A Fast Method for US EPA 8270 Using Direct Heating Column Technology

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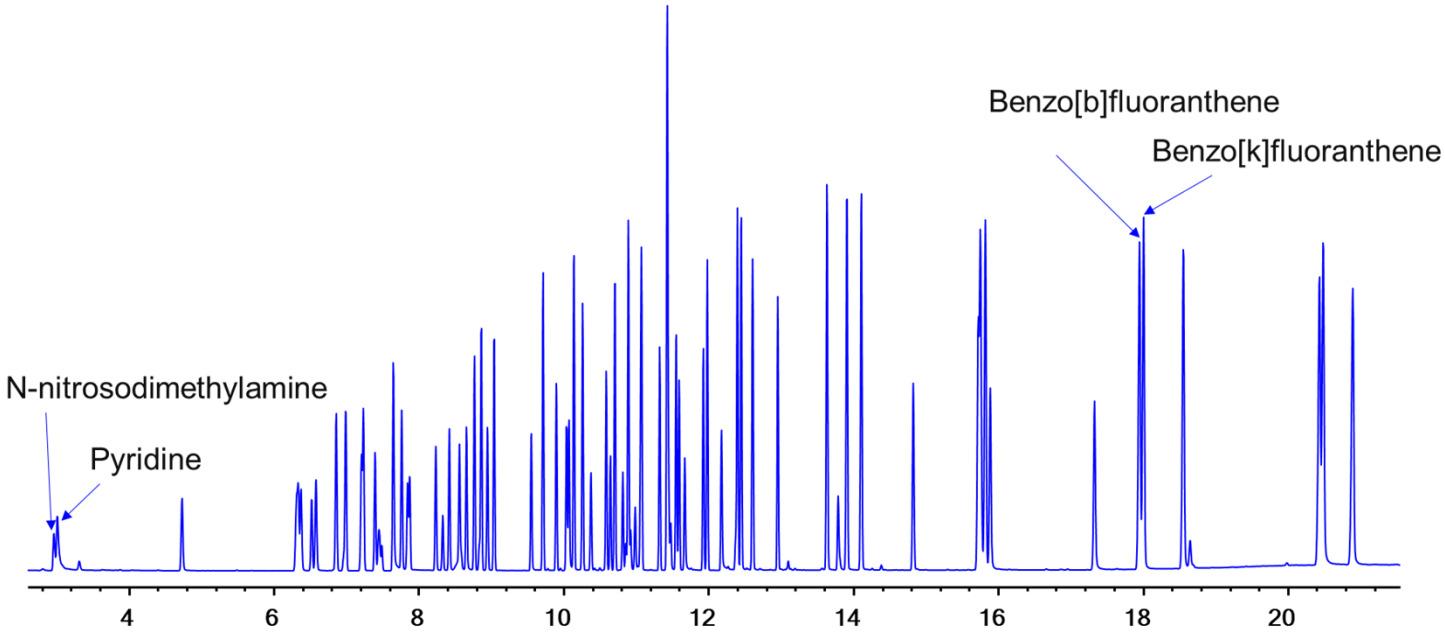
Agilent Technologies

Environmental Protection Agency (EPA) Method 8270D

- Provides procedures and requirements for the quantitation of semivolatile organic compounds (SVOCs) extracted from solid waste, soil, water, or air by GC/MS
- Typical GC/MS analysis run time with a 30 m x 0.25 mm ID column is >20 min



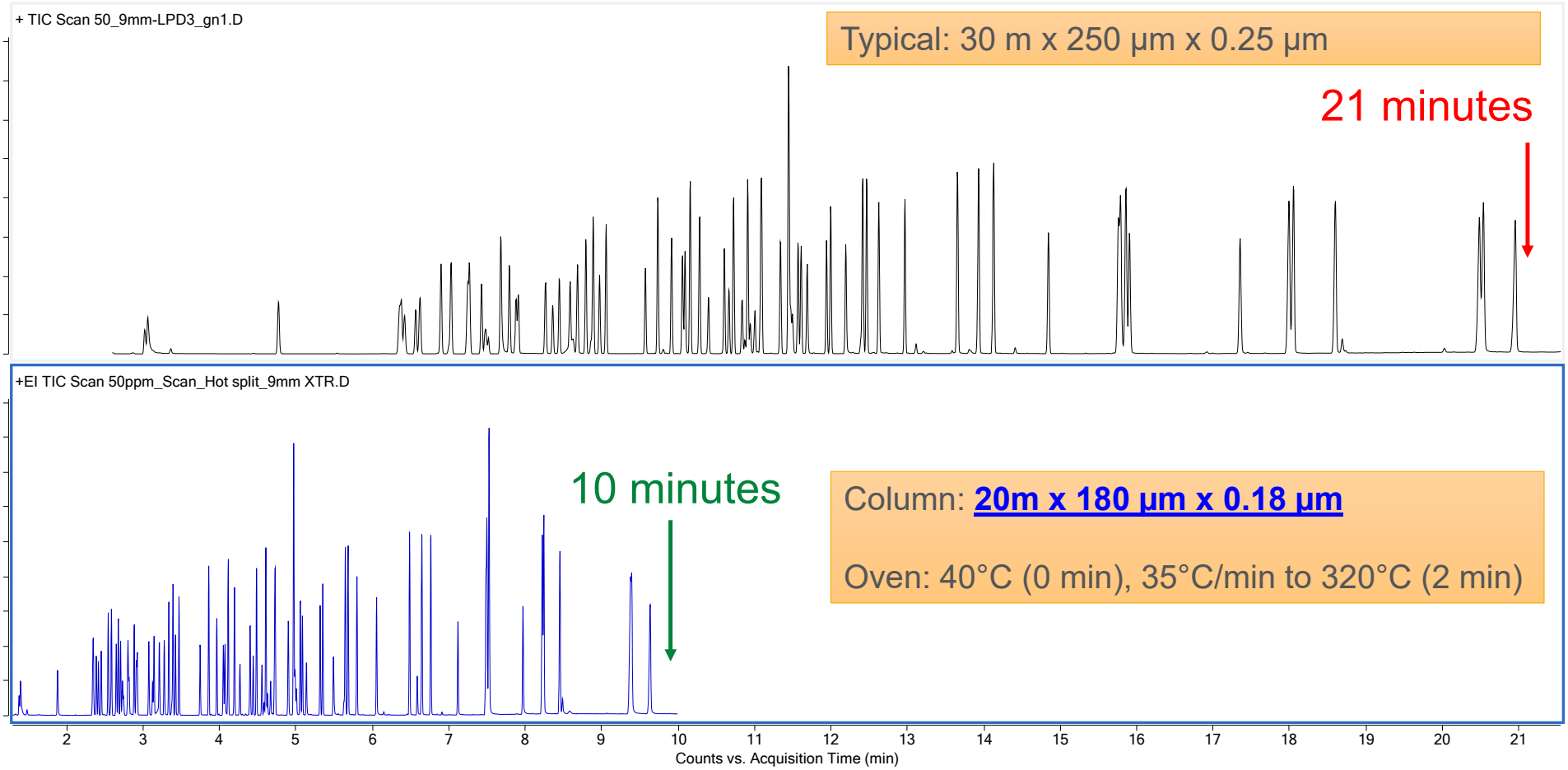
Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); Method 8270E; United Stated Environmental Protection Agency, Revision 6, June 2018.

Environmental Protection Agency (EPA) Method 8270D

- Provides procedures and requirements for the quantitation of semivolatile organic compounds (SVOCs) extracted from solid waste, soil, water, or air by GC/MS
- Typical GC/MS analysis run time with a 30 m x 0.25 mm ID column is >20 min

- Demand for high throughput drives the interest in faster analysis:
  - columns with smaller ID
  - direct column heating
    - ✓ increased temperature programming rates
    - ✓ faster cooling

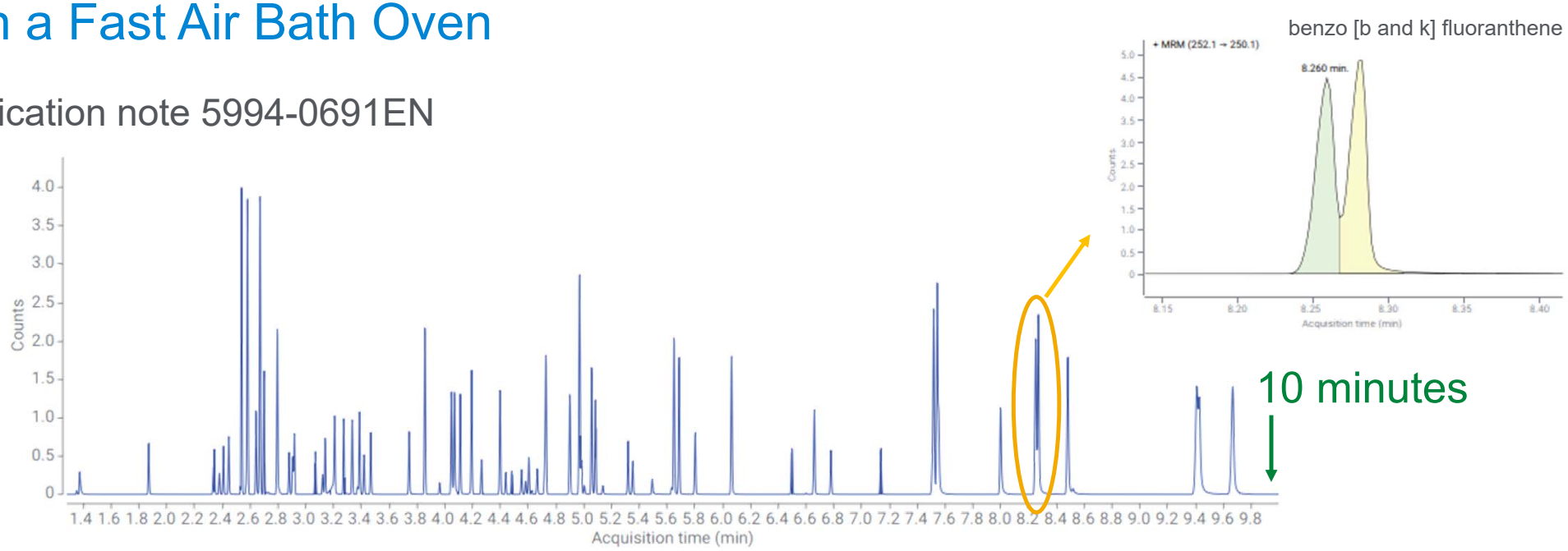
Fast EPA 8270 Method with a 20 m Column and a Fast Air Bath Oven



Churley, M. Agilent Technologies, publication number 5994-0691EN, 2019.

Fast EPA 8270 Method with GC/MS/MS in MRM Acquisition Mode with a Fast Air Bath Oven

Application note 5994-0691EN



- 20m x 180 µm x 0.18 µm 5% phenyl (polysiloxane) column
- Sufficient chromatographic resolution for benzo [b and k] fluoranthene
- **A 240-V air bath GC oven** that enables ramp rate up to 35°C/min to 320°C (or using an oven insert)

Environmental Protection Agency (EPA) Method 8270D

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- Demand for high throughput drives the interest in faster analysis:
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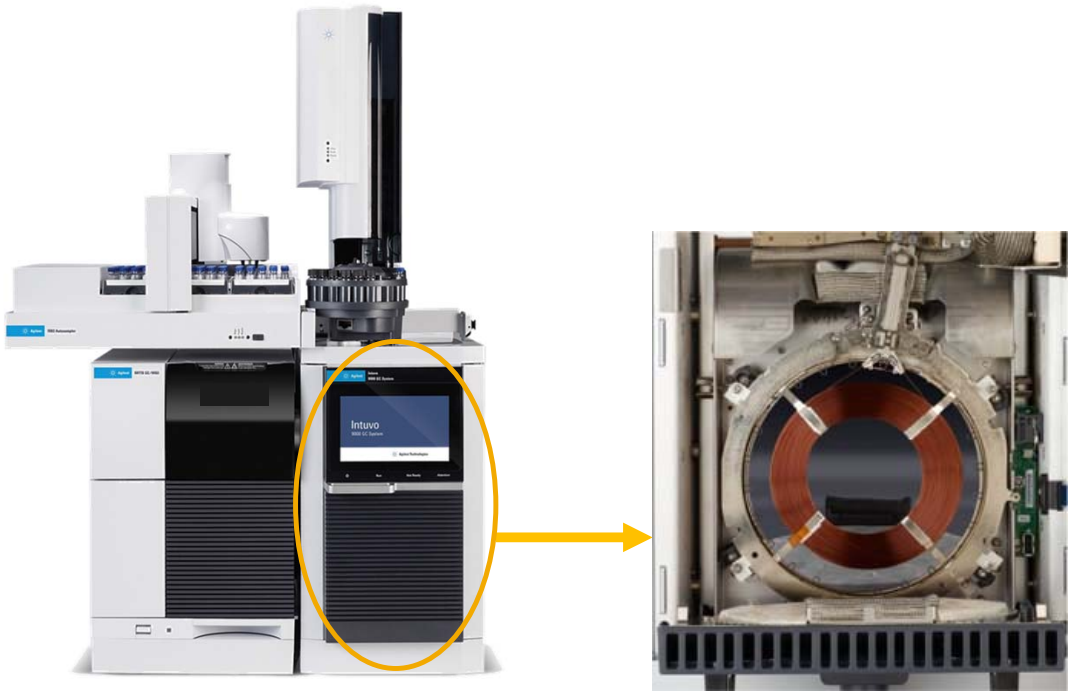
  - direct column heating
    - ✓ increased temperature programing rates
    - ✓ faster cooling
    - ✓ no special electrical service requirements (V or A)

Direct Column Heating

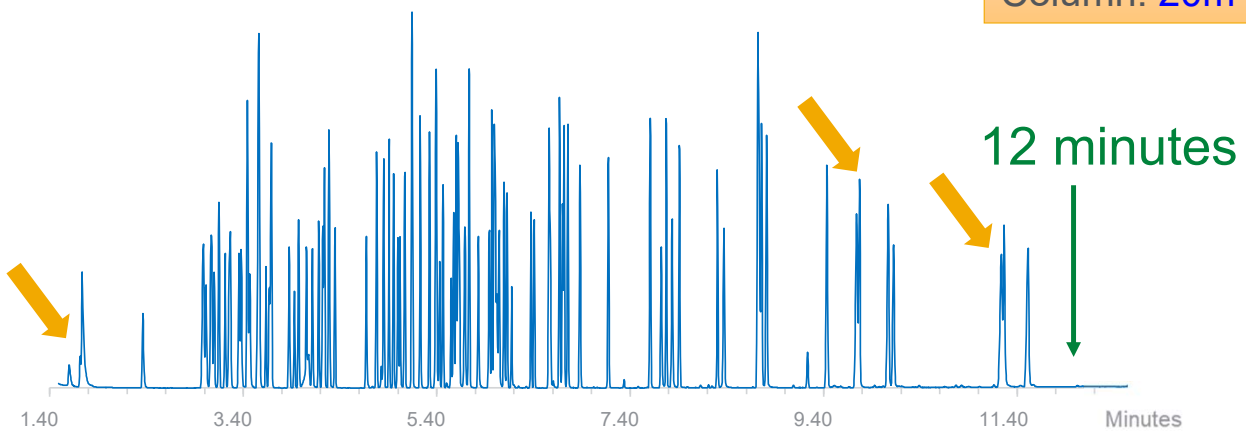


Intuvo 9000 GC with 5977B MSD

- Over 50% reduction in the electrical power consumption compared to a conventional air bath GC
- Reduction in the emitted heat energy
- No special electrical requirements
- Ultra-fast GC heating and cooling (up to 250 °C/min)
- Small footprint

Fast EPA 8270 Method with Direct Column Heating GC/MS

Column: 20m x 180µm x 0.18 µm

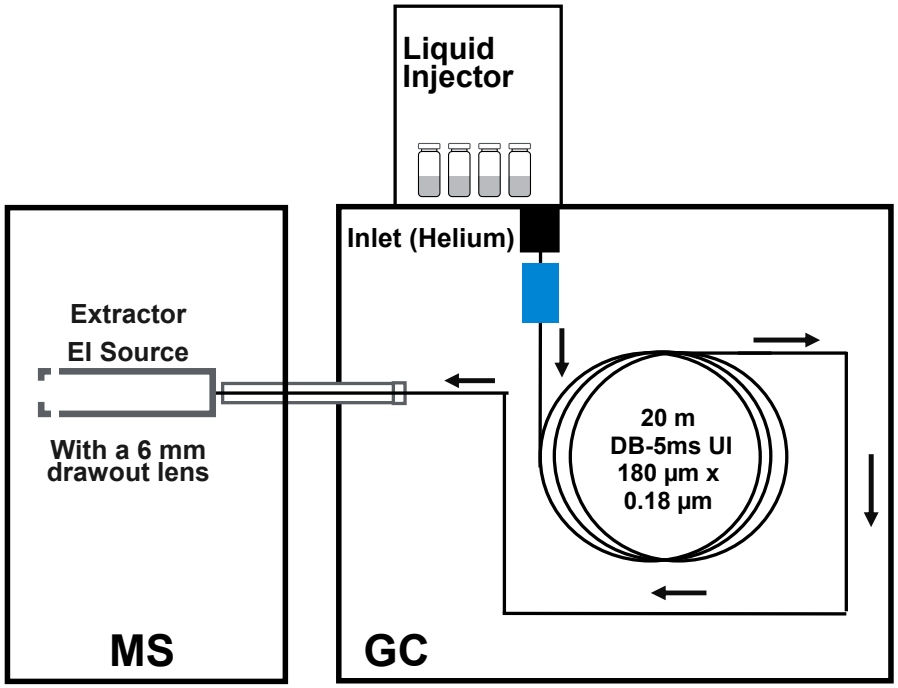


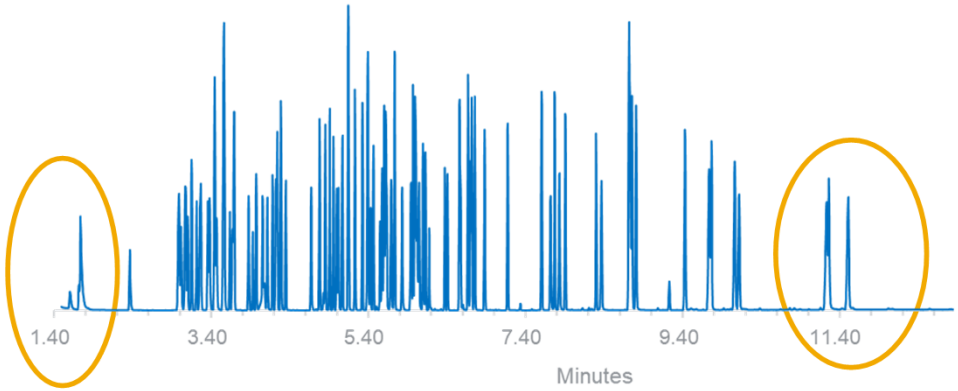
Direct heating GC that does not require special electrical service at the bench

Sufficient chromatographic resolution for:

- benzo [b and k] fluoranthene
- the latest eluters: indenopyrene and dibenzoanthracene
- the earliest eluters: 1,4-dioxane, N-nitrosodimethylamine, and pyridine

Direct Heating GC/MS Instrument Configuration

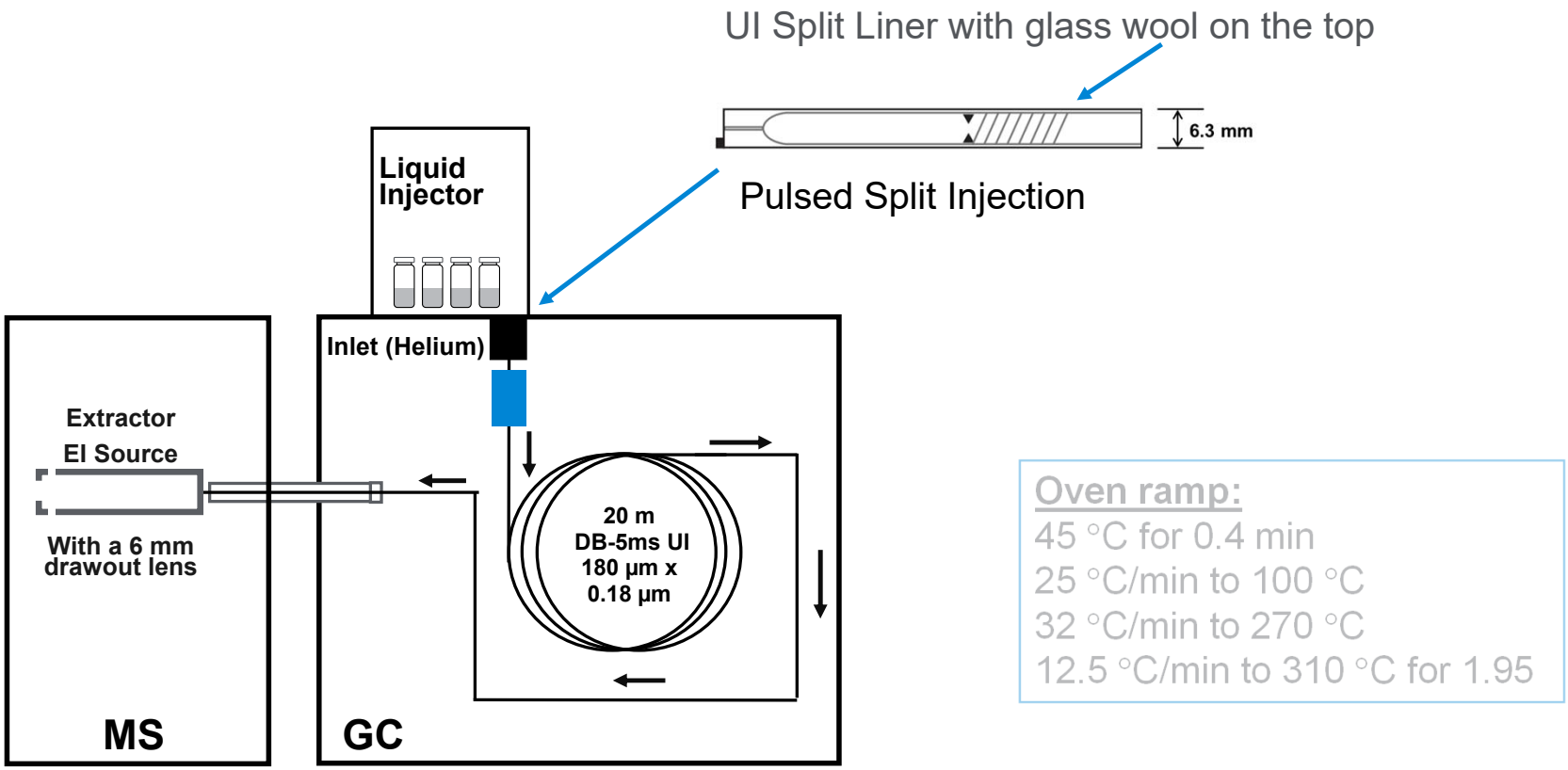




Oven ramp:
45 °C for 0.4 min
25 °C/min to 100 °C
32 °C/min to 270 °C
12.5 °C/min to 310 °C for 1.95

Optimized for chromatographic resolution of all the targets, including the earliest- and the later-eluters

Direct Heating GC/MS Instrument Configuration



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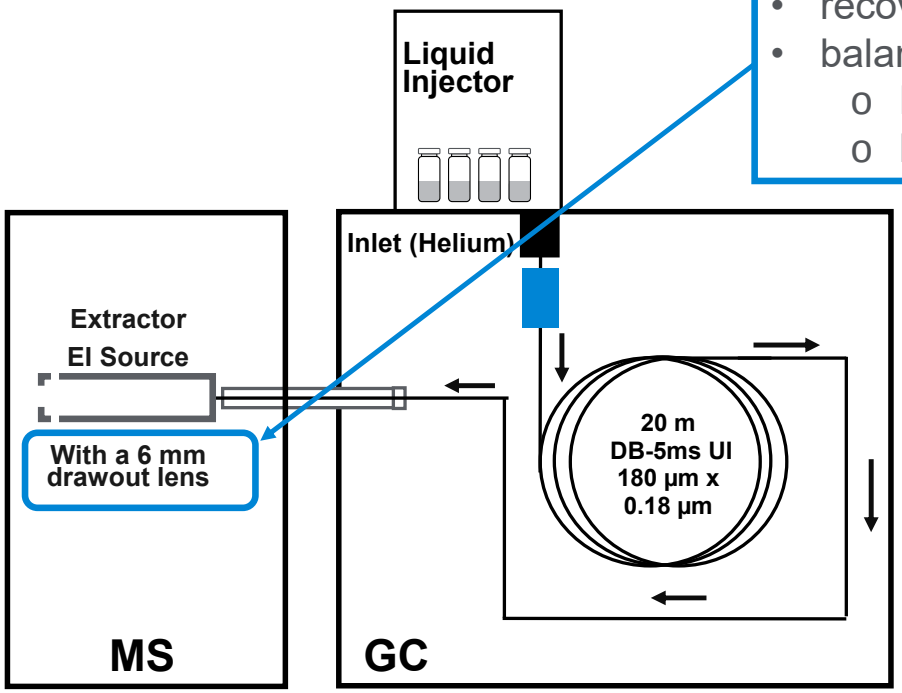
Churley, M. Agilent Technologies, publication number 5994-0350EN, 2018.

Direct Heating GC/MS Instrument Configuration



6 mm drawout lens is advantageous in terms of
- recovery of the most challenging classes of compounds
- balance between:
  - High sensitivity
  - Linearity over the widest overall calibration range

Oven ramp:
45 °C for 0.4 min
25 °C/min to 100 °C
32 °C/min to 270 °C
12.5 °C/min to 310 °C for 1.95

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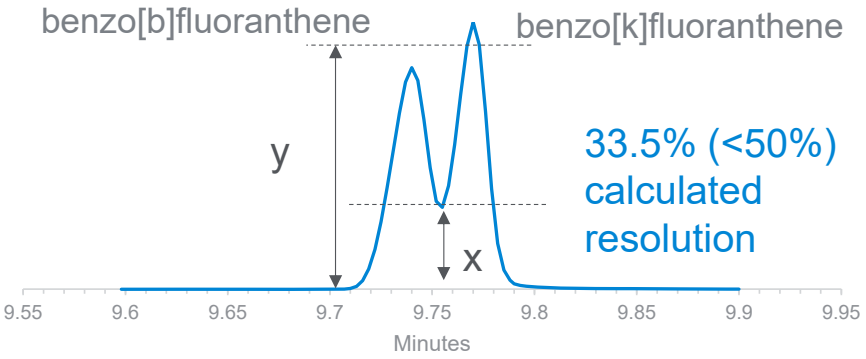
Optimized Direct Heating GC/MS Method Enabled Peak Resolution

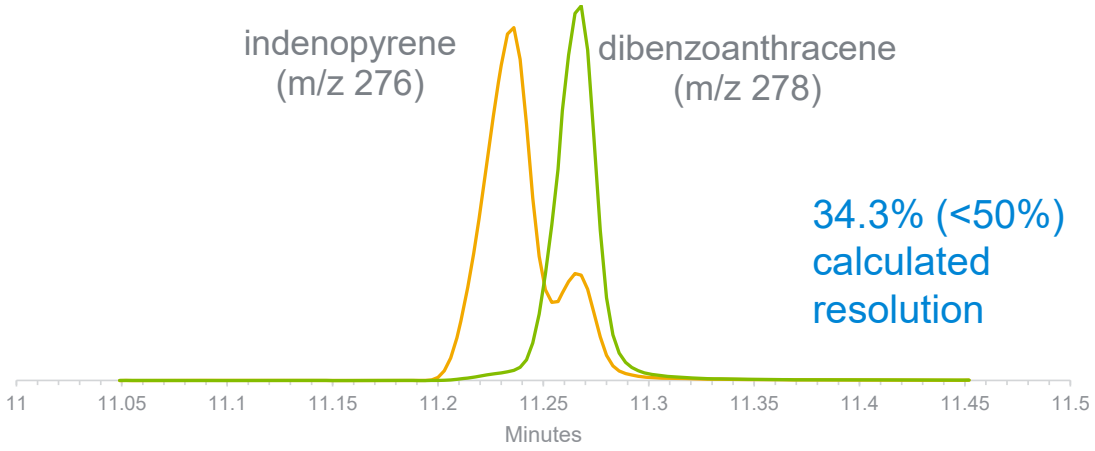
- "Benzo [b and k] fluoranthene must be resolved to a maximum value of 50% as calculated by the ratio of the valley height between the isomers (X) to the average of the two height maxima (Y)".

  Here it is measured at the highest concentration standard.

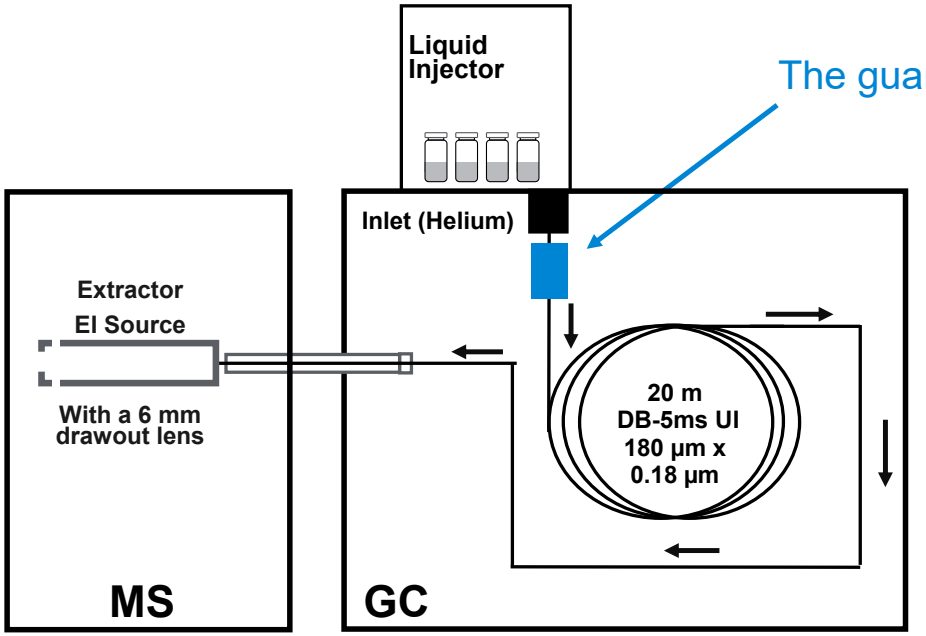


- Indenopyrene and dibenzoanthracene.

  The oven ramp was intentionally optimized (reduced after 270 °C) to enable the separation.



Microfluidic Temperature Programmable Retention Gap (Guard Chip)



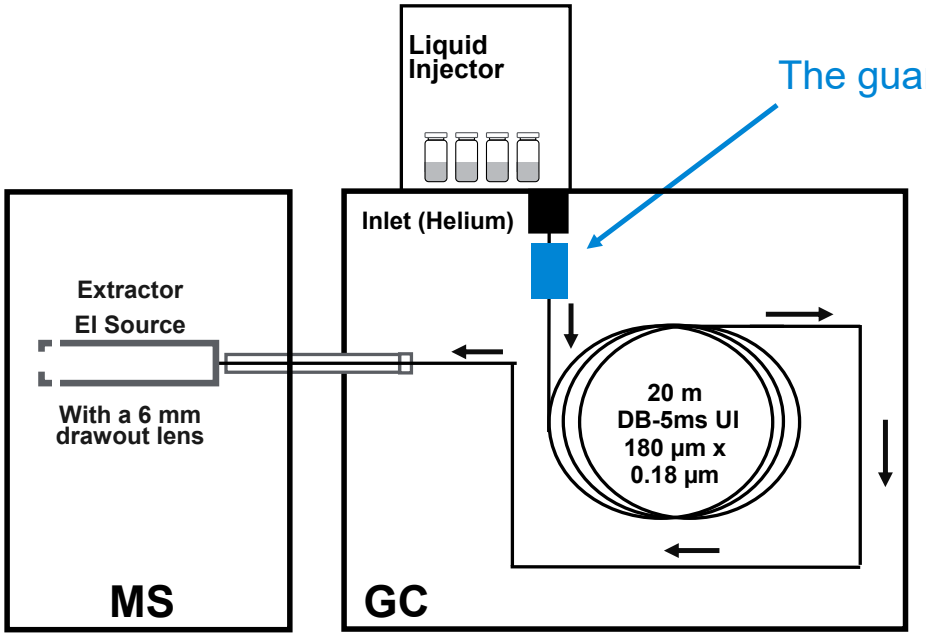
The guard chip (guard column/retention gap)

acts as sacrificial trap to prevent low volatility matrix from reaching the column

Oven ramp:
45 °C for 0.4 min
25 °C/min to 100 °C
32 °C/min to 270 °C
12.5 °C/min to 310 °C for 1.95

Benefits of Using the Guard Chip

- Use as retention gap with temperature programmability separate from that of the GC oven



The guard chip (guard column/retention gap)

Guard chip ramp:
200 °C for 0.4 min
25 °C/min to 100 °C
32 °C/min to 270 °C
12.5 °C/min to 310 °C

Oven ramp:
45 °C for 0.4 min
25 °C/min to 100 °C
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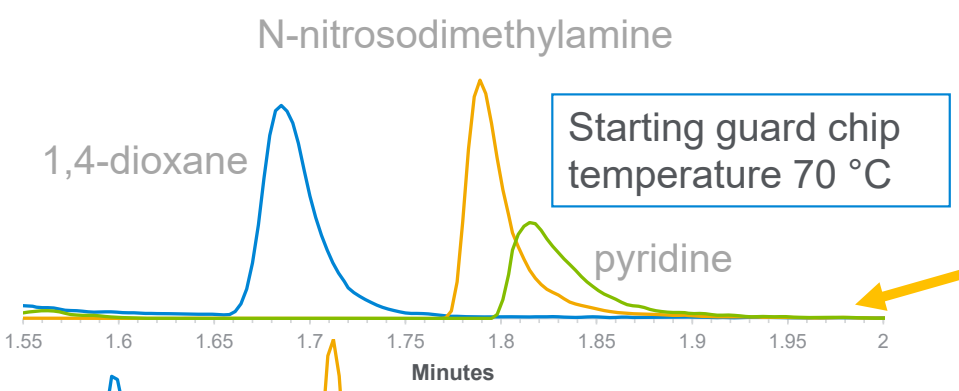
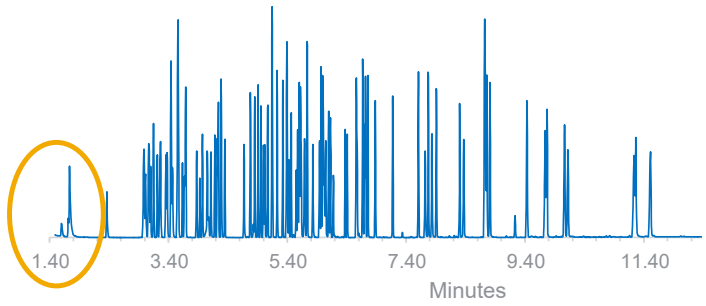
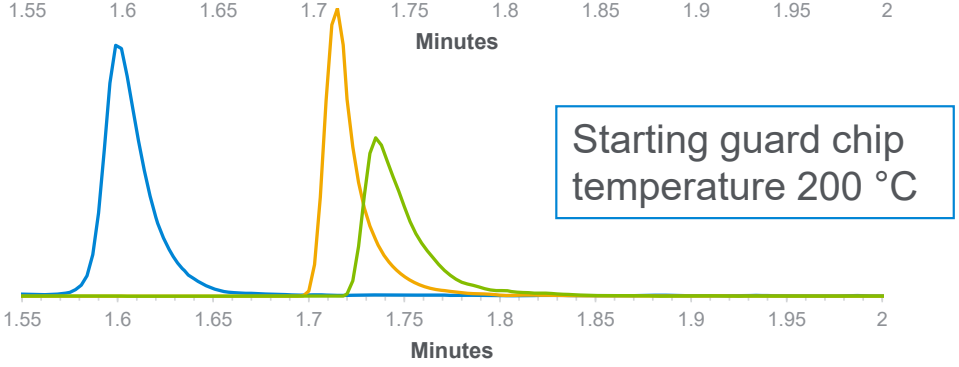
Benefits of Using the Guard Chip

Improved Peak Shape and LODs for the Early-Eluters

- Use as retention gap with temperature programming separate from that of the GC oven





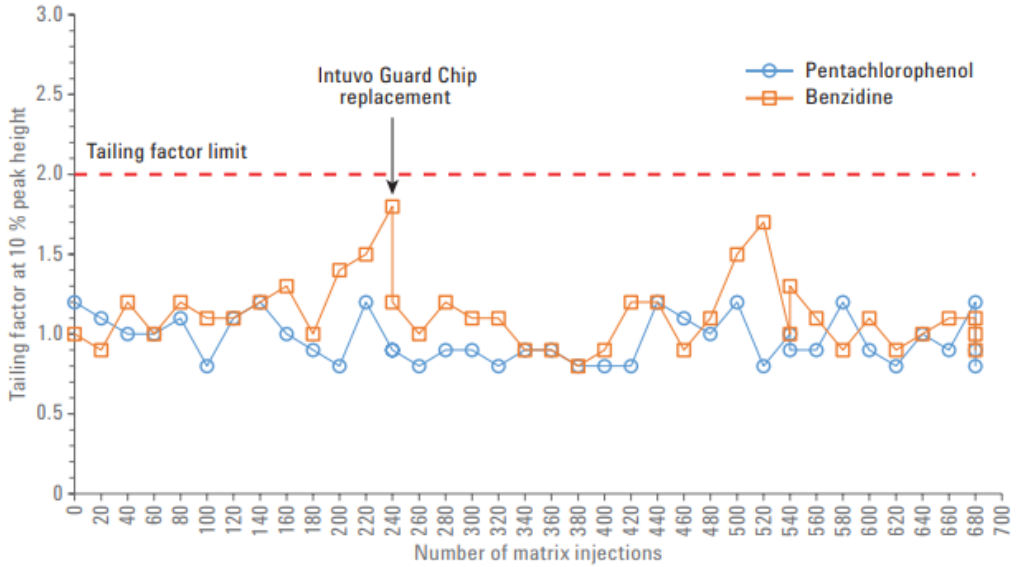


- Peak shape was improved by allowing the compounds to stay in vapor phase until they reach the column
- Reliable quantitation for these compounds down to 0.1 µg/mL

Benefits of Using the Guard Chip

Expedited Maintenance and Increased Uptime

- Use as retention gap with temperature programming
- Simplified maintenance – no column trimming needed
- Maintaining retention times with guard chip replacement



Dichloromethane soil extract

Giardina, M. Agilent Technologies, publication number 5991-7256EN, 2018.

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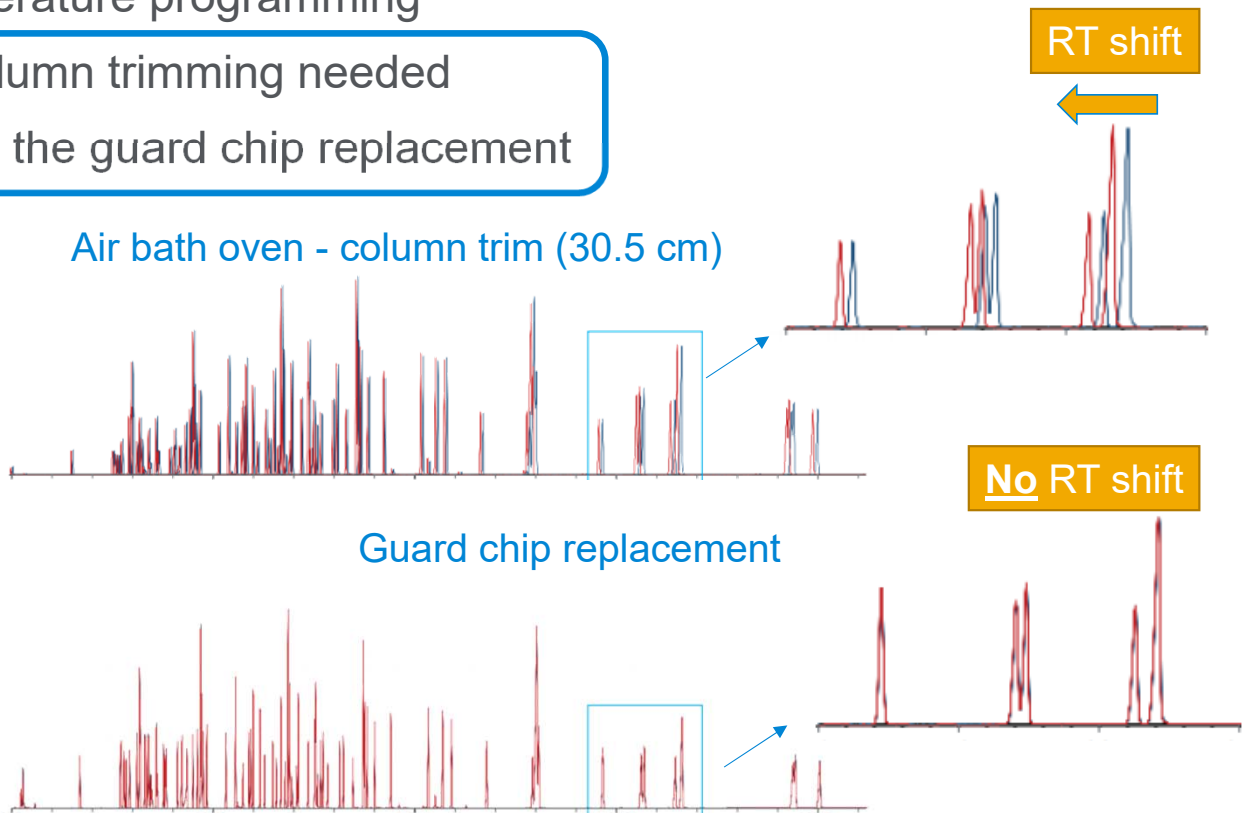

RT stability, hence, no need to:

- Update quantitation method
- Change SIM or MRM data acquisition method

Giardina, M. Agilent Technologies, publication number 5991-7256EN, 2018.

Conclusions

The direct heating GC/MS method was optimized for the separation of SVOCs for EPA 8270 in 12 minutes

The resolution requirements of the method were maintained, including for benzo [b and k] fluoranthene and for indenopyrene and dibenzoanthracene

The peak shape of the early-eluting targets was improved by temperature programming the microfluidic retention gap (the guard chip)

Replacement of the guard chip was more expedient than column trimming in terms of maintenance time, and did not require retention time re-adjustment

Acknowledgement

Mark Johnston, TestAmerica

Thank you!