Agilent Solutions for Analyzing Polycyclic Aromatic Hydrocarbons in Seafood

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PAH analytical method summary

- **NOAA** NMFS-NWFSC-59, 2004 **GPC + GC/MS** is the current method specified for PAH analysis in seafood. It is a complex and time-consuming method so many customers are looking for an alternative.

- **QuEChERS + GC/MS** or **GC/MS/MS PAH analyzers** are the proposed solutions from Agilent to simplify sample preparation, reduce cycle time, and provide increased sensitivity. Backflush, Multi Mode Inlet, and MS/MS are the technologies contributing to the performance and sample throughput.

- **QuEChERS + HPLC/Fluorescence** is an excellent screening tool. This Agilent solution was chosen by the US Food Emergency Response Network.
Why QuEChERS for Sample Prep?

When Compared to traditional sample prep methods:

- 25-50%+ time savings
- Reduced solvent usage: 10-15 mL/sample
- No chlorinated solvents required
- Extract multiple families of compounds with one extraction method
- Does not require advanced sample preparation experience
QuEChERS Seafood Extraction Method:

**Step 1: Extraction**

- Finfish, shellfish
- Weigh 3 g into 50 mL tube, add 2 ceramic homogenizers
- Add 12 mL of water, vortex 30 sec
- Vertically shake for 1 min, centrifuge at 4000 rpm for 5 min

**Step 2: d-SPE (dispersive-SPE)**

- Transfer 8 mL of extract from Step 1 to d-SPE (fatty sample)
- Vortex 1 min
- Centrifuge 400 rpm, 5 min
- Aspirate extract, filter through 0.45 um Nylon filter, transfer to GC or HPLC Vial

**Step 3: Analyze**

- GC/MS or GC/QQQ
- LC/UV/FLD or LC/QQQ
  (require dilution with water 1:4 or 1:5 prior to LC)
QuEChERS: PAH Determination in Fish

Amenable to both GC and LC, UV/FLD and MS detection

Note: Solvent exchange is not required for GC analysis
PAH Analysis: GC/MS with Column Backflush

-- Improved reliability and speed

Oven Program
50 °C for 0.8 min
then 70 °C/min to 180 °C for 0 min; then 7 °C/min to 230 °C for 1 min
then 40 °C/min to 280 °C for 1 min; then 25 °C/min to 335 °C for 3 min

Run Time 18.25 min

MM Inlet
Mode Pulsed Splitless  Temperature: 320 °C

Column DB-EUPAH, 20 m x 180 μm x 0.14 μm

Column Flow constant flow at 1 mL/min (pressure = 25.885 psi)

MSD Transfer line: 320 °C  MS Source: 350 °C  MS Quad: 200 °C

Internal Std
1 Naphthalene-d8  4 2-Methylnaphthalene  15 Phenanthrene
2 1-methylnaphthalene  5 Biphenyl  16 Anthracene
3 2-Methylnaphthalene  6 Phenylmethylnaphthalene  17 1-methylphenanthrene
4 2-Methylnaphthalene  7 HMB  18 Fluoranthene
5 Biphenyl  8 Acenaphthylene  19 Pyrene
6 Phenylmethylnaphthalene  9 Acenaphthene  20 Benz[a]anthracene
7 HMB  10 Acenaphthene  21 Benz[a]anthracene
8 Acenaphthylene  11 2,3,5-trimethylnaphthalene  22 Triphenylene
9 Acenaphthe  12 Fluorene  23 Chrysene
13 Dibenzothiophene  14 Phenanthrene-d10  24 Benzo[b]fluoranthene
14 Phenanthrene-d10  15 Phenanthrene  25 Benzo[k]fluoranthene
16 Anthracene  17 1-methylphenanthrene  26 Triphenylene
18 Fluoranthene  19 Pyrene  27 Dibenz[b,k]fluoranthene
20 Pyrene  21 Benz[a]anthracene  28 Benzo[b]fluoranthene
22 Triphenylene  23 Chrysene  29 Benzo[a]pyrene
24 Benzo[b]fluoranthene  25 Benzo[k]fluoranthene  30 Perylene
26 Triphenylene  27 Dibenz[b,k]fluoranthene  31 Dibenz[a,h]anthracene
28 Benzo[b]fluoranthene  29 Benzo[a]pyrene  32 Dibenz[a,h]anthracene
30 Perylene  31 Dibenz[a,h]anthracene  33 Indeno[1,2,3-cd]pyrene
32 Dibenz[a,h]anthracene  33 Indeno[1,2,3-cd]pyrene  34 Benzo[ghi]perylene
Low detection limit with GC/MS/MS

NIST Certified Reference Material  1974b Mussel sample
Ready to use PAH Analyzers configured and tested at the factory for fast lab start-up

At Factory

- Retention Time Locking (RTL)
- Capillary Flow Device for Backflushing
- Dynamic MRM
- Sample Prep. QuEChERS
- MRM method for PAHs
- PAH optimized column

At User’s Lab

Plug and Play

Ready for the 1st run

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GC/MS/MS Analyzers
Solutions from Agilent for PAH Analysis in Seafood

-- for higher productivity, better performance on Day ONE

- QuEChERS: *simple and fast* extraction and sample clean-up
- GC/MS with Backflush: using innovative Capillary Flow Technology *shortens GC cycle time* while keeping column and source cleaner longer!
- GC/MS/MS with Backflush: highest *sensitivity* for PAH analysis.
- PAH Optimized Column: *optimized PAH separation* in shortest cycle time
- Factory setup and tested analyzers: Reduces method development time!
- HPLC Fluorescence - sensitive quick screening method