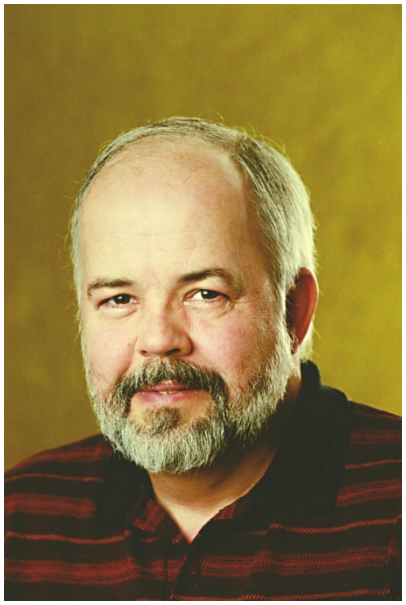


Capillary Flow Technology

-- solves difficult application problems easily & opens up many new (and old) possibilities for GC & GC/MS

Bruce D. Quimby Ph.D.
Agilent Technologies
Wilmington, Delaware, USA

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)
- **Deans 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

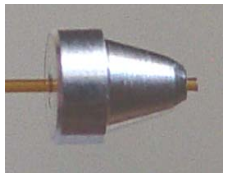
		Advantages	Limitations
Metal Fittings		**Packed columns, reliable**	**Not inert, no ferrule for capillary columns**
Press Fit Glass		**Low dead volume, inert, low cost**	**Difficult to assemble, comes apart**
Graphite		**High temperature**	**Sheds active graphite particles into sample path**
Polyimide		**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 400°C

Manifold Plates

Complex flow structures with low thermal mass

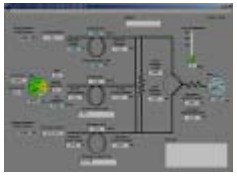
Deactivation of Metal

Makes metal surfaces as inert as column

EPC

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

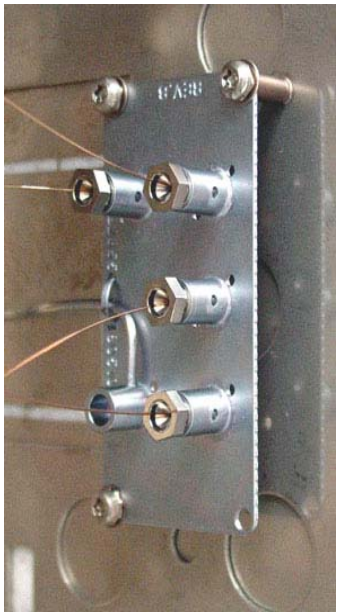
Calculators



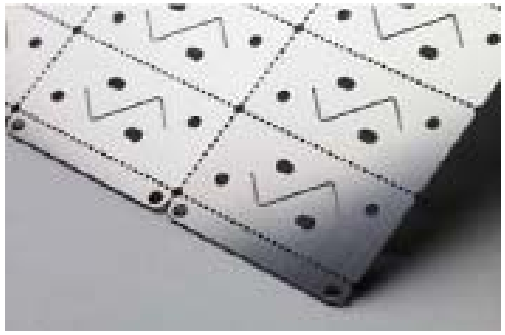
Accurately predict flows and pressures BEFORE installing devices

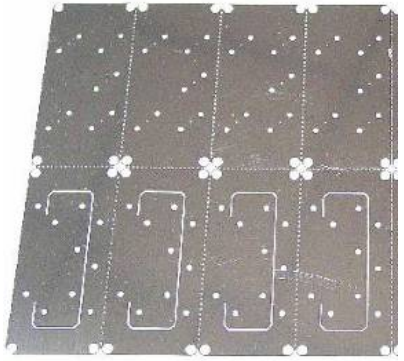
Capillary Flow Technology- Design

… 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

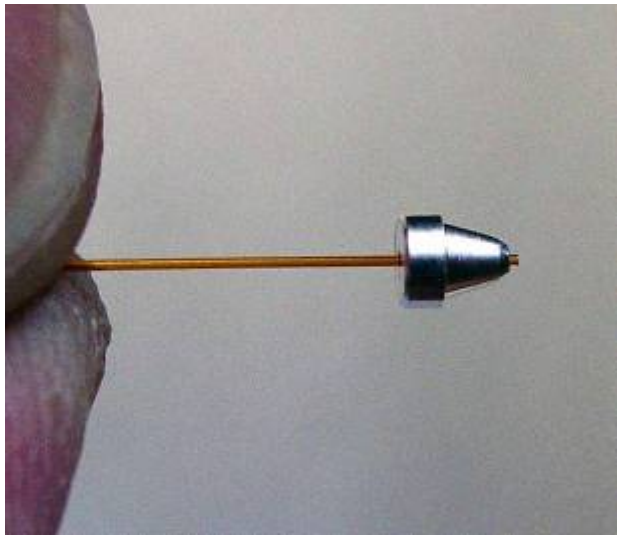


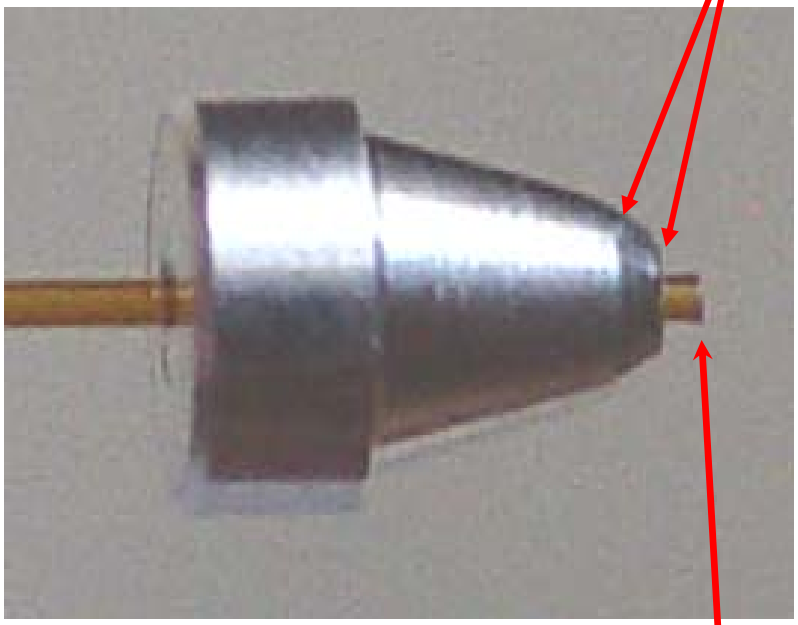


The Metal Ferrule

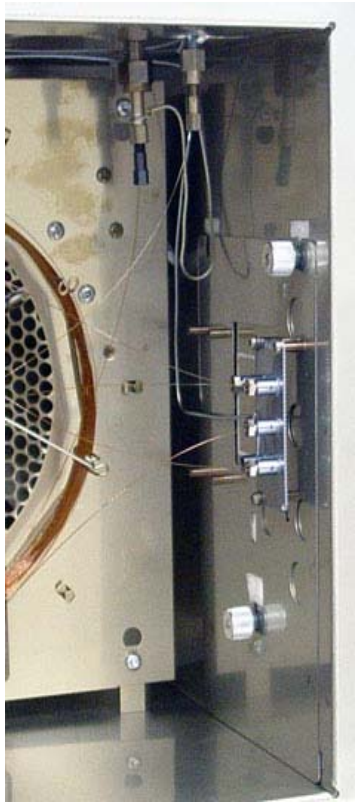
Does not loosen (leak) even with *thousands* of runs to 350C

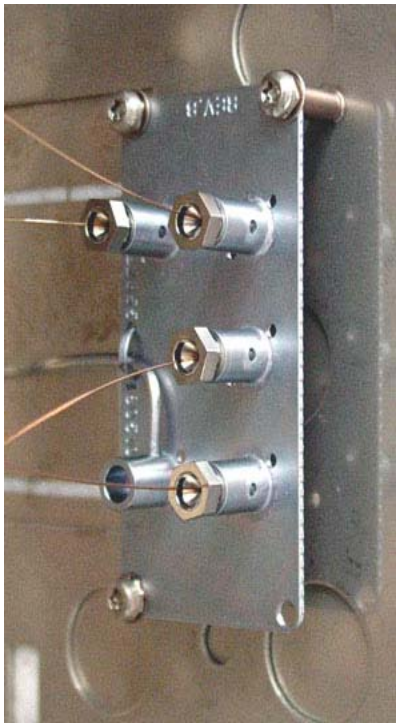
Does not shed particles

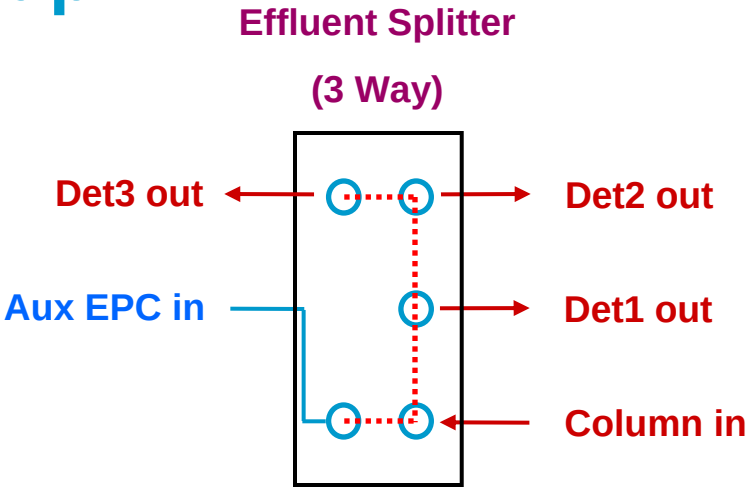


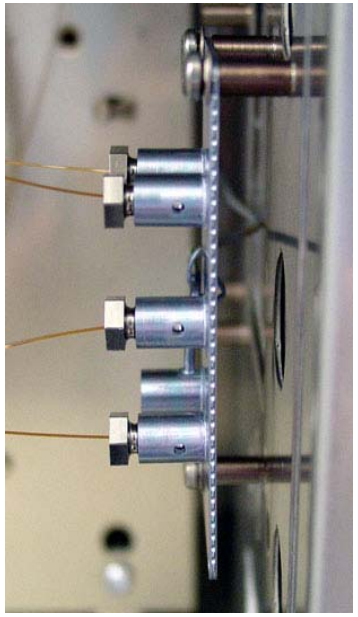


3-Way Splitter With Makeup

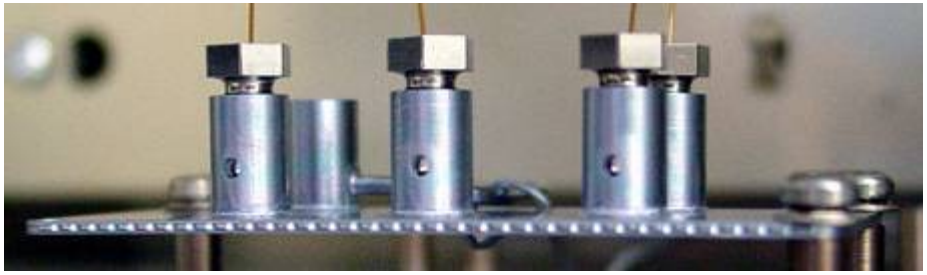


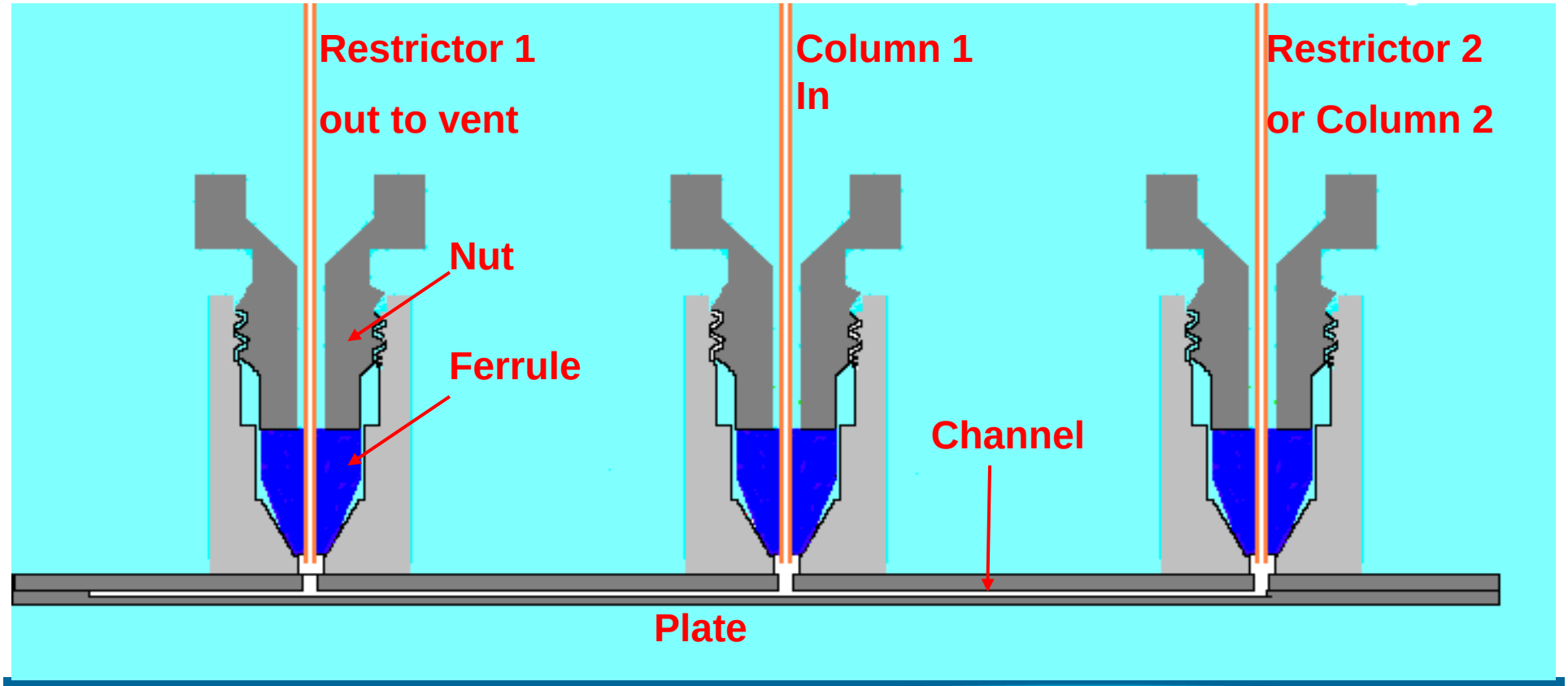






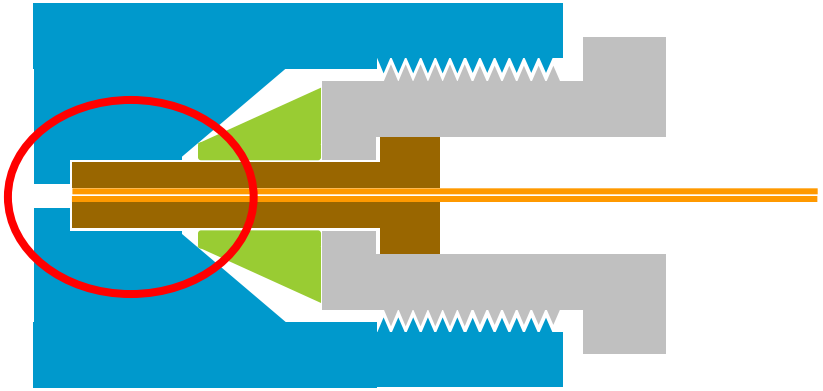
Capillary Flow Technology





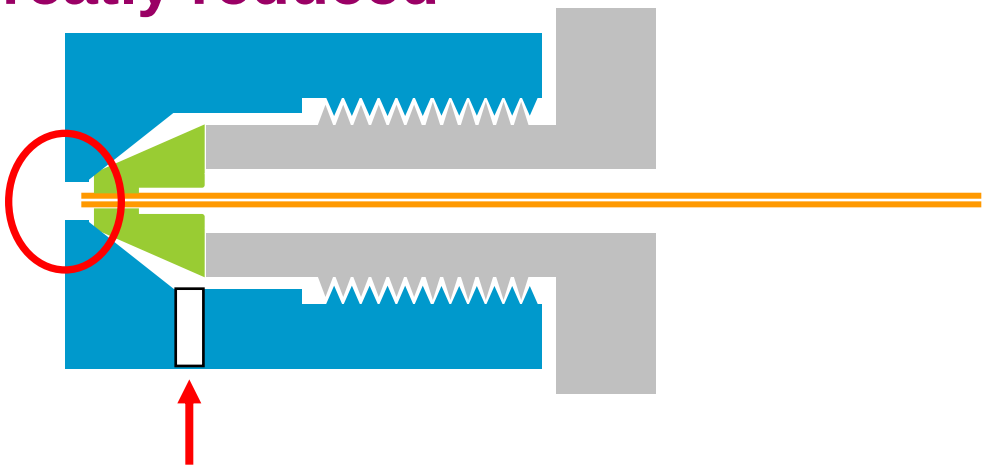
Comparison of New Fitting with Polyimide Fitting

Polyimide Fitting



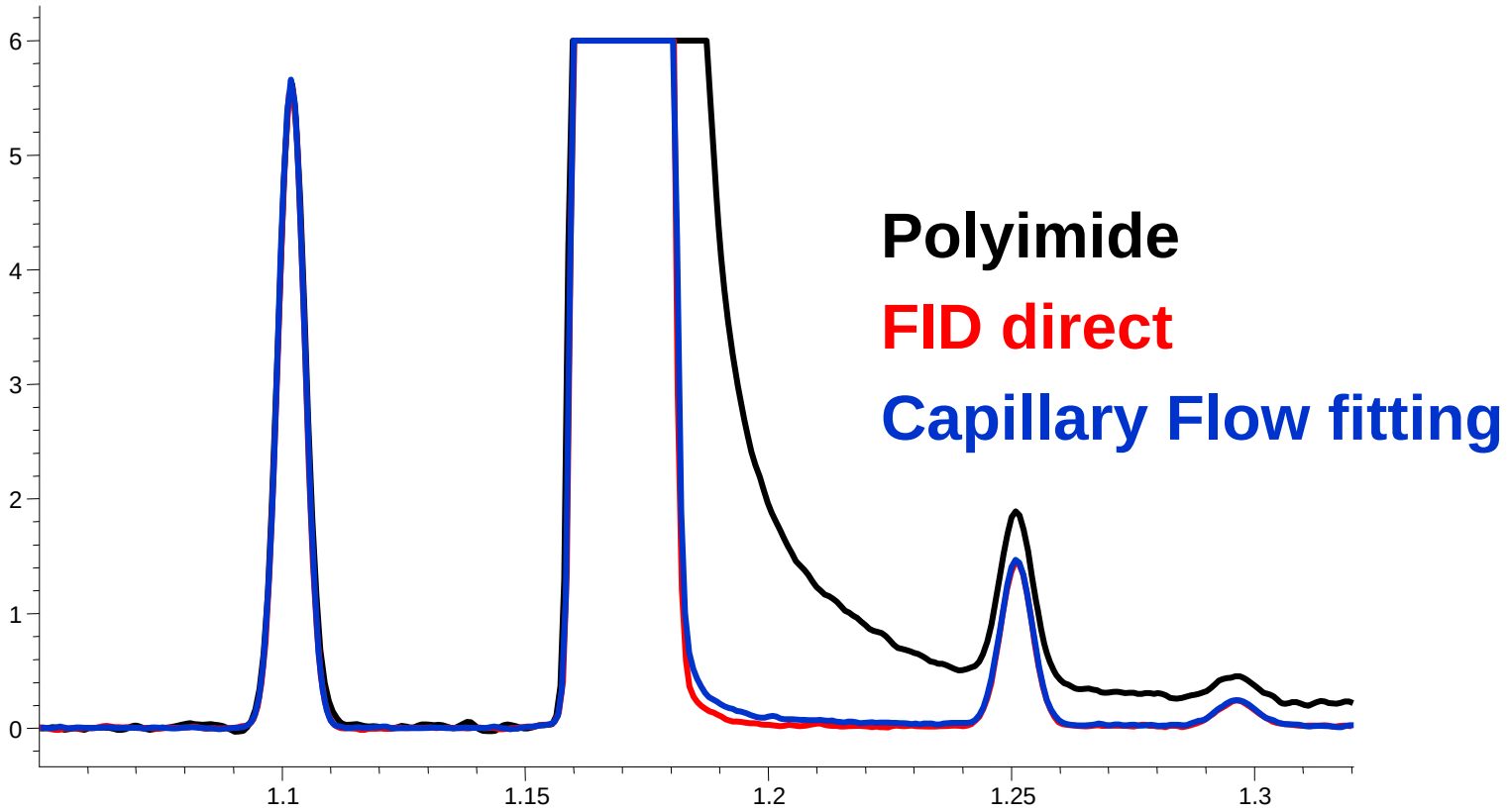
Exposure to polyimide and unpurged annular spaces is greatly reduced

New Fitting



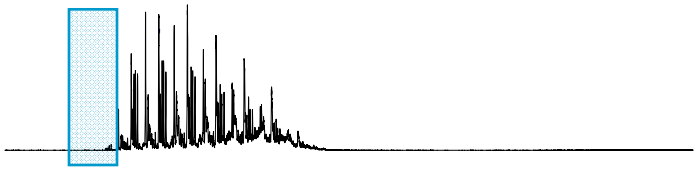
Fitting Design Minimizes Tailing

Pentane test chromatogram

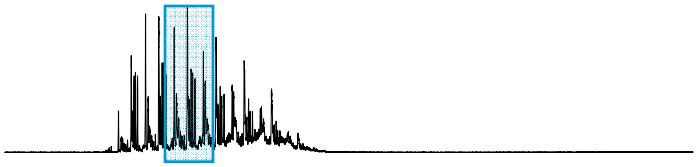


Capillary Flow Technology fittings avoid tailing with small but well swept dead volume

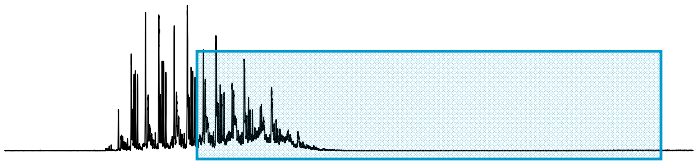
Capillary Flow Technology- Capabilities



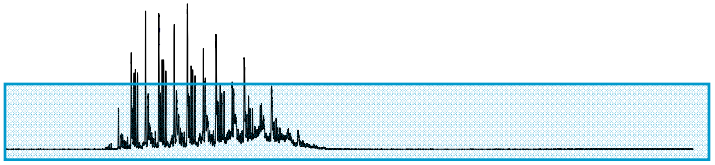
Solvent Bypass



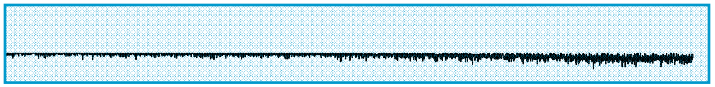
Heart Cutting (Deans Switch)



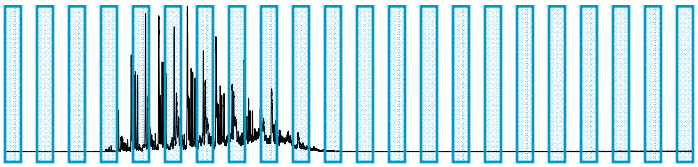
Backflush



Detector Splitting

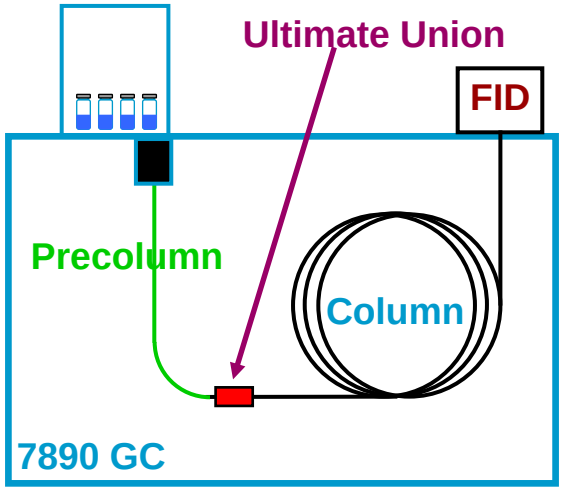


QuickSwap



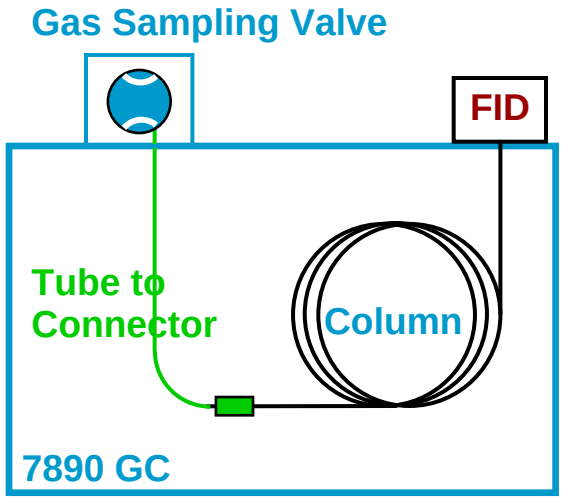
Modulation (GCXGC)

Capillary Flow Technology Devices



Ultimate Union

Reliable precolumn connector



Tube Connector

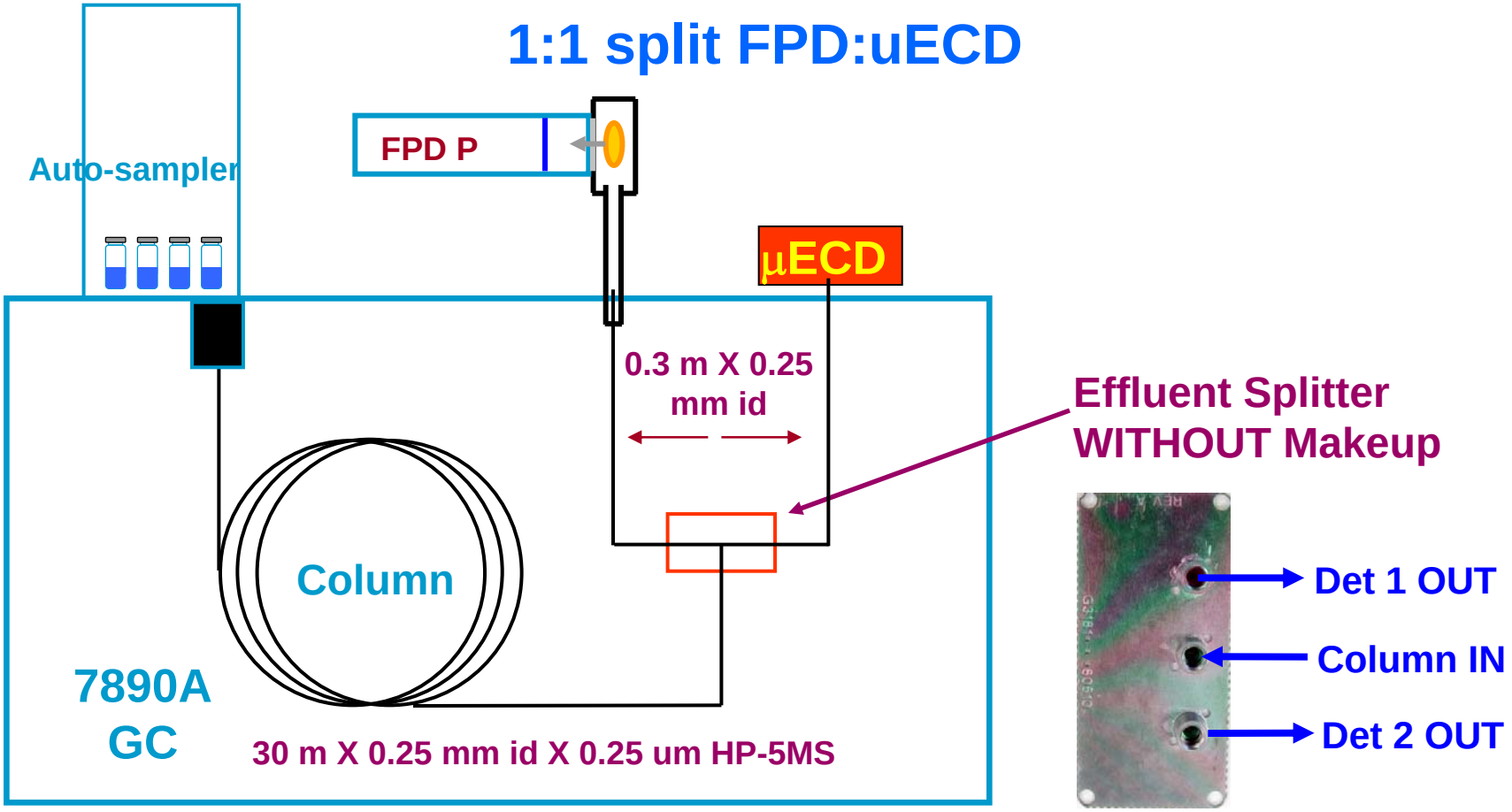
Easy valve to capillary column connector

Tube is 0.25 mm id and is deactivated

Splitters: Unpurged Tee

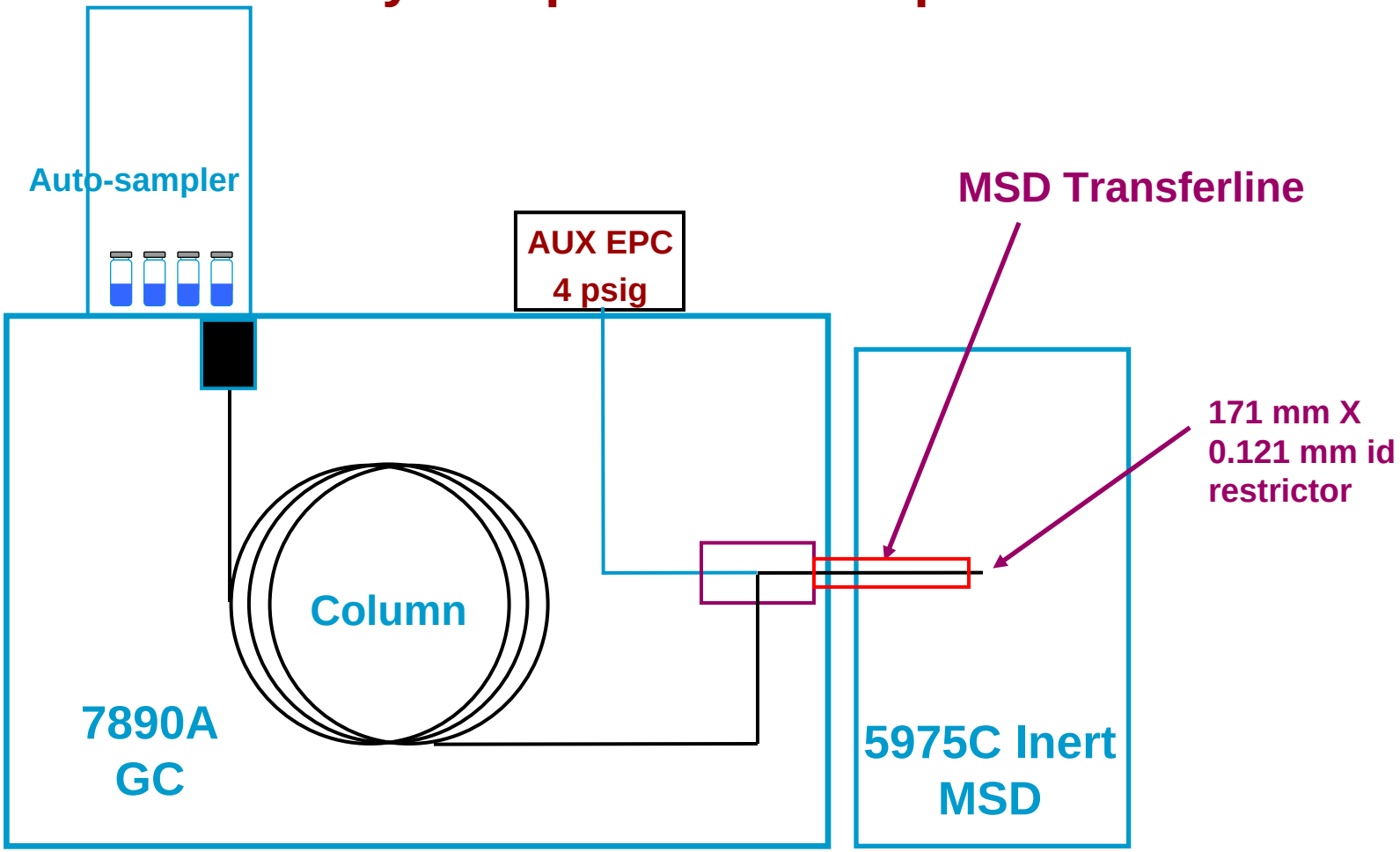
Simultaneous detection with 2 detectors (but NOT MSD)

Cannot do backflushing



QuickSwap

Change MSD columns without venting
Backflush heavy components out split vent



QuickSwap MSD Interface

Remove column w/o venting

- Air & H_2O blocked

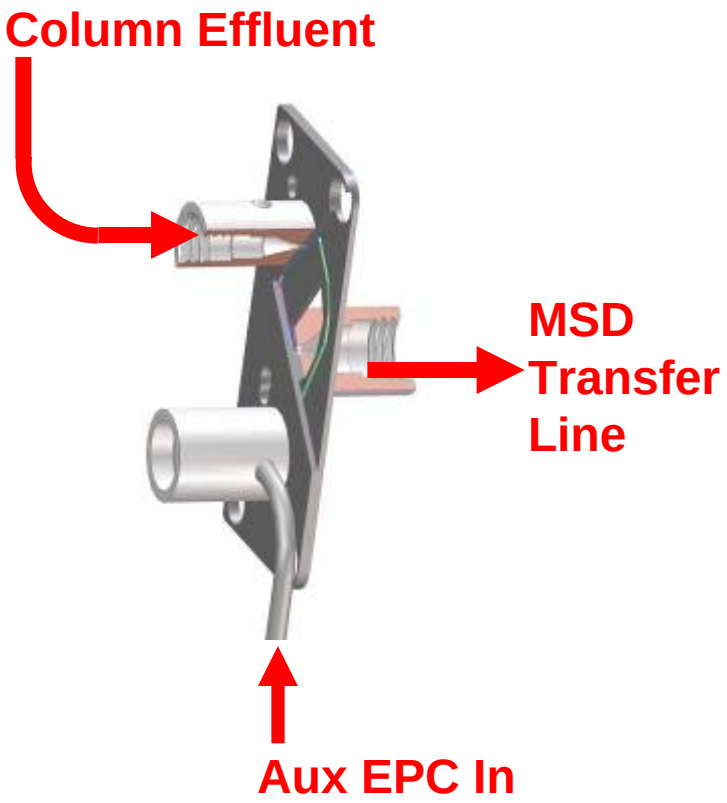
Safe disconnection of column from inlet for inlet maintenance

- Reversed flow through column during inlet maintenance

Backflushing

- Removes heavies from column

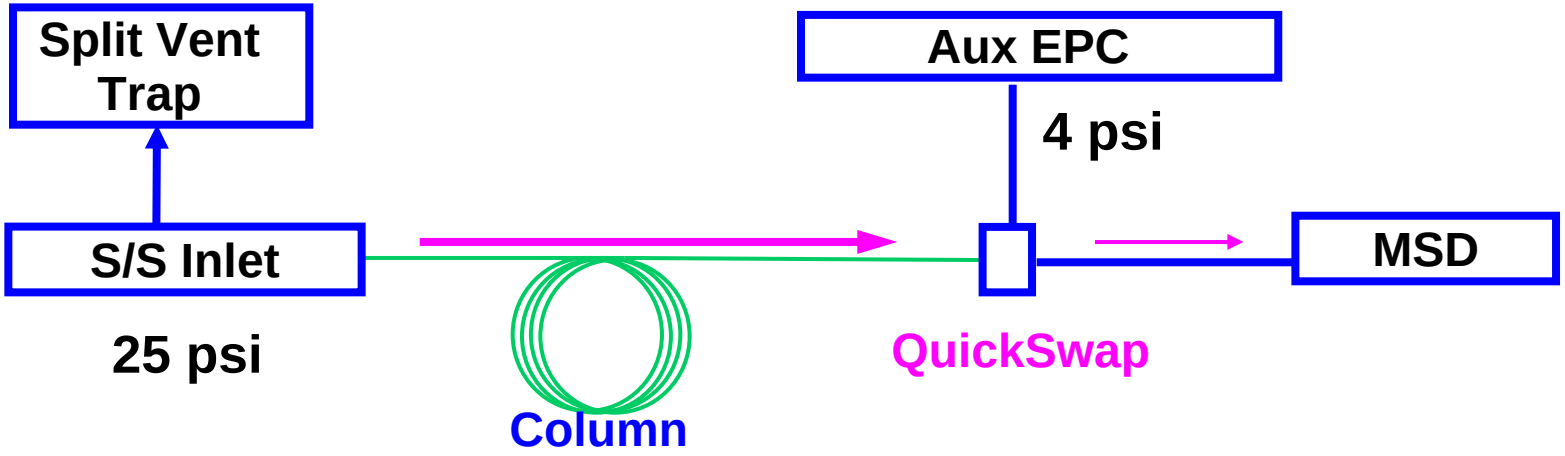
Maintain constant flow to MSD



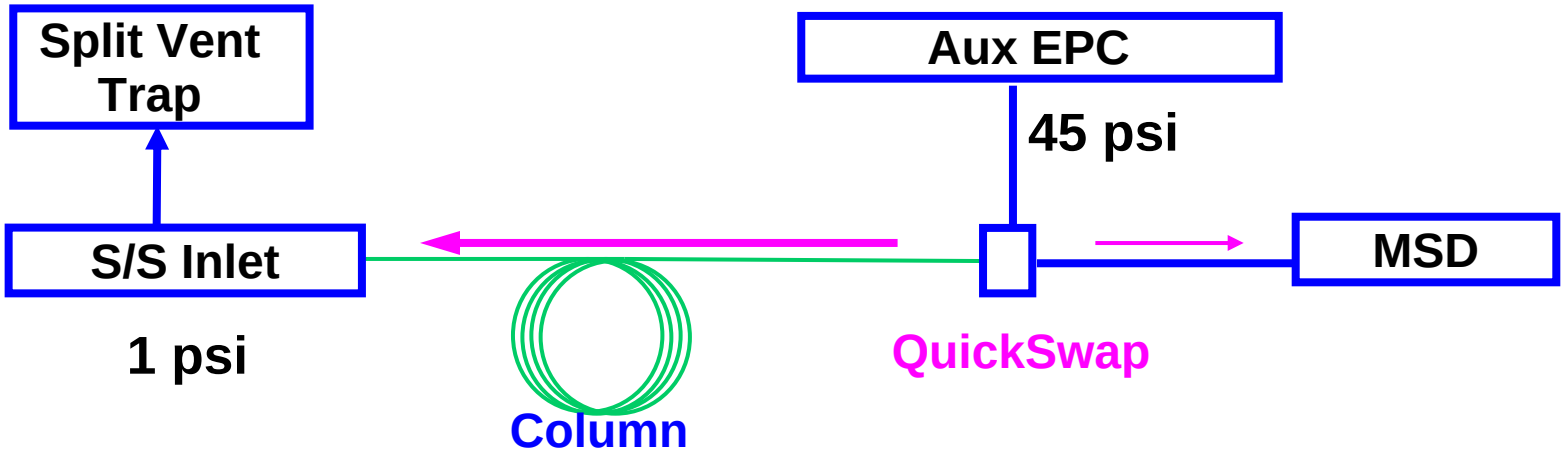
(flow rates exceeding 2 mL/min require an MSD with Performance Turbo)

Backflush with QuickSwap

During GC Run



After GC Run



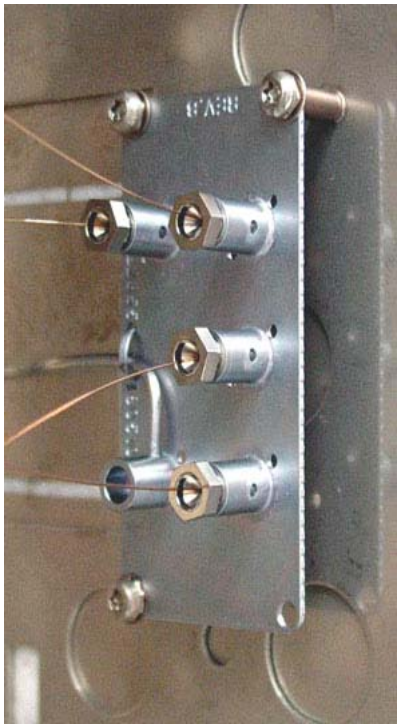
Benefits of Backflushing

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

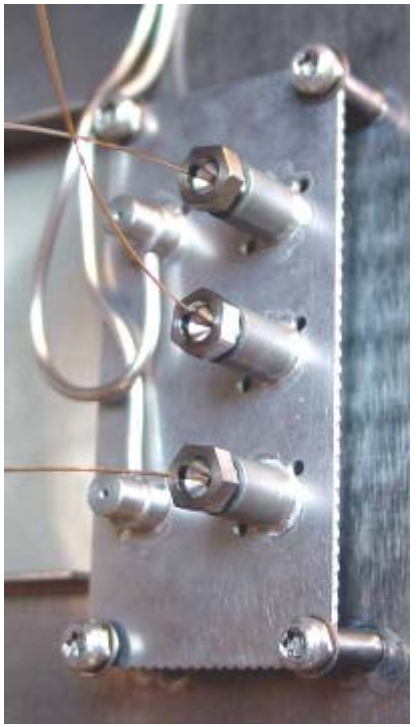
Three Other Devices Provide Backflush Capability

2-Way Splitter with Makeup

3-Way Splitter with Makeup

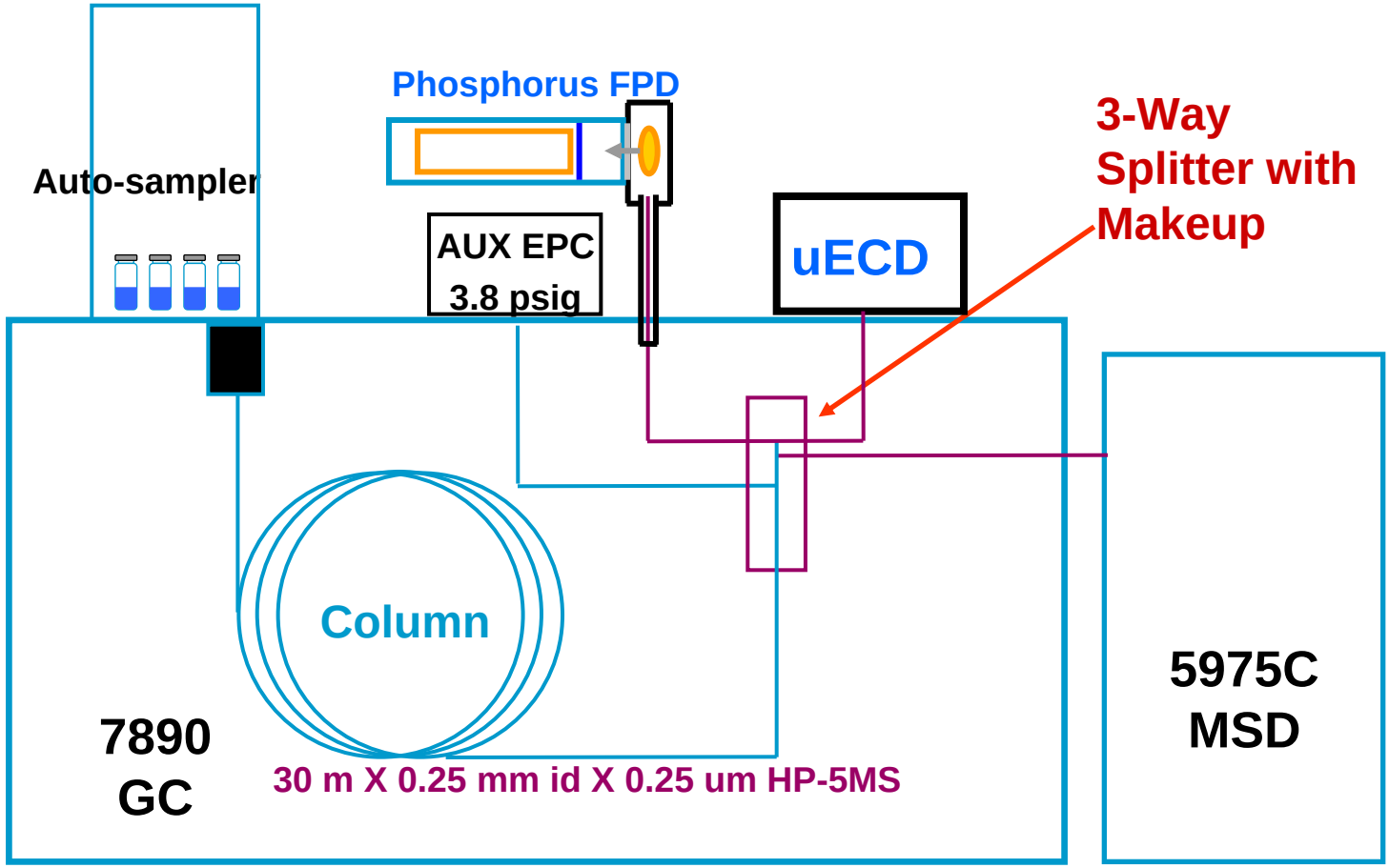


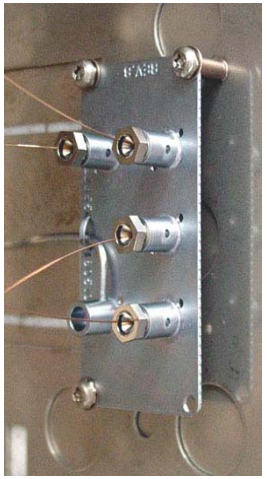
Deans Switch



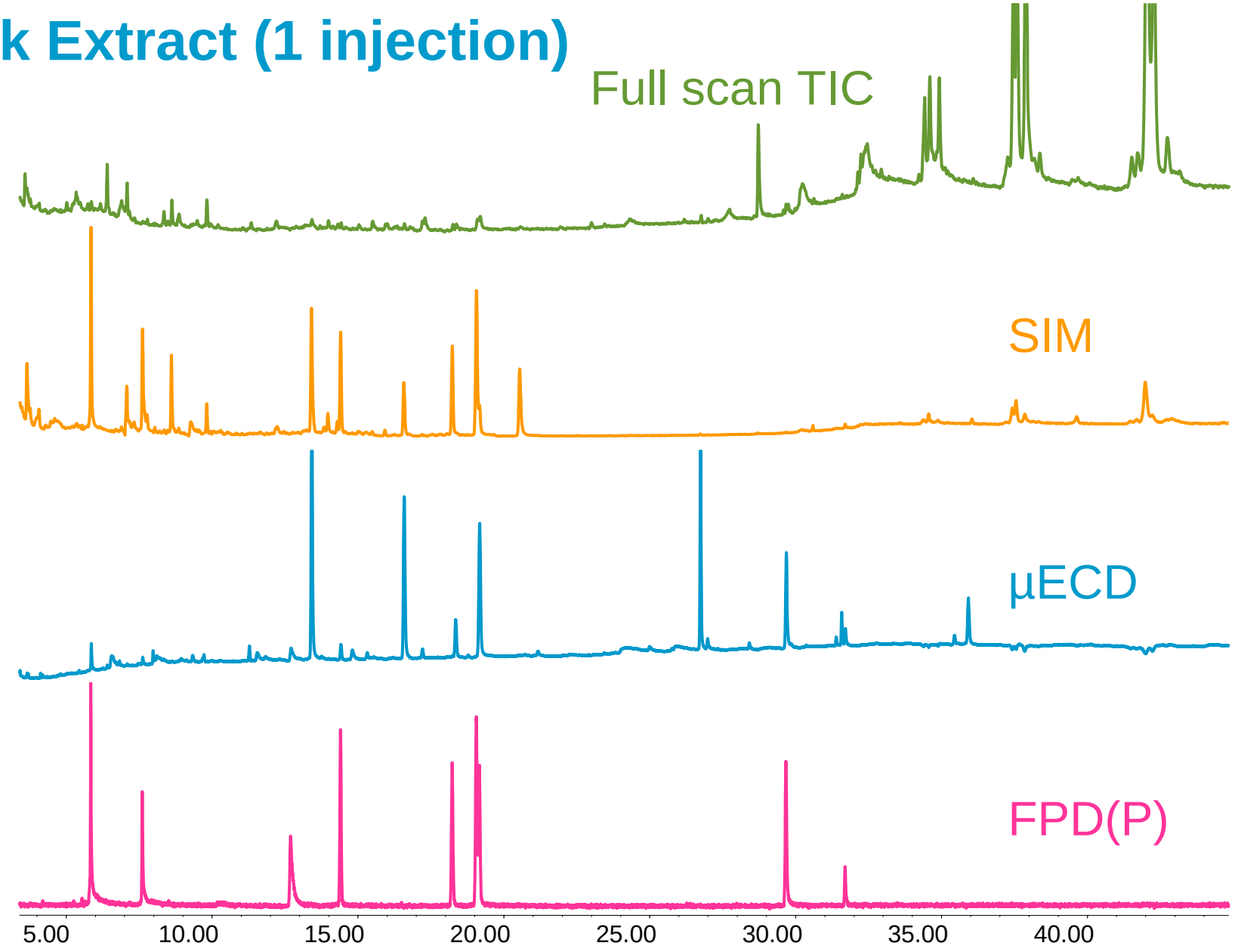
Pesticides: Three Way Splitter with Makeup

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

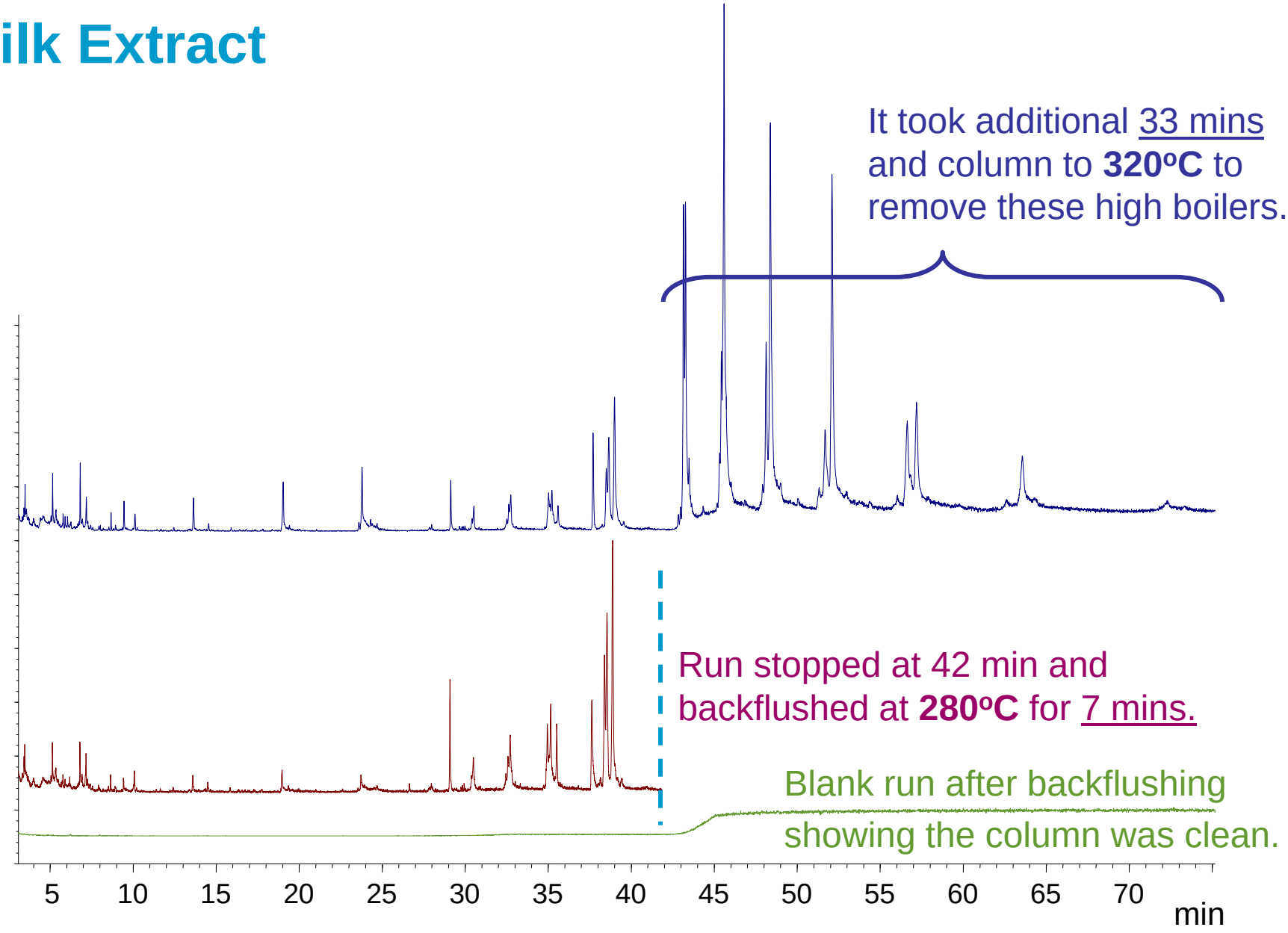




Milk Extract (1 injection)

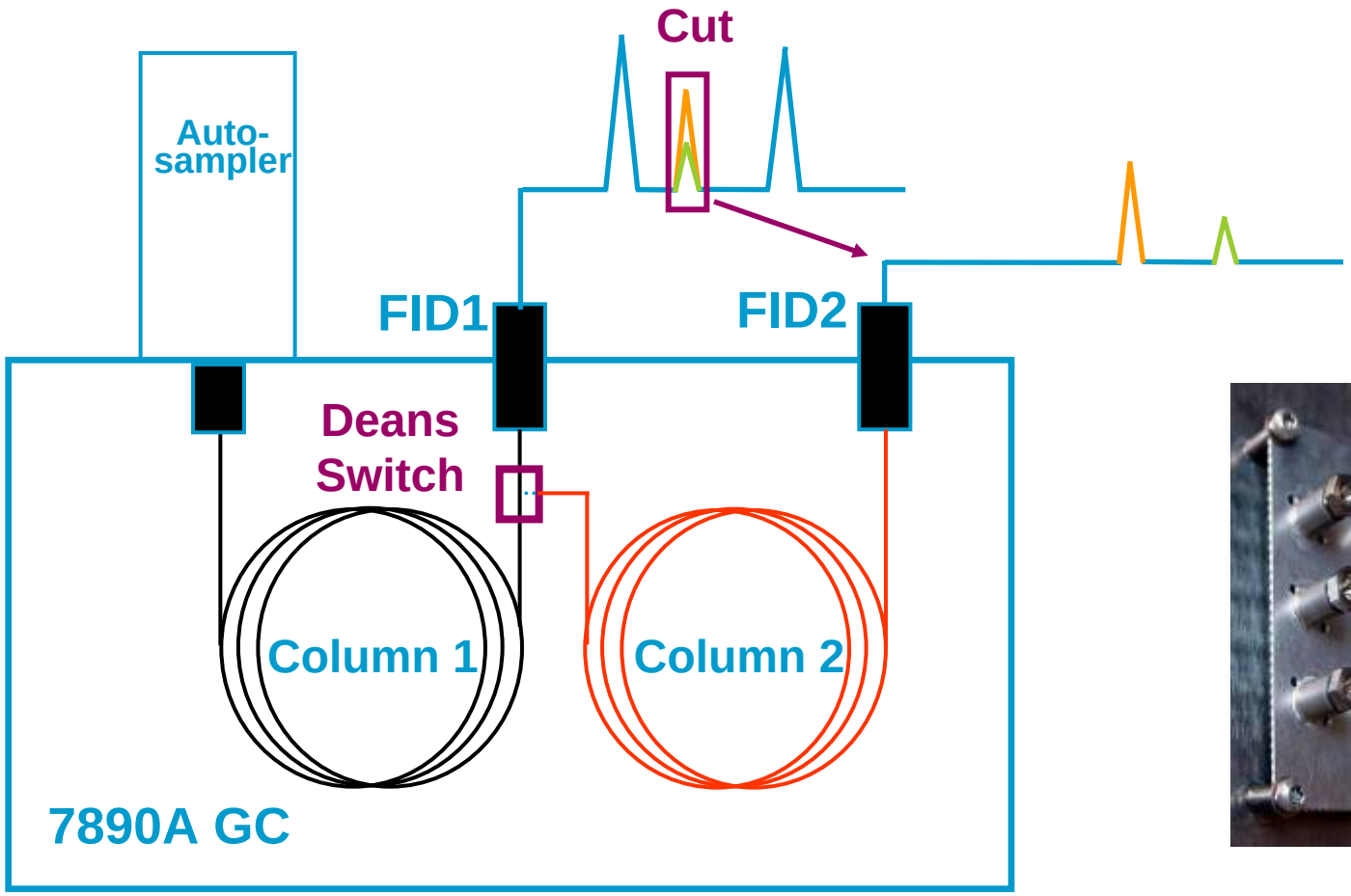


Milk Extract



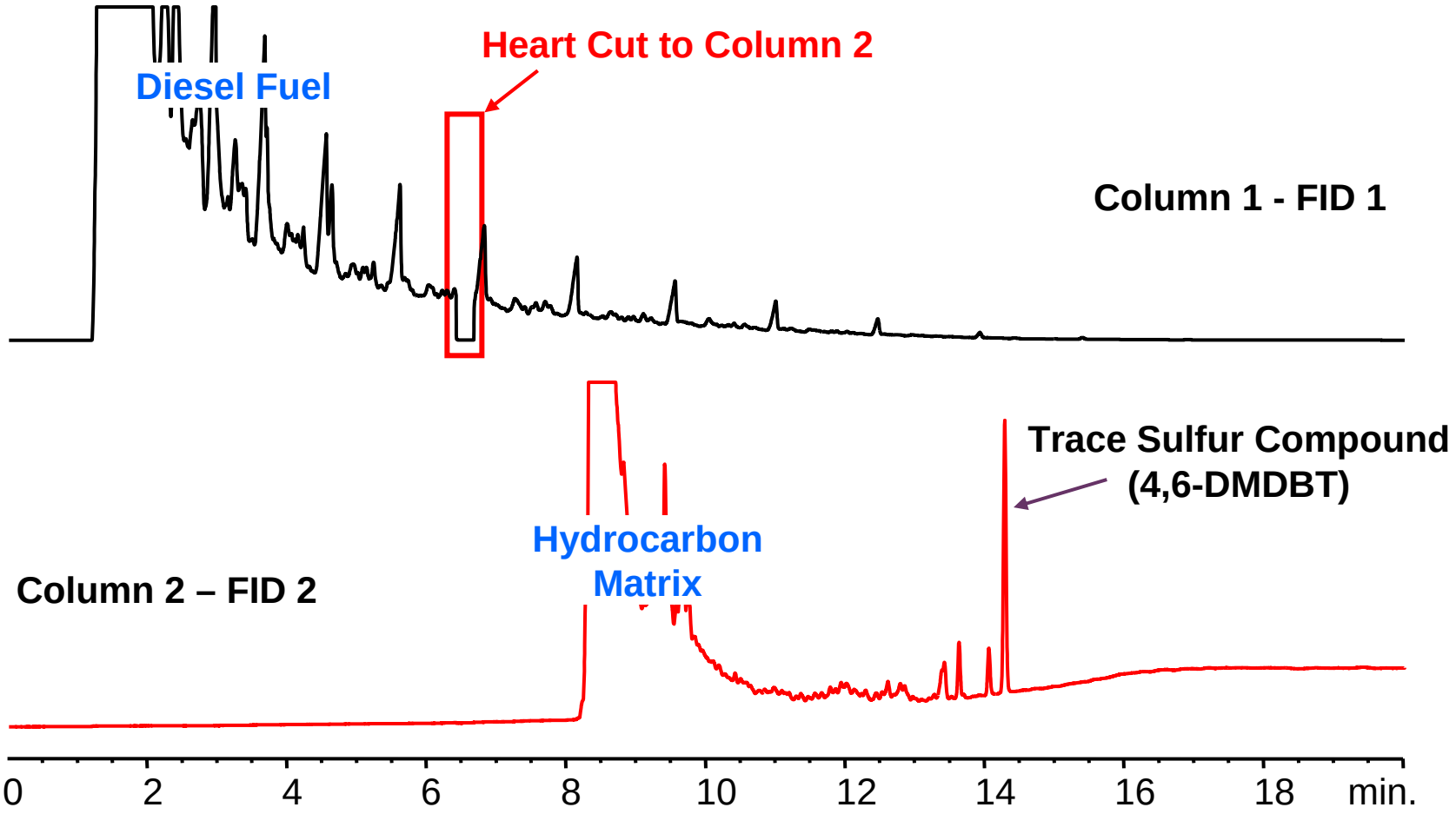
Dean Switch

Heartcutting 2-D GC provides extremely high chromatographic resolution

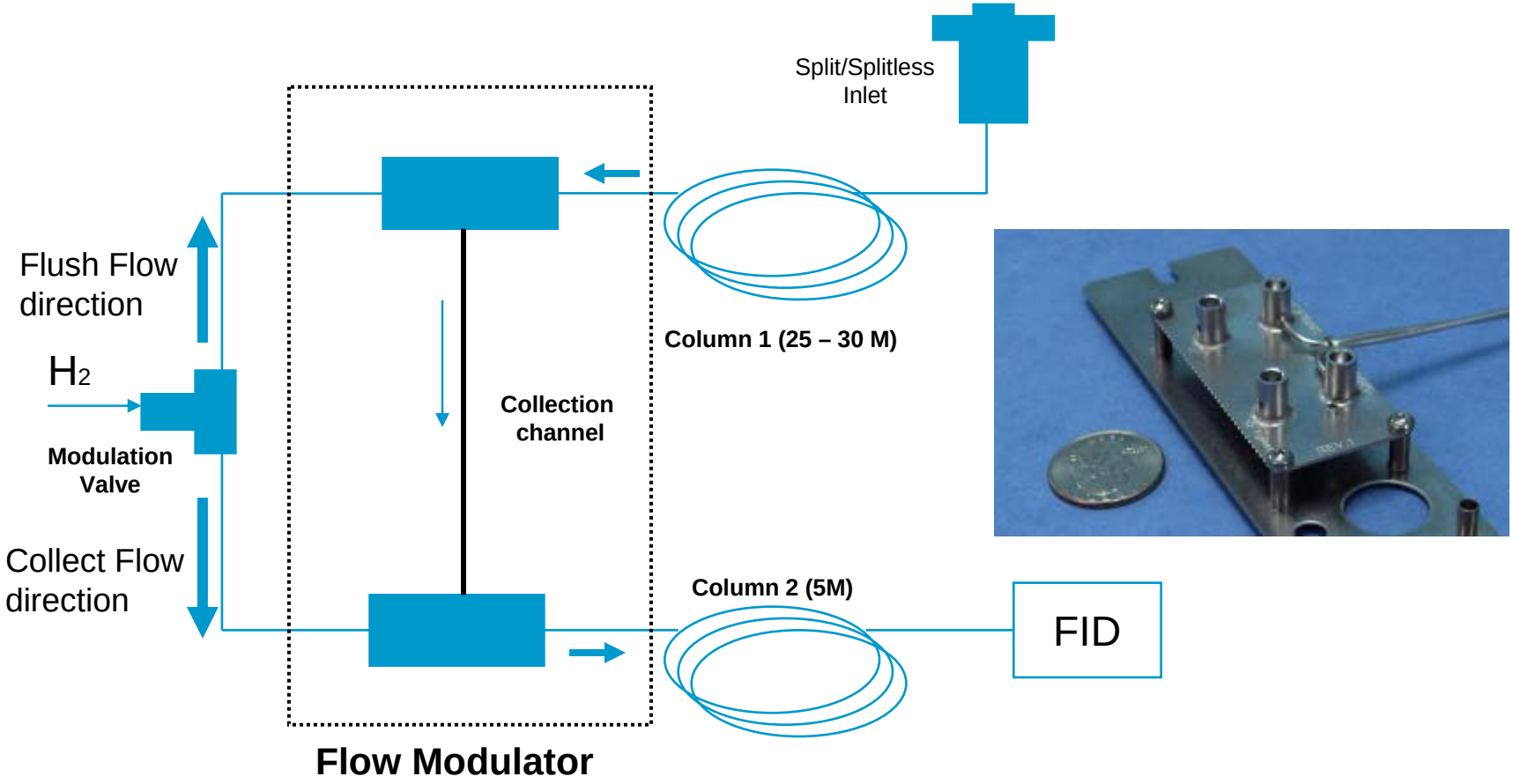


2-D Separation of Sulfur Compound in Diesel Fuel

Compound is completely resolved and can be analyzed with FID



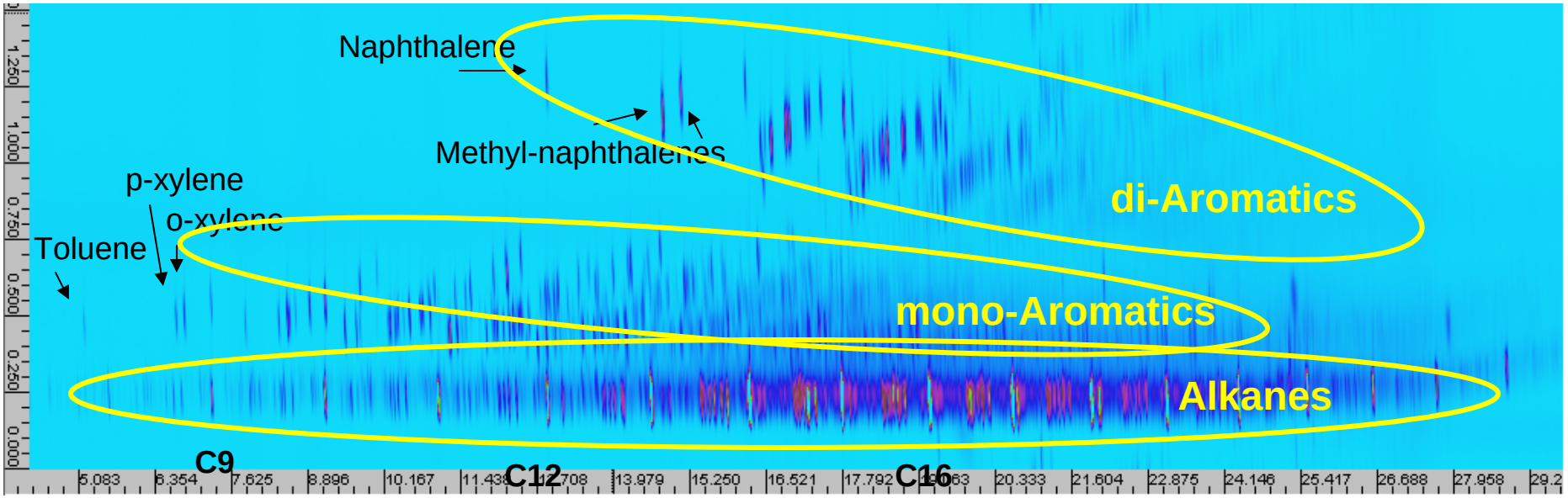
Agilent's flow modulator design : Differential Flow



Differential flow concept is designed 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 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 solves difficult application problems easily.

It opens up many new (and old) possibilities for GC and GC/MS systems.