

Extending analyte boiling point range using thinner film porous layer open tubular columns paired with GC×GC-MS

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University of Washington

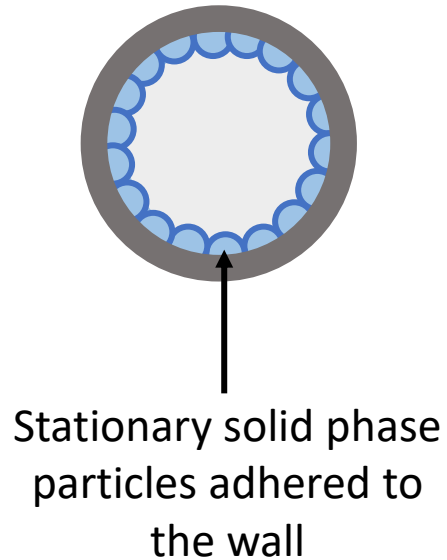
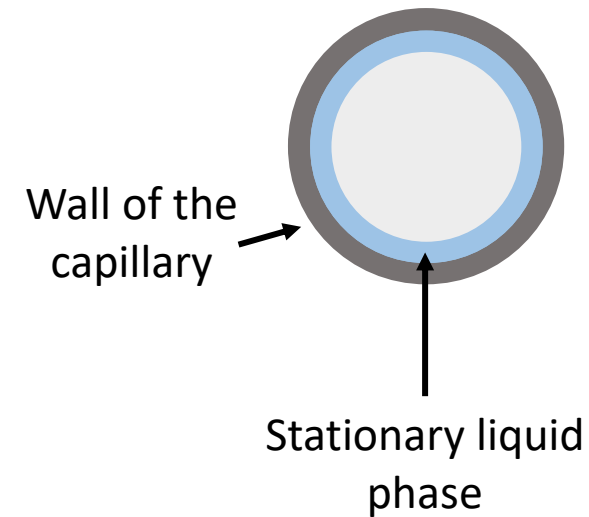
15th Multidimensional Chromatography Workshop

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WCOT vs PLOT columns

Wall Coated Open
Tubular Column

Porous Layer Open
Tubular Column

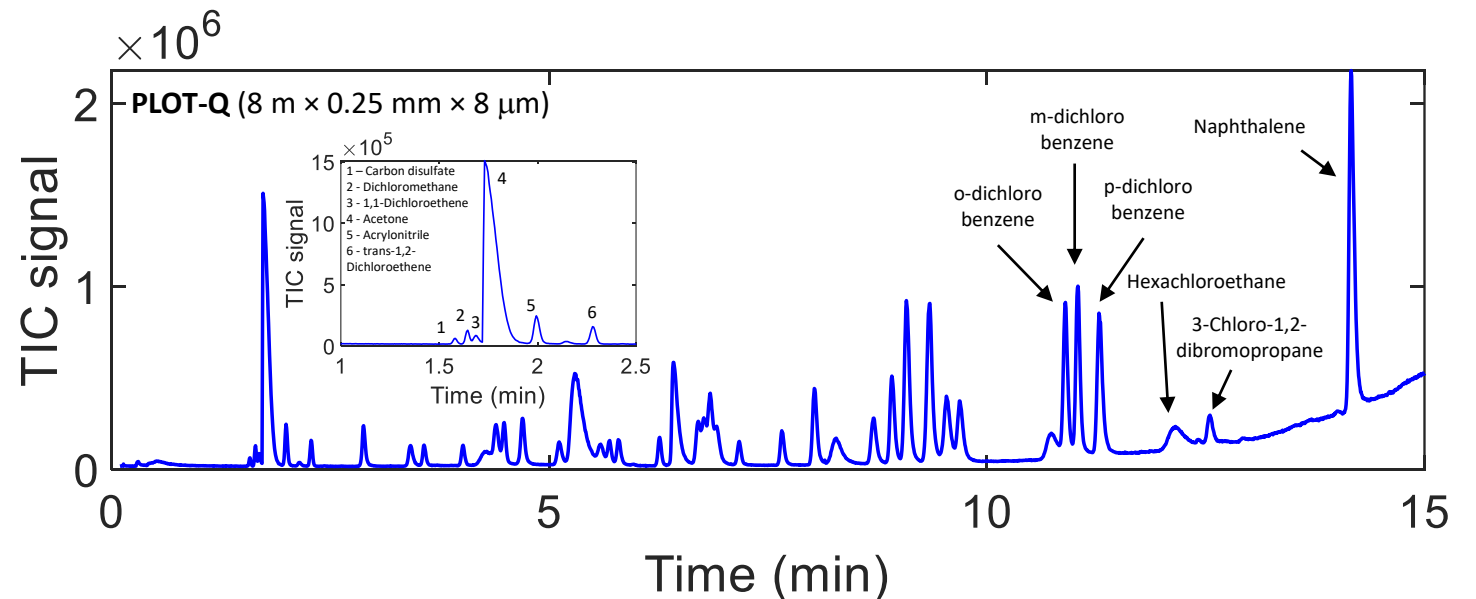
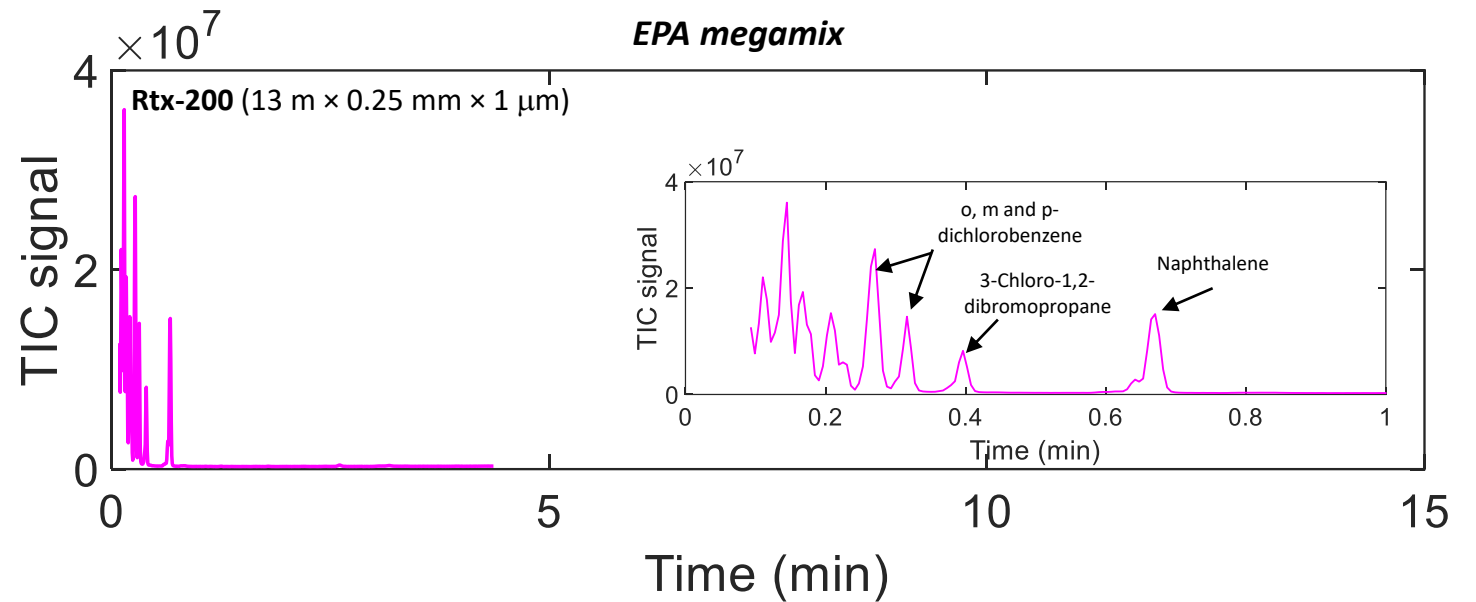


- PLOT columns have maximum flow rate limitation
- Usually used with FID and not MS
- They have temperature limitations
- ***However, much better for low boiling point analytes***

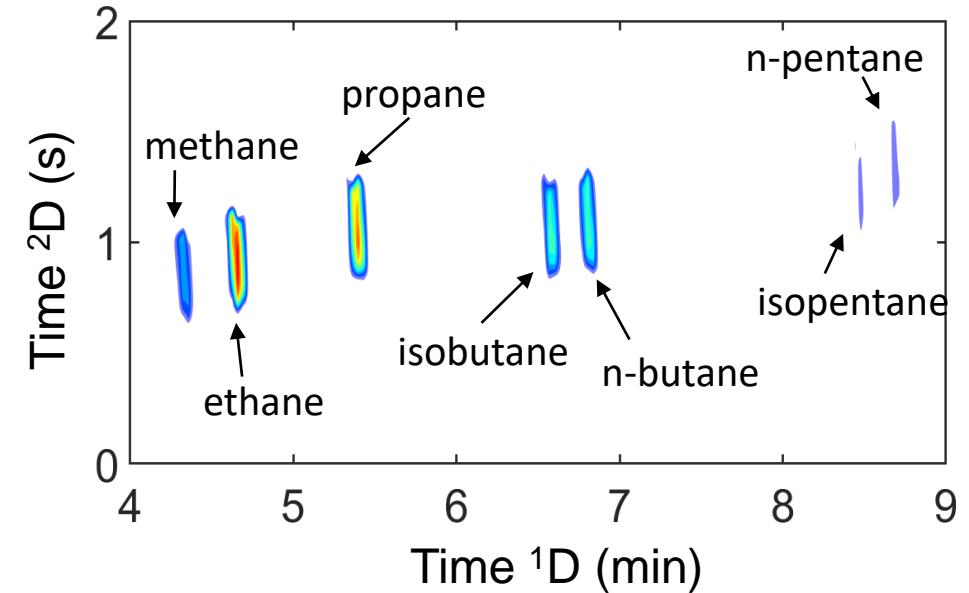
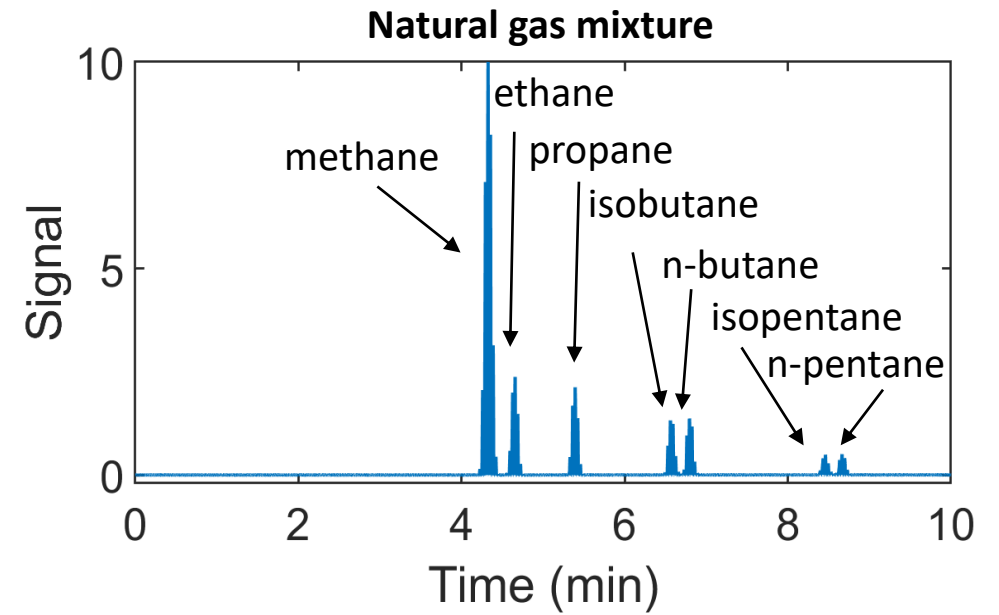
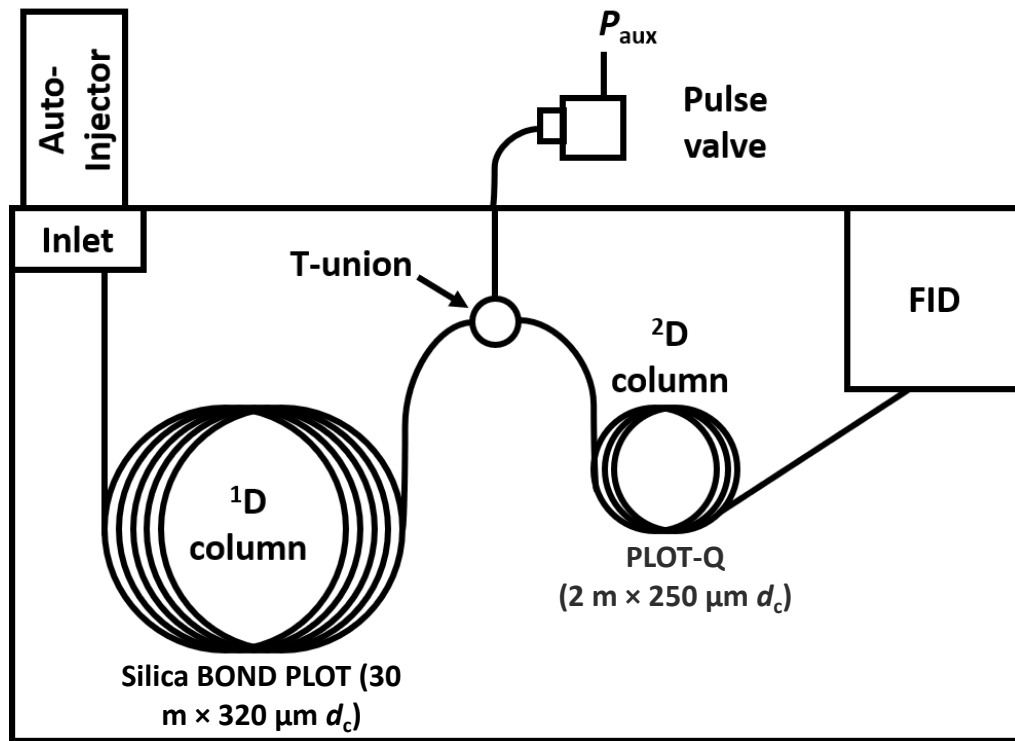
WCOT vs PLOT columns for light compounds

Experimental conditions:

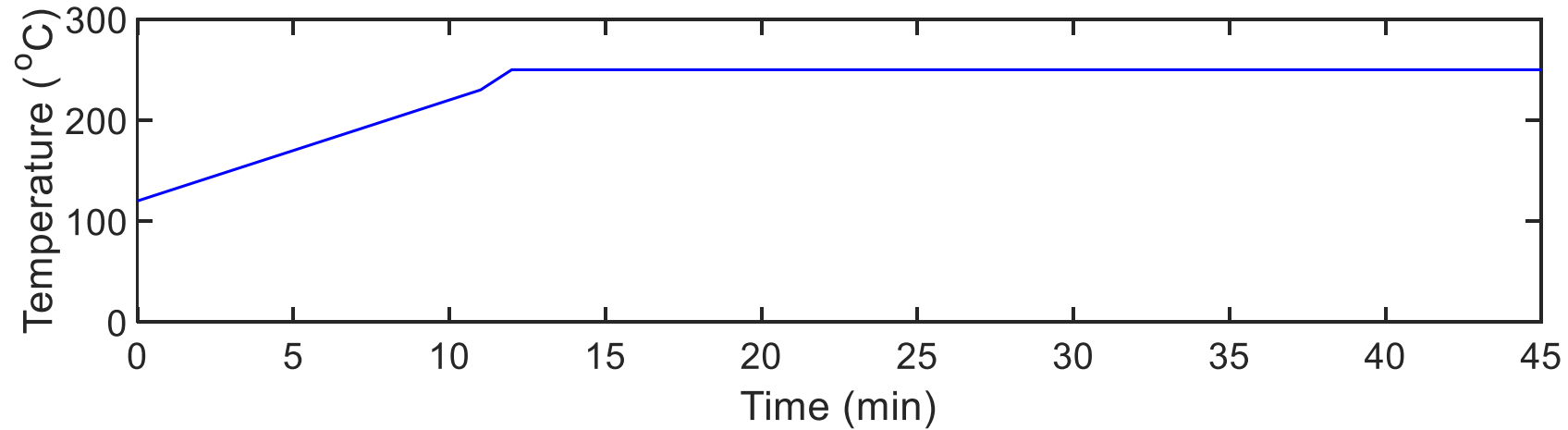
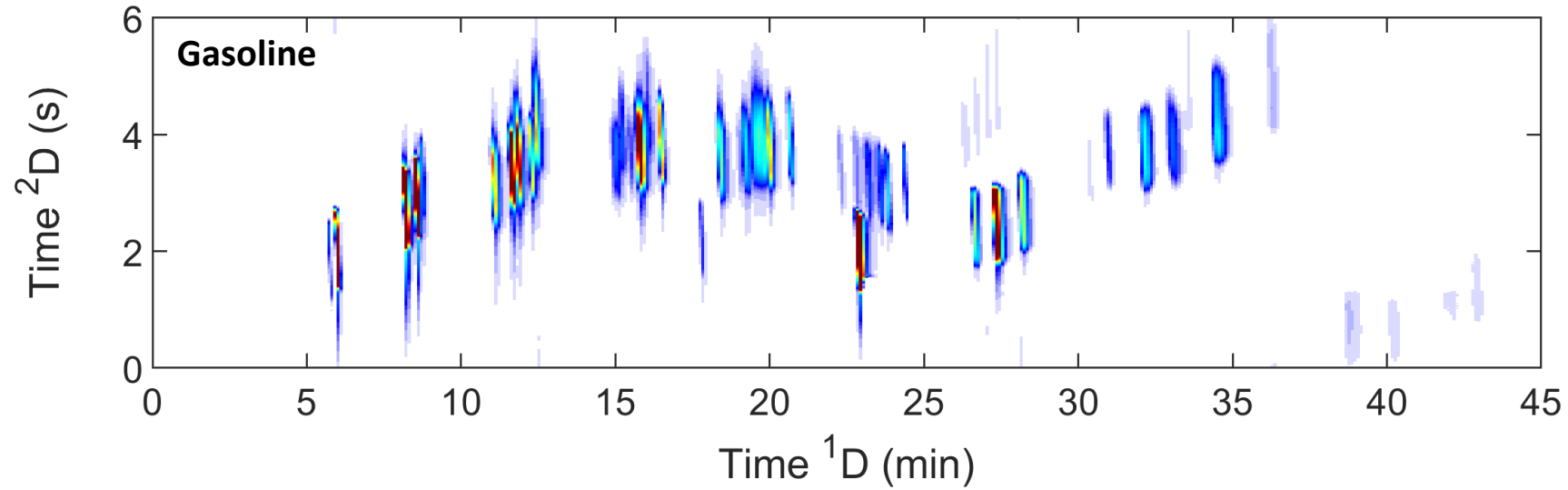
- temperature program was started at 100 °C and ramped at 10 °C/min to 250 °C;
- 2 μL of sample were injected both times using 1:25 split;
- columns were run at 1.5 ml/min He carrier gas.



Application of PLOT columns

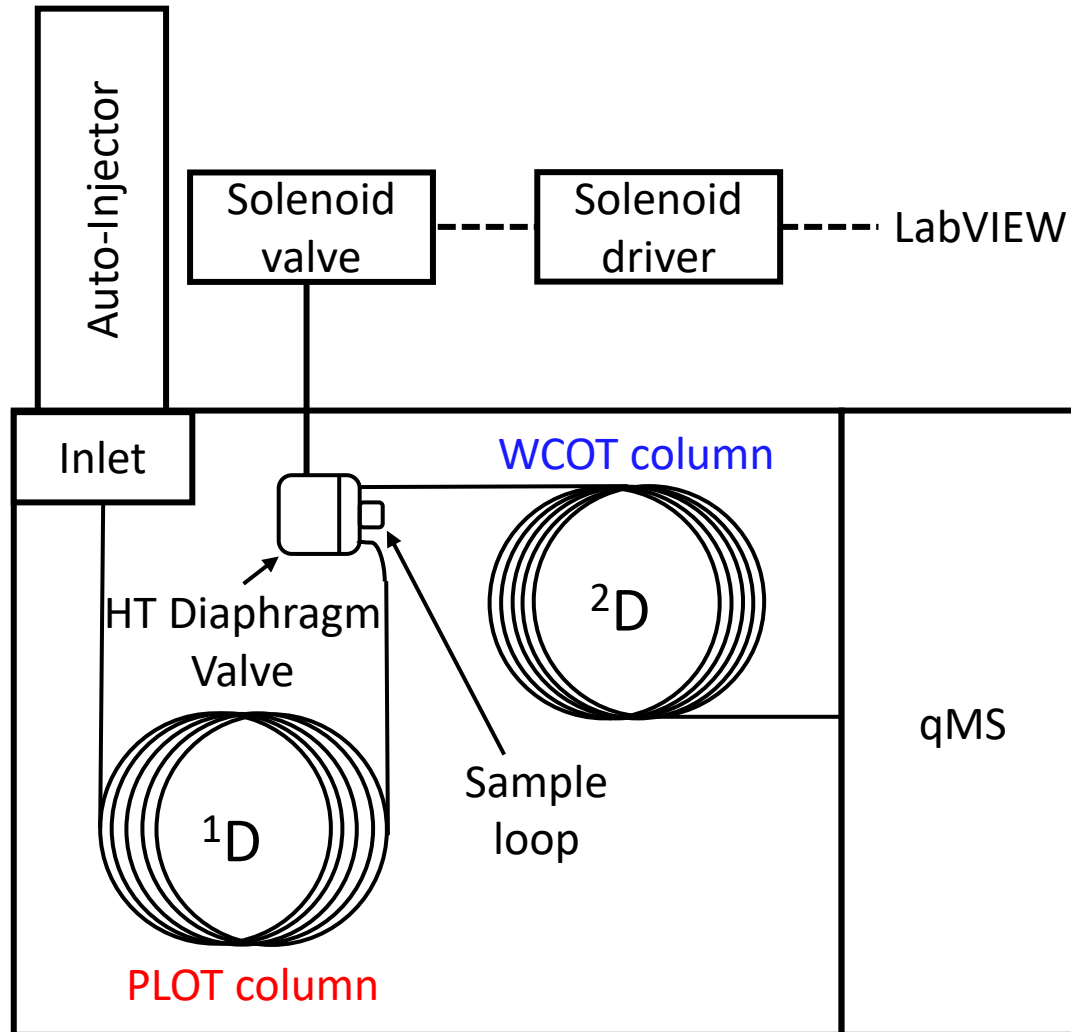


Light sample on PLOT columns



Is there a way to use PLOT columns for heavier compounds?

Next generation PLOT-based GC×GC using qMS for added selectivity: ultralight to semi-volatile compounds



Sample loop: 10 μL

1D column: **PLOT – S** (5 m x 250 μm x 8 μm)

Flow 1D: 1.4 ml/min

2D column: **Rtx – 200** (5 m x 150 μm x 2 μm)

Flow 2D: 2.0 ml/min

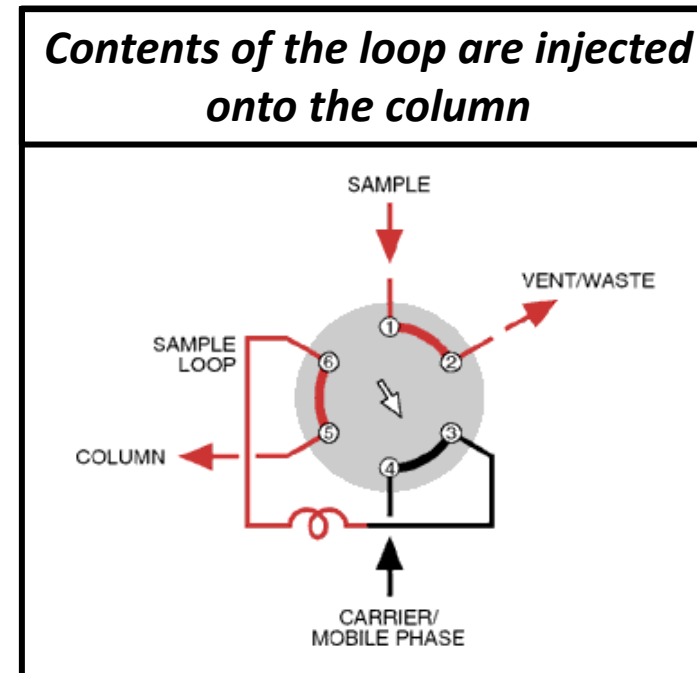
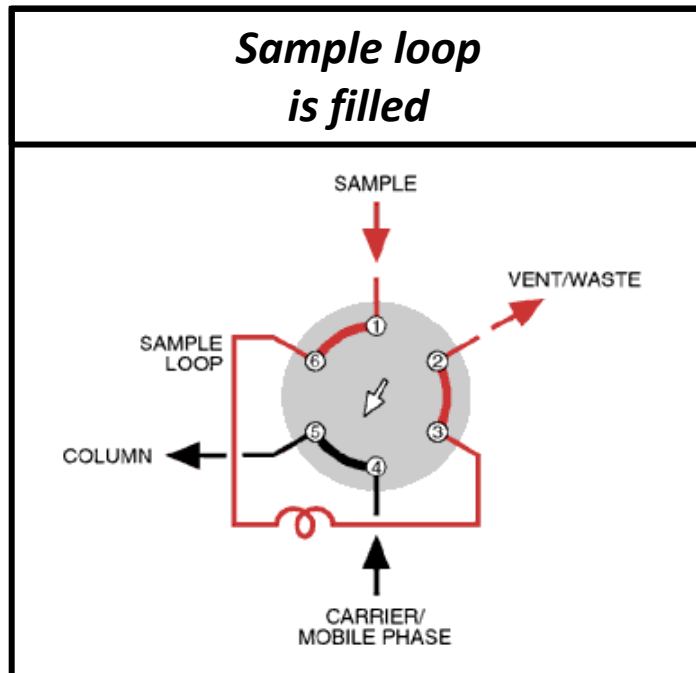
Temperature program:

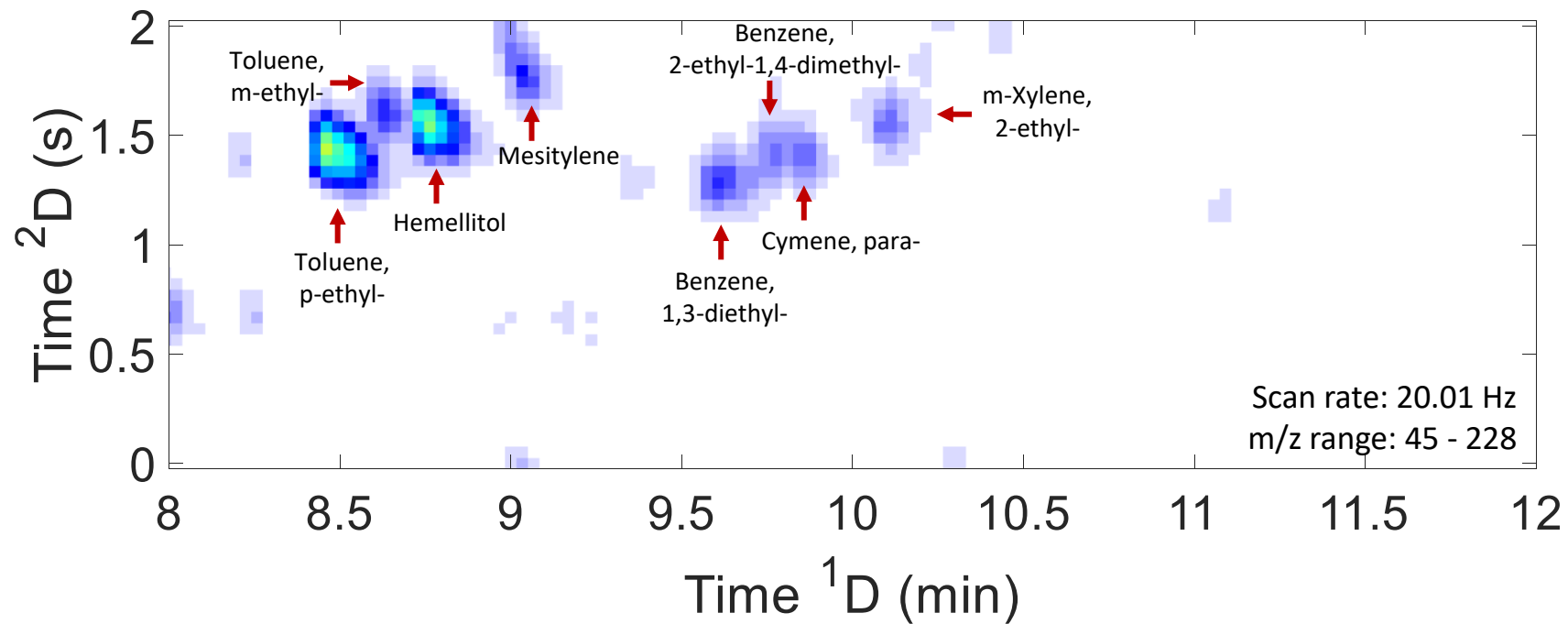
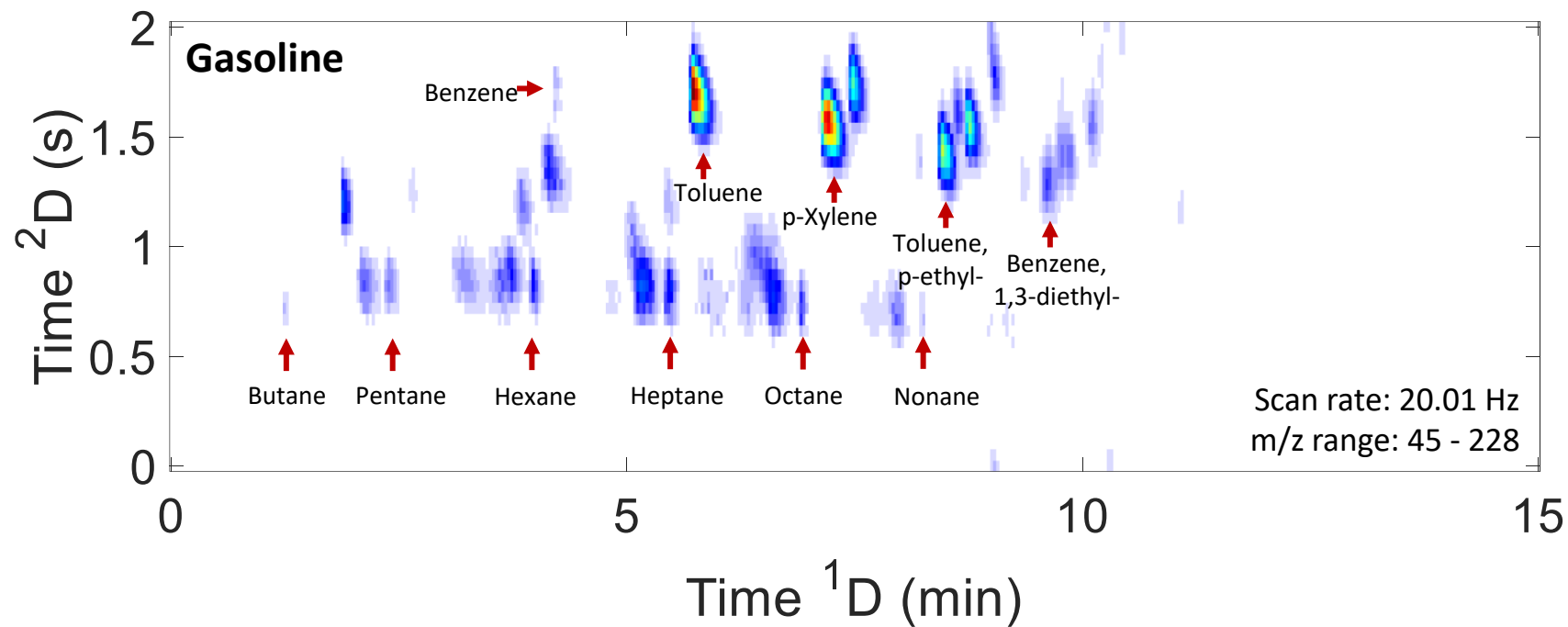
100 °C to 250 °C @ 15 °C/min

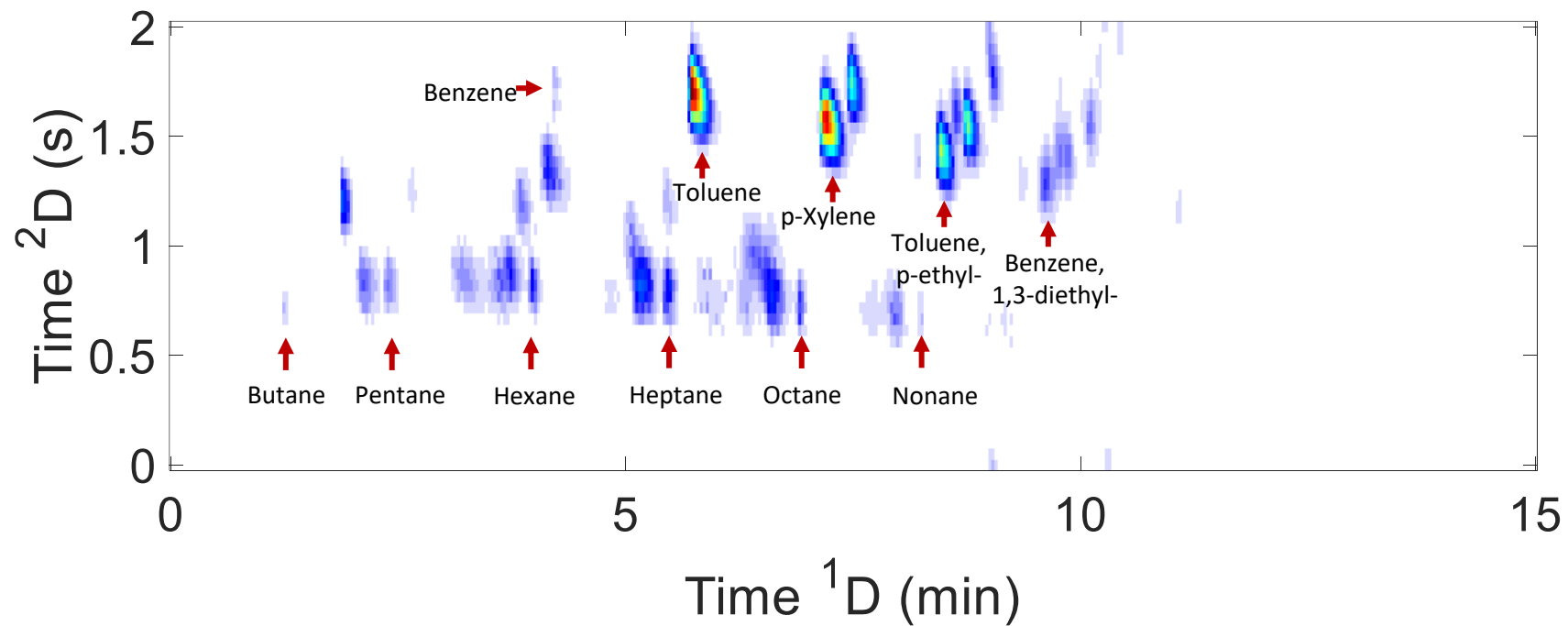
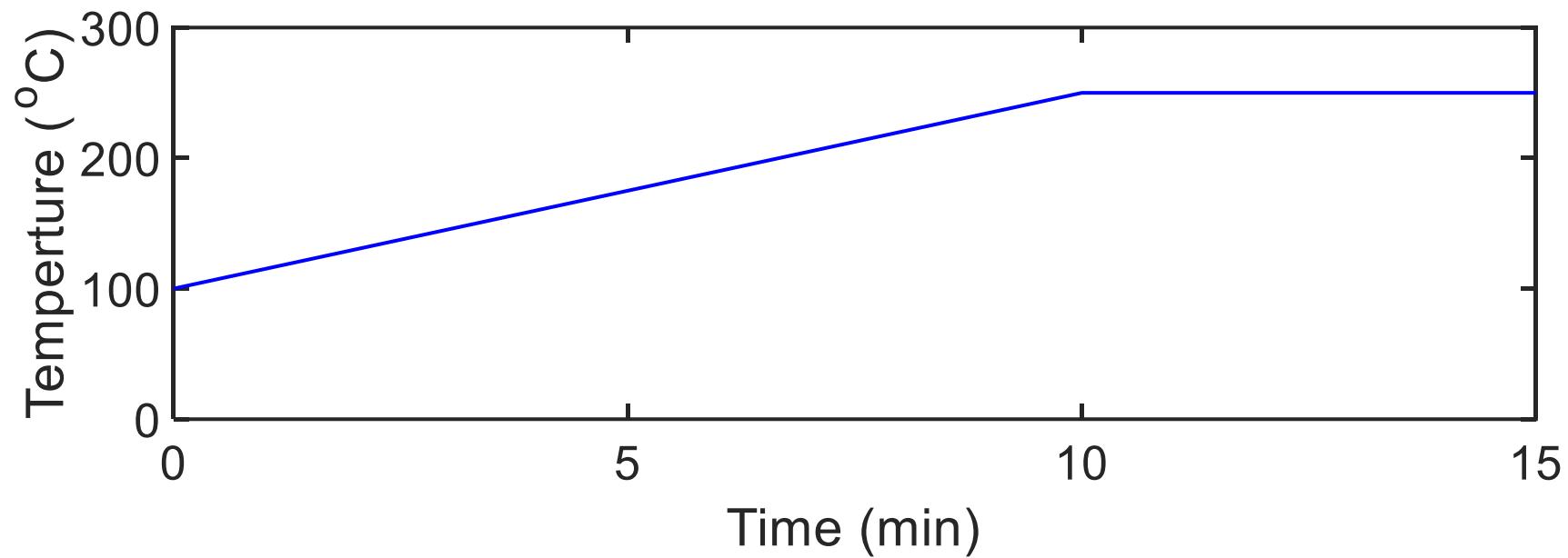
$P_M = 2$ s and $p_w = 250$ ms

Diaphragm Valve for process GC x GC

Custom made diaphragm valve:
Can be used up to 325 °C, as temperature sensitive O-rings have been replaced with Kalrez O-rings.

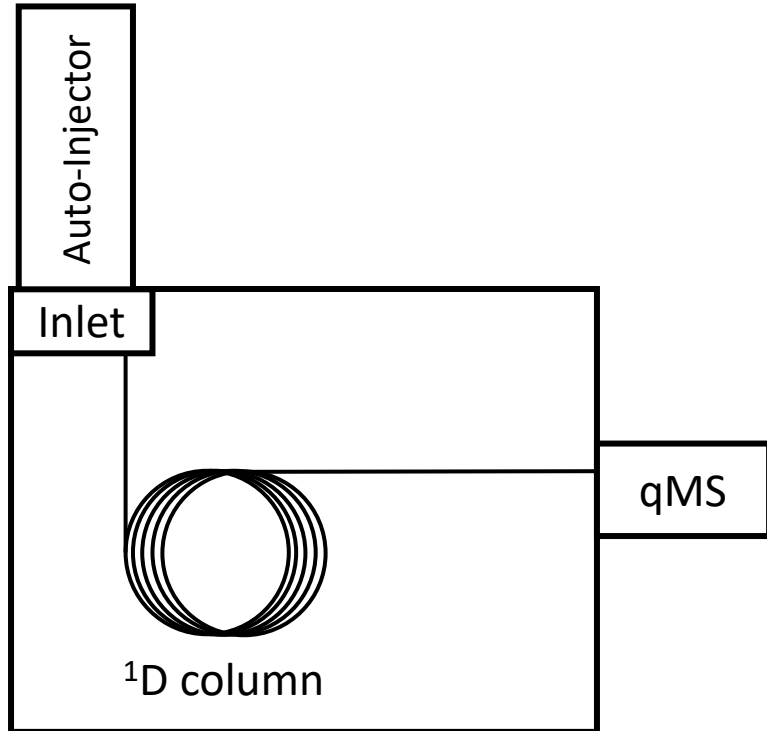






Can we do even better?

Reducing PLOT column film thickness (d_f) for extended analyte boiling point range



PLOT-S columns:

- 30 m x 0.25 mm x **8 μm**
- 30 m x 0.25 mm x **4 μm**
- 30 m x 0.25 mm x **2 μm**

Samples:

- Calibration MegaMix (75 compounds)
 - Refinery mix (27 compounds)

Flow: 1.4 mL/min

Injection volume:

Calibration mix: 0.1 μL

Refinery mix: 40 μL

Split: 50:1

Mass channel range:

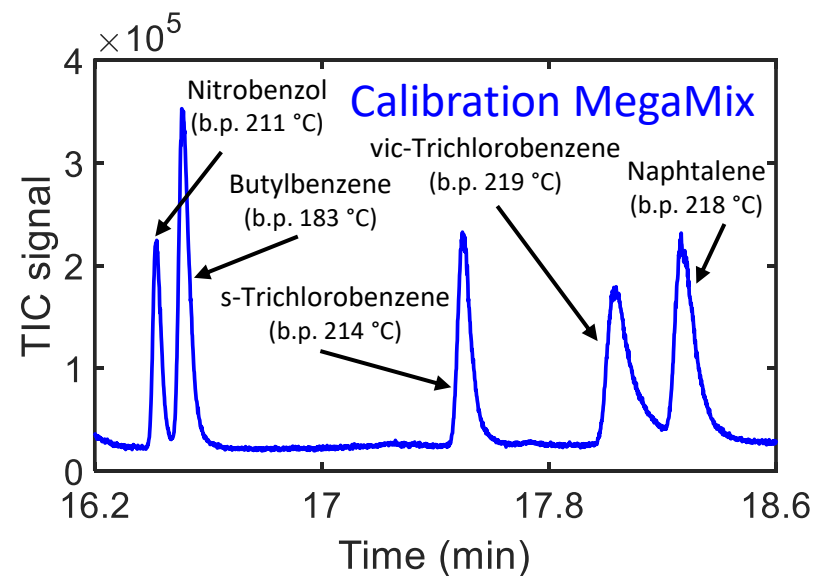
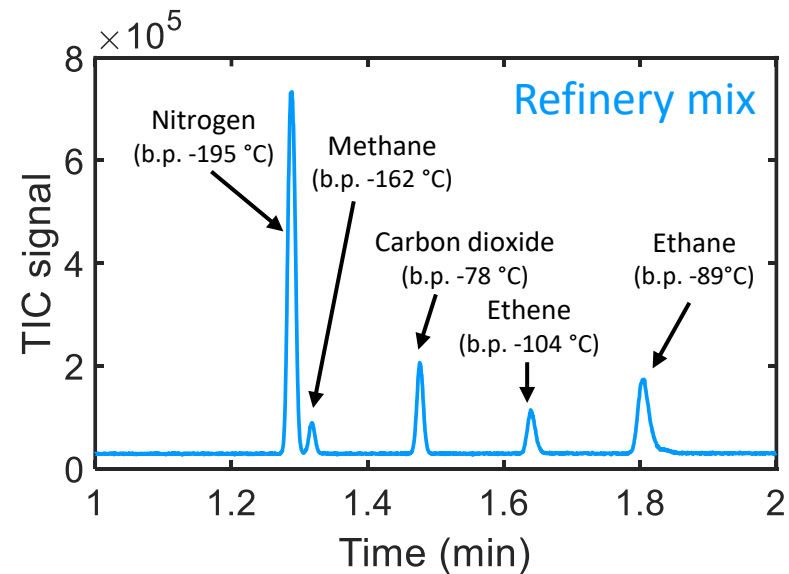
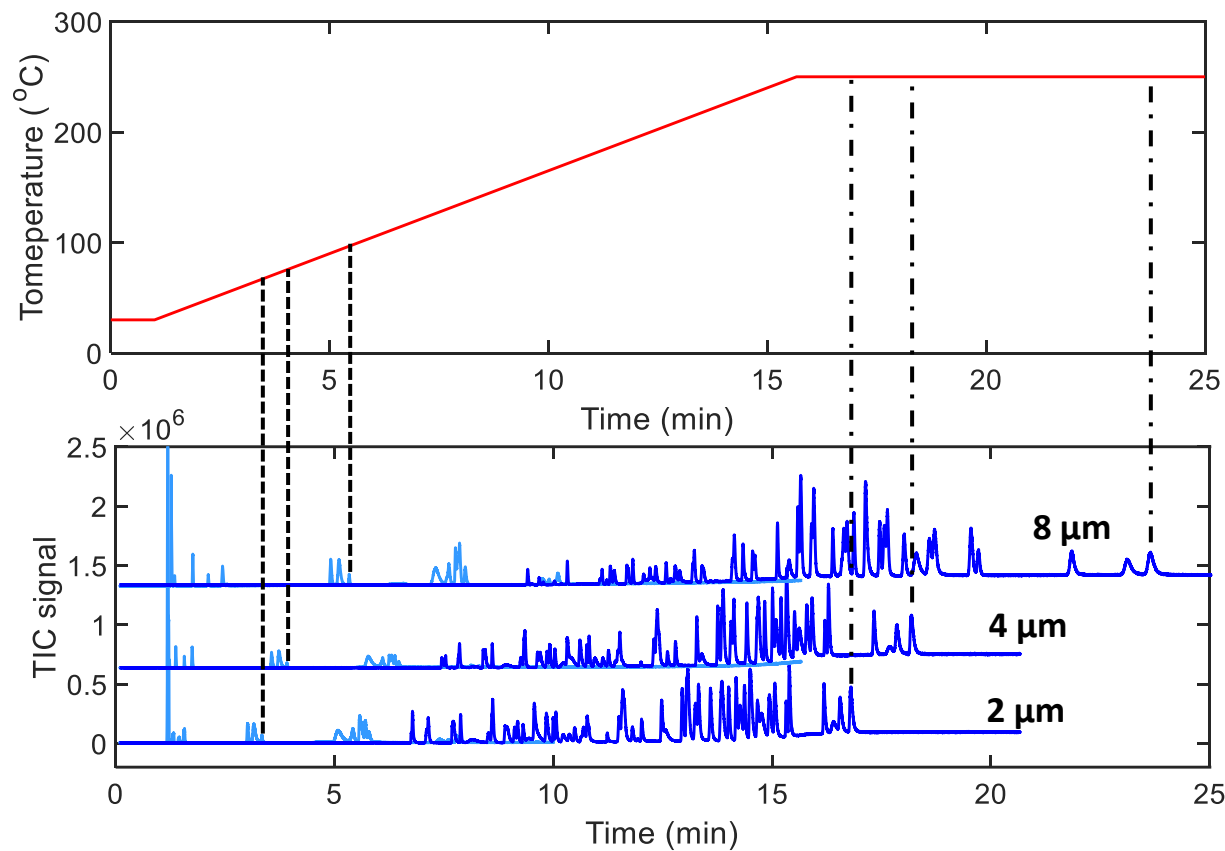
Calibration mix: 40 – 334

Refinery mix: 2 – 100

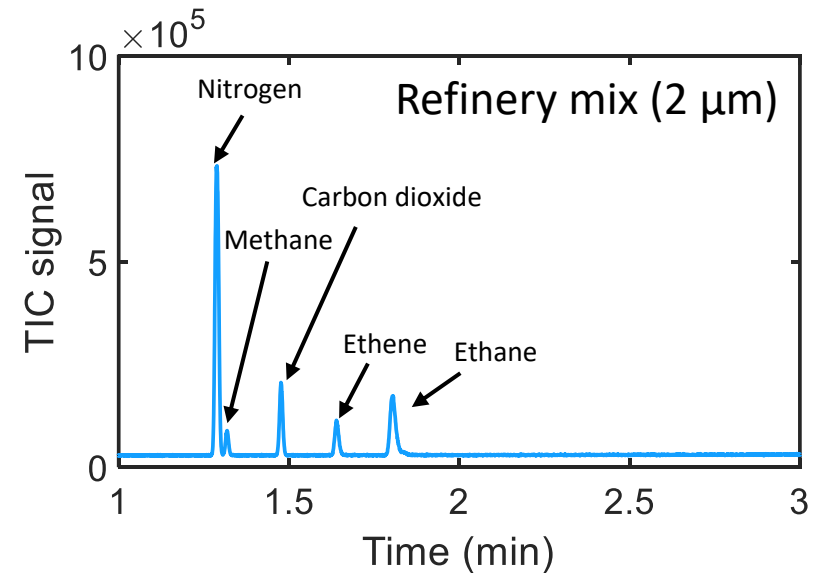
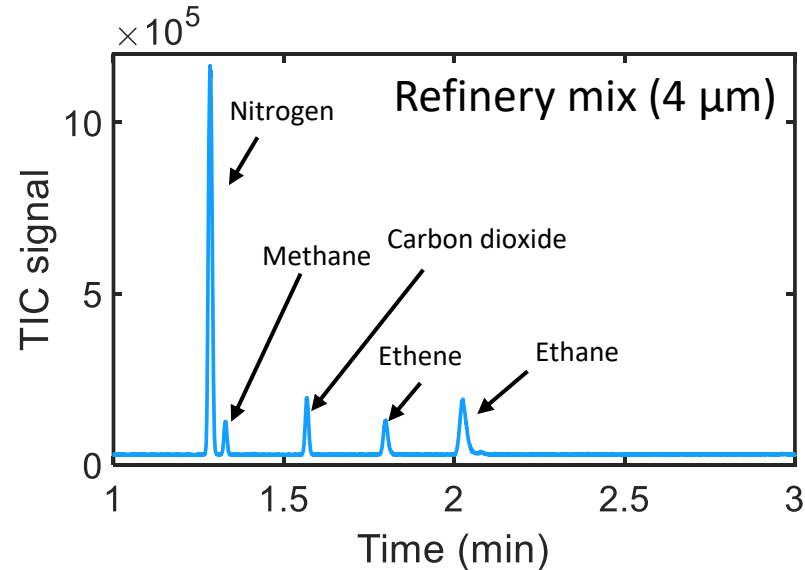
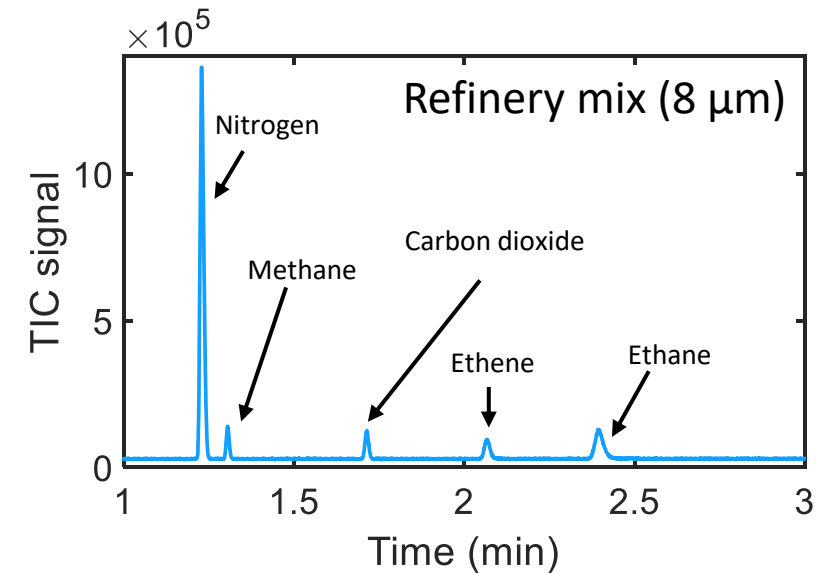
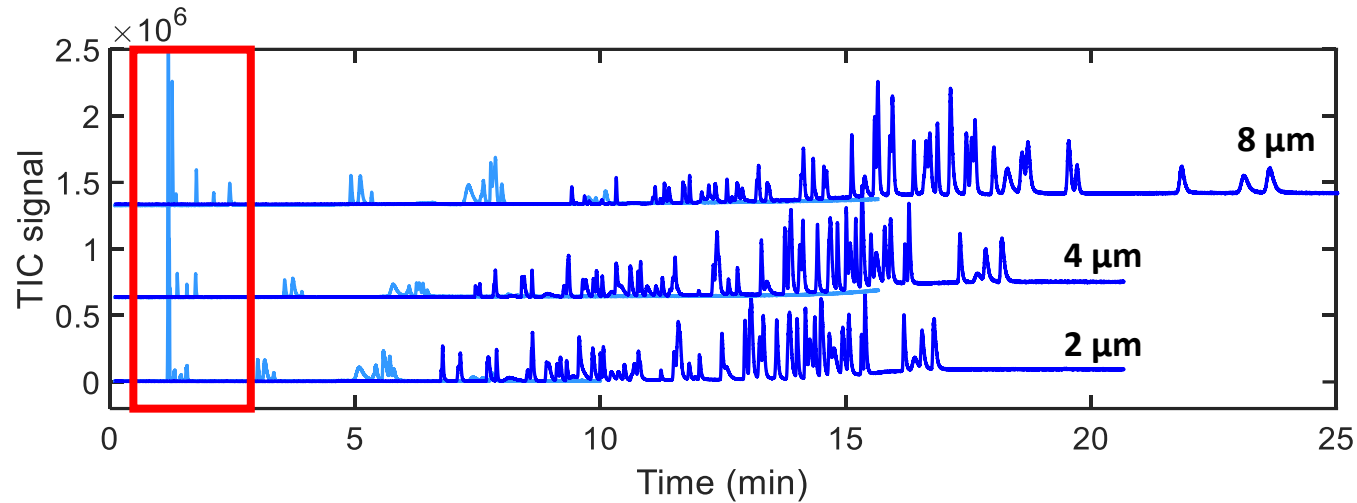
Temperature program:

30 °C to 250 °C @ 15 °C/min

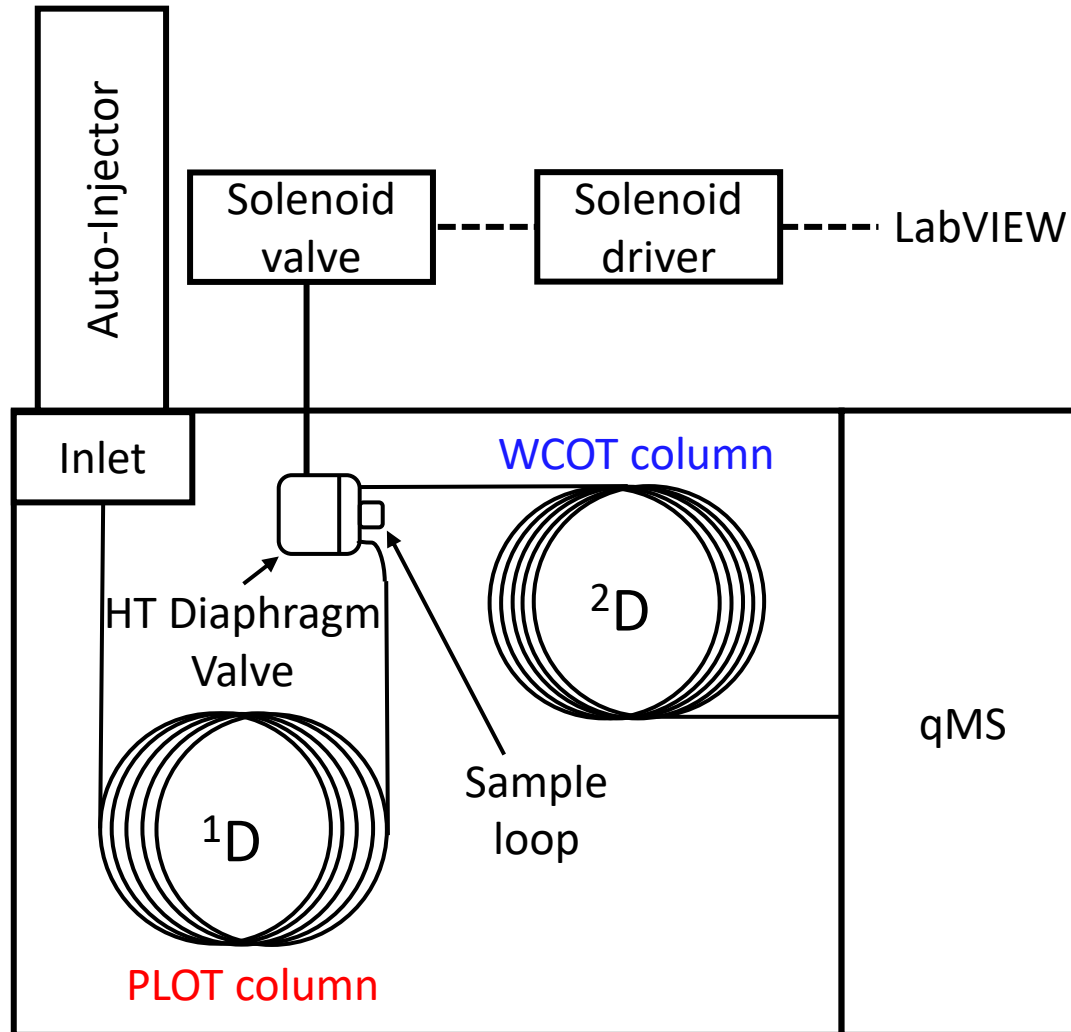
Results of reducing PLOT column film thickness



Results of reducing PLOT column film thickness



Next generation PLOT-based GC×GC using qMS for added selectivity: ultralight to semi-volatile compounds



Sample loop: 10 μL

1D column: **PLOT – S** (30 m x 250 μm x 4 μm) or
PLOT – S (30 m x 250 μm x 2 μm)

Flow 1D: 1.4 ml/min

2D column: **Rtx – 200** (5 m x 150 μm x 2 μm)

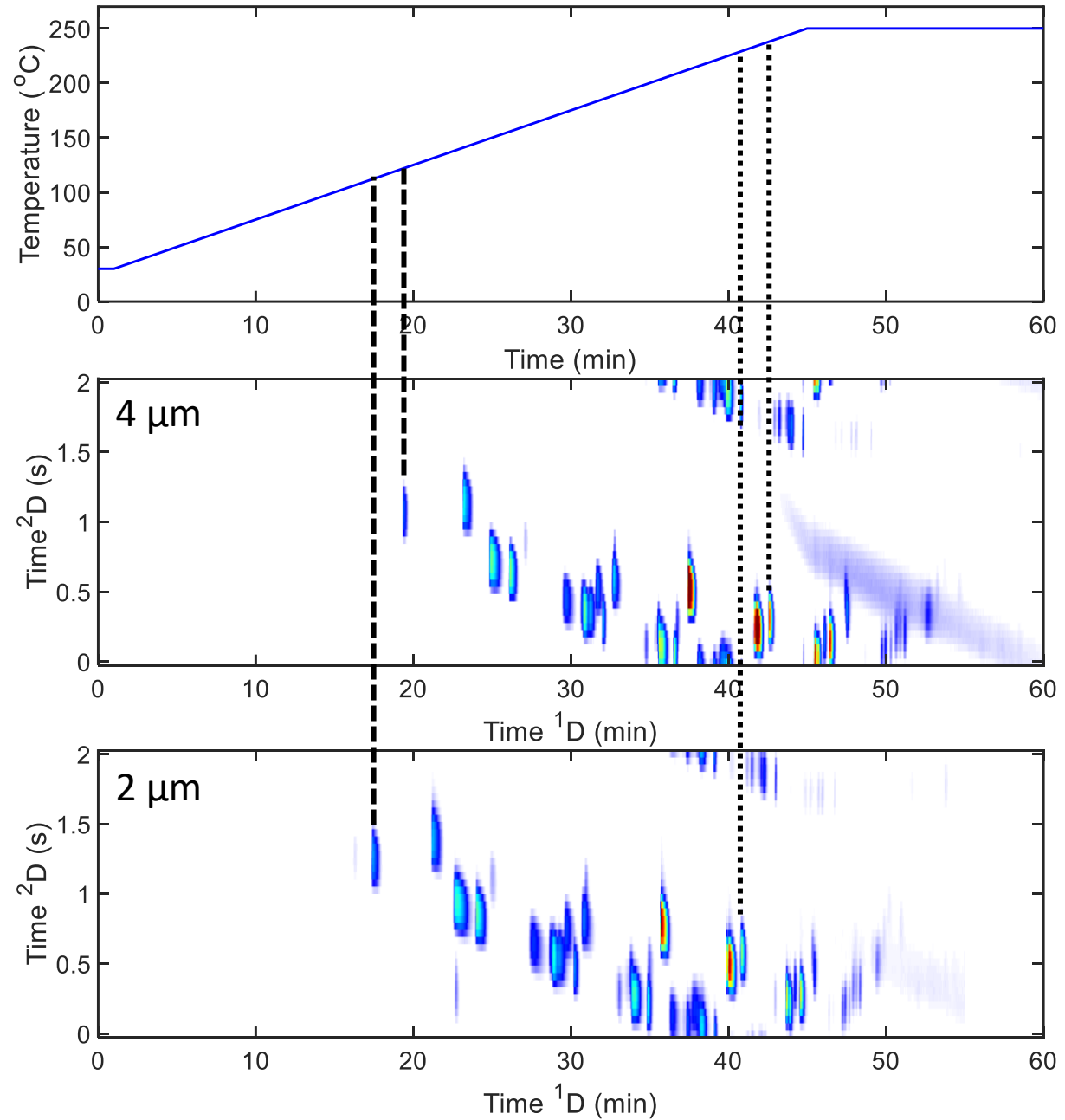
Flow 2D: 2.0 ml/min

Temperature program:

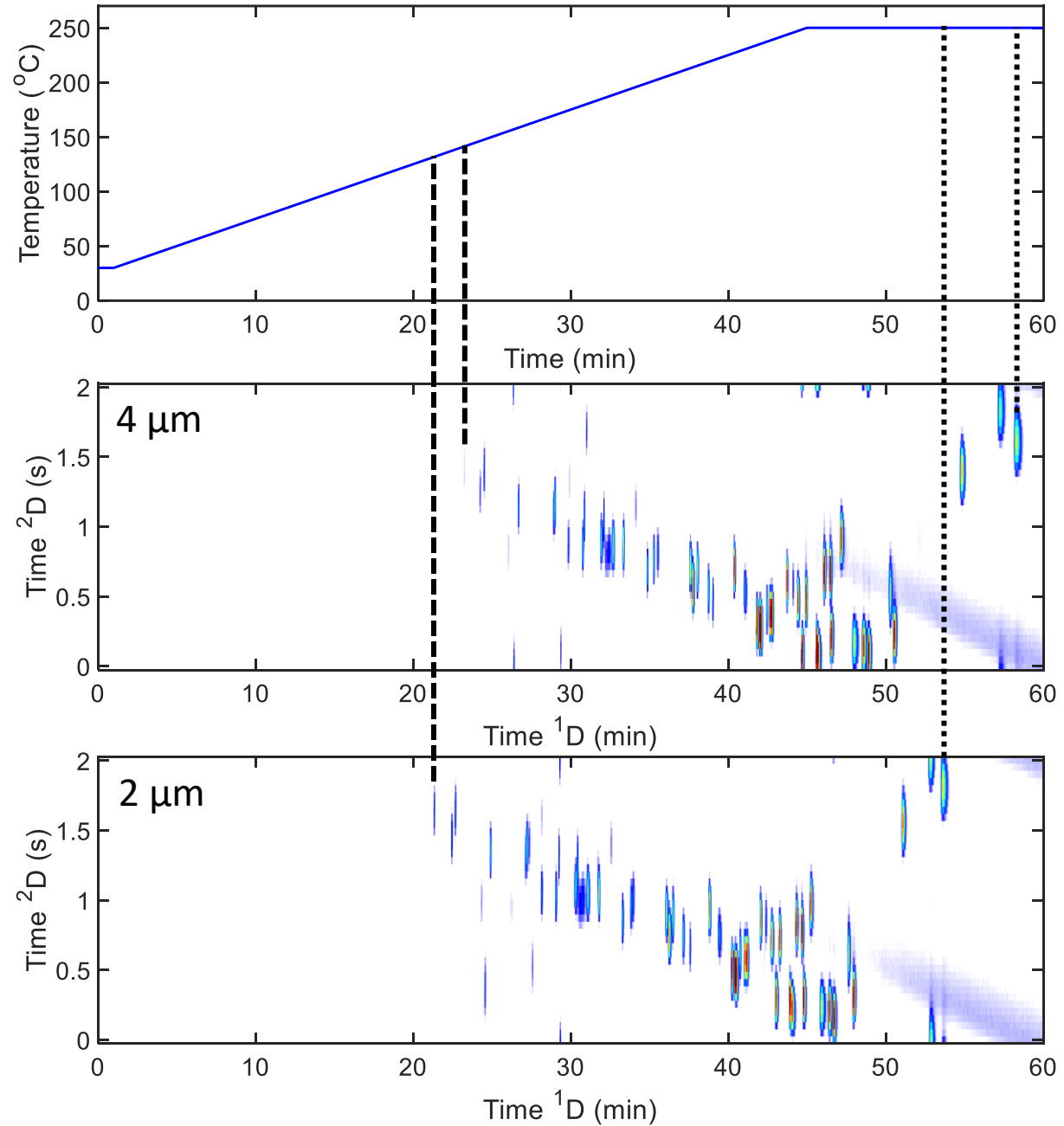
30 $^{\circ}\text{C}$ to 250 $^{\circ}\text{C}$ @ 5 $^{\circ}\text{C}/\text{min}$

$P_M = 2$ s and $p_w = 500$ ms

PLOT-based
GC×GC – qMS
using thinner
columns:
Gasoline

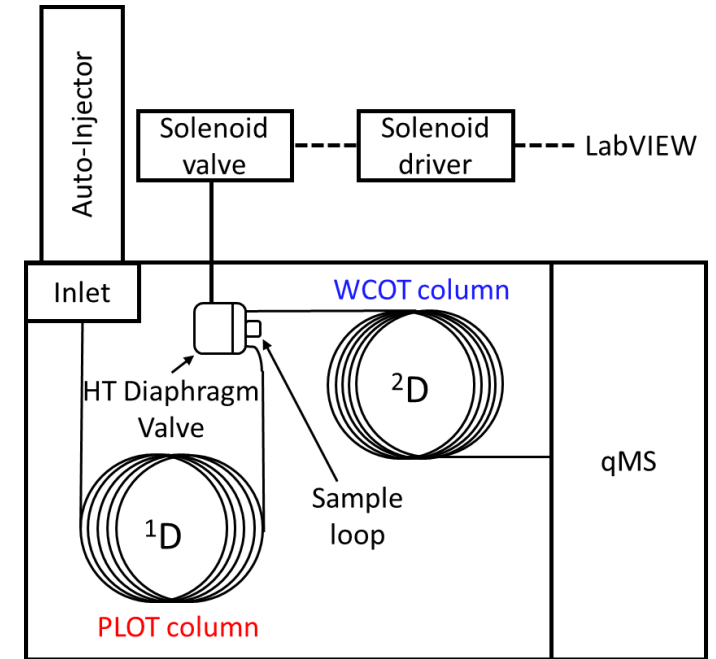


PLOT-based
GC×GC – qMS
using thinner
columns:
Calibration Mix



Conclusions and future work

- Running GC × GC-qMS with thinner film PLOT columns at 5 m length instead of 30 m
- Trying some samples that would show the full potential of using thinner PLOT columns
- Testing retention time reproducibility with thinner film PLOT columns



Acknowledgements

Principal Investigator:

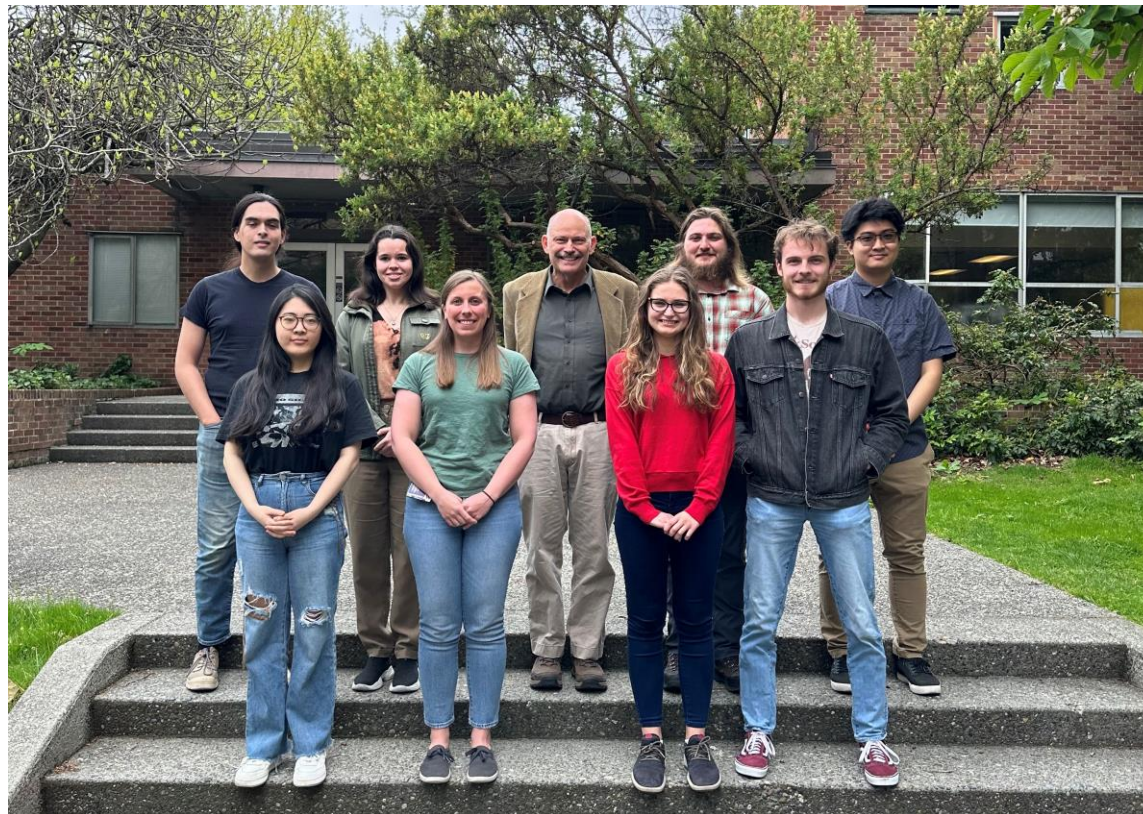
- Dr. Robert Synovec

Synovec Lab Members:

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- Cassandra Padilla
- Owen Lee
- Arty Manafe
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- Jakob Klein

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Any questions?