

The Future of Environmental Analysis by GC/MS: Combining New Deactivation Chemistries, Microfluidics, and Precision Pneumatics

Agilent Intuvo 9000 GC

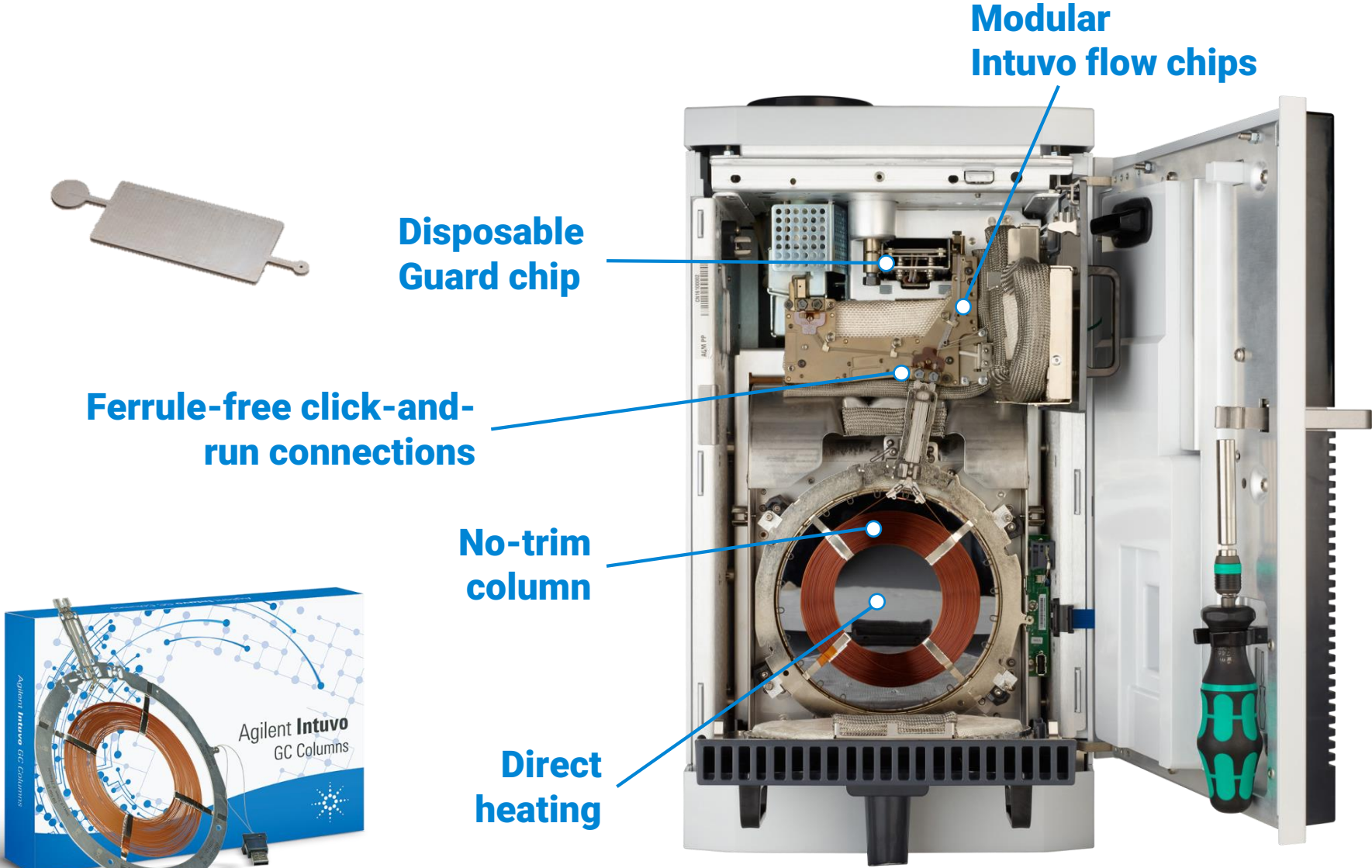
Matthew Giardina, Ph.D.

October 24, 2017

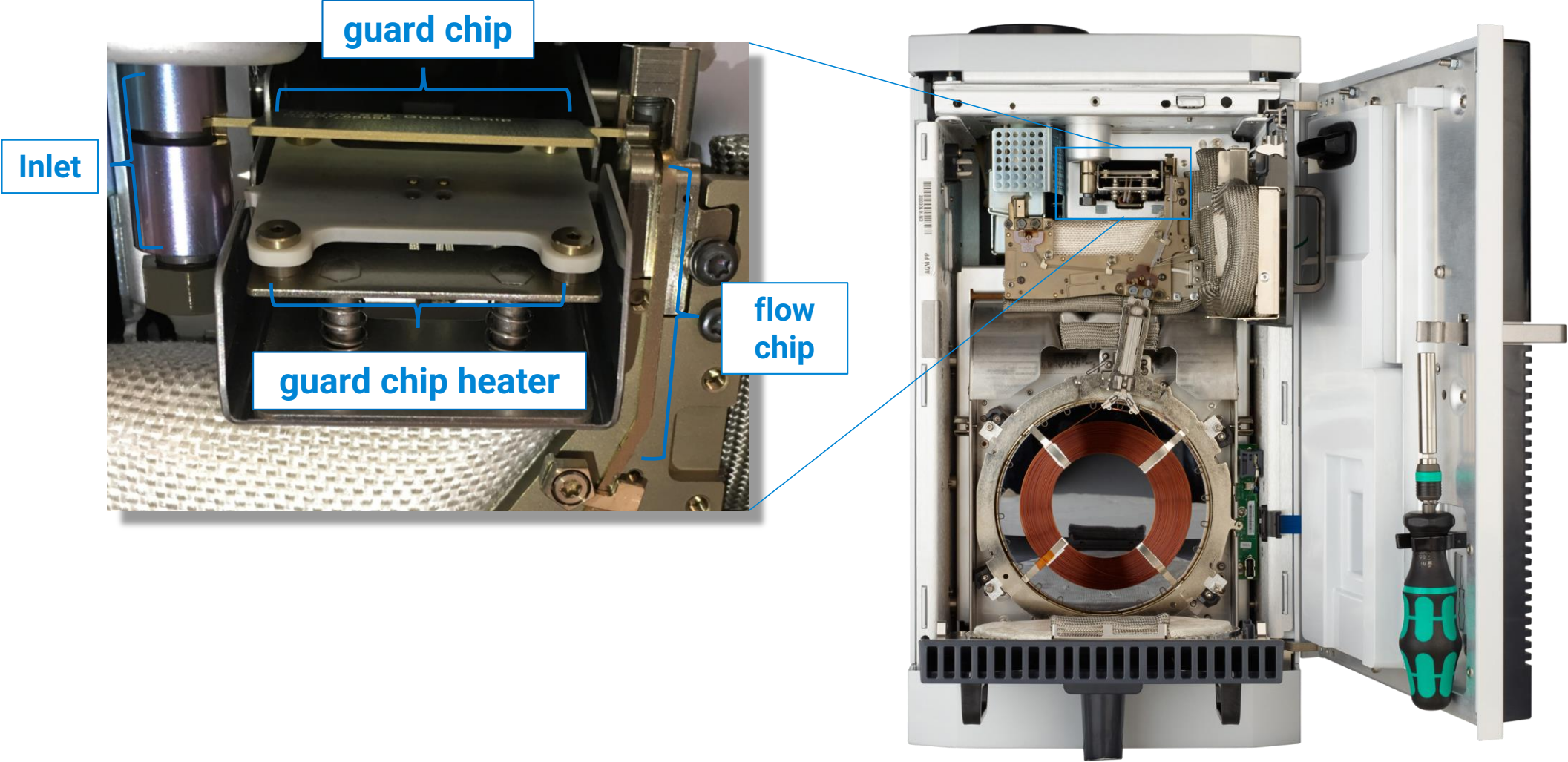
Outline

- **Brief review of the Intuvo flowpath**
- **Guard chip modes of operation**
- **Applying guard chip modes of operation to environmental analysis**

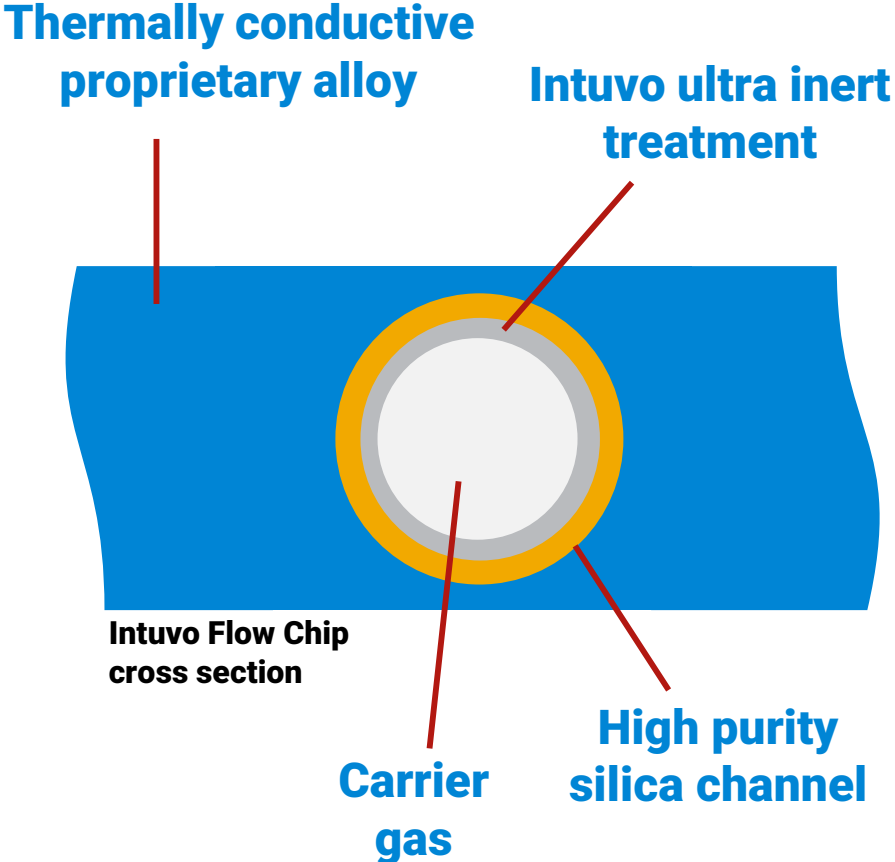
Intuvo 9000 GC Flow Path



Intuvo 9000 GC Flow Path

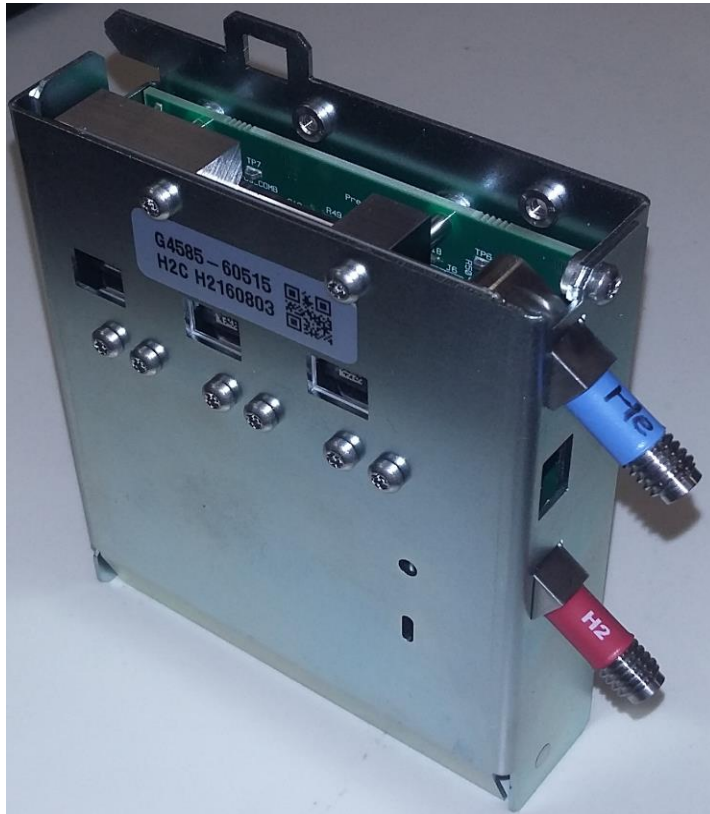


Intuvo 9000 GC Flow Path – Guard Chip

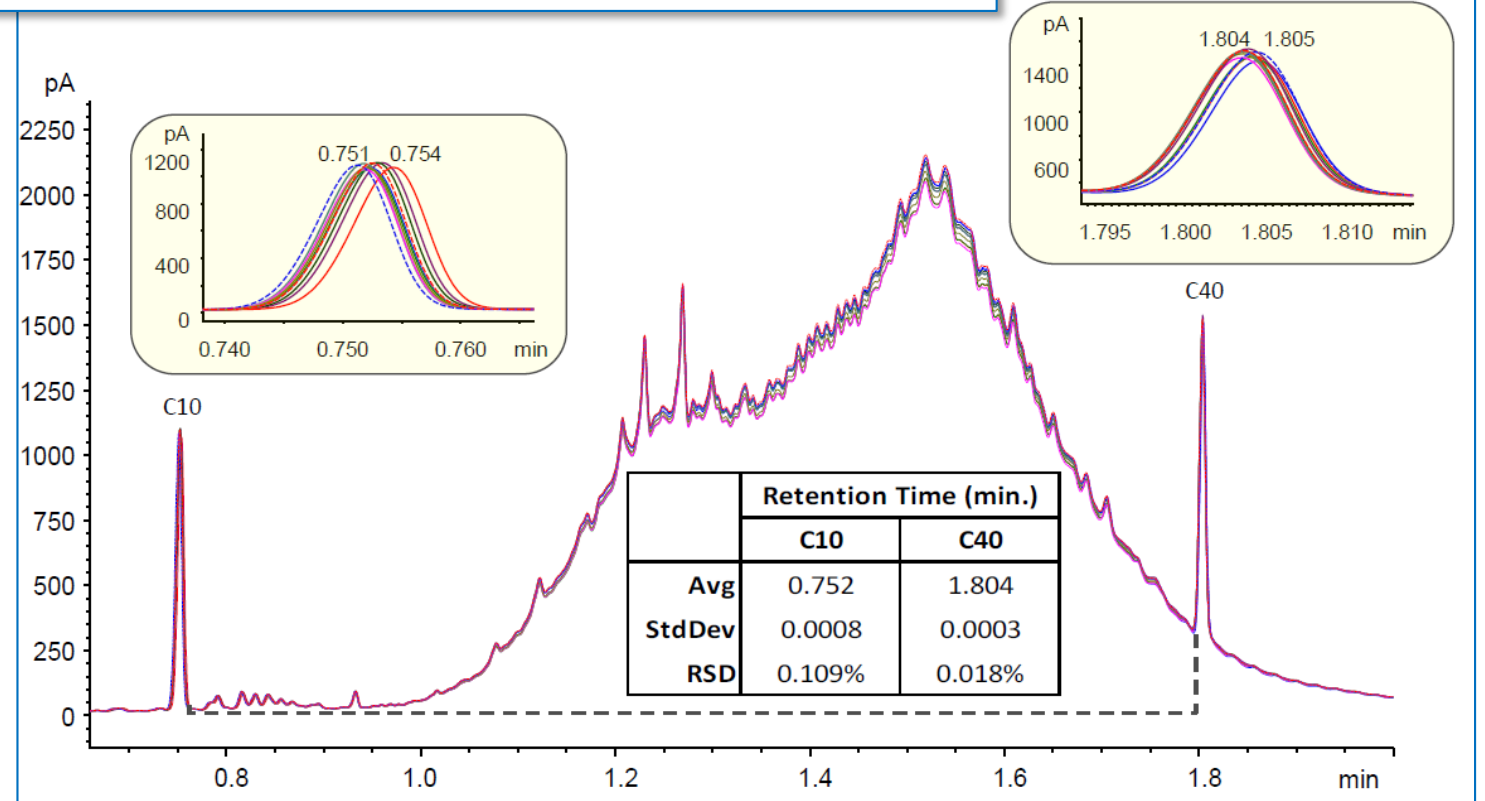


Intuvo 9000 GC Pneumatics

Microfluidic-enabled 6th generation EPC modules.

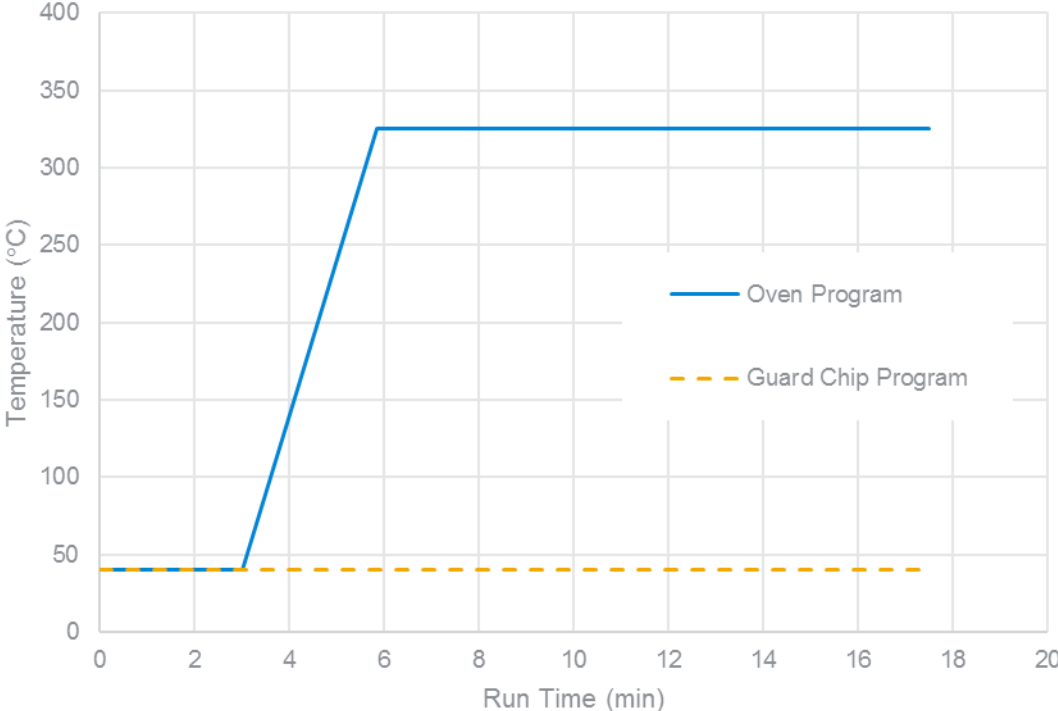


TPH Analysis: 250 °C/min and 10 mL/min

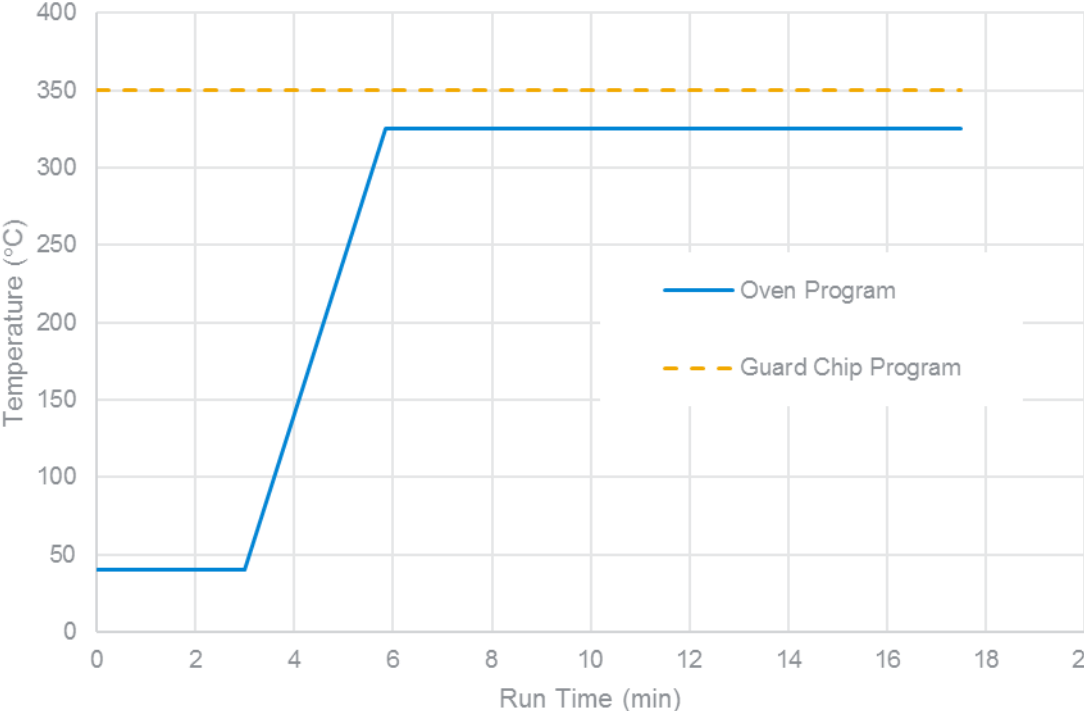


What is the guard chip is trapping?

Guard chip 40 °C isothermal

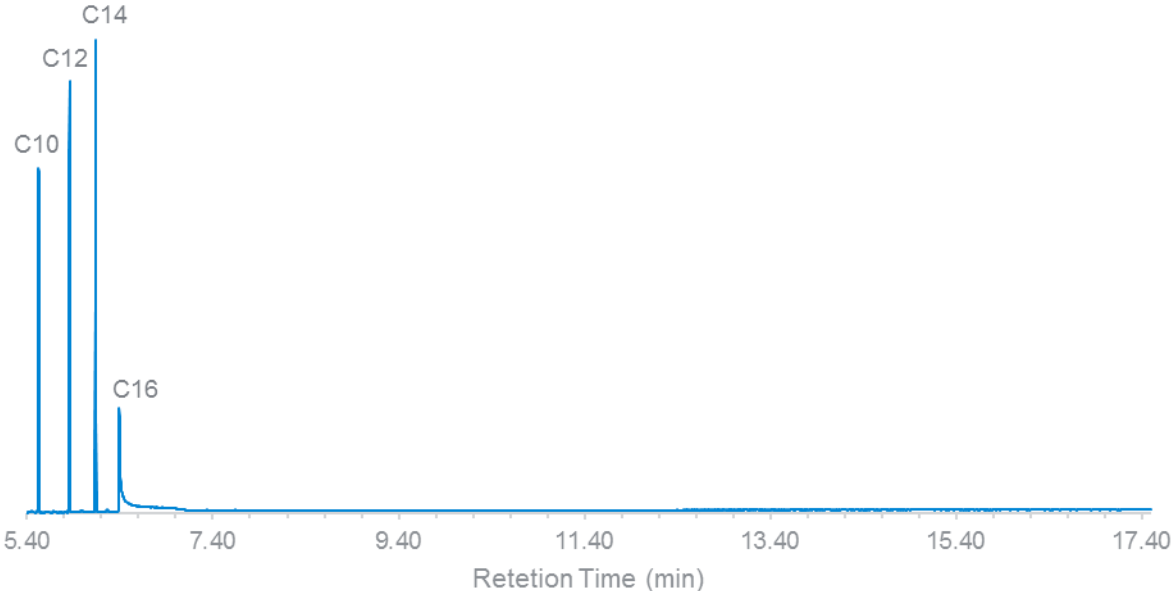


Guard chip 350 °C isothermal

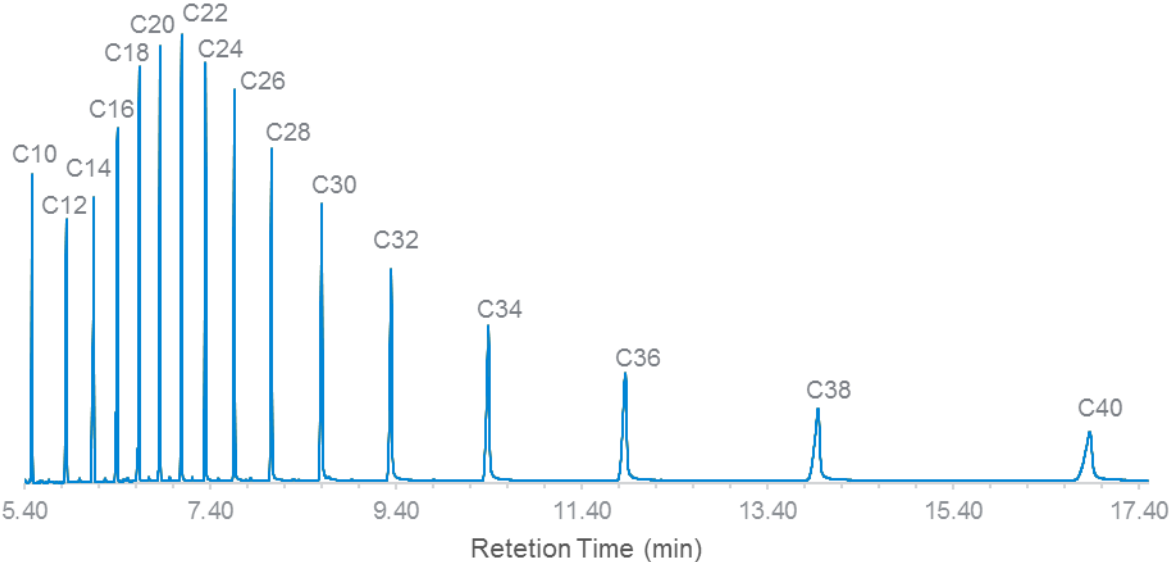


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Guard chip 40 °C isothermal

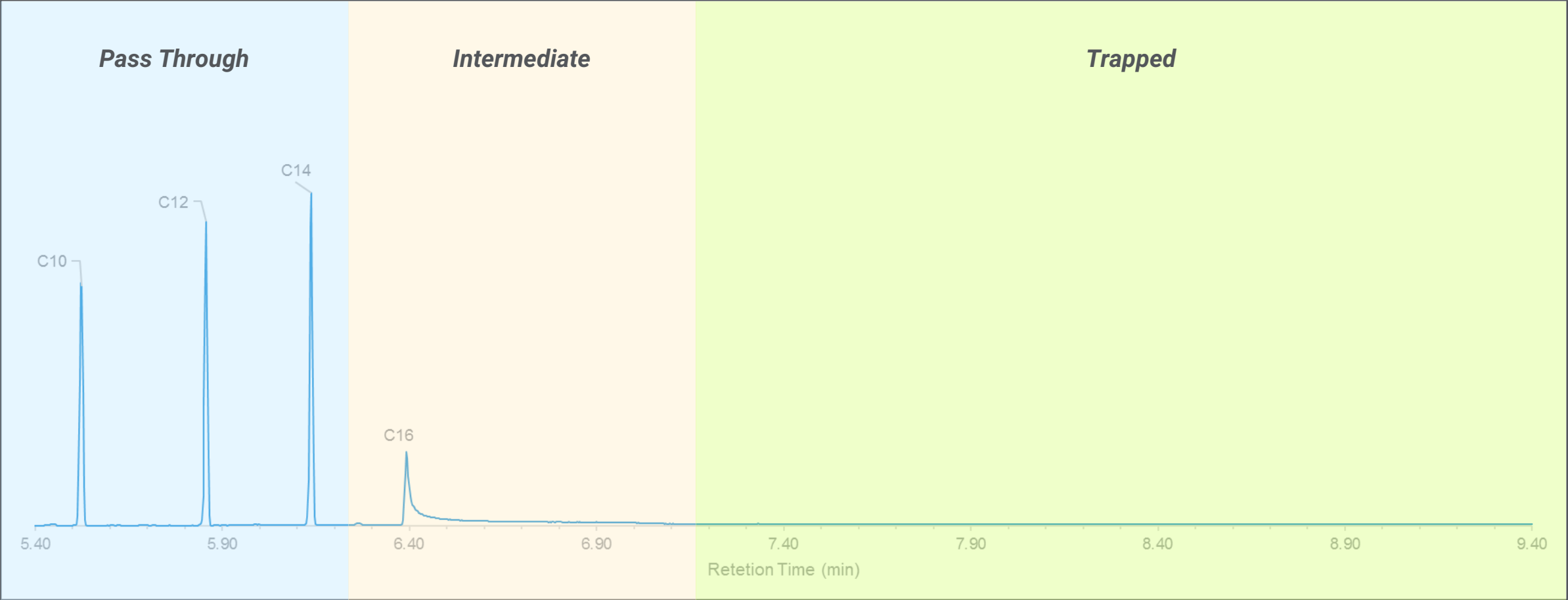


Guard chip 350 °C isothermal



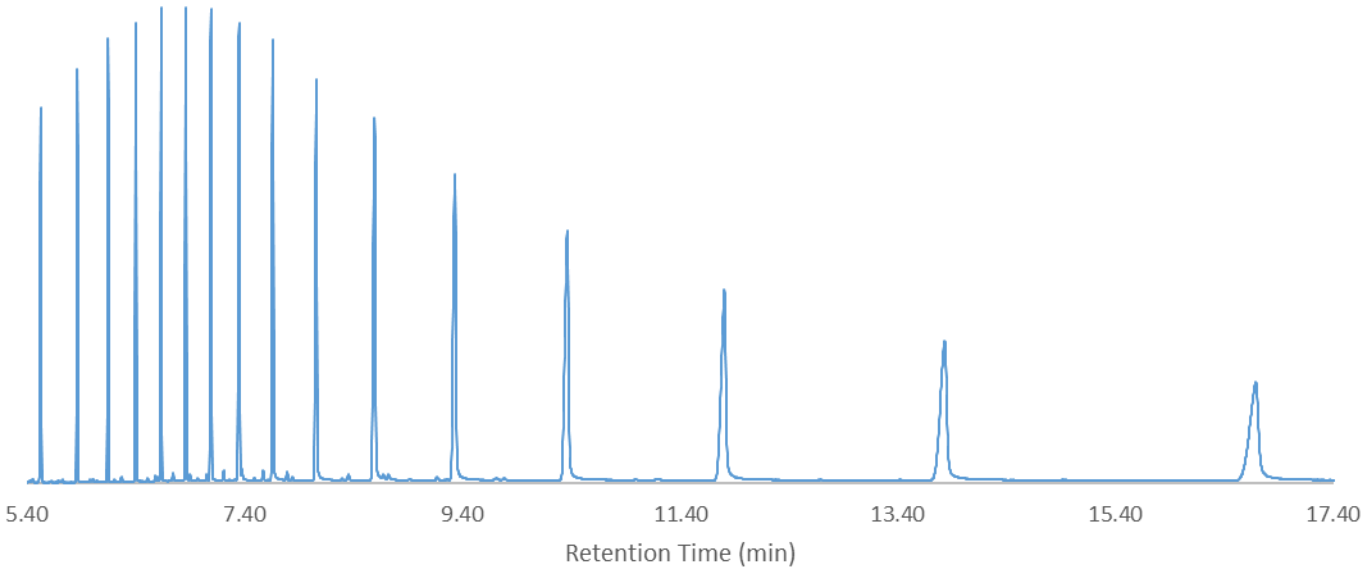
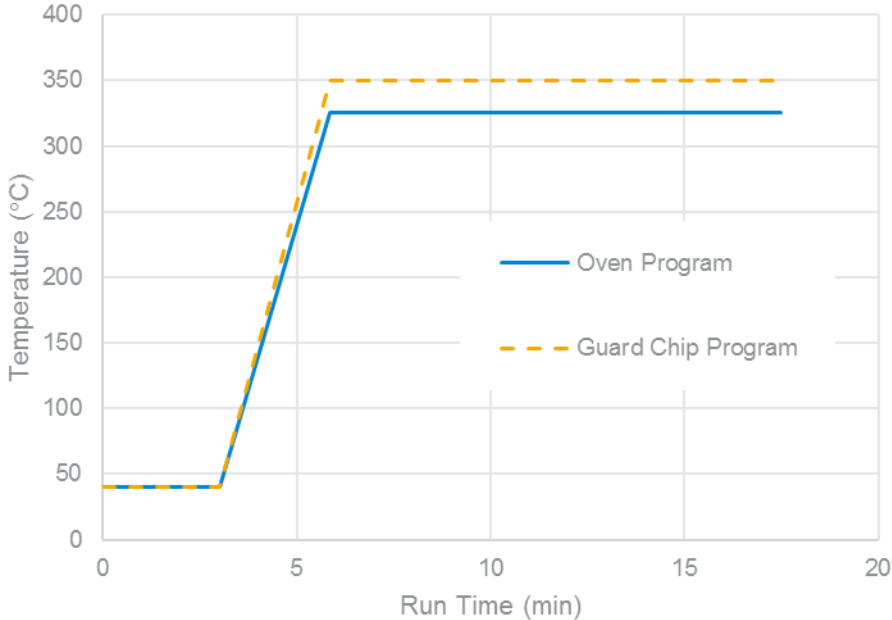
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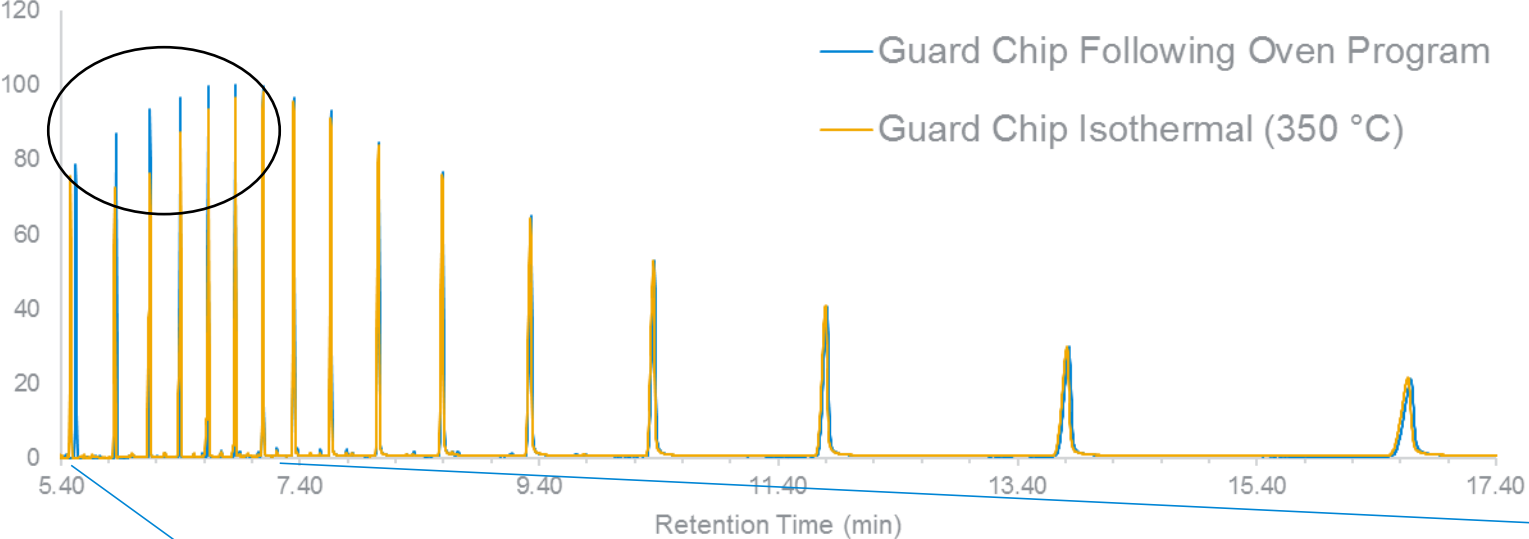


What about ramping the guard chip temperature?

Guard chip following the column temperature program

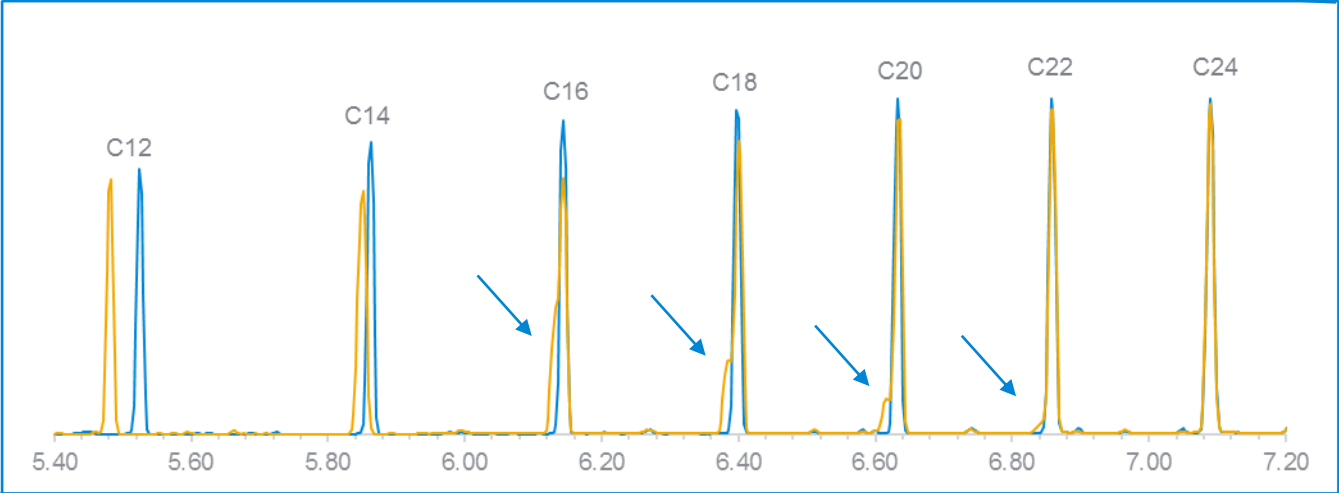


What about ramping the guard chip temperature?



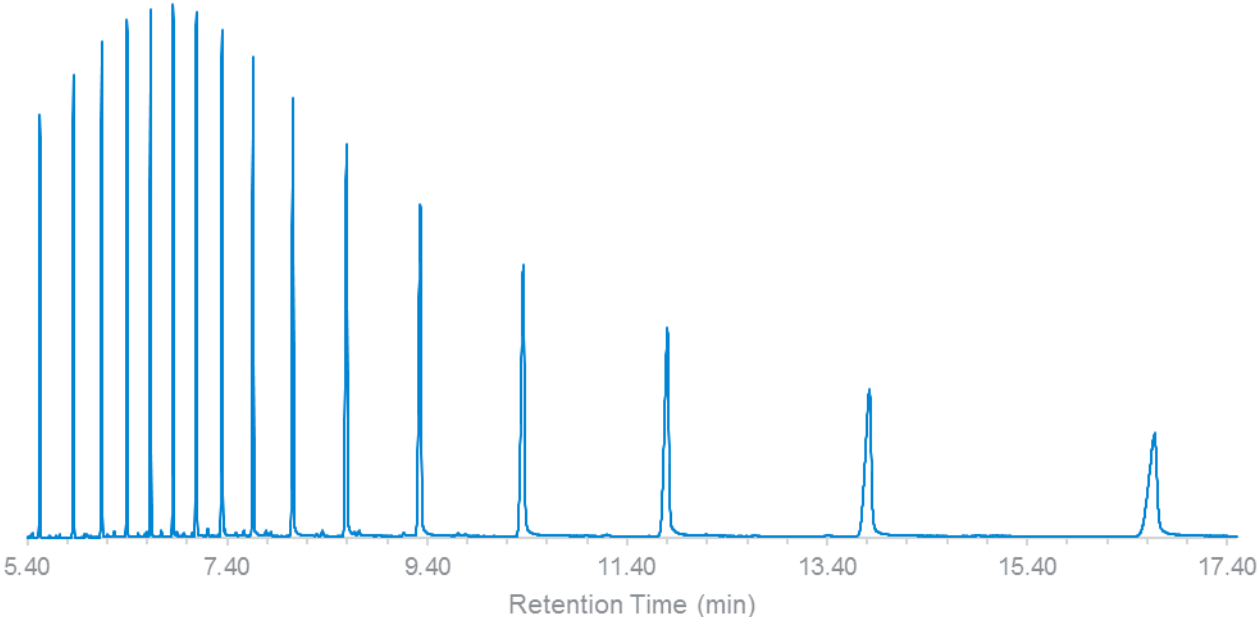
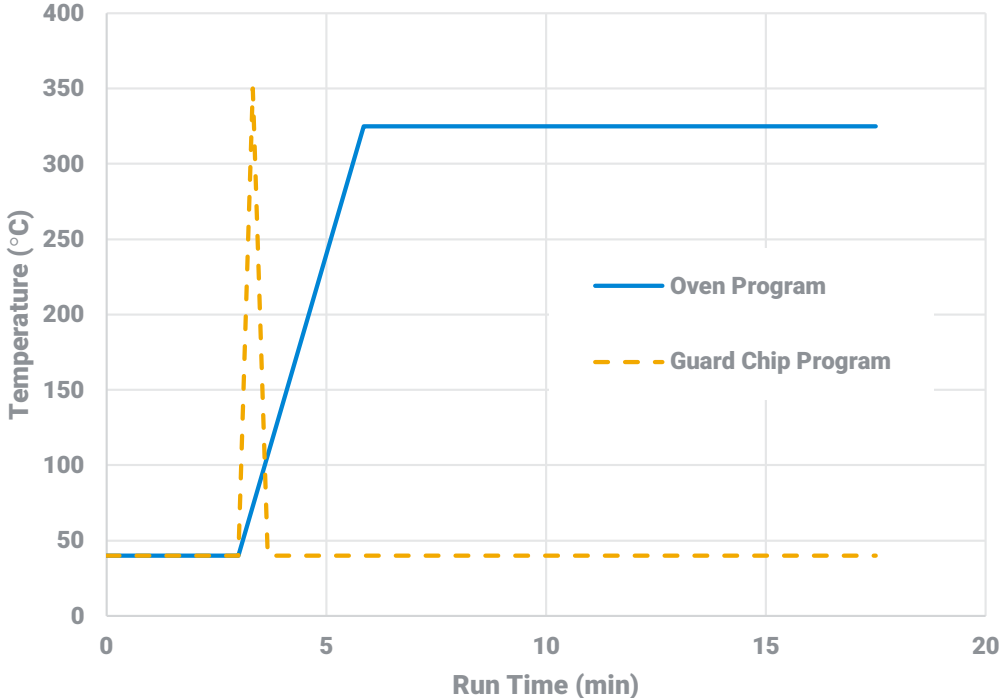
Isothermal guard chip 350 °C

- ***Peak broadening for some compounds***
- ***Better peak shape ramping guard chip***



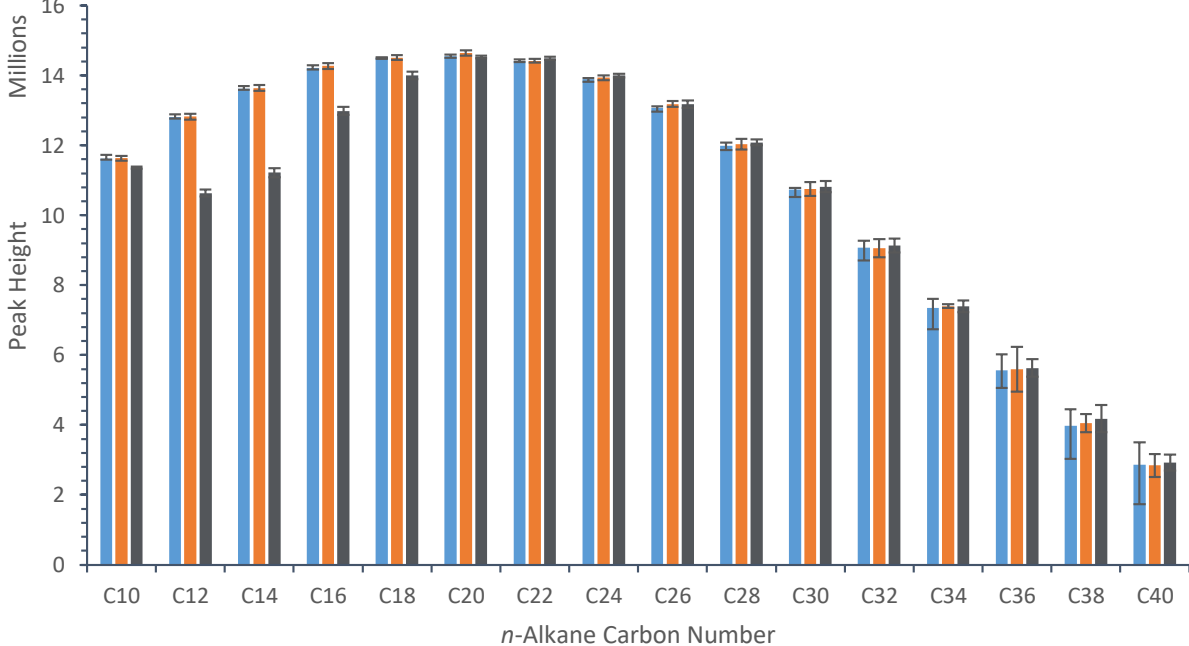
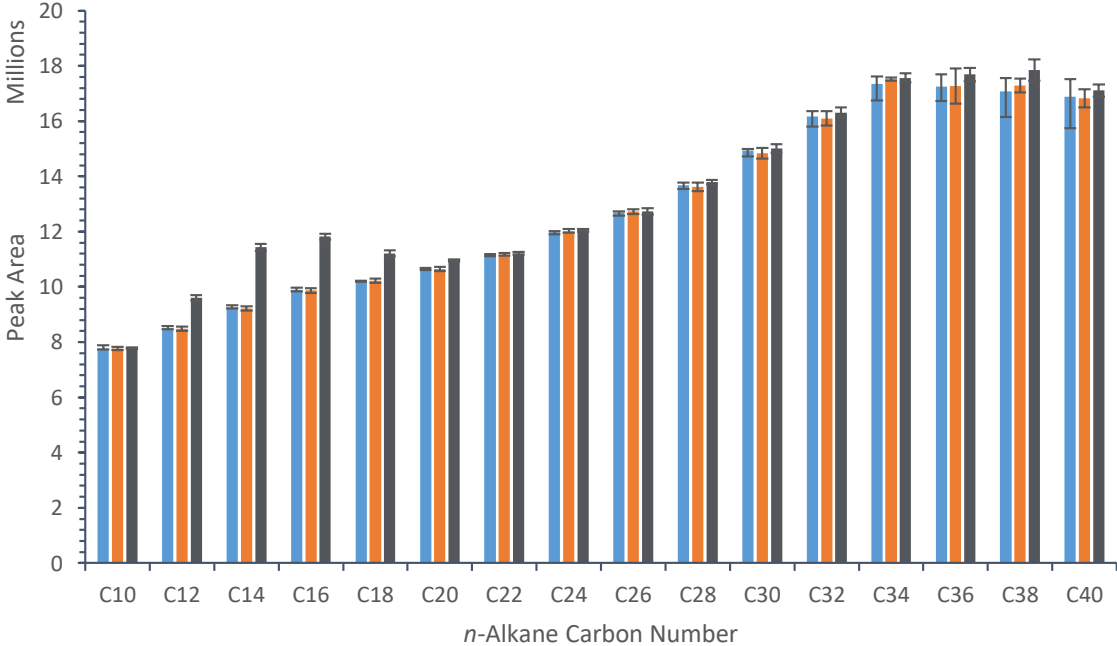
What about pulsing the guard chip temperature?

Rapid guard chip heating and cooling

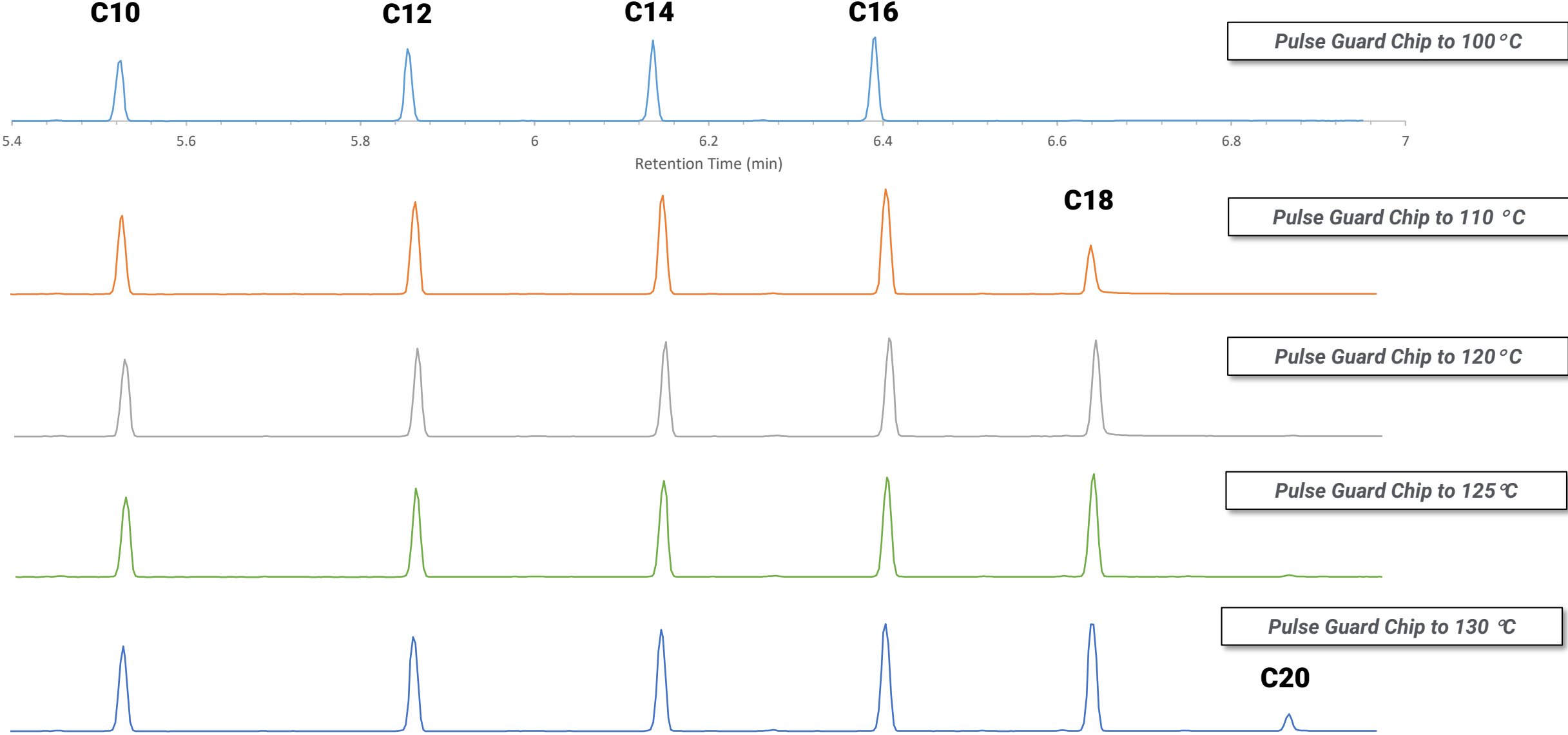


How do the peak area and height compare?

- Guard Chip Fast Pulse (300 °C/min)
- Guard Chip Follow Column Ramp (100 °C/min)
- Guard Chip Isothermal (350 °C)

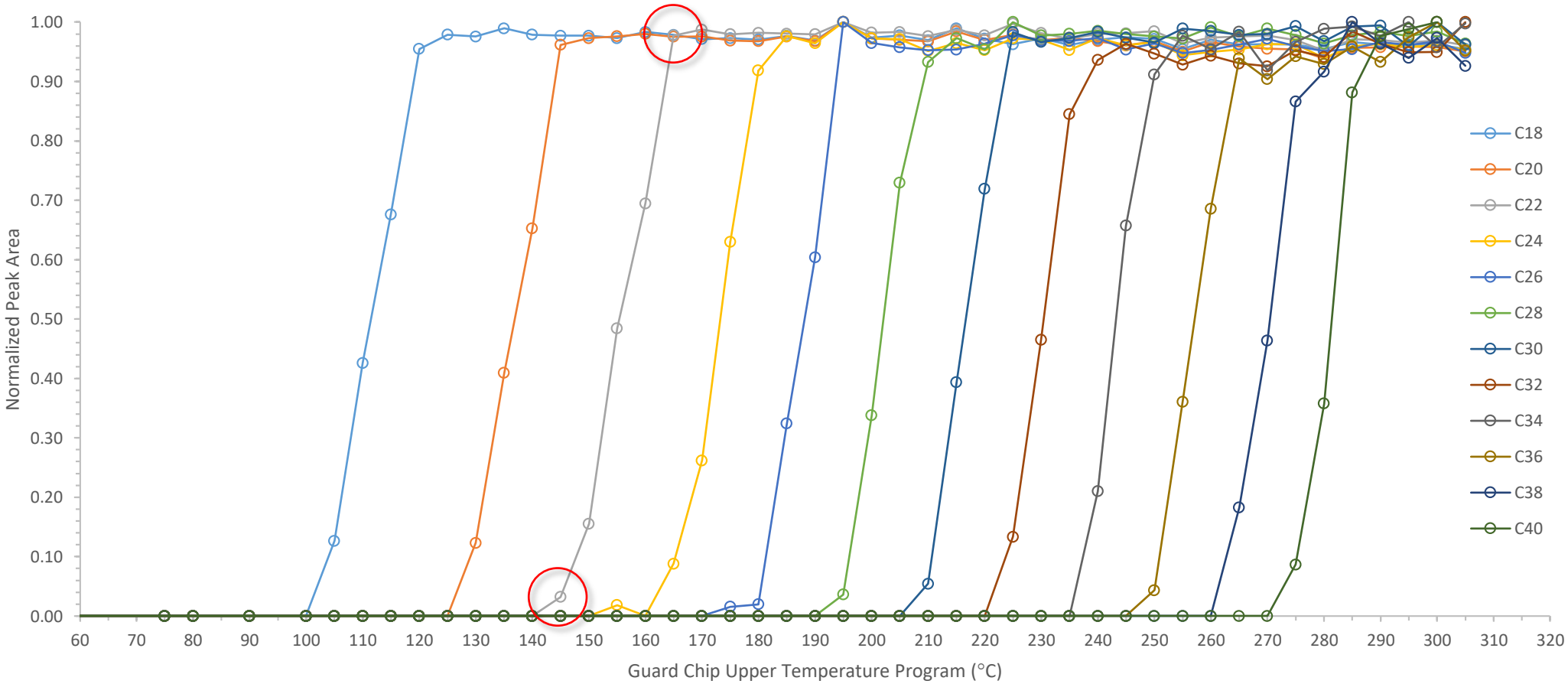


How selective is guard chip pulsing?



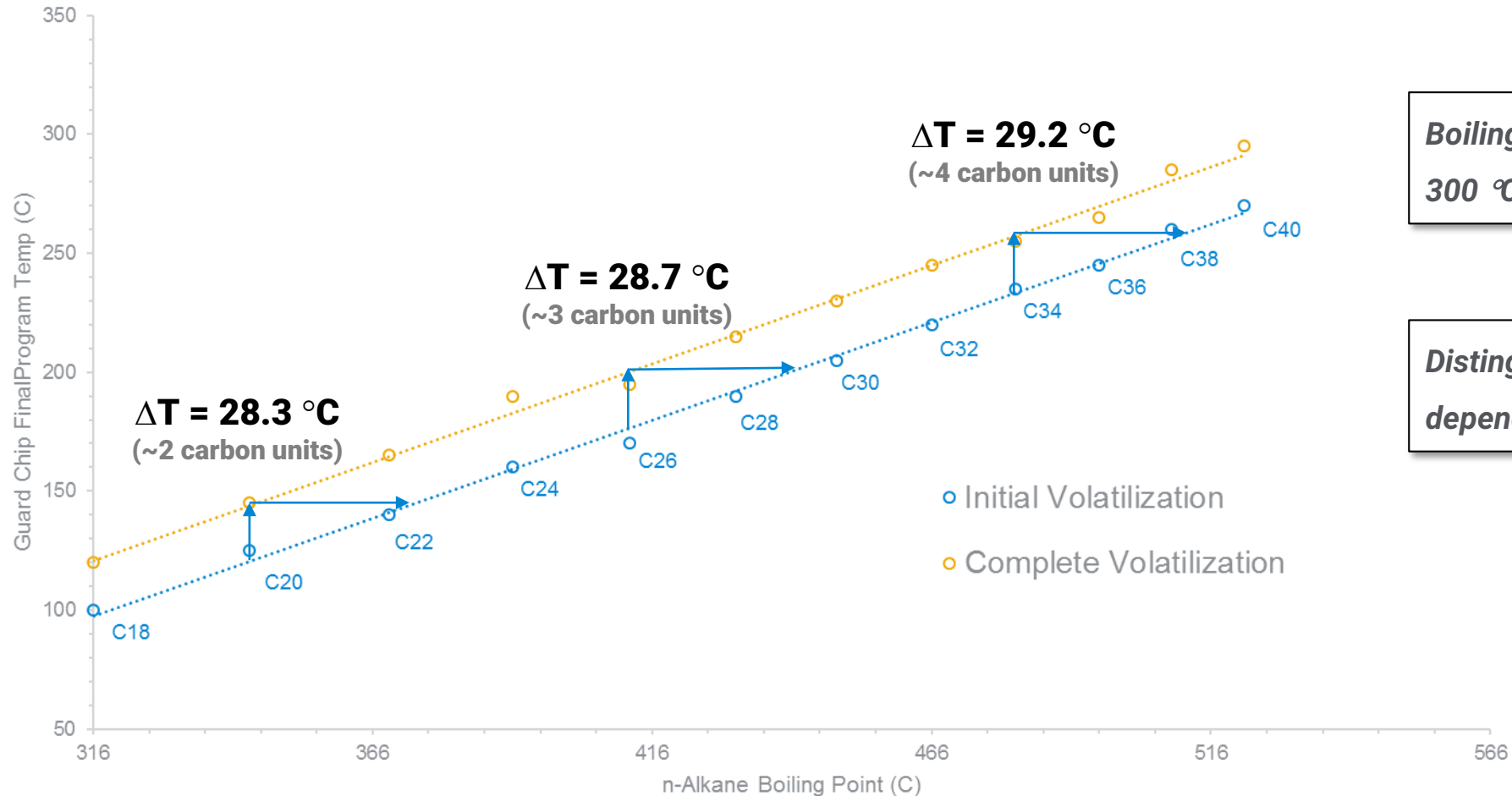
How selective is guard chip pulsing?

Pulsed guard chip to endpoints from 100 to 305 °C



How selective is guard chip pulsing?

Pulsed guard chip to endpoints from 100 to 305 °C



Boiling point resolution about 30 °C at 300 °C/min

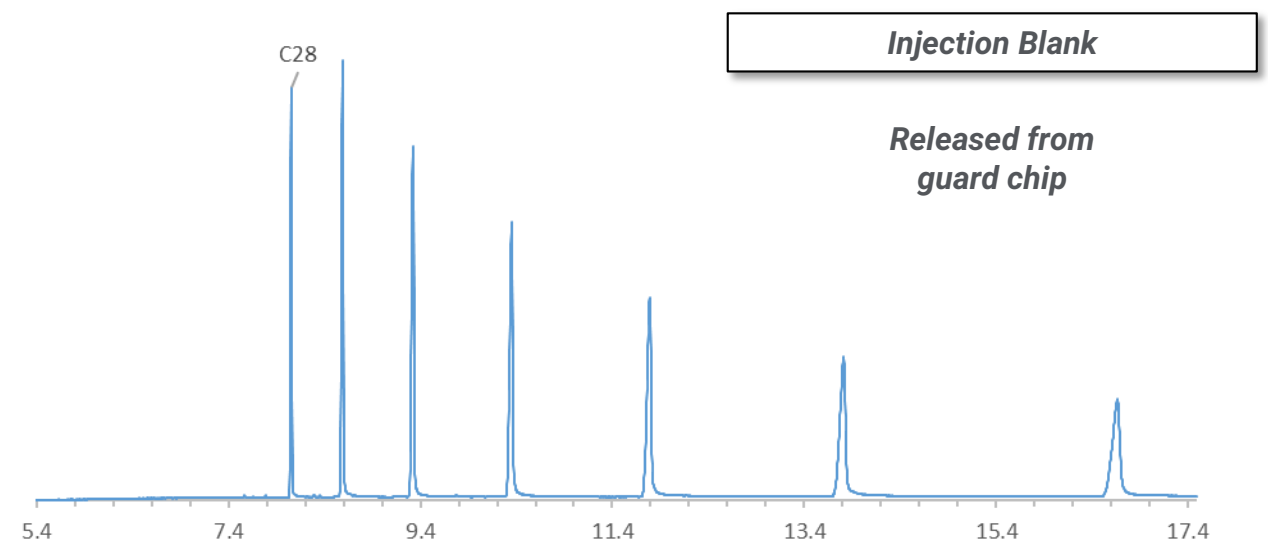
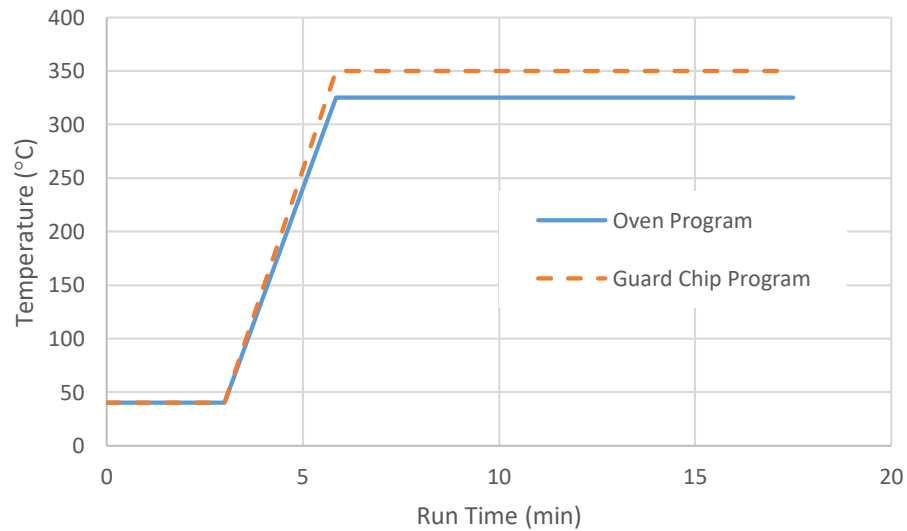
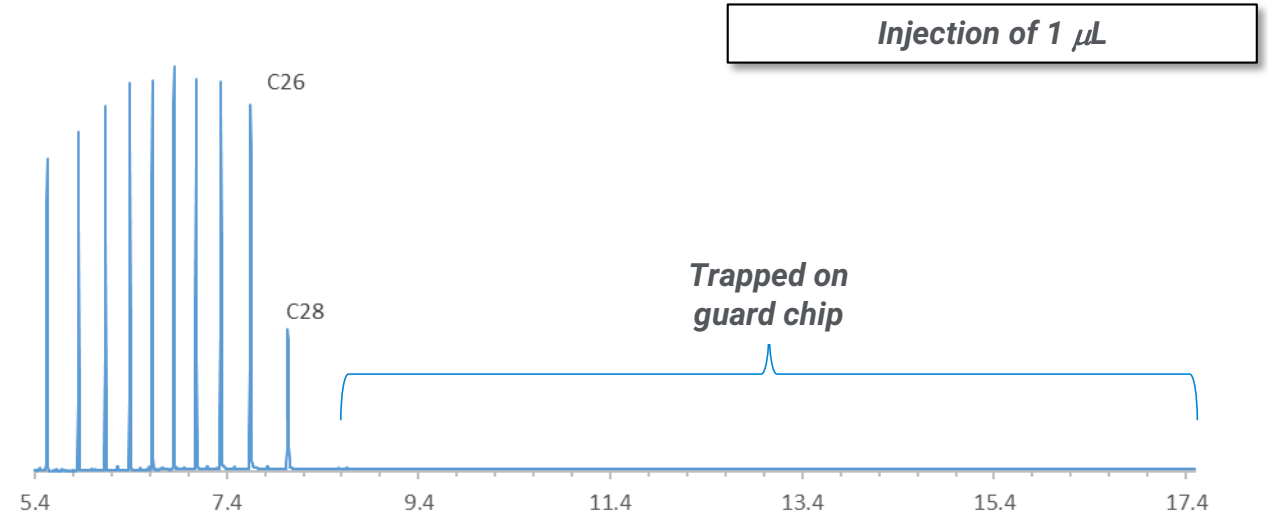
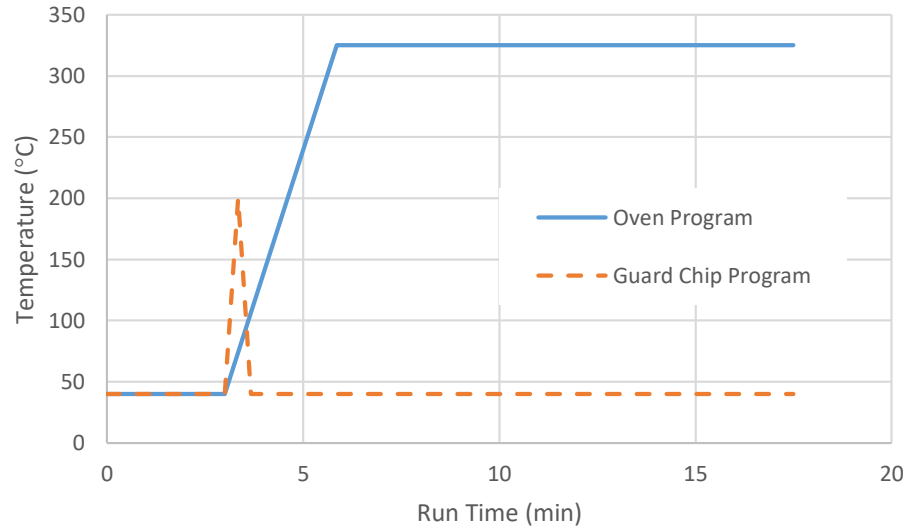
Distinguish between 2 to 4 carbon units depending on boiling point

Guard chip pulsing selectivity

What is gained with boiling point selectivity of the guard chip?

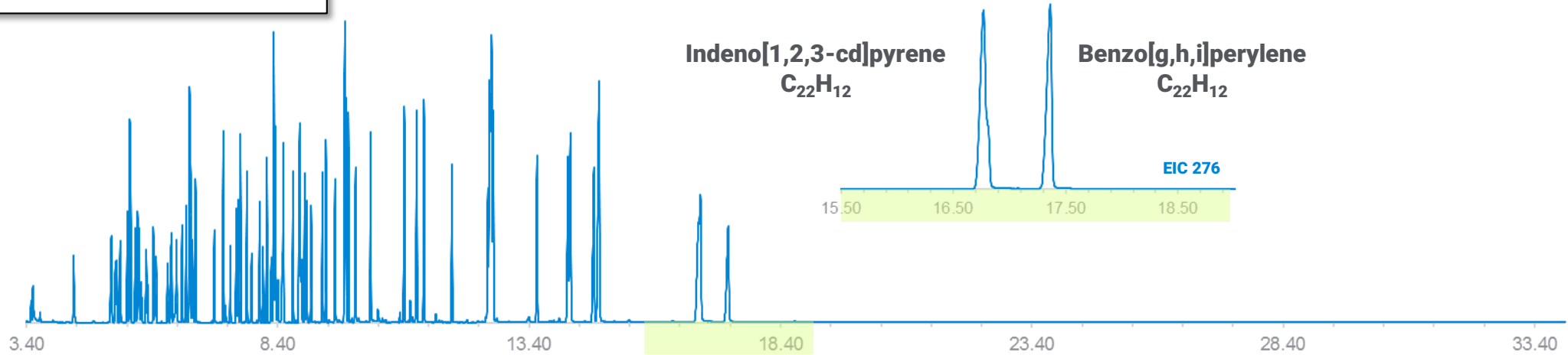
- I. **Can be used trap heavy matrix on the guard chip while passing analytes to the column.**
- II. **In combination with post-column backflush can provide more efficient use of time and prevent high boilers from reaching the column.**
- III. **Selective enough for pseudo heart-cutting.**

I. Can be used to trap heavy matrix

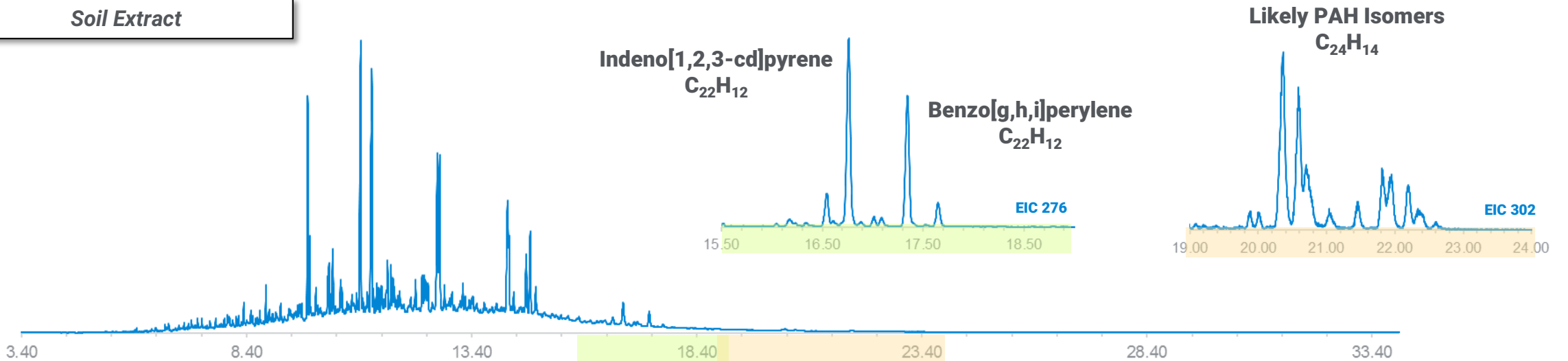


I. Can be used to trap heavy matrix

Semivolatile Standard

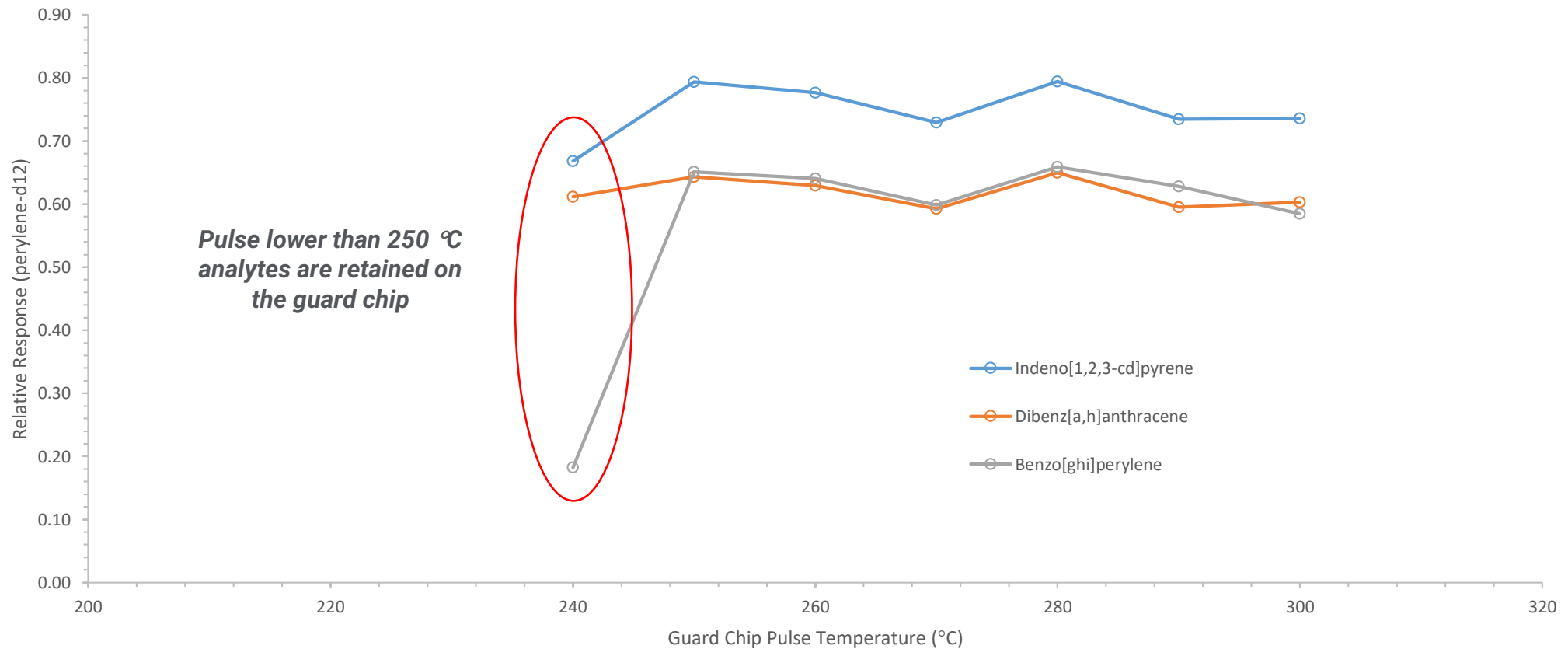


Soil Extract

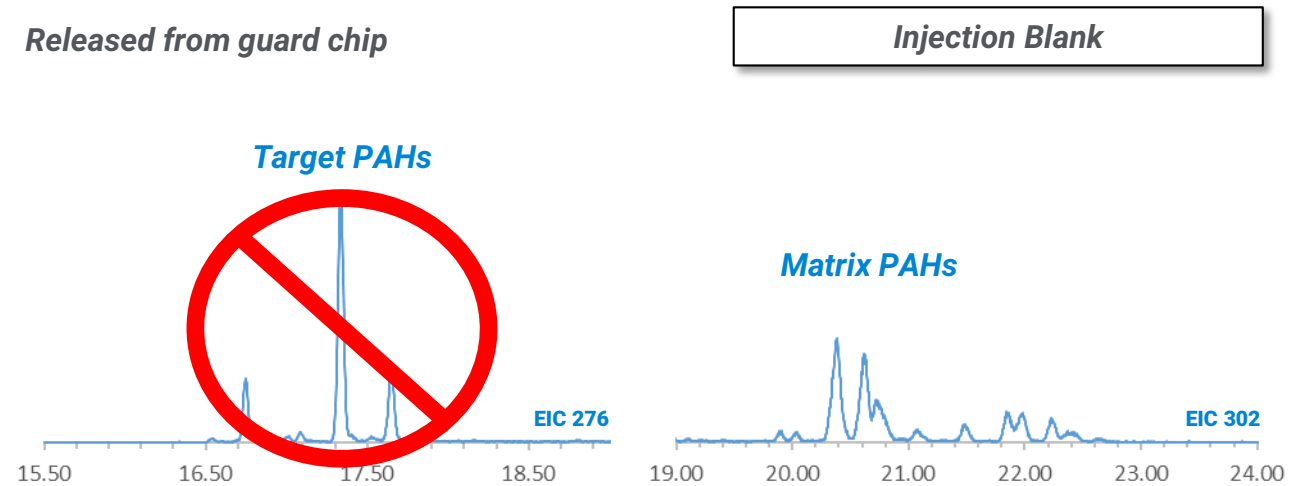
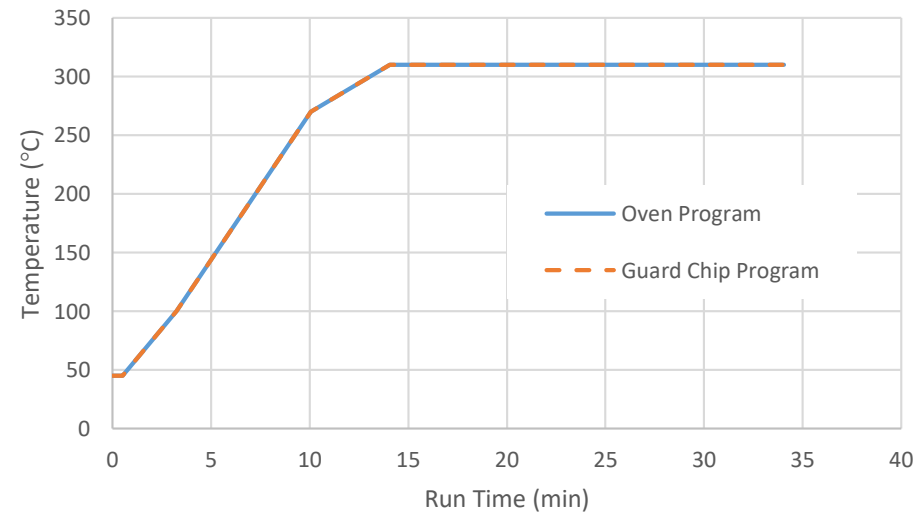
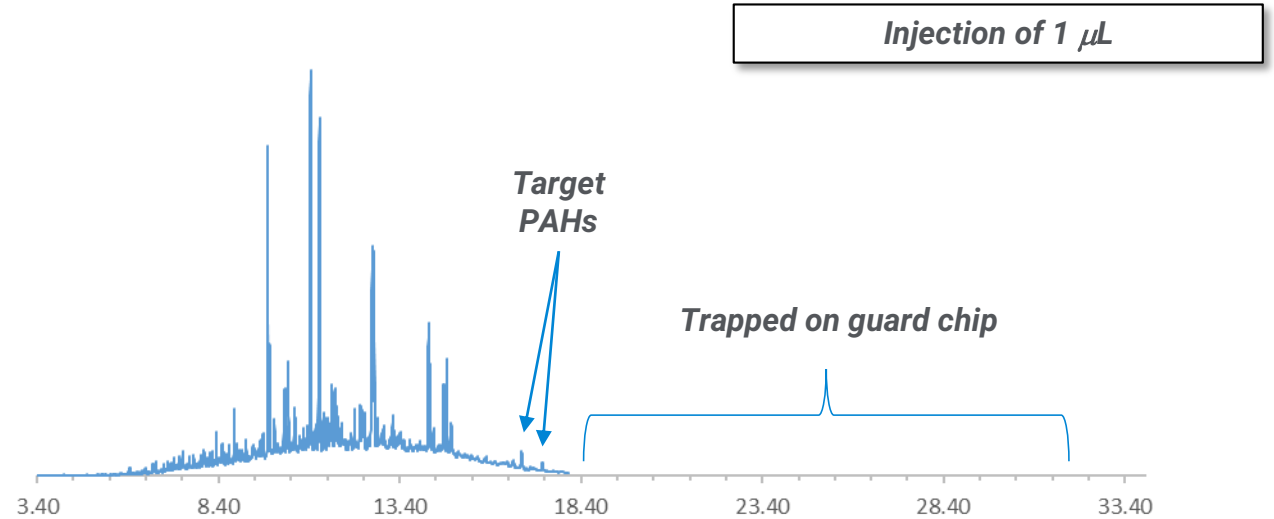
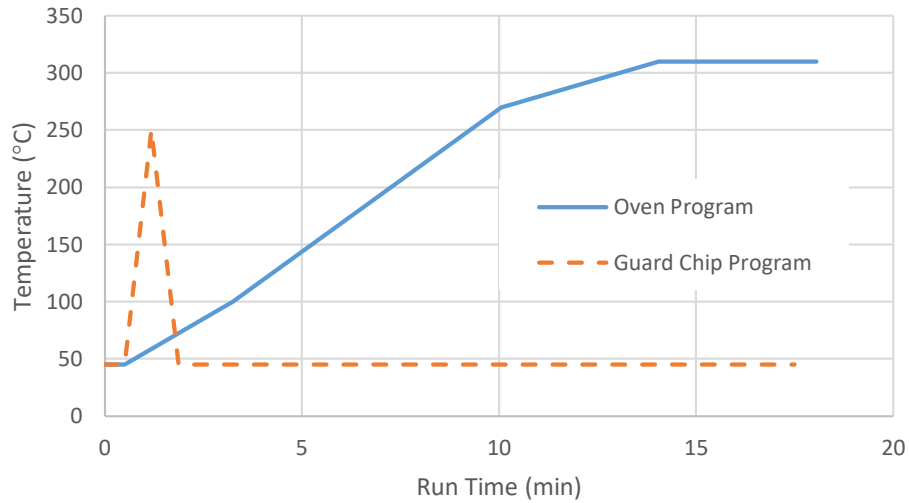


I. Can be used to trap heavy matrix

How high a temperature do you need to pulse the guard chip to release the last compounds of interest?



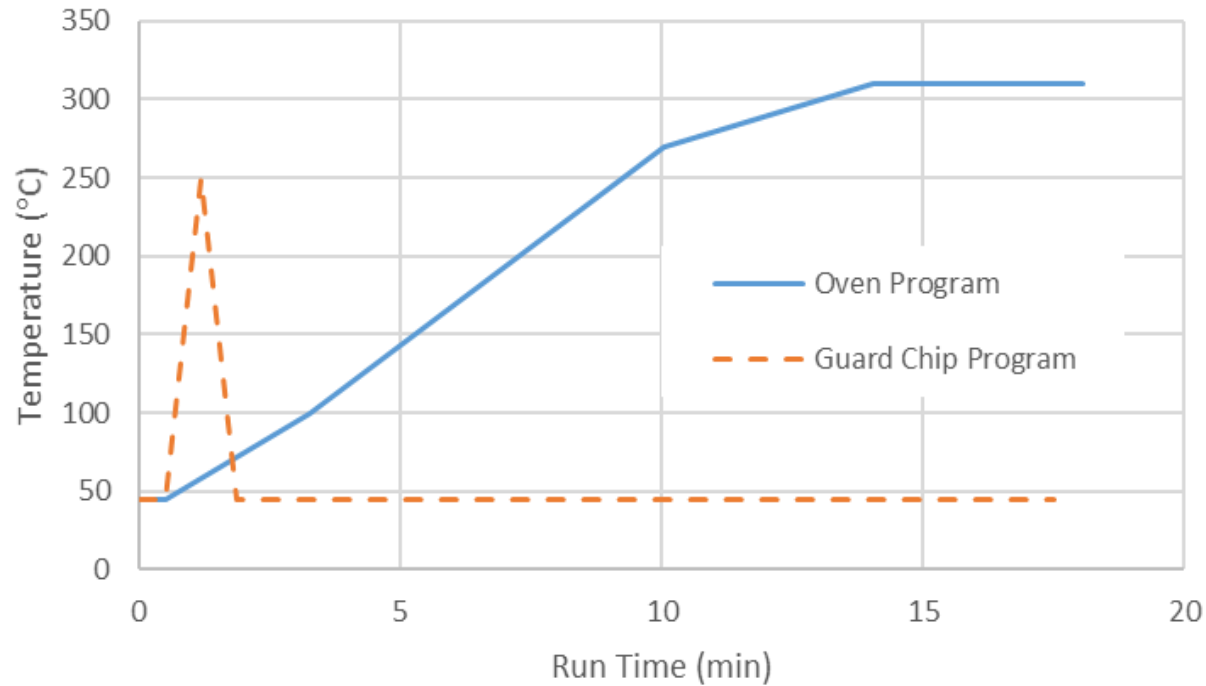
I. Can be used to trap heavy matrix



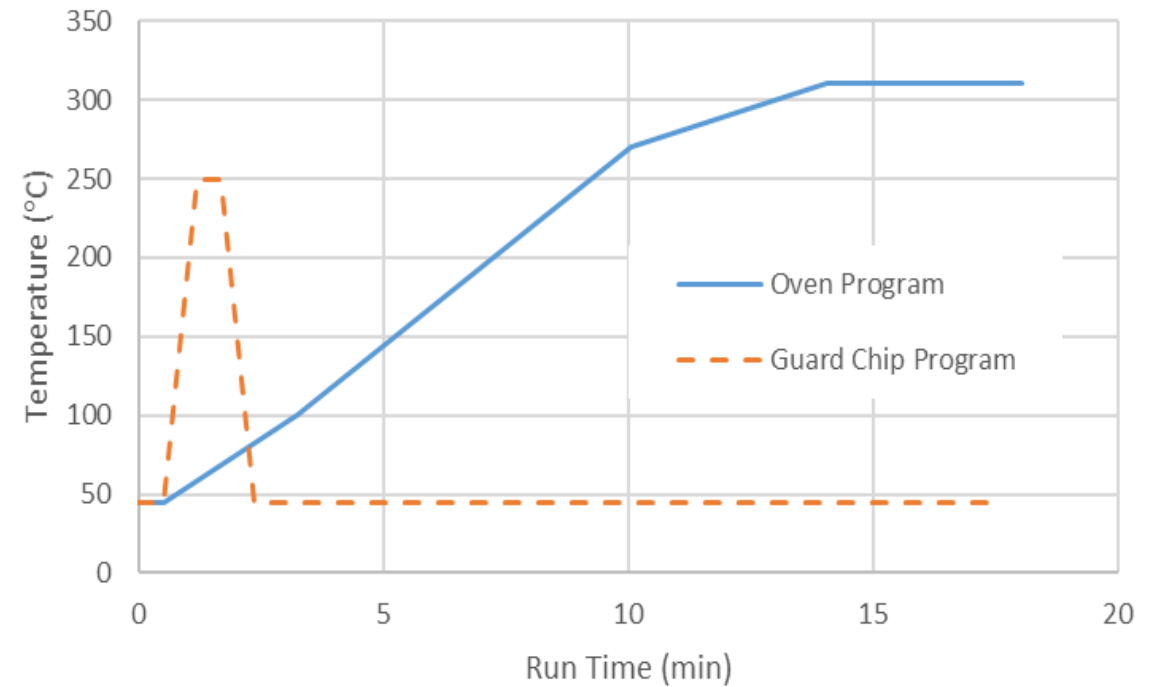
I. Can be used to trap heavy matrix

How can selectivity be further increased with guard chip pulsing?

Guard Chip Pulse



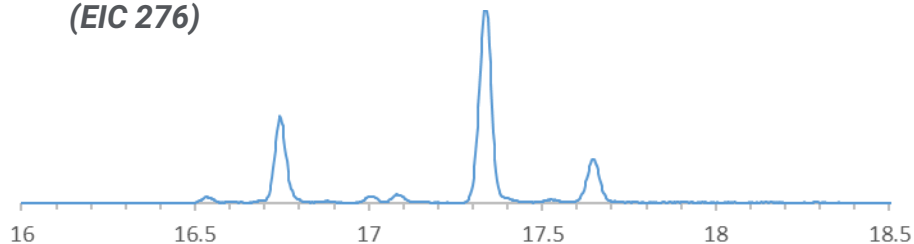
Add Short Isothermal Period



I. Can be used to trap heavy matrix – Injection Blanks After Pulsing

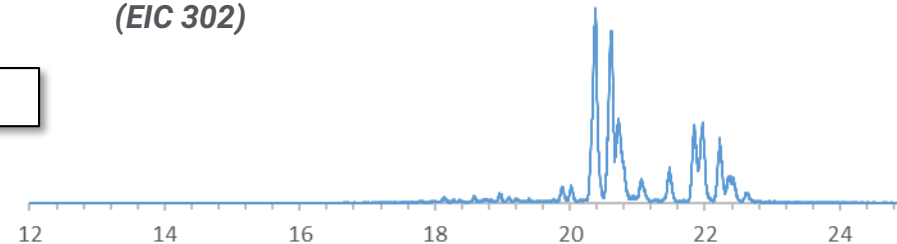
Target PAHs
(EIC 276)

Too short!



Hold Time 1.5 seconds

Matrix PAHs
(EIC 302)

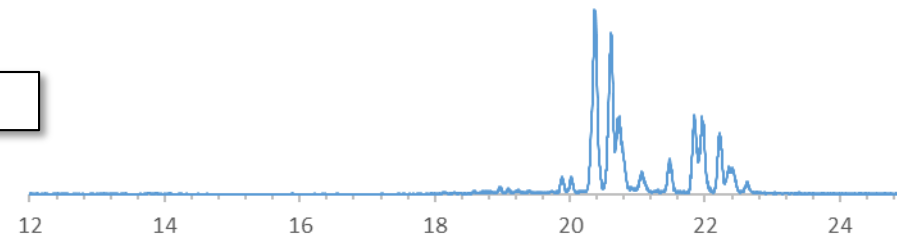


**Best
Compromise!**

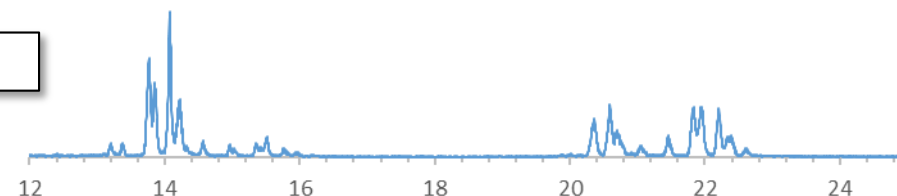
~1% carryover



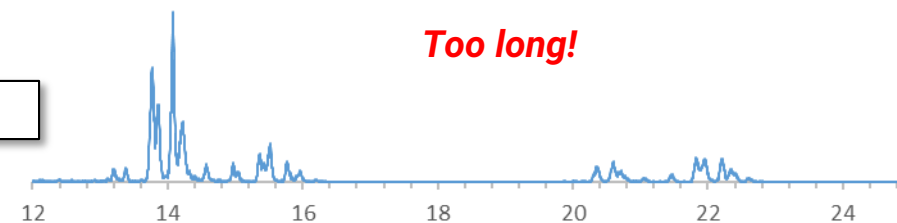
Hold Time 3.0 seconds



Hold Time 6.0 seconds



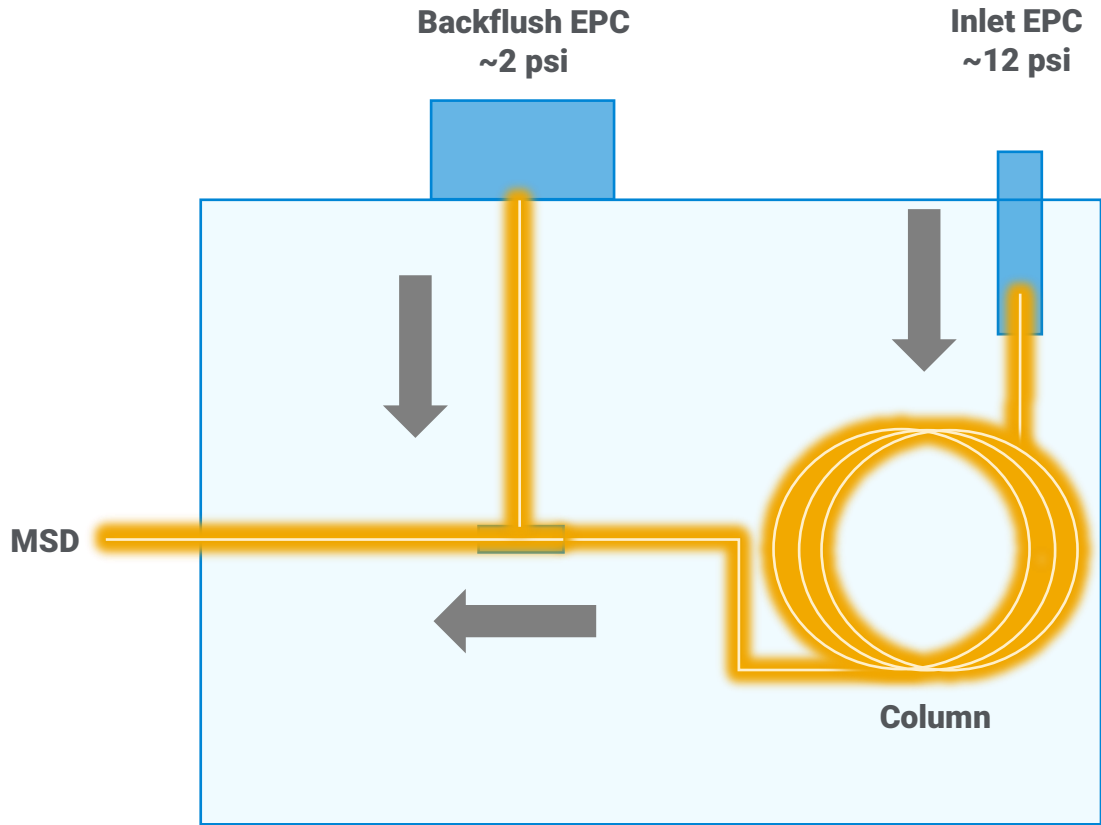
Hold Time 12.0 seconds



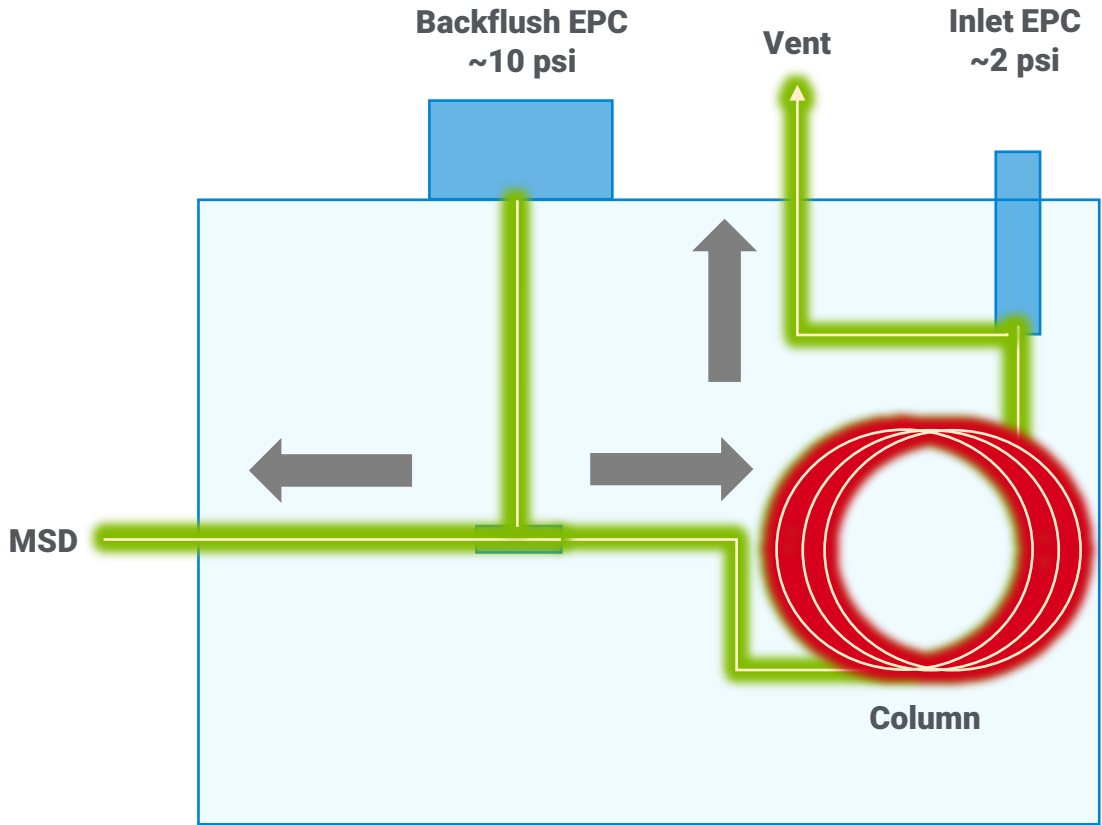
Too long!

II. Guard chip pulsing with backflush

Traditional Air Bath Oven

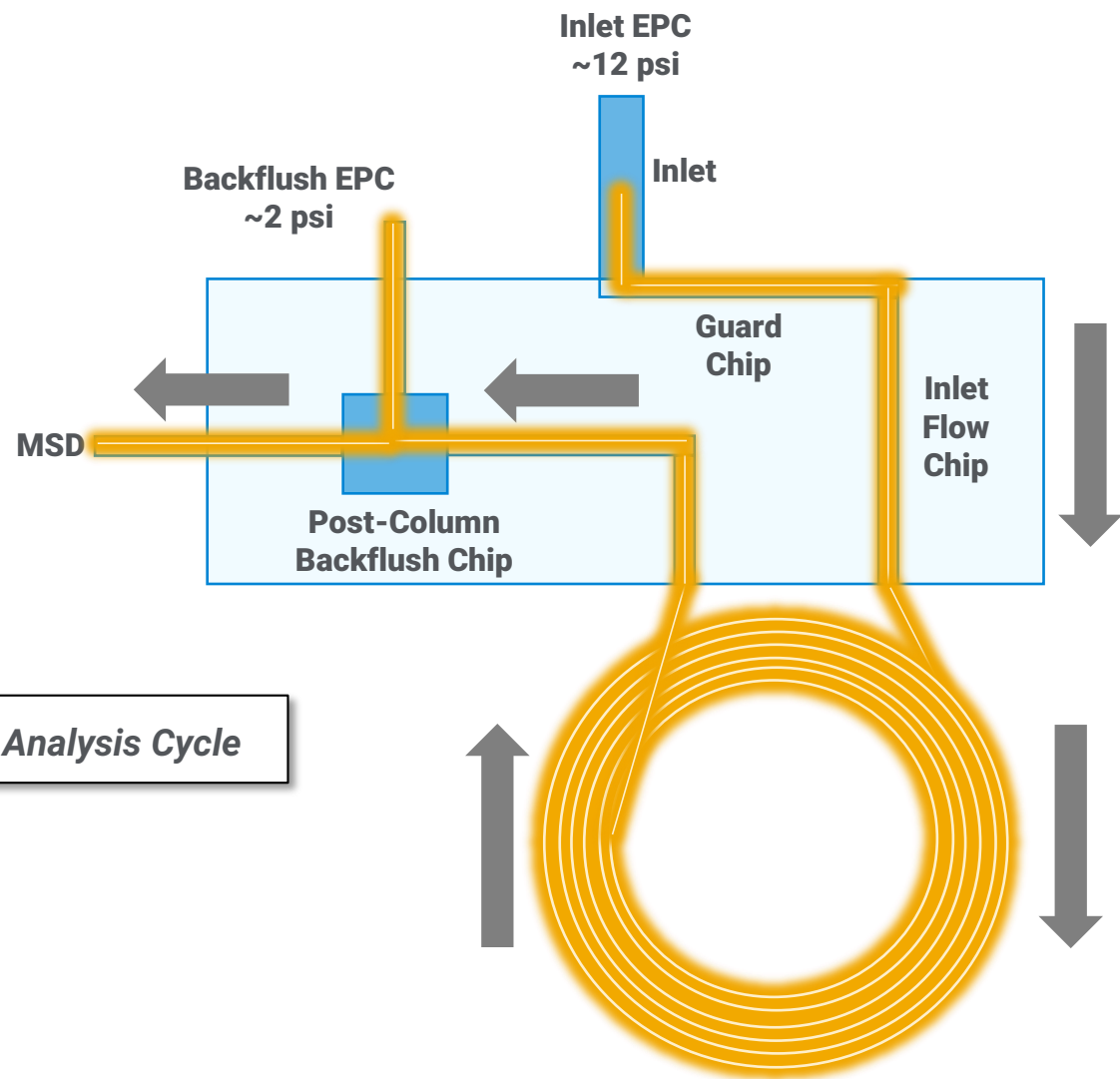


Analysis Cycle

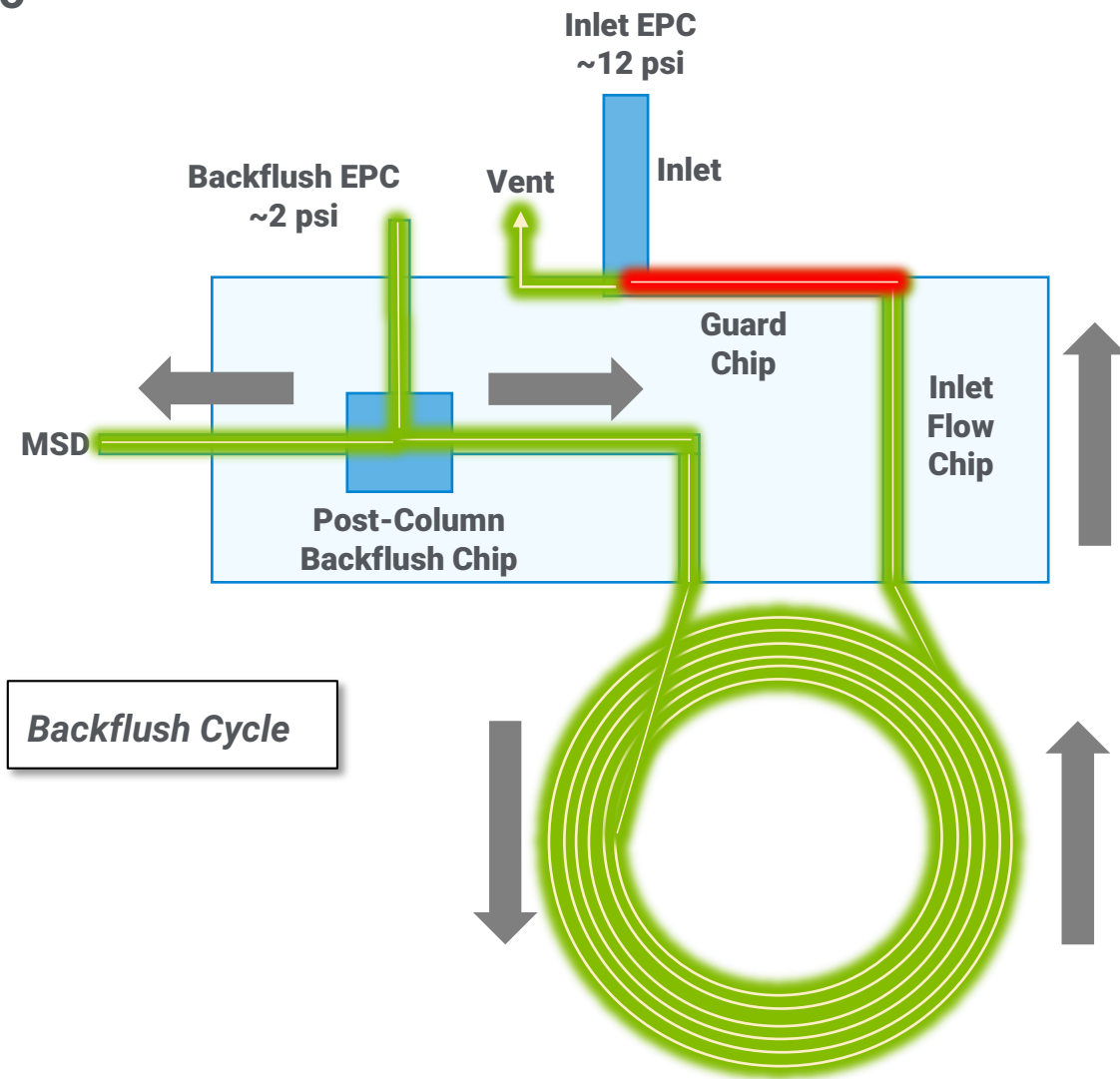


Backflush Cycle

II. Guard chip pulsing with backflush

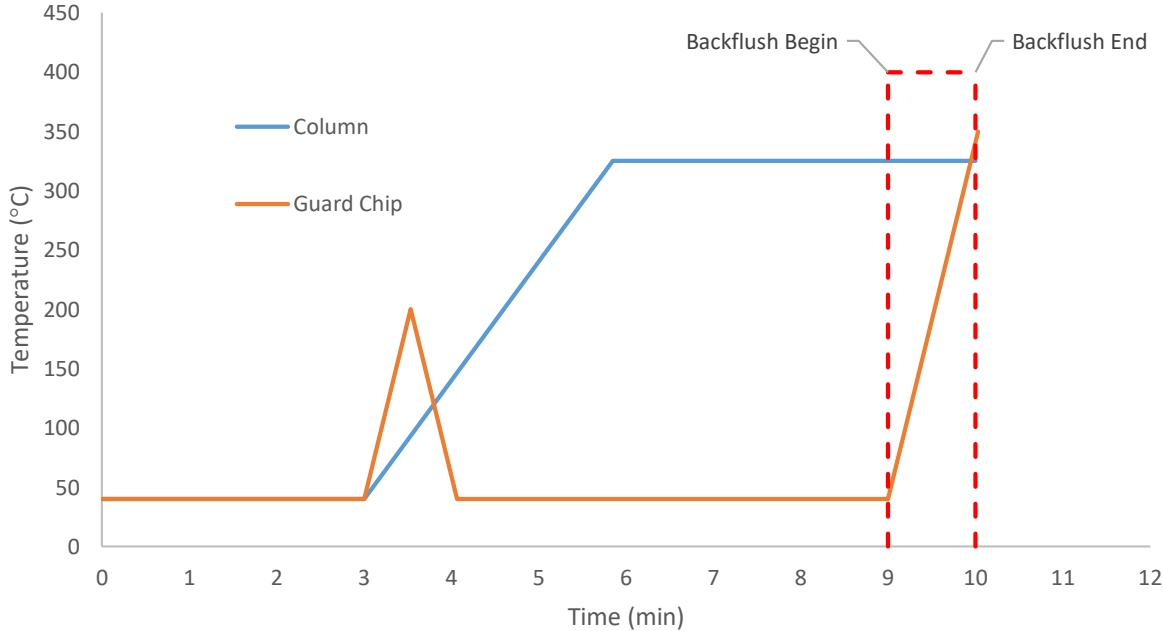


Intuvo

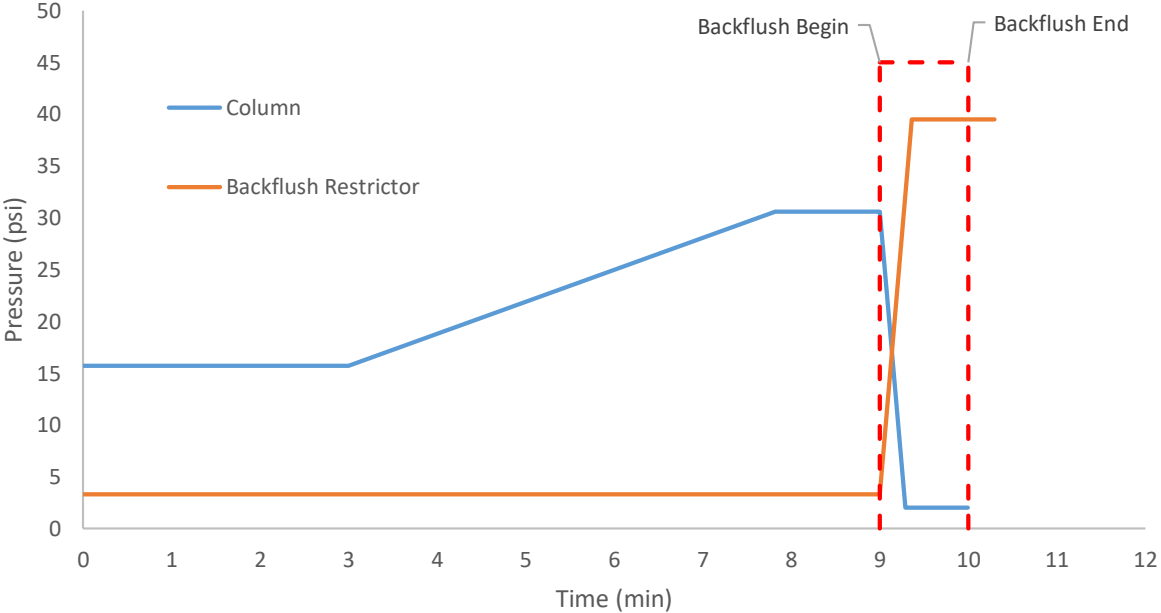


II. Guard chip pulsing with backflush

Temperature Programs

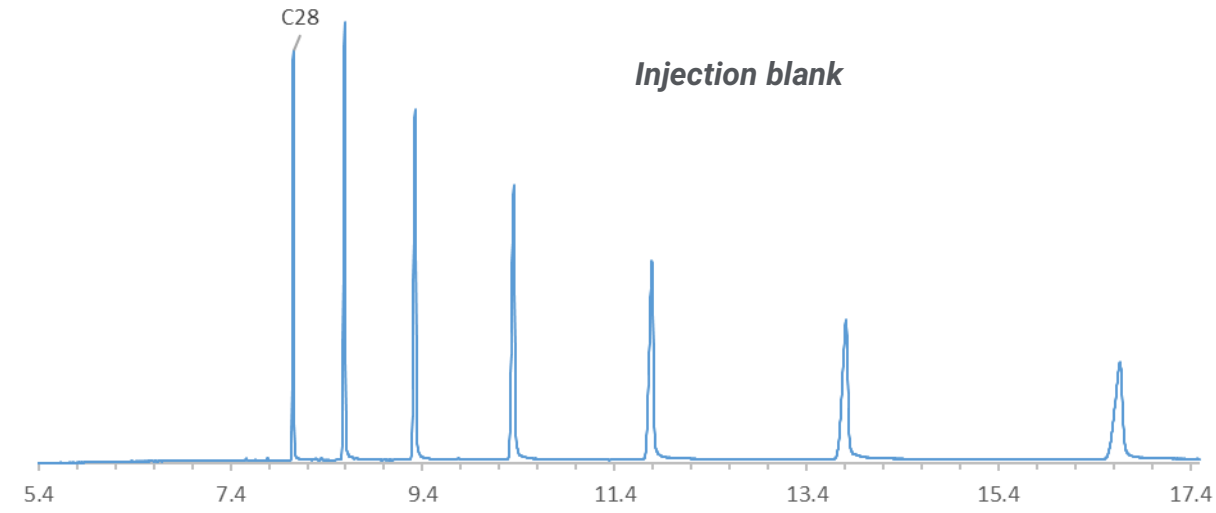
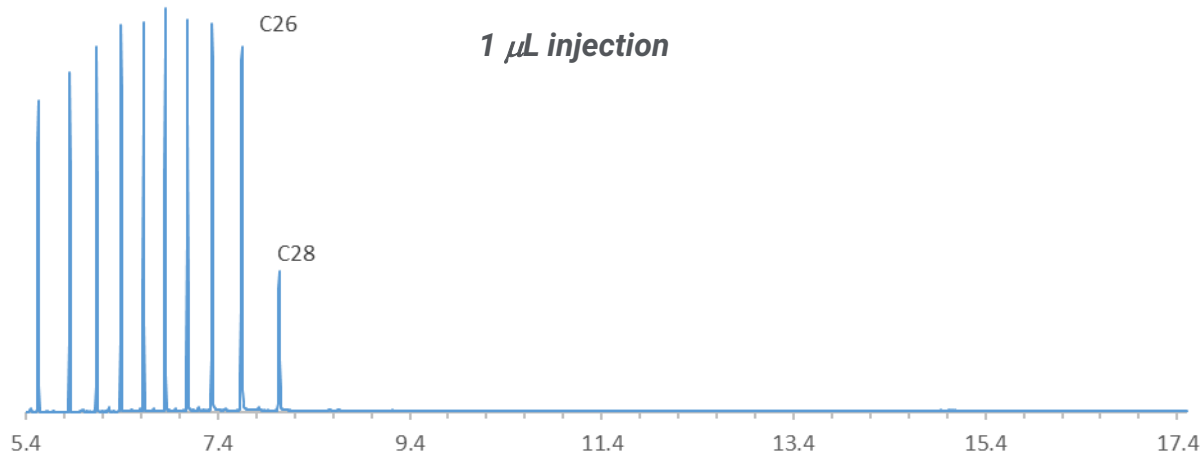


Pressure Programs

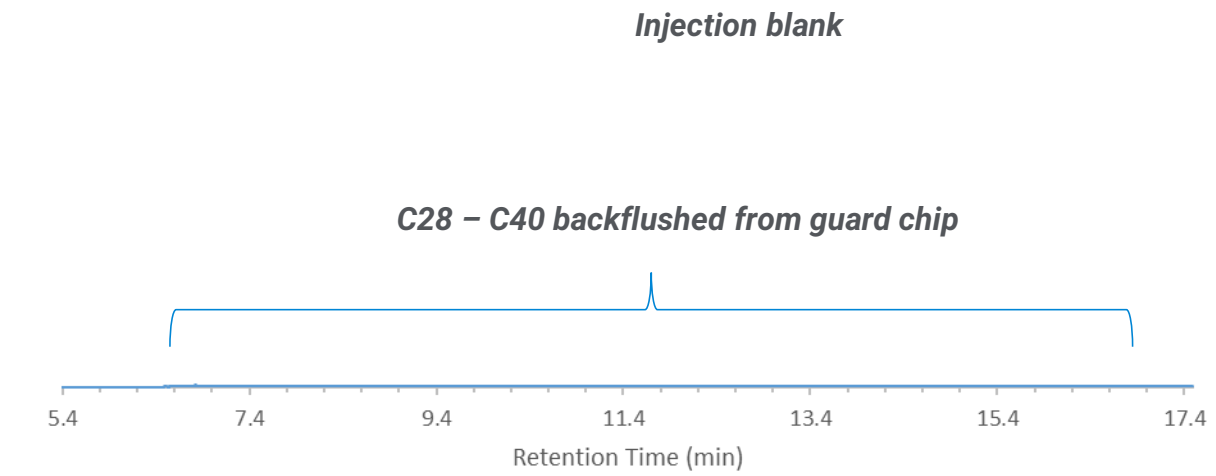
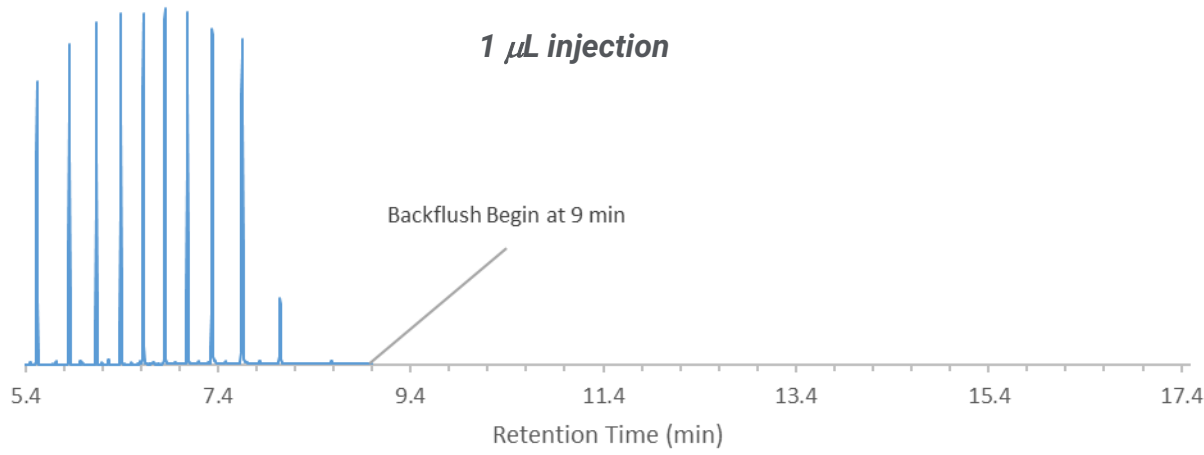


II. Guard chip pulsing with backflush

Without Backflush

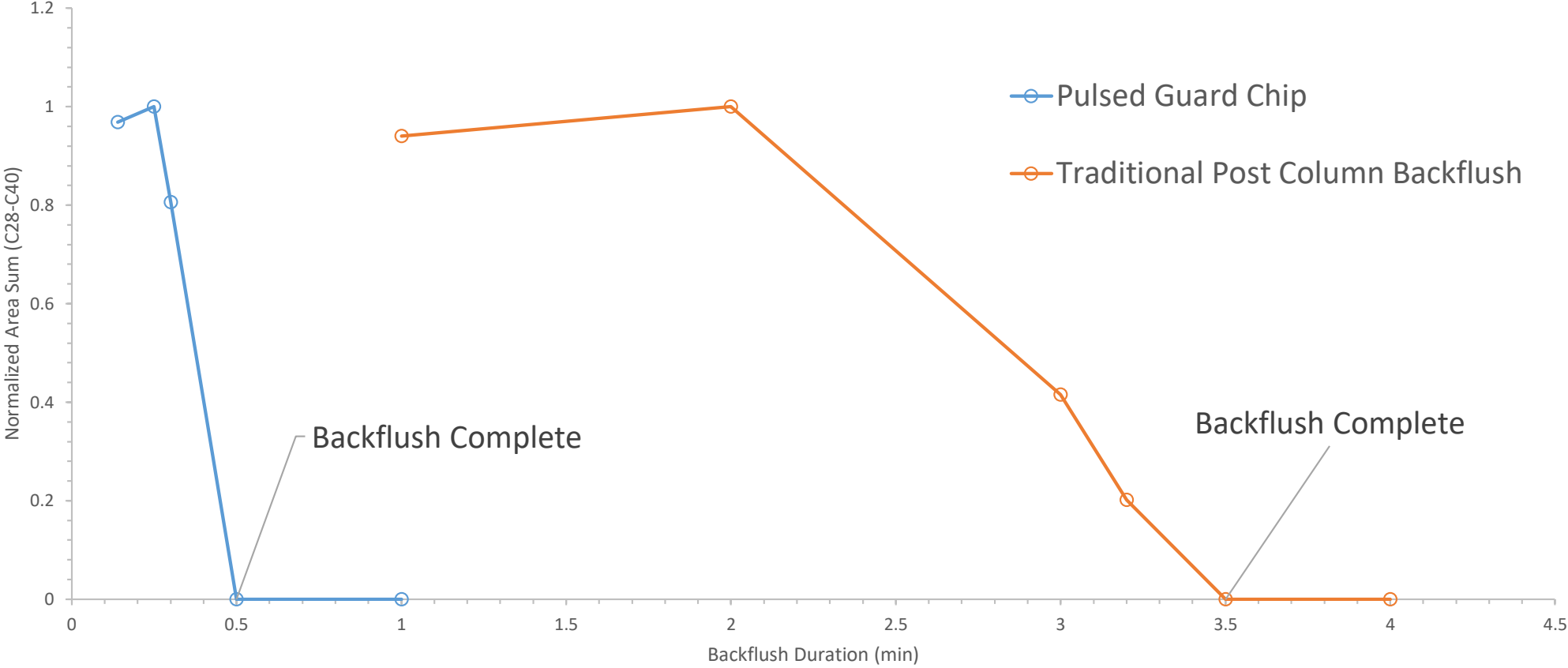


With Backflush (1 min duration)



II. Guard chip pulsing with backflush

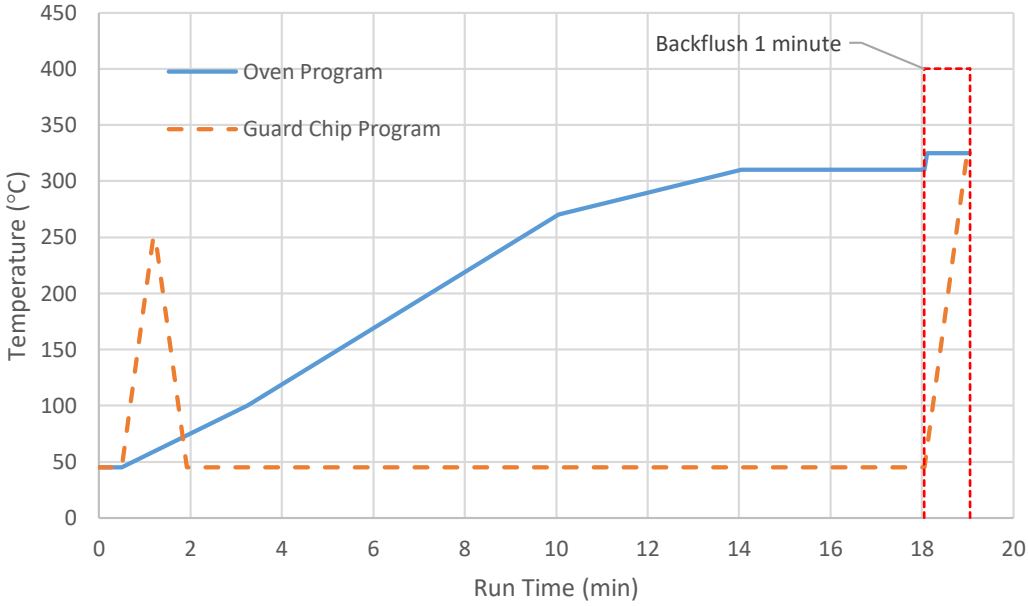
Pulse guard chip backflush – 7x improvement in time required to backflush



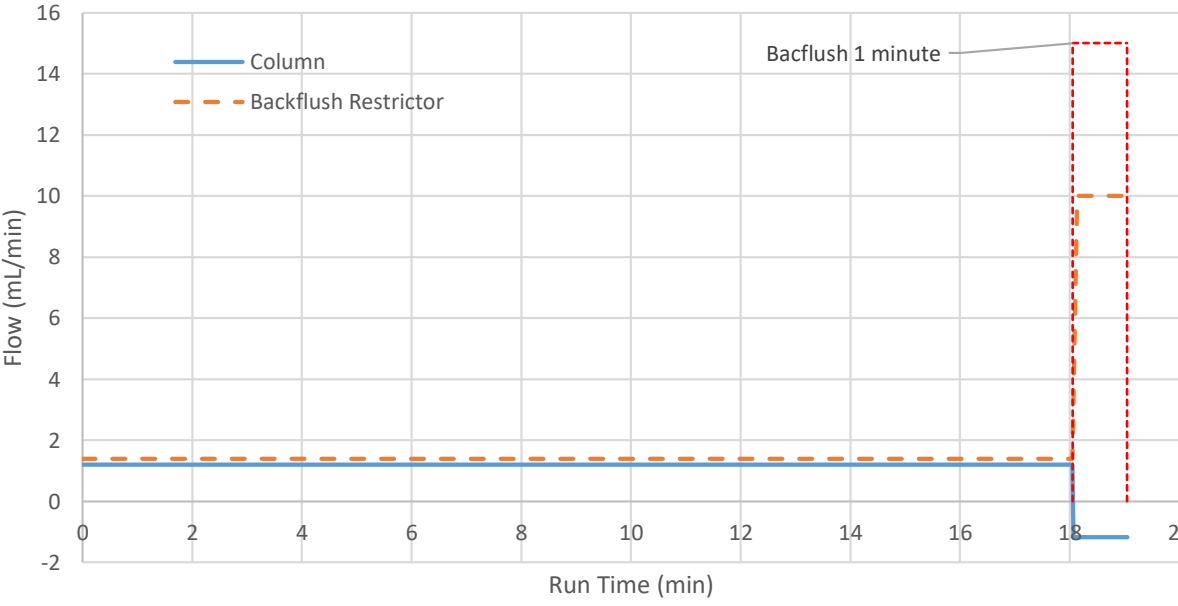
II. Guard chip pulsing with backflush

Matrix backflushing pulsed guard chip with 3 second hold.

Temperature Program



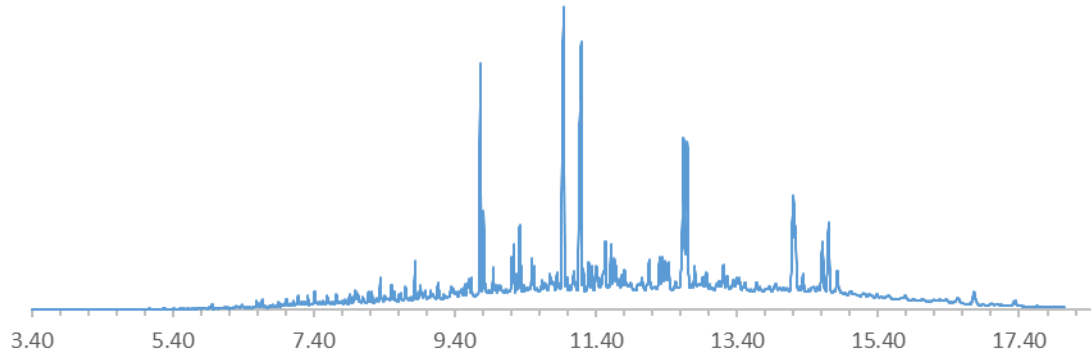
Flow Program



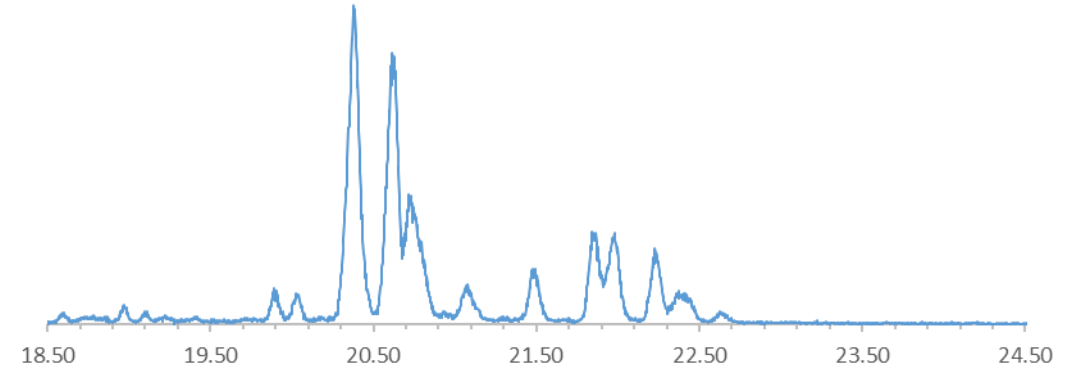
II. Guard chip pulsing with backflush – matrix injections

Without Backflush

1 μ L injection

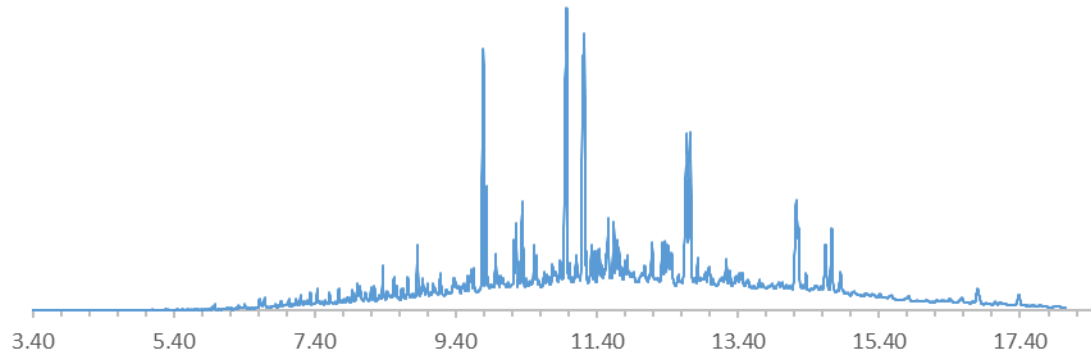


Injection blank

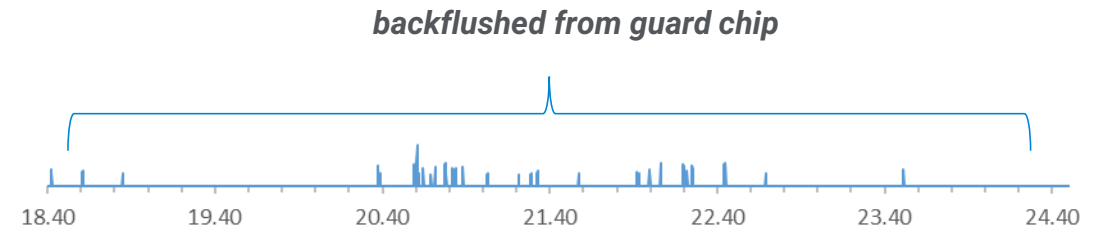


With Backflush (1 min duration)

1 μ L injection

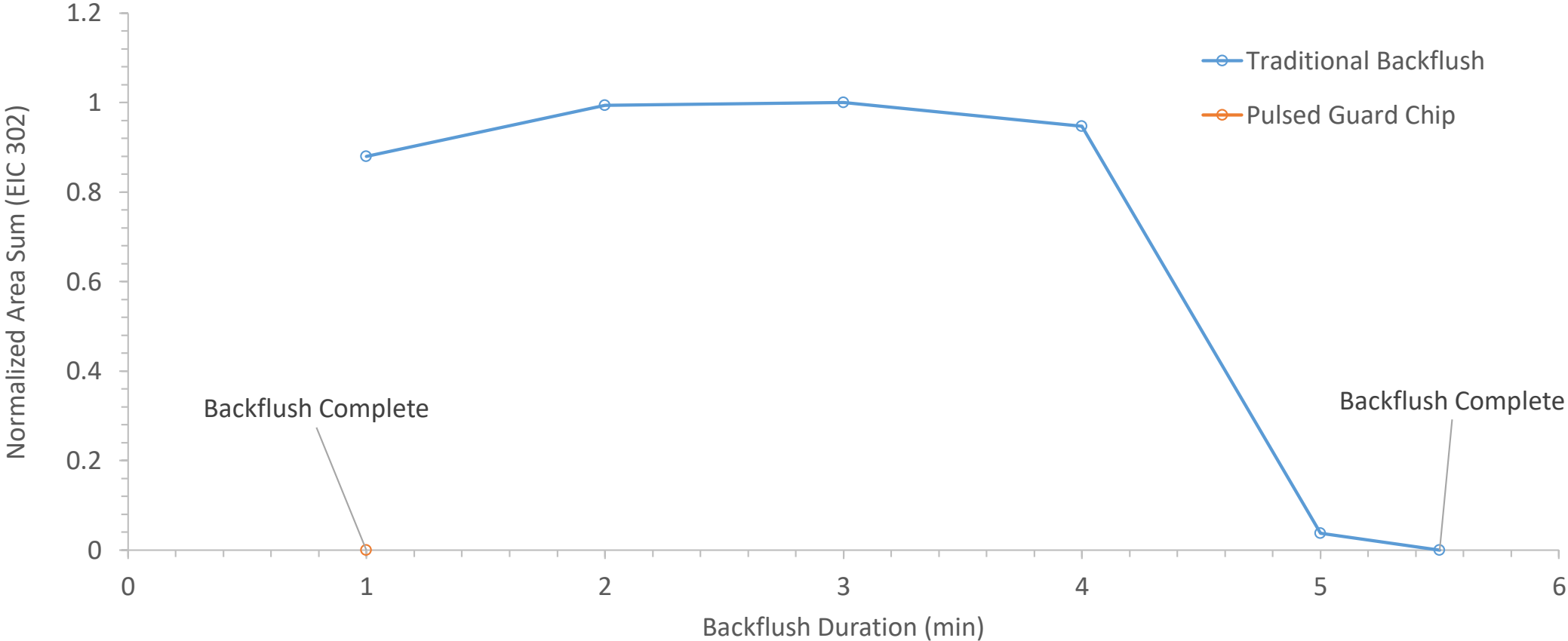


Injection blank



II. Guard chip pulsing with backflush

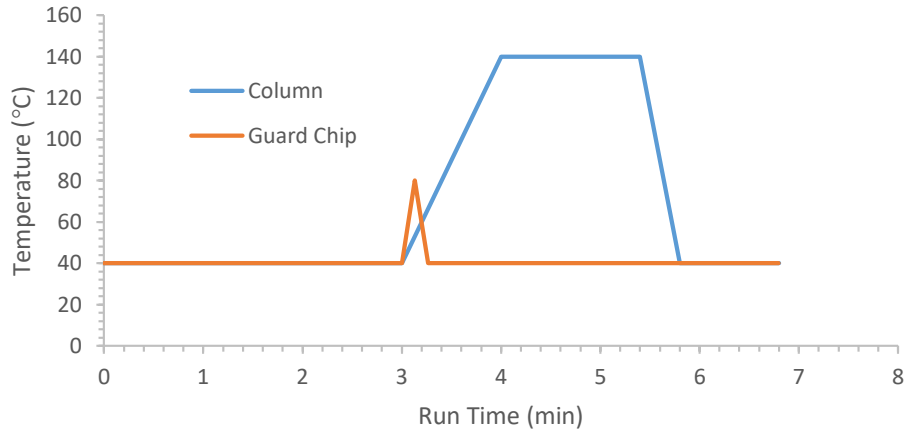
Pulse guard chip backflush – 5x improvement in time required to backflush



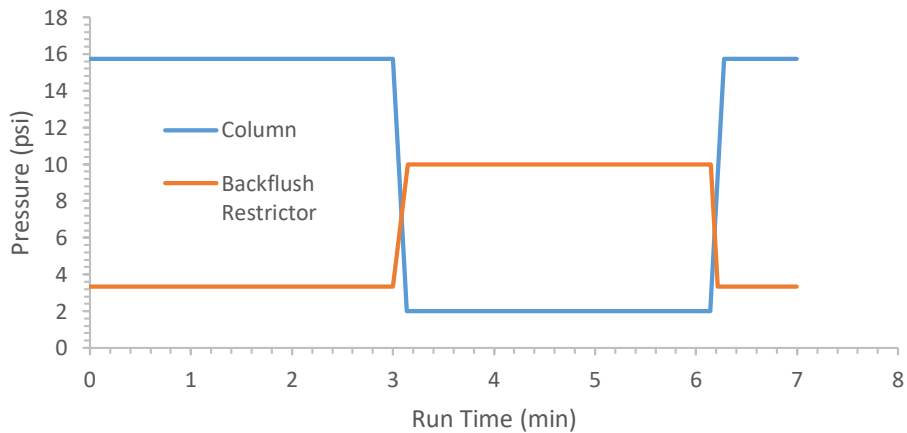
III. Pseudo heart-cutting – requires two GC runs

Sample loading and backflushing below 80 °C (~C18-C10)

Temperature Program

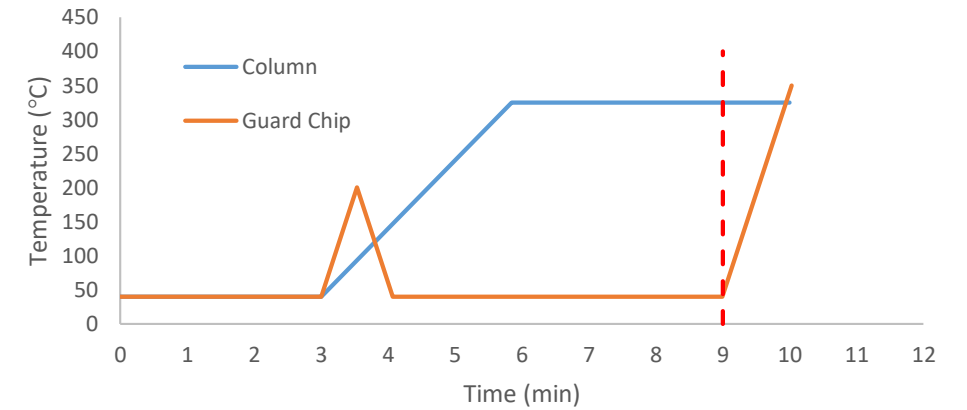


Pressure Program

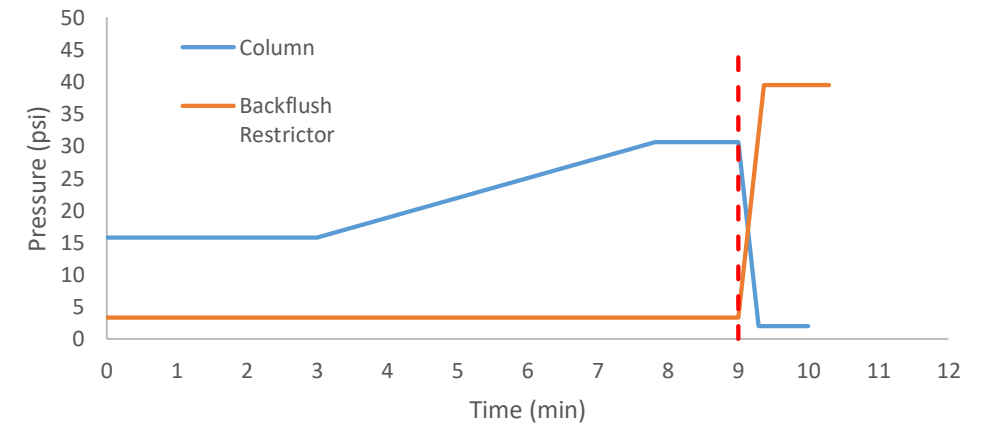


Analyze and backflush above 200 °C (C28-C40)

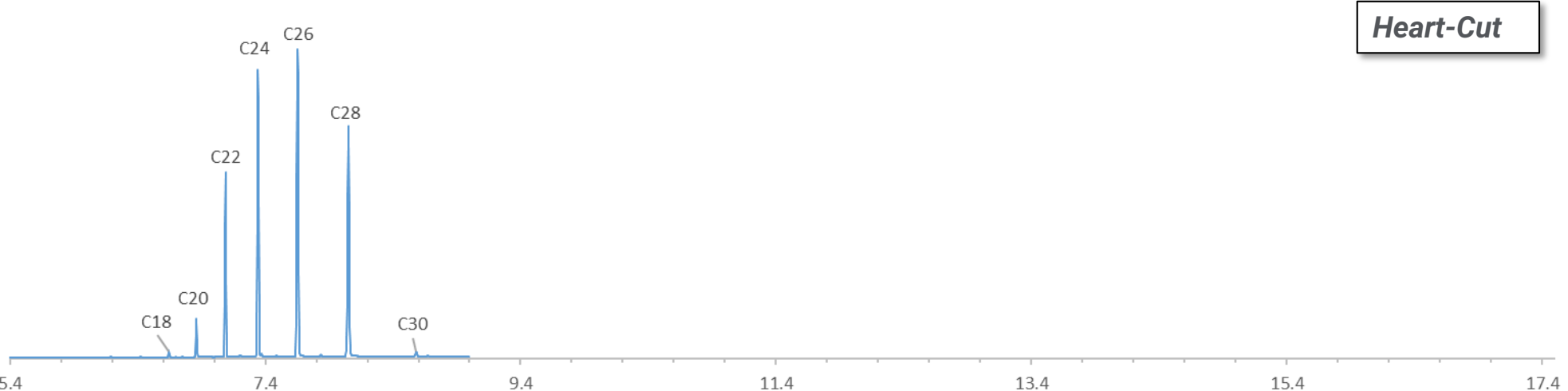
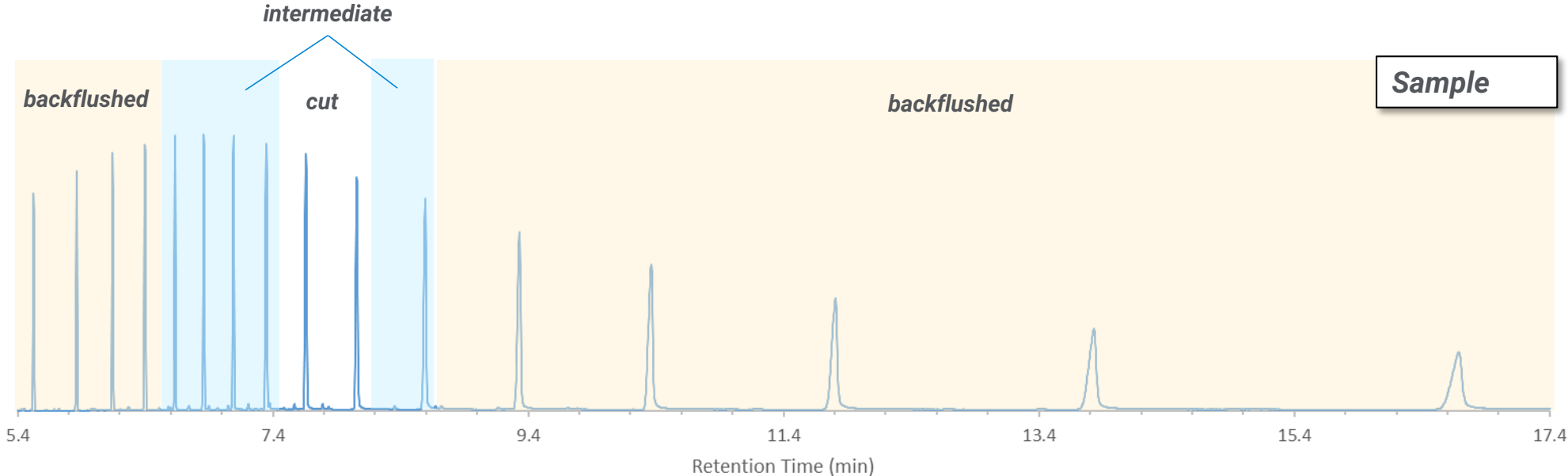
Temperature Programs



Pressure Programs



III. Pseudo heart-cutting



Conclusions

- **Provided some fundamental information on how the guard chip functions in the Intuvo**
- **How precise temperature control can be used to selectively trap compounds on the guard chip**
- **Combining guard chip temperature pulsing with precise flow programming can be used to backflush lower volatility compounds from the guard chip before they enter the column**
- **This provides more efficient use of backflushing compared to traditional post-column backflush**
- **Precise control of guard chip temperature and pressure can be used for pseudo-heart cutting**