Extraction of Polycyclic Aromatic Hydrocarbons from Fish Using the QuEChERS Approach

UCT Part Numbers

ECMSSC-MP
4000 mg mag. Sulfate, 1000 mg sodium chloride

ECMPSC1815CT
900 mg $MgSO_4$, 300 mg PSA and 150 mg endcapped C18

ECPAHFR50CT
ENVIRO-CLEAN® PAH centrifuge tubes

Procedure:

1. Extraction: Protect samples from exposure to light to avoid degradation

a) 5 grams of homogenized fish are transferred to a 50 mL centrifuge tube.
b) Add 10 mL of acetonitrile.
c) Mix by shaking.
d) Add the contents of pouch containing 4 grams $MgSO_4$ and 1 gram of NaCl to the centrifuge tube.
e) Immediately vortex the mixture for 3 minutes.
f) Centrifuge samples for 3 minutes at 3400 rpm.
g) Recover the clear supernatant for cleanup.

2. Clean-up, Dispersive Solid-phase (dSPE)

a) Use 3 mL of the supernatant for clean-up.
b) Add supernatant to centrifuge tube **ECMPSC1815CT**.
c) Shake for 1 minute.
d) Centrifuge for 1 minute at 3400 rpm.
e) Filter supernatant through 0.20 µm PTFE membrane filter
f) Samples are ready for analysis.

3. Elution

a) Separation of the compounds is performed in a C18 column (CC 150/4 Nucleosil 100-5 C18 PAH, 150 x 4.0 mm; 5 µm particle size; Macherey-Nagel, Duren, Germany) or equivalent maintained at room temperature.
b) Inject 15 µL.
c) The initial composition of the mobile phase is 50% of ACN and 50% water.
d) A linear gradient to 100% is programmed in 15 minutes.
e) Final hold 13 minutes.
f) Initial conditions are achieved within 1 minute and maintained for 6 min before next run.
g) Total run time is 40 minutes.
h) Flow rate of 0.8 mL/minute.
i) Fluorescence wavelength program:

Each compound is detected at its optimum excitation/emission wavelength pair:
315/260 nm naphthalene, acenaphthene and fluorene
366/260 nm Phenanthrene
430/260 nm anthracene, fluoranthene, pyrene, benz(a) anthracene, chrysene, benzo(b)fluoranthene, benzo(k) fluoranthene, benzo(a)pyrene, dibenz(a,h)anthracene, benzo(g,h,i)perylene and dibenzo(a,l)pyrene
505/290 nm (indeno(1,2,3-cd)pyrene)

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References:

[1] *Adapted from, Ramalhosa, Maria Joao et al, "Analysis of polycyclic aromatic hydrocarbons in fish: evaluation of a quick, easy, cheap, effective, rugged, and safe extraction method", J. Sep. Sci. 2009, 32, 3529 – 3538

DCN-117090-116

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