Capillary Flow Technology

Improving Your GC/MS and GC Analyses with Agilent's Capillary Flow Technology

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IF We Only Had A Technology That Provided Easy, Reliable Flow Structures In The GC Oven...

It would open up many new (and old) capabilities for GC

- **Column connections** (connect pre-column)
- **Change MSD columns** (without venting)
- **Backflush** (Reverse flow through column)
- **Detector splitter** (effluent split to two or more detectors)
- **Merge flows** (2 columns to 1 MSD)
- **Dean switch** (heart cut select peaks to 2nd column)
- **Comprehensive 2-D GC** (cut all peaks to 2nd column)
- **etc.**

Types of Connectors Used In The GC Oven

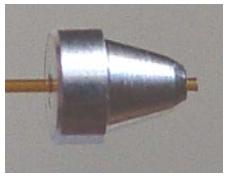
		Advantages	Limitations
Metal Fittings		**Packed columns, reliable**	**Not inert, no ferrule for capillary columns**
Press Fit Glass		**Low dead volume, inert, low cost**	**Difficult to assemble, comes apart**
Graphite		**High temperature**	**Sheds active graphite particles into sample path**
Polyimide		**Low initial leakage**	**Loosens and leaks with oven cycling, solvent tailing**

Challenges For Inside the Oven Devices

- **Inertness** (it is in the sample path)
- **Low dead volume** (it is in the separation path)
- **Leak free** (especially with repeated temp cycling)
- **Fast thermal response** (follow rapid oven ramping)
- **High temp tolerance** (GC oven can go over 350C)
- **Reliable and easy to use**

5 Key Developments in Capillary Flow Technology

Metal Ferrules



Easy to use, do not loosen or leak with oven cycling to 350°C

Manifold Plates

Complex flow structures with low thermal mass

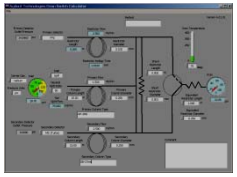
Deactivation of Metal

Makes metal surfaces as inert as column

EPC

Backflushing now possible, change MSD columns without venting, known column outlet pressure

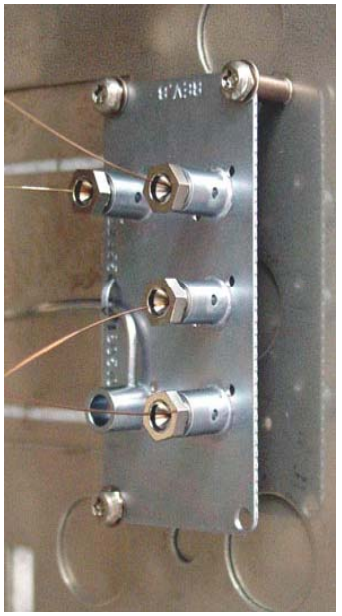
Calculators



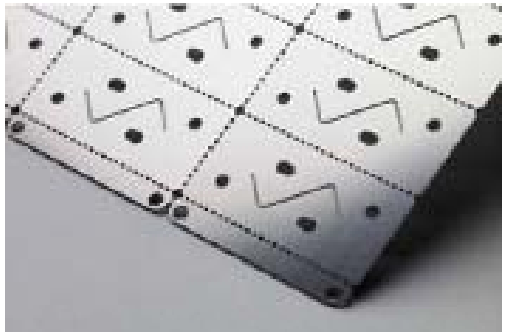
Accurately predict flows and pressures BEFORE installing devices

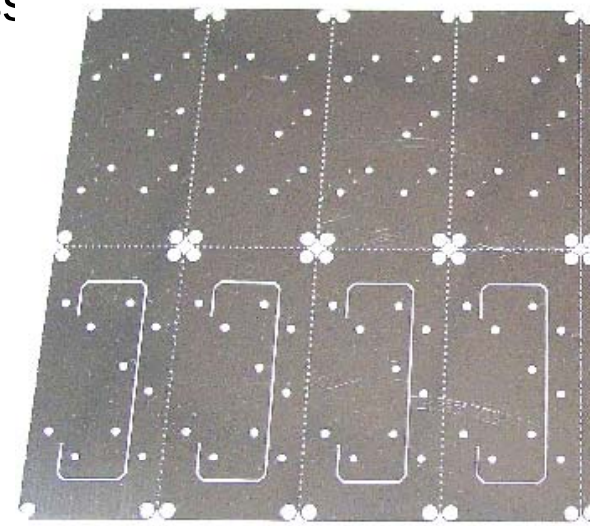
Capillary Flow Technology- Design

… a proprietary Agilent Technology



- Photolithographic chemical milling for low dead volume
- Diffusion bond two halves to form a single flow plate
- Small, thin profile provides fast thermal response
- Projection welded connections for leak tight fittings
- Deactivation of all internal surfaces for inertness

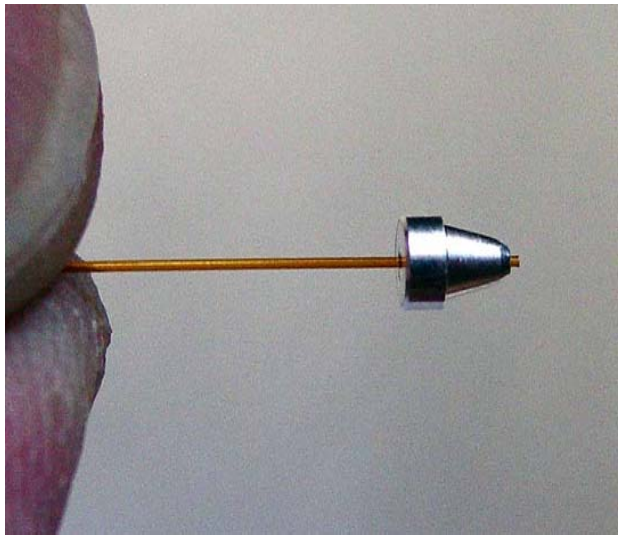


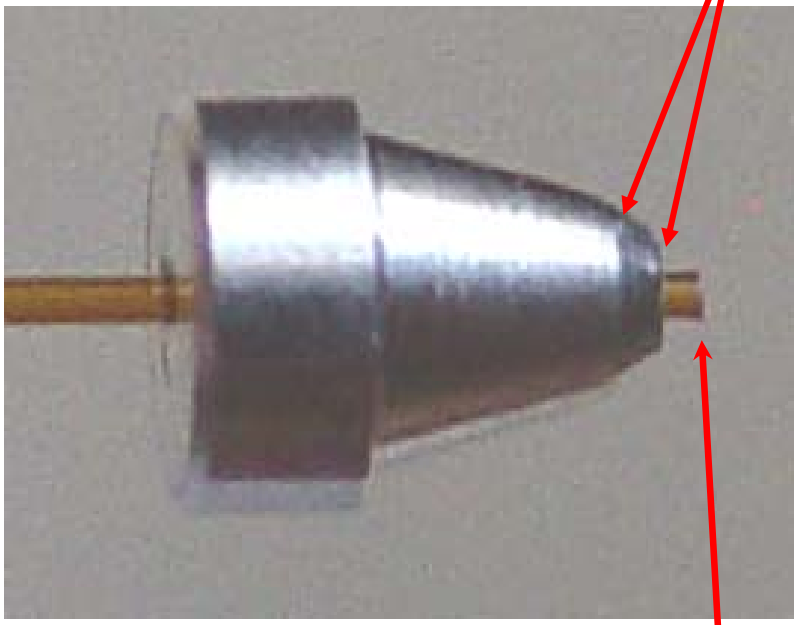


The Metal Ferrule

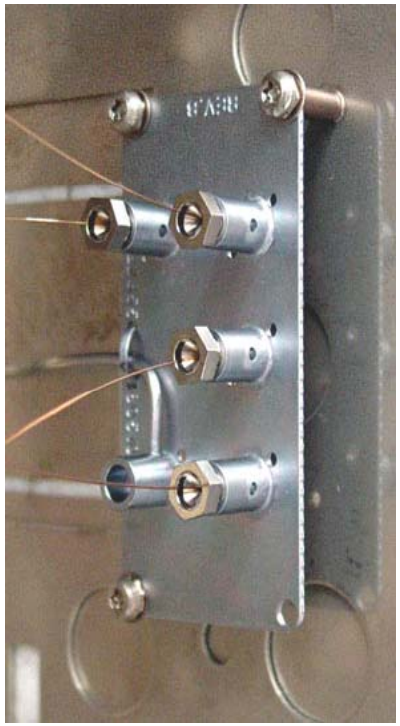
Does not loosen (leak) even with *thousands* of runs to 350C

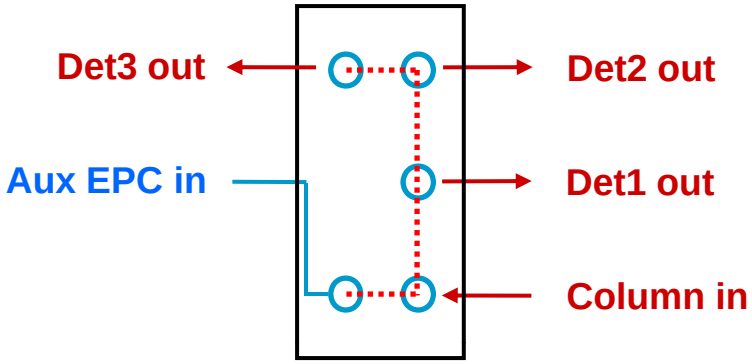
Does not shed particles

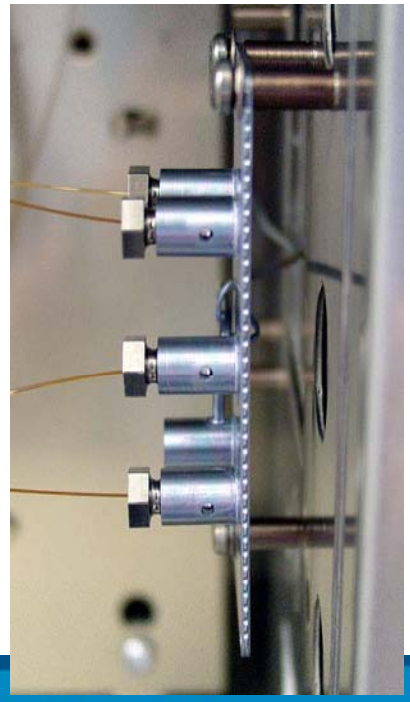




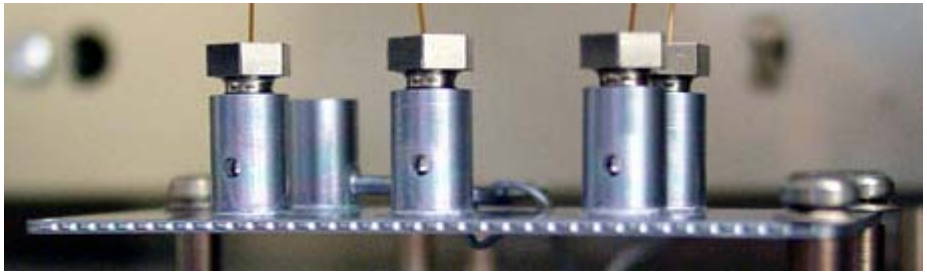
3-Way Splitter With Makeup

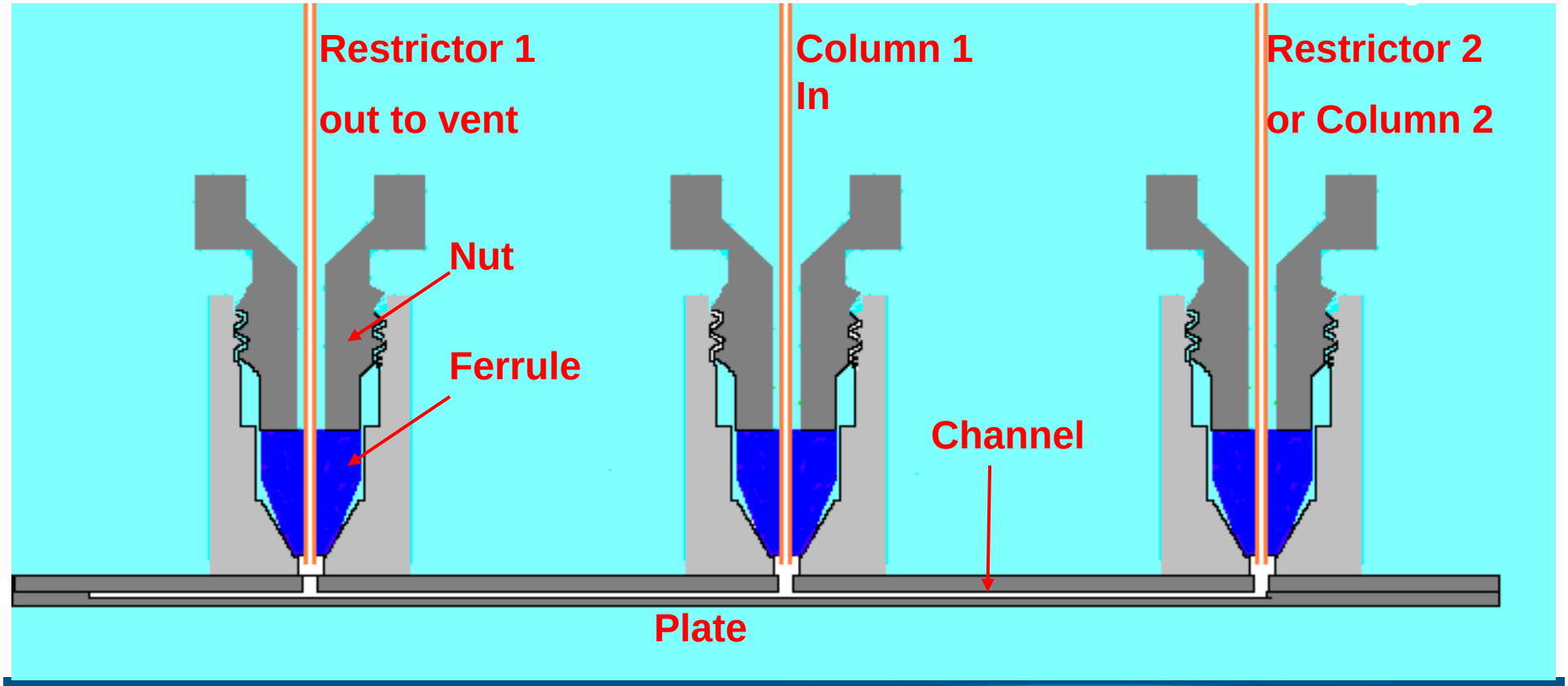






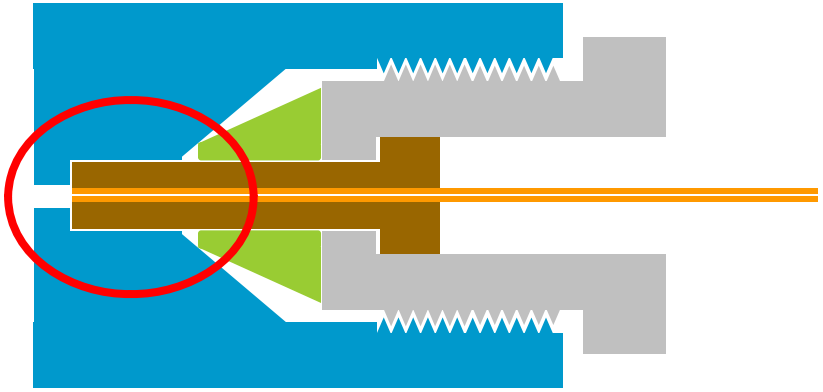
Capillary Flow Technology





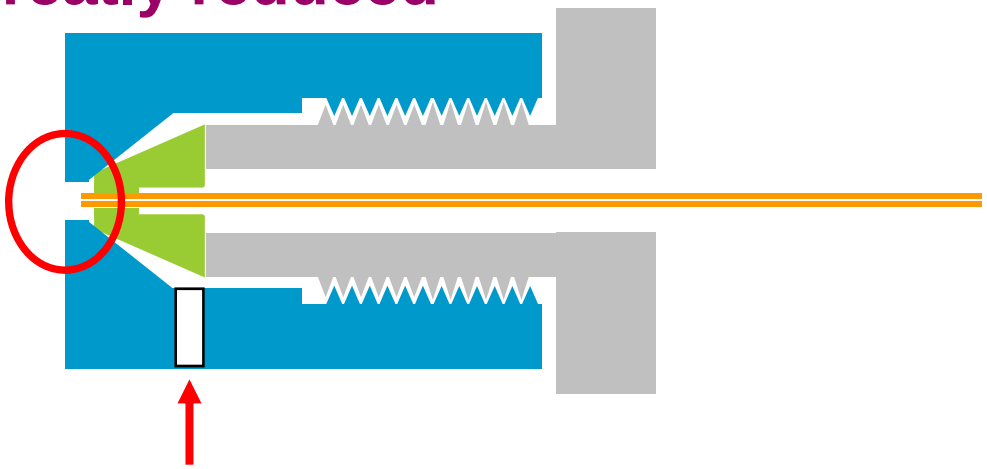
Comparison of New Fitting with Polyimide Fitting

Polyimide Fitting



Exposure to polyimide and unpurged annular spaces is greatly reduced

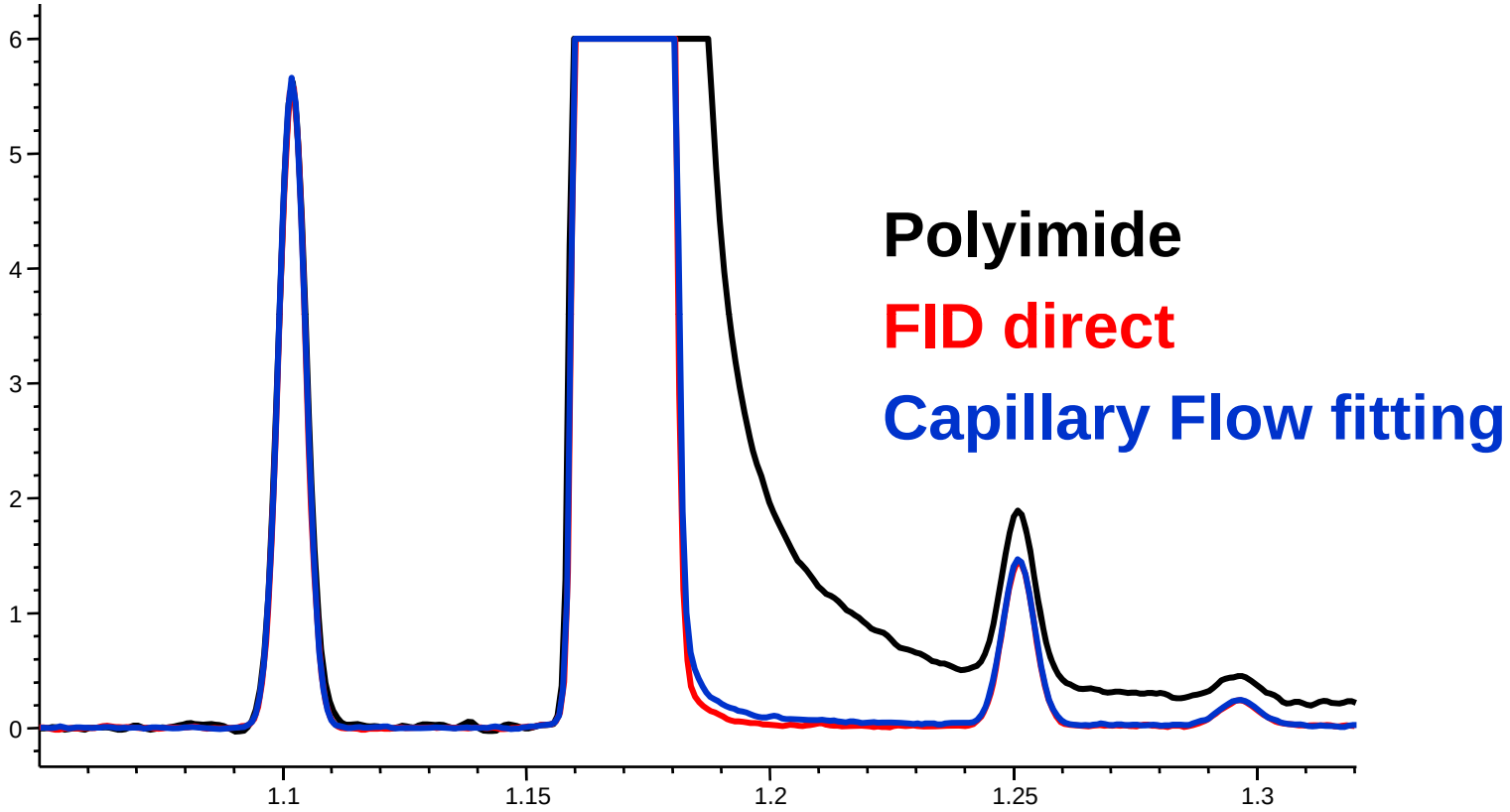
New Fitting



Ferrule Ejector Hole

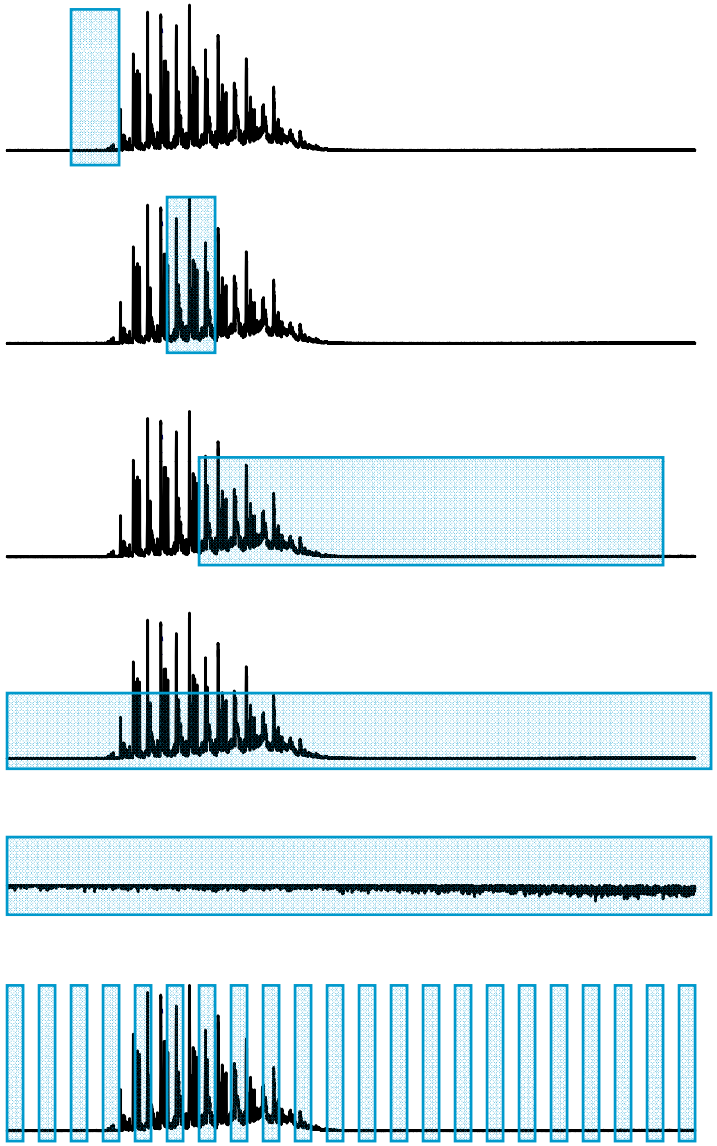
Fitting Design Minimizes Tailing

Pentane test chromatogram



Capillary Flow Technology fittings avoid tailing with small but well swept dead volume

Capillary Flow Technology- Capabilities



Solvent Bypass

Heart Cutting (Deans Switch)

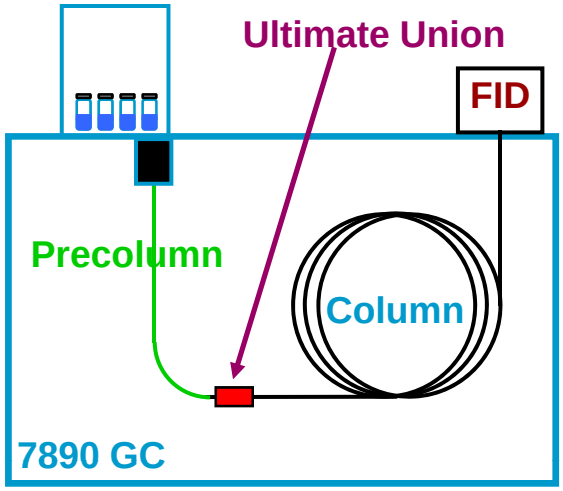
Backflush

Detector Splitting

QuickSwap

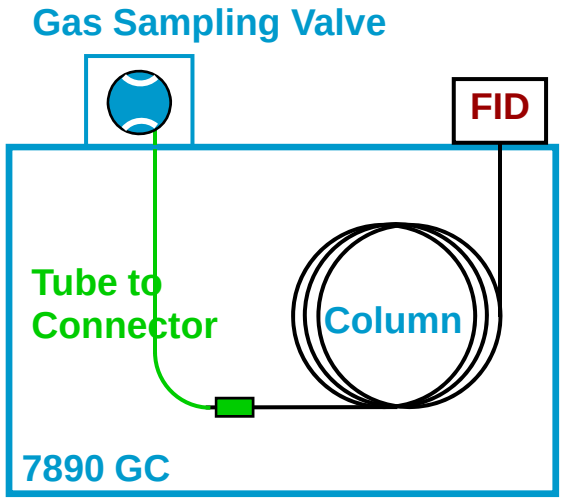
Modulation (GCXGC)

Capillary Flow Technology Devices



G3182 Ultimate Union

Reliable precolumn connector



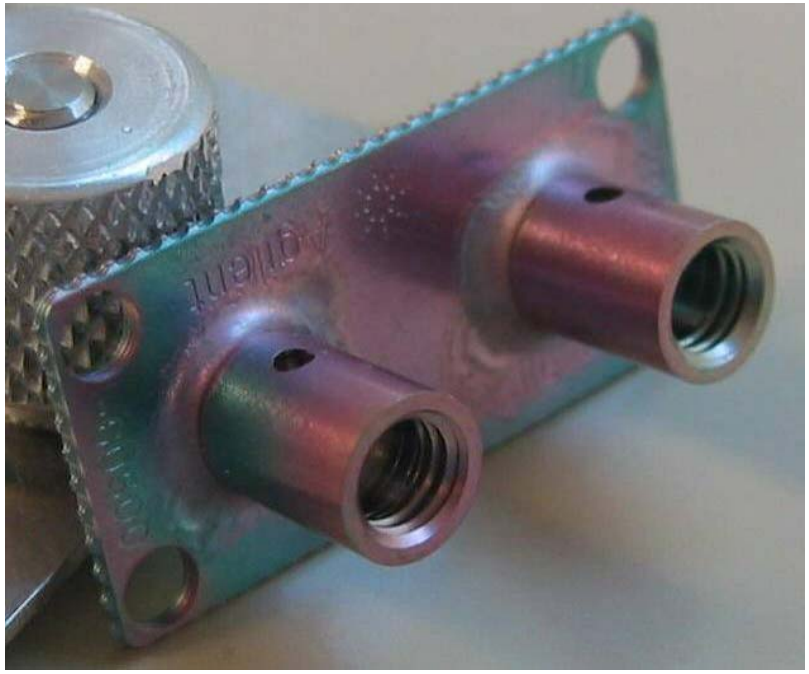
Tube Connector

Easy valve to capillary column connector

Tube is 0.25 mm id and is deactivated

Union and Tee On Wall Mounted Plate

G3187 Union-on-Plate wall mounted with both column fittings facing toward the column basket.

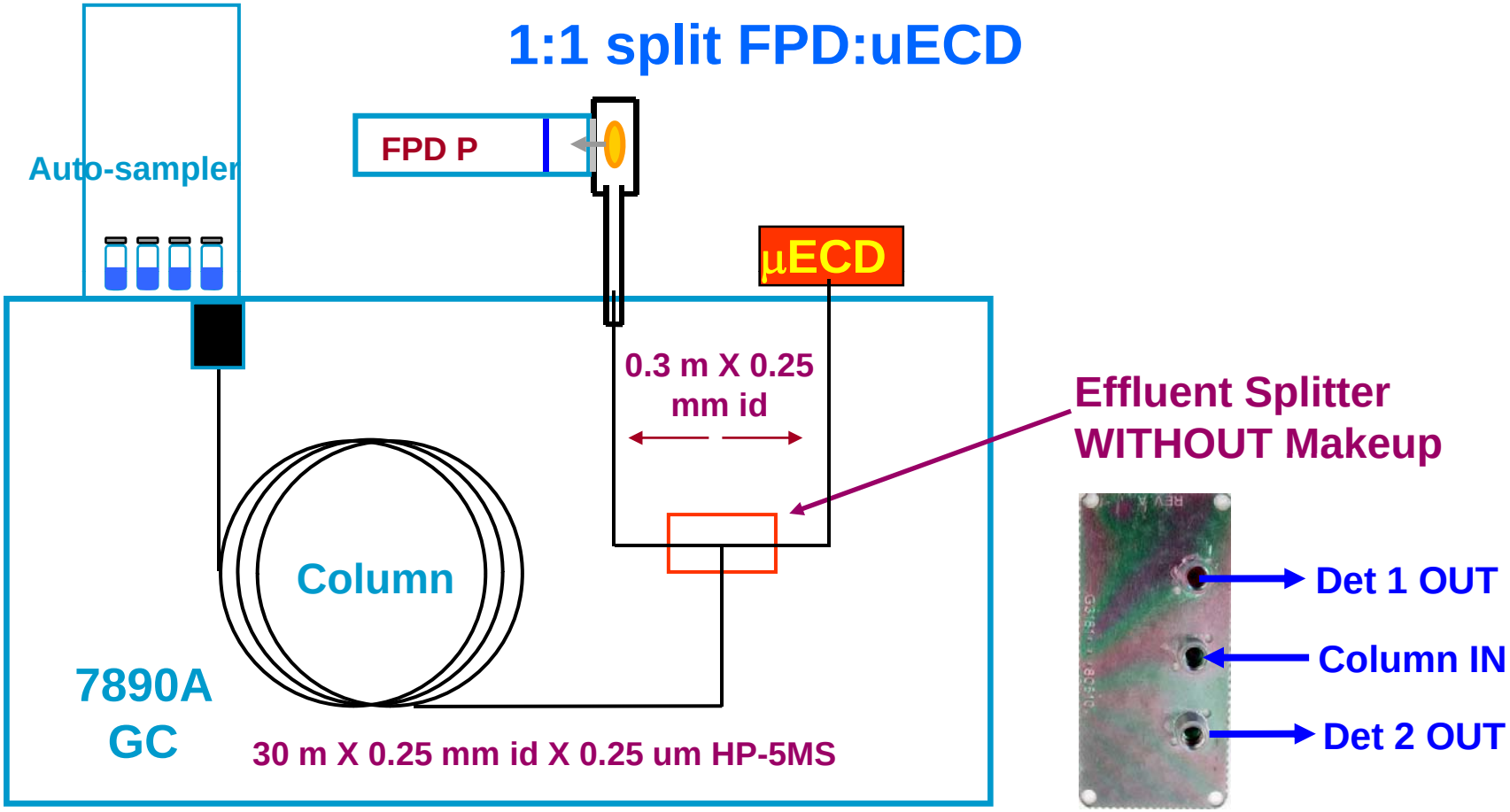


G3181B Tee-on-Plate wall mounted with three column fittings facing toward the column basket.

Splitters: Unpurged Tee

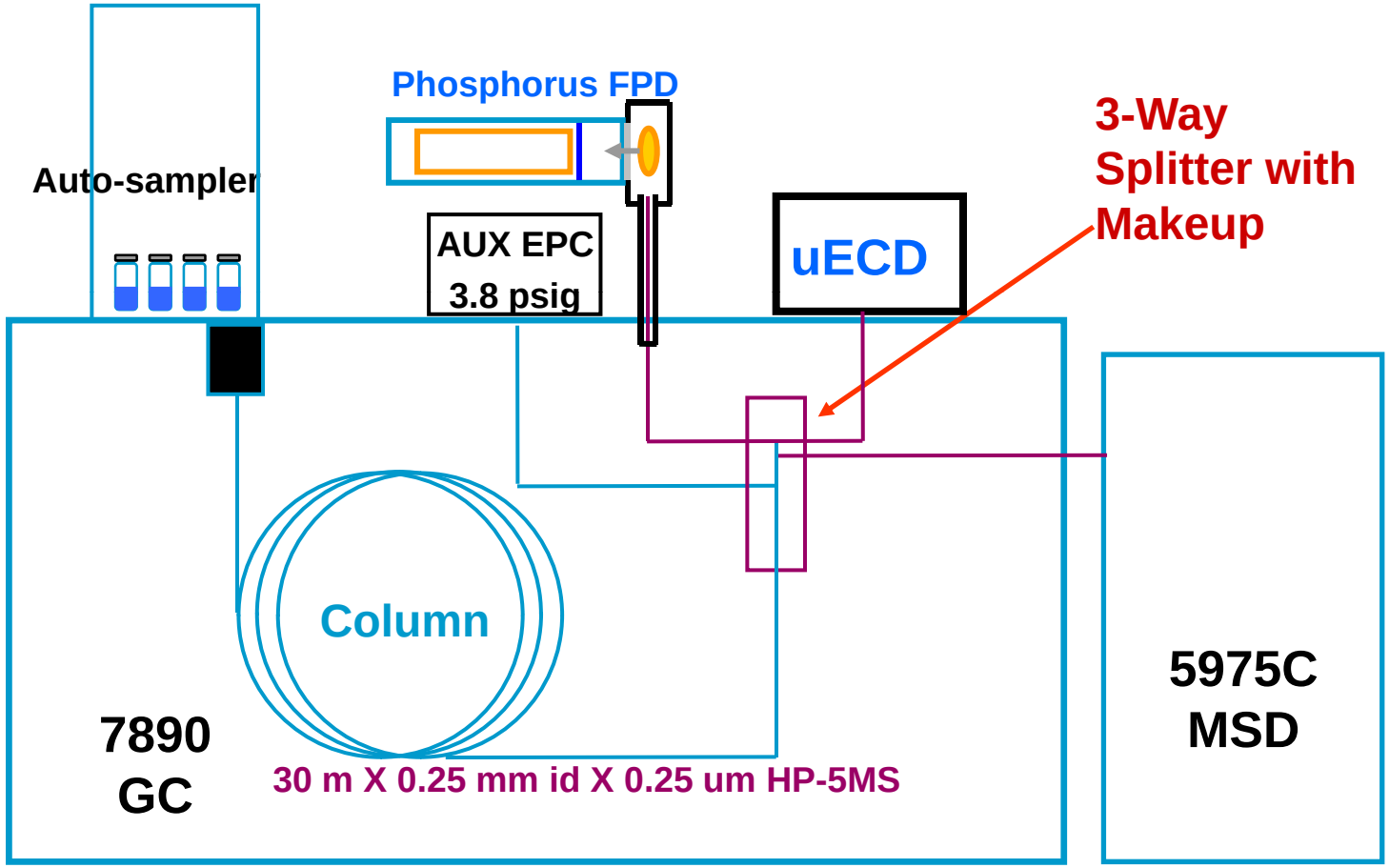
Simultaneous detection with 2 detectors (but NOT MSD)

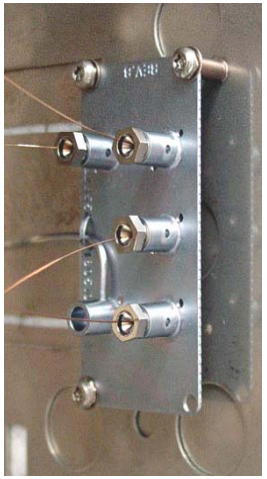
Cannot do backflushing



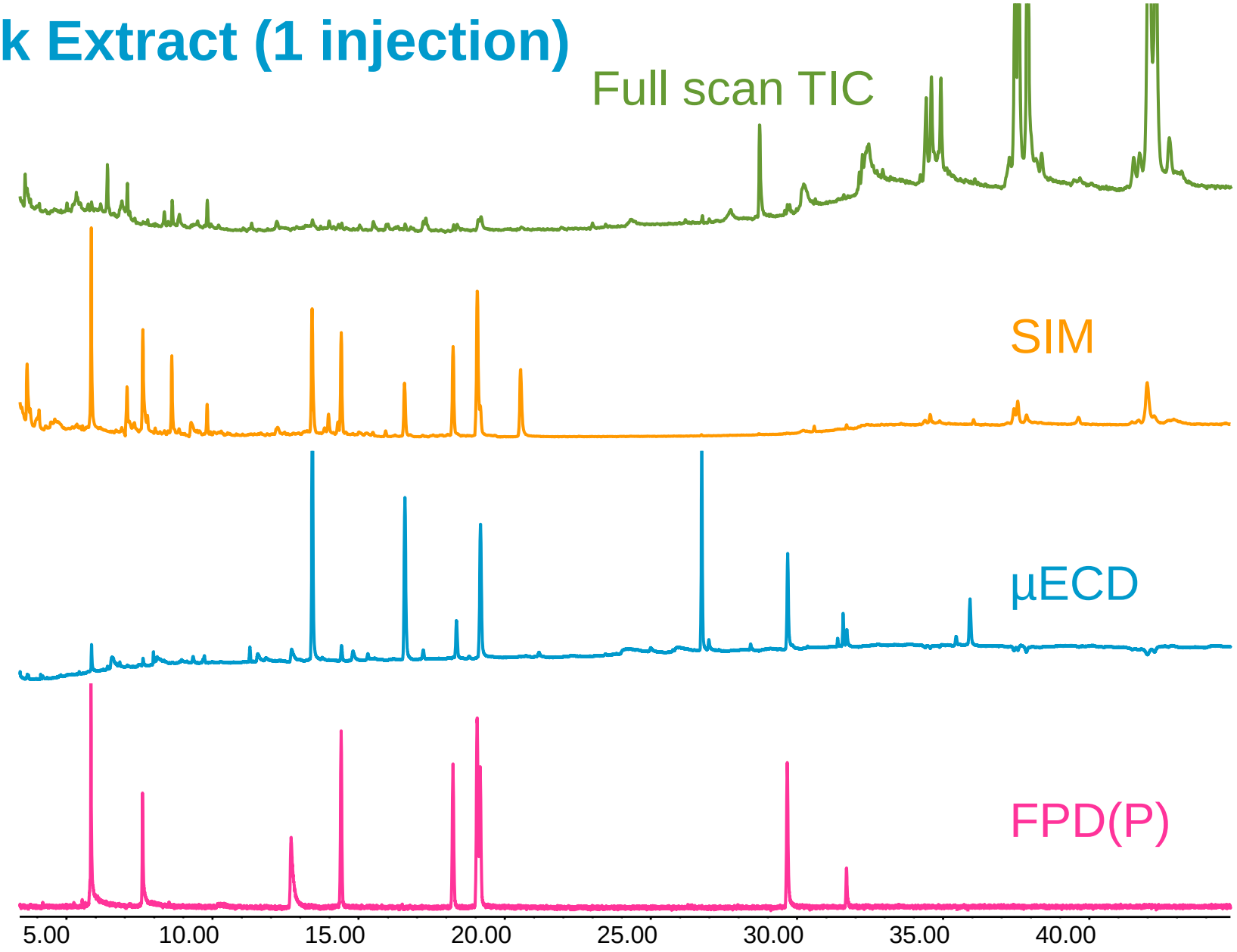
Pesticides: Three Way Splitter with Makeup

1X method with 1:1:0.1 split FPD:MSD:ECD

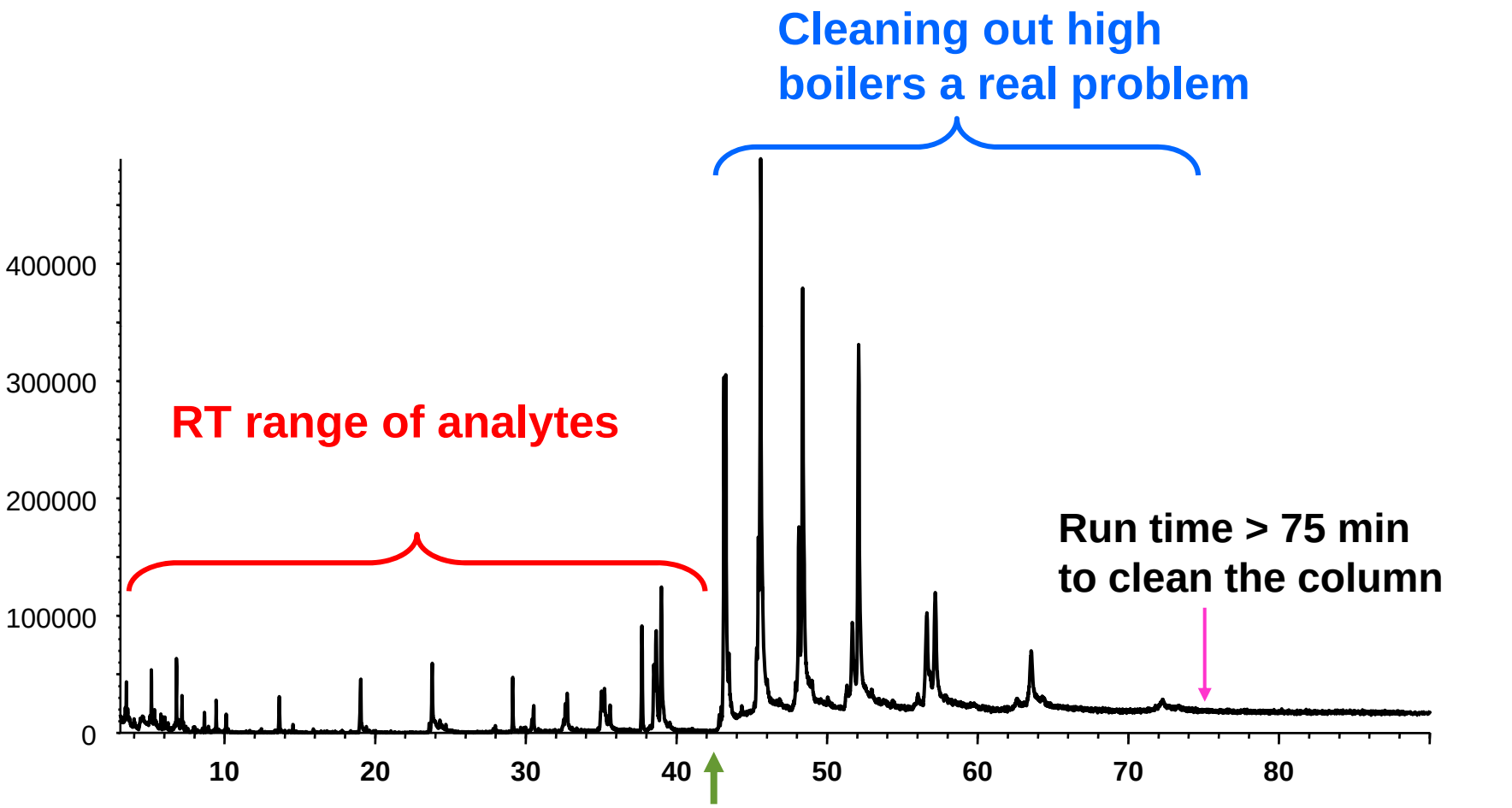




Milk Extract (1 injection)



Milk Extract (MSD TIC)

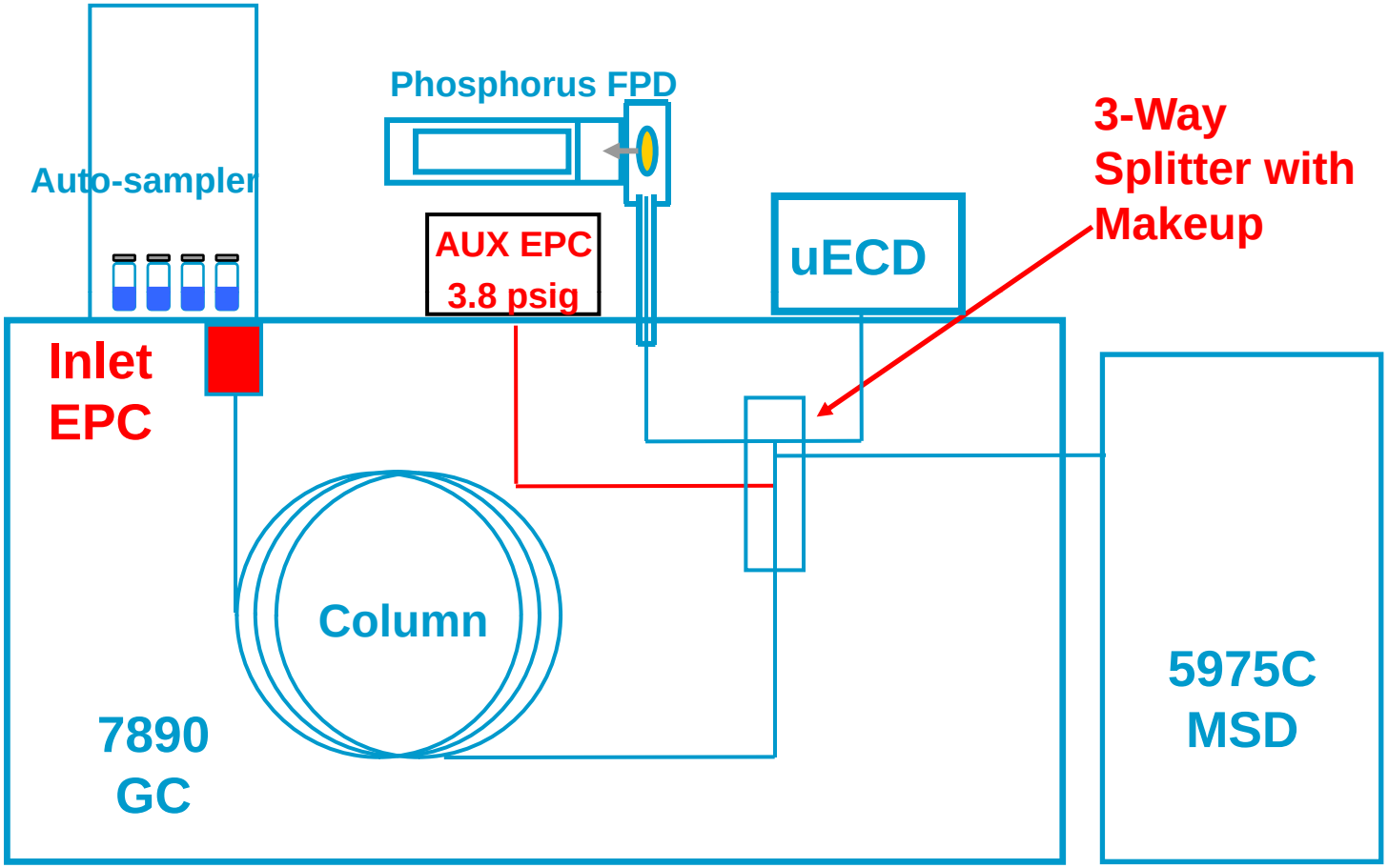


Problems With High Boilers

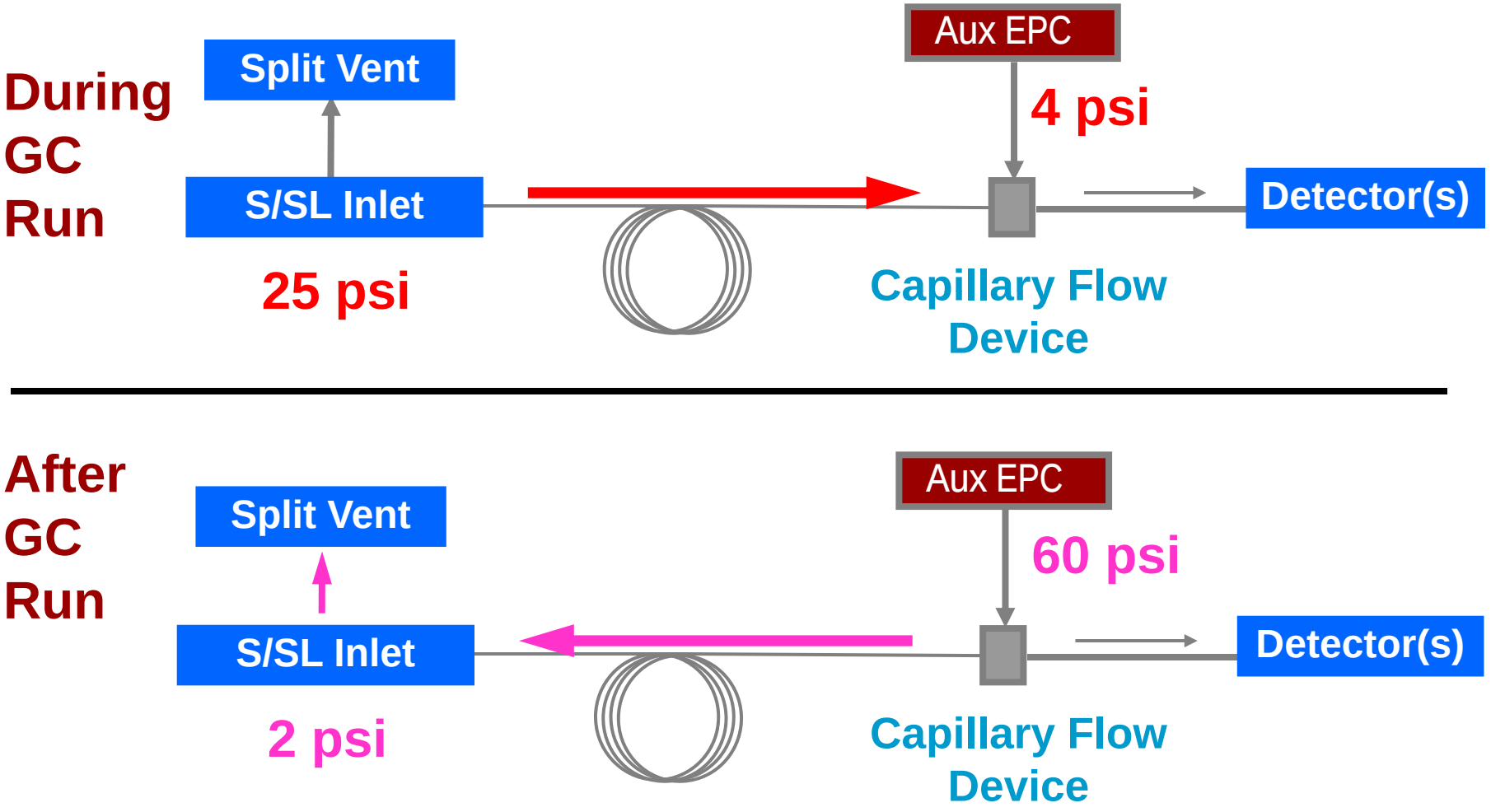
- Fewer samples/day/instrument because of required long bakeouts
- Bakeouts must be long enough for worst case samples
- More chemical background due to ghost peaks from previous injections
- More frequent column head trimming
- Shorter column life from cutting and roasting column
- More frequent MSD source cleaning from high boilers *and* column bleed bakeout
- Higher operating costs

We Have Control Of *Both* The Inlet And The Outlet Pressure Of The Column

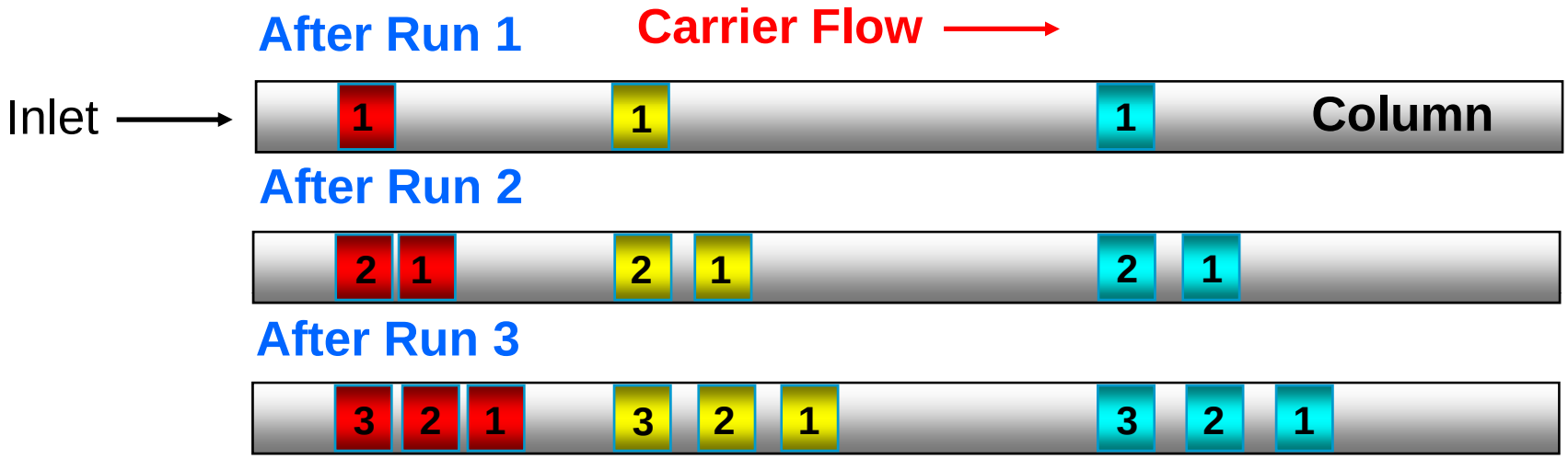
We can reverse the column flow during bakeout



Principle Of Backflushing



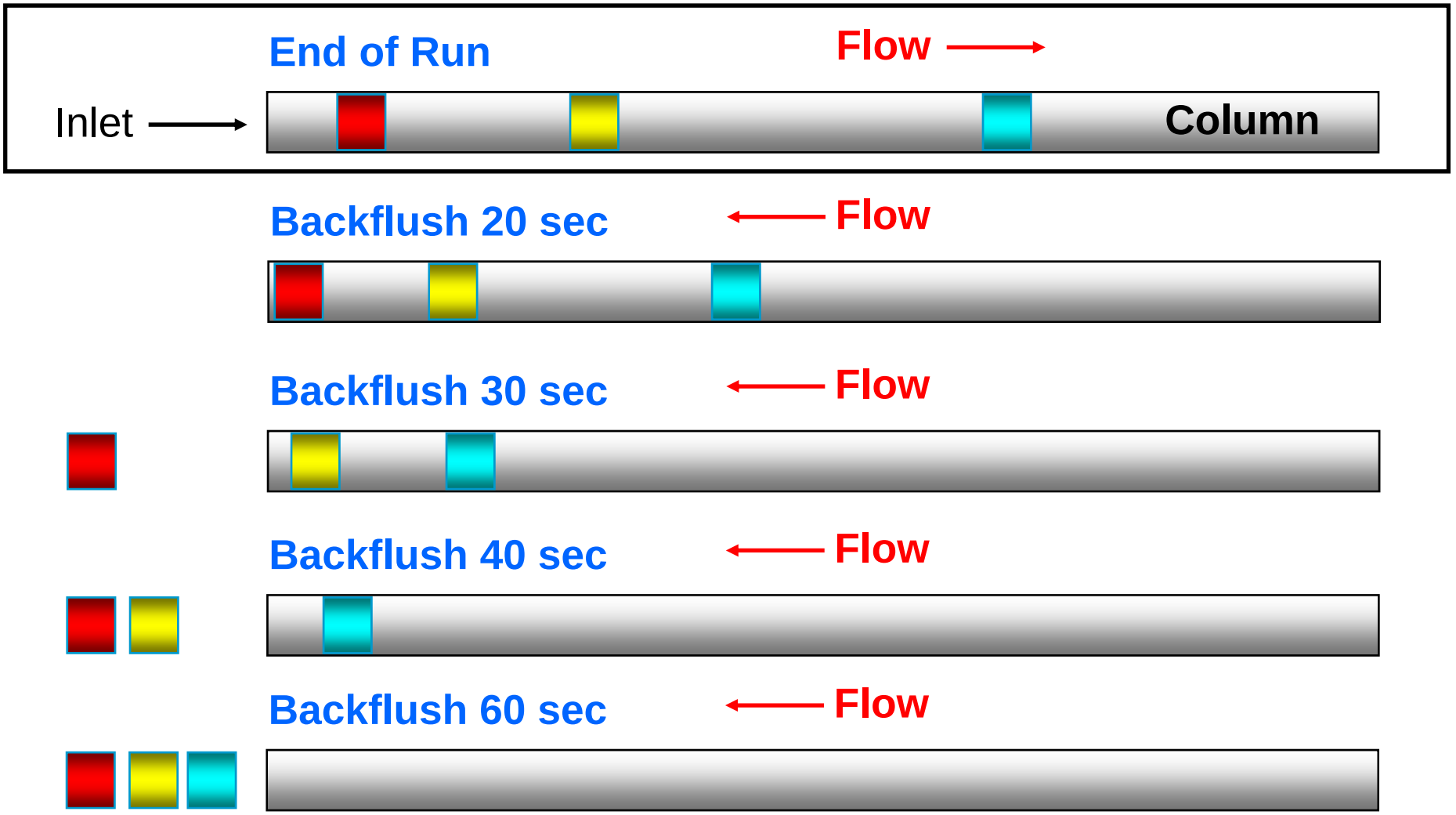
Heavy Compounds May Be Left in Head of Column After Each Injection



These heavy materials build up and travel further into the column with each injection.

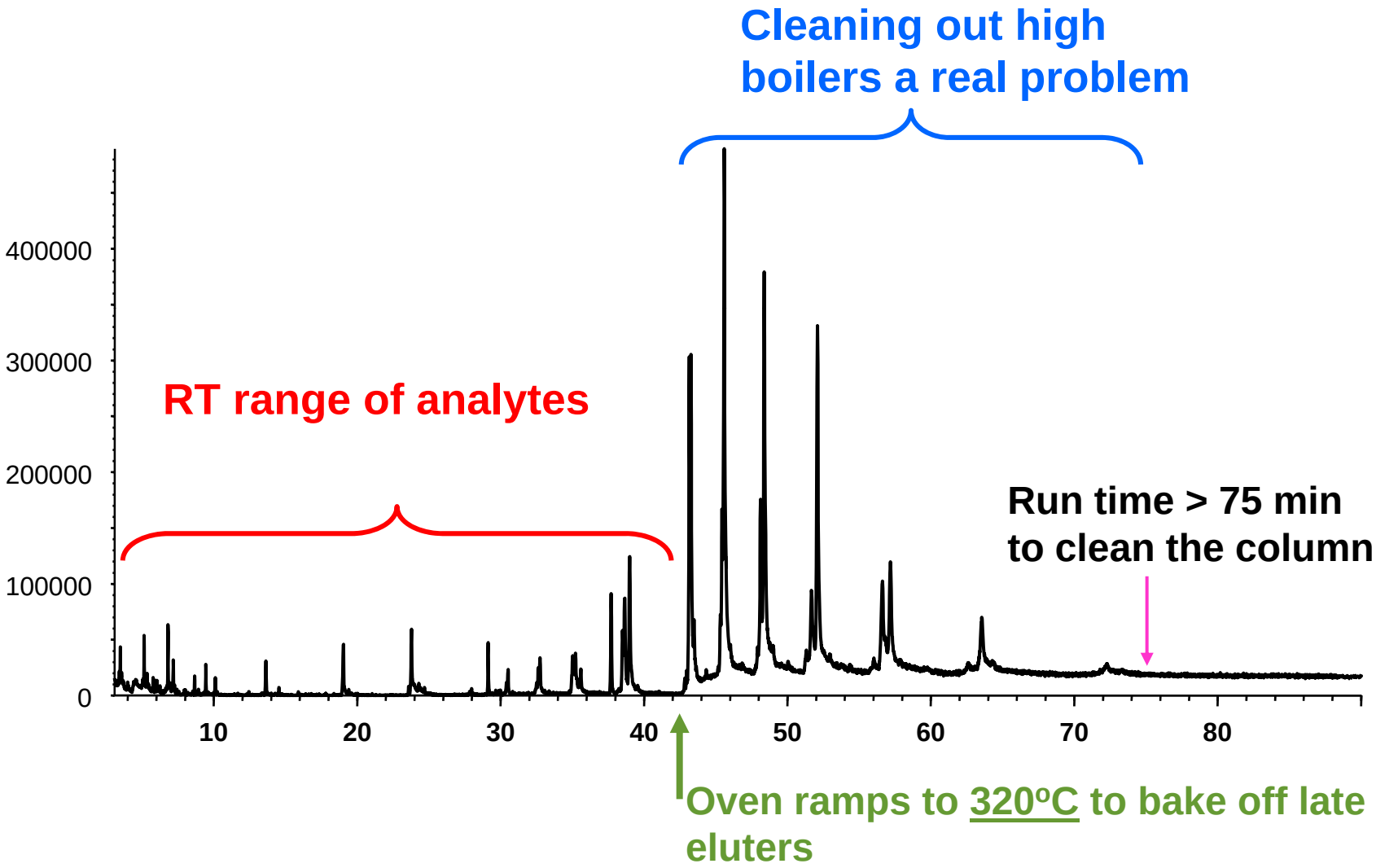
This buildup of heavy materials causes retention time shifts, peak distortion, higher bleed, and loss of sensitivity

Backflushing After Each Injection



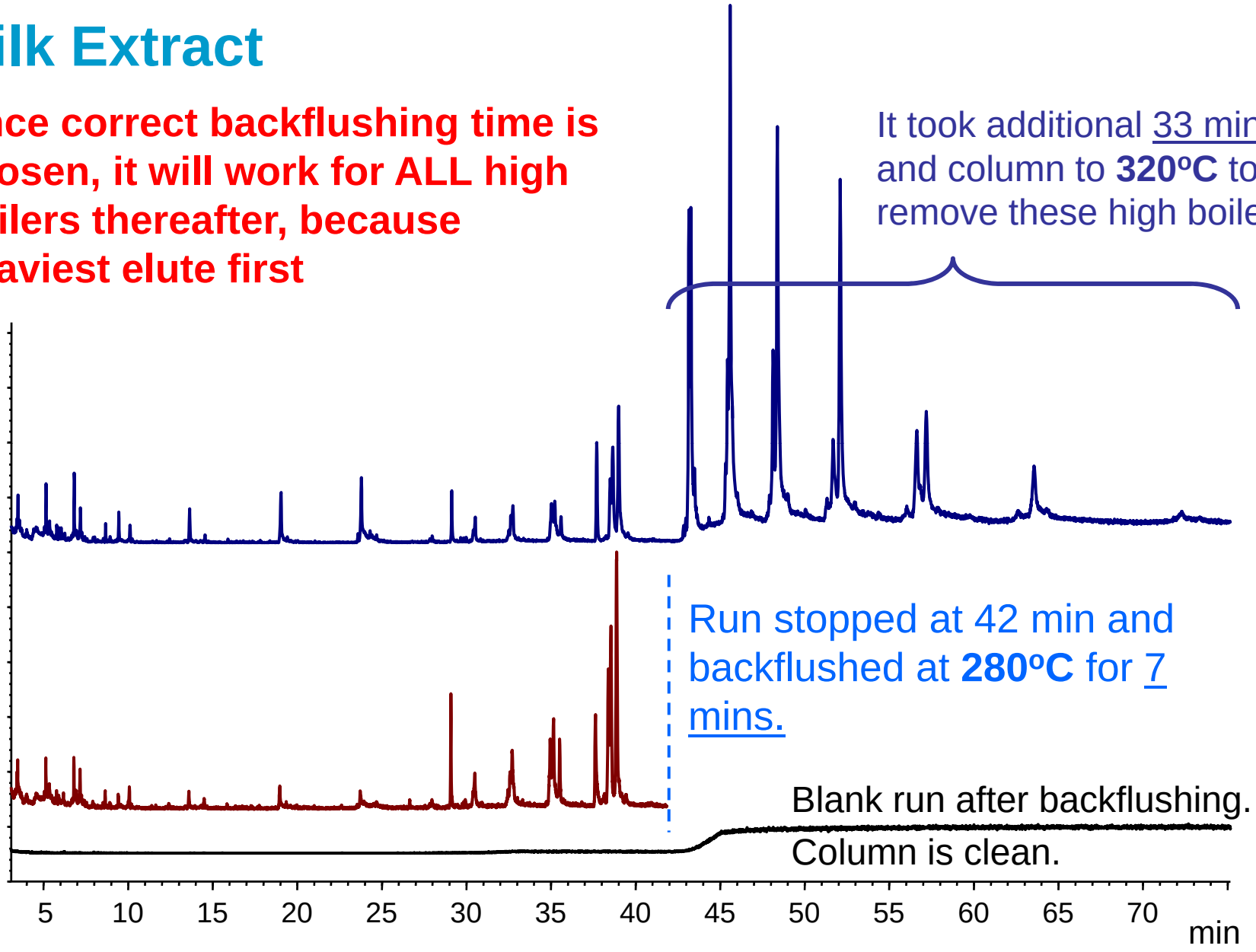
Backflushing removes heavy materials after each injection.

Milk Extract Using Bakeout To Remove High Boilers

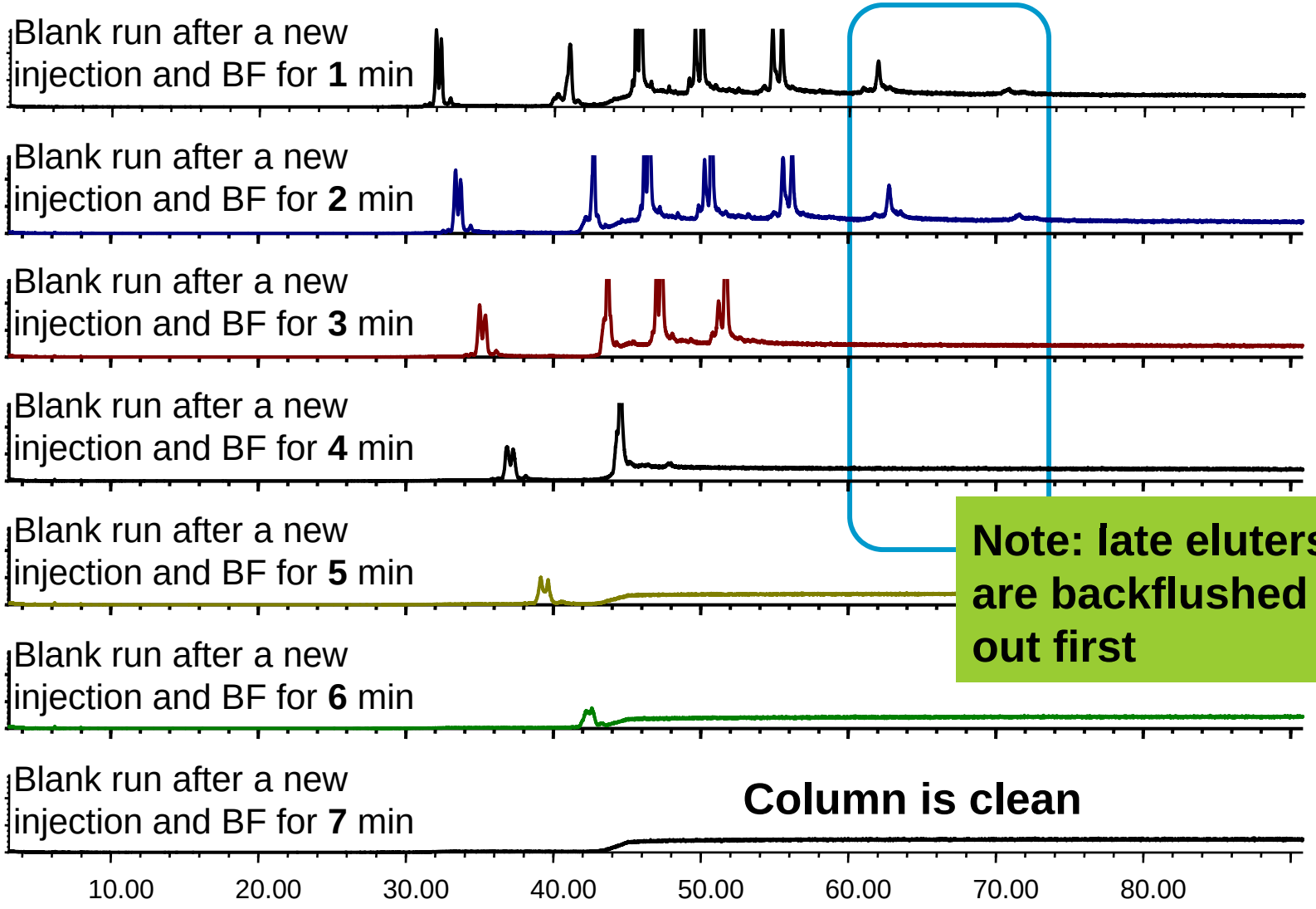


Milk Extract

Once correct backflushing time is chosen, it will work for ALL high boilers thereafter, because heaviest elute first



Blank Runs After Backflushing @ 60 psi and 280C



Note: late eluters are backflushed out first

Backflushing Example 2: Grapefruit Oil

Instrument Configuration: Same, but with 10m column

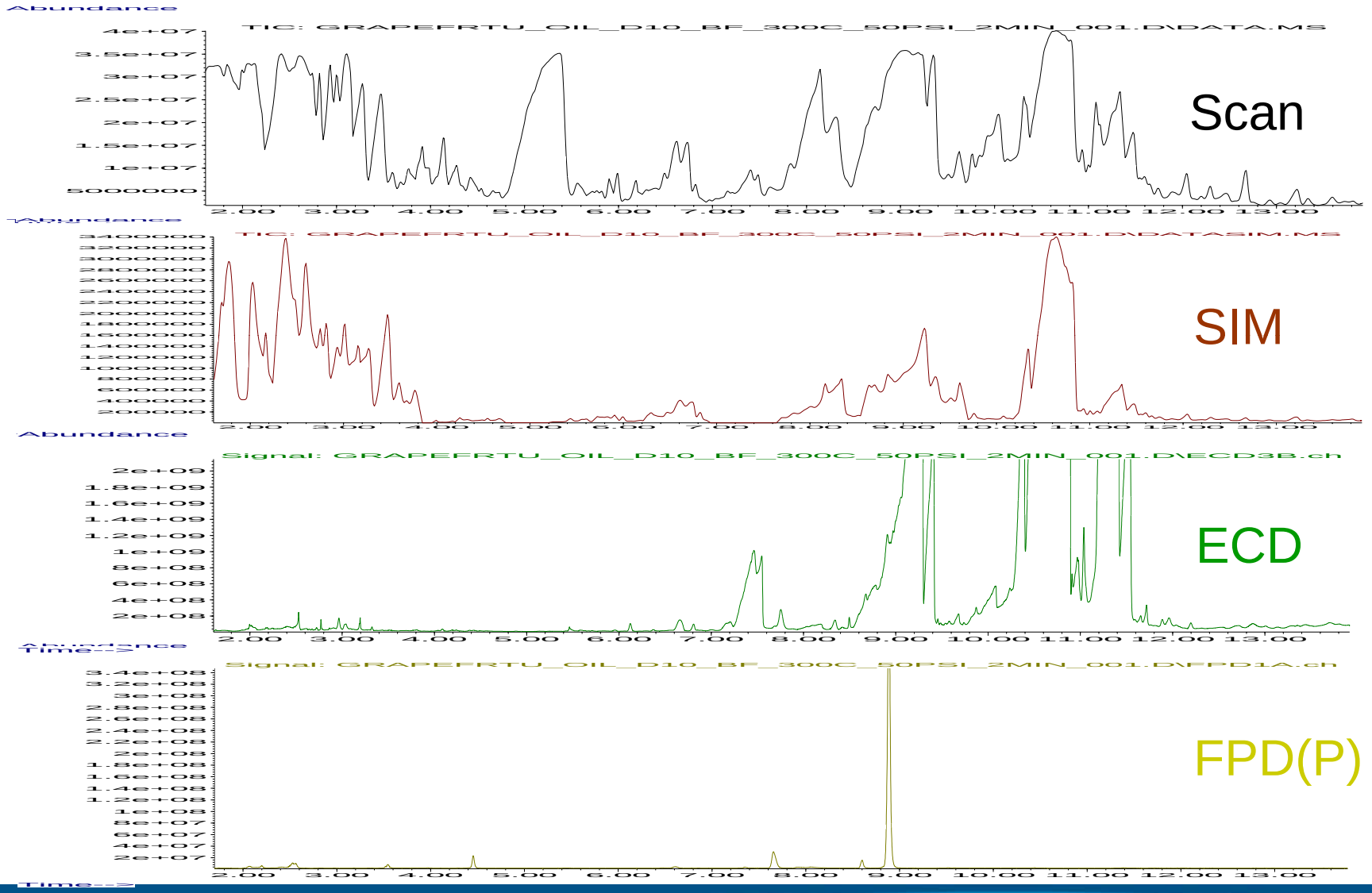
Sample: 10 Fold Cold Pressed Grapefruit Peel Oil

Dilute 10 to 1 with Acetone

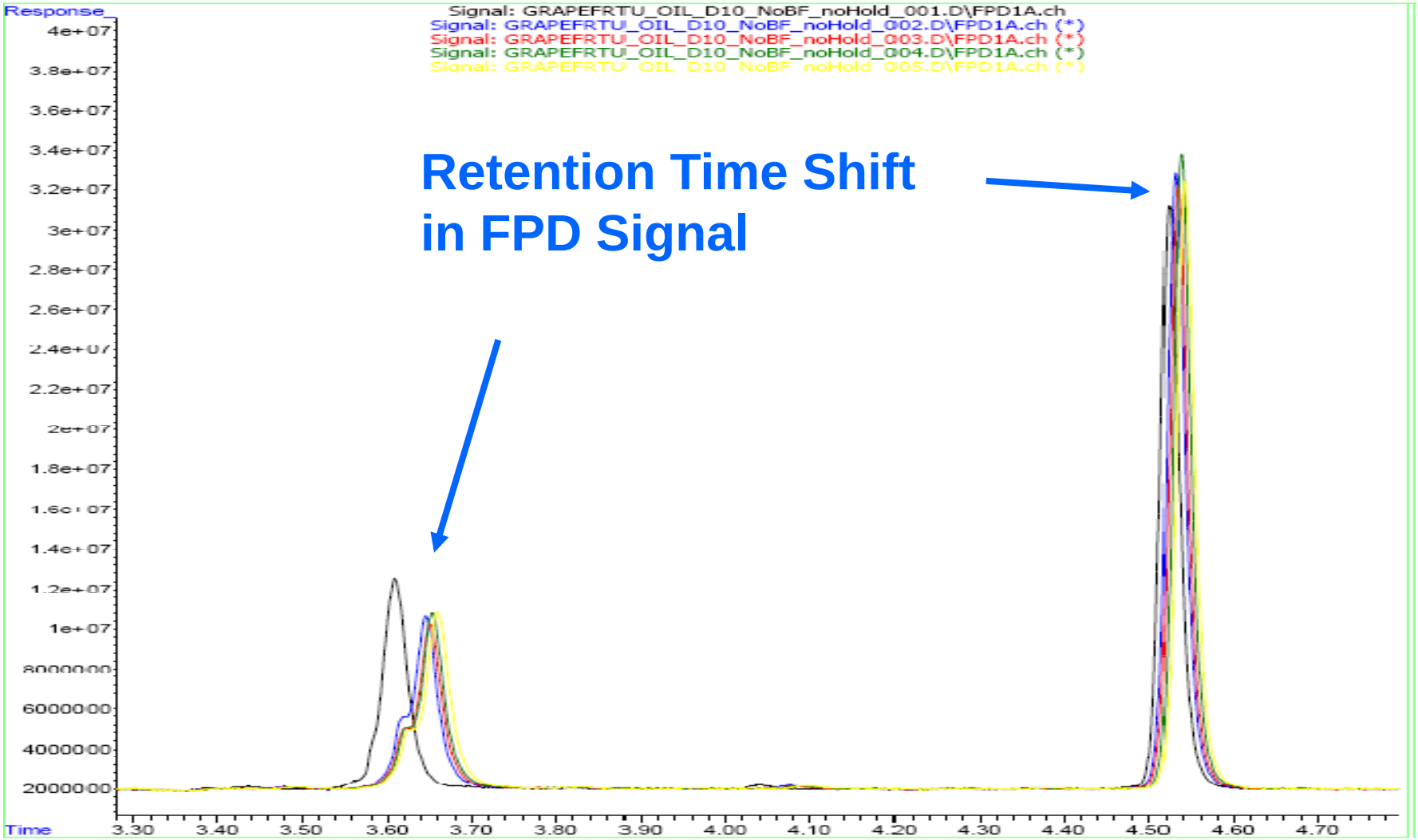
Problem: Heavy oil compounds deposit at head of column

- Requires excessively long post run bake time
- High backgrounds after running samples
- Retention times start to shift immediately

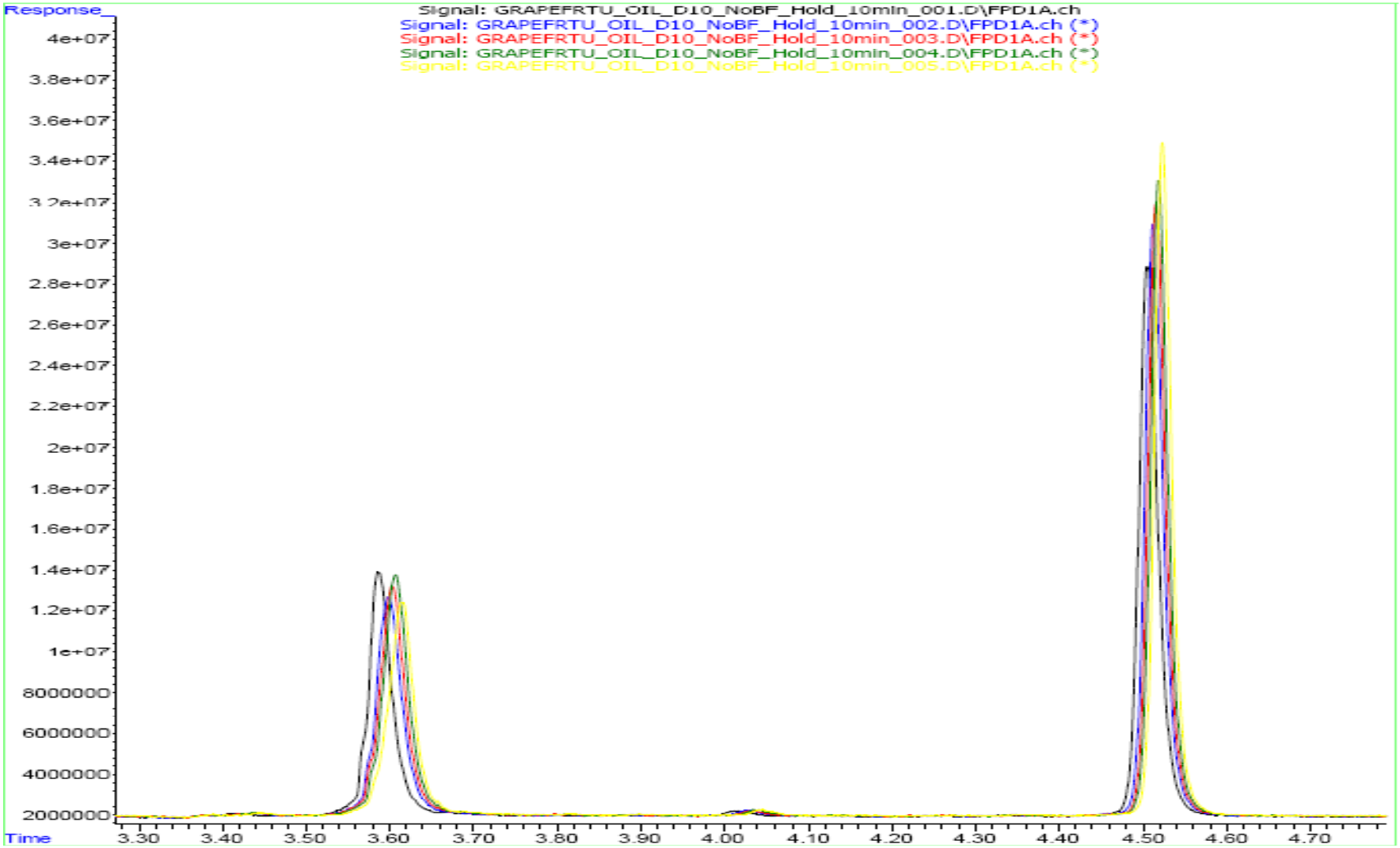
Grapefruit Oil: Very Bad Matrix Effects



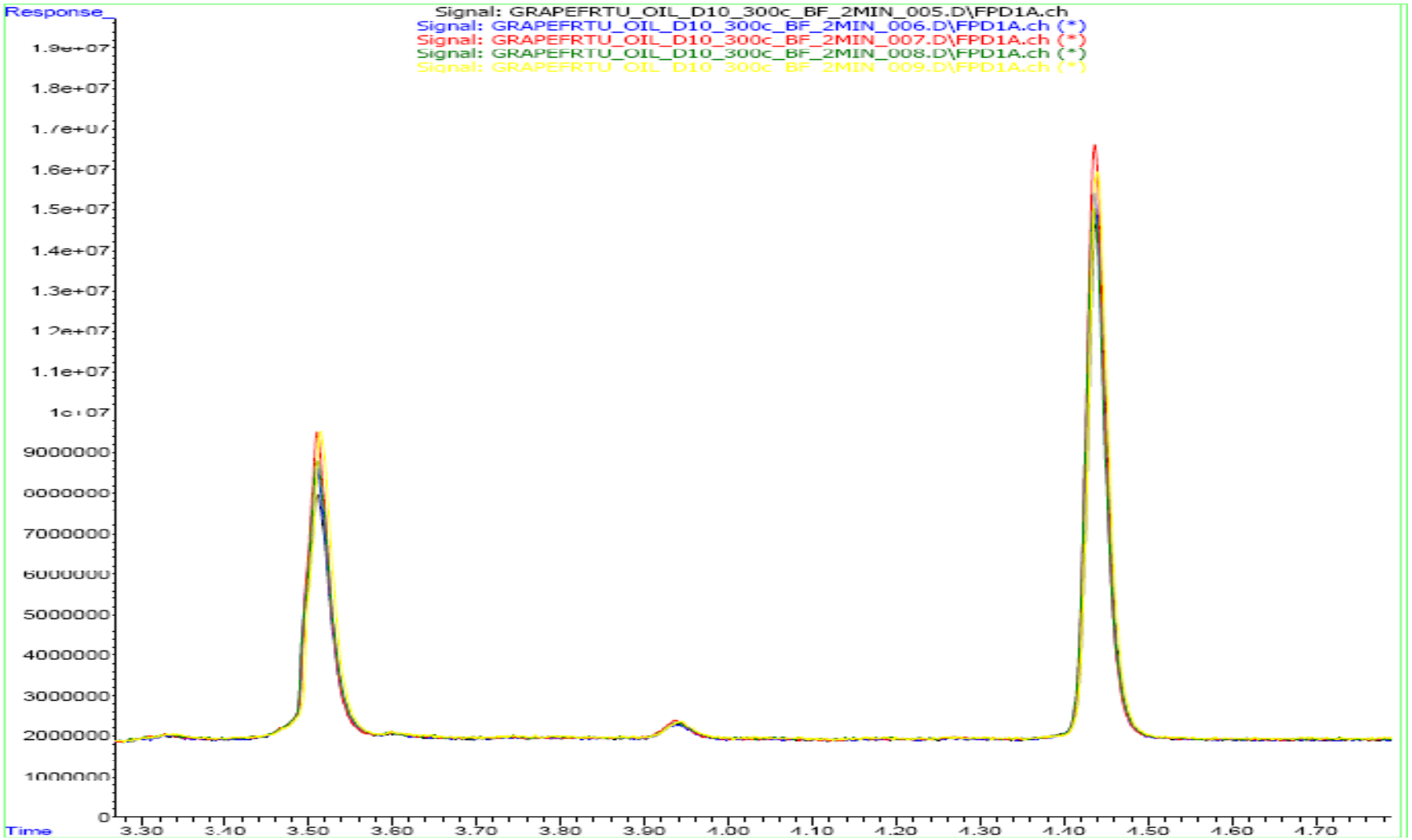
5 Injections With Original Method: No Bakeout Or BackFlushing



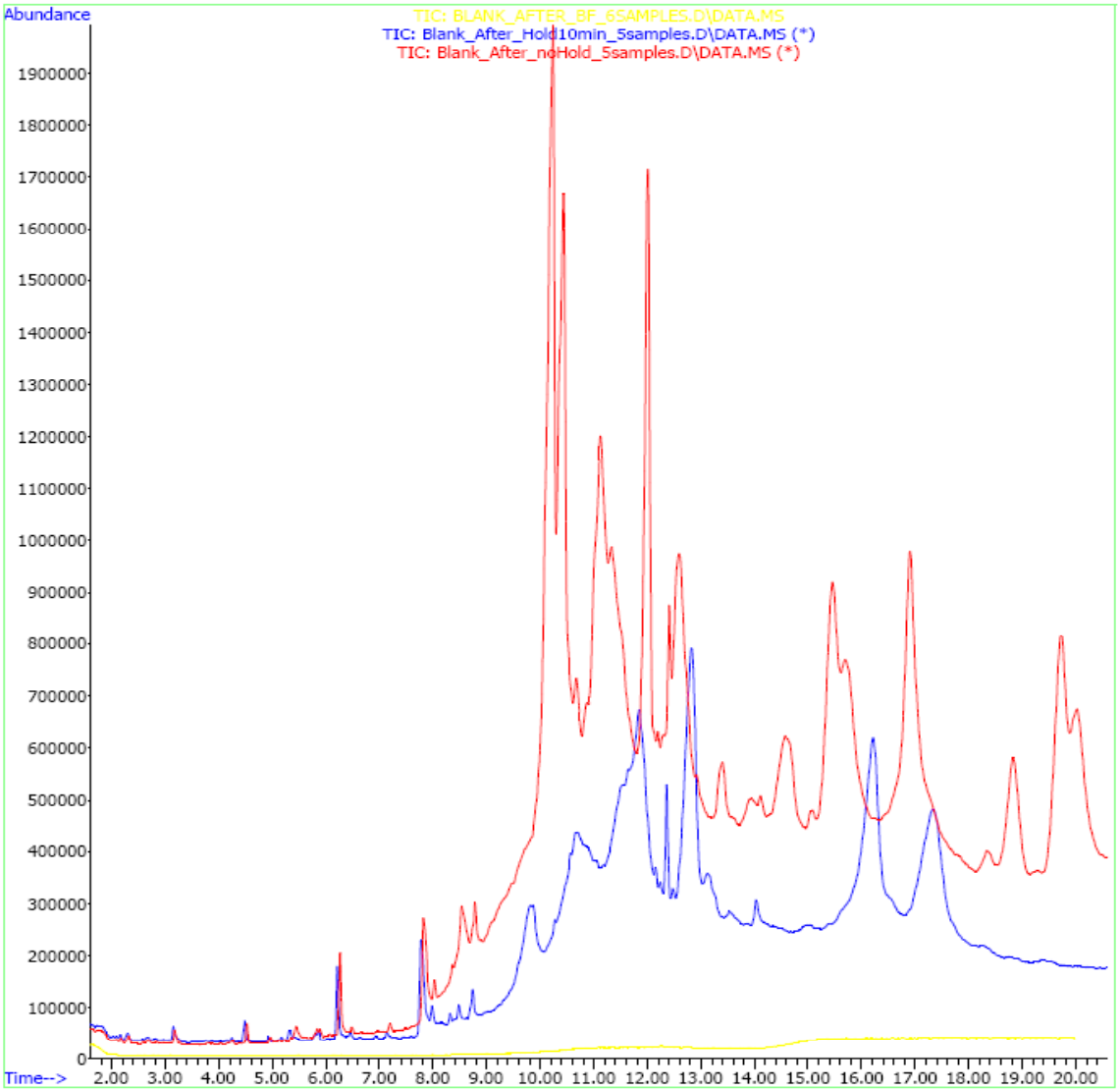
5 Injections With 10min Bake at 280C Shows Smaller Retention Time Shifts In FPD Signal



5 Injections With 2 Min Backflush (50 psi) at 300C Shows No Retention Time Shifts (FPD Signal)



Backflush Gives Clean Baseline



Red: Blank after 5 injections normal method

Blue: Blank after 5 injections with 10 min bake at 280 C

Yellow: Blank after 5 injections with 2min backflush at 50 psi and 300 C

Example 3: Fish Oil, A Very Difficult Matrix

Problems when trying to GC fish oil

High boilers stay on column causing:

- carryover

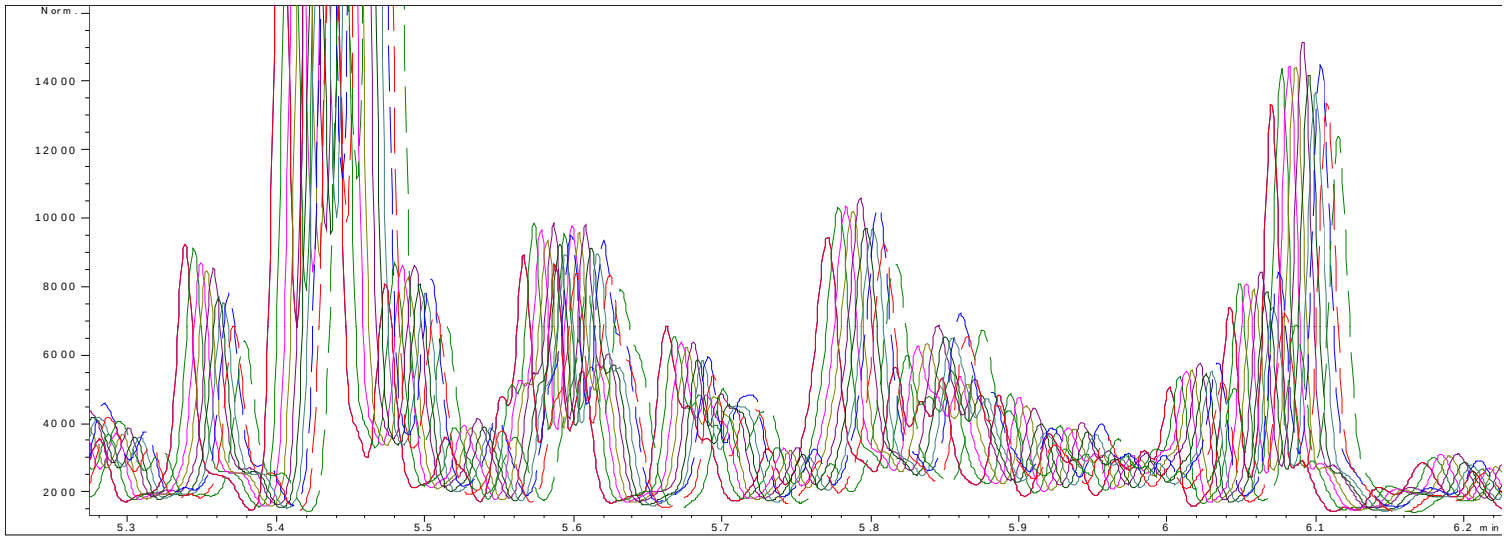
- dramatic shifts in retention times (like PCBs)

Backflushing eliminates retention time shifting by removing high boilers that cause the problem

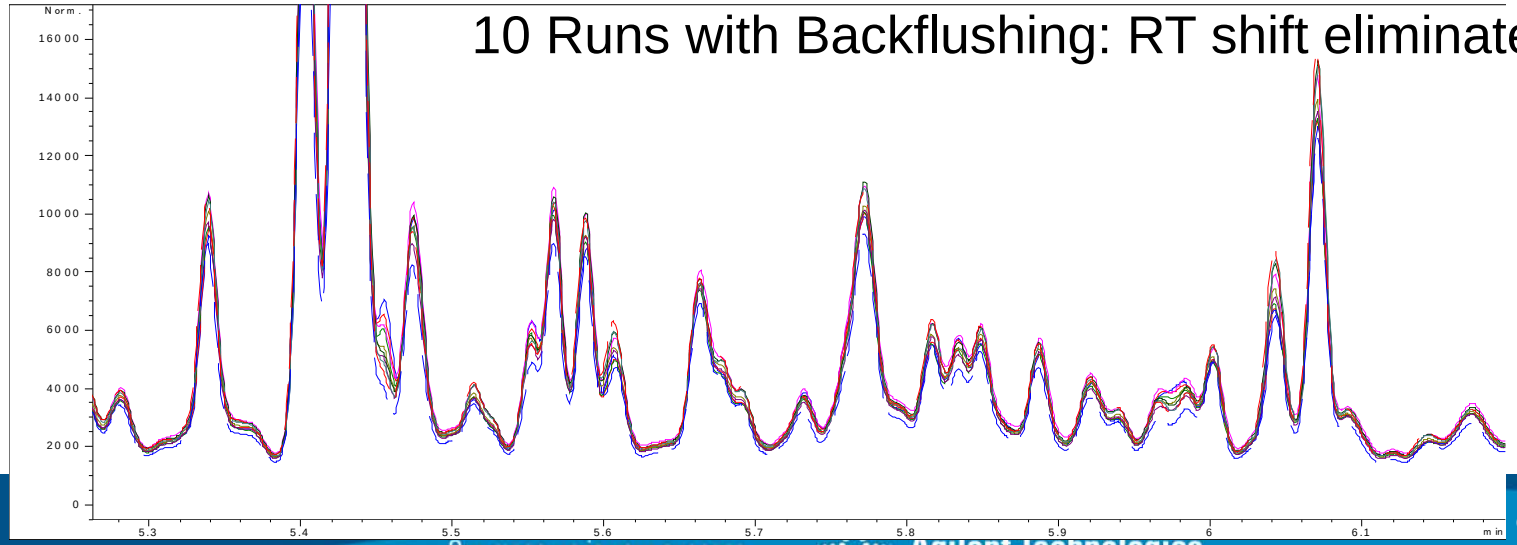
– (for more details, see Application Note: 5989-6095EN, Direct Injection of Fish Oil for the GC-ECD Analysis of PCBs: Results Using a Deans Switch With Backflushing)

10% Fish Oil In Acetone: Retention Time Shifts Eliminated With Backflushing

10 Runs without Backflushing: Retention times shift ~4-5 sec during 10 runs



10 Runs with Backflushing: RT shift eliminated



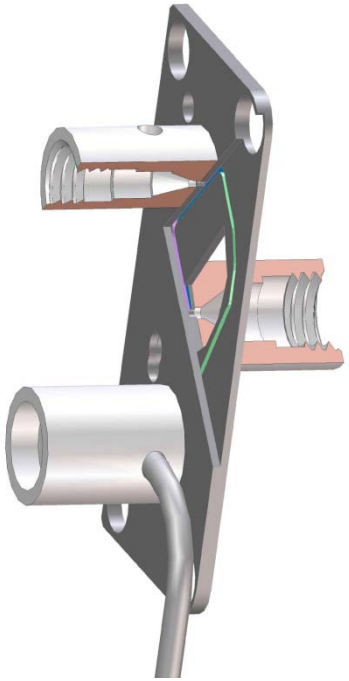
Benefits of Backflushing

- More samples/day/instrument
- Better quality data
- Lower operating costs
- Less frequent and faster GC & MSD maintenance
- Longer column life
- Less chemical background

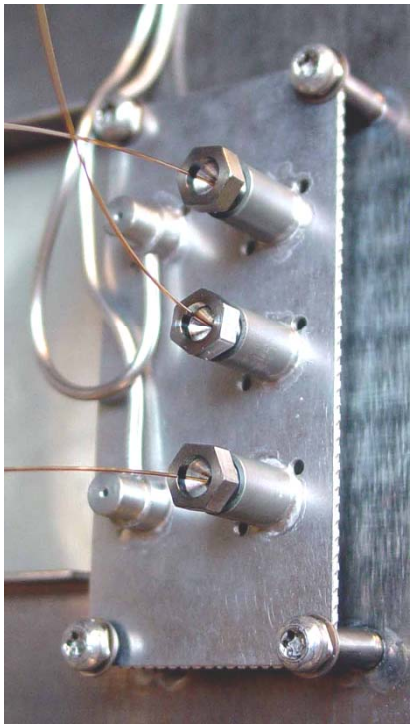
Other Devices Can Provide Backflush Capability

2-Way Splitter with Makeup

QuickSwap

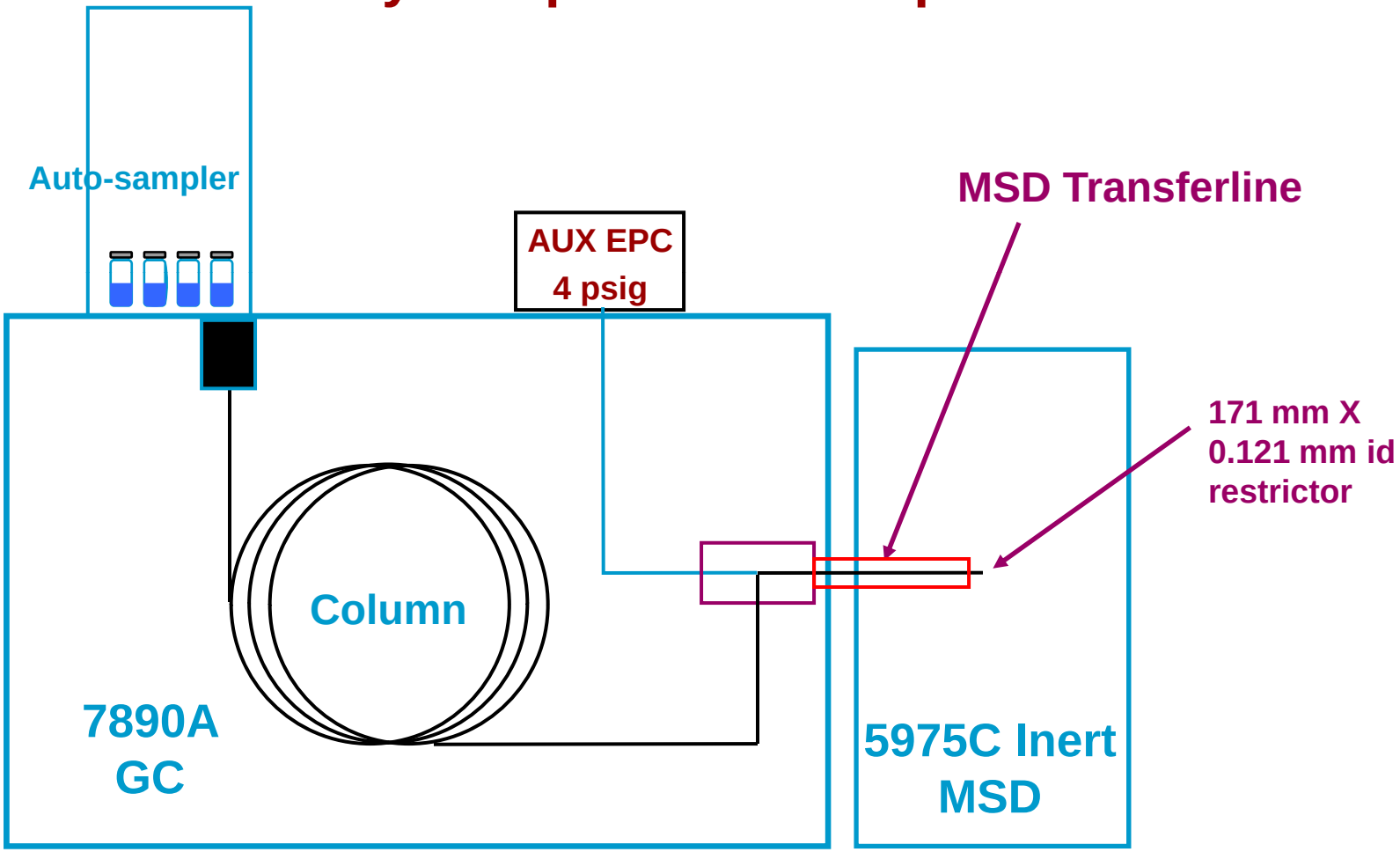


Deans Switch



QuickSwap

Change MSD columns without venting
Backflush heavy components out split vent



QuickSwap MSD Interface

Remove column w/o venting

- **Air & H_2O blocked**

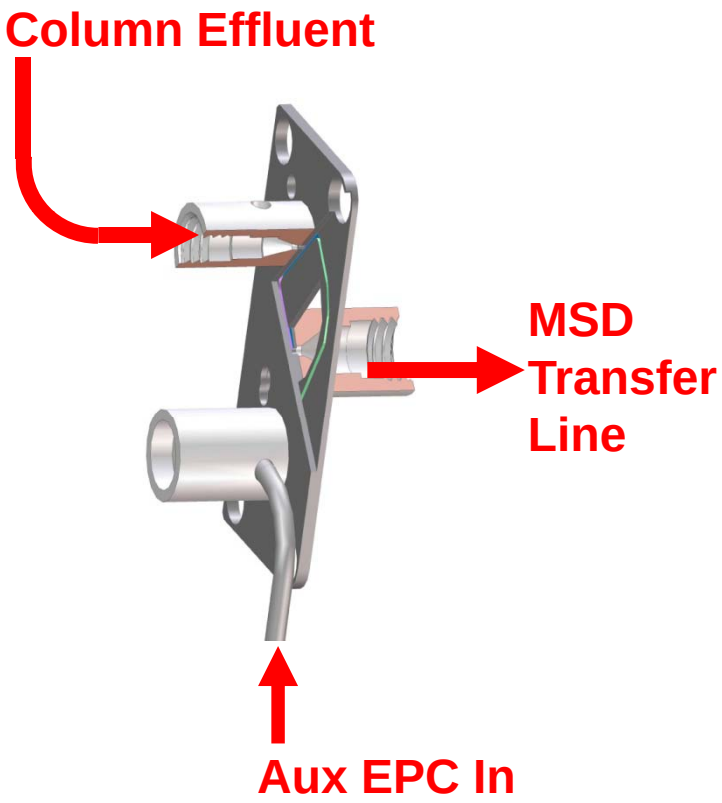
Safe disconnection of column from inlet for inlet maintenance

- **Reversed flow through column during inlet maintenance**

Backflushing

- **Removes heavies from column**

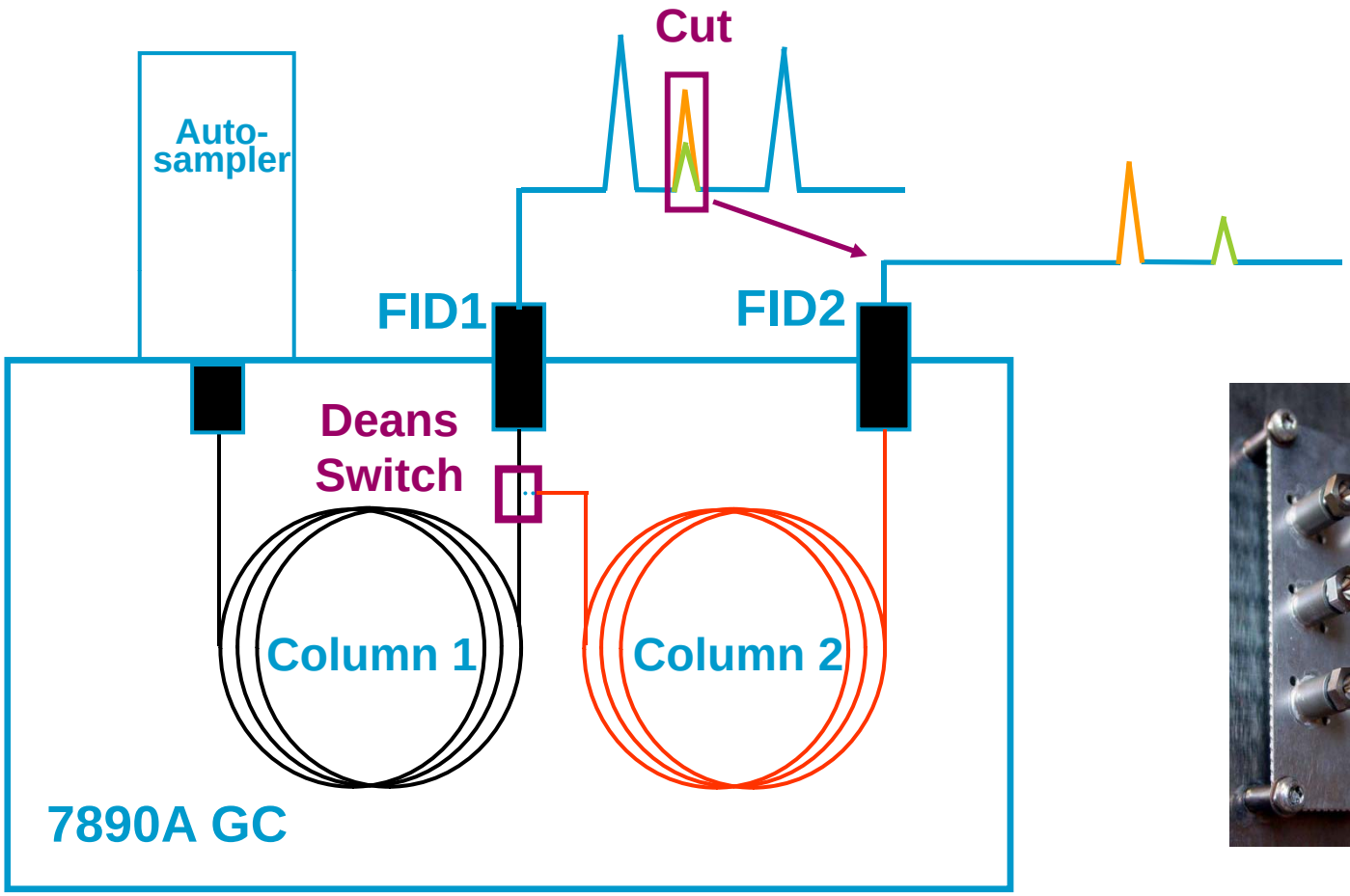
Maintain constant flow to MSD

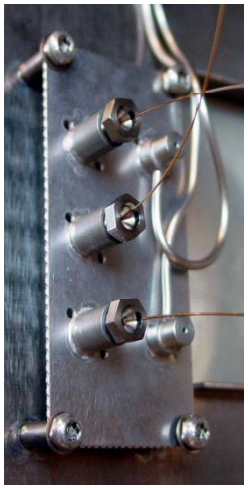


(flow rates exceeding 2 mL/min require an MSD with Performance Turbo)

Dean Switch

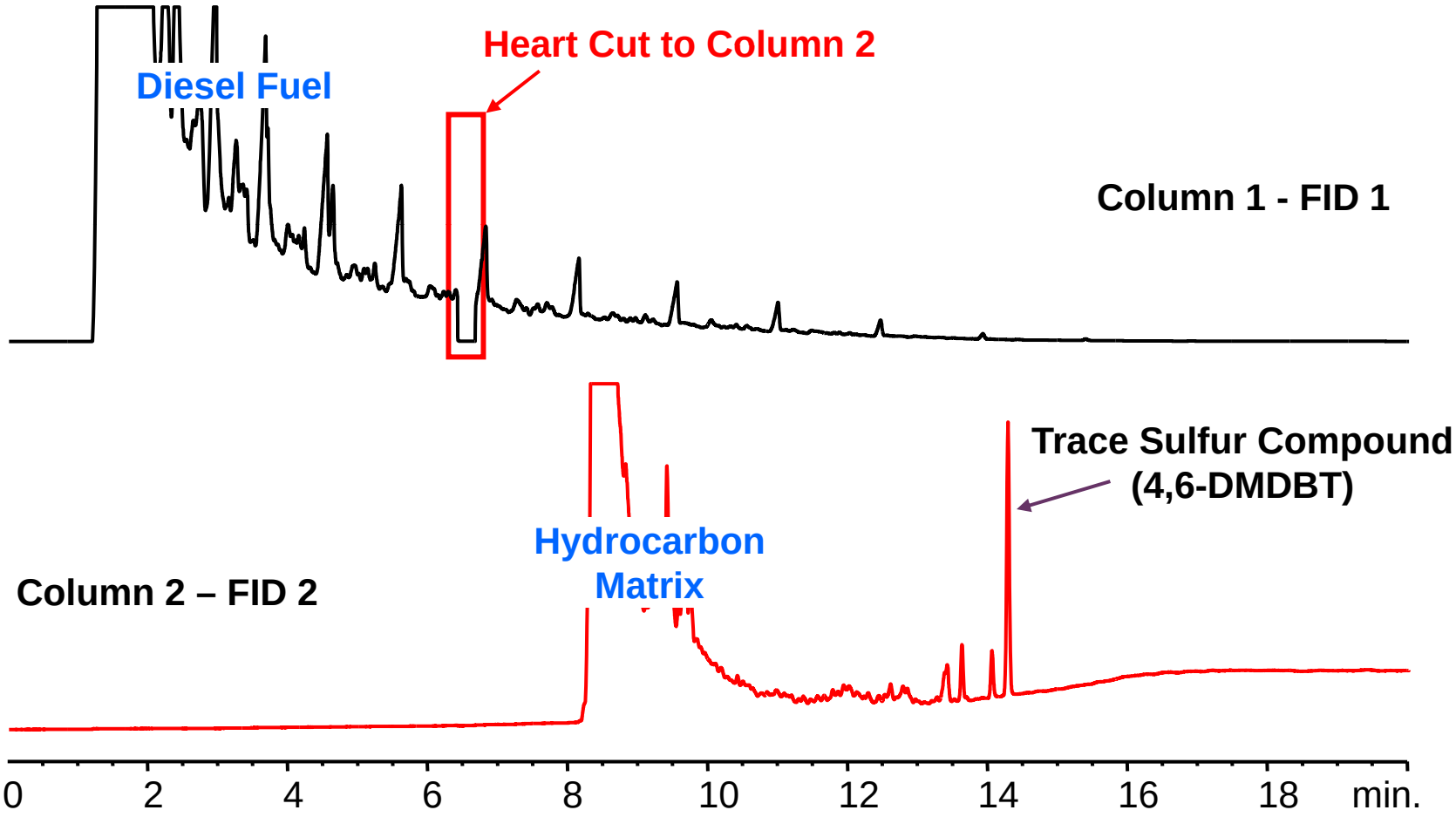
Heartcutting 2-D GC provides extremely high chromatographic resolution



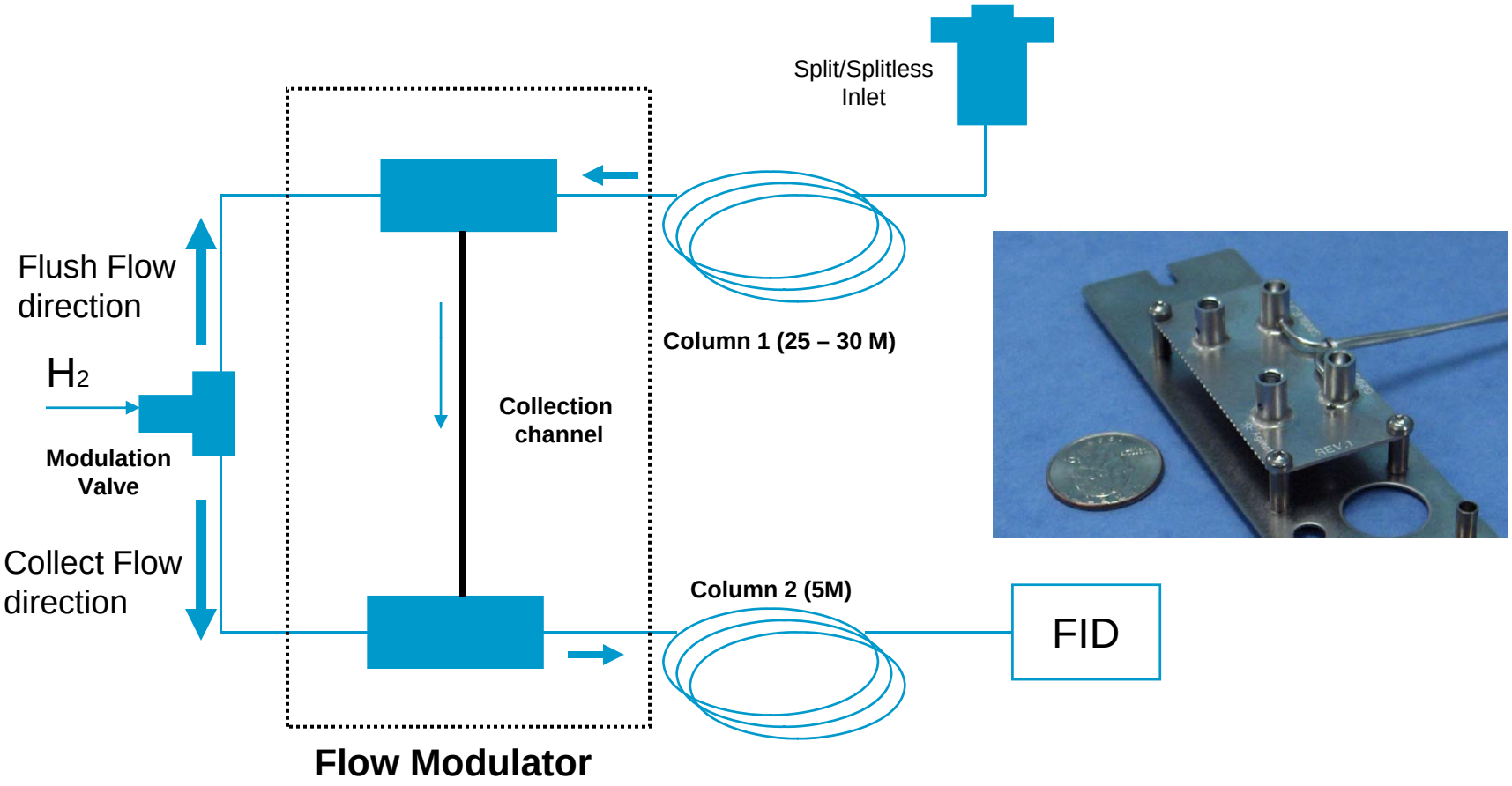


2-D Separation of Sulfur Compound in Diesel Fuel

Compound is completely resolved and can be analyzed with FID

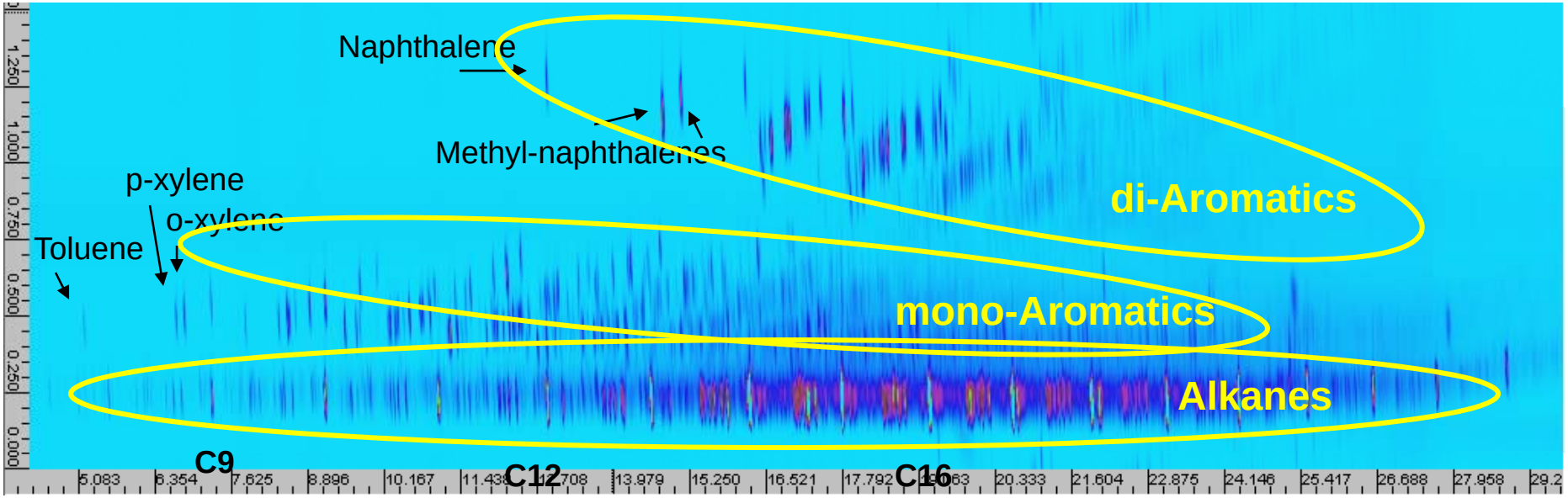


Agilent's flow modulator design : Differential Flow Using Design by John V. Seeley, Oakland University



Flow modulator eliminates the need for cryo. Sample compression controlled by flow ratios occurs in the collection loop and is quickly injected into the second column, resulting in very narrow and tall peaks.

Flow modulation:
(GC x GC) of diesel fuel: 7890A



GC x GC Chromatogram:

- Showing the normal B.P. distribution (1st dimension)
- Also shows hydrocarbon classes in clusters

- Consistent RT for alkanes in 1st dimension showing precise modulation
- Comparable peak in 2nd dimension band shows minimum peak broadening with flow modulation

Agilent Flow Modulation GC x GC

- **Reliable Setup:** Based on capillary-flow- technology, easy to setup, high performance chromatography, and reliable.
- **No Cryogen Required:** Flow modulation means no tanks of Liquid N_2 or CO_2
- **7890A Enabled GC x GC:** Capillary- flow-technology ready, synchronized periodic events ensure precise modulation, control from a modified TCD board
- **Comparable resolution without Liquid N_2:** Cap Flow Technology allows low dead volume and precise flow control, resulting in minimum peak broadening even without cryo-focusing . Peak widths on the second column are typically 70 to 100 ms at half maximum.
- **Sensitivity:** Approaches that obtained by thermally modulated systems

Summary

Capillary Flow Technology opens up many new (and old) possibilities for GC and GC/MS systems.

Application Notes

5989-1840EN Using a New Gas Phase Micro-Fluidic Deans Switch for the 2-D GC Analysis of Trace Methanol In Crude Oil by ASTM Method D7059

5989-6082EN Simultaneous Analysis of Trace Oxygenates and Hydrocarbons in Ethylene Feedstocks Using Agilent 7890A GC Capillary Flow Technology

5989-8060EN Comprehensive GC System Based on Flow Modulation for the 7890A GC

5989-6018EN Improving Productivity and Extending Column Life with Backflush

5989-8107EN Analysis of Fatty Acid Methyl Ester (FAME) Content and Distribution in Biodiesel Blends Using Heart-Cutting 2D Gas Chromatography

5989-8582EN Improved Forensic Toxicology Screening Using A GC/MS/NPD System with a 725-Compound DRS Database

5989-5668EN Confirmation of THC in Oral Fluids Using High-Resolution 2-D GC/MS

5989-7670EN Replacing Multiple 50-Minute GC and GC-MS/SIM Analyses with One 15-Minute Full-Scan GC-MS Analysis for Nontargeted Pesticides Screening and >10x Productivity Gain

5989-4834EN Screening for Hazardous Chemicals in Homeland Security and Environmental Samples Using a GC/MS/ECD/FPD with a 731 Compound DRS Database

5989-6026EN Significant Cycle Time Reduction Using the Agilent 7890A/5975 GC/MSD for EPA Method 8270