TRAJAN

TECHNICAL ARTICLE

PAH analysis by GC

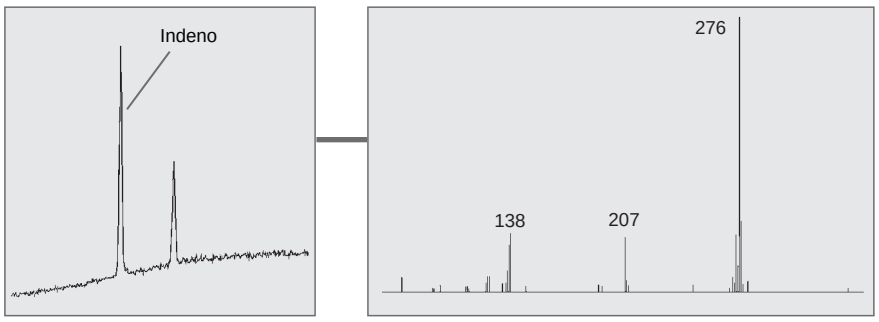
BPX5

Introduction

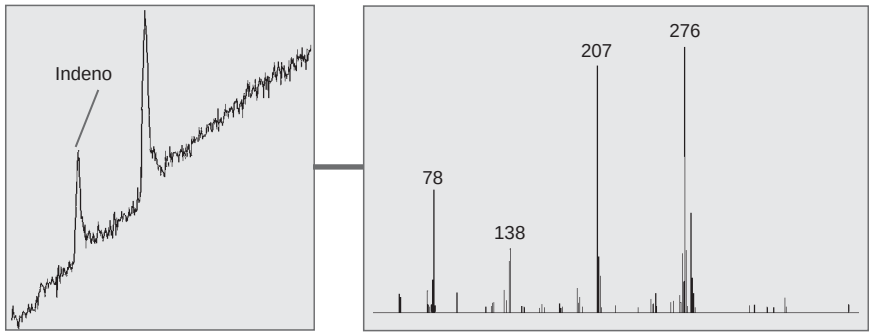
As benchtop quadrupole GC-MS systems continue to improve, there is a greater tendency to analyse PAHs in full scan mode as opposed to selective ion monitoring (SIM) mode. If the instrumentation is sensitive enough to meet the criteria set down by the USEPA, then full scan offers increased versatility. For example, it is common in routine GC maintenance to cut the injector side of the column to eliminate the collection of non-volatile material which can lead to increased activity, peak tailing and decreased sensitivity. The downside of this procedure is that retention times become progressively shorter leading to a shift outside the acquisition window in SIM mode. If this occurs, data from analysed samples could potentially be lost for particular PAHs. The danger of this occurring is eliminated in full scan mode. In addition, other contaminants will be missed in the analysis in SIM mode but the positive response of unknowns in full scan mode, means mass spectral searching is possible against a library containing standard mass spectra.

The extremely low bleed BPX5 column is ideal for the analysis of PAH compounds. This low bleed column results in less contamination of phase on the source in the mass spectrometer, hence less need for maintenance. It results in better quantitation (through improved signal to noise ratios) and therefore better peak identification due to improved spectral clarity. This latter advantage is important especially when the identification of unknowns is required and the identification and quantitation of PAHs near the limit of quantitation. This is clearly demonstrated from an examination of the chromatograms in Figure 1. The signal-to-noise ratio of indeno(1,2,3-cd)pyrene, when analysed on the BPX5 column, is clearly better when compared against a competitor's conventional polysiloxane column. This is evident from the ratio of the abundance of the molecular ion at 276 m/z against the 207 m/z ion which is derived from column bleed. The end result is not only greater sensitivity of the PAH, but improved spectral purity. This results in better quality 'hit' against standard contained in a commercial library of mass spectra.

BPX5



Competitor A



Final temperature	320°C
Component	Indeno(1,2,3-cd)pyrene (PAH)
Amount	0.5 ng

Figure 1: Chromatograms comparing the signal-to-noise ratio and the spectral clarity of the PAH compound Indeno[1,2,3-cd]pyrene on a BPX5 column against a competitor's conventional dimethyl polysiloxane column.

Note: the ratio of the molecular ion at 276 m/z to the 207 m/z ion which is from column bleed.

Figure 1. Chromatograms show improved signal to noise of BPX5 due to its lower bleed

Part number	054101		
Phase	BPX5	Final temperature	315°C
Column	30 m x 0.25 mm x 0.25 µm	Detector	MS, 350°C
Initial temperature	45°C, 1 min	Carrier gas	He, 1.0 mL/min constant flow
Rate 1	30°C/min to 200°C, 5 min	Carrier gas flow	20 cm/sec, 130°C
Rate 2	7°C/min to 315°C		

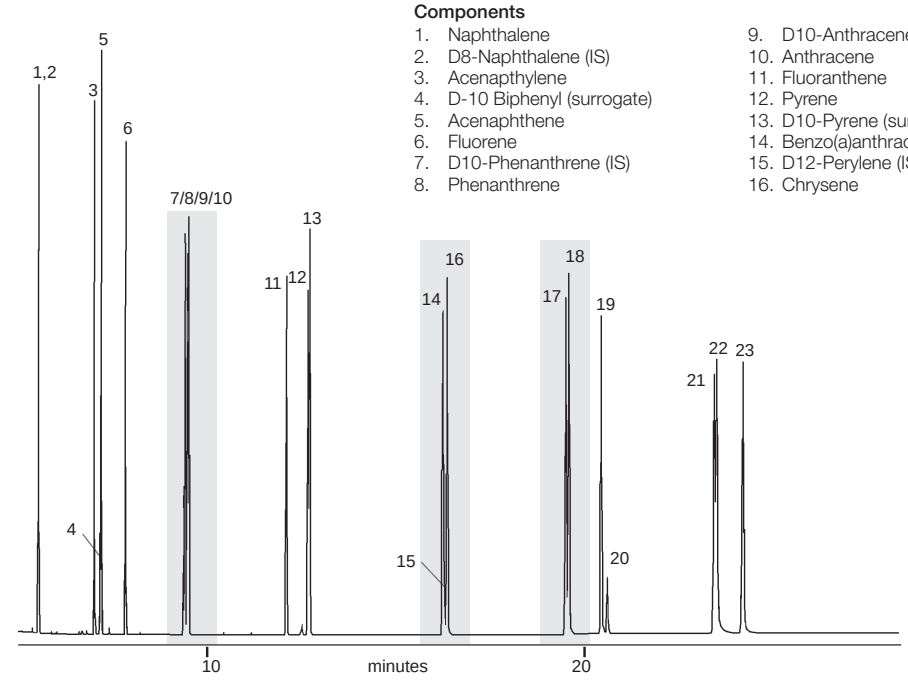


Figure 2 shows the separation of the 16 target compounds, 5 internal standards and 2 surrogate compounds. A 30 m x 0.25 mm x 0.25 µm column is used for this analysis. The chromatogram shows separation of phenanthrene / anthracene, chrysene / benzo[a]anthracene, benzo[a] / benzo[k] fluoranthene and indeno[1,2,3cd] pyrene / dibenzo[ah]anthracene. The first three pairs have been enlarged and reproduced in Figure 3.

Figure 2. Analysis of PAH mix and internal standard using a BPX5 column

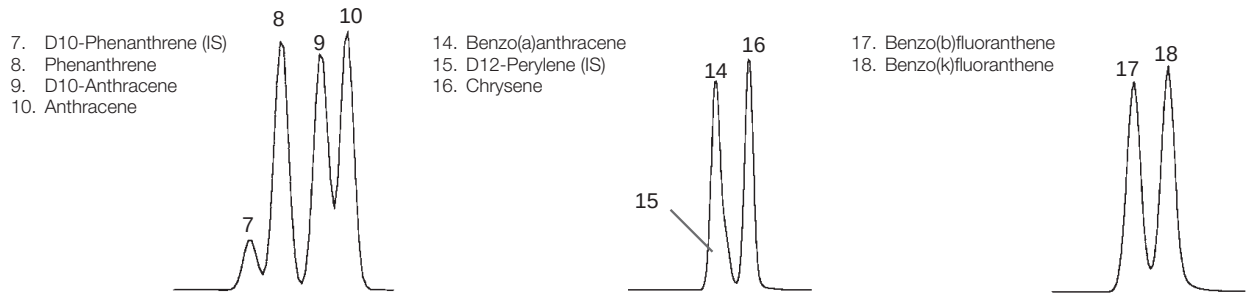


Figure 3. Chromatograms showing enlarged view of separation of various PAH pairs from Figure 2.

The BPX5 column shows excellent separation of the 16 targeted PAH compounds as shown in figure 2. Figure 3 shows the expanded separation of the PAH pairs which are the most difficult in this analysis. These pairs are phenanthrene / anthracene; the benzofluoranthene isomers; benzo[b] / benzo[k] and the PAH pair chrysene / benzo[a]anthracene. Because these three pairs of PAHs have identical mass spectra, chromatographic resolution is required for the analysis.

PAHs are a class of compounds targeted by the US Environmental Protection Agency (USEPA). Within this class, 16 are marked as priority pollutants and these range from the two-ringed naphthalene to multi-ring structures such as benzo[a]pyrene. Although there are methods available using HPLC and GC-FID for the analysis of this class of compounds, Gas Chromatography with Mass Spectrometry is the method of choice for the analysis in water and soil.

Information and support

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Specifications are subject to change without notice.