

Application Note

Food and Beverages

Quantitative Analysis of Polycyclic Aromatic Hydrocarbons in Five Edible Oil Samples by GCMS-TQ8050 NX

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1. Introduction

Polycyclic Aromatic Hydrocarbons (PAHs) are a group of organic compounds containing two or more fused aromatic rings, formed primarily during the incomplete combustion of organic materials. PAHs are lipophilic organic contaminants that readily accumulate in lipid-rich foods such as edible oils and fats. Long-term exposure to PAHs increases the risk of cancer, harms the heart and lungs, and weakens the immune system. To protect consumer health, the European Commission (EU) 2023/915 has established maximum levels (MLs) for several PAHs^[1] in edible oils and fats. Among all, benzo[a]pyrene (BaP) serves as the primary marker compound, while compliance is based on the sum of four priority PAHs (PAH4): BaP, benz[a]anthracene (BaA), benzo[b]fluoranthene (BbF), and chrysene (CHR) (Fig. 1). The maximum levels are 2 µg/kg for benzo[a]pyrene (BaP) and 10 µg/kg for the sum of the four marker PAHs (PAH4). For coconut oil and cocoa butter, the maximum level for PAH4 is 20 µg/kg.

The analysis of PAHs in edible oils is challenging because the high triglyceride content and other matrix components can interfere with chromatographic analysis. Therefore, efficient sample extraction and cleanup procedures are recommended for trace-level determination. In the present study, Molecularly Imprinted Polymer Solid-Phase Extraction (MIP-SPE) was employed for extraction and cleanup. A highly sensitive GC-MS/MS method was developed for 14 PAHs including regulated PAHs in five commonly used edible oils namely corn oil, olive oil, sesame oil, coconut oil and rice bran oil.

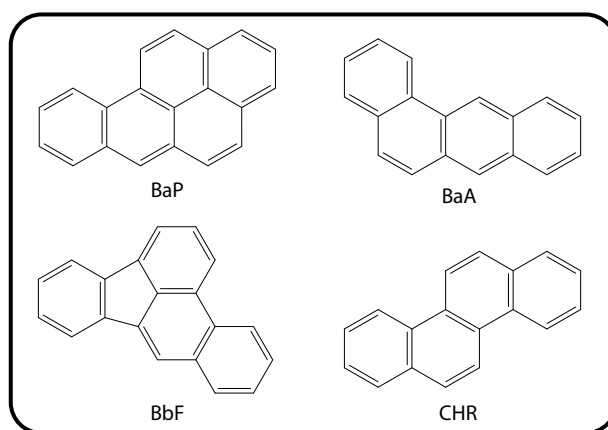


Fig. 1 Structures of four priority PAHs

2. Materials and methods

The PAH calibration standard mixture, containing target analytes in methylene chloride at individual concentrations ranging from 500 to 1000 µg/mL, was procured from Restek Corporation (Cat. No. 31264). The isotopically labelled internal standard mixture was obtained from AccuStandard, Inc. (Cat. No. EN-17132-IS).

A high purity grade cyclohexane, ethyl acetate and toluene were used for sample extraction. AFFINIMIP® SPE PAHs cartridges (3 mL), was purchased from

Affiniseq. Commercially available samples of olive oil, coconut oil, sesame oil, rice bran oil, and corn oil were procured from the local market and analyzed for the target PAHs.

The quantification of PAHs was carried out using a Shimadzu GCMS-TQ8050 NX (Fig. 2), equipped with the HTA 2800T "all-in-one GC autosampler". This autosampler can perform liquid injections, headspace analysis as well as Solid - Phase Microextraction (SPME).



Fig. 2 GCMS-TQ™8050 NX

2.1. Analytical Condition

Preparation of instrument and analysis.

A scan was performed using a standard solution containing the target PAHs and isotopically labelled internal standards. The NIST Mass Spectral Library was used to identify the peaks for target analytes and internal standards. The retention time and precursor ion for each compound were established. This can be used for creating a multiple reaction monitoring (MRM) method.

However, optimization of the voltages for individual MRM transitions requires separate acquisition methods for each transition. Subsequently, each dataset must be independently processed and the optimized parameters manually consolidated into a single MRM method, which significantly increases the method development time.

To overcome this, "Create MS Table" feature of LabSolutions™ GCMS software was used which automatically generates a product ion scan method through a guided, step-by-step workflow.

The product ion scan method generated above was used in the MRM Optimization Tool of Smart Pesticides Database™ Ver.2.1, which automatically creates an acquisition batch consisting of multiple methods. After batch acquisition, all data files were uploaded to the MRM Optimization Tool, which automatically processes the individual datasets, optimize the voltages for each MRM transition, and compile them into a single MRM method, thereby eliminating the need for manual data evaluation and method compilation. The final acquisition method parameters are summarized in Table 1.

2.2. Standard and Sample Preparation

Preparation of calibration standards.

A seven-level calibration standard was prepared using matrix-matched solvent derived from corn oil, covering a concentration range of 0.5-100 µg/L for the native PAHs, with the deuterated internal standards (ISTD) maintained at a constant concentration of 25 µg/L.

Preparation of oil samples and spiked samples.

1 mL aliquot of each edible oil sample (olive, coconut, sesame, rice bran, and corn oil) was transferred to a suitable vial and spiked with 50 µL of ISTD (500 µg/L).

Table. 1 Analytical conditions

GC Inlet Parameters			GC Column Oven Program		
Injector temperature	250 °C	Analytical column: SH-I-5Sil MS (P/N:221-75954-30) (30 m x 0.25 mm; df: 0.25 µm)	Rate (°C/min)	Temperature (°C)	Hold Time (min)
Injection mode	Splitless		-	90.00	1.00
Injection volume	1.0 µL		30.00	130.00	0.00
Flow control mode	Linear Velocity (55.0 cm/s)		10.00	320.00	3.00
Purge flow	5.0 mL/min		MS Parameters		
Total flow	30.0 mL/min		Ion Source temperature	230 °C	
			Interface temperature	250 °C	
			Solvent cut time	1.5 min	
			Detector voltage	+ 0.6 kV (relative to tune result)	

Target	Retention time (min)	Type	ISTD Group	Target transition	Reference Transitions
Acenaphthylene	5.674	Target	2	152.00>150.00	152.00>126.10; 152.00>102.10
Fluorene	7.016	Target	2	165.00>163.00	165.00>115.10; 165.00>139.10
Phenanthrene	9.077	Target	2	178.00>152.10	178.00>176.10; 176.00>150.20
Anthracene	9.191	Target	3	178.00>176.10	178.00>152.20; 176.00>150.20
Phenanthrene-D10	9.025	ISTD	2	158.00>156.10	158.00>82.10; 185.00>157.20
Anthracene-D10	9.149	ISTD	3	188.00>160.10	184.10>156.05; 160.10>158.10
Fluoranthene	11.806	Target	4	202.00>200.00	101.00>88.00; 202.00>152.50
Pyrene-D10	12.266	ISTD	4	210.00>208.10	209.00>207.20; 210.00>206.10
Pyrene	12.307	Target	4	202.00>200.10	101.00>88.00; 200.00>199.00
Benz[a]anthracene (BaA)	15.185	Target	5	228.00>226.00	114.00>101.00; 226.00>224.00
Chrysene (CHR)	15.263	Target	5	228.00>226.00	226.00>223.90; 228.00>202.20
Benzo[b]fluoranthene (BbF)	17.581	Target	5	252.00>250.00	250.00>248.00; 253.00>251.00
Benzo[k]fluoranthene	17.639	Target	5	252.00>250.00	250.00>248.10; 252.00>249.00
Benzo[a]pyrene (BaP)	18.228	Target	5	252.00>250.00	126.00>113.10; 250.00>247.30
Benzo[a]pyrene - D12	18.179	ISTD	5	261.00>257.00	116.00>102.10
Perylene-D12	18.348	ISTD	6	264.00>260.10	132.10>118.10; 132.10>130.10
Indeno[1,2,3-cd]pyrene	20.327	Target	6	276.00>274.10	138.00>125.10; 138.00>136.10
Dibenz[a,h]anthracene	20.395	Target	6	278.00>276.10	279.00>277.10; 278.00>275.00
Benzo[ghi]perylene	20.759	Target	6	276.00>274.00	138.00>125.00; 274.00>272.00

The sample was then diluted with 1 mL of cyclohexane and mixed thoroughly using a vortex mixer.

An AFFINIMIP® SPE PAHs cartridge was conditioned with 3 mL of cyclohexane prior to sample loading. Subsequently, 2 mL of the prepared sample solution was loaded onto the cartridge and passed through at an approximate flow rate of 0.3 mL/min using a positive-pressure manifold. The cartridge was then washed with 1 mL of cyclohexane to remove residual matrix components. Finally, the retained PAHs were eluted with 3 mL of ethyl acetate. The eluate was evaporated to dryness under a gentle stream of nitrogen in a water bath maintained below 30 °C. The residue was subsequently reconstituted using 1 mL of toluene and transferred to an autosampler vial for GC-MS/MS analysis.

To evaluate the performance of the method, spike recovery studies were carried out using all five edible oil matrices. The same extraction and cleanup procedure was followed for spike samples too.

3. Results and Discussion

The matrix-matched calibration curves obtained demonstrated excellent linearity over the concentration range of 0.5–100 µg/L for all target PAHs with correlation coefficients (r^2) greater than 0.99 and accuracy between 80–120 %. This confirms a strong linear relationship between analyte concentration and instrument response across the evaluated calibration range. The calibration curves for the four PAHs are presented in Fig. 3. In addition, a signal-to-noise ratio (S/N) of ≥ 10 was achieved at the lowest calibration level, demonstrating adequate sensitivity of the developed method for the determination of PAHs at low concentration levels.

Among the five edible oil samples analysed, corn oil and rice bran oil showed no detectable levels of any of the 14 target PAHs. In olive oil, chrysene was detected at a concentration of approximately 1 µg/kg. Meanwhile, in coconut oil benzo[a]anthracene and chrysene were detected at concentrations of 1.1 and 1.4 µg/kg, respectively. Sesame oil showed the highest PAH contamination, with benzo[a]pyrene detected at 1 µg/kg and the total concentration of the four marker PAHs (PAH4) measured at 6 µg/kg. The concentrations measured in all analysed edible oil samples were below the applicable maximum levels specified in Commission Regulation (EU) 2023/915, indicating compliance with the established regulatory requirements for PAHs in food.

The recovery results are summarized in Table 2 and graphically presented in Fig. 4. Among the target analytes, acenaphthylene exhibited comparatively lower recoveries of approximately 60 %, whereas fluorene generally recovered recoveries in the range of 70–85 % for most of the oil matrices, with the exception of coconut oil and olive oil. The relatively lower recoveries observed for these compounds may be attributed to their weaker interaction and retention on the MIP-SPE sorbent and/or potential analyte losses during the solvent evaporation step. The remaining PAHs demonstrated generally satisfactory recoveries, indicating effective extraction and enrichment of the target compounds from the edible oil matrices. However, a few analytes showed recoveries in the range of 120–150 %, which may be associated with residual matrix effects that got enhanced by analytical response of co-extracted matrix components.

The developed GC-MS/MS method for the determination of PAHs in edible oils demonstrated satisfactory sensitivity and linearity. The use of molecularly imprinted polymer (MIP)-based SPE provides enhanced selectivity toward PAHs compared with conventional SPE sorbents and facilitates the effective removal of lipid-rich matrix components. This results in cleaner extracts, reduced matrix-related interference and improved suitability for sensitive GC-MS/MS determination.

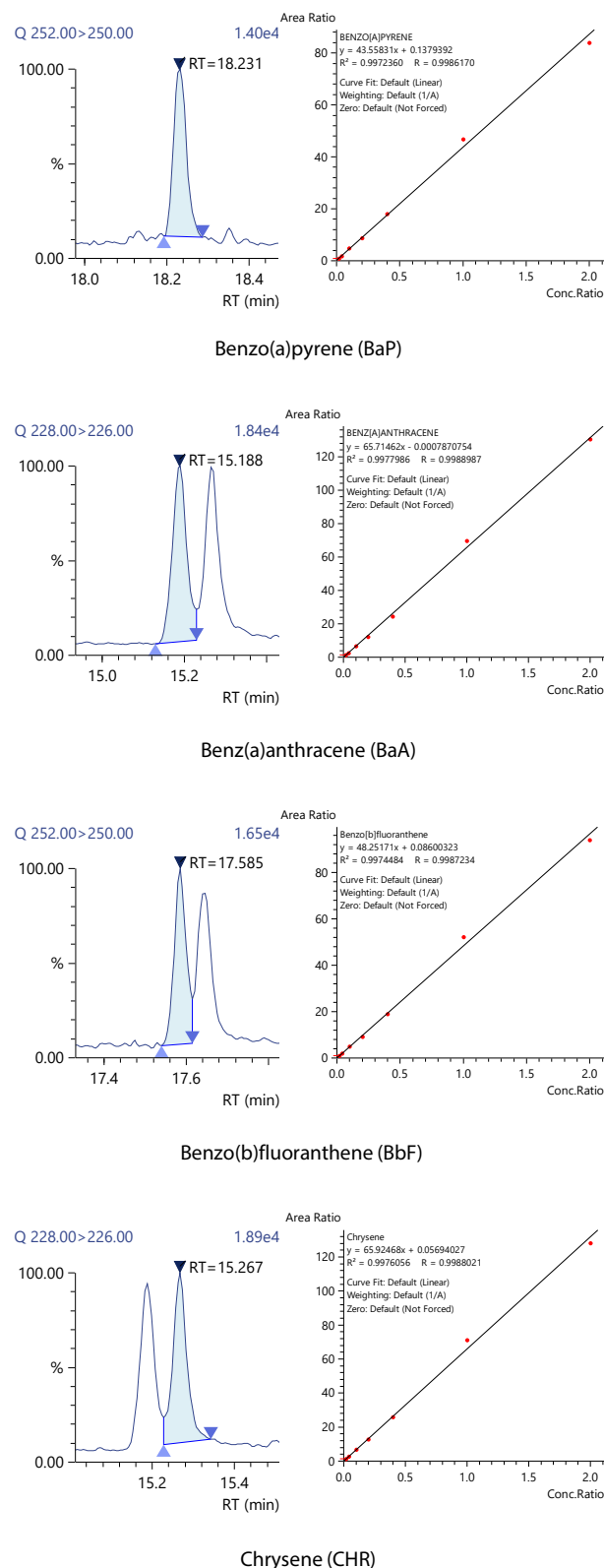


Fig. 3 Representative chromatograms and calibration curves for PAHs

Table. 2 Summary results of oil samples

Target	Spiked Amount (µg/kg)	Corn Oil		Sesame Oil		Rice Bran Oil		Coconut Oil		Olive Oil	
		Sample	Recovery (%)	Sample	Recovery (%)	Sample	Recovery (%)	Sample	Recovery (%)	Sample	Recovery (%)
Acenaphthylene	4	ND	61.9	1.1	61.9	ND	52.7	2.2	58.4	ND	64.4
Fluorene	4	ND	82.7	4.8	76.2	ND	72.2	2.0	64.1	1.2	67.5
Phenanthrene	2	ND	116.4	27.4	130.2	ND	119.7	15.3	107.6	8.4	130.6
Anthracene	4	ND	111.1	2.0	127.9	ND	109.7	3.3	111.4	ND	130.8
Fluoranthene	2	ND	121.0	5.0	133.9	ND	114.3	9.1	135.7	3.8	126.3
Pyrene	2	ND	136.6	6.4	154.4	ND	118.2	8.0	124.9	4.3	136.9
Benz[a]anthracene (BaA)	2	ND	135.0	1.7	114.6	ND	118.7	1.1	102.5	ND	145.3
Chrysene (CHR)	2	ND	120.2	2.3	116.1	ND	124.1	1.4	101.1	1.0	134.1
Benzo[b]fluoranthene (BbF)	2	ND	114.0	1.3	105.3	ND	133.6	ND	135.4	ND	111.8
Benzo[k]fluoranthene	2	ND	109.6	ND	121.4	ND	106.8	ND	111.3	ND	101.3
Benzo[a]pyrene (BaP)	2	ND	113.3	1.0	109.1	ND	120.7	ND	120.6	ND	108.9
Indeno [1,2,3-cd]pyrene	2	ND	105.4	ND	133.3	ND	106.1	ND	112.1	ND	94.7
Dibenz[a,h]anthracene	2	ND	109.7	ND	108.5	ND	111.4	ND	107.0	ND	92.8
Benzo[ghi]perylene	2	ND	92.7	ND	111.5	ND	94.8	ND	87.6	ND	77.2

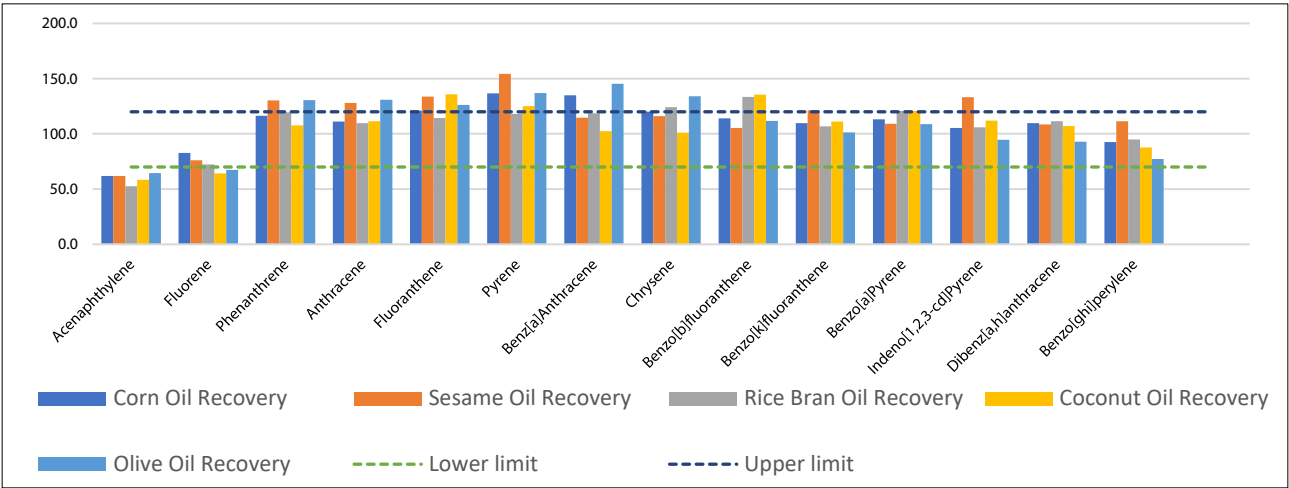


Fig. 4 Recoveries of PAHs from five edible oil samples

4. Conclusion

- ◆ A highly sensitive GC-MS/MS method was developed for the determination of 14 PAHs in five edible oils namely; olive oil, coconut oil, sesame oil, rice bran oil, and corn oil using the Shimadzu GCMS-TQ8050 NX.
- ◆ Sample preparation was performed using AFFINIMIP® SPE PAHs Molecularly Imprinted Polymer Solid-Phase Extraction (MIP-SPE) columns, which provided effective cleanup and selective extraction of PAHs from the complex oil matrix.
- ◆ The developed protocol is suitable for analyzing PAHs in complex, highly pigmented, and high-fat cooking oil matrices containing triglycerides, plant sterols, vitamins, and natural antioxidants.

- ◆ Better recoveries were obtained for the heavier PAHs across the tested oil matrices, demonstrating the reliable performance of the MIP-SPE method. The results also satisfied the criteria specified in Commission Regulation (EU) 2023/915 for PAHs in edible oils.

5. References

- 1) Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006. <https://eur-lex.europa.eu/eli/reg/2023/915/oj/eng> (Accessed on September 14, 2026).

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