

This test method describes a high performance liquid chromatography (HPLC) procedure for the determination of polycyclic aromatic hydrocarbons (PAHs) in an oil matrix.

INTRODUCTION

PAHs (*Figure 1*) are environmental and food-borne contaminants. Edible oils and fats may be contaminated by environmental pollutants and during the processing steps of crude oils prior to refining. The presence of PAHs in fats and oils is a health concern due to the carcinogenicity of the PAHs. Different levels of PAHs have been observed in crude edible oils. The refining process of the oils, which involves deodorization, bleaching and charcoal treatment under appropriate conditions, reduces the contents of individual PAHs to levels measured in $\mu\text{g kg}^{-1}$.

The known methods of analysis of PAHs in edible oils and fats comprise complex and laborious extraction and clean-up steps to isolate the low levels of PAHs present in the oil matrix. The donor-acceptor complex chromatography (DACC) method, when compared to the conventional methods, uses a significantly reduced amount of solvent and shortens the analysis time considerably. The total analysis time for one

sample is approximately 55 min compared to the traditional methods of 8-10 hr.

PRINCIPLE

PAHs are electron donors (π -electron) and hence can interact strongly with an electron acceptor stationary phase, resulting in the retention of PAHs and elution in the oil components. DACC is an automated on-line method (*Figure 2*) for the determination of PAHs in edible oils and fats. The system consists of liquid chromatography-liquid chromatography (LC-LC) coupling, where a clean-up DACC column is linked to an analytical column for the separation of PAHs. The PAHs are quantified using fluorescence detection.

RESULTS

The 16 PAHs compound were successfully separated from oil matrix (*Figure 3*) using DACC method. Method validation was carried out by determining the linearity, reproducibility and repeatability of the method. The results show that the R^2 for 16 compounds were in the range of 0.9958 – 0.9974. The recovery for reproducibility and repeatability was in the range of 70%-92%. The standard deviation (SD) for both repeatability and reproducibility was $< 10\%$. The limit of quantification was $0.1 \mu\text{g kg}^{-1}$ for individual PAHs.

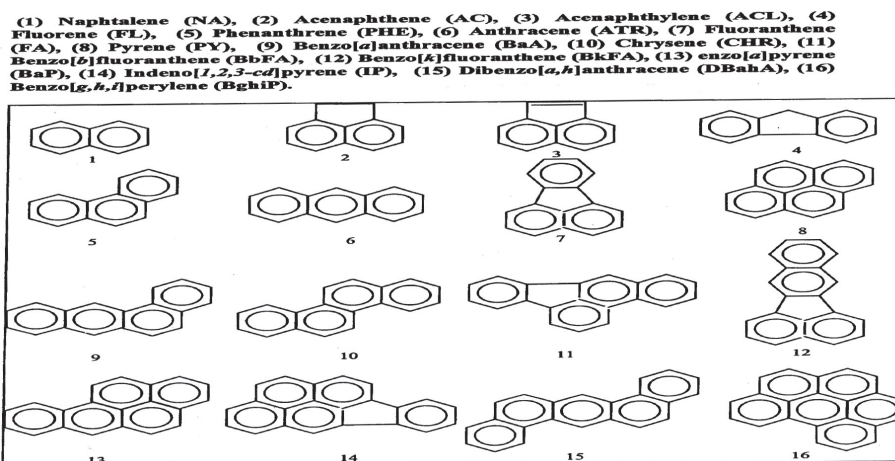


Figure 1. Chemical structure of 16 polycyclic aromatic hydrocarbons (PAHs) under US-EPA Priority Pollutant list.



Figure 2. High performance liquid chromatography (HPLC) instrumentation system for the donor-acceptor complex chromatography (DACC) method.

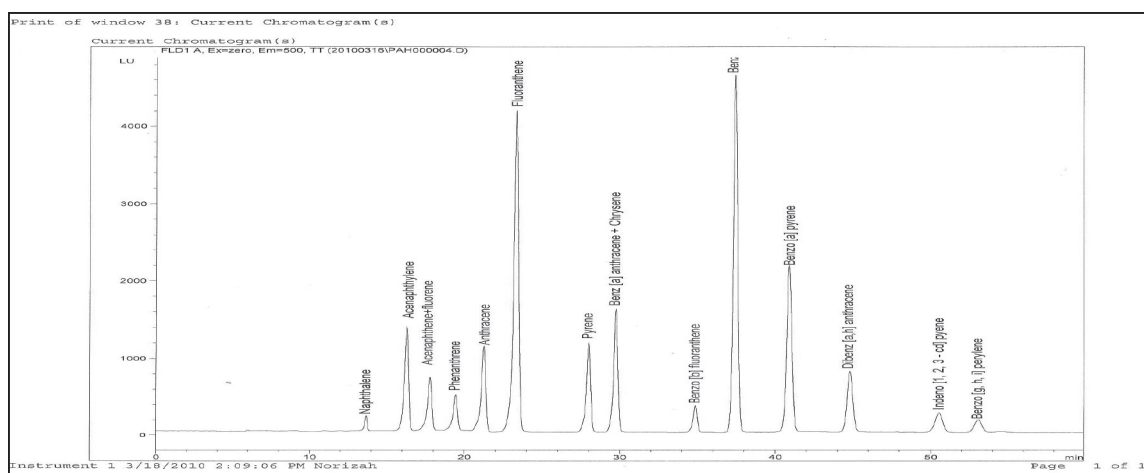


Figure 3. High performance liquid chromatography (HPLC) chromatogram of 16 polycyclic aromatic hydrocarbons (PAHs) separated from oil matrix using the donor-acceptor complex chromatography (DACC) method.

CONCLUSION

The DACC method can run for 24 hr a day and be easily applied as a routine analysis. The losses of volatile PAHs during solvent evaporation are eliminated because no sample preparation steps are involved in this method.

For more information, kindly contact:

Director-General
MPOB
P. O. Box 10620
50720 Kuala Lumpur, Malaysia.
Tel: 03-8769 4400
Fax: 03-8925 9446
www.mpob.gov.my