Accelerated PAH screening using Intuvo GC with MS Detection

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Introduction

Polycyclic Aromatic Hydrocarbons (PAHs) are a group of contaminants that are commonly monitored in food and environmental samples.

PAHs originate from three sources: Petrogenic (from petroleum inputs), Pyrogenic (from combustion sources and Biogenic (from natural processes). This class of compounds is persistent, meaning they do not break down under outdoor conditions, and are known to bioaccumulate in living organisms.

Regional regulations exist, but vary greatly from country to country, including compound lists and detection thresholds..

Traditional airbath GC are often implemented in laboratory methods, with run times often in excess of one hour per sample in order to obtain optimal resolution of the various isomers.

In this work, we look at the possibility of a fast screening method that can be applied to adequately resolve PAH compounds using a multi-component standard internal standard correction.

Experimental

System Setup:

Agilent Intuvo 9000GC
Agilent 5977B Ext MSD w/ 9mm draw out lens
Agilent 7650A Automatic Liquid Sampler
Agilent Select PAH 15 m x 150 µm x 0.1 µm Column
Agilent UI Split Liner 5190-2295

Total Run Time: 6.08 minutes

Oven Ramp:

Ramp Speed	Temperature	Hold Time
	90°C	0.5min
200°C/min	230°C	0.5
250°C/min	285°C	1min
10°C/min	300°C	0min
250°C/min	340°C	1.5min

Inlet parameters:

Injection mode	Pulsed Split (60psi for 0.5min)
Split Ratio	30:1
Injection Volume	0.5uL
Inlet Temperature	350°C

Intuvo Specific:

Guard Chip	330°C
Bus Temperature	340°C
MSD Tail (Transfer Line)	330°C

Results and Discussion

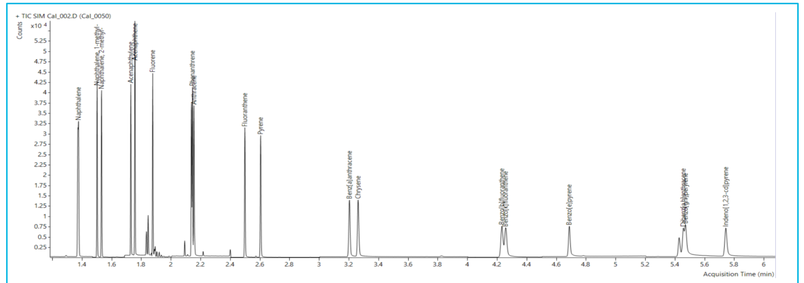


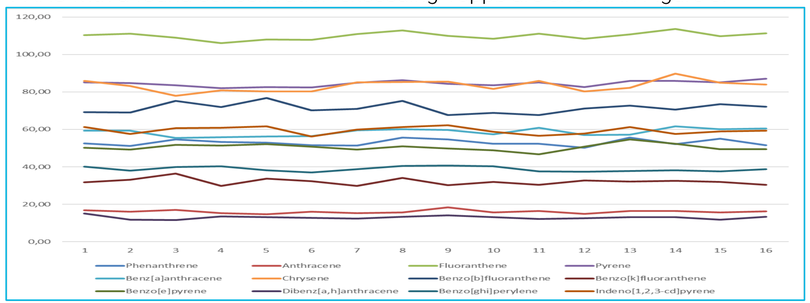
Figure 1-TIC SIM Chromatogram of a 10ppb standard in just over 6 minutes

A five point calibration curve, spanning from 10ppb to 1ppm was performed to prove the linearity of the method. Each calibration point was injected three times. These are the typical concentration ranges for Industrial Soil Analysis.

All compounds showed a linearity with an R^2 of at least 0.999 and accuracies between 94% and 104% for all concentrations.

Soil samples, extracted with hexane, were used to check the reproducibility of the method. One sample was split in to 4 vials and four replicates of each vial were analyzed.

PAHs with a found concentration exceeding 10ppb are shown in Figure 3.



Results and Discussion

On this SelectPAH column with a maximum column heating rate, ideal resolution of 1.0 was not achieved on some critical pairs, as shown below in Figure 4.

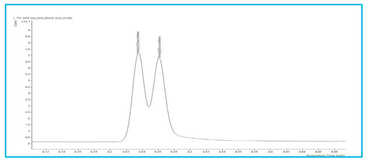


Figure 4: BbF/BkF Resolution of 0.8 (EU Calculation)

A less aggressive oven program and a DB-EUPAH 20m x 180µm x 0.14µm column increase the resolution of the above mentioned pair to 1.1 (from 0.8) using the EU resolution formula (fig 5). The run time is extendend to 10.2 minutes, which is still an impressive fast analysis time, with a run to run time of 13 minutes.

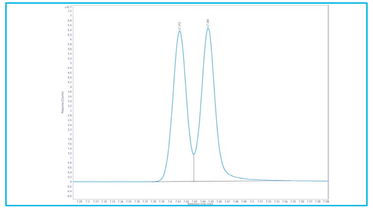


Figure 5 – Extracted ion 252 using a 20m column to achieve a 1.1 resolution for Benzo(b) and Benzo(k)fluoranthene.

Using H_2 as carrier gas, with the same configuration, gives a speed and resolution increase. The total run time drops to 8.4 minutes but the BbF/BkF resolution also drops to 0.96.

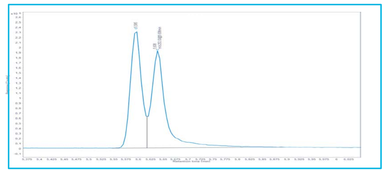


Figure 5 – Extracted ion 252 using H_2 as the carrier gas.

Conclusions

The direct heating within Intuvo GC combined with high efficiency columns yields a 6 minute PAH screening method. Shorter screening runs, while not optimized for complete resolution, can positively affect backlog as well as increase instrument efficiency and resource utilization. Improvements in resolution are easily attained with a simple column change and slighty less aggressive temperature program.

References

1. EU Priority PAH Analysis in Pumpkin Seed Oil Using Bond Elut EMR—Lipid Cleanup by GC/MS/MS – Agilent app note 5994-0593EN
2. PAH Analysis in Fish by GC/MS using Agilent Bond Elut QuEChERS dSPE Sample Preparation and a High Efficiency DB-5ms Ultra Inert GC Column – Agilent app note 5990-6668EN