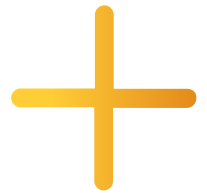


Application Note



**Determination of PAHs in Olive and Rapeseed oils
by GC-MS/MS using AFFINIMIP®SPE PAHs**



INTRODUCTION :

Polycyclic Aromatic Hydrocarbons (PAHs) are a large group of organic compounds with two or more fused aromatic rings and are known to be carcinogenic. Human beings are exposed to PAHs mostly by intake of food. As these are highly soluble in lipophilic matrices, edible oils can be an important source of contamination by PAHs. In 2011, EU Commission Regulation No 835/2011, amending Regulation 1881/2006, set maximum levels in edible oils to 2 $\mu\text{g}/\text{Kg}$ of benzo[a]pyrene individually, and 10 $\mu\text{g}/\text{Kg}$ of benzo[a]pyrene, benzo[b]fluoranthene, chrysene and benzo[a]anthracene combined.

This application note describes the extraction and GC-MS/MS analysis of 8 PAHs (including the four regulated ones) in olive oil and rapeseed (canola) oil using **AFFINIMIP®SPE PAHs 3mL**.

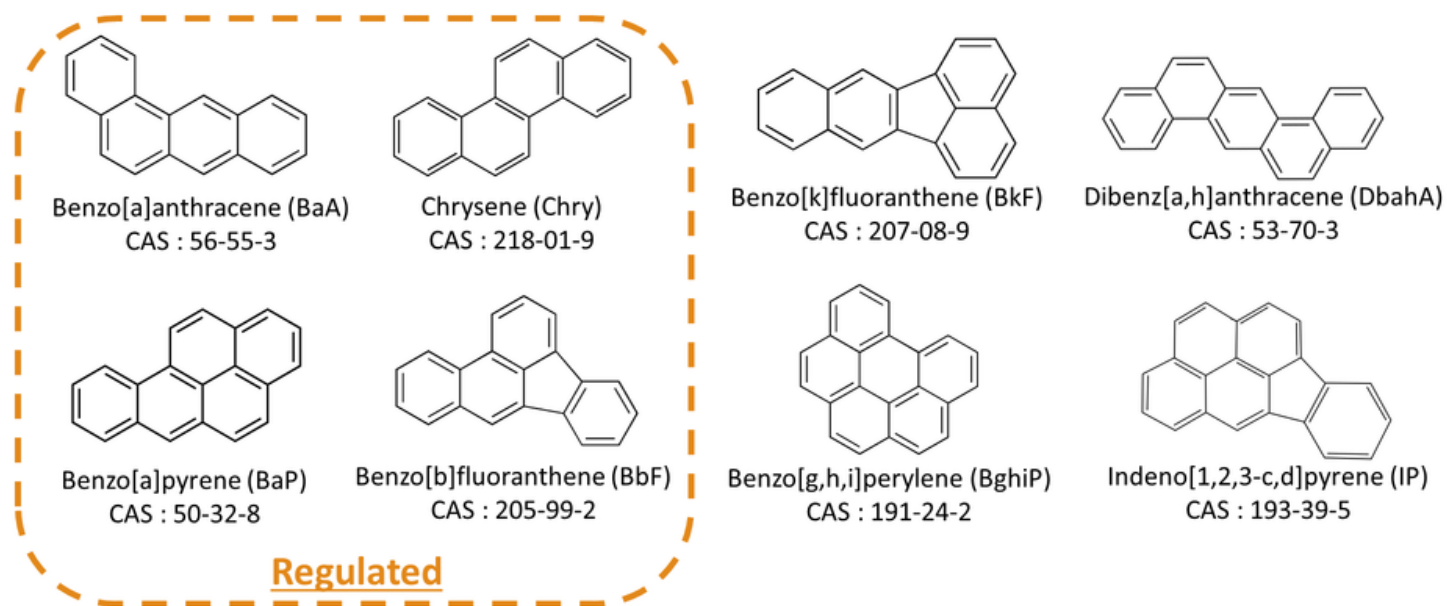


Figure 1. List of the tested PAHs

Sample preparation

In this application note, canola oil and olive oil were both spiked with 8 PAHs and then purified by SPE using **AFFINIMIP®SPE PAHs 3 mL**. Each spiked oil was tested three times. A blank (non-spiked) was also tested to determine the potential presence of PAHs in oils.

Preparation of the loading solution:

10 mL of oil is spiked at 2 µg/Kg with a mix of 8 PAHs, then 10 mL of cyclohexane is added. The mixture is stirred to form the loading solution.

Extraction and cleaning procedure

CONDITIONING

- 3mL cyclohexane

LOADING

- 2 mL loading solution (~0.3mL/min)

WASHING

- 1mL cyclohexane

ELUTION

- 3mL Ethyl Acetate

The elution solutions were evaporated to dryness under vacuum at 40°C for 20 minutes, then dissolved in 1mL of toluene prior to analysis.



Conditions of analysis

Device : Agilent 7000 series GC-MS/MS.

GC Column: Select PAH (30m x 0.25 x 0,15µm) (Agilent Technologies)

Temperature 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)

Temperature gradient

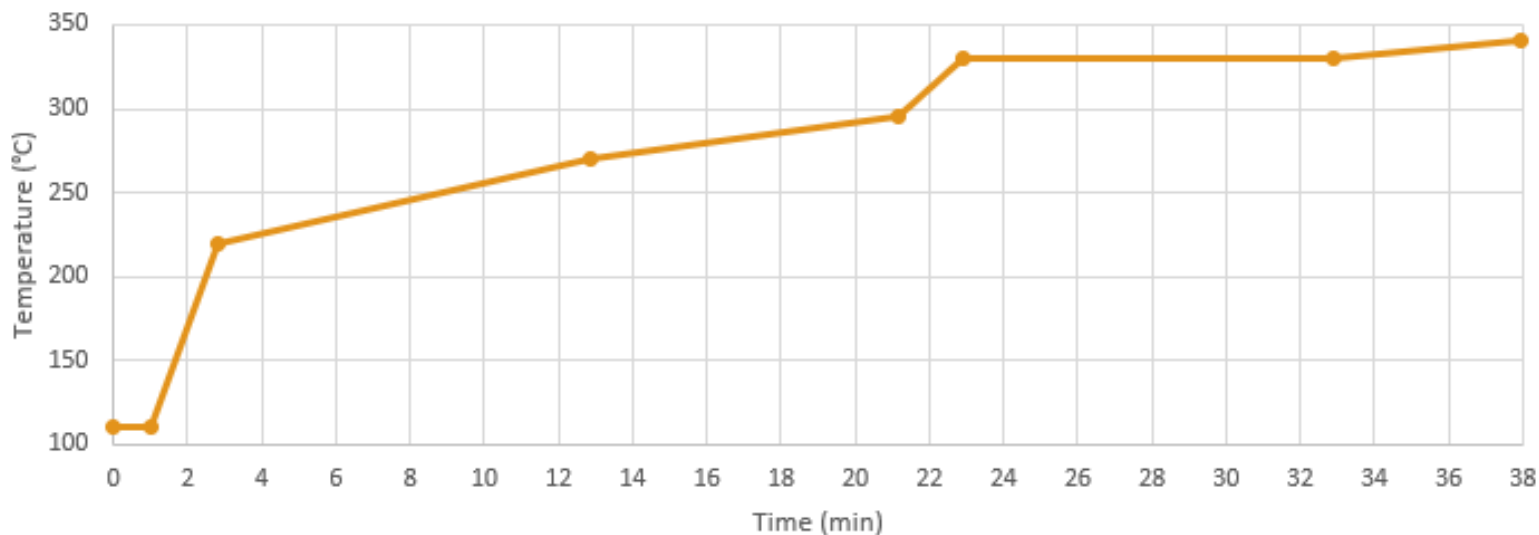


Figure 2. Temperature gradient

Molecules	Transition 1	Collision energy T1 (eV)	Transition 2	Collision energy T2 (eV)	Tr (min)
Benz[a]anthracene	228.1 > 226.1	30	228.1 > 202.1	20	14.66
(IS) Benz[a]anthracene 13C6	234.1 > 232.1	30			14.66
Chrysene	228.1 > 226.1	30	228.1 > 202.1	20	15.00
(IS) Chrysene 13C6	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 13C6	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 13C6	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 13C4	256.1 > 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 13C6	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 13C6	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 13C12	288.1 > 286.1	45			27.06

Table 1. List of transitions for the 8 PAHs and corresponding Internal Standards (IS).

Results

COMPOUNDS	RAPESEED OIL			OLIVE OIL		
	BLANK µg/Kg	% RECOVERY IN CANOLA OIL	RSDr (n = 3)	BLANK µg/Kg	% RECOVERY IN OLIVE OIL	RSDr (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%

Table 2. Concentration found in blank (non-spiked oils), recovery yield with blank subtracted, and repeatability for rapeseed oil and olive oil obtained using **AFFINIMIP®SPE PAHs - 3mL**. (N.D = Not detected)

CONCLUSION

AFFINIMIP®SPE PAHs cartridges have shown excellent performances for the extraction and analysis of 8 PAHs in rapeseed (canola) and olive oils. Excellent recovery yields were obtained between 83% and 95% with a very satisfying repeatability. Moreover, the sample preparation allows cleaner samples and reduces the risk of premature GC-MS/MS fouling. It also makes the protocol suitable for LC-fluorescence analysis (see application note AN-0015-01).

Product references :

AFFINIMIP®SPE PAHs cartridges :

- **FS119-03-NG for AFFINIMIP®SPE PAHs - 3mL - 50/pk**
- **FS119-03B-NG for AFFINIMIP®SPE PAHs - 6mL - 50/pk**