

GC Analysis of PCBs in Fish Oil by Direct Injection Without Any Sample Cleanup

Philip L. Wylie, Ph.D.
Sr. Applications Chemist
Agilent Technologies

Pittsburgh Conference, 2008
Paper 1120-1
Tuesday, 8:30 am



Background Information

Fish are an excellent source of omega-3 fatty acids (DHA & EPA)

American Heart Association recommends eating fatty fish at least two times per week or taking a daily fish oil supplement

0.5-1.8 g of DHA + EPA /day “significantly reduces deaths from heart disease and all causes”

But fish and fish oil may contain pollutants:

PCBs, PBDEs, PCDDs, PCDFs, & heavy metals

American Heart Association (<http://www.americanheart.org/presenter.jhtml?identifier=4632>)

Analysis of PCBs in Fish Oil – The Challenges

209 possible PCB congeners

No single-column method separates them all

“Indicator” congeners measured in fish oil are: 28, 52, 101, 138, 153, 180 – can be difficult to resolve from other PCBs

Recommended method uses extraction followed by GC with High Resolution MS \$\$\$\$\$

Problems when trying to GC fish oil

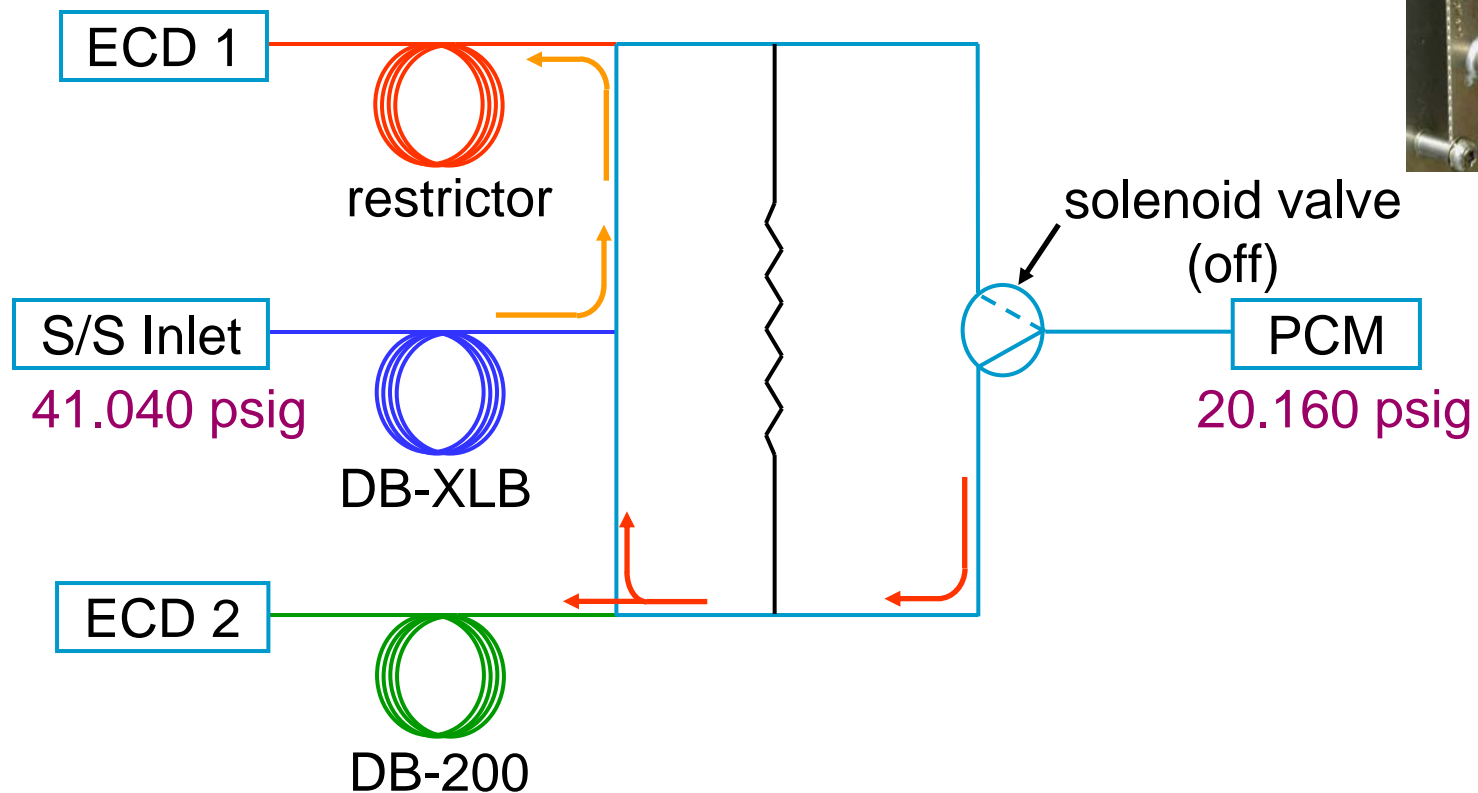
High boilers stay on column causing:

- carryover
- dramatic shifts in PCB retention times



The Solution: Agilent 7890A with Deans Switch for Heart Cutting and Backflushing

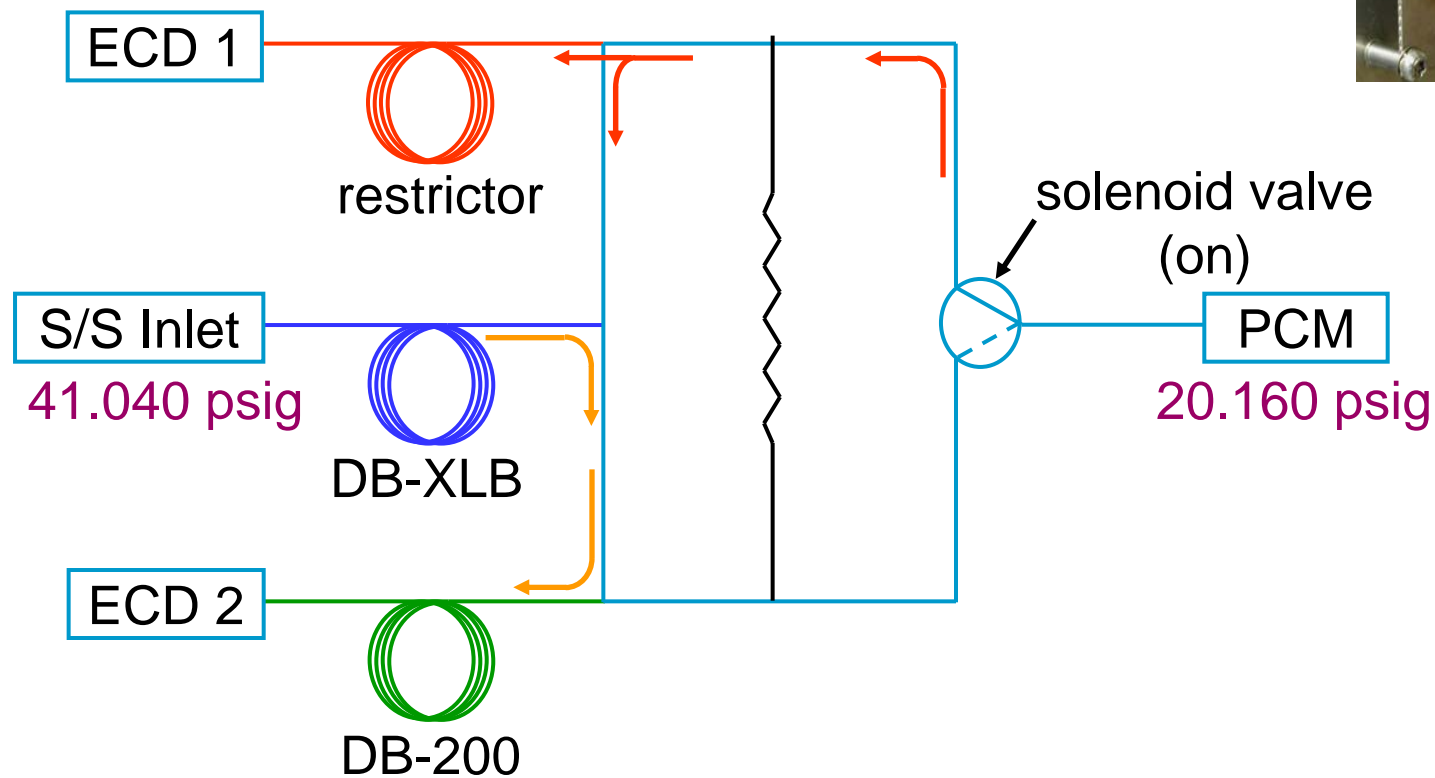
No Cut



Solenoid Valve "On" for Heart Cutting



Cut to Column 2

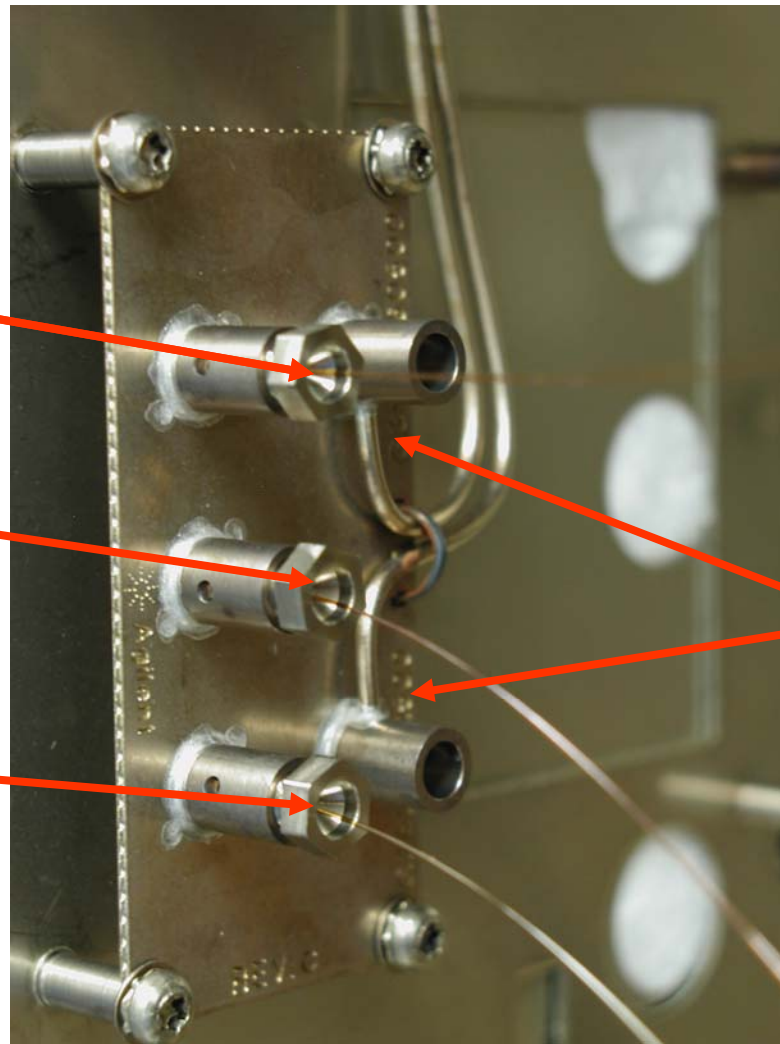


Deans Switch – One of Agilent's Capillary Flow Technology Devices

Restrictor to
back detector

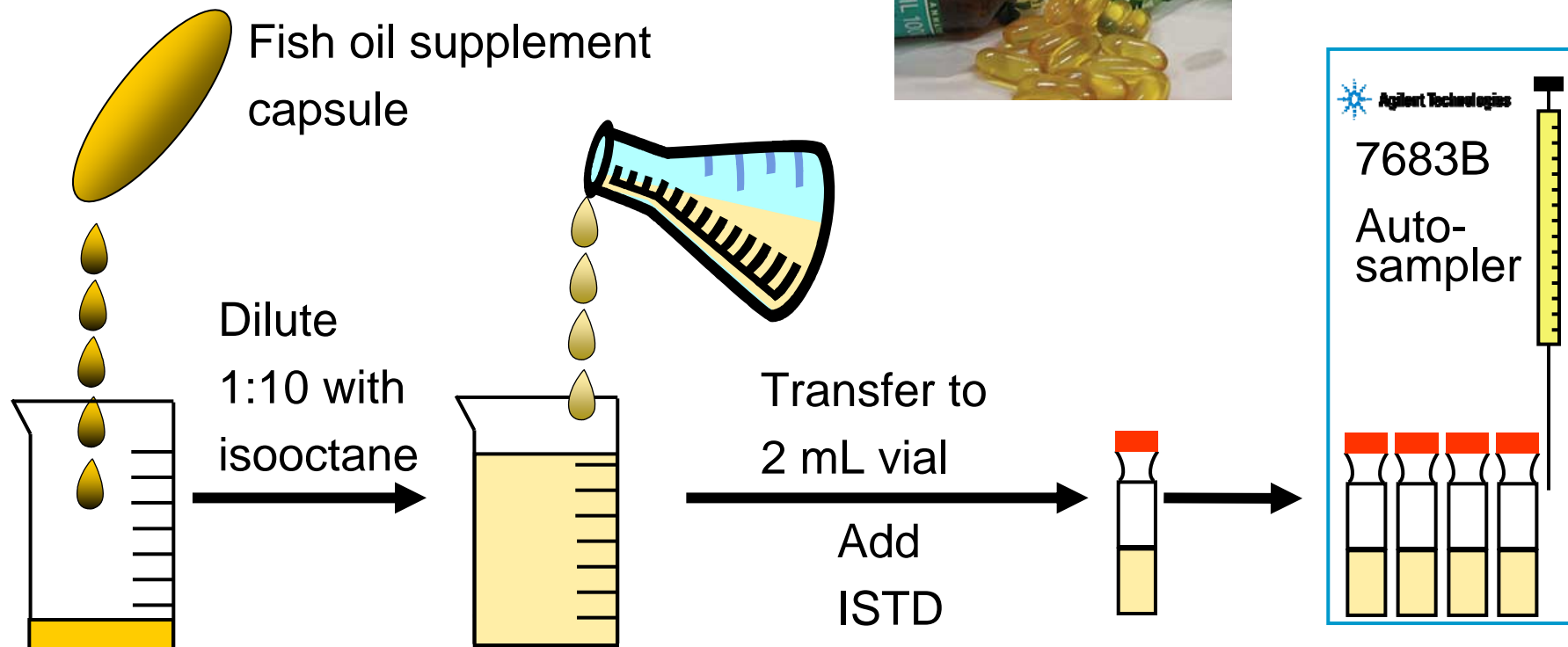
Primary column
DB-XLB

Secondary
Column DB-200

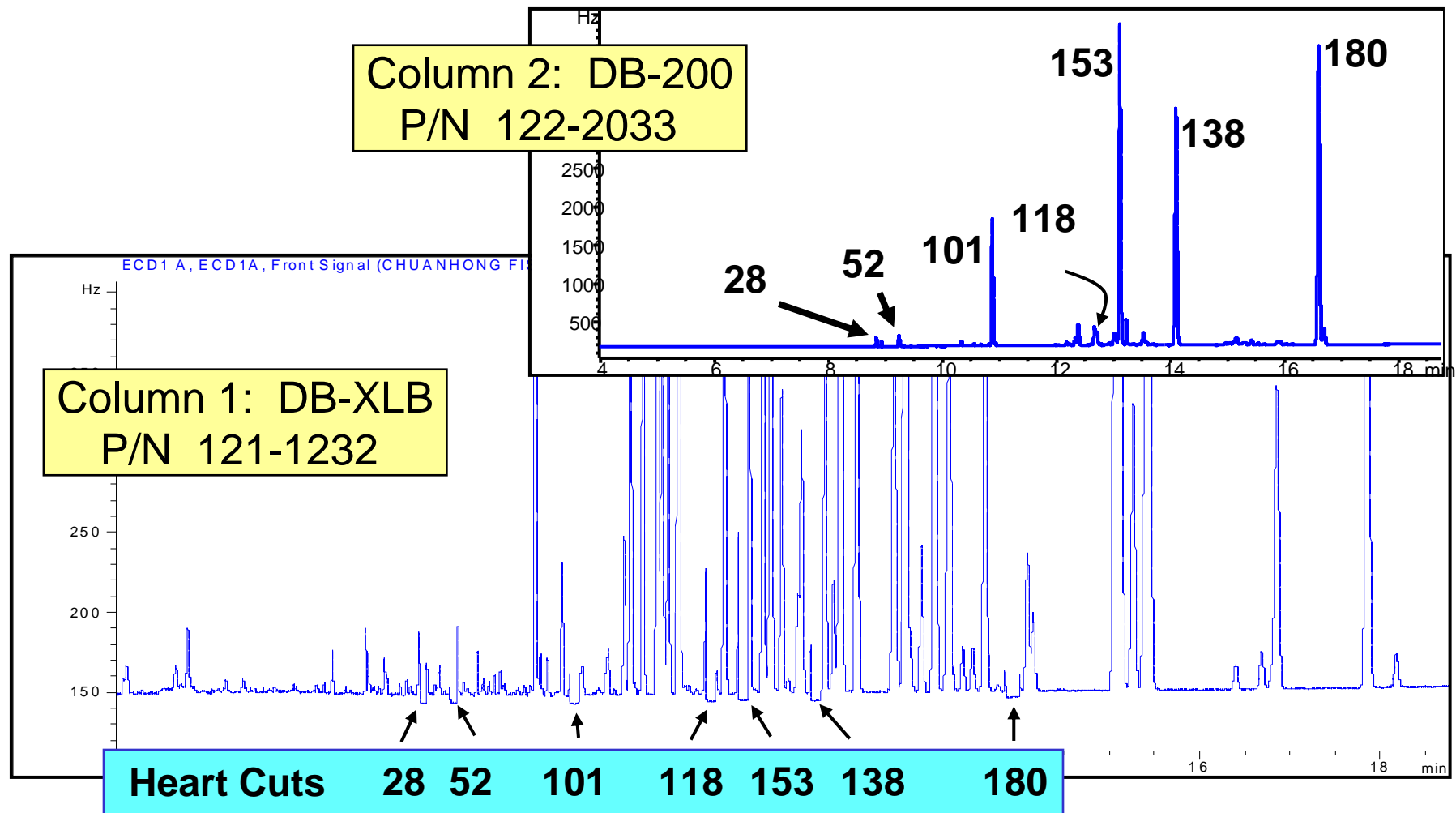


Pneumatic
connections to
the solenoid valve

Fish Oil Sample Preparation is “Dilute and Shoot”

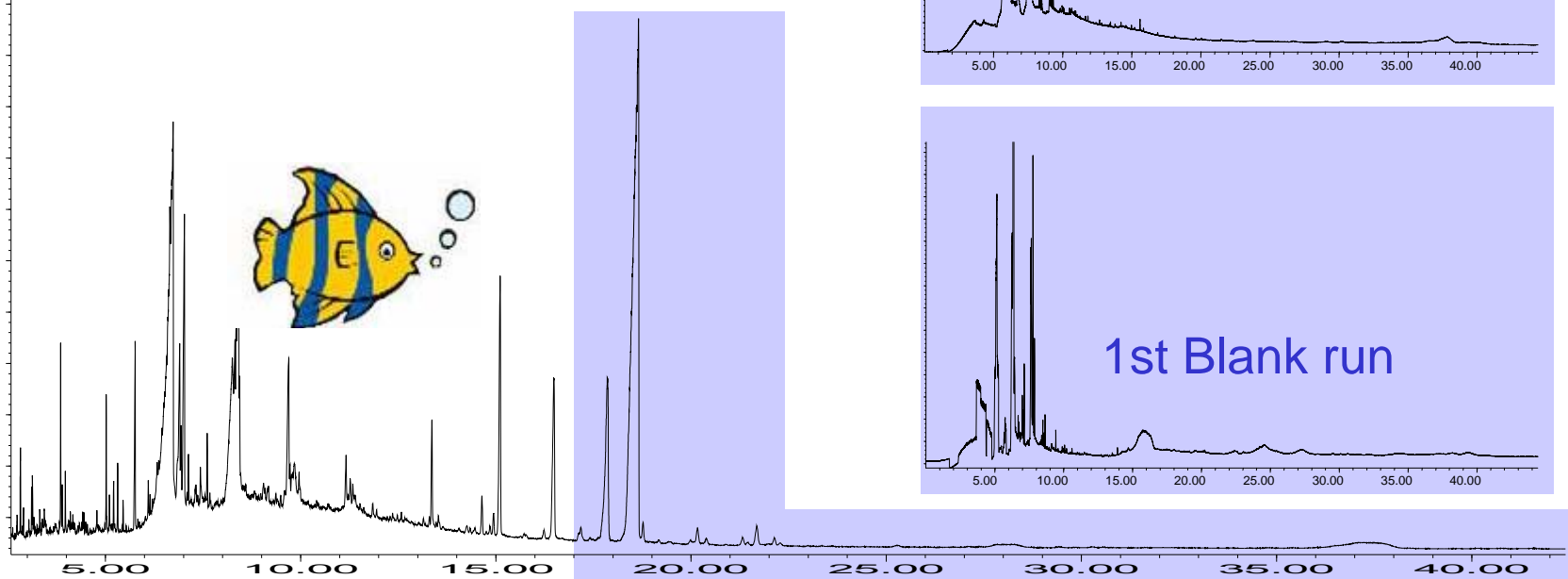


Heart Cutting with the Deans Switch to Isolate Target PCBs from Fish Oil Spiked with Aroclor 1260



GC-FID Chromatogram of Fish Oil with No Backflushing

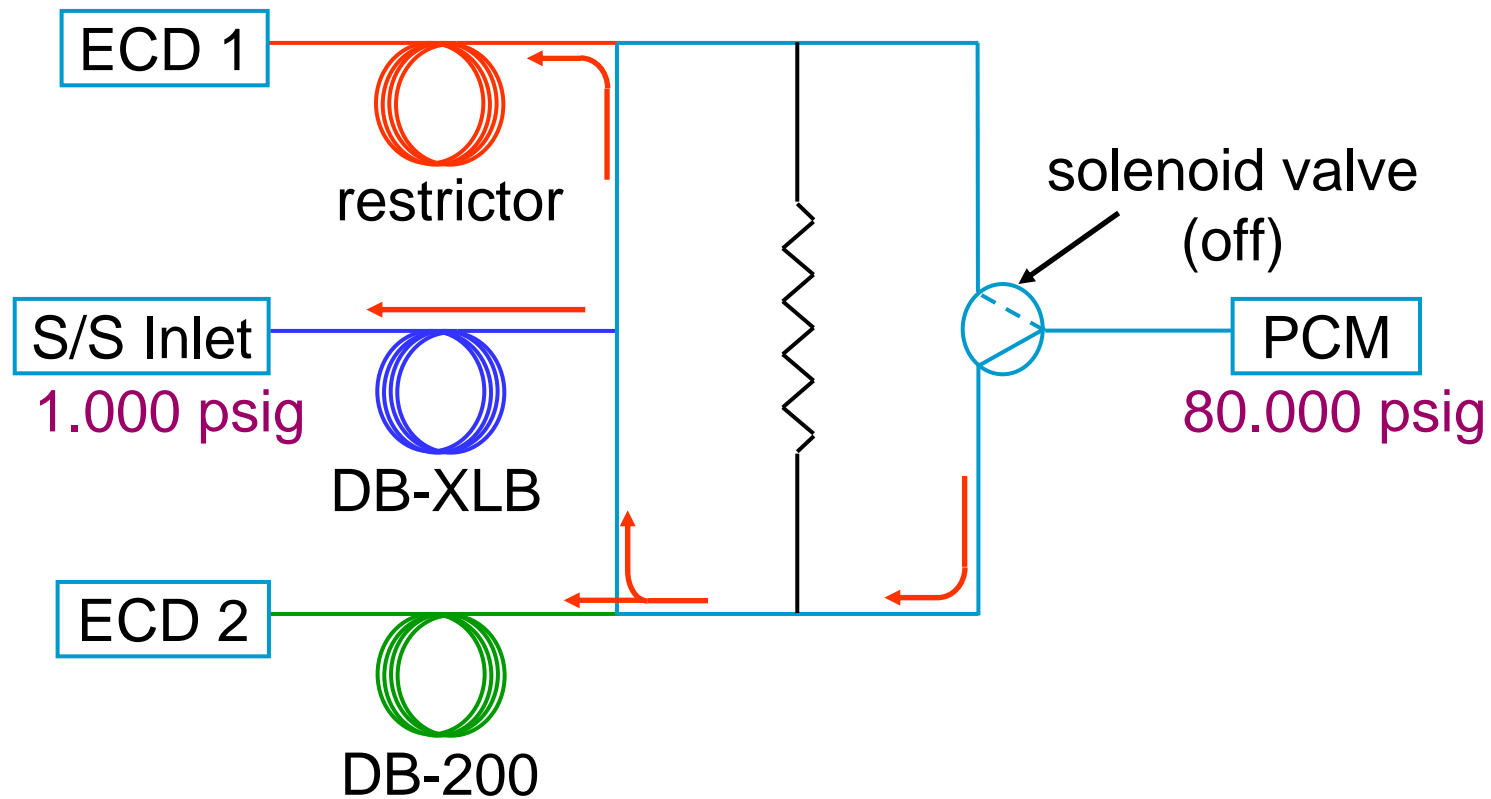
1- μ L splitless injection of 10% fish oil without backflushing. GC oven was held at 290 °C for 30 min at the end of the run.



Highlighted areas are backflushed

Deans Switch in Backflush Mode

Backflushing Column (3 min @ 295 °C)



New ChemStation Software Makes it Easy to Set Up Backflushing

Setting Up Backflush Method

Edit Method: Filename (D:\VCCSnaps\Ohana\LFS_Drivers\parts\78SeriesGC\BinBuilt\DualECD.config.xml)

Backflush - Front Backflush - Back

SS Inlet
PCM B-A
Column 1: 30 mx180 μm x 0.18 μm
295 °C
Column 2: 30 mx250 μm x 0.5 μm
Column 3: 0.77 mx100 μm x 0 μm
Back - ECD
Front - ECD

Evaluate...

Summary of Backflush Calculations

Oven Temperature
295 °C

| Detector | Maximum Flow | Allowable Pressure | Flow at Chosen Pressure |
|-------------|--------------|--------------------|-------------------------|
| Back - ECD | 60 | 174.02 | 14.834 |
| Front - ECD | 60 | 172.77 | 15.034 |

Backflush Pressure
80 psi

Inlet Pressure during Backflush
1 psi

Void Volumes
6.8474

Backflush Flow to Inlet
4.0045 mL/min

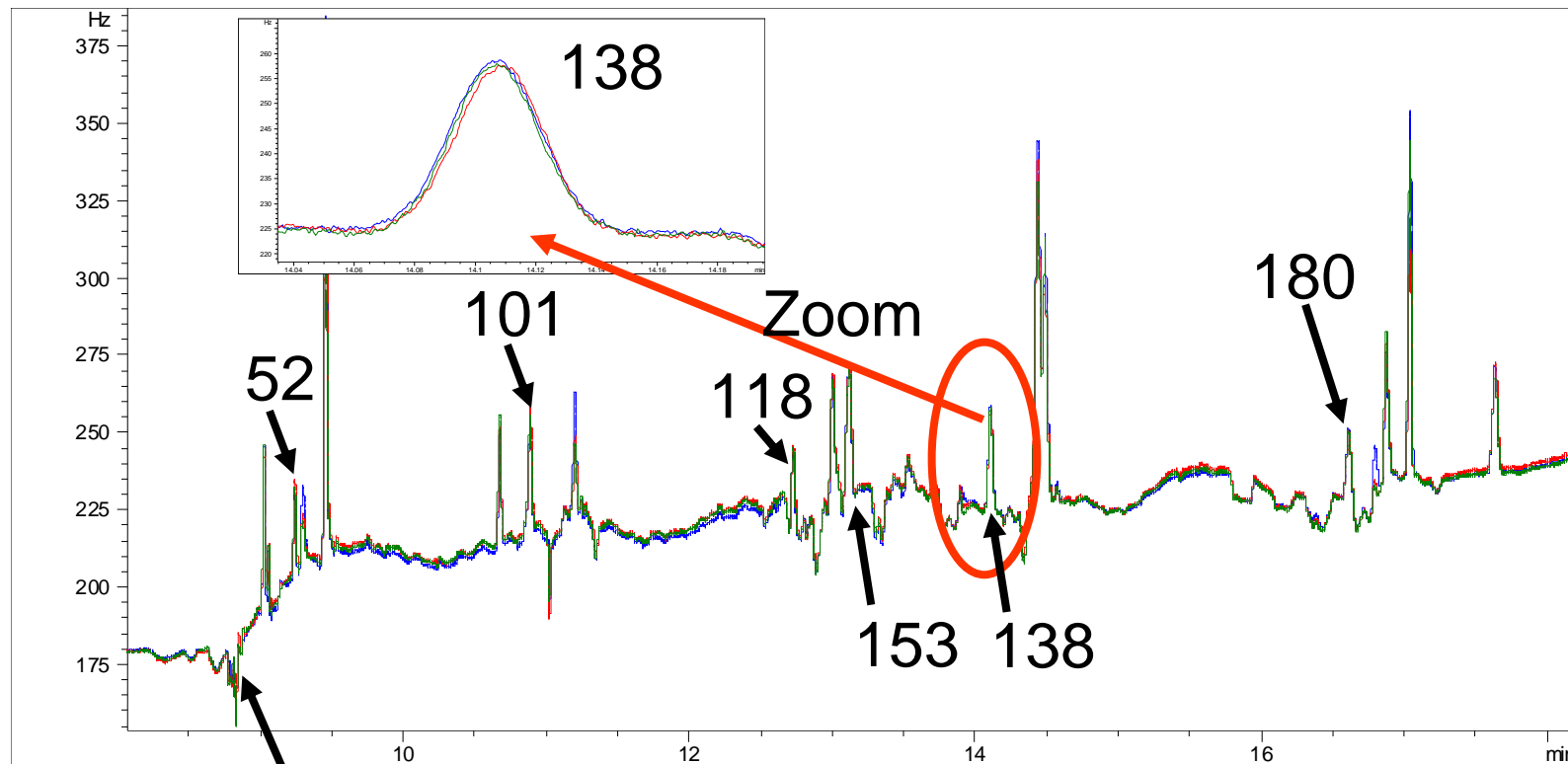
Backflush Time
3 min

Void Time
0.43812 min

OK Cancel Help

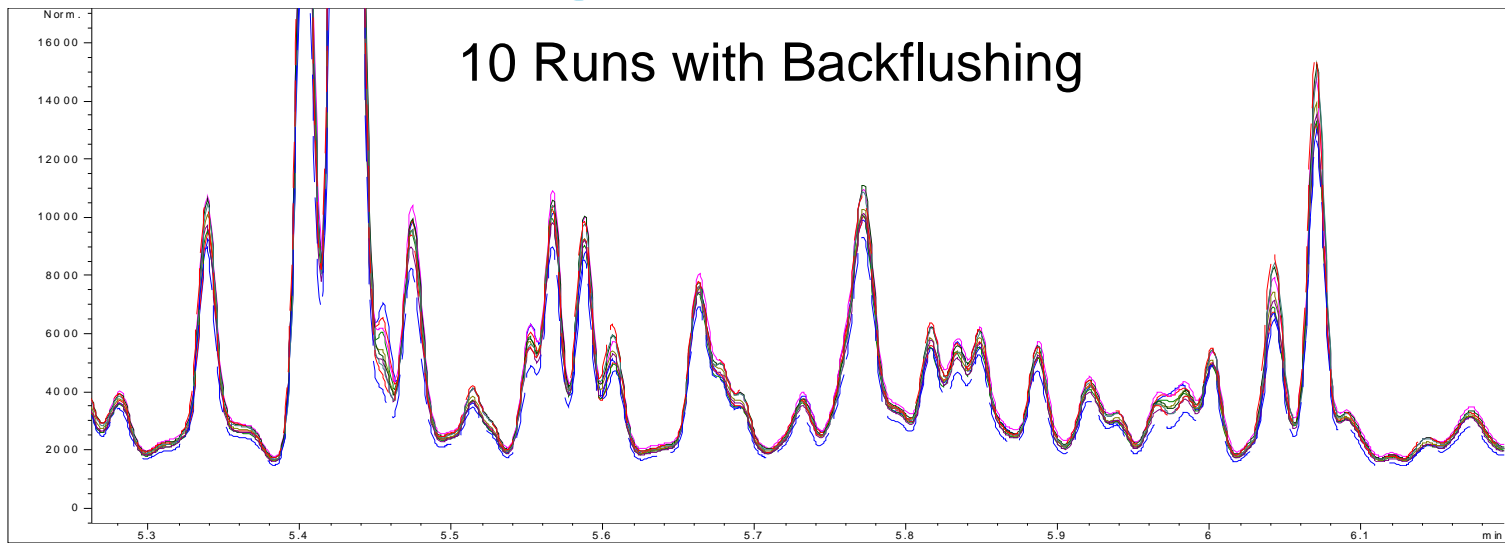
From a 35-Sample Sequence: Chromatograms 2, 20, & 35 Overlaid

PCBs Spiked at 0.5 ppb Each

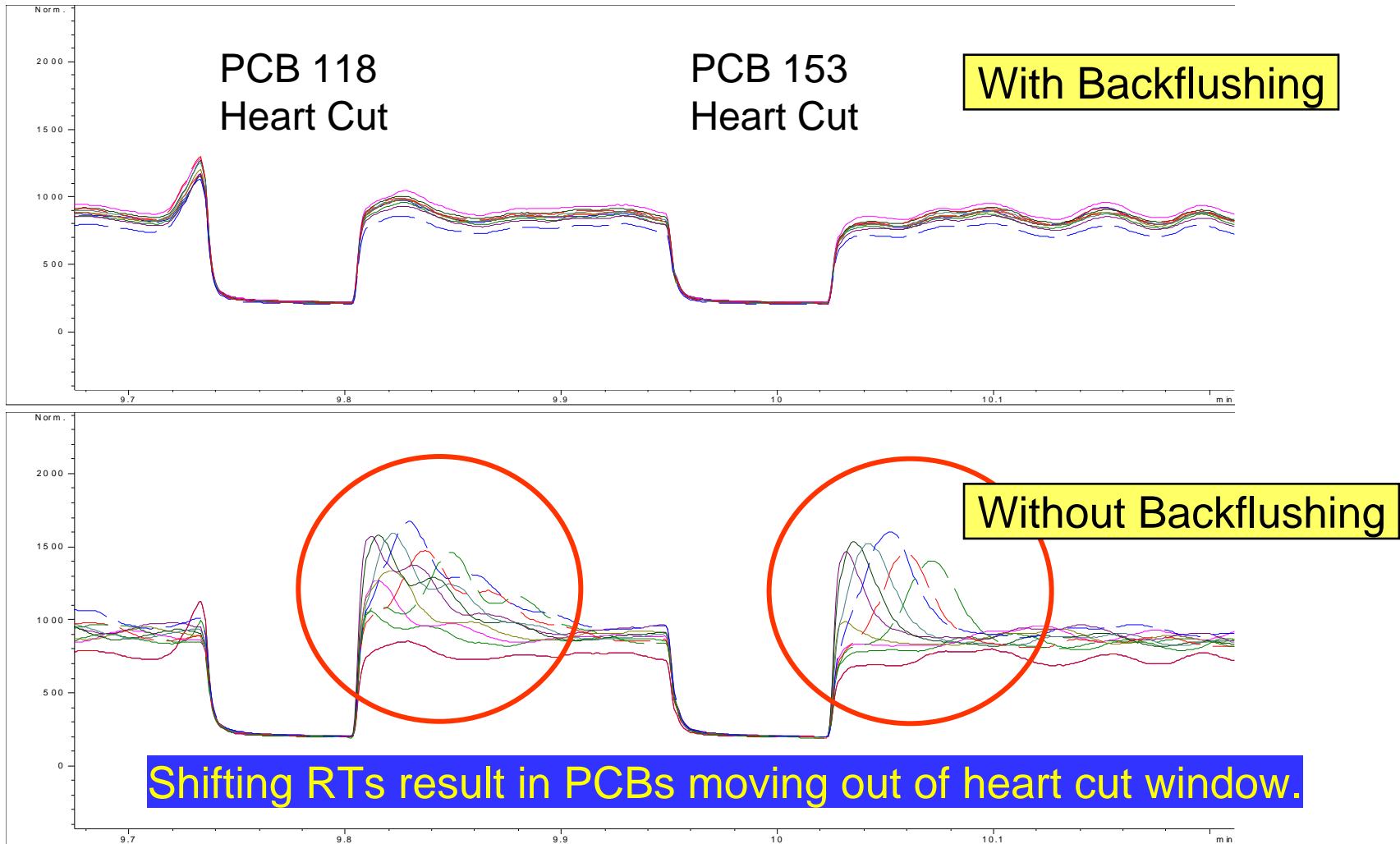


At 0.5 ppb, PCB 28 is obscured by an interference in the Fish oil

Retention Time Precision Column 1 with and without Backflushing

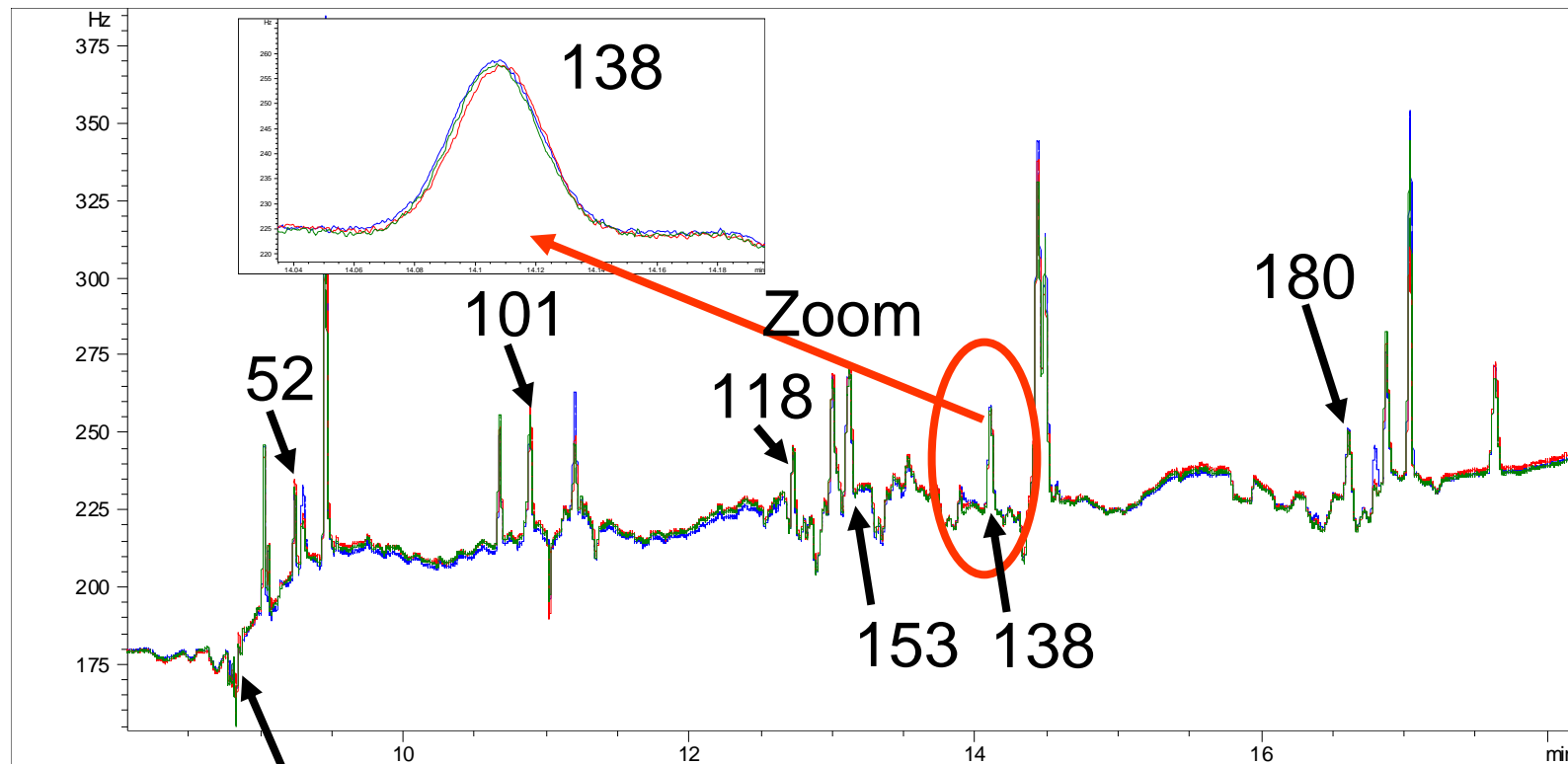


Retention Time Precision Column 1 with and without Backflushing –



From a 35-Sample Sequence: Chromatograms 2, 20, & 35 Overlaid

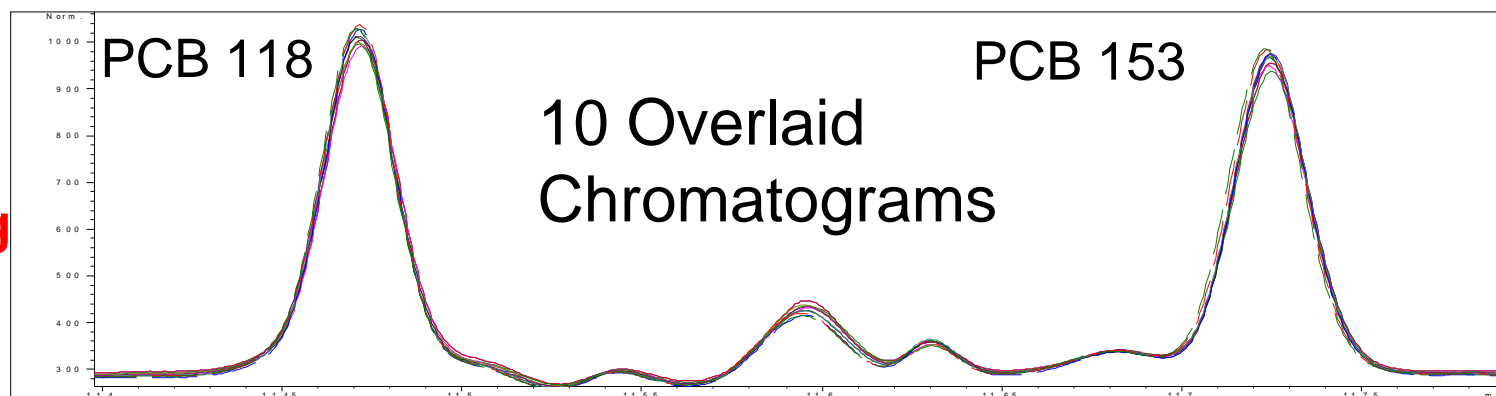
PCBs Spiked at 0.5 ppb Each



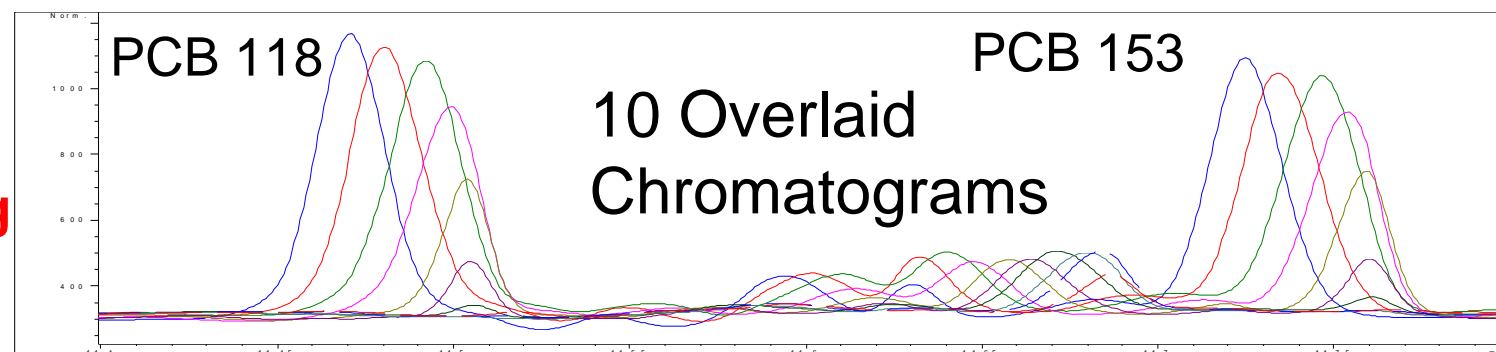
At 0.5 ppb, PCB 28 is obscured by an interference in the Fish oil

PCBs 118 & 153 Heart Cut to DB-200 Column – with & without Backflushing

**With
Backflushing**

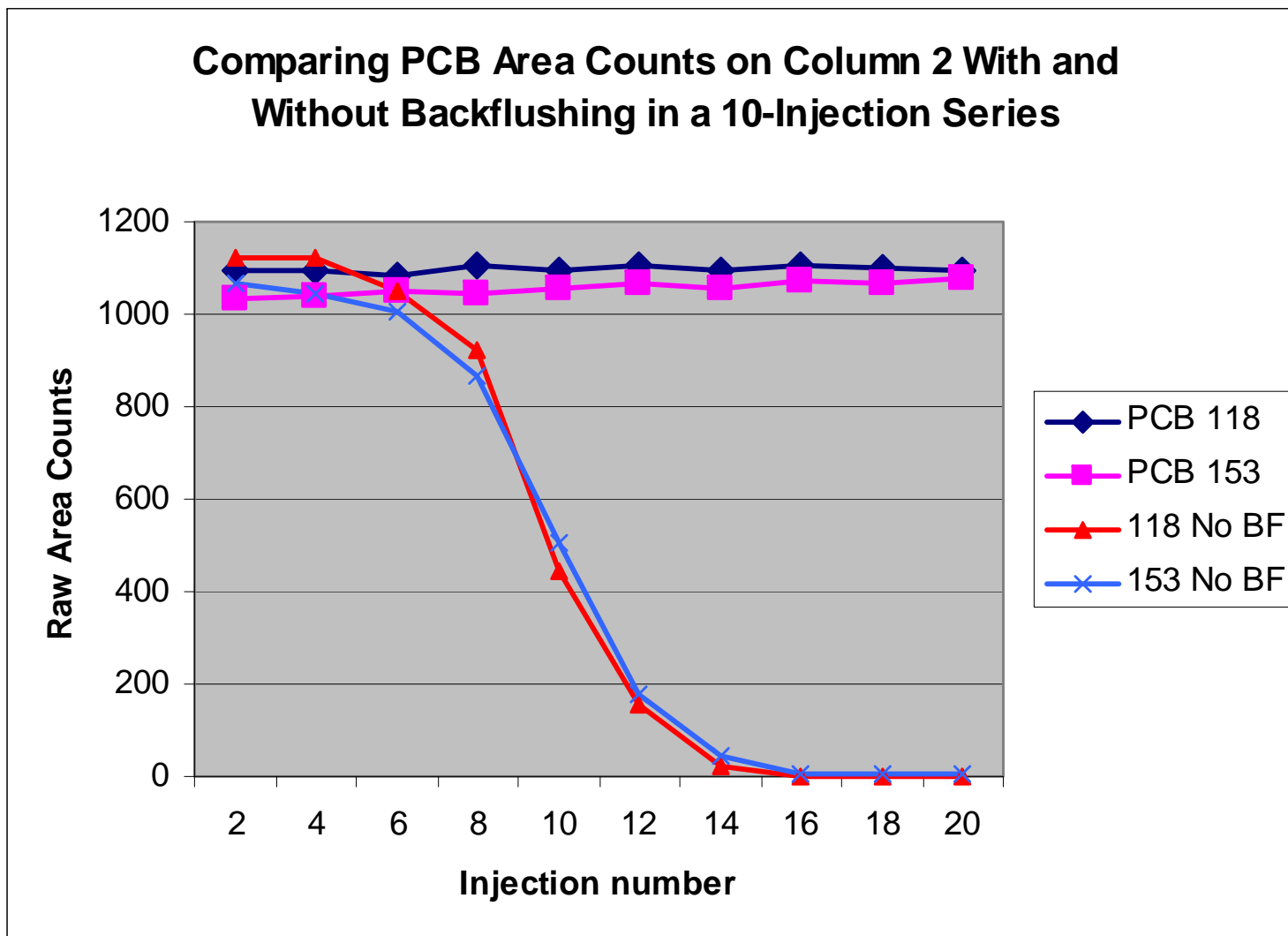


**Without
Backflushing**

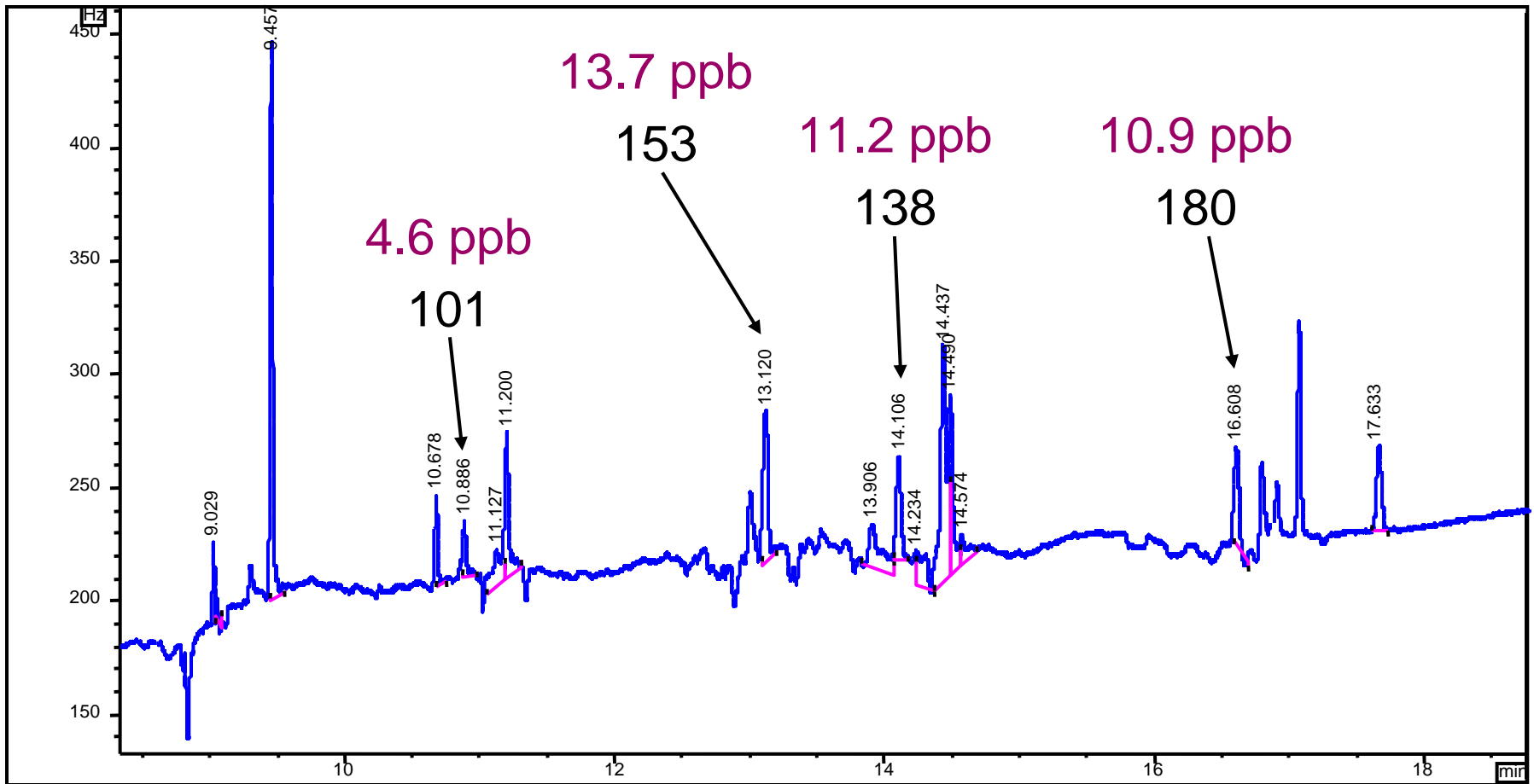


Retention times shift by 2.0 seconds in first 7 runs on the secondary column (DB-200)

Comparing Area Count Stability on Secondary Column for 2 PCBs – With and Without Backflushing



Quantitative Analysis of Indicator PCBs in a Real Fish Oil Sample (Un-spiked)



Conclusions

Capillary Flow Technology – Deans switch

- extremely low dead volume
- inert
- does not leak
- tracks oven temperature
- easy column installation



Backflushing made easy by the ChemStation software

Can configure columns to Capillary Flow Devices and easily set pressures/flows – 7890A is extremely versatile!

For this application...

- No fish oil extraction – **saves time and \$**
- Low column maintenance - **saves time and \$**
- HR/MS or HR/MS/MS not needed – **Saves ~ \$400,000**

More than 100 fish oil injections made without inlet or column maintenance

Thank You



<http://www.headington.org.uk/shark/>

