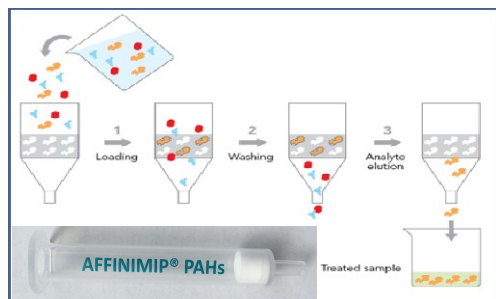


Selective Solid Phase Extraction for PAHs Analysis from olive oil using AFFINIMIP®SPE



Polycyclic aromatic hydrocarbons: a major concern for human health and a challenge in food safety analysis

Polycyclic aromatic hydrocarbons (PAHs) are a large group of organic compounds with two or more fused aromatic rings and are known to be cancer causing agents. Human beings are exposed to PAHs mostly by intake of food. As these compounds are highly soluble in lipophilic compounds, edible oils can be an important source of contamination by PAHs.

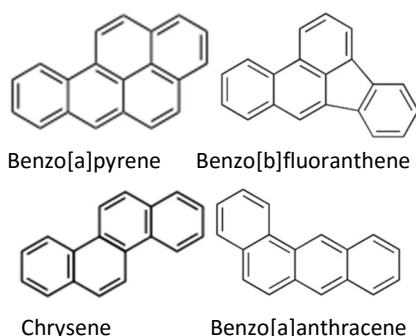


Figure 1. Chemical structure of regulated PAHs

In 2011, EU Commission Regulation No 835/2011, amending Regulation 1881/2006, set maximum levels in edible oils to 2 ng/g of benzo[a]pyrene individually, and 10 ng/g of benzo[a]pyrene, benzo[b]fluoranthene, chrysene and benzo[a]anthracene combined.

How to solve this?

AFFINISEP has developed a new class of intelligent polymers based on molecularly imprinted polymers (AFFINIMIP®). AFFINIMIP® SPE PAHs cartridge is a simple, fast, sensitive and selective tool for the extraction of PAHs (including benzo[a]pyrene, benzo[b]fluoranthene, chrysene and benzo[a]anthracene) from fatty foods such as oil.

In this application note, we demonstrate a reliable

quantification of these PAHs from olive oil using AFFINIMIP® SPE PAHs with an LC-Fluo analysis suitable for routine analysis.

We obtained good recovery yields with a fluorescence detection proving the efficiency of AFFINIMIP® SPE PAHs cleanup.

Good recoveries in olive oil

Molecules	C° ng/mL	R%
Benz[a]anthracene	9	98
Benzo[a]pyrene	12	95
Chrysene	12.5	105
Benzo[b]fluoranthene	10	80

Table 1. Recovery of main PAHs after AFFINIMIP® SPE PAHs clean-up of olive oil spiked at concentration C°

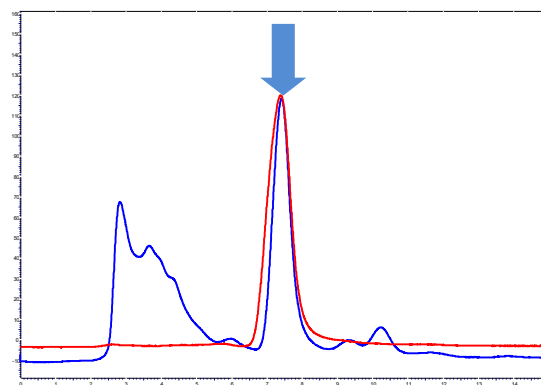


Figure 2. Fluo Chromatograms obtained after clean-up with AFFINIMIP® SPE PAHs of olive oil spiked at 9ng/mL of Benzo[a]anthracene (blue) and standard solution (red)

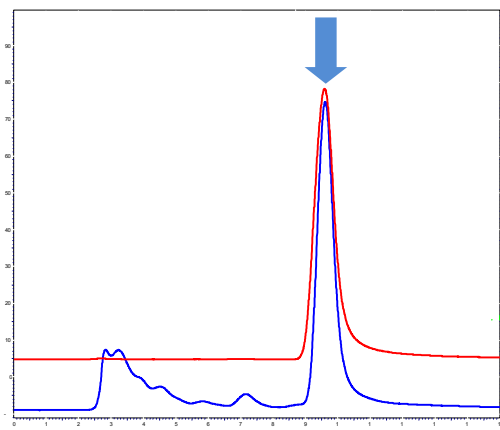


Figure 3. Fluo Chromatograms obtained after clean-up with AFFINIMIP® SPE PAHs of olive oil spiked with 12ng/mL of Benzo[a]pyrene (blue) and standard solution (red). Dilution of oil by 5 with cyclohexane.

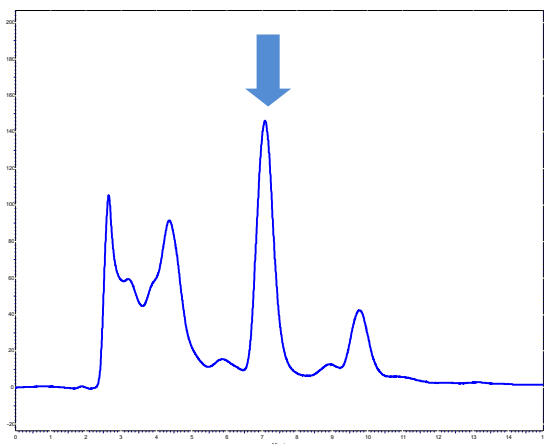


Figure 4. Fluo Chromatograms obtained after clean-up with AFFINIMIP® SPE PAHs of olive oil spiked with 12.5ng/mL of Chrysene (blue). Dilution of oil by 5 with cyclohexane.

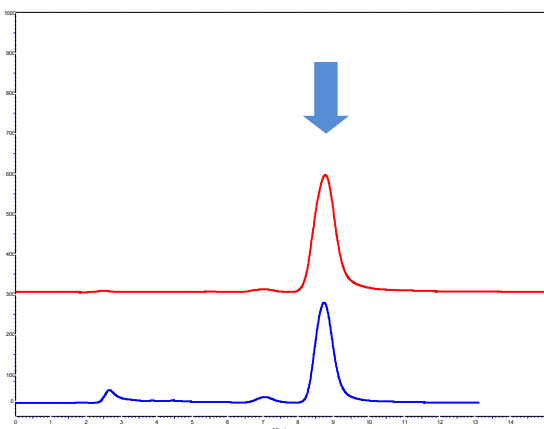


Figure 5. Fluo Chromatograms obtained after clean-up with AFFINIMIP® SPE PAHs of olive oil spiked with 10ng/mL of Benzo[b]fluoranthene (blue) and standard solution (red). Dilution of oil by 5 with cyclohexane.

Experimental conditions

Materials

All reagents and chemicals were ACS grade quality or better. Olive oil was purchased at a supermarket.

Solid phase extraction (SPE) protocol

The SPE procedure used a 3mL AFFINIMIP® SPE PAHs cartridge or 3mL cartridge. The details of each step are as follow:

- No equilibration step
- Load 2mL of the olive oil diluted by 5 or 10 with cyclohexane (0.5 drop/s)
- Wash the cartridge with 2 x3 mL of cyclohexane (1 drop/s)
- Elute PAHs with 3mL Ethyl acetate/ cyclohexane 15-85 (1 drop/s) (alternatively, a higher ratio of ethyl acetate can be used for a higher eluting solution)

The SPE procedure lasted less than 30 minutes. The elution fraction was then evaporated and dissolved in the mobile phase.

Analysis

HPLC was performed on a ThermoFinnigan Spectra System with a Thermo Hypersil Gold column (150mm x 2.1mm; 3µm). Mobile phase at 75 ACN 25 H₂O with a flow rate of 0.2mL/min.

The detection system was a Fluorimeter Jasco FP2020 plus (λ_{ex}/em 290/430nm). The injection volume was 20µL.

Product reference

• AFFINIMIP® SPE PAHs

Catalog number: FS119-02 for 25 cartridges - 3mL
FS119-03 for 50 cartridges - 3mL

SPE for Polycyclic Aromatic Hydrocarbons (PAHs)

AFFINIMIP® SPE PAHs	AFFINIMIP® SPE Phenolics	AttractSPE™ HLB	SilactSPE™ CN/SiOH
For the cleanup of PAHs in FATTY food and liquid such as oil	For the cleanup of Hydroxylated PAHs	For the cleanup of PAHs in WATER	For the cleanup of PAHs in SOIL
Molecularly imprinted polymer for PAHs	Molecularly imprinted polymer for Phenolics	HLB	A two layer sorbents with cyano modified silica and silica sorbents

Product	Vol	25 cartridges/box	50 cartridges/box
AFFINIMIP® SPE PAHs	3mL	FS119-02	FS119-03
AFFINIMIP® SPE Phenolics	3mL	FS103-02	FS103-03
AttractSPE™ HLB	6mL	HLB-25.S.6.200	HLB-50.S.6.200
SilactSPE™ CN/SiOH	3mL	CNSiOH-25.S.3.500.1g	CNSiOH-50.S.3.500.1g
	6mL	CNSiOH-25.S.6.500.1g	CNSiOH-50.S.6.500.1g
	6mL glass	CNSiOH-25.G.6.500.1g	CNSiOH-50.G.6.500.1g

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