Polycyclic Aromatic Hydrocarbon (PAH) Evaluation in Fatty Food Matrix using Gas Chromatography Triple Quadrupole Mass Spectrometry (GC-MS/MS)

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Introduction

Polycyclic Aromatic Hydrocarbons (PAHs)

- Food contaminants that originate from preparation processes (high temperature grilling of fatty matrix)
- Persistent and bioaccumulate in the environment
- Highly monitored by EU and US Regulatory Agencies
- Exposure is associated with health concerns

Sample Preparation

- Enhanced Matrix Removal-Lipid (EMR-Lipid) is a sorbent material that removes major lipid classes from sample extract to detect the analyte of interest
- High lipids in food cause interference, matrix effects, and accumulate in the analytical flow path

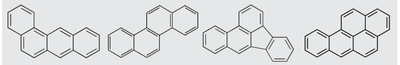
GC EI-MS/MS Analysis

- DB-EUPAH column separates isomeric PAHs
- Self-Cleaning Ion Source (SCIS) prevents PAH deposition in the source with Hydrogen
- Backflush (BF) maintains column lifetime by removing heavy matrix interference between sample injections

Poster Evaluates

- Recoveries and Deviation of 1 ppb PAH in food
- EMR-Lipid as sample cleanup
- GC-MS/MS analysis

EU Commission 1881/2006 Monitors Four PAHs[1]



Benz[a]anthracene	Chrysene	Benzo[b]fluoranthene	Benzo[a]pyrene
MW: 228	MW 228	MW 252	MW 252
228→228 CE 50	228→228 CE 50	252→252 CE 50	252→252 CE 50
228→226 CE 50	228→226 CE 50	252→250 CE 50	252→250 CE 50

EU Commission Maximum Levels (µg/kg)[1]

Points	Benzo[a]pyrene	Sum of Four PAH
Oil and Fats (6.1.1)	2	10
Cocoa Bean (6.1.2)	2	30
Smoked Meat (6.1.4)	2	12
Smoked Bivalve Mollusks (6.1.7)	2	12

Enhanced Matrix Removal – Lipid

Extraction

PreSpike (QC)
Vortex

Add
Acetonitrile
Homogenizers

Vortex
Centrifuge

EMR-Lipid

Add water to
EMR-Lipid dSPE
Vortex

Add Supernatant
Vortex
Centrifuge

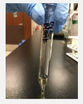
Decant to
EMR-Lipid Polish
Vortex
Centrifuge

PSA/C18 EC

Vortex
Centrifuge

Concentration

Post-spike (CAL)



Dry with N_2
Reconstitute
Sonicate
Centrifuge

Consumables	
EMR-Lipid dSPE	1g in 15-mL tube (p/n 5982-1010)
EMR-Lipid Polish	NaCl/anhydrous $MgSO_4$; 2g in 15mL tube (p/n 5982-0101)
PSA/C18EC dSPE EN method	25mg PSA, 25mg C18, 150mg MgSO4, in 2mL tube (p/n 5982-5121)

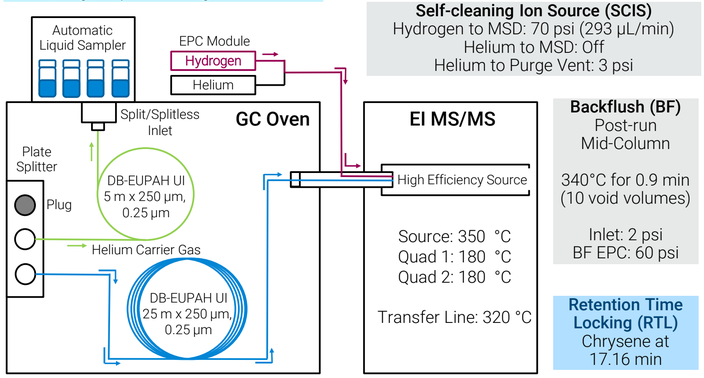
GC-EI-MS/MS Parameters

7890 GC Coupled with 7010 Triple Quad MSD

Inlet Parameters
0.5-2.0 µL injection
320 °C inlet temperature
Pulsed Splitless at 20 psi for 0.9 min
4-mm, single-tapered with glass wool Liner

GC Oven Parameters
Initial: 60 °C (Hold 1 min)
Rate 1: Ramp at 25 °C/min to 200 °C
Rate 2: Ramp at 8 °C/min to 335 °C (Hold 11 min)

Self-cleaning Ion Source (SCIS)
Hydrogen to MSD: 70 psi (293 µL/min)
Helium to MSD: Off
Helium to Purge Vent: 3 psi



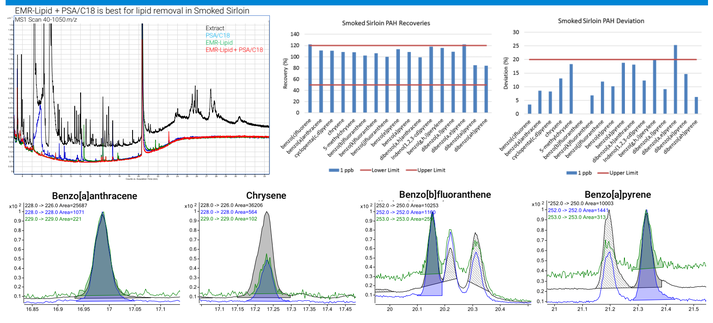
Backflush (BF)
Post-run
Mid-Column

340°C for 0.9 min
(10 void volumes)

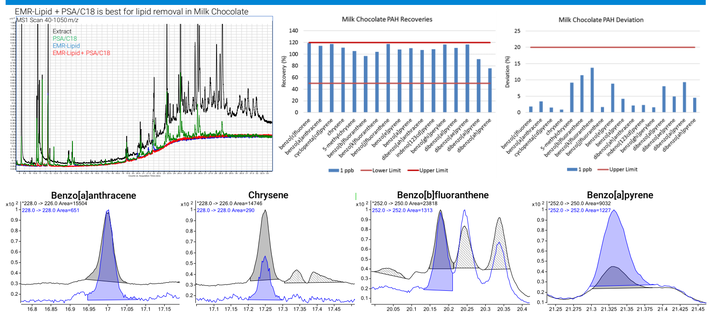
Inlet: 2 psi
BF EPC: 60 psi

Retention Time Locking (RTL)
Chrysene at 17.16 min

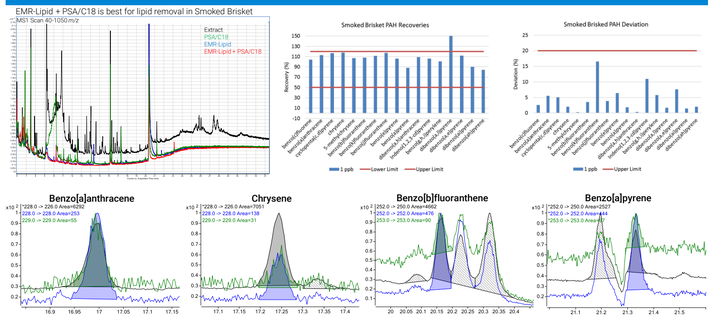
Smoked Sirloin



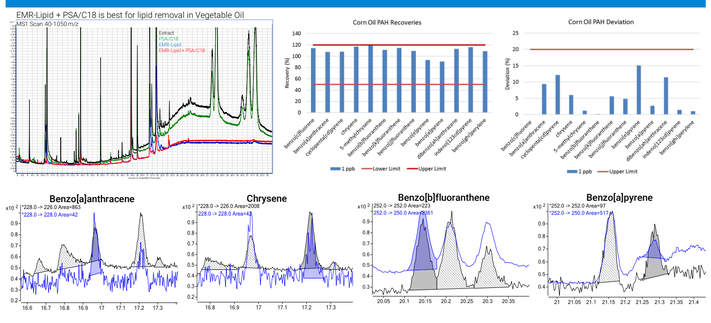
Milk Chocolate



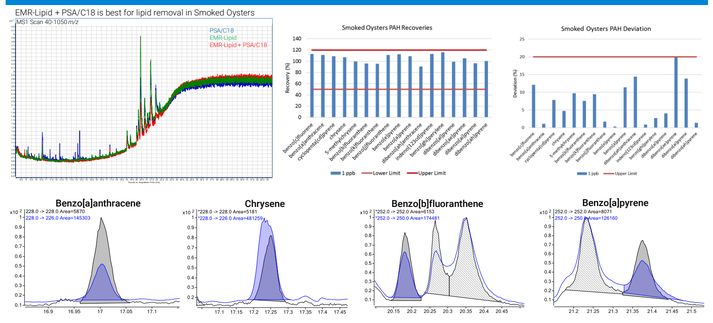
Smoked Brisket



Vegetable Oil



Smoked Oysters



Conclusions

For recovery of 1 ppb PAH in fatty food matrices:

- EMR-Lipid with PSA/C18 sample preparation
- GC-MS/MS with Self-Cleaning Ion Source (SCIS) and Backflush (BF)
- Source temperature 320-350 °C and both Quadrupoles at 150-180 °C
- Single-tapered with wool ultra-inert liner with Pulsed Splitless injection
- DB-EUPAH UI column for separation of isomeric and coeluting PAH
- Collision energy at 50 eV to destroy matrix interference

References

[1]Legislation on Polycyclic Aromatic Hydrocarbons (PAHs) - EU Science Hub - European Commission. *EU Science Hub*, November 17, 2015. https://ec.europa.eu/jrc/en/eurl/pahs/legislation.

[2]Anderson, K.A., Szelewski, M.J., Wilson, G., Quimby, B.D., Hoffman, P.D., 2015. Modified ion source triple quadrupole mass spectrometer gas chromatograph for polycyclic aromatic hydrocarbon analyses. J. Chromatogr. A 1419, 89–98.

[3]Optimized PAH Analysis using the Agilent Self-Cleaning Ion Source and the Enhanced PAH Analyzer. Agilent Application Note 5991-3003EN

[4]PAH analysis in Salmon with Enhanced Matrix Removal. Agilent Application Note 5991-6088EN