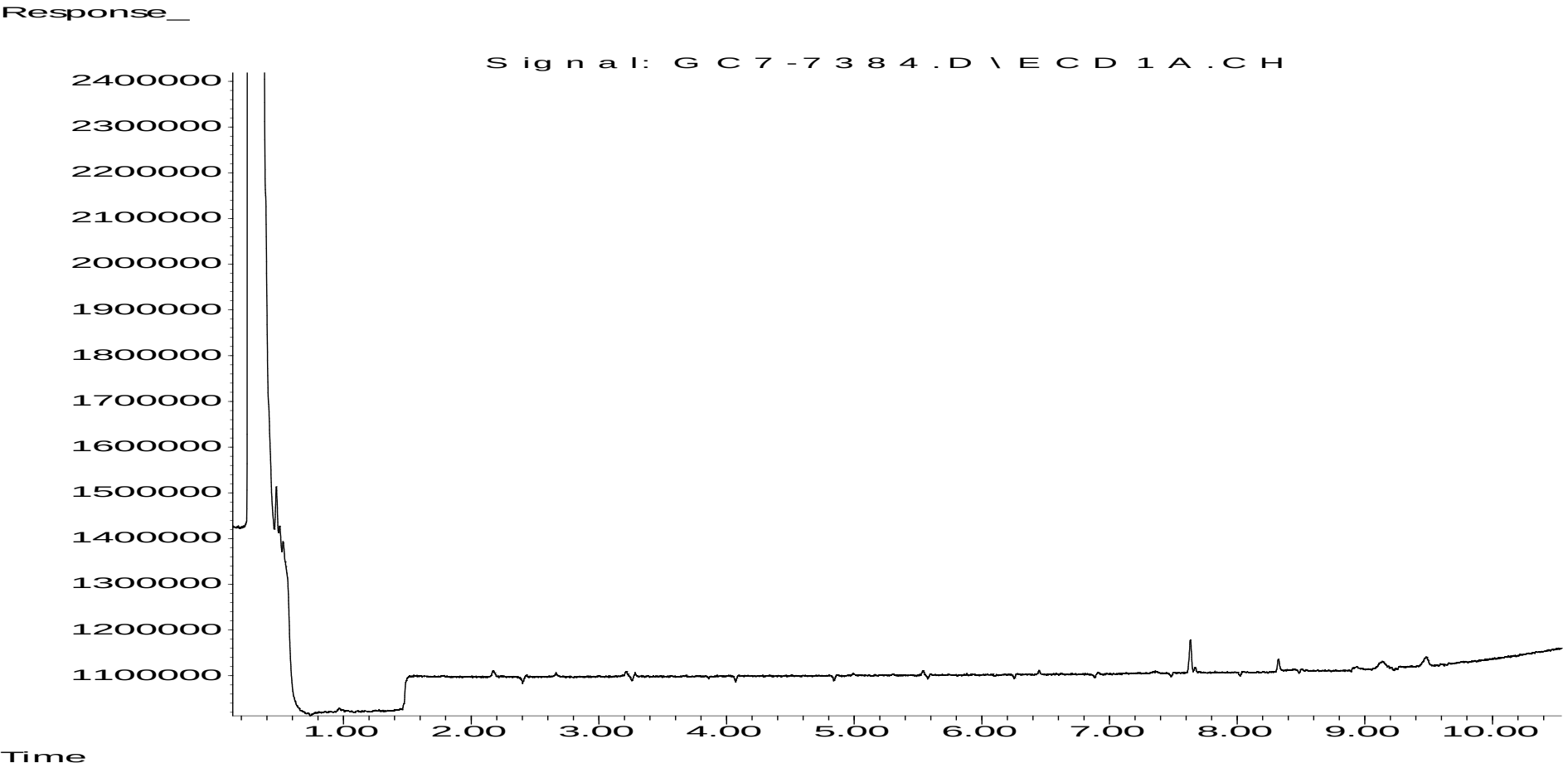
**Problem Resolution**:
Agilent Non-Stick Liner O-Ring
p/n 5188-5365, 10PK

# Contaminated Vial Inserts, New, Bag 1 and Bag 2



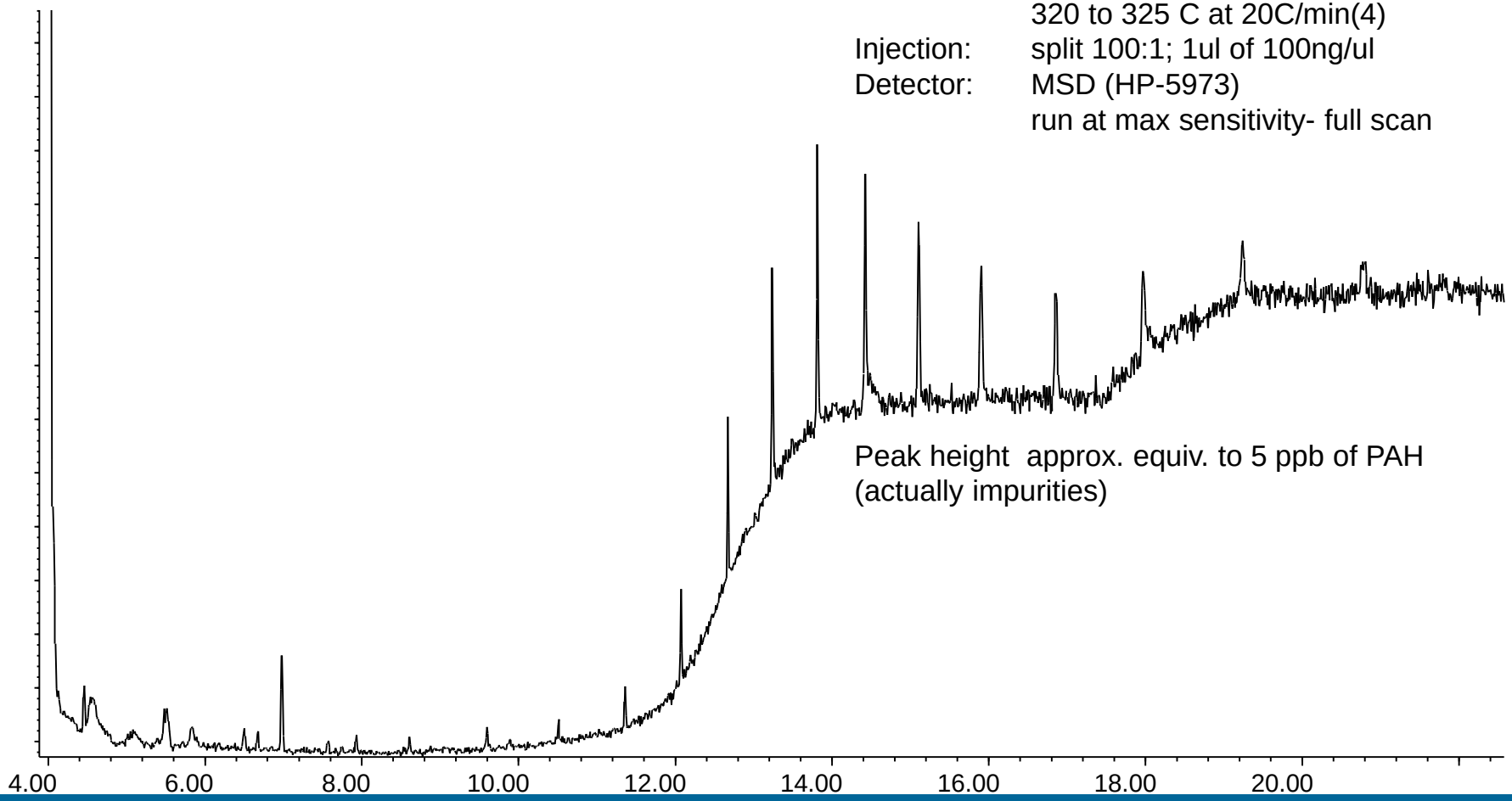
# Hexane Rinsed Insert



# Septa Bleed vs Column Bleed (MSD)

## Source of peaks from outside of the column

| | |
|---|---|
| Columns: | HP 5MS<br>30mx0.25mmx0.25um |
| Oven: | 80 to 160C at 25 C/min,<br>160 to 320 C at 3 C/min(4),<br>320 to 325 C at 20C/min(4) |
| Injection: | split 100:1; 1ul of 100ng/ul |
| Detector: | MSD (HP-5973)<br>run at max sensitivity- full scan |



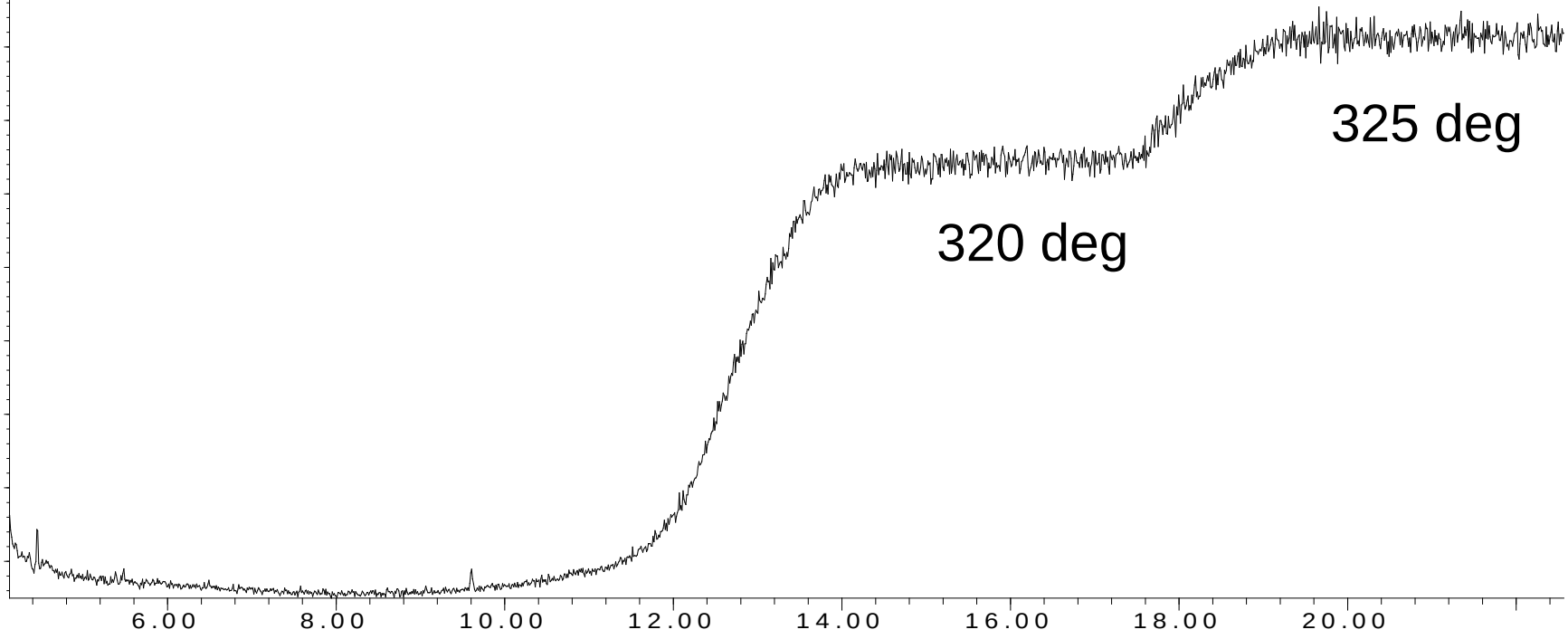

Peak height approx. equiv. to 5 ppb of PAH (actually impurities)

# Septa Bleed vs Column Bleed (MSD)

Source of peaks from outside of the column *ELIMINATED*

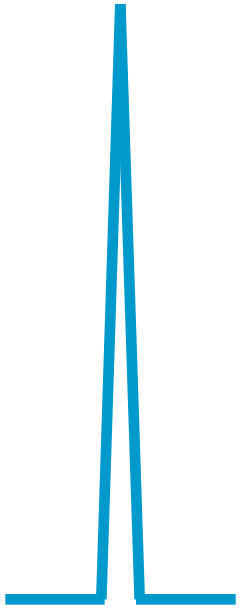
| | |
|---|---|
| Columns: | HP 5 MS<br>30mx0.25mmx0.25um |
| Oven: | 80 to160 C at 25 C/min,<br>160 to 320 C at 3 C/min(4)<br>320 to 325 C at 20C/min(4) |
| Injection: | split 100:1; 1ul of 100ng/ul |
| Detector: | MSD (HP-5973)<br>run at max sensitivity- full scan |

**New Septa Installed**
HP Advanced Green Septa
P/N 5183-4594 ( 11 mm)



# Peak Tailing

**Before**



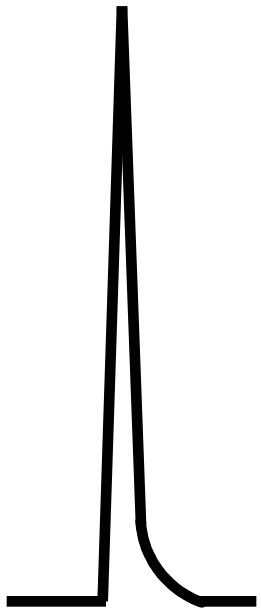
INJECTOR or COLUMN is Active

-Reversible adsorption of active compounds

(-OH, -NH, -SH)

FLOW problem
- dead volume, obstruction, poor installation, or severe column contamination

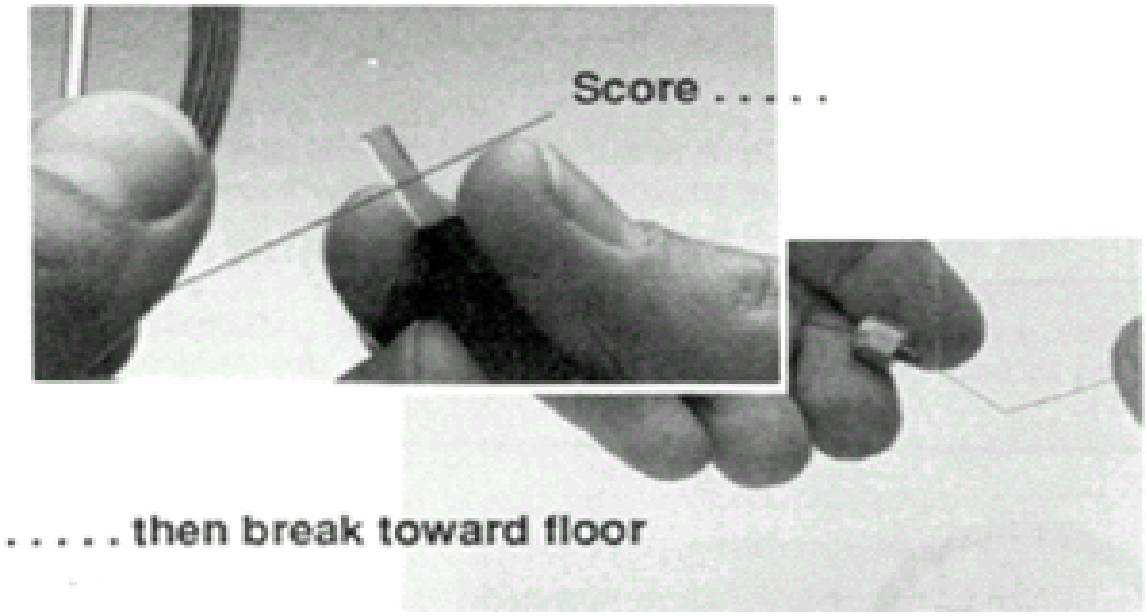
**After**

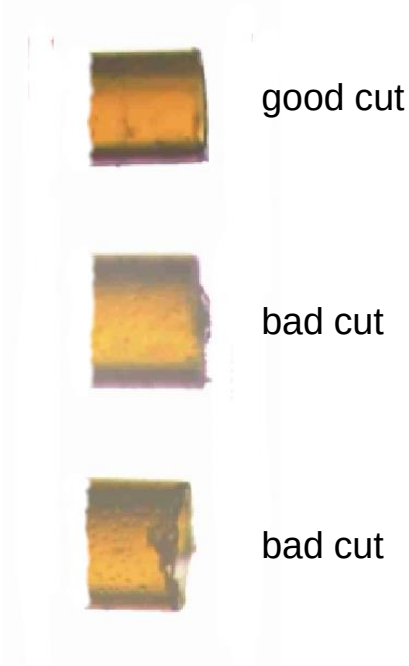


Miscellaneous – temperature issues for late eluters, overloading of PLOT columns, co-elution, polarity mismatch between phase, solute or solvent, and some compounds always tail

*Tip = Inject a light hydrocarbon, should not tail unless flow path problem.

# Flow Path Matters



good cut

bad cut

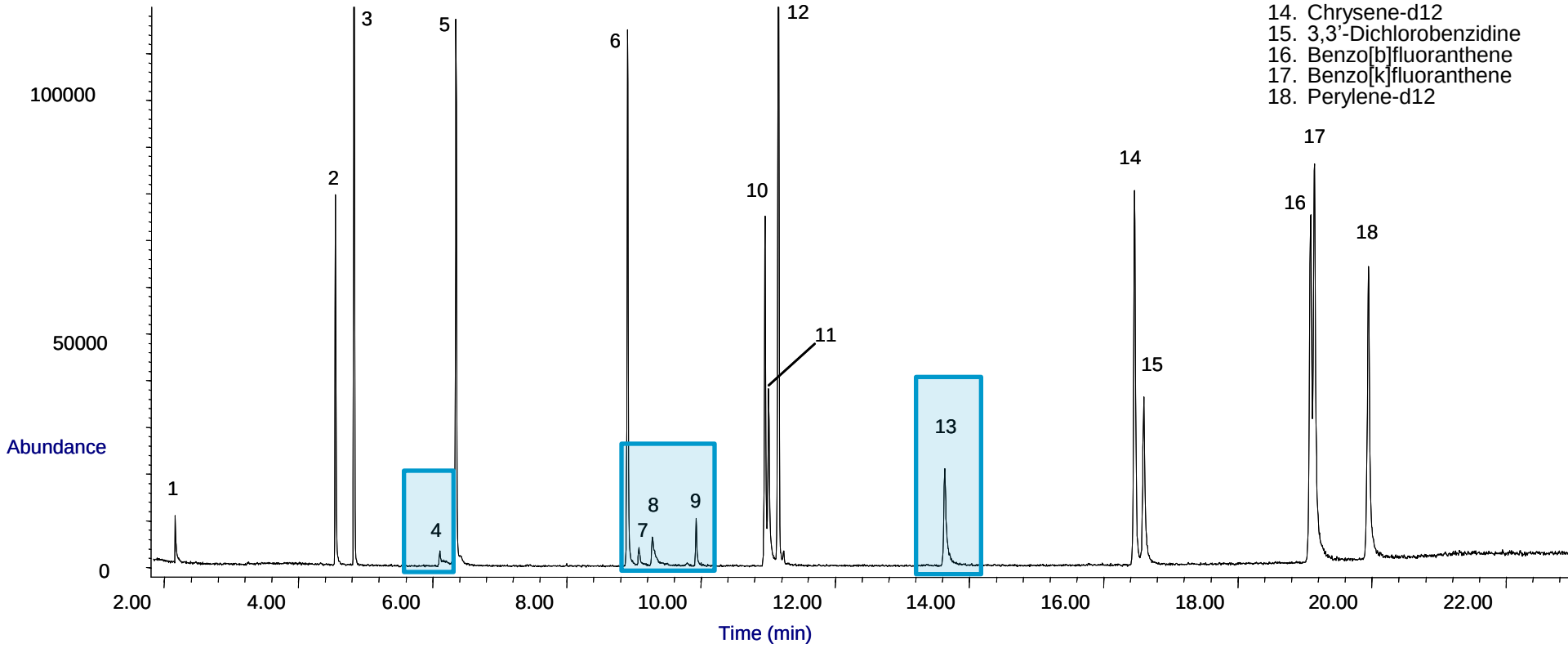
bad cut

A flat,  90° square cut  will be optimal for all connections

# Symptom – Tailing of Active Compounds

Sample: 0.5 ng on column loading with ISTD
Column: 20m 0.18mm 0.18µm
Carrier: Helium 37cm/sec, Ramped flow; 0.7ml/min (0.1min) to 1.3ml/min (15ml/min$^2$)
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: 0.5µl, Splitless, 280°C, purge flow 30ml/min at 0.75 min
MSD: Transfer Line 290°C, Source 300°C, Quad 180°C

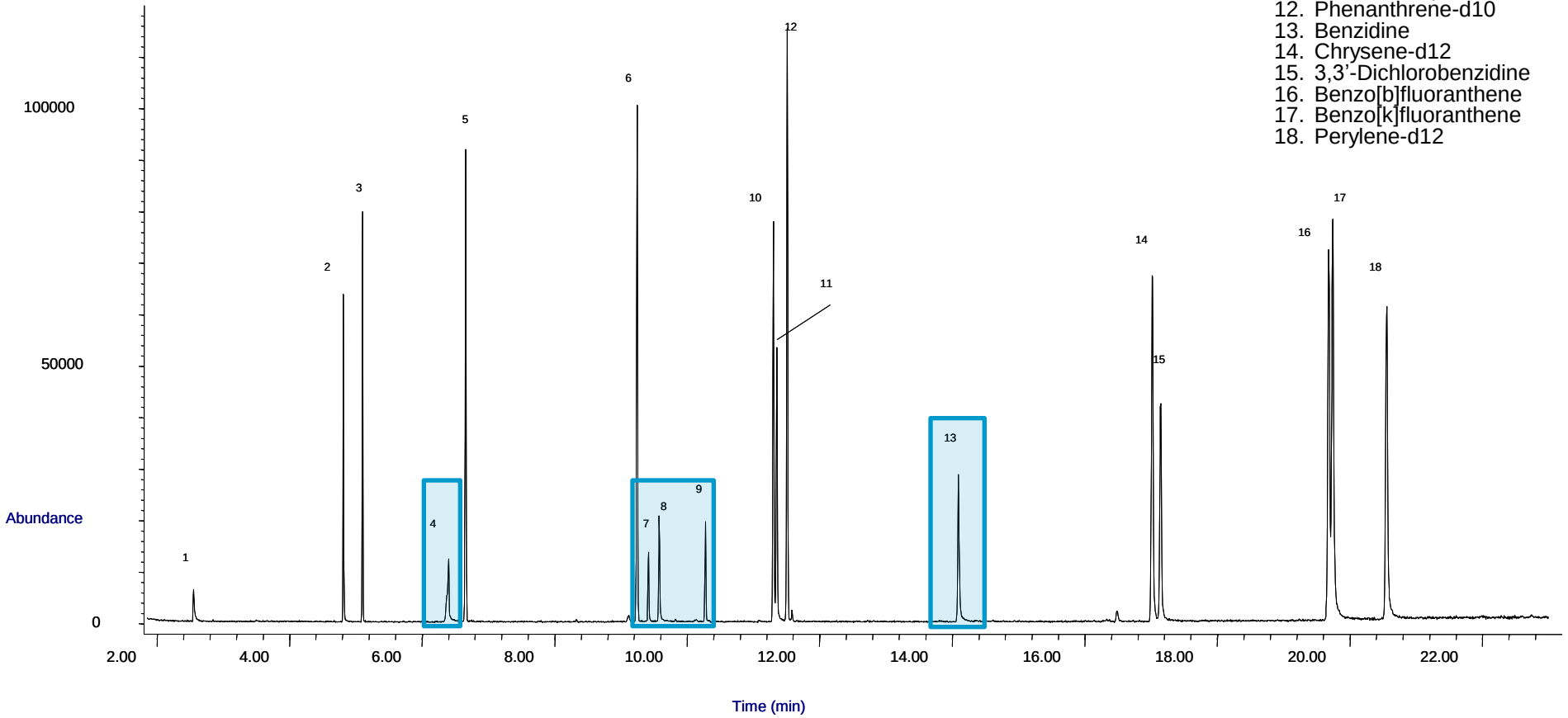
1. n-Nitrosodimethylamine
2. Aniline
3. 1,4-Dichlorobenzene-d4
4. Benzoic Acid
5. Naphthalene-d8
6. Acenaphthene-d10
7. 2,4-Dinitrophenol
8. 4-Nitrophenol
9. 2-Me-4,6-dinitrophenol
10. 4-Aminobiphenyl
11. Pentachlorophenol
12. Phenanthrene-d10
13. Benzidine
14. Chrysene-d12
15. 3,3'-Dichlorobenzidine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



# Solution – Ultra Inert GC Column

| | |
|---|---|
| Sample: | 0.5 ng on column loading with ISTD |
| Column: | 20m 0.18mm 0.18µm |
| Carrier: | Helium 37cm/sec, Ramped flow; 0.7ml/min (0.1min) to 1.3ml/min (15ml/min$^2$) |
| 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: | 0.5µl, 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






Agilent Technologies

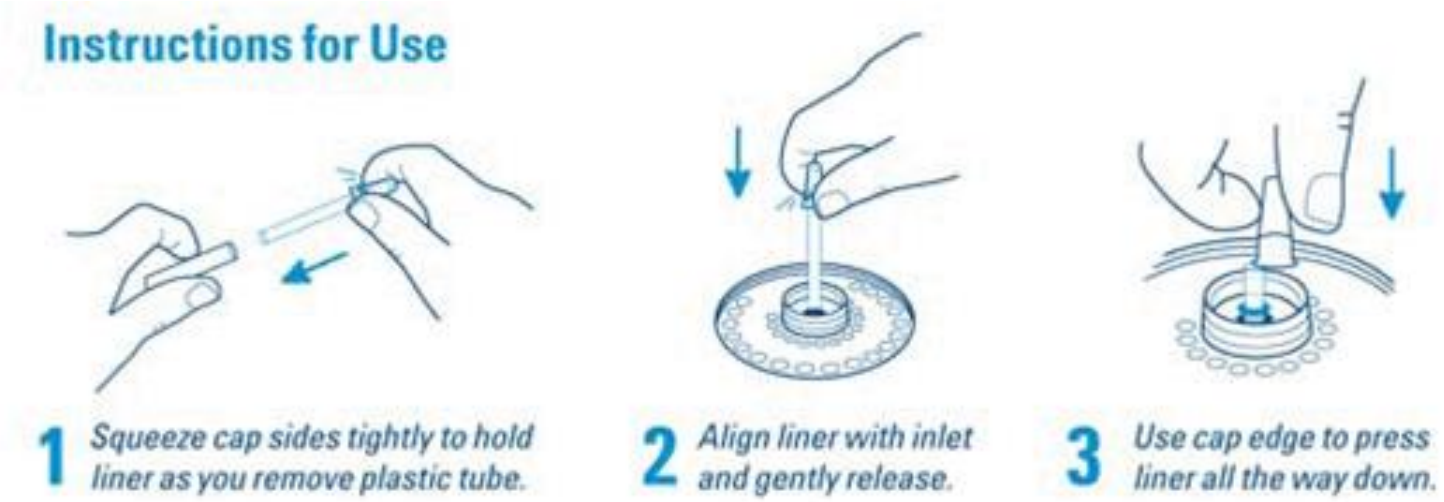
# Agilent Ultra Inert Liners


## Touchless packaging

Easy installation of new, clean liner

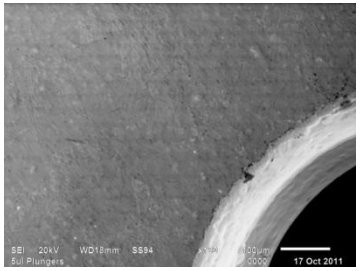
without risk of contamination from touching

Includes non-stick plasma treated O-ring

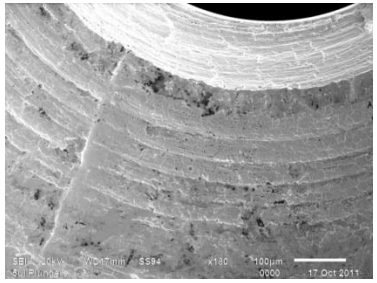


# Agilent UI Gold Seal: Deactivated Gold Surface

- Soft gold plating is essential for proper sealing
- Ultra Inert chemistry blocks active sites (gold is NOT inert)
- Smooth surface doesn't leak



Agilent MIM seal



Competitor's machined seal

*Reliable ppb and ppt measurements require attention to the little things!*

# New Product: Agilent Inert Inlet

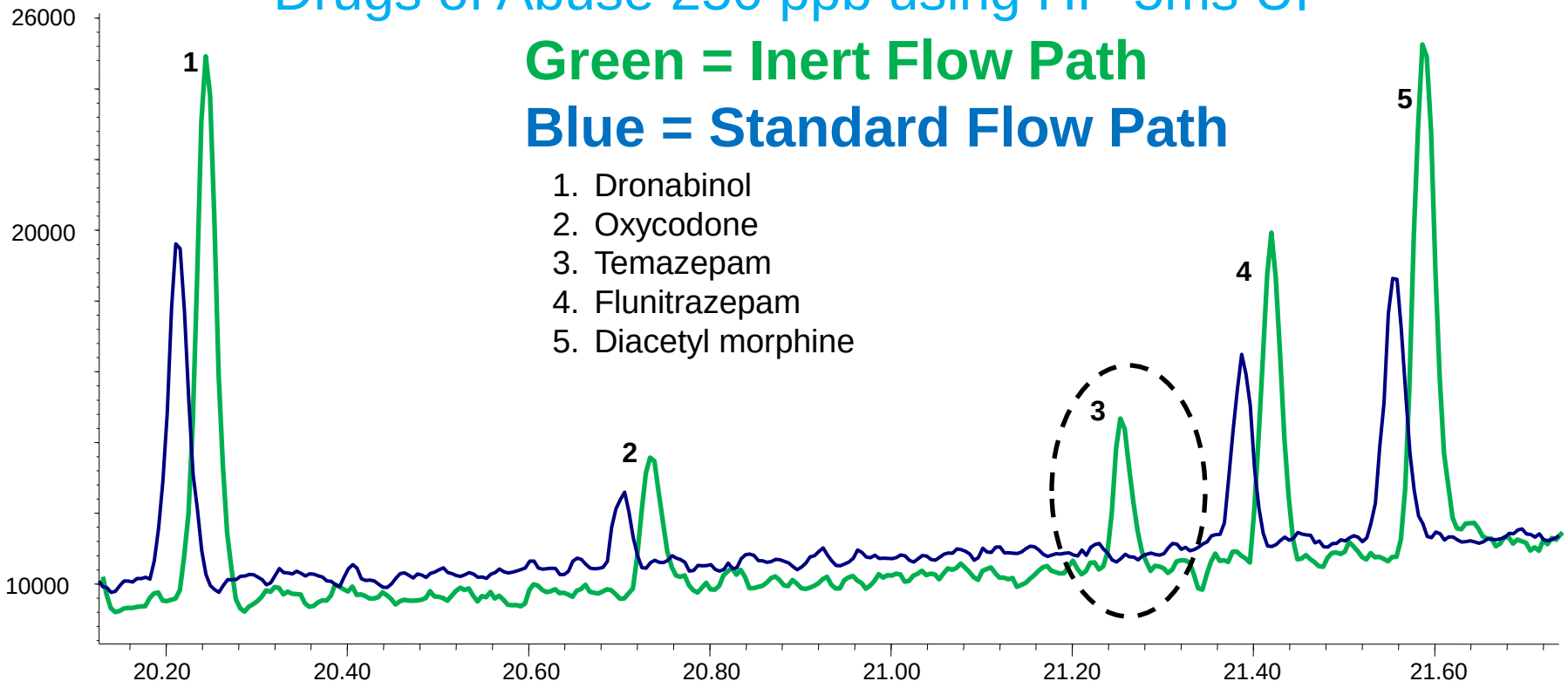
***UltiMetal Plus*** treatment
creates inert surface 7890 inlet weldment & shell

- Limits adsorption/degradation of active analytes
- Optimum for Trace GC/MS and GC-ECD pesticide analyses

***Agilent's proven proprietary Ultimetal Plus surface treatment***

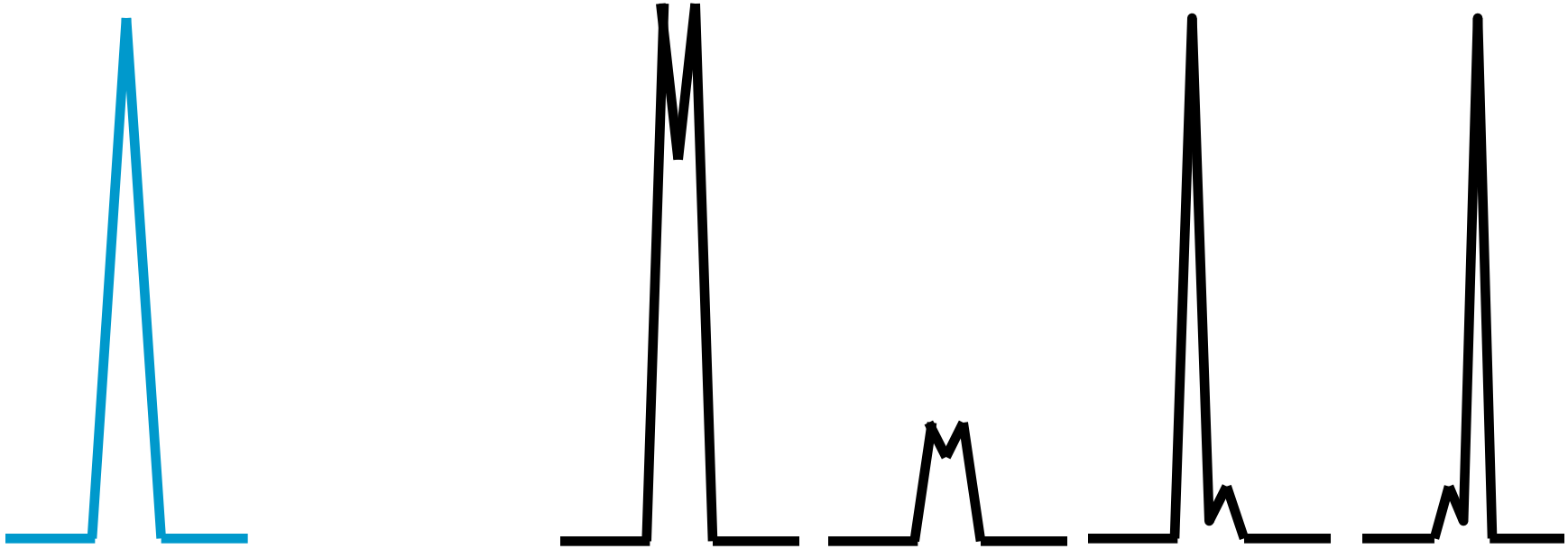
# Ultra UltiMetal Plus Inert Inlet

## Drugs of Abuse 250 ppb using HP-5ms UI




| | |
|---|---|
| Column: | Agilent J&W HP-5ms UI 30 m x 0.25 mm x 0.25 um |
| Oven: | 100°C 4 min hold, 10°/min to 280°C, 6 °/min to 300°C (4.67 min hold), |
| Carrier : | Helium 52.7 cm/s (2 mL/min) set at 100°C, EPC-Constant Flow |
| Inlet: | Pulsed Splitless 35 PSI pulse until 0.73 min, 0.75 min purge 50 ml/min, gas saver 20, ml/min at 2 min |
| Inlet liner: | Ultra Inert with wool / Standard single taper liner with wool (p/n 5190-3165) |
| Gold Seal: | UI Gold Seal / Standard gold seal |
| Detector: | MSD Scan mode 40 t0 450 m/z, 230 °C source temp, 150 °C Quad temp, 310 °C transfer line |

# Split Peaks



INJECTOR (poor sample introduction)

-Injecting the sample twice (some how?)

-Mixed sample solvent (polarity difference)

-Sample in syringe needle (manual inject)

INJECTOR (activity)

-Breakdown (not really a split peak, 2 peaks)

-Sample degradation in injector

VOLATILITY

High boilers dropping out on Cold Spots

-Transfer line temps

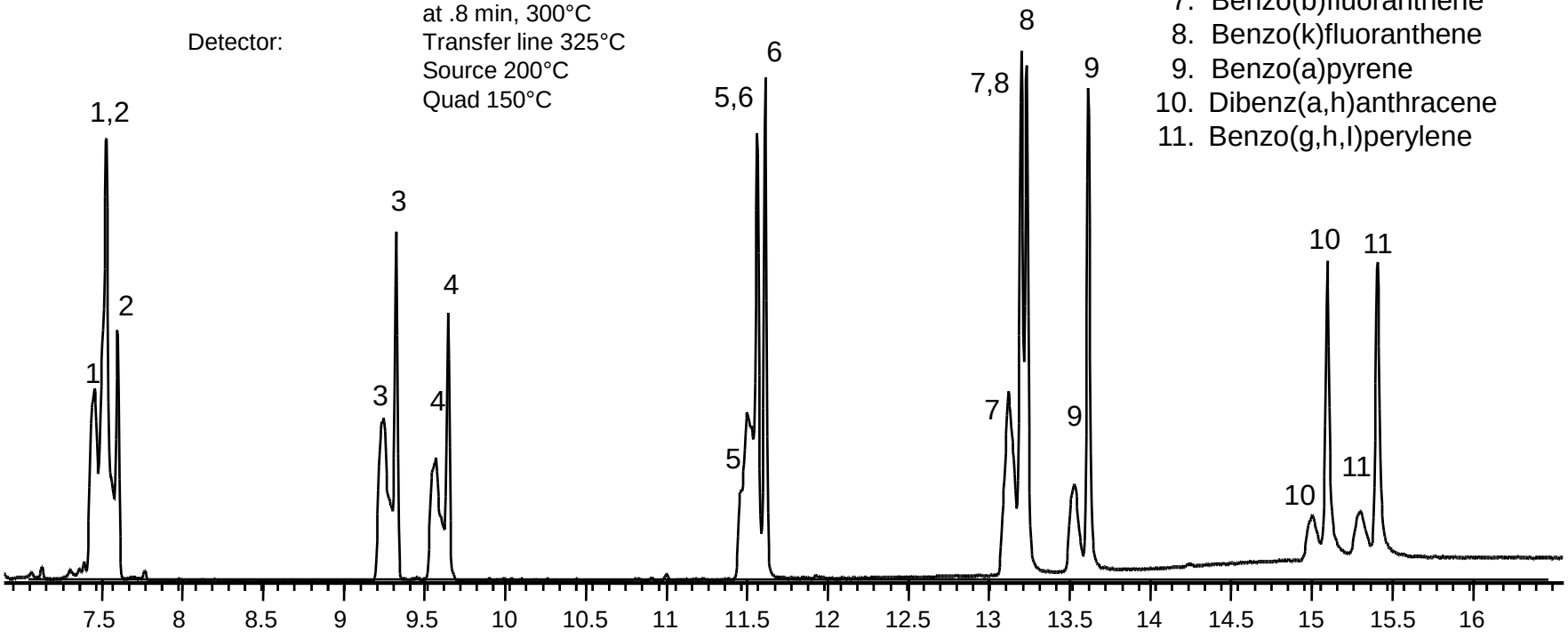
-Unions or fittings not tracking column temp

# PAH's in Toluene

## Splitting Peaks Immediately After Installation

| | |
|---|---|
| **Column:** | **Agilent J&W DB-1ms** |
| | **30 m x 0.25 mm I.D., 0.25 µm** |
| **P/N:** | **123-1522** |
| Test Temperature: | 80°C for 0.8 min |
| | 80-325°C at 15°/min |
| | 325°C for 2 min |
| Carrier Gas: | Helium at 40 cm/sec |
| Injector: | Splitless, purge on |
| | at .8 min, 300°C |
| Detector: | Transfer line 325°C |
| | Source 200°C |
| | Quad 150°C |

1. Phenanthrene
2. Anthracene
3. Fluoranthene
4. Pyrene
5. Benzo(a)anthracene
6. Chrysene
7. Benzo(b)fluoranthene
8. Benzo(k)fluoranthene
9. Benzo(a)pyrene
10. Dibenz(a,h)anthracene
11. Benzo(g,h,I)perylene



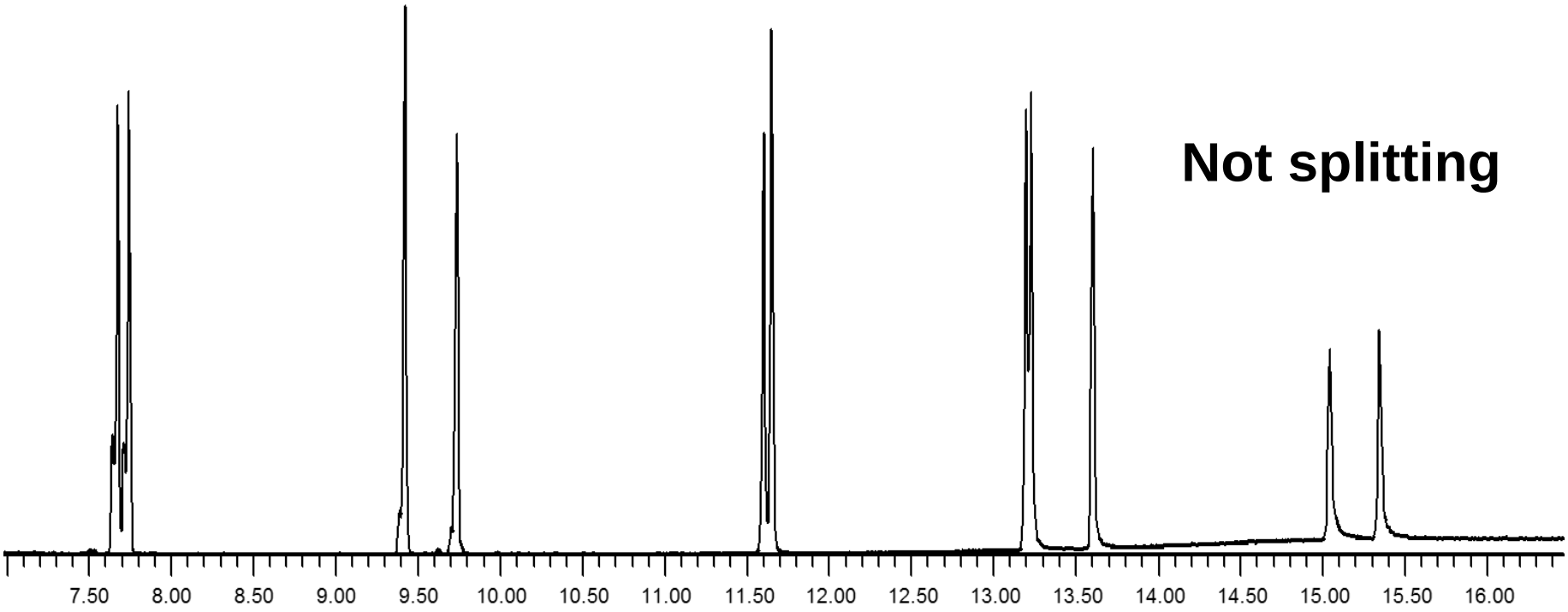



Agilent Technologies

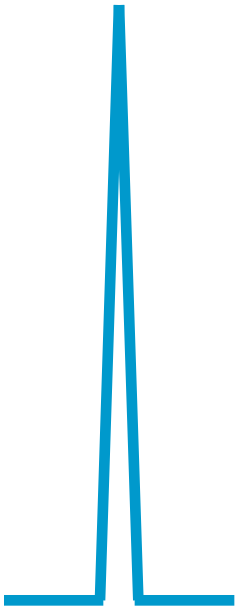
# Reinstalled the Column

**Distance was off - previously installed too far**

**Improved but still splitting**



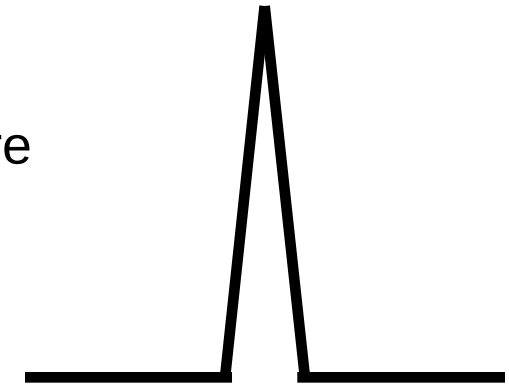
# Broad Peaks



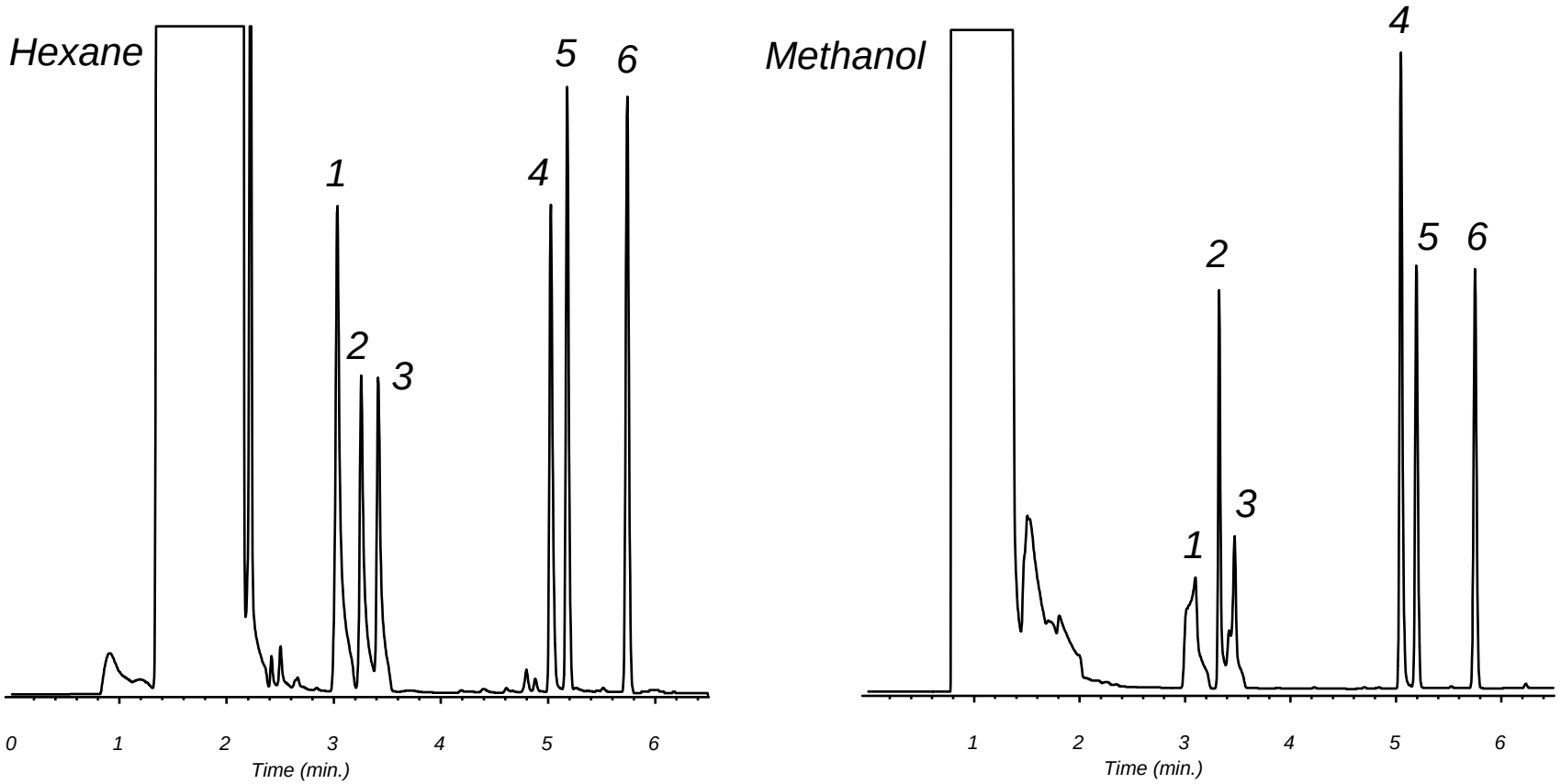
INJECTOR
-Poor Installation
-Change in settings (temps/flows)
-Poor sample focusing
-Large change in sample concentration

FLOW
-Change in gas velocity
-Constant Flow vs Constant Pressure

COLUMN
-Contamination
-Damaged/old stationary phase
-Reverse Solvent Effect



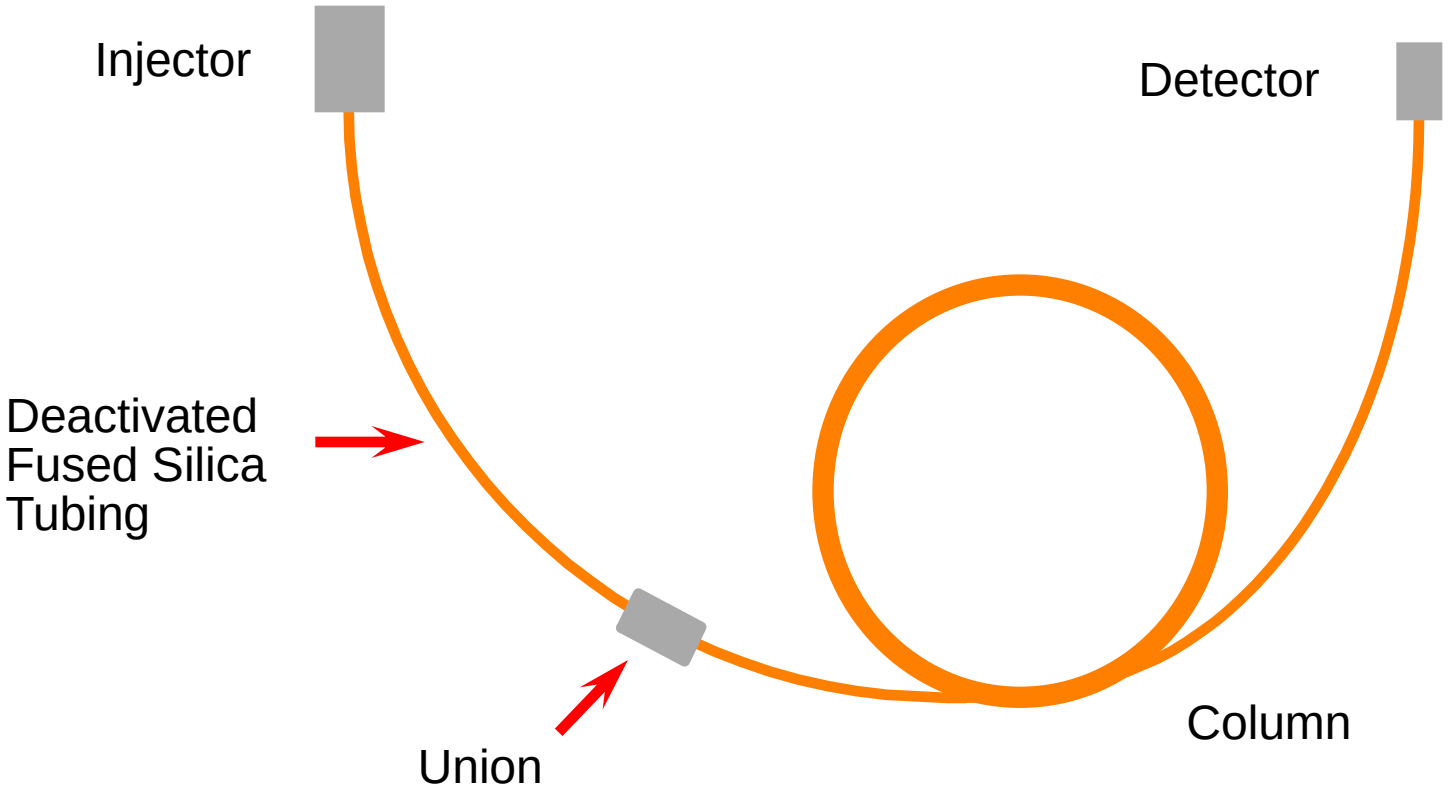
# Splitless Injection
## Reverse Solvent Effect




**DB-1, 15 m x 0.25 mm I.D., 0.25 µm**
**50°C for 1 min, 50-210°C at 20°/min;  Helium at 30 cm/sec**
**1. 1,3-DCP  2. 3-hexanol  3. butyl acetate  4. 1-heptanol  5. 3-octanone  6. 1,2-dichlorobenzene**

# Retention Gap

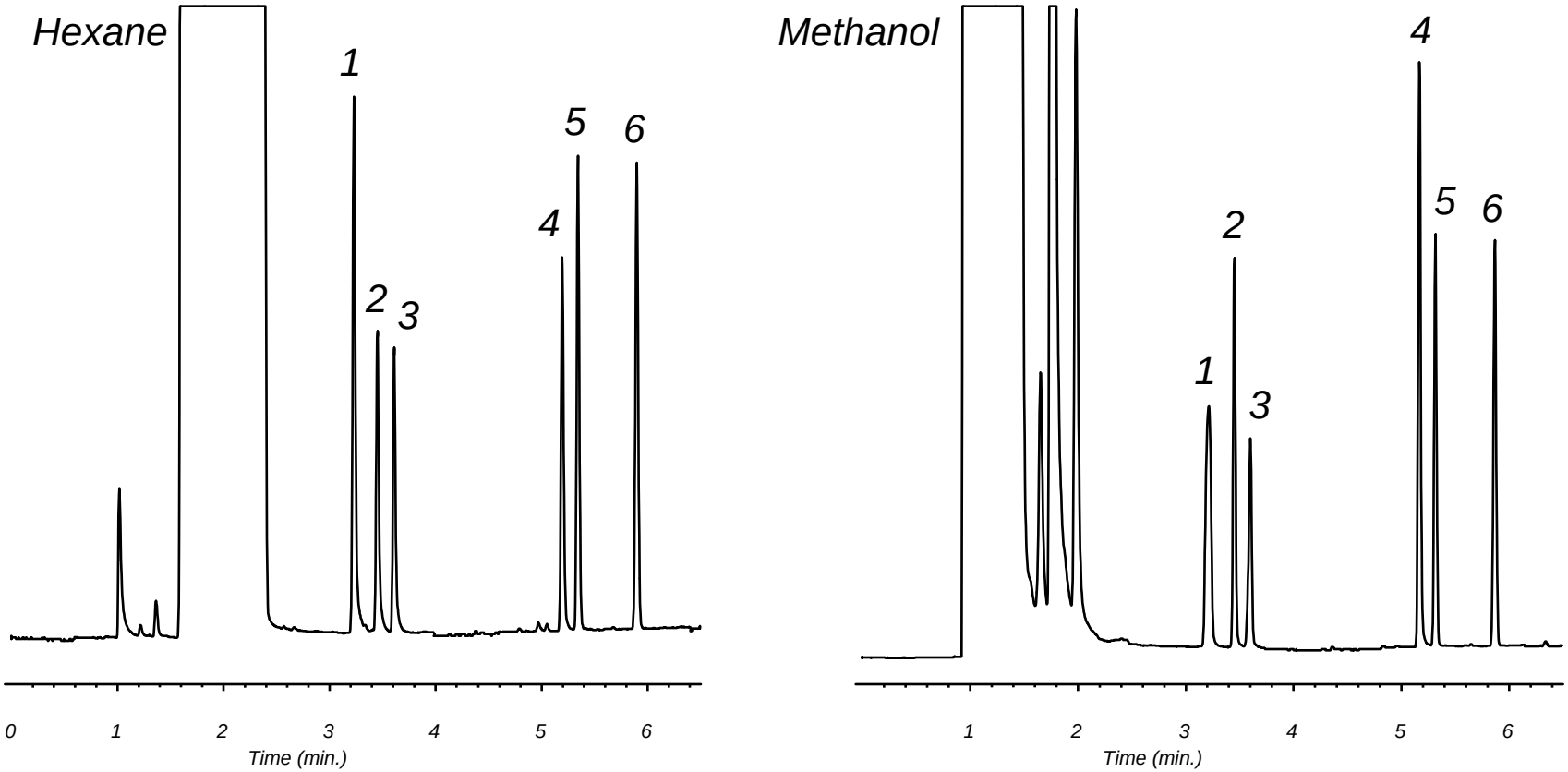
## Also Called A Guard Column




Usually 2-10 meters long and same diameter as the column (or larger if needed)

# Fixing the Reverse Solvent Effect

## 3 m x 0.25 mm I.D. Retention Gap

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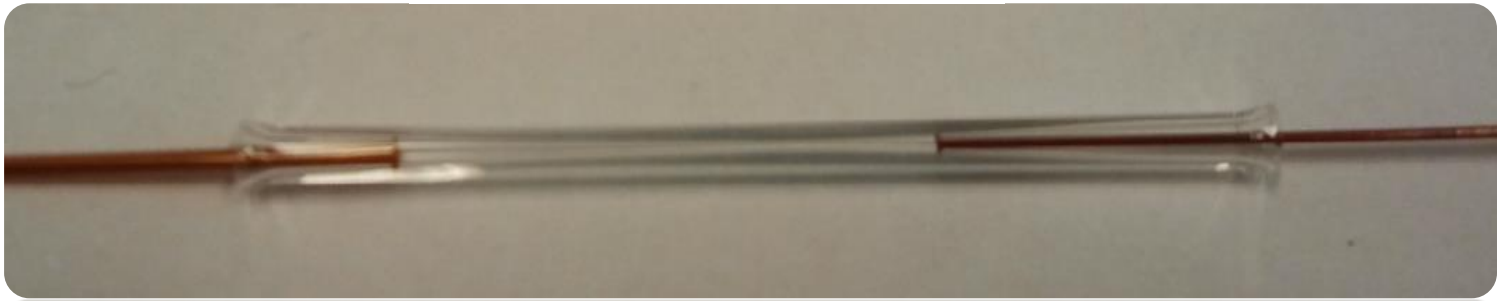
# Press Fit Connectors

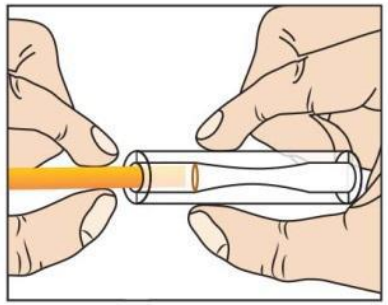
**Competitive Press-Tight**

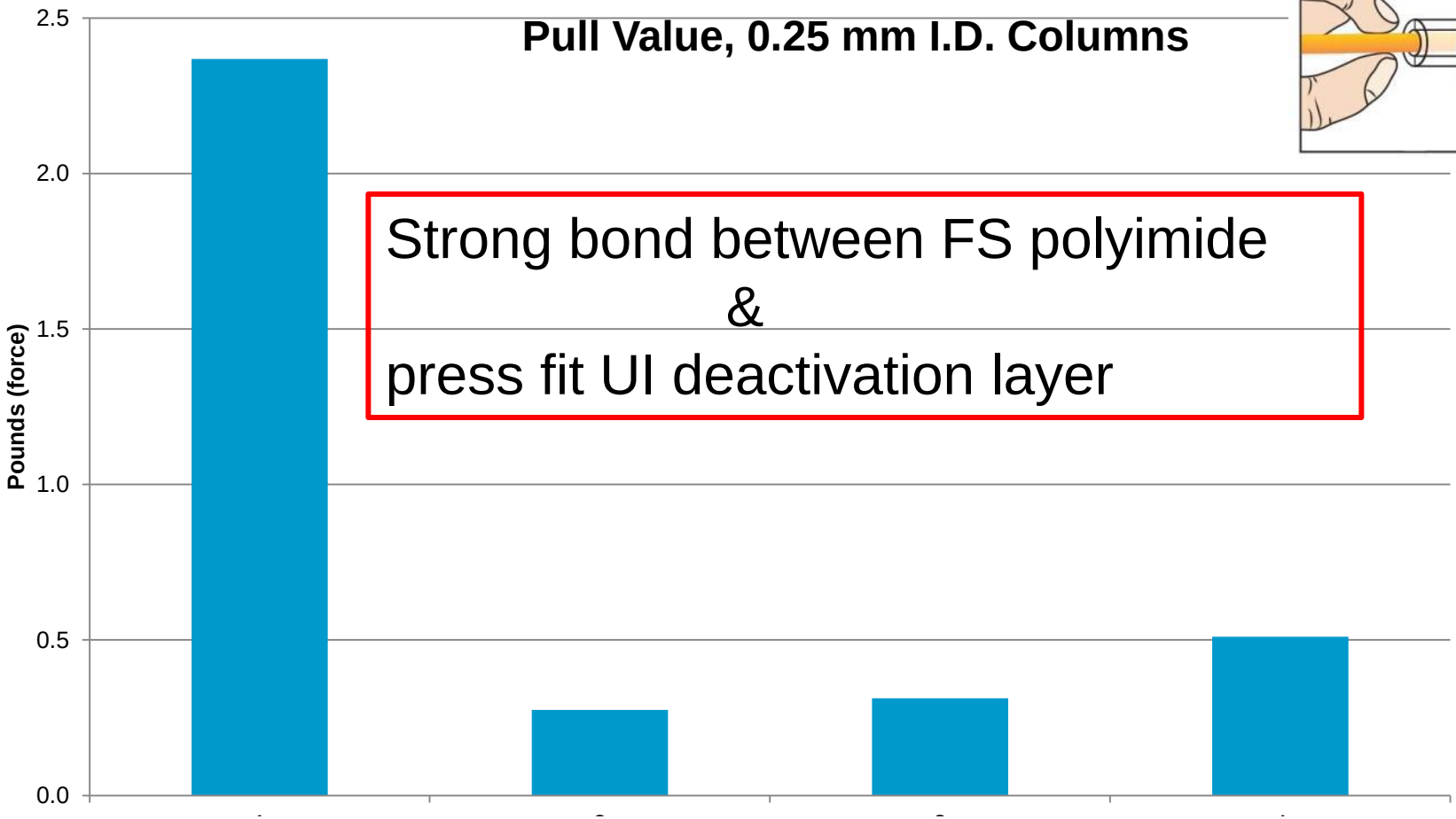
**Agilent Ultra Inert Press Fit**



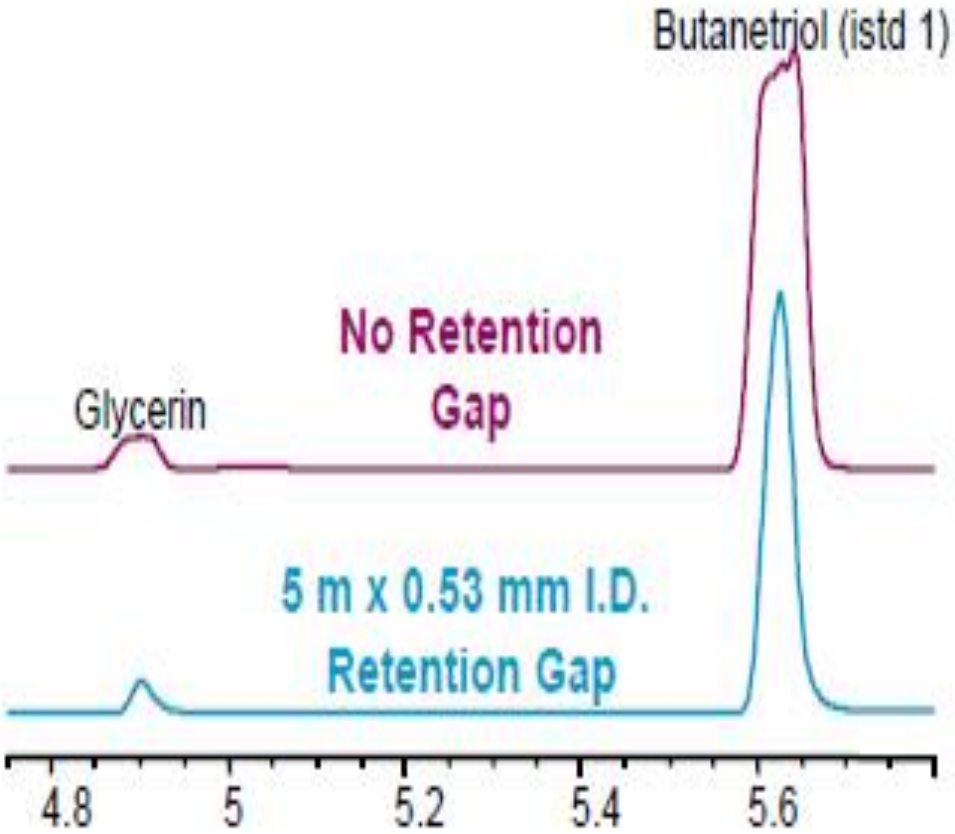
Inertness, seal and transparency are key parameters

# Maximum Force Required to “pull apart”

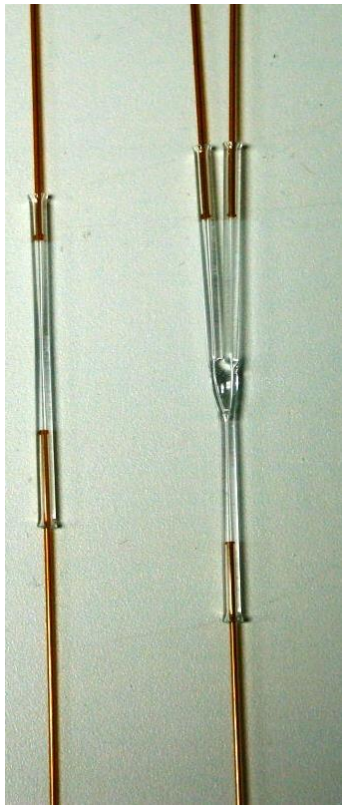




# Ultimate Union and Retention Gap

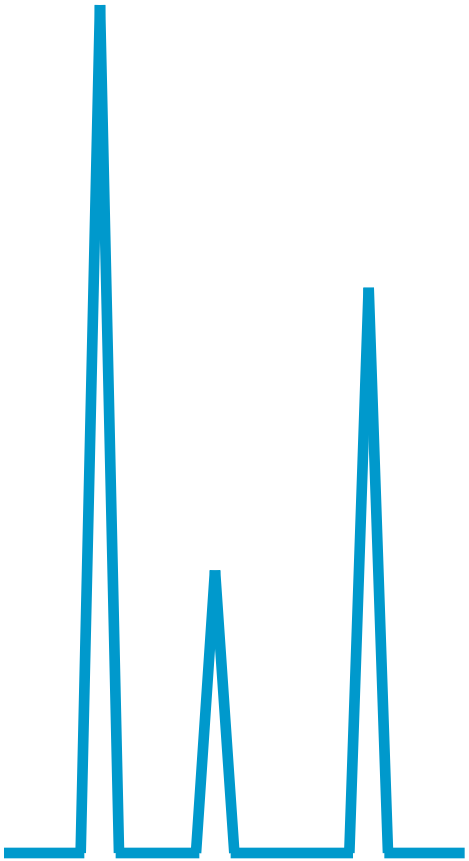
# Two Connection Options from Agilent



Ultra Inert Press Fit Connectors

Capillary Flow Technology

# No Peaks



DETECTOR (not on or not operational)

INJECTOR (not working)

-Plugged syringe/plunger not moving

-Wrong injector (or detector)

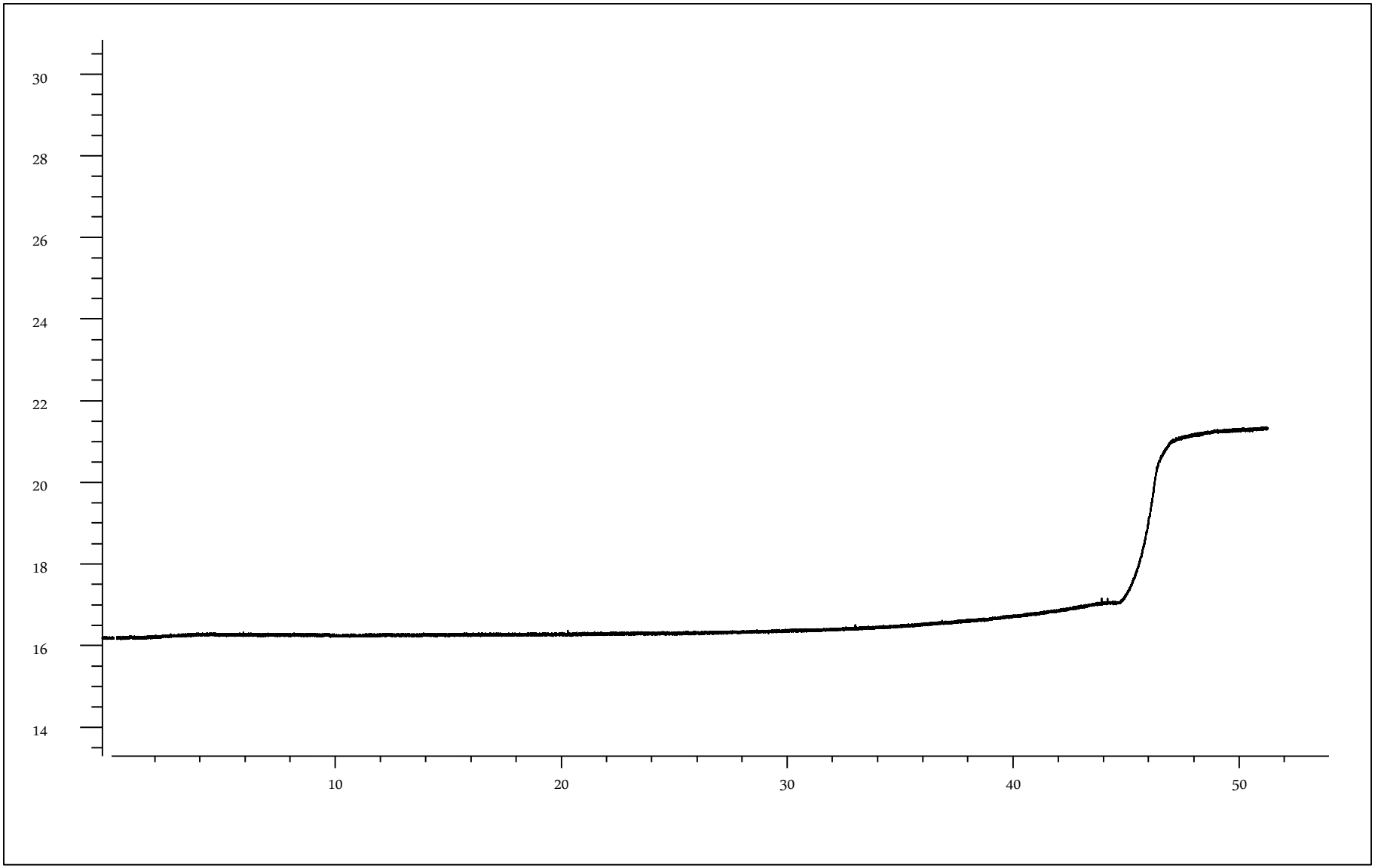
-Huge leak/no carrier gas flow (older systems)

NOT the COLUMN Unless…

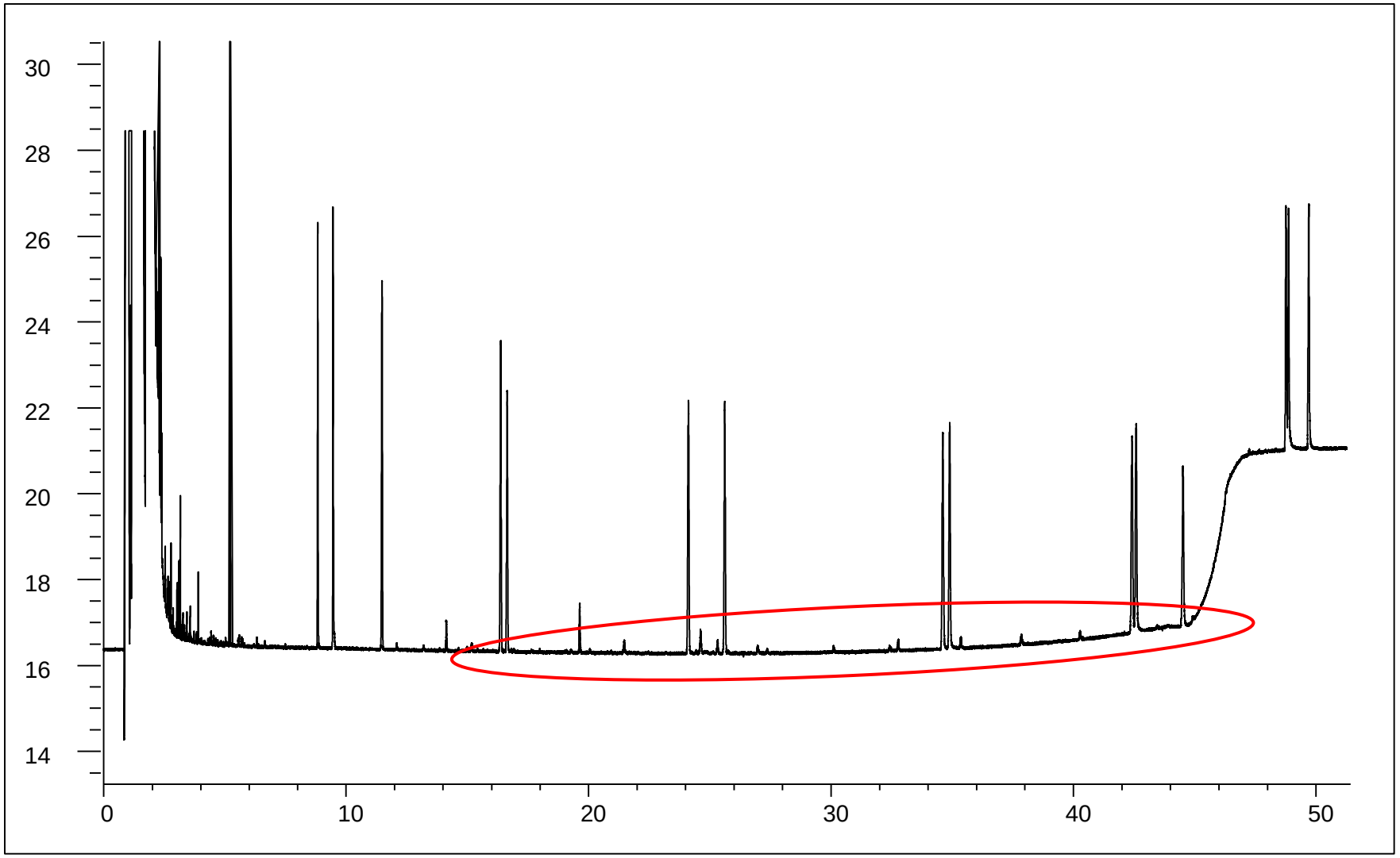
-Broken column

-No column

# Symptom – No Peaks

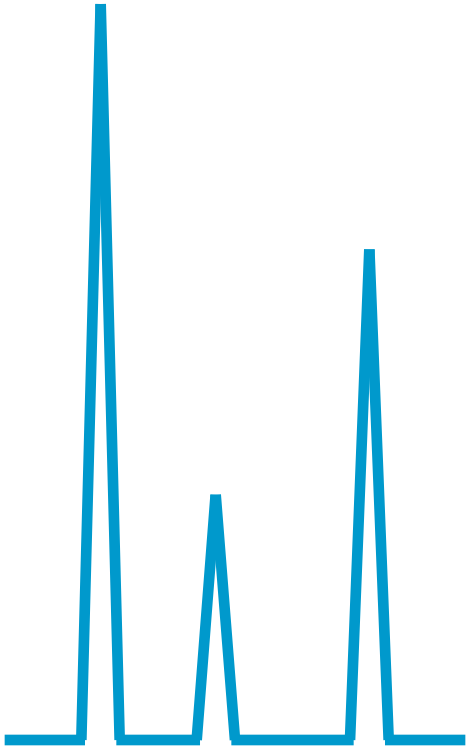


# Solution - Unplugged Syringe



# Peak Response

## All Change in Size



INJECTOR

-Leaky syringe

-Split ratio set incorrectly

-Wrong purge activation time

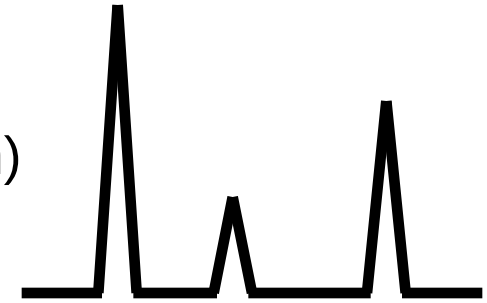
-Septum purge flow too high

-Injector temperature too low*

DETECTOR (response problem)

-Settings or flows changed

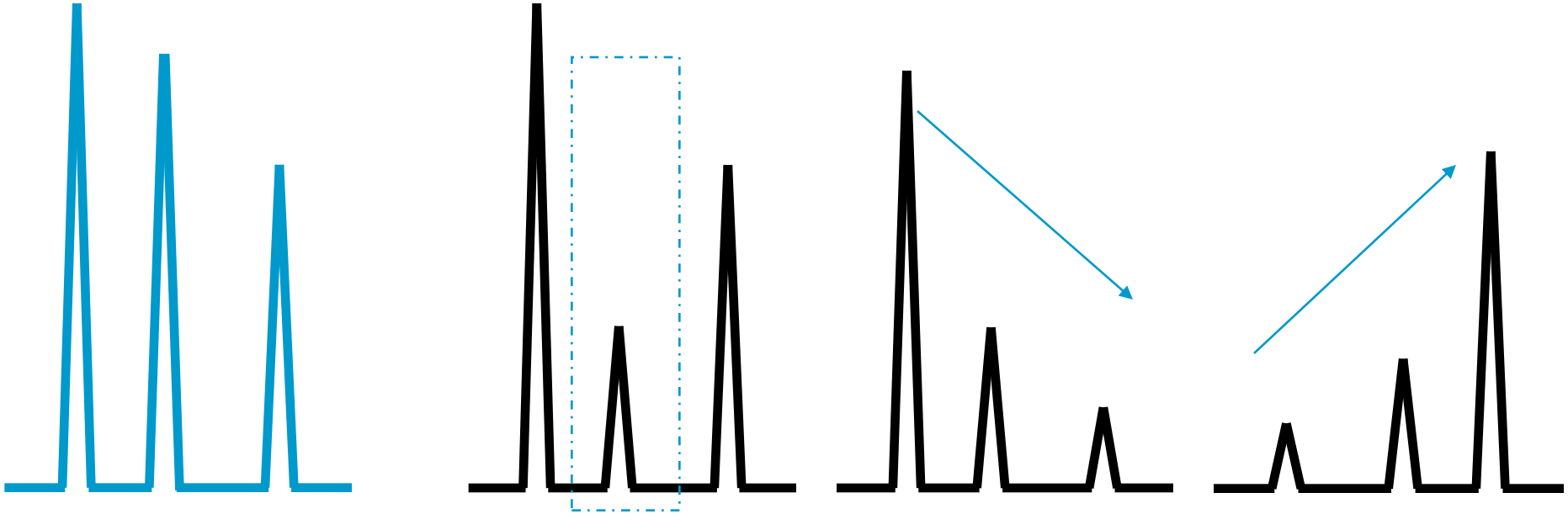
-Electronics failing



*Tip = Ask is it all of them or some of them, if all then injector or detector

# Peak Response

## Some Change in Size



INJECTOR or COLUMN is active/contaminated

-Irreversible adsorption of active compounds (-OH, -NH, -SH)

-Decomposition of sample

-Temperature Change – Discrimination

-Evaporation from sample

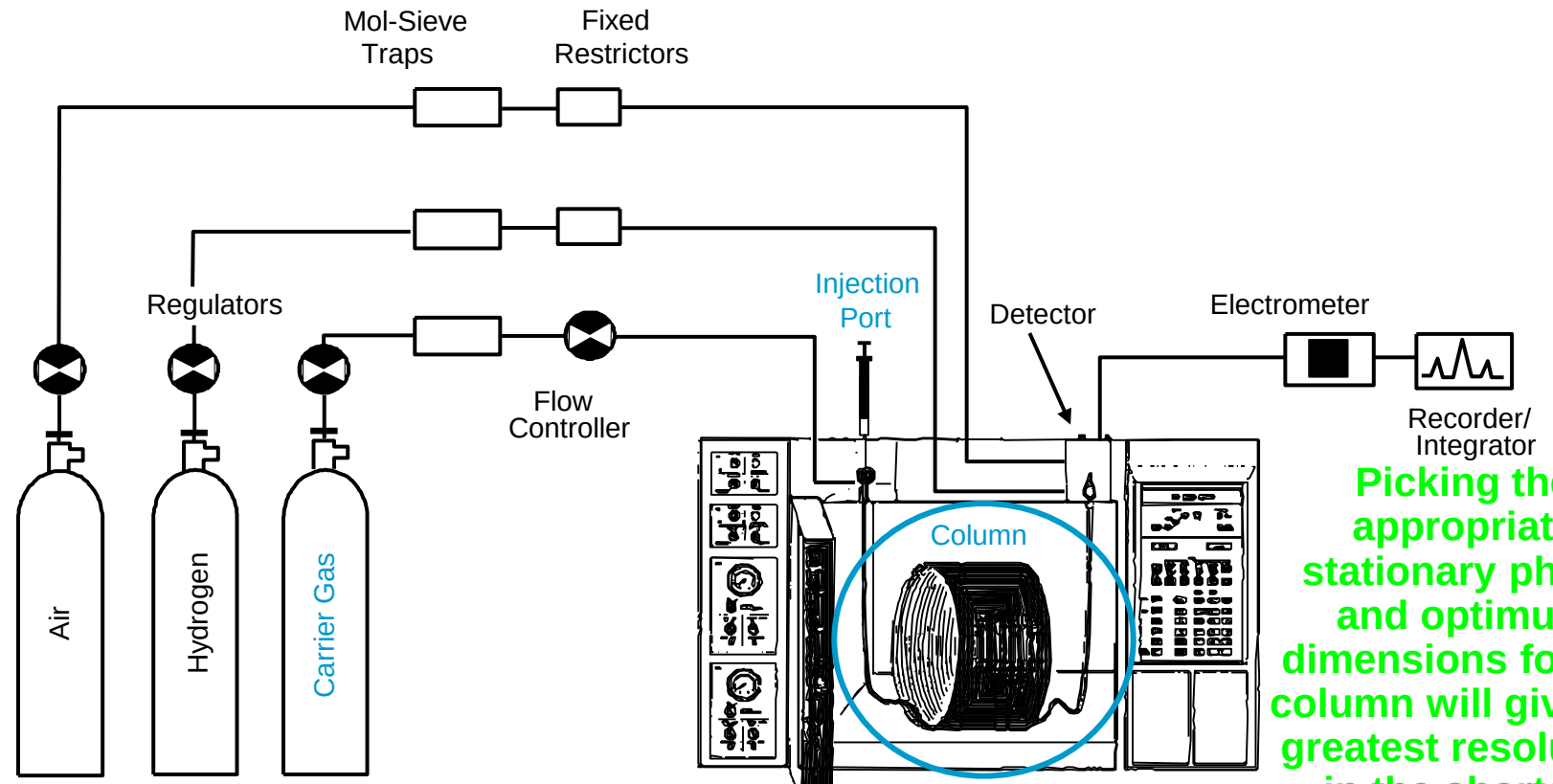
*Tip = If only some change, then ask which ones? If active compounds then activity. If tracks volatility then cold spots or inlet discrimination.

# Advan 6 C 12.001 ed

# Troubleshoo 22 Ti 47.867 ng

# Part 2 The Rest of the 16 S 32.065 tory

# Typical Gas Chromatographic System

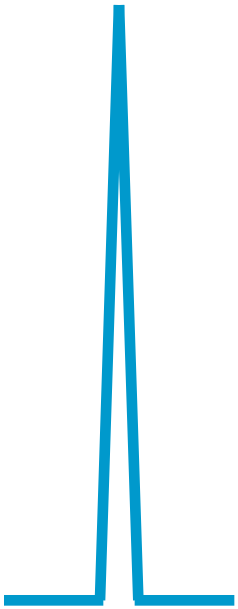



**Cylinders or Generators**

**Picking the appropriate stationary phase and optimum dimensions for the column will give the greatest resolution in the shortest analysis time.**

# Peak Fronting

## Shark Fin Shaped or Just Slight



COLUMN (contaminated)

-Overload (More pronounced with large solute and phase polarity differences)

INJECTOR

-Poor efficiency (flow/temp)

-Column installation

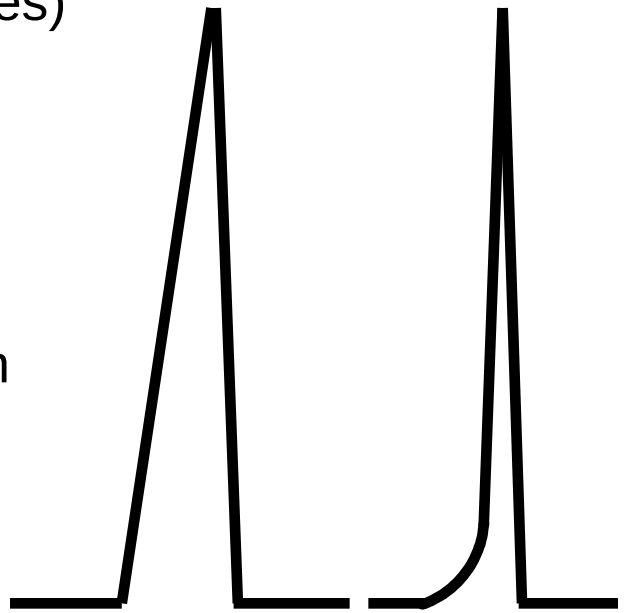
-Compound very soluble in injection solvent (need retention gap)

-Mixed sample solvent

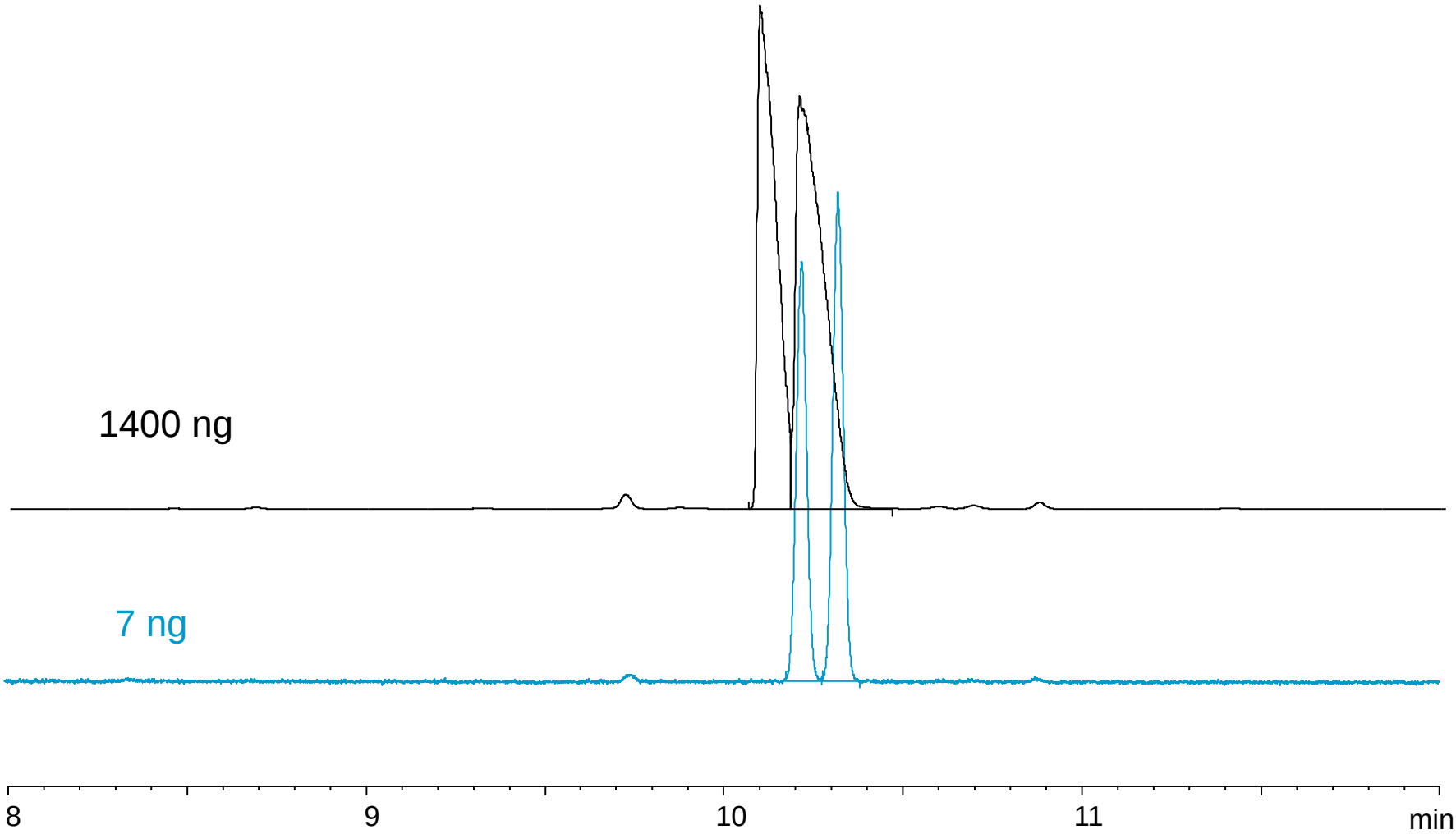
OTHER

-Co-elution

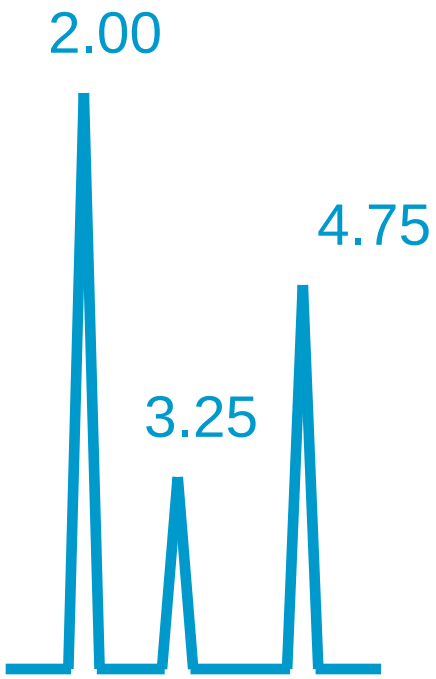
-Breakdown



# Effect of Sample Overload on Retention Time and Peak Shape



# Retention Time Shift




INJECTOR

-Change in injection solvent

-Large change in sample concentration

FLOW

-Leak in the septum

-Change in gas velocity

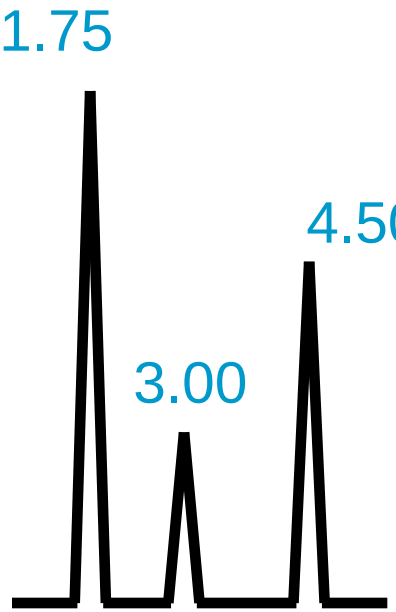
COLUMN

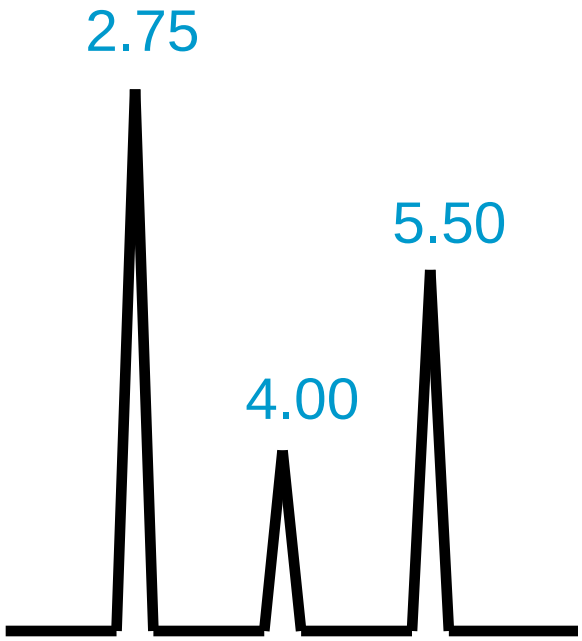
-Contamination

-Damaged stationary phase

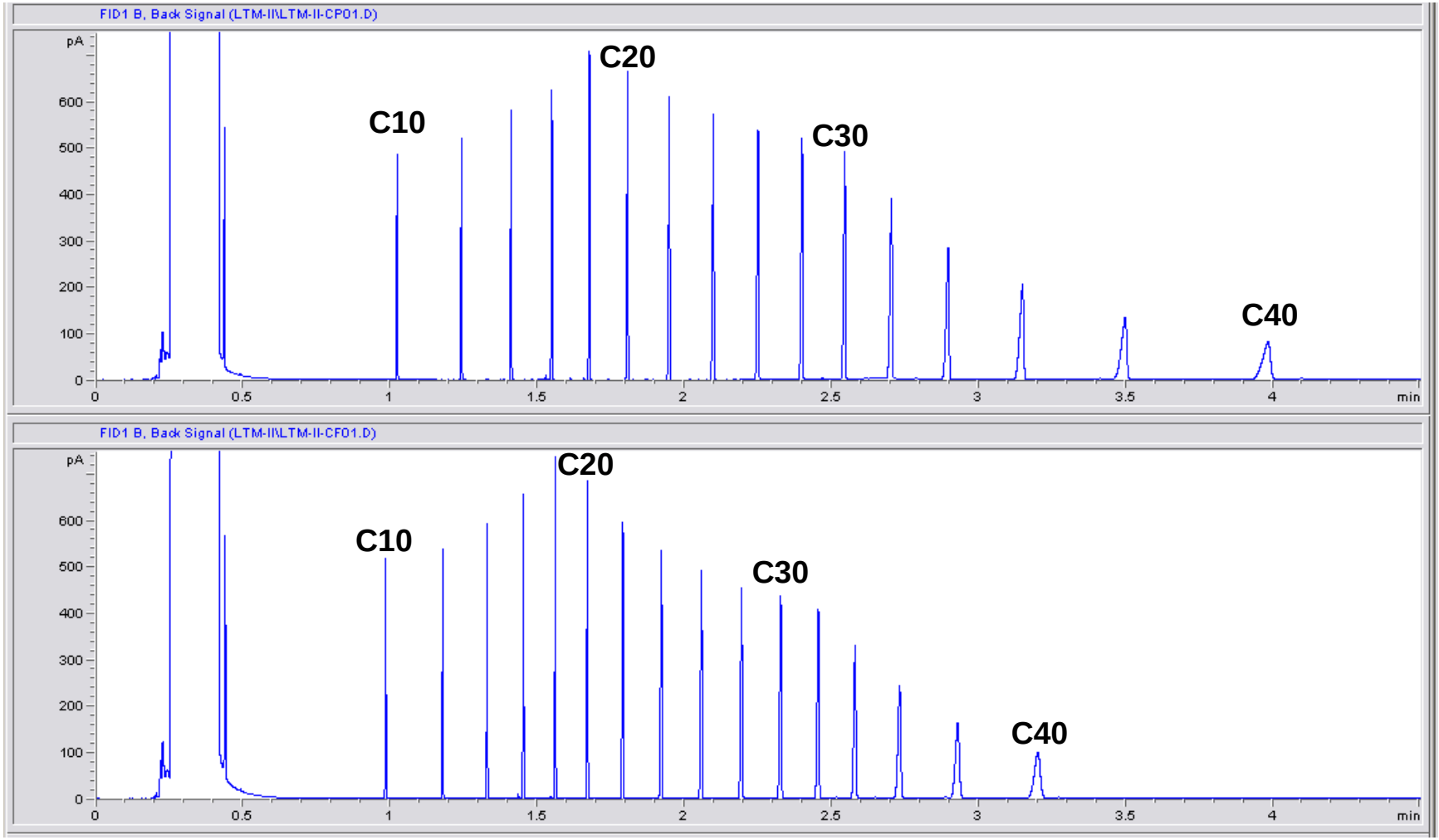
-Loss of stationary phase

-Change in temperature



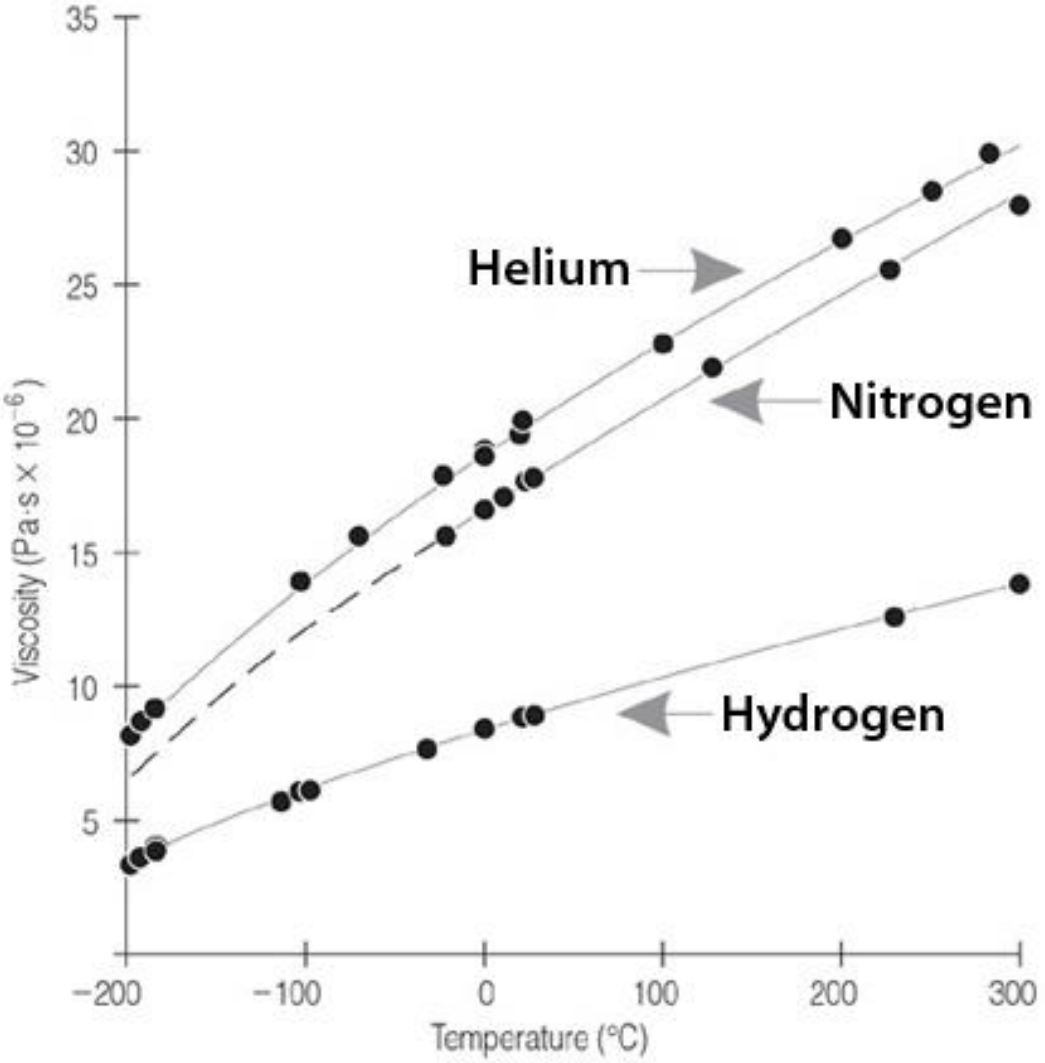



# Constant Pressure vs Constant Flow Retention




Under constant pressure conditions, flow decreases as temperature increases. (viscosity of a gas increases as temperature increases)

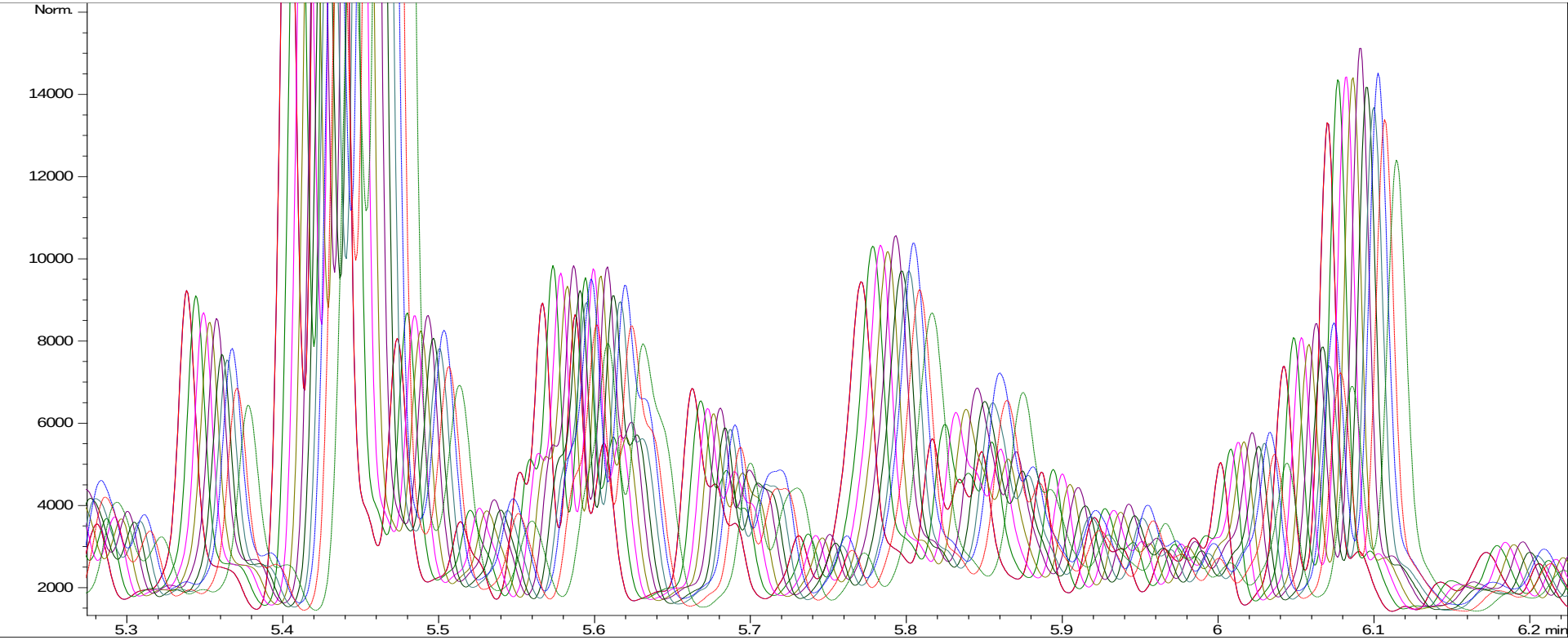
# Gas Viscosity vs Temperature




*J.V. Hinshaw, Column Connections, LCGC Asia Pacific, 12(2), 1100 (2009).*

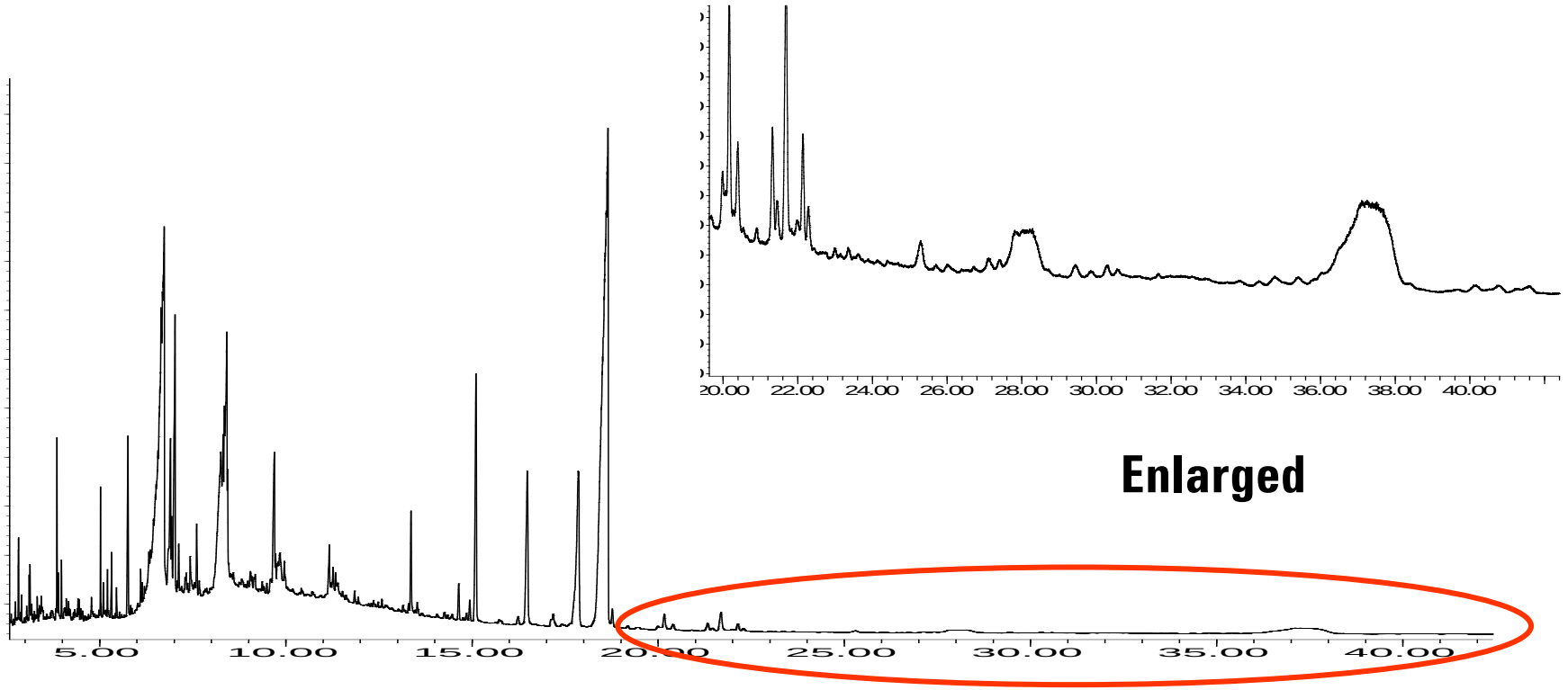
# Retention Times Shift 4-5 sec During the First 10 Runs

**Overlay of first 10 runs**



# GC-FID Chromatogram of Fish Oil

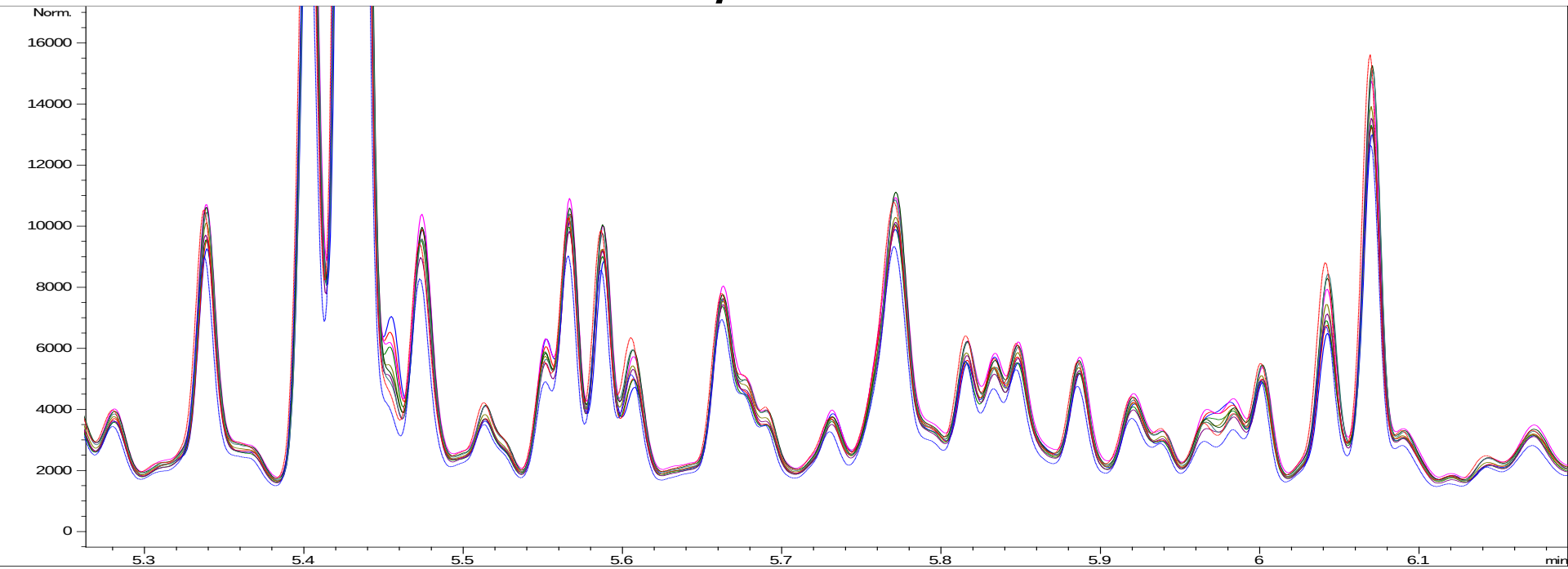
**1-µL splitless injection of 10% fish oil**
**GC oven was held at 290 °C for 30 min at the end of the run.**



# Get High M.W. Fish Oil Off the Column After Every Run = No More Shifting Retention

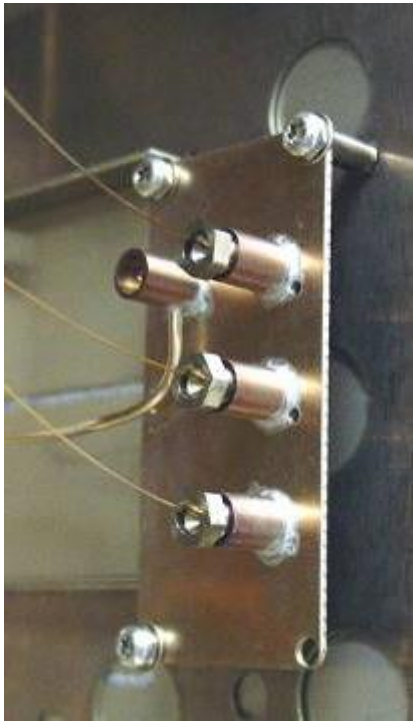
Retention Time Precision with Backflushing

**Overlay of 10 Runs**



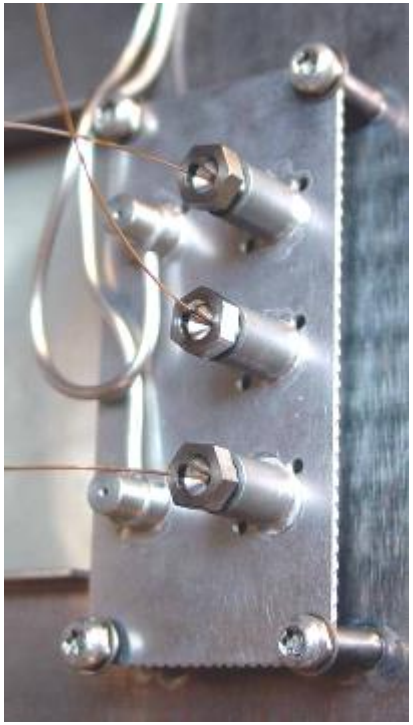
# Purged Capillary Flow Devices

**2-Way Splitter with Makeup**



**3-Way Splitter with Makeup**

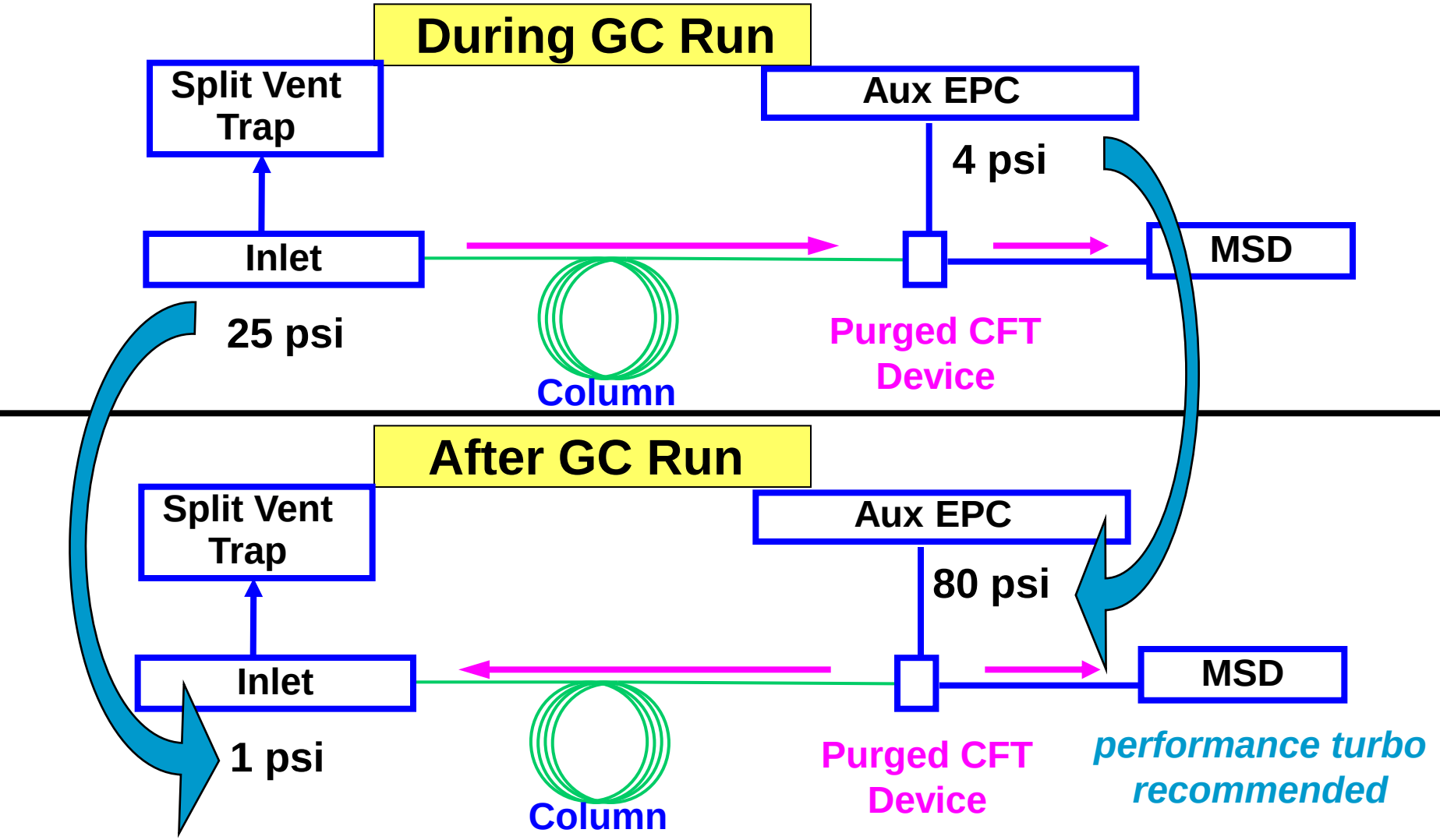
**Deans Switch**



**Purged Union (most recent)**

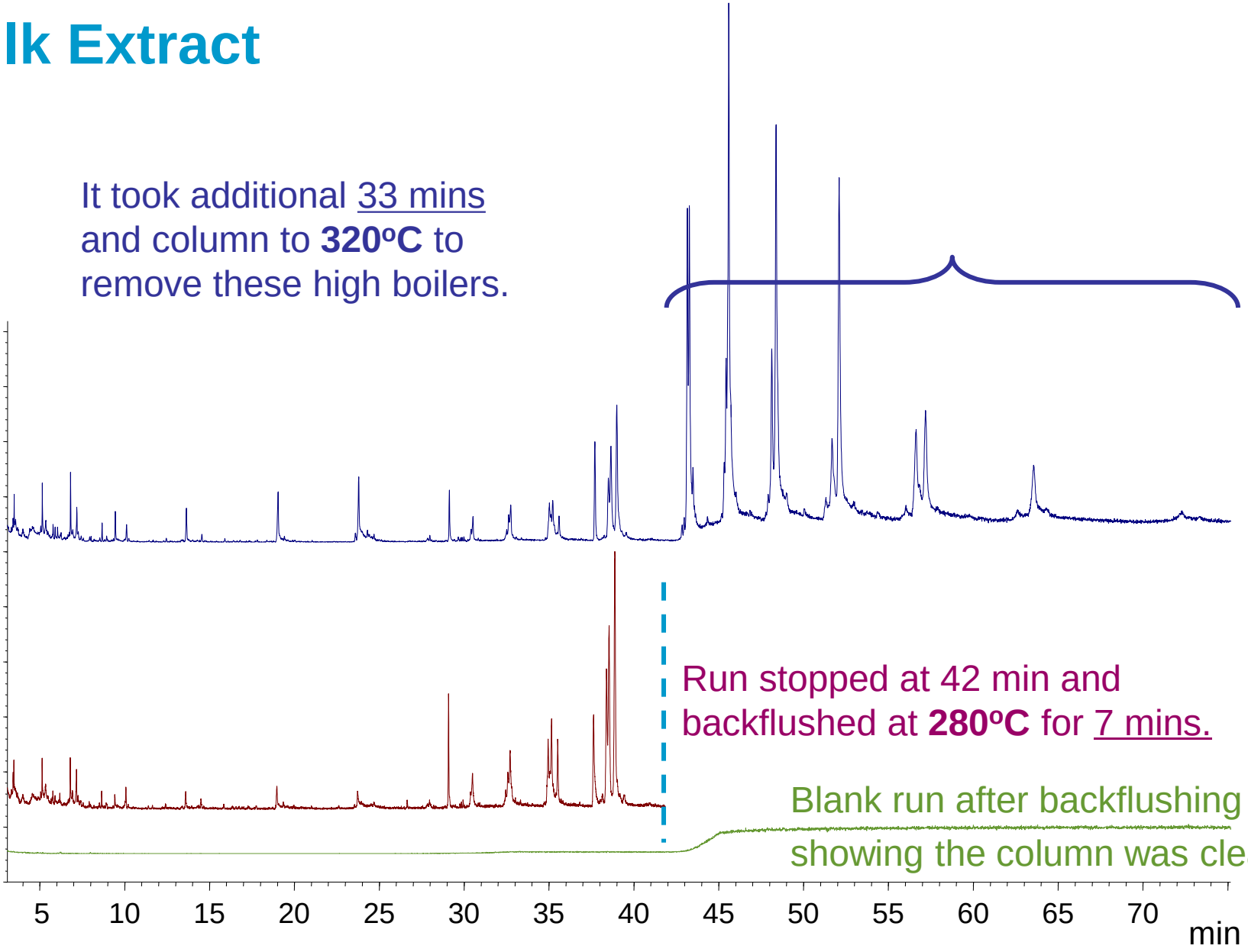
All Purged Capillary Flow Devices are Capable of Backflushing

# Backflush with Purged Union or Any Purged Capillary Flow Technology Device

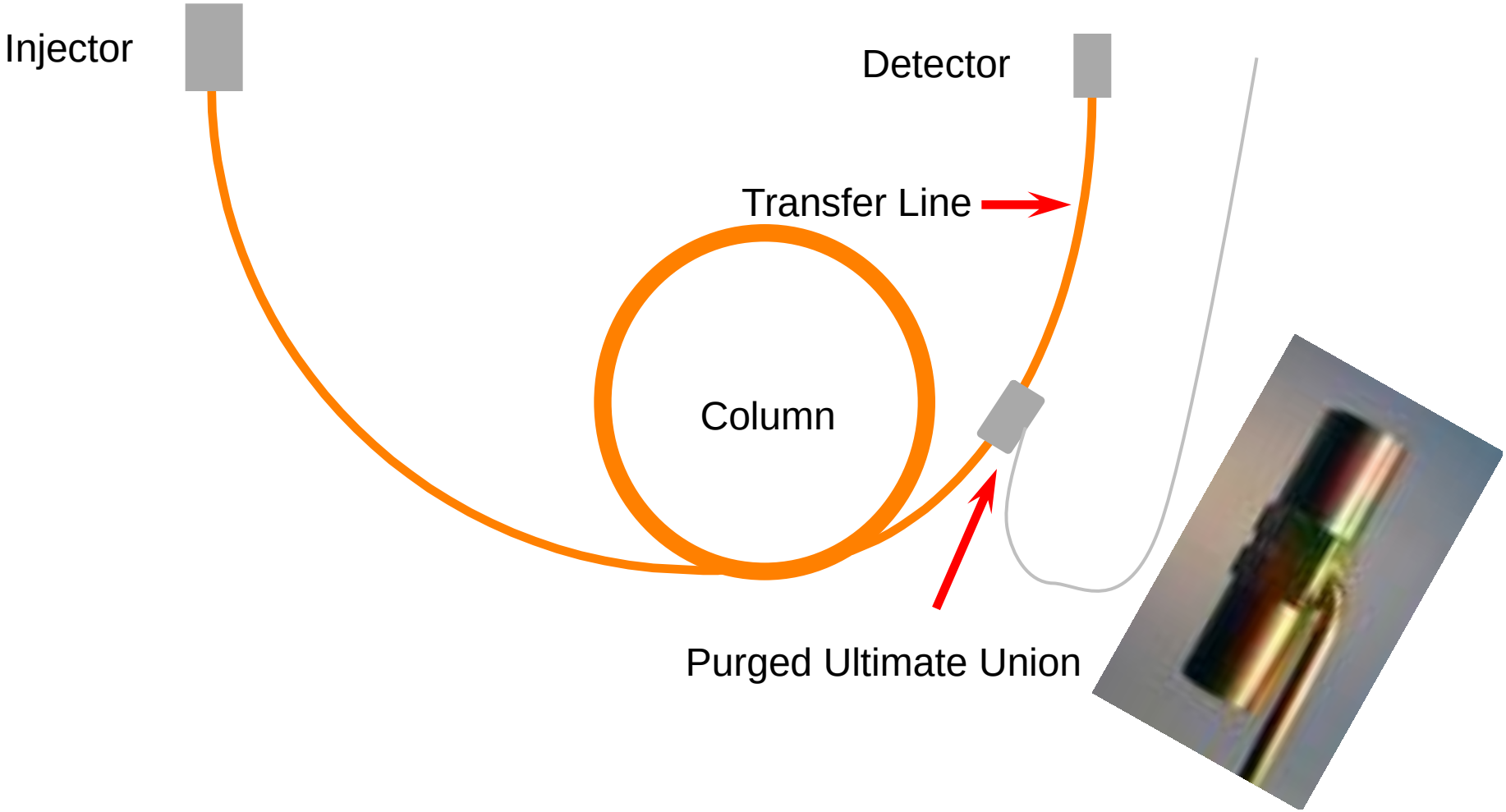


# Milk Extract

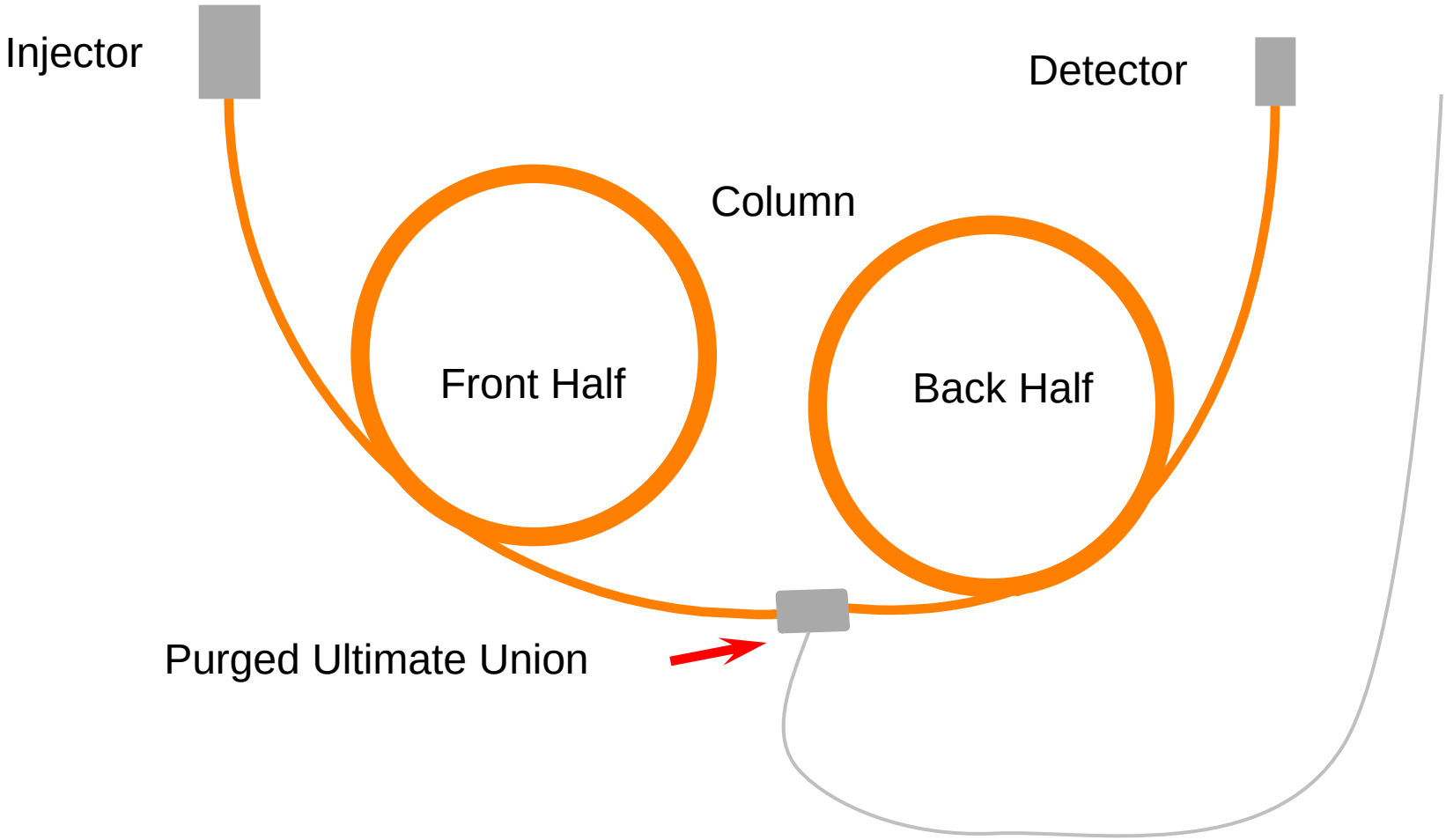
It took additional 33 mins and column to **320ºC** to remove these high boilers.



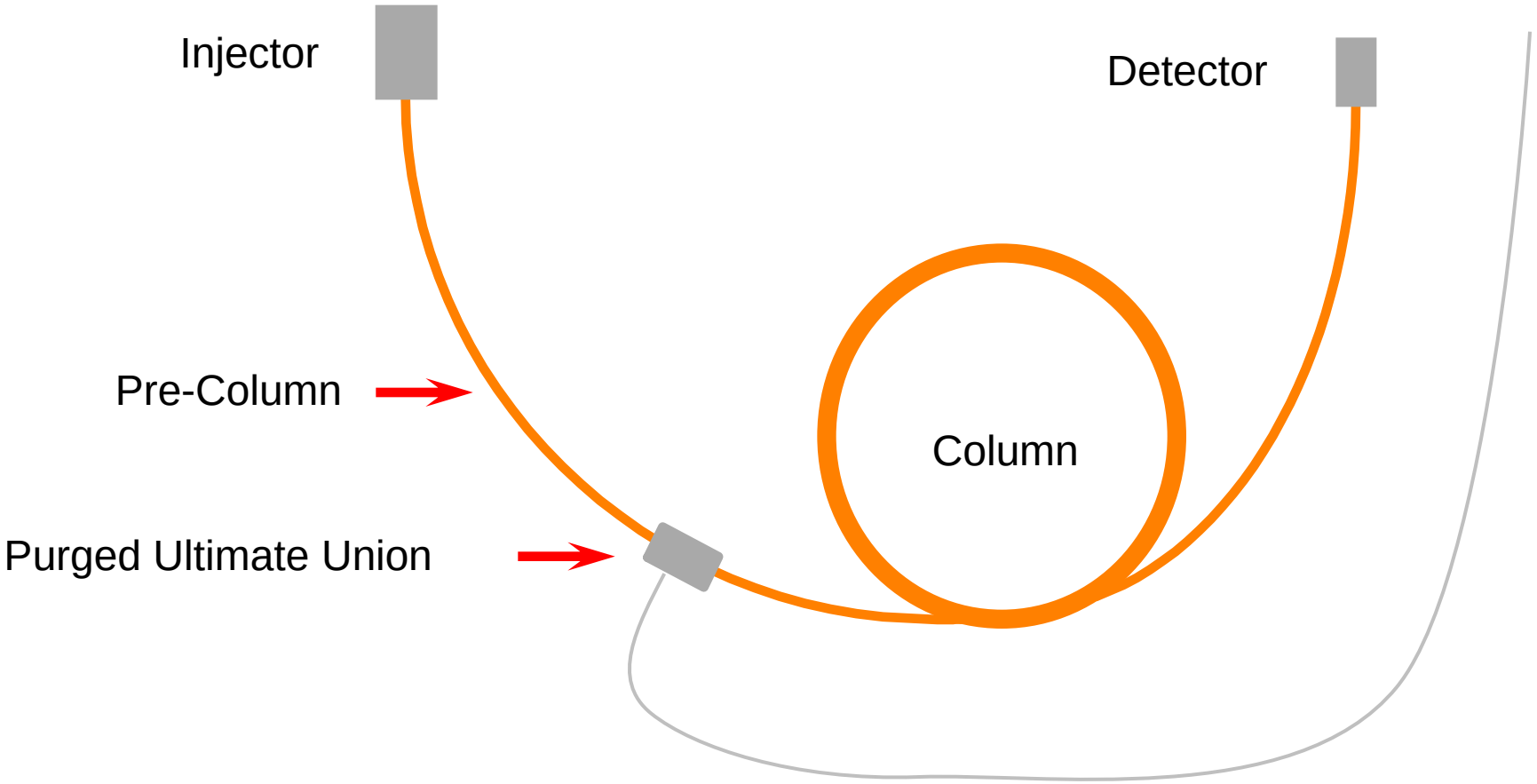
# Post-Column Backflush



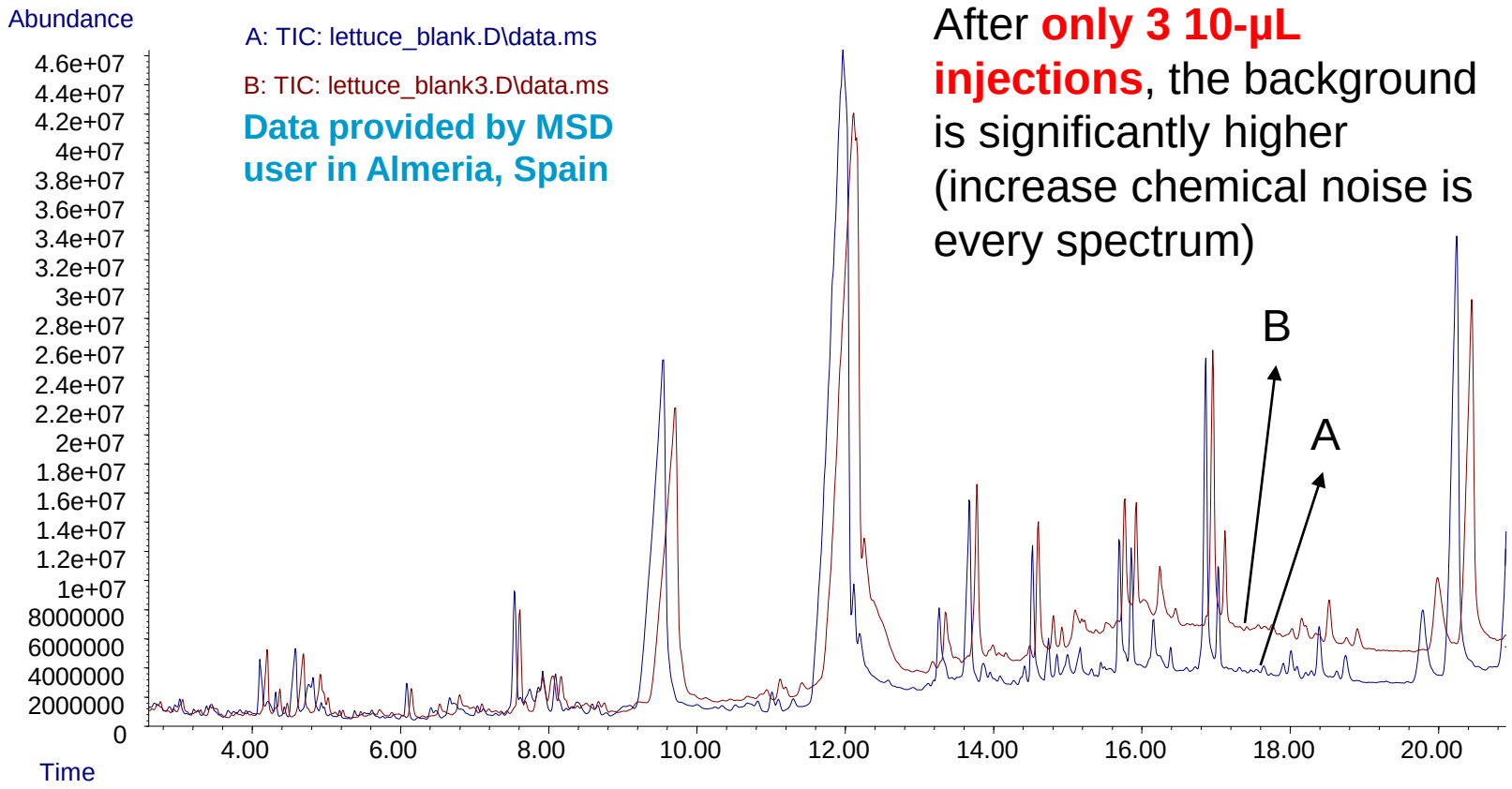
# Mid-Column Backflush



# Pre-Column Backflush

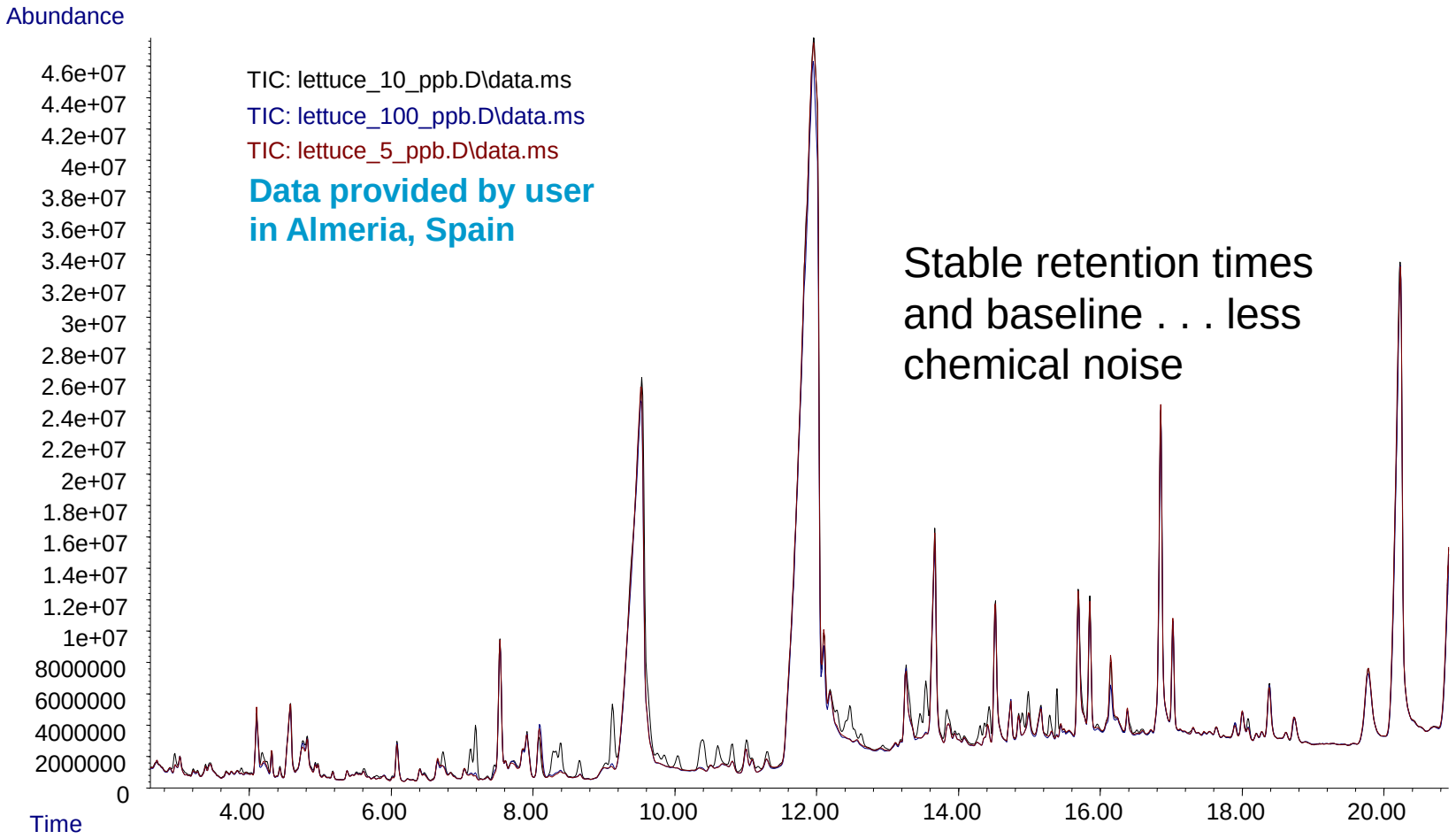


# Without Backflush: A Serious Problem



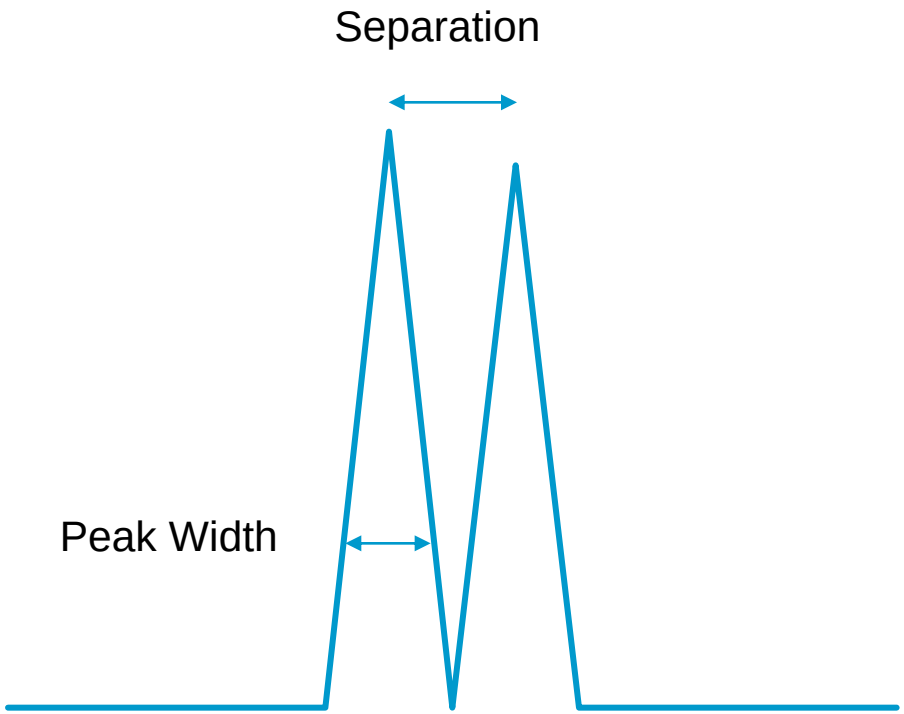

**Overlay of two chromatograms of a blank extract injected BEFORE (A) and AFTER (B) three injections without backflush**

# With Backflush: No Increased Background (Less Spectral Noise) and Consistent Retention Times




**Overlay of three chromatograms of lettuce extract run with 2 min of back flush**

# Loss of Resolution




**Resolution is a function of separation and peak width**

# Loss of Resolution - Separation Decrease

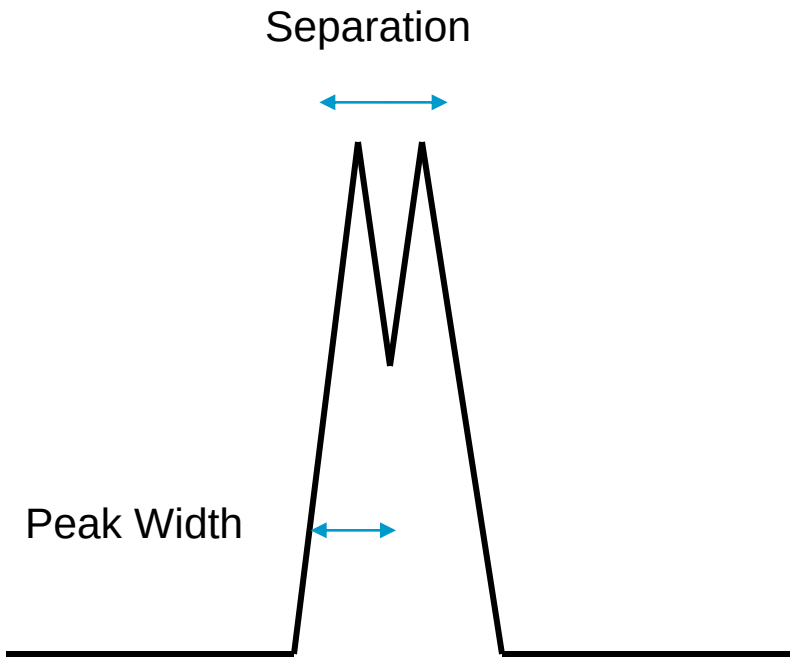
COLUMN

-Different column temperature

-Contamination (more phase?)

-Matrix components co-eluting

-Different column phase?



# Loss of Resolution - Peak Broadening

FLOW

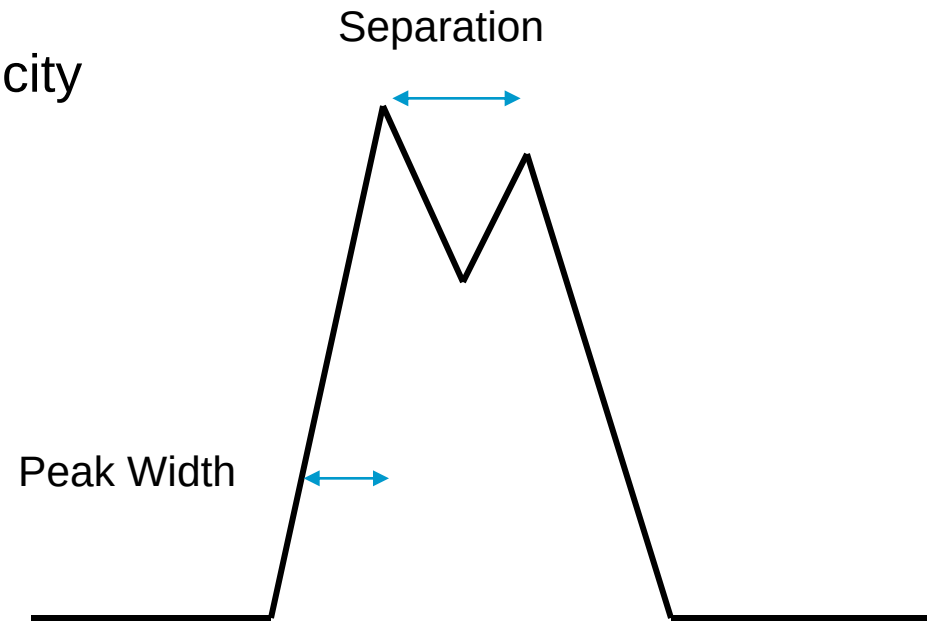
-Change in carrier gas velocity

-Make-up gas

COLUMN

-Contamination

-Phase degradation




INJECTOR (efficiency)

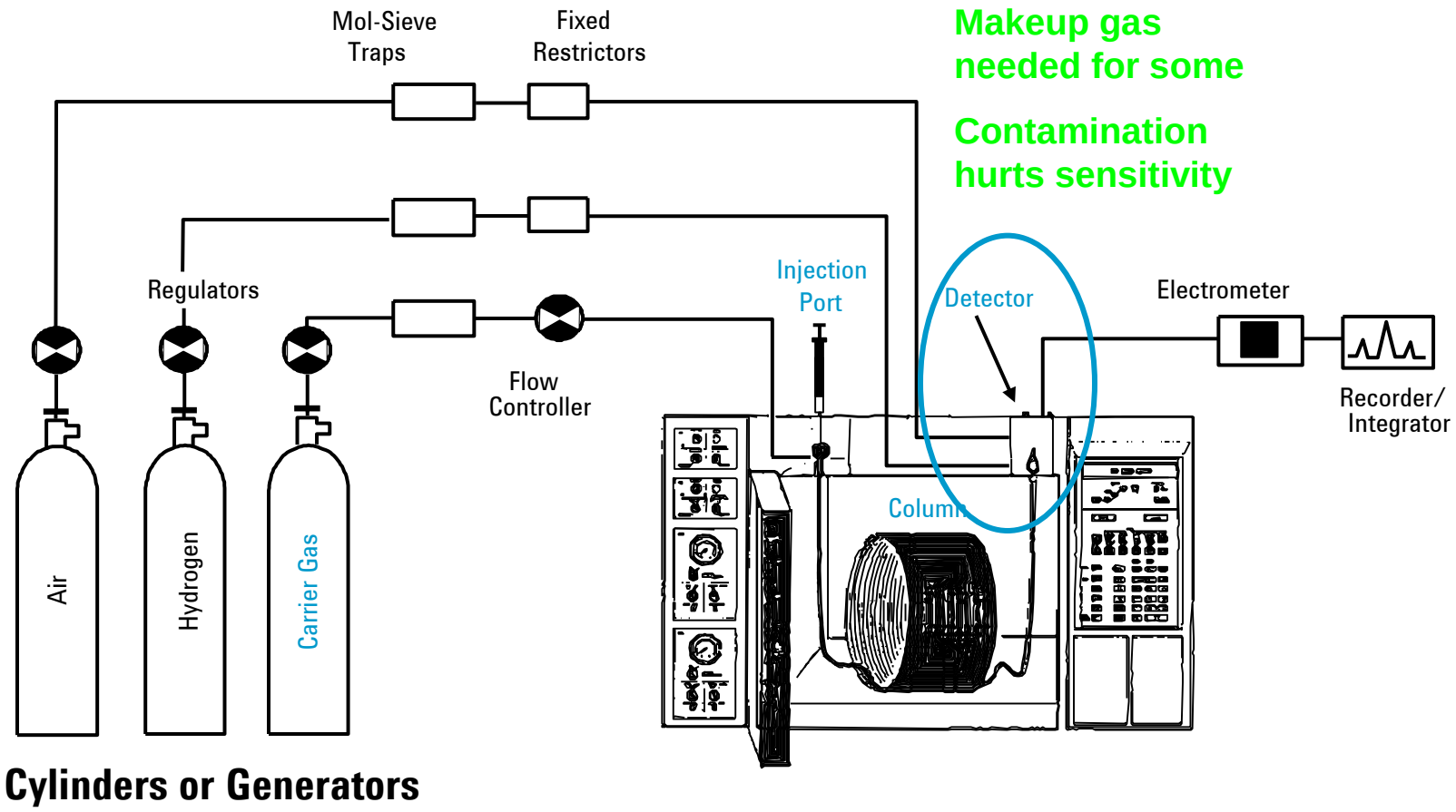
-Settings, Liner, Installation, etc.

# Typical Gas Chromatographic System

Gas settings have to be correct

Makeup gas needed for some

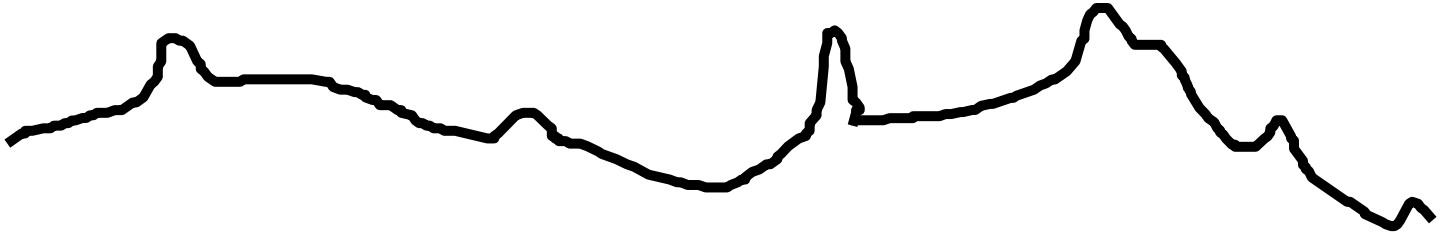
Contamination hurts sensitivity



# Baseline Disturbances

## Sudden Changes, Wandering, or Drifting

WANDER



COLUMN or DETECTOR

-Not fully conditioned or stabilized (electronics)

-Contamination

FLOW

-Changes in carrier and/or detector gas flows

-Valves switching, leaks

DRIFT

# Noisy Baseline

MILD

SEVERE

FLOW

-Contaminated gas

-Incorrect detector settings

COLUMN

-Bleed if at high temperature

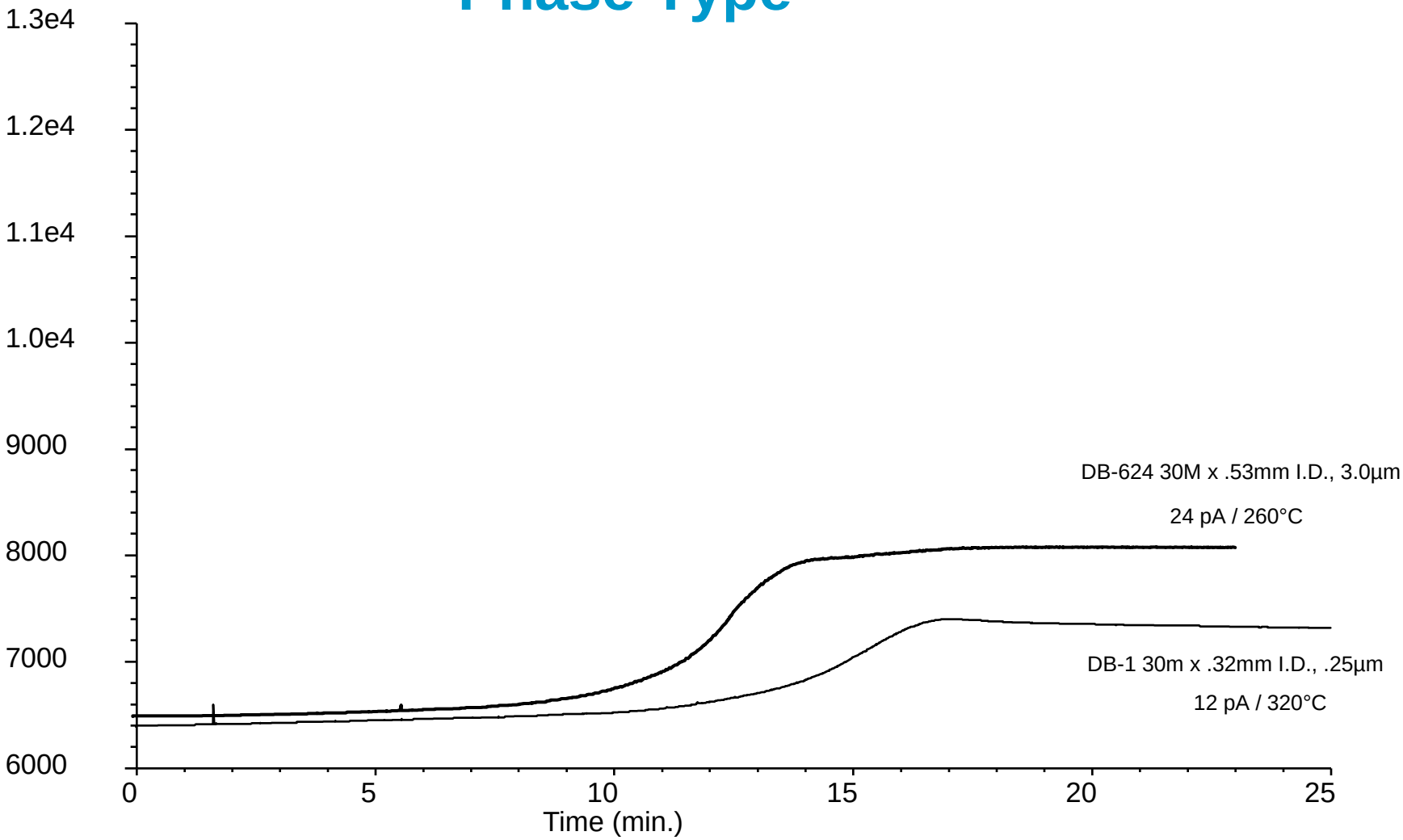
-In detector flame (poor installation)

DETECTOR

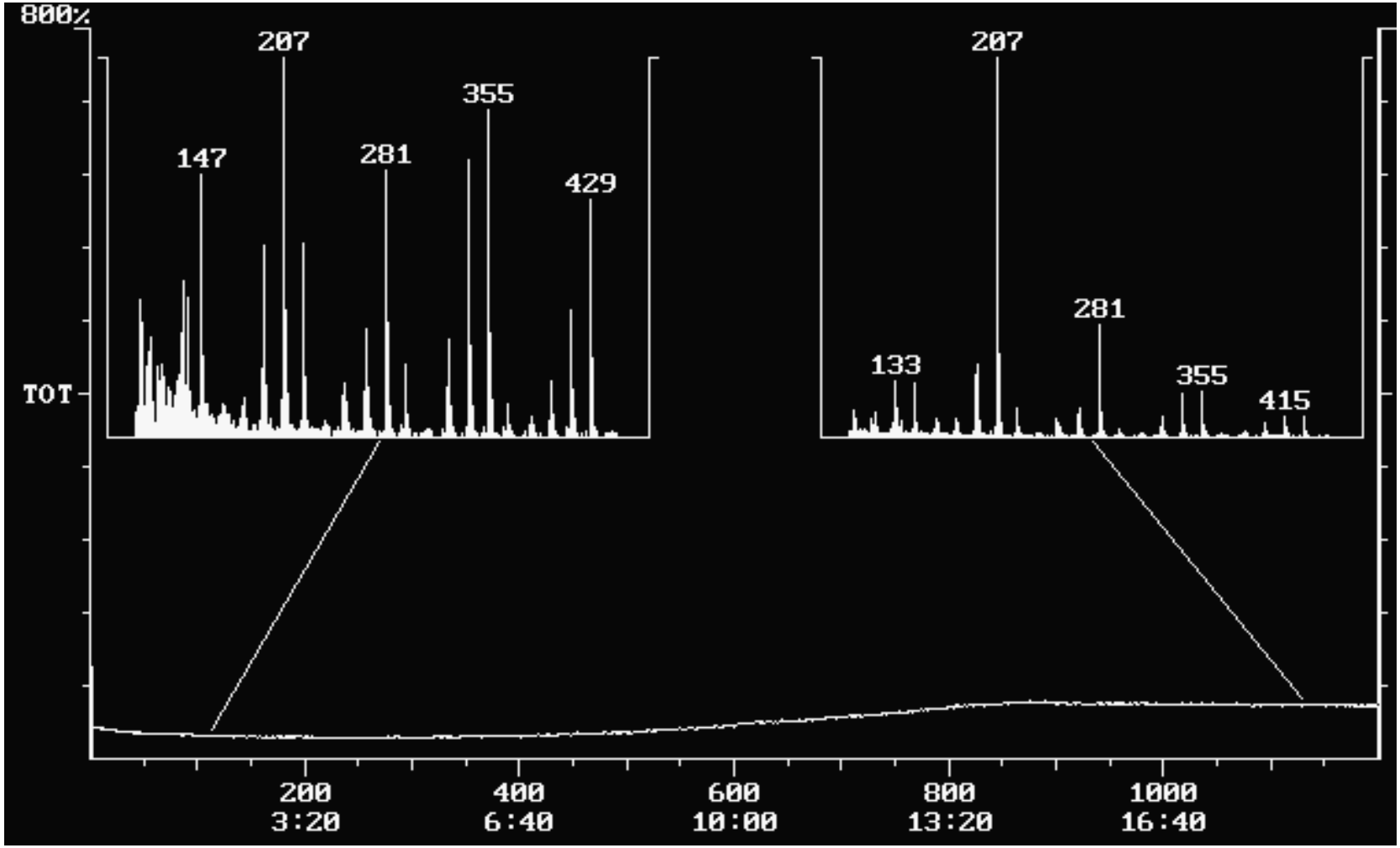
-Air leak - ECD, TCD

-Electronics malfunction

# Column Bleed – Affected by Dimensions and Phase Type



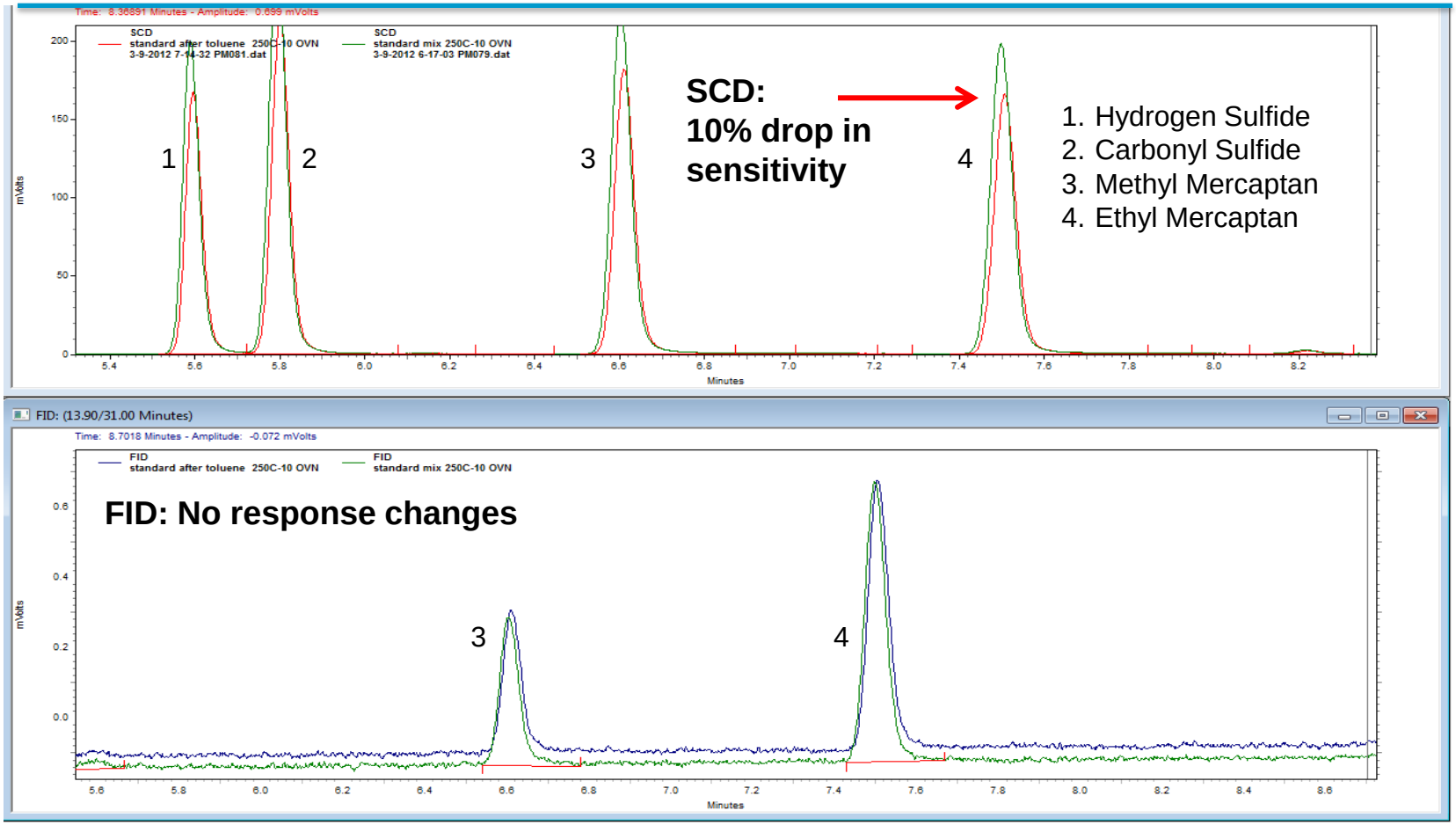
# Mass Spectrum of Phenylmethylpolysiloxane Column Bleed (Normal Background)




Mass spectral library search is not always accurate

# Traditional PDMS column- Coking (desensitization) of Reactor Tubes

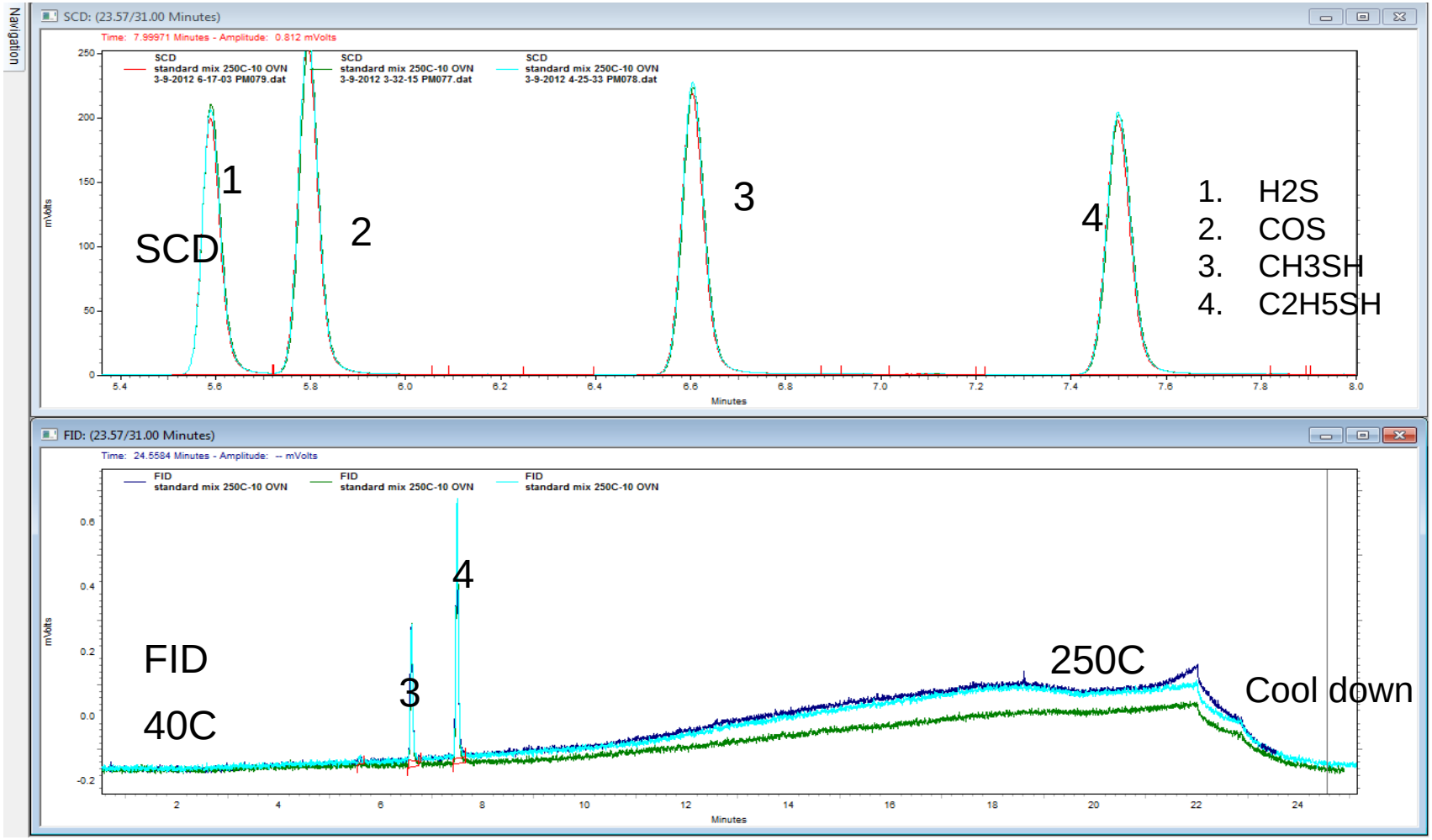
Overlay of before (green) and after 2 x 2 uL neat toluene Injection (red)




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

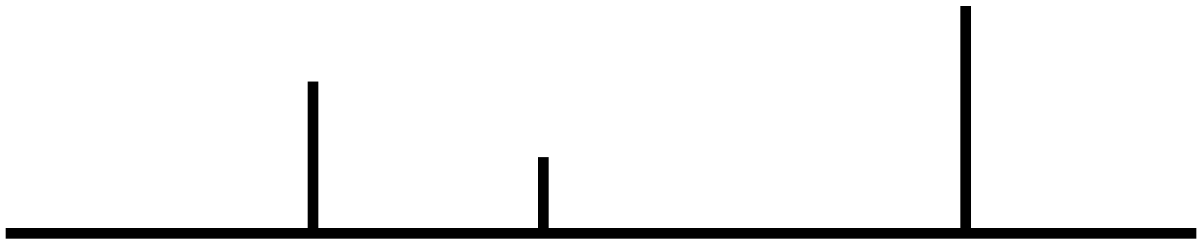
# New DB-Sulfur SCD column

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




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

# Spiking Baseline



DETECTOR

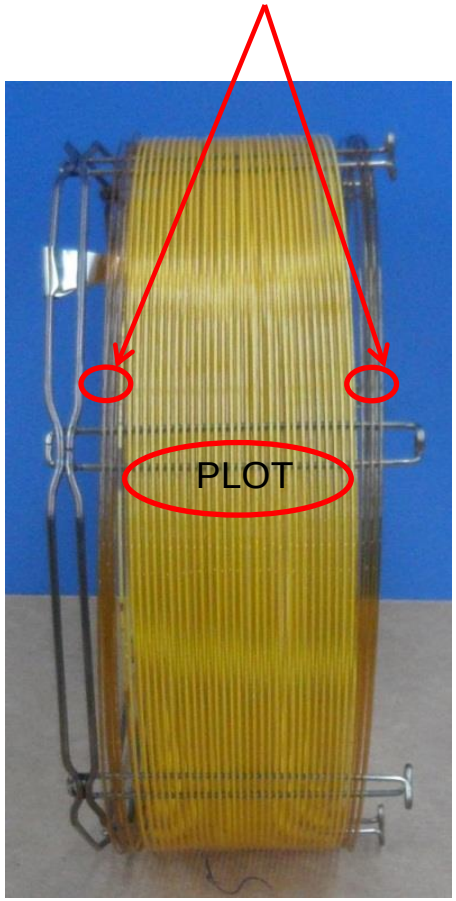
-Particles entering the detector

-Random:  poor connection

-Regular:  nearby "cycling" equipment (electronics)

# NEW Integrated Particle Trap PLOT Column

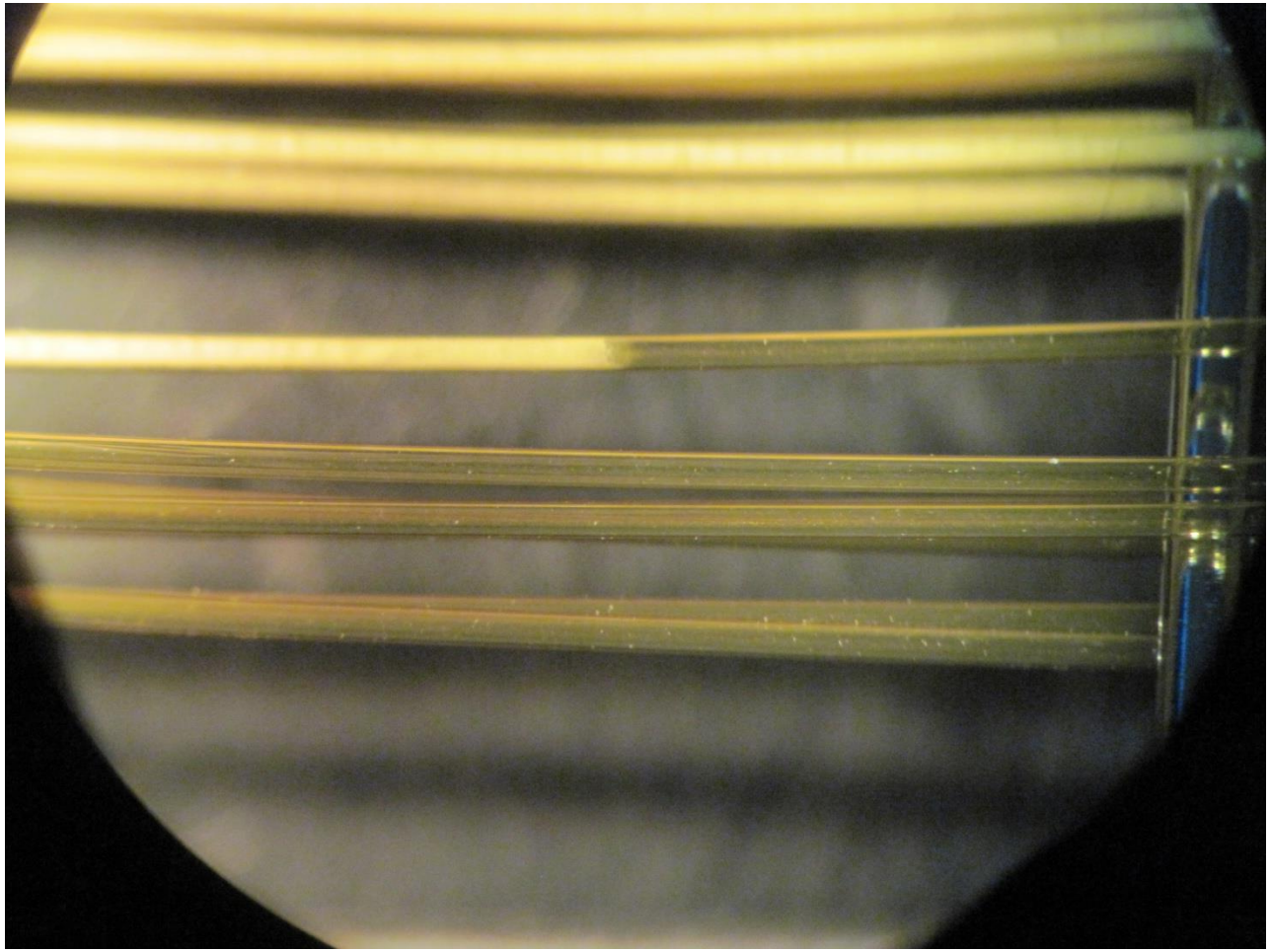
2.5 meter integrated particle traps on both ends



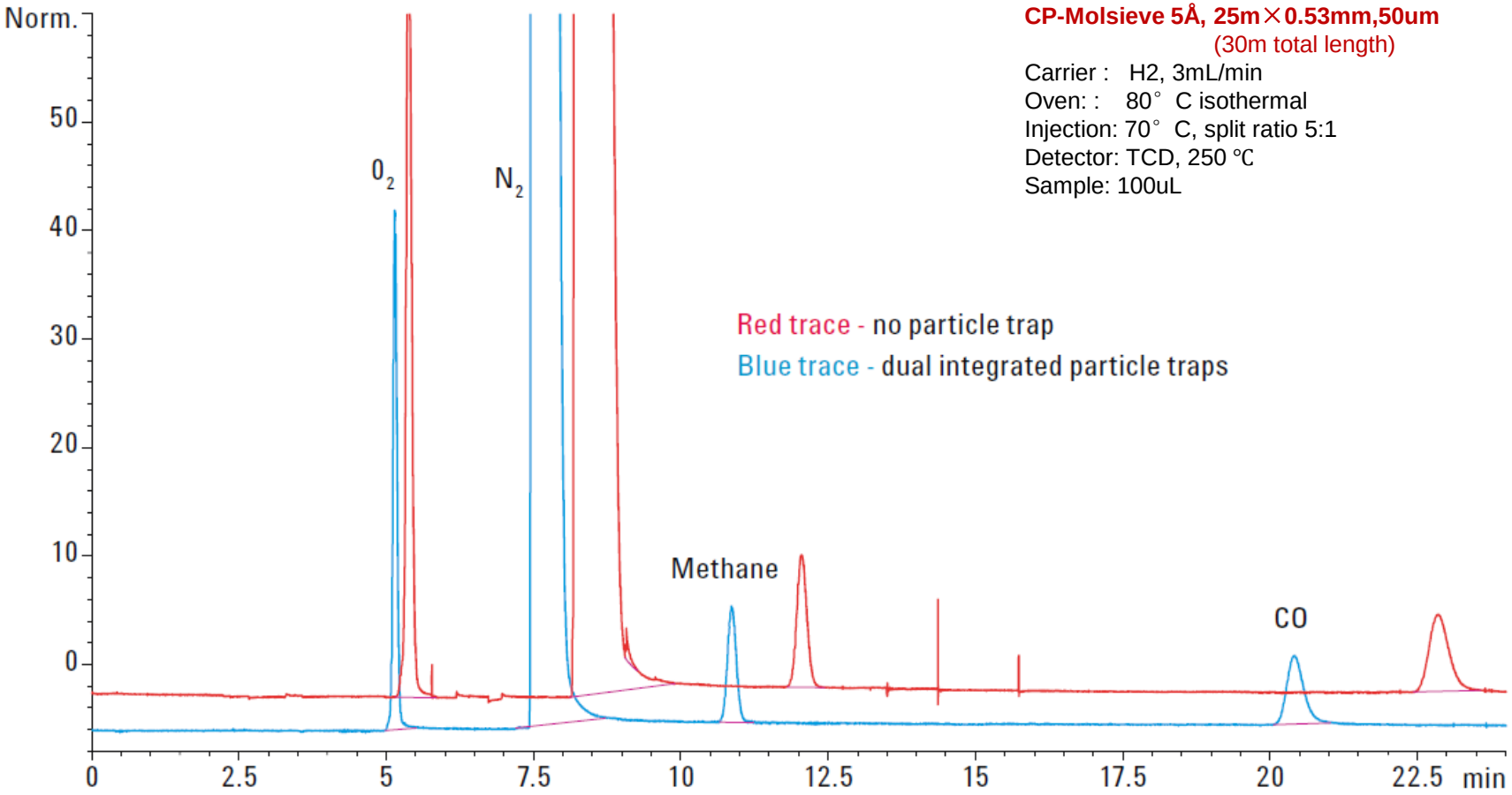

Compatible with GC/MS and valve switching systems, and Capillary Flow Technology

Very similar selectivity, plates and peak shape to existing Agilent PLOT columns

# The PLOT Column and Integrated Particle Trap



# 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

Red trace - no particle trap
Blue trace - dual integrated particle traps

# Quantitation Problems

DETECTOR

-Poor stability (electronics) or Baseline disturbances (contamination)

-Outside detector's linear range or wrong settings

Activity (adsorption) in INJECTOR or COLUMN

INJECTOR

-Technique, settings, conditions

-Syringe worn

OTHER

-Co-elution

-Matrix effects

-Sample evaporation – leaky vials

-Sample decomposition

# Agilent 5975C GC/MS

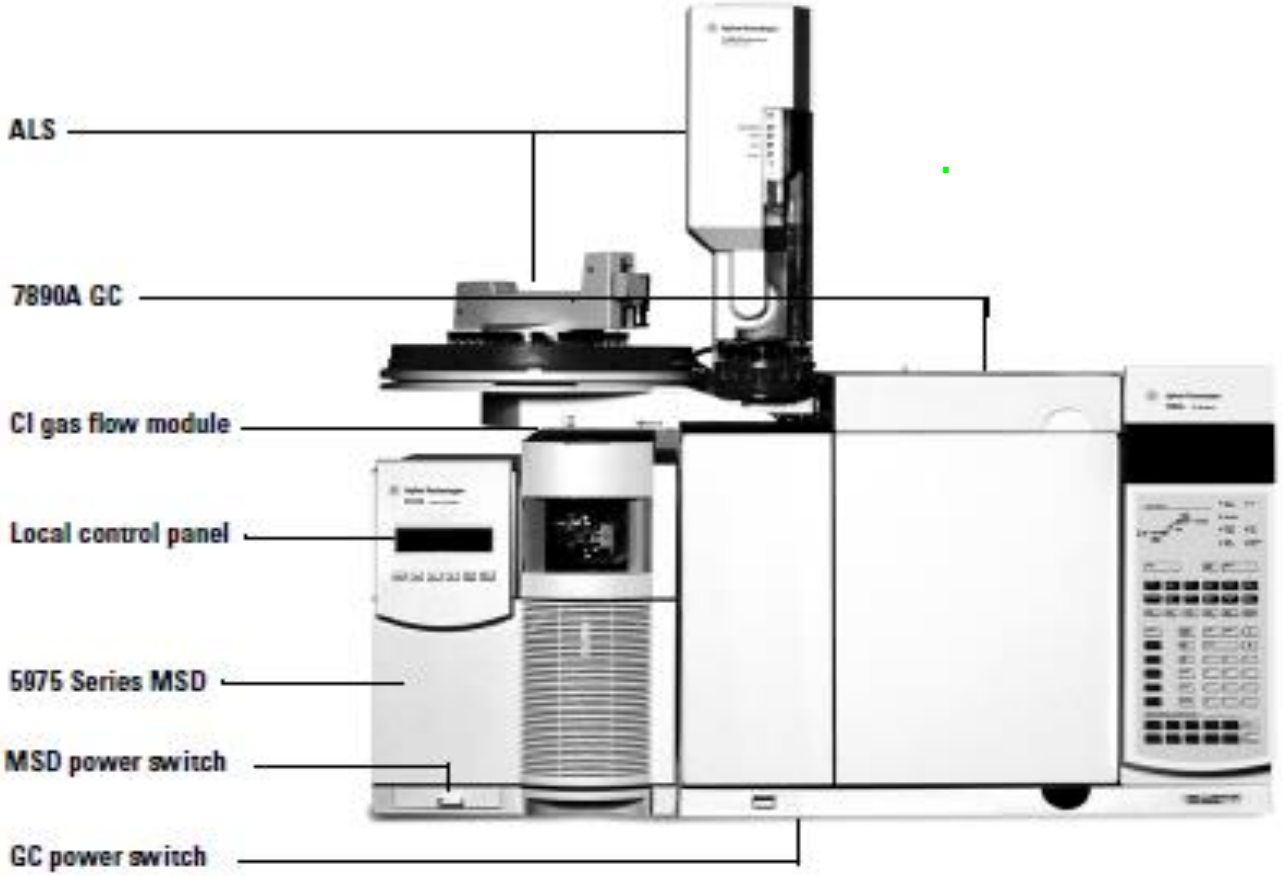
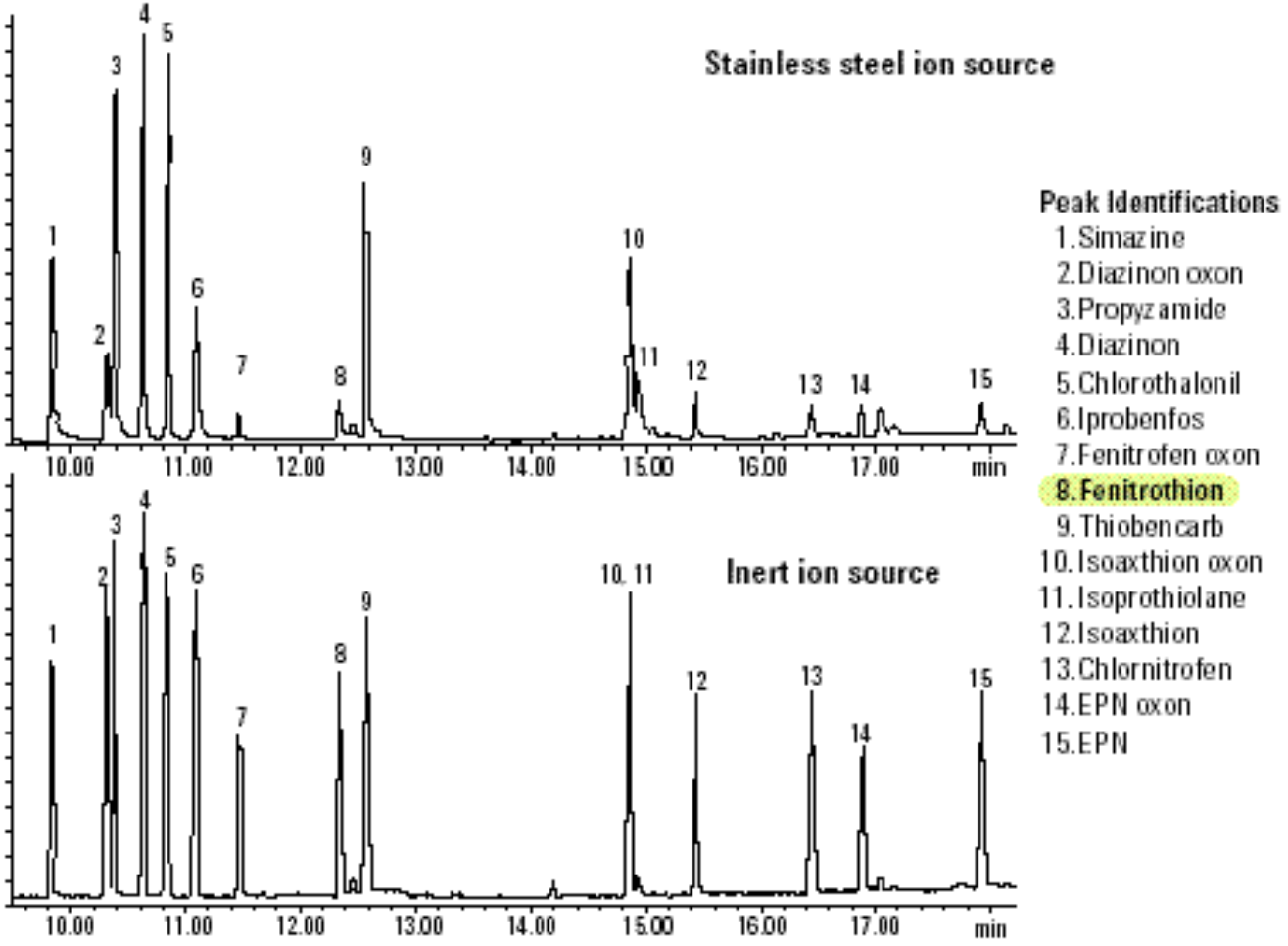


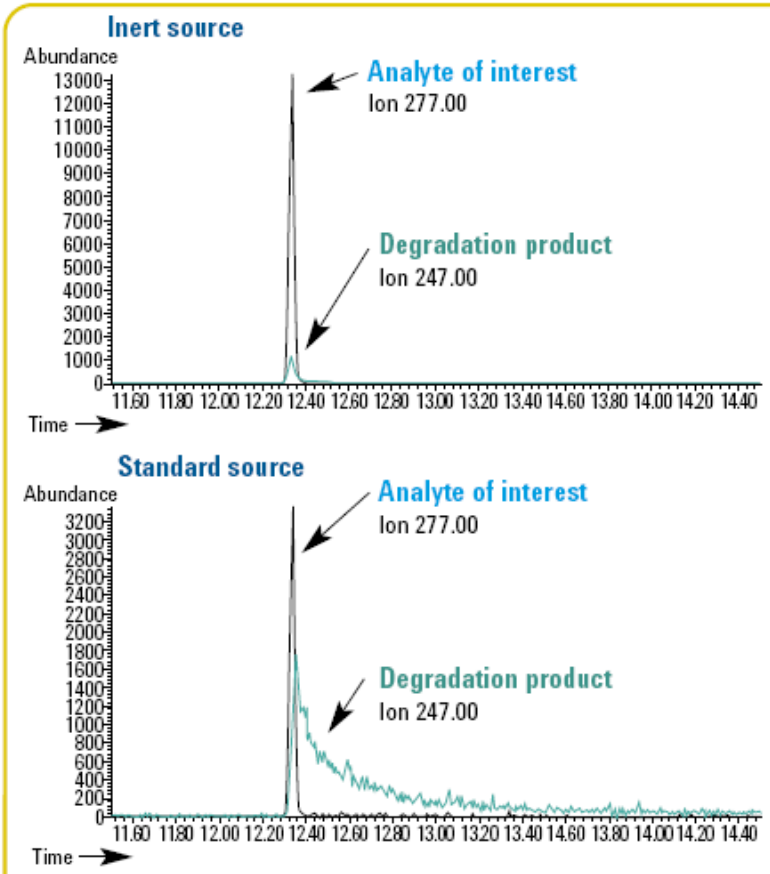

**Figure 1** 5975 Series GC/MSD system

# MSD Source Inertness – an old problem revisited

Inert MSD source increases response of chemically active analytes



# Inert MSD Source




Mass chromatograms for the pesticide Fenitrothion acquired via the inert source (upper) and a standard source design (lower). The black line indicates the ion abundance of the molecular ion of Fenitrothion (m/z 277) and the green line is attributed to a degradation product (m/z 247).

# Mass Spectral Problems

## High background

- Excessive pressure in the analyzer chamber
- Air leak
- Contamination

# Mass Spectral Problems

## High abundances at m/z 18,28,32 and 44

- System recently vented (pumping down)
- Masses at 14,16 and 19 are indicative of a massive leak

# Common Contaminant Ions

| Ions (*m/z*) | Compound | Possible source |
|---|---|---|
| 18, 28, 32, 44 or 14, 16 | $H_2O$, $N_2$, $O_2$, $CO_2$ or N, O | Residual air and water, air leaks, outgassing from Vespel ferrules |
| 31, 51, 69, 100, 119, 131, 169, 181, 214, 219, 264, 376, 414, 426, 464, 502, 576, 614 | PFTBA and related ions | PFTBA (tuning compound) |
| 31 | Methanol | Cleaning solvent |
| 43, 58 | Acetone | Cleaning solvent |
| 78 | Benzene | Cleaning solvent |
| 91, 92 | Toluene or xylene | Cleaning solvent |
| 105, 106 | Xylene | Cleaning solvent |
| 151, 153 | Trichloroethane | Cleaning solvent |
| 69 | Foreline pump oil or PFTBA | Foreline pump oil vapor or calibration valve leak |
| 73, 147, 207, 221, 281, 295, 355, 429 | Dimethylpolysiloxane | Septum bleed or methyl silicone column bleed |
| 77, 94, 115, 141, 168, 170, 262, 354, 446 | Diffusion pump fluid and related ions | Diffusion pump fluid |
| 149 | Plasticizer (phthalates) | Vacuum seals (O-rings) damaged by high temperatures, vinyl gloves |
| Peaks spaced 14 *m/z* apart | Hydrocarbons | Fingerprints, foreline pump oil |

# But at the End of the Day…It's in the Inlet!

Do Inlet Maintenance

- Trim the Column
- Replace the Liner
- Replace the Gold Seal
- Replace the Syringe
- Replace the Split Vent Trap, Split Vent Line
- Rinse the Inlet

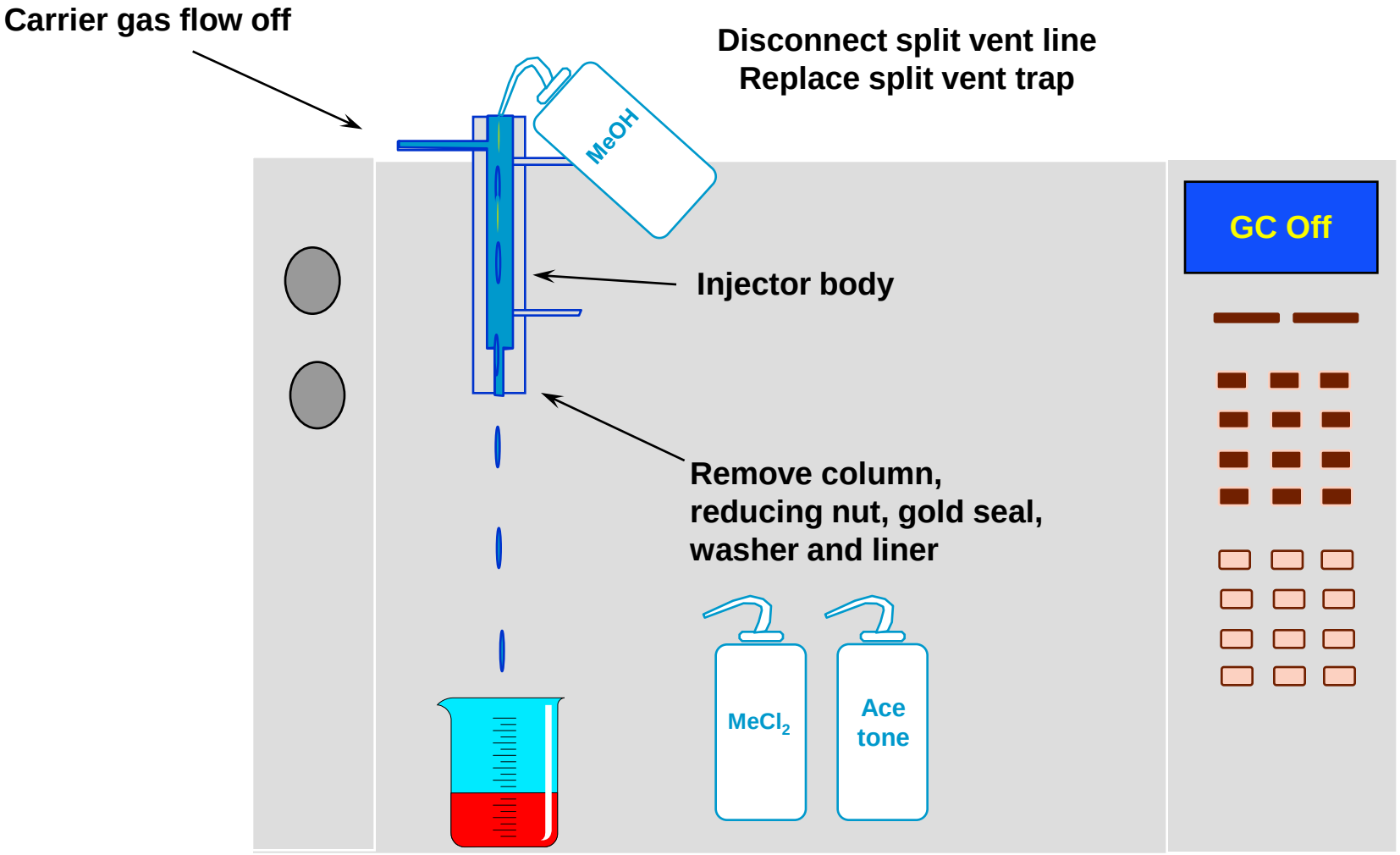
# Common Care and Maintenance Scheme for GC Columns

1. Bake out the column for no more than 2 hours.

2. Cut off 6”-1ft of the inlet end of the column.

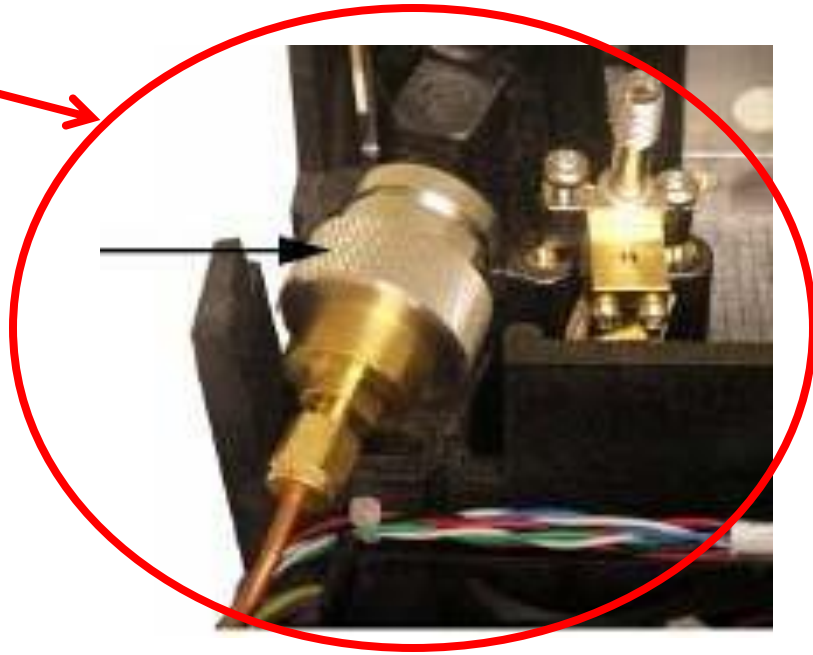
3. Cut off more column.  (repeat as necessary)

# Cleaning the Split/Splitless Injector



# Finding the Split Vent Trap

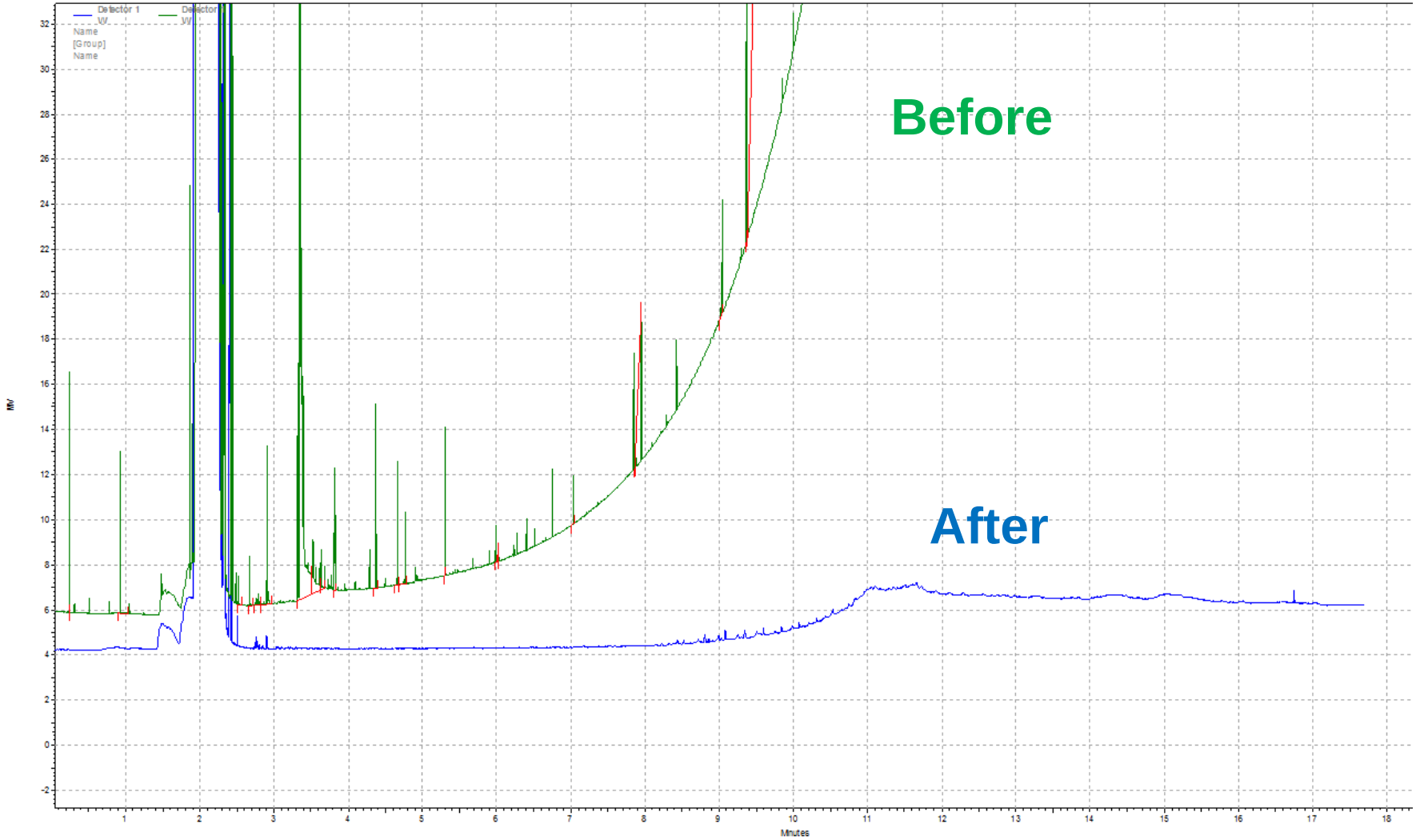
Remove cover at Split Vent



# Replacing the Split Vent Trap

Finger Tight Knurled Nut

# Split Vent Trap Changed (Column Bleed?!?)



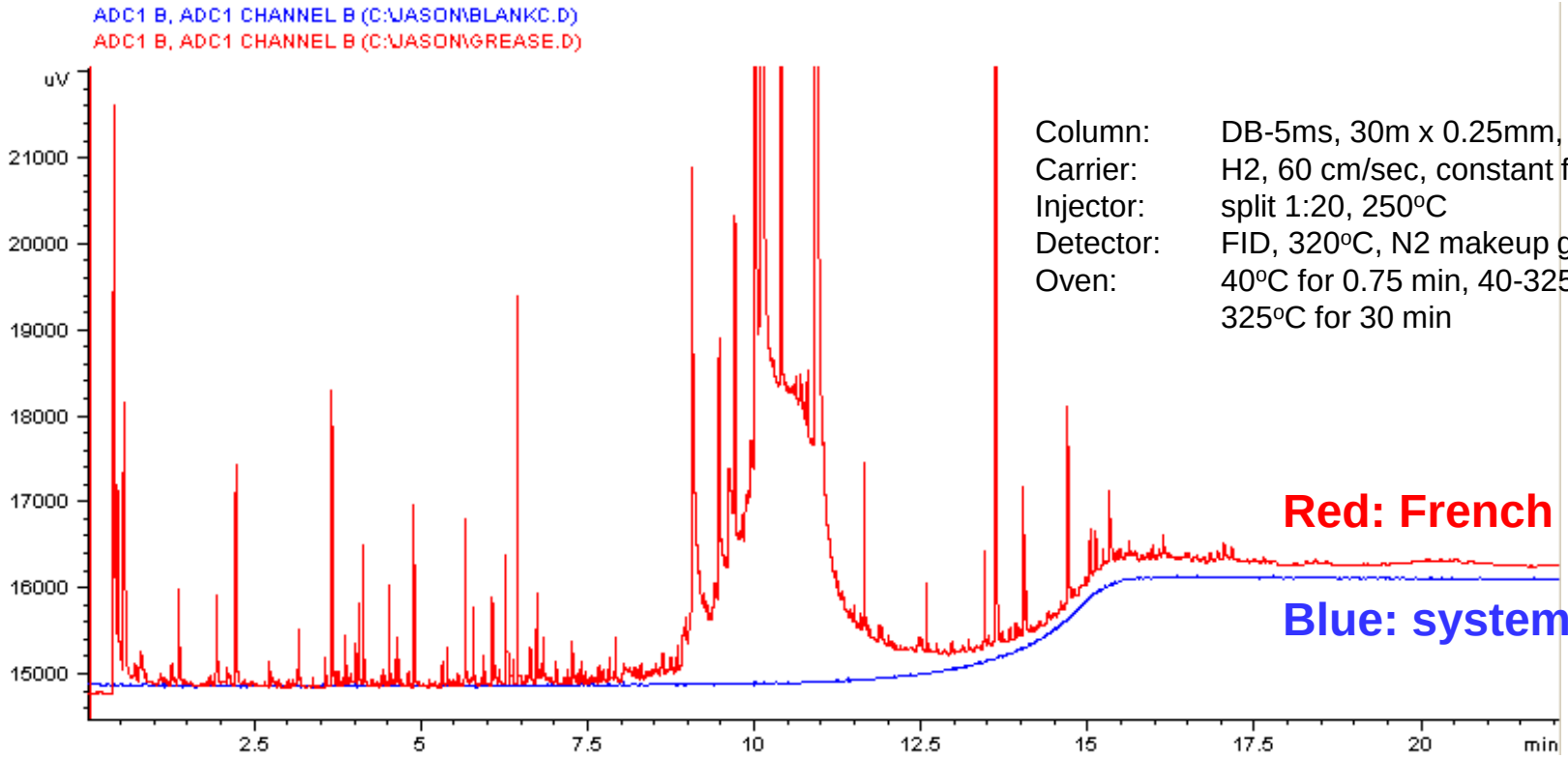



Agilent Technologies

# Be Careful When Doing Maintenance…

**You may be the CONTAMINATOR!**

# Contamination of system by residue on fingers during column installation




| | |
|---|---|
| Column: | DB-5ms, 30m x 0.25mm, 0.25um |
| Carrier: | H2, 60 cm/sec, constant flow |
| Injector: | split 1:20, 250°C |
| Detector: | FID, 320°C, N2 makeup gas |
| Oven: | 40°C for 0.75 min, 40-325°C at 20°C/min, 325°C for 30 min |

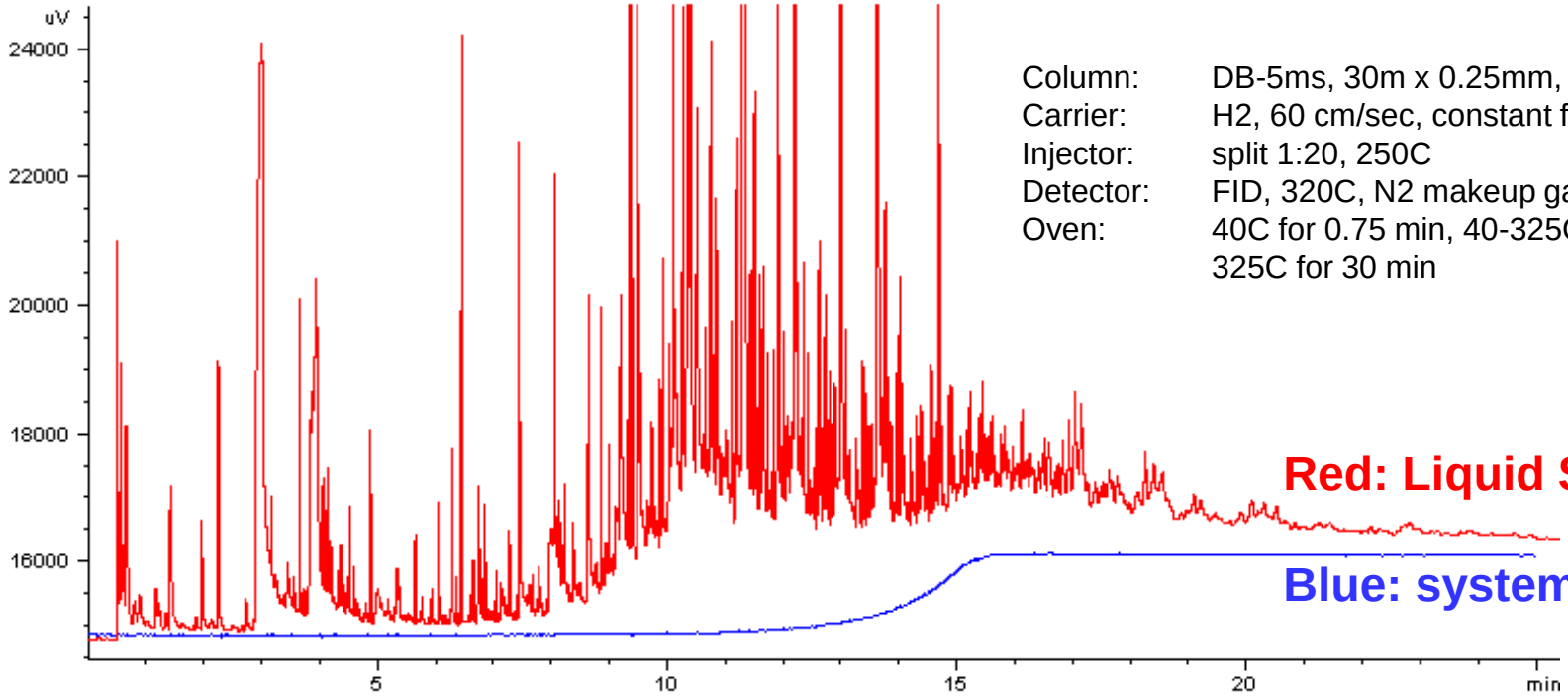
**Red: French Fry**

**Blue: system blank**

Procedure:

(1) Held French fry for 5 seconds.
(2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
(3) Lightly touched the part of the column sticking up above the ferrule.
(4) Installed column into injector.
(5) Set oven temperature to 40°C.
(6) Started oven temperature program as soon as oven reached 40°C.

# Contamination from Liquid Soap




| | |
|---|---|
| Column: | DB-5ms, 30m x 0.25mm, 0.25um |
| Carrier: | H2, 60 cm/sec, constant flow |
| Injector: | split 1:20, 250C |
| Detector: | FID, 320C, N2 makeup gas |
| Oven: | 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min |

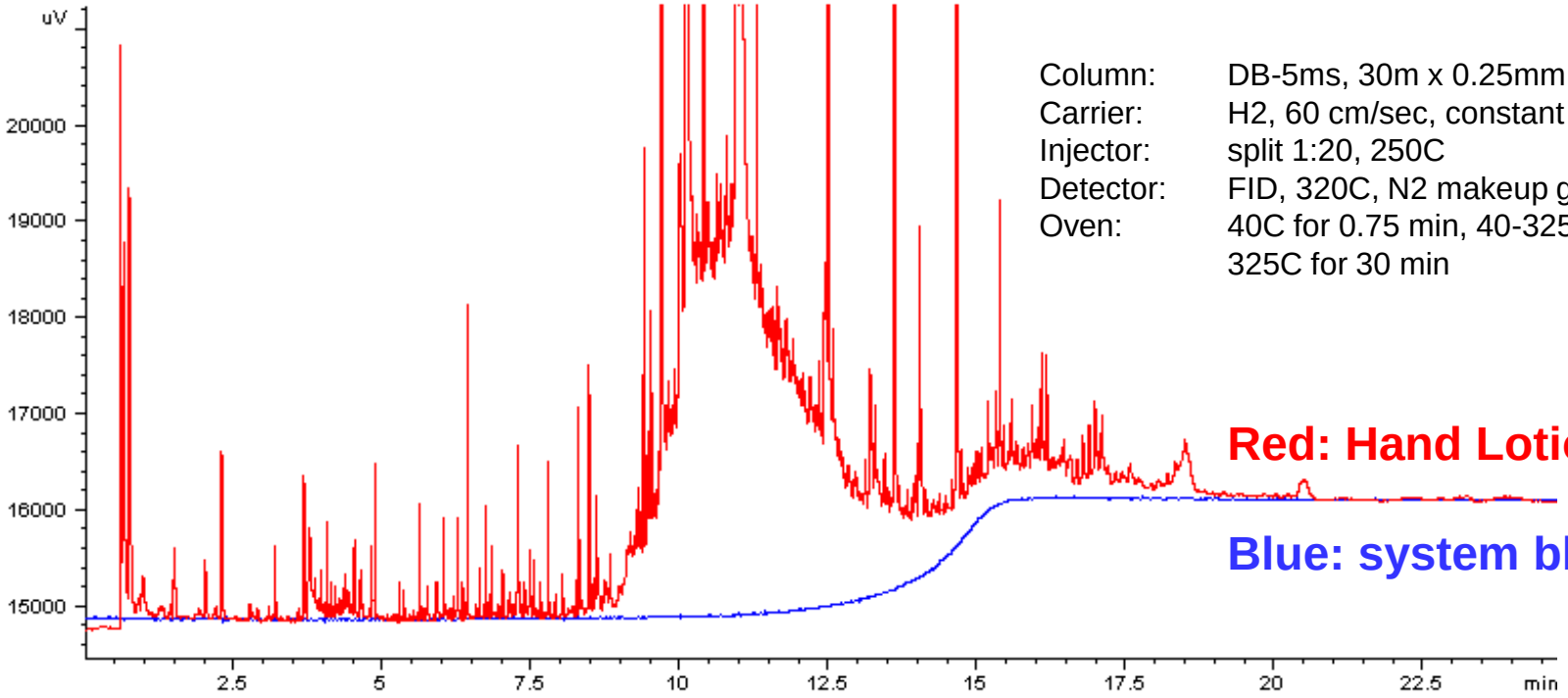
**Red: Liquid Soap**

**Blue: system blank**

Procedure:

(1) One very small drop of liquid soap placed on one fingertip.
(2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
(3) Lightly touched the part of the column sticking up above the ferrule.
(4) Installed column into injector.
(5) Set oven temperature to 40C.
(6) Started oven temperature program as soon as oven reached 40C.

# Contamination from Hand Lotion




| | |
|---|---|
| Column: | DB-5ms, 30m x 0.25mm, 0.25um |
| Carrier: | H2, 60 cm/sec, constant flow |
| Injector: | split 1:20, 250C |
| Detector: | FID, 320C, N2 makeup gas |
| Oven: | 40C for 0.75 min, 40-325C at 20C/min, 325C for 30 min |

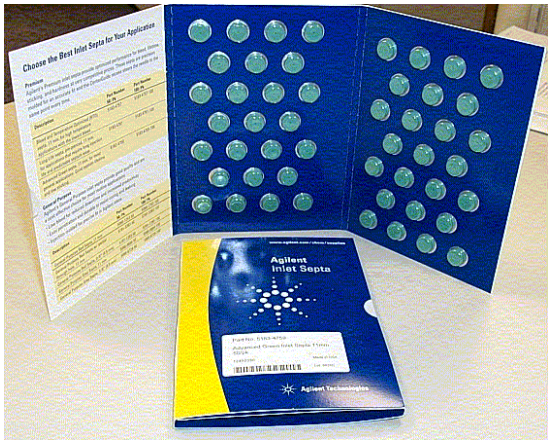
**Red: Hand Lotion**

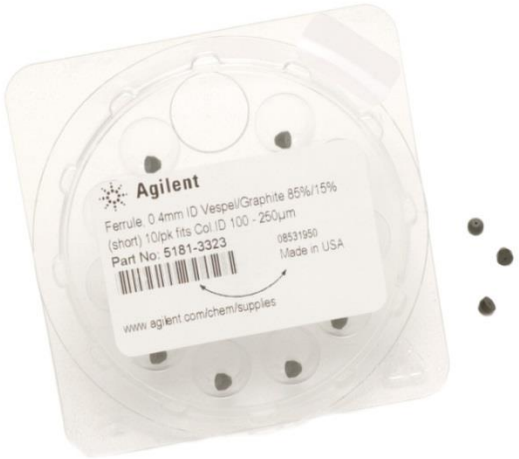
**Blue: system blank**

Procedure:

(1) One very small drop of hand lotion placed on one fingertip.
(2) Fingertip was wiped with paper towel to remove as much of the offending material as possible.
(3) Lightly touched the part of the column sticking up above the ferrule.
(4) Installed column into injector.
(5) Set oven temperature to 40C.
(6) Started oven temperature program as soon as oven reached 40C.

# More and More “Touchless” Packaging





# Troubleshooting “Tools”

**Bleed Profile:** ***baseline problems***

**Inject a non-retained peak:** ***peak shape problems***

**Leak Test:** ***all problems***

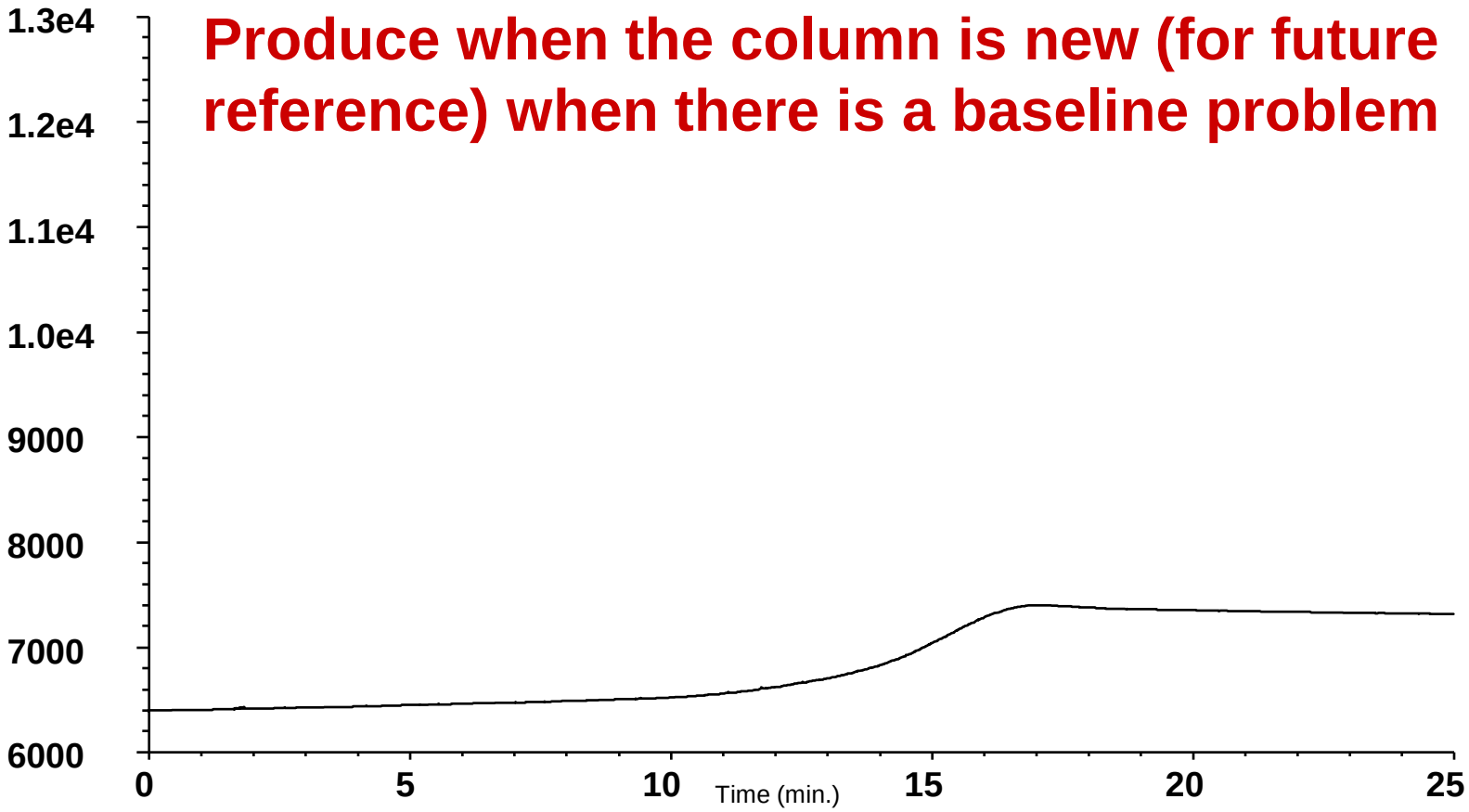
**Test mix:** ***all problems***

**Isolate the components:** ***all problems***

**Jumper Tube Test:** ***baseline problems***

# Generating a Bleed Profile

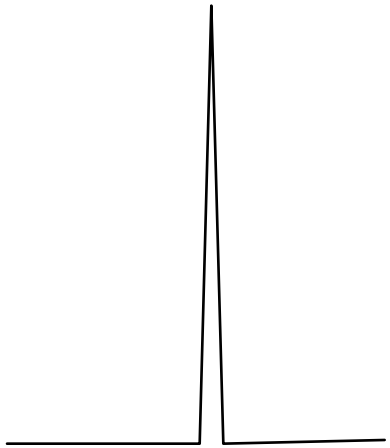
**Produce when the column is new (for future reference) when there is a baseline problem**



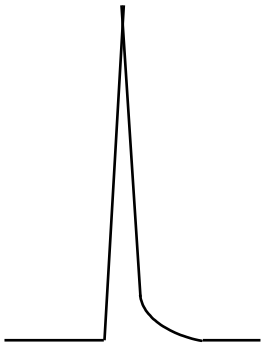

***DB-1 30m x .32mm I.D., .25µm**
**Temperature program // 40°C, hold 1 min // 20°/min to 320°C, hold 10 min.**

# Non-Retained Peak Shapes

**Used to Check Flowpath**



Good Installation



Improper Installation or Injector Leak

Potential problems:

- Injector or septum leak
- Too low of a split ratio
- Liner problem (broken, leaking, misplaced)
- Column position in injector and detector

# Leak Test


- MS – Air/Water

- Leak Detector (G3188B)
  - Portable, handheld unit – only 105g
  - USB cable for recharging
  - Audible and visual alerts for 12 gases
  - Minimum detection limit of 0.0005 mL/minute for hydrogen and helium
  - Rechargeable lithium ion battery with over 5 hours of life

# Ferrule Pre-swaging & MS Interface Installation Tools

- Ensures proper length of column into the fittings, every time
- For graphite or metal ferrules

Metal ferrule
G3440-80218


Graphite ferrule
G3440-80217

# Self-Tightening Column Nuts

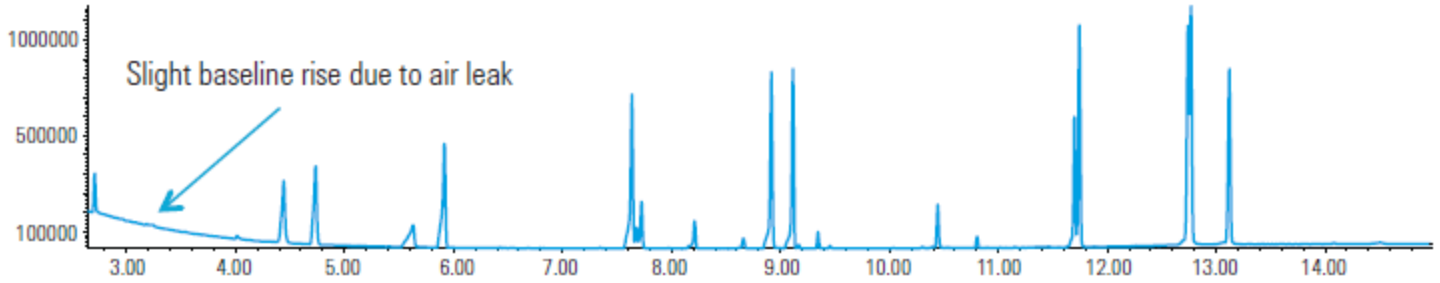
Innovative spring-driven piston continuously presses against ferrule

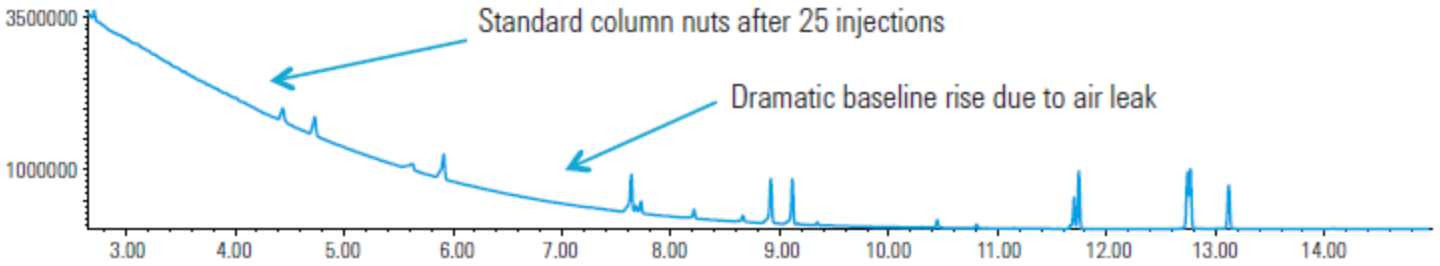
- **Less wasted time:** No retightening needed after repeated thermal cycles
- **Ease of use:** Finger-tight, consistent connections *without tools*
- **Leak Free = Lower column bleed:** Longer column life

Video at **agilent.com/chem/STnutvideo**

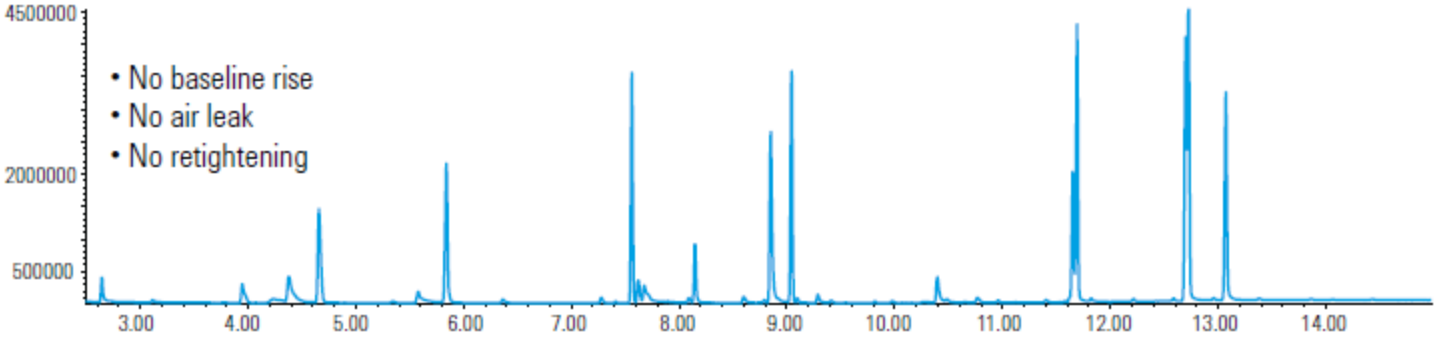
# Self-Tightening Column Nuts

**Standard column nuts new fitting**



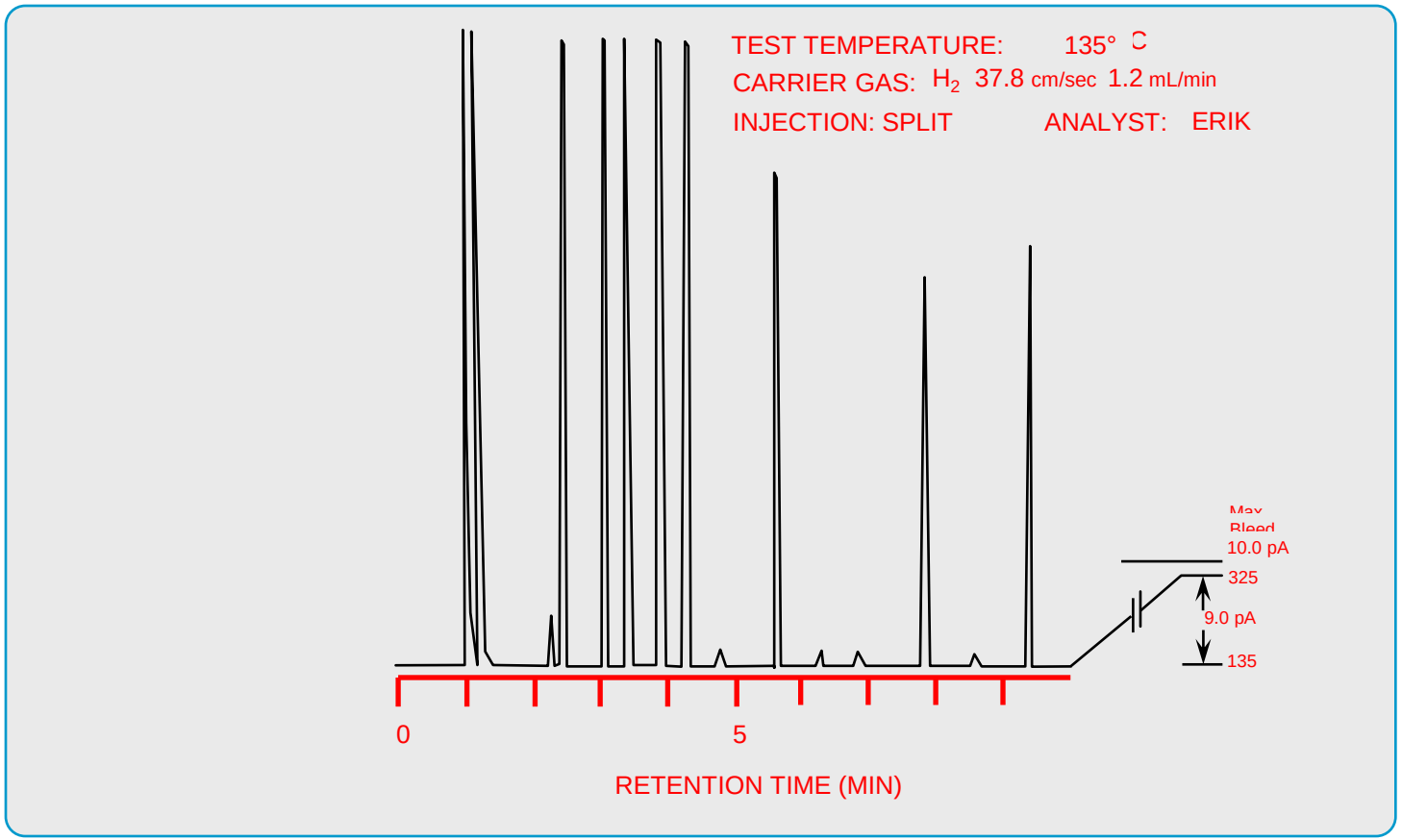




**Agilent Self Tightening Column Nuts after 400 injections**



# Test Mix

**Used to determine how “good” the column is or if the problem is related to the chemical properties of the analytes.**



# Test Mixture Components

| Compounds | Purpose |
| --- | --- |
| Hydrocarbons | Efficiency |
| | Retention |
| Alcohols | Activity |
| FAME's, PAH's | Retention |
| Acids | Acidic Character |
| Bases | Basic Character |

# Own Test Mixture

**More specific**

**Selective detectors**

**Actual concentrations**

**No conditions or instrument changes**

# Jumper Tube Test

## Purpose

**Helps to locate the source of contamination or noise**

**Isolates GC components**

# Jumper Tube Test

## Isolate the Detector

**Remove column from the detector**

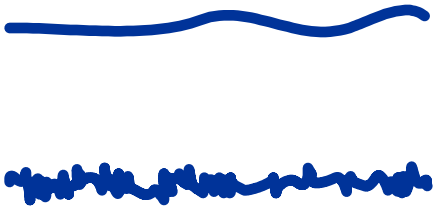
**Cap detector and turn on**

**Blank run**

# Jumper Tube Test

## Isolation of Detector - Results

**Detector OK**



**Detector is the problem**

# Jumper Tube Test

## Isolate the Injector

**Connect the injector and detector**

- **1-2 meters deactivated fused silica tubing**

**Turn on carrier gas**

**Blank run**

# Jumper Tube Test

## Isolate the Injector - Results

**Injector OK**

**Injector, lines or carrier gas contaminated**

# Jumper Tube Test

## Isolate the Column

**Reinstall the column**

**Setup as before**

**Blank Run**

# Jumper Tube Test

## Isolate the Column - Results

**Problem returns:  It's the column**

**Problem gone:  Previous leak, solid debris, or installation problem**

# Jumper Tube Test

## Used to Isolate Source of Contamination

- Cap off the detector and establish normal gas flows and temperature.
- Plot the baseline using a temperature program. If flat.....
- Connect 1 meter of deactivated tubing between the injector and detector
- Plot the baseline using a temperature program. If flat.....
- Install the column.
- Plot the baseline using a temperature program.

# Remember

Complete system = Carrier Gas + Injector + Column + Detector + Data System

**Multiple cause and effect**

**Do not change too many variables at once**

# Symptoms and Reasons (Cheat Sheet)

## *Solution = Get Rid of the Reason

- Peak Tailing – Flow Path or Activity
- Bonus Peaks – In Sample or Back Flash (Carry Over)
- Split Peaks – Injector Problems, Mixed Solvent, Cold Spots
- Broad Peaks – Injection Problems, Temperature/Flow, Solubility Problems
- No Peaks – Wasn't Introduced, Wasn't Detected
- Response Changes – Activity, Injector Discrimination, Detector Problem
- Peak Fronting – Overload or Solubility Mismatch, Injector Problems
- Shifting Retention – Leaks, Column Aging, Contamination or Damage
- Loss of Resolution – Separation Decreasing, Peak Broadening
- Baseline Disturbances – Column Bleed, Contamination, Electronics
- Noisy or Spiking Baseline – Electronics or Contaminated Detector
- Quantitation Problems – Activity, Injector or Detector Problems

# Troubleshooting Resources

## Online Troubleshooting and Maintenance Videos

http://www.chem.agilent.com/en-US/Technical-Support/Instruments-Systems/Gas-Chromatography/Pages/troubleshootingvideos.aspx

## GC Troubleshooting Guide

http://www.chem.agilent.com/en-US/Products-Services/Instruments-Systems/Gas-Chromatography/pages/gp6770.aspx

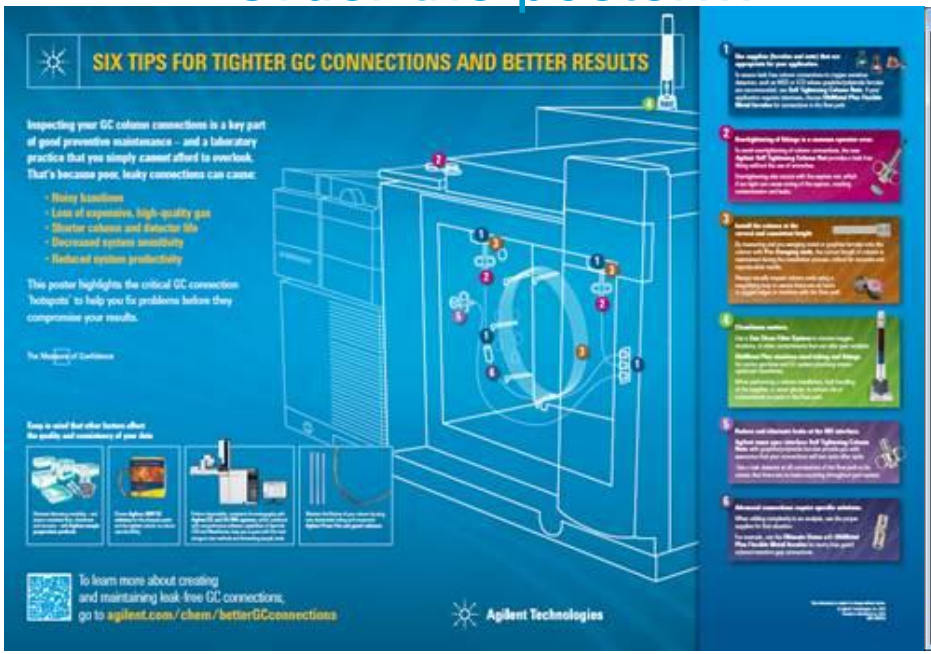
## Method Translation Software

http://www.chem.agilent.com/en-US/Technical-Support/Instruments-Systems/Gas-Chromatography/utilities/Pages/gcmethodtranslation.aspx

# Agilent Better GC Connections

## www.agilent.com/chem/betterGCconnections

Order the poster…




View the video…

# 52 Te 127.60 chnical 16 S 32.065 upport

# 1-800-227-9770, #3

**1 (972) 699-6423 (Daron)**

**1 (214) 883-2260 (Eric)**

**E-mail:**

**gc-column-support@agilent.com**

**Eric.pavlich@agilent.com**

**Daron_Decker@agilent.com**