Polycyclic Aromatic Hydrocarbon (PAH) evaluation in complex food matrix using Triple Quadrupole Gas Chromatography Mass Spectrometry (GC EI-MS/MS)

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Introduction

Polycyclic Aromatic Hydrocarbons (PAHs)

- Food contaminants consisting of fused aromatic rings with shared carbons that originate from preparation processes such as high temperature grilling of fatty matrix
- Persistent and bioaccumulate in the environment
- Highly monitored by the EU and US regulatory agencies as exposure is associated with health concerns

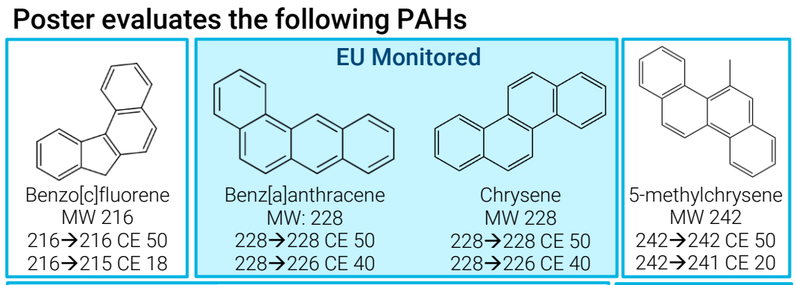
Sample Preparation for PAH Analysis in Fatty Foods

- Enhanced Matrix Removal-Lipid (EMR-Lipid) is a sorbent material that selectively removes major lipid classes from sample extract without removing the analyte of interest
- High lipid in food causes interference, matrix effects, and accumulate in the analytical flow path

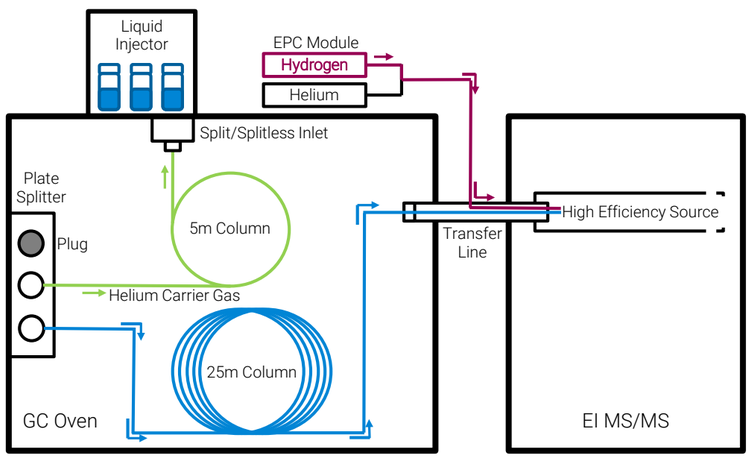
GC EI-MS/MS Analysis of PAHs

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

Poster evaluates the following PAHs



GC-EI-MS/MS Configuration

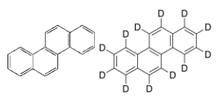


GC and MSD Parameters	
GC EI-MS/MS	7890B GC Coupled with 7010 Triple Quad MSD
Inlet Mode	Pulsed Splitless at 20 psi for 0.9min at 320 °C
Injection Volume	0.5 to 2 µL
Injection Liner	4-mm single tapered with glass wool, ultra inert (p/n 5190-2293)
Oven	60 °C (1 min), 25 °C/min to 200 °C, 8 °C/min to 335 °C (11 min)
Column	DB-EUPAH UI (p/n 122-9632), 30m x 250 µm, 0.25 µm
Column Setup	Inlet to Backflush EPC: 5m x 250 µm, 0.25 µm at 1.2 mL/min Backflush EPC to MSD: 25m x 250 µm, 0.25 µm at 1.5mL/min
BF (post-run)	Oven: 340 °C for 0.9min (10 void volumes) Inlet: 2 psi, Backflush EPC: 60 psi
SCIS	70 psi hydrogen (293 µL/min) to MSD, Helium off 3 psi Helium to Purge vent
RTL	System retention time locked to chrysene at 17.16 min
MSD	Transfer line 320 °C, Source 320-350 °C, Quads 150-180°C

Enhanced Matrix Removal – Lipid

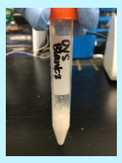
Extraction

PreSpike (QC)
Vortex

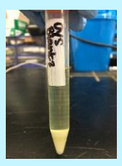


Add Acetonitrile
Add homogenizers
Vortex
Centrifuge

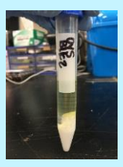
EMR-Lipid



Add water to EMR-Lipid dSPE
Vortex



Add supernatant
Vortex
Centrifuge

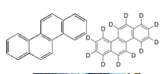


Decant to Polish tube
Vortex
Centrifuge

Extra Cleanup

Transfer to PSA/C18 EC dSPE
Vortex
Centrifuge

Concentration



Post-spike (matrix matched calibration)
Vortex
Centrifuge
Reconstitute
Sonicate
Centrifuge

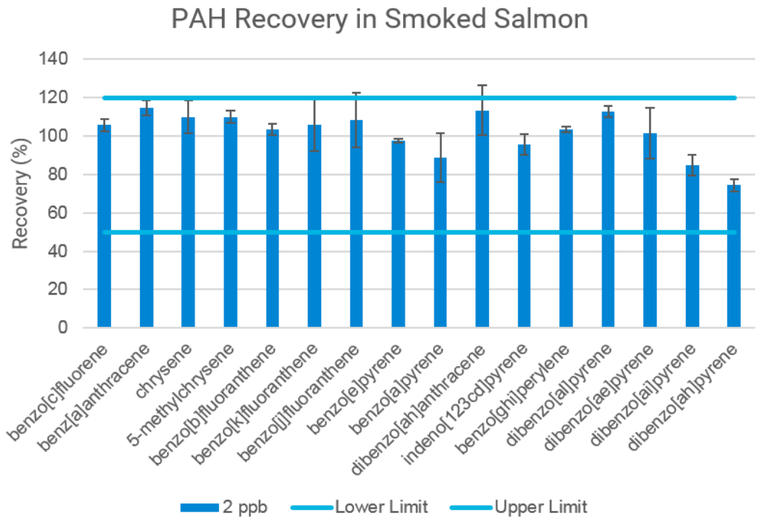
GC EI-MS/MS

≥5ppb
Except dibenzopyrenes

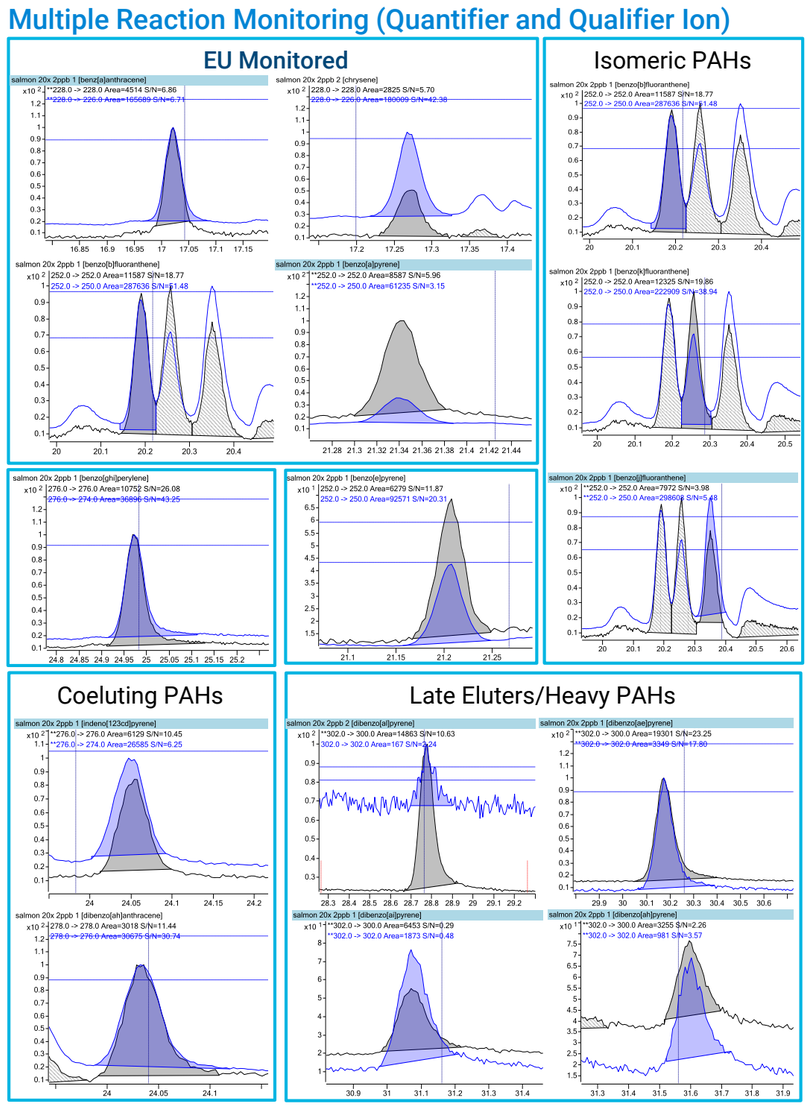
<5ppb
dibenzopyrenes

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)

Smoked Salmon



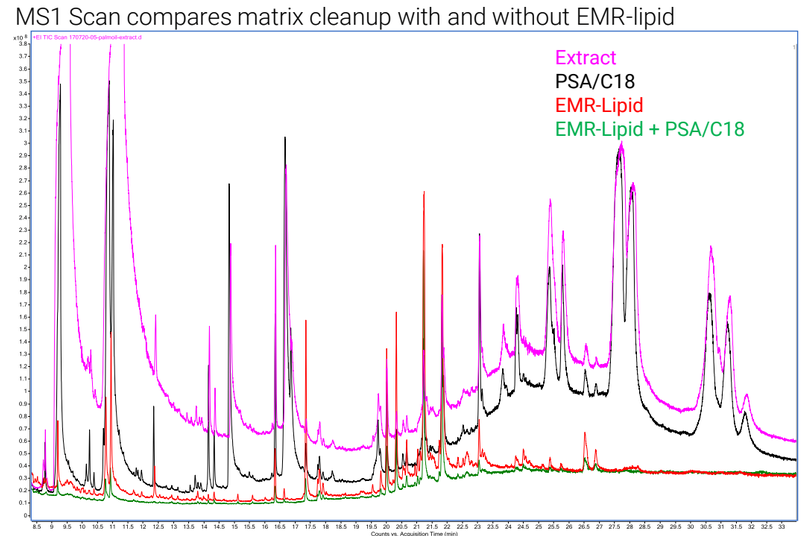
Multiple Reaction Monitoring (Quantifier and Qualifier Ion)



Matrix Cleanup

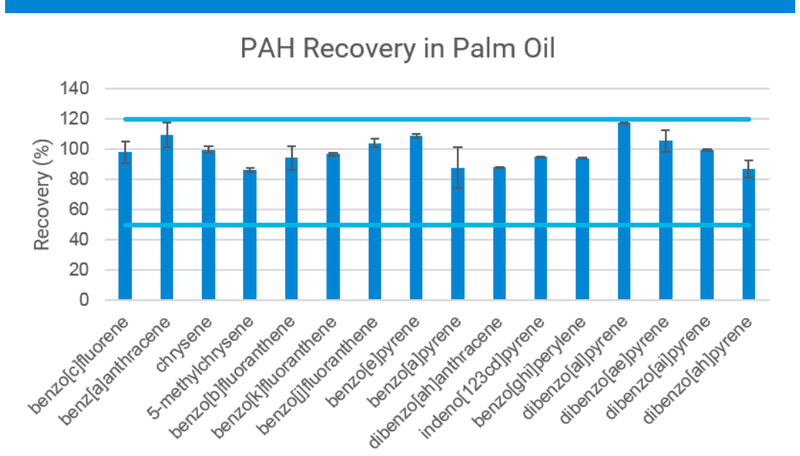
EMR-Lipid combined with PSA/C18 dSPE removes lipid and matrix interferences

MS1 Scan compares matrix cleanup with and without EMR-lipid



MS1 scan range 40-1050 m/z

Palm Oil



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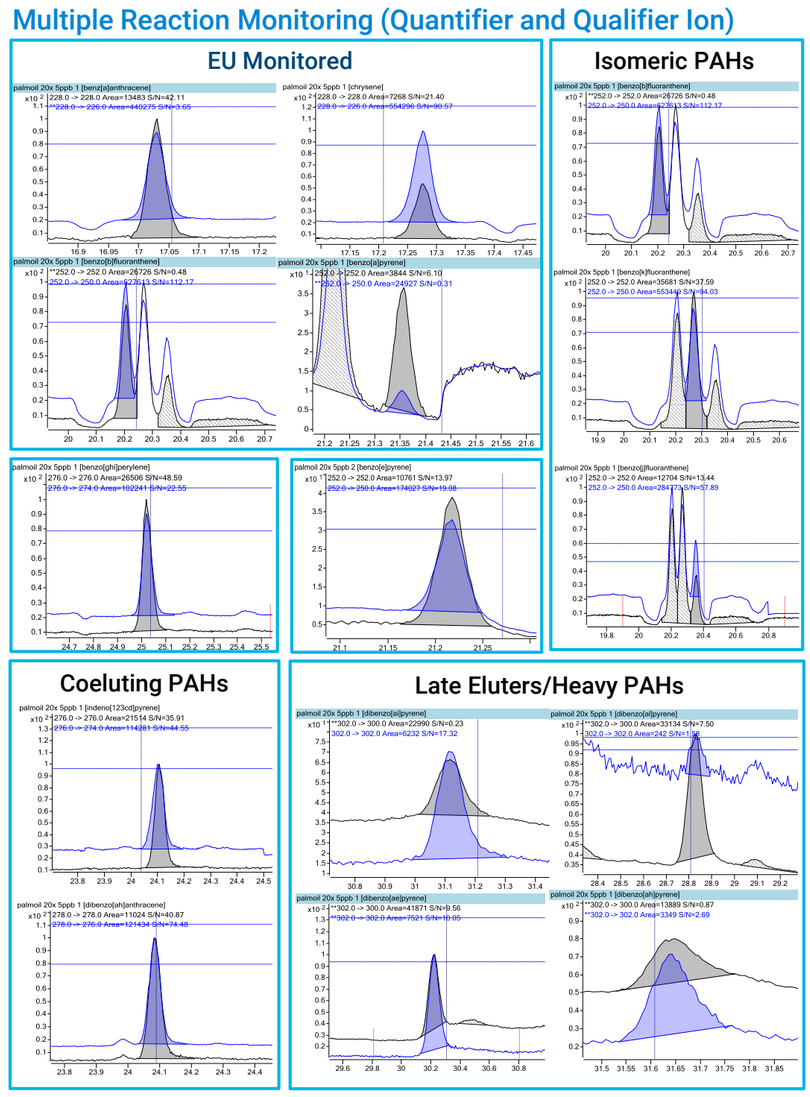

Conclusions

Optimum procedure for ≤5ppb PAH detection in fatty foods

- EMR-Lipid combined with PSA/C18 dSPE to removed lipid and matrix interference in fatty matrix
- GC EI-MS/MS consisting of self-cleaning ion source, post-run mid-column backflush, inlet and source at 320°C, DB-EUPAH column, and pulsed splitless injection with single-tapered wool liner
- High collision energy of 50 eV to destroy matrix interference

References

- 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.
- PAH analysis in Salmon with Enhanced Matrix Removal. Application Note 5991-6088EN
- Optimized PAH Analysis using the Agilent Self-Cleaning Ion Source and the Enhanced PAH Analyzer. Application Note 5991-3003EN