

Analysis of PAHs in olive, canola, and CBD oils by a simple and fast cleanup method based on Molecularly Imprinted Polymers

Sami Bayoudh¹, Michel Arotçarena¹, Corentin Germain¹, Bruno Veyrand², Rémi Roger², Emmanuelle Bichon¹, Kaynoush Naraghi¹

¹Affinisep, Le Houlme, France (contact@affinisep.com)
²National reference laboratory LABERCA Oniris, Nantes, France

Polycyclic Aromatic Hydrocarbons (PAHs) are likely to be found in edible oils. Solid Phase Extraction (SPE) is an efficient strategy for sample preparation of challenging matrices as it helps to reach certain limits of detection and preserve the integrity of the analytical devices.

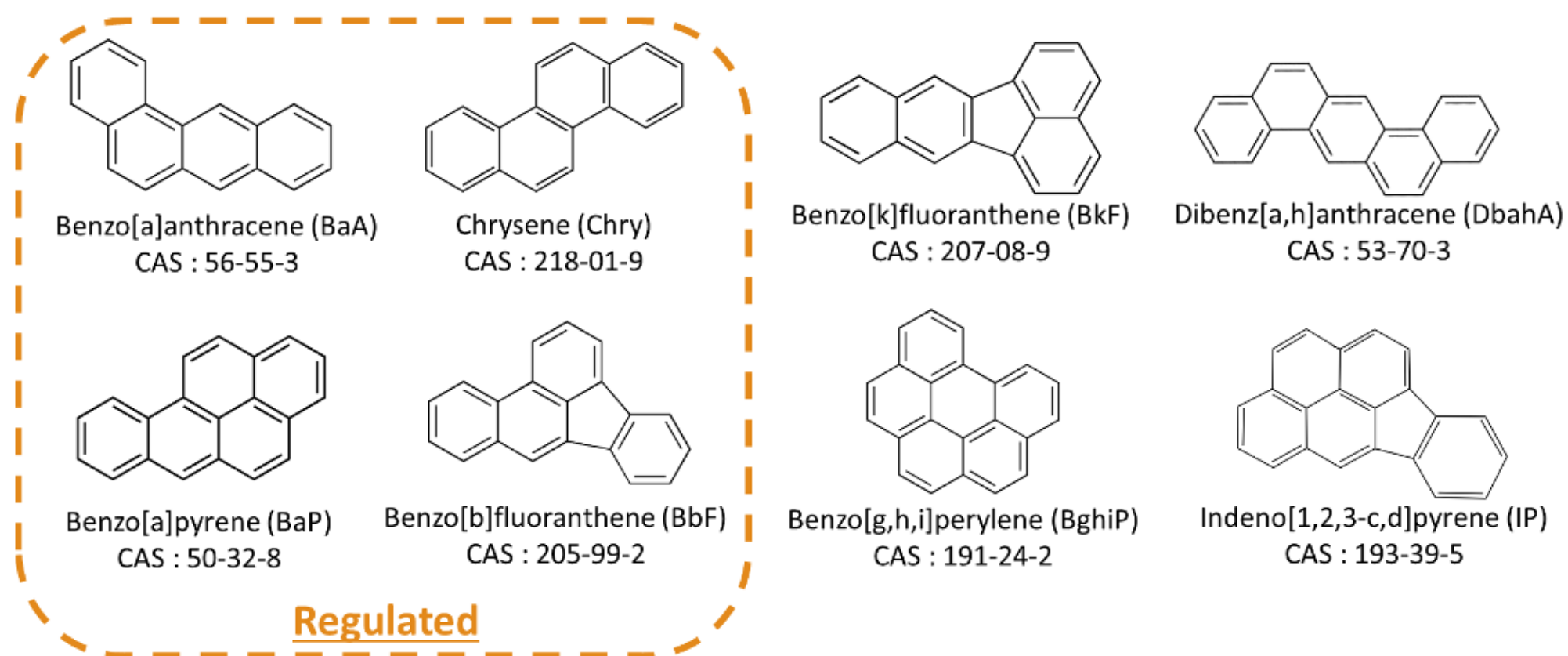
This poster describes a simple and fast protocol for the extraction and cleanup of 8 PAHs in canola and olive oils using **AFFINIMIP® SPE PAHs - 3mL**. It also describes the cleanup and analysis of 7 PAHs in CBD oil, a more challenging matrix than conventional edible oils.



Extraction and cleanup of PAHs in oils using AFFINIMIP® SPE PAHs

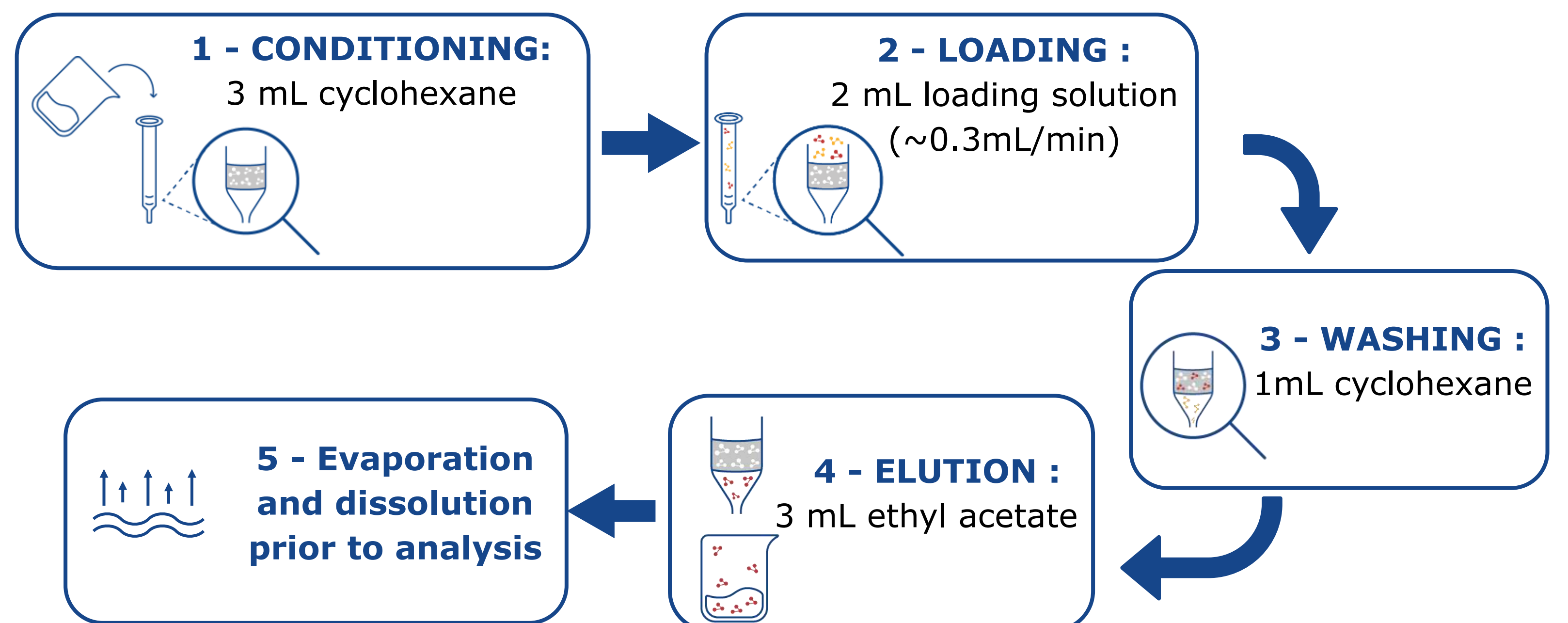
① What is AFFINIMIP® SPE PAHs

AFFINIMIP® SPE is based on Molecularly Imprinted Polymers (MIP) technology. MIPs are affinity columns made with very stable polymers. They are intended to target a specific group of molecules. During the synthesis, a template molecule with a structure close to the analytes of interest is used. The template is then removed, leaving cavities that allow an increased selectivity for targeted analytes in comparison with standard SPE and thus, allows to get higher recovery yields and better sample cleanups.



② Sample preparation and extraction procedure

SAMPLE PREPARATION :
 1 mL of oil is diluted with 1 mL of cyclohexane to form the loading solution for SPE protocol with **AFFINIMIP® SPE PAHs - 3mL**



③ Results for canola and olive oils

% Recovery yields obtained for canola oil and olive oil spiked at 2µg/Kg with 8 PAHs.

Compounds	Canola oil			Olive oil		
	Blank µg/Kg	% recovery in Canola oil	RSD (n = 3)	Blank µg/Kg	% recovery in olive oil	RSD (n = 3)
Benzo[a]anthracene	0.12	86%	6%	0.26	89%	3%
Chrysene	0.15	89%	6%	0.64	95%	6%
Benzo[a]pyrene	N.D	93%	5%	0.12	89%	3%
Benzo[b]fluoranthene	0.08	83%	4%	0.15	86%	3%
Benzo[k]fluoranthene	0.03	86%	7%	0.08	84%	3%
Dibenz[a,h]anthracene	N.D	88%	7%	N.D	91%	6%
Benzo[g,h,i]perylene	0.09	87%	2%	0.16	91%	5%
Indeno[1,2,3-c,d]pyrene	0.03	83%	6%	0.05	90%	6%

(N.D = not detected)

Analysis with agilent 7000 series GC-MS/MS

GC-MS/MS analysis were carried out by the national reference laboratory LABERCA, Nantes (France)

GC separation

Column: Select PAH (30m x 0.25 x 0.15µm) (Agilent technologies)

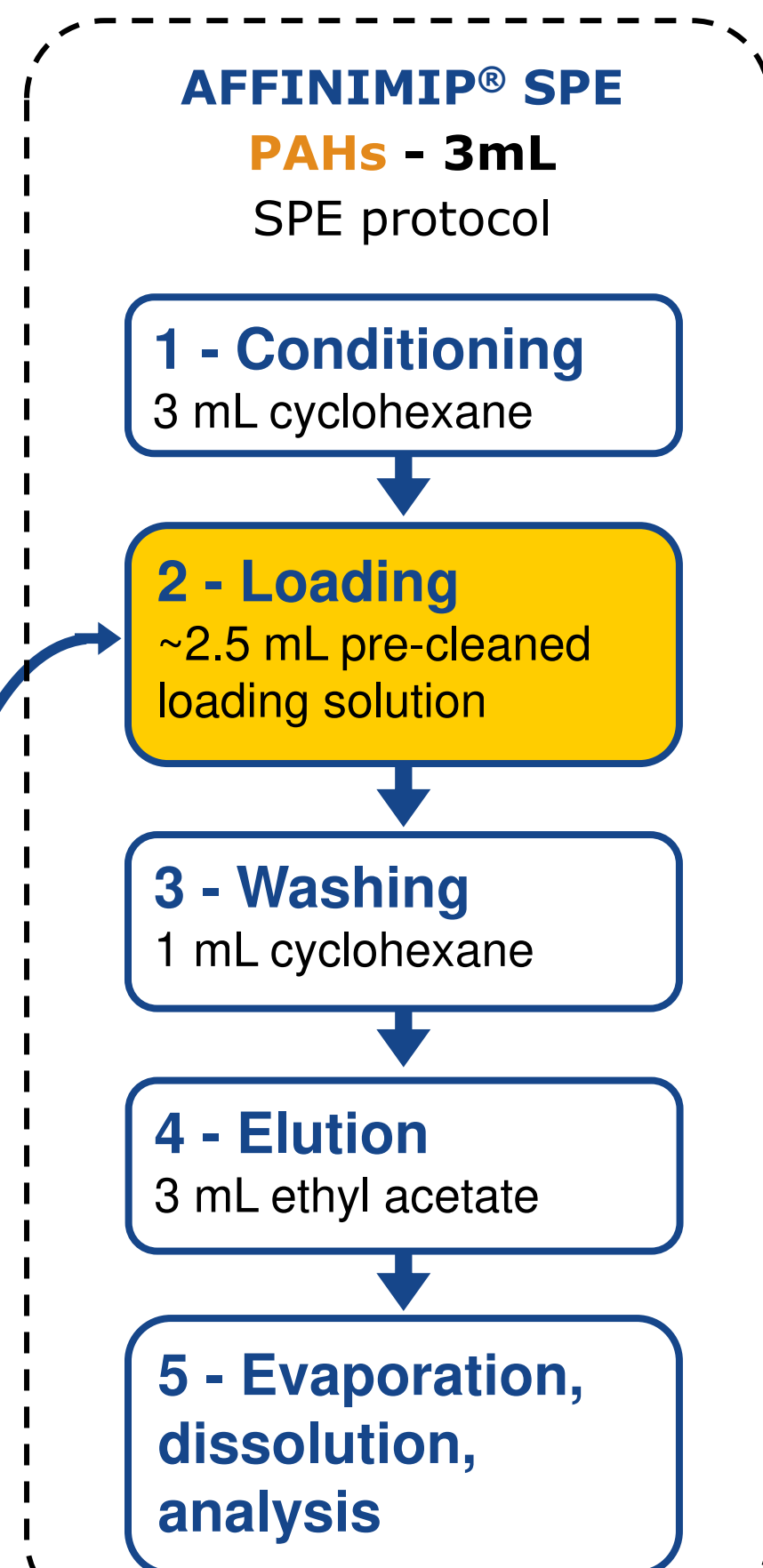
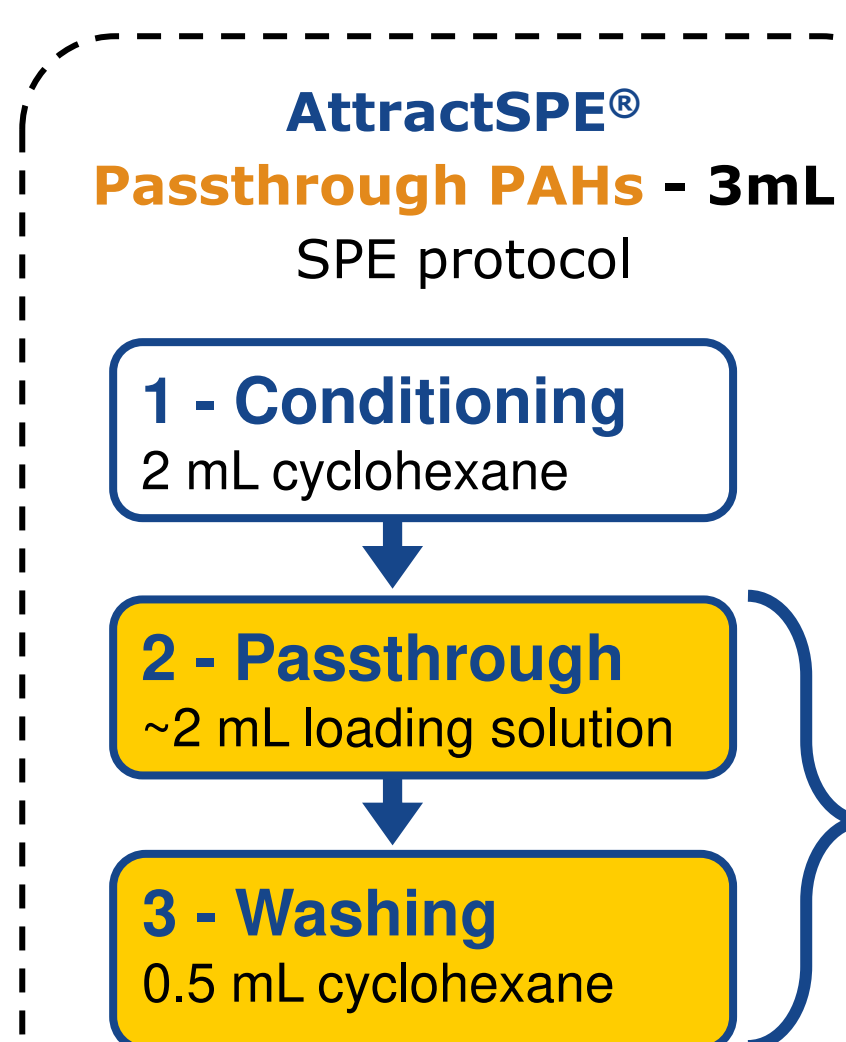
Gradient: (total duration : 37.92 min) : 110°C (1 min); 60°C/min up to 220°C(0 min); 5°C/min up to 270°C (0 min); 3°C/min up to 295°C (0 min); 20°C/min up to 330°C(10 min); 2°C/min up to 340°C (0 min)

MS/MS detection (QqQ)

Molecules	Transition 1	CE T1 (eV)	Transition 2	CE T2 (eV)	Tr (min)
Benzo[a]anthracene	228.1 >226.1	30	228.1 >202.1	20	14.66
(IS) Benzo[a]anthracene ¹³ C6	234.1 >232.1	30			14.66
Chrysene	228.1 >226.1	30	228.1 >202.1	20	15.00
(IS) Chrysene ¹³ C6	234.1 >232.1	30			15.00
Benzo[b]fluoranthene	252.1 >250.1	30	252.1 >226.1	20	20.28
(IS) Benzo[b]fluoranthene ¹³ C6	258.1 >256.1	30			20.28
Benzo[k]fluoranthene	252.1 >250.1	30	252.1 >226.1	20	20.40
(IS) Benzo[k]fluoranthene ¹³ C6	258.1 >256.1	30			20.40
Benzo[a]pyrene	252.1 >250.1	30	252.1 >226.1	20	22.23
(IS) Benzo[a]pyrene ¹³ C4	258.1 >254.1	30			22.23
Indeno[1,2,3-c,d]pyrene	276.1 >274.1	45	276.1 >272.1	50	25.79
(IS) Indeno[1,2,3-c,d]pyrene ¹³ C6	282.1 >280.1	45			25.79
Dibenz[a,h]anthracene	278.1 >276.1	30	278.1 >252.1	20	25.79
(IS) Dibenz[a,h]anthracene ¹³ C6	284.1 >282.1	30			25.79
Benzo[g,h,i]perylene	276.1 >274.1	45	276.1 >272.1	50	27.06
(IS) Benzo[g,h,i]perylene ¹³ C12	288.1 >286.1	45			27.06

Work with CBD oil – Full spectrum (CBD 15%)

SAMPLE PREPARATION :
 1 mL of CBD oil is diluted with 1 mL of cyclohexane to form the loading solution.



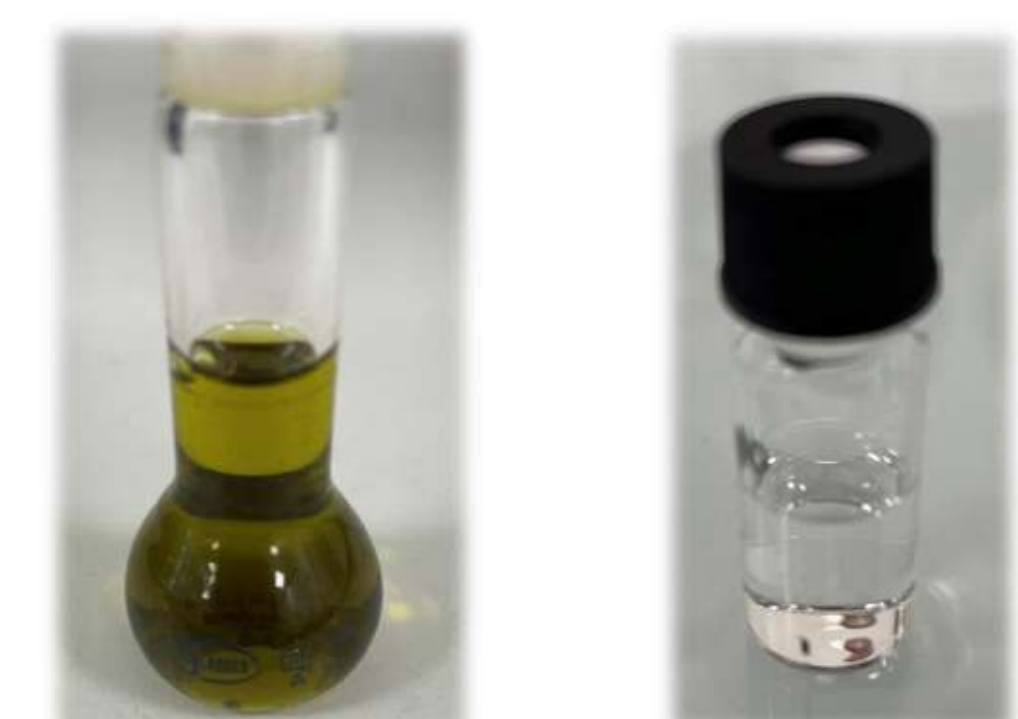
% Recovery yields obtained for CBD oil spiked at 5µg/Kg with 7 PAHs.

Compounds	CBD oil		
	Blank µg/Kg	% recovery in CBD oil	RSD (n = 3)
Benzo[a]anthracene	1.57	103%	3%
Chrysene	0.82	94%	3%
Benzo[a]pyrene	1.39	92%	7%
Benzo[b]fluoranthene	2.08	97%	1%
Benzo[k]fluoranthene	0.87	85%	4%
Dibenz[a,h]anthracene	N.D	86%	1%
Benzo[g,h,i]perylene	0.82	86%	3%

(N.D = not detected)

Analysis by LC-FLD

- Column:** Zorbax Eclipse PAH (50mm x 4.6mm x 1.8µm) (Agilent technologies)
- Temperature:** 30°C
- Injection volume:** 50µL
- Gradient:** 25 minutes isocratic 15/85 (v/v) water/acetonitrile
- Detection wavelengths:** λ_{ex/em} 252/400nm



CBD oil diluted with cyclohexane (left) and sample after cleanup (right).

Conclusion

AFFINIMIP® SPE PAHs cartridges have shown excellent performances for the extraction and analysis of 8 PAHs. Excellent recovery yields were obtained between **83 and 95%** with a very satisfying repeatability. Moreover, the protocol was also proved to be very effective for a more complex oil (CBD oil – full spectrum) with the addition of a passthrough cartridge. Recovery yields were obtained between **85% and 103%** with also a very satisfying repeatability.