TRAJAN

TECHNICAL ARTICLE

Fast GC for TPH analysis

Fast GC

Total Petroleum Hydrocarbon (TPH) analysis is one of the most common analytical methods used in environmental laboratories. This method is applicable to either water or soil and involves an extraction (usually with dichloromethane) followed by GC analysis with a flame ionization detector (FID). The method is applicable to compounds which elute with vapor pressures between the aliphatic hydrocarbons of C8 and C36. Quantitation is carried out by relative response to either one aliphatic standard or relative to hydrocarbons at the elution mid-point of TPH fractions. The more volatile hydrocarbons are often analysed by purge and trap or by headspace methods.

The total instrument time of this analysis is crucial to high volume throughput laboratories. This time includes the actual analytical time and the GC oven cool-down time. These two added together are the cycle time of the analysis. TPH analysis is typically carried out on 0.25 or 0.32 mm ID columns with a film thickness of 0.25 µm. Depending on experimental conditions employed, run times can vary between 15 and 30 minutes. This article describes short ultra narrow bore columns with 0.1 µm films used to analyse TPH standards in around 6 minutes.

The primary advantage of very short ultra narrow bore columns with very thin films is the very fast analysis time possible. Run times can be halved compared to 0.25 and 0.32 mm ID columns. Using 0.1 mm ID columns does, however, present some challenges to the chromatographer. Inlet pressures do not necessarily need to be at optimum but need to be high for fast run times and this places more stress for a leak-free system. Also for fast GC, temperature program rates are limited by the ability of the GC oven to accurately follow the selected program rate. The very thin films equates with reduced capacities i.e. not as much sample can be injected into the column before overloading occurs.

The speed of elution with 0.1 mm ID columns and peak width also places a higher requirement on detectors. A Gaussian peak needs 20 data points across the peak for representative sampling. For bench-top quadrupole mass spectrometers, systems are often limited to a sampling rate of ≈15 Hz (at best with a narrow mass range) or 15 points / second. For peaks less than one second wide, this sampling rate is less than optimum for good integration.

Conditions

Phase	BPX5
Column	5 m x 0.1 mm x 0.1 µm
Initial temperature	50°C
Rate 1	45°C/min
Final temperature	300°C, 0 min
Detector temperature	270°C
Detector	FID
Carrier gas	He, Inlet pressure 40 psi (constant flow mode, linear velocity of 75 cm/sec)

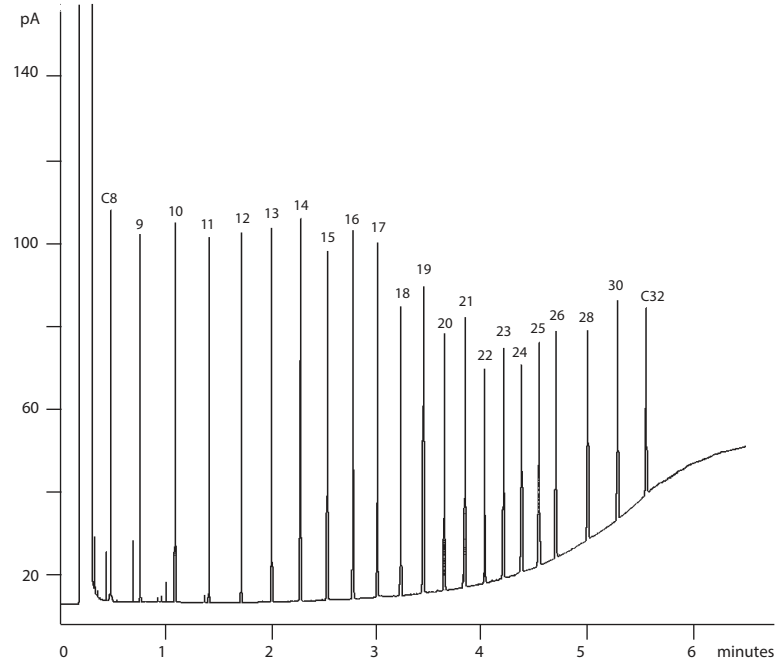

Figure 1. Chromatogram of TPH standards from C8 to C32

There is still a great deal of interest in fast GC. Figure 1 shows why this is the case. This is a chromatogram showing excellent resolution of aliphatic hydrocarbons from C8 to C32 in around six minutes. The data was acquired using a temperature program from 50°C (hold time 0.5 minutes) to 300°C at a temperature program rate of 45°C/min. The cycle time for this analysis is less than 12 minutes with an oven cool down time of 5.5 minutes from 300°C to 50°C.

Figure 2 shows a chromatogram showing excellent resolution of Benzene, Toluene, Ethyl benzene, para and meta-Xylene (co-eluting), ortho-Xylene and (BTEX) and n-octane (C8) in under one minute using the conditions described above.

Fast GC

There has been an increasing interest in 'fast GC'. What is fast GC and what are its advantages? Fast GC is about using short and ultra small diameter (usually 0.1 mm ID) columns with thin phase films and instrumental conditions of inlet pressure and temperature which give short analysis times. The attractiveness of the technique is greatly reduced run times. For example, run times can often be halved in fast GC leading to considerably reduced analysis times and laboratory costs.

Fast GC has always placed heavy demands on GC instrumentation. Fast GCs need rapid sample introduction, handle high inlet pressures, high GC temperature program rates, high inlet split ratios and fast detection rates. Instrument companies, however, have met this challenge and modern GCs offer many improvements such as electronic pressure controls and excellent oven temperature control to operate using fast GC conditions.

The phase ratio (Beta) is also an important consideration and to maintain a phase ratio the same as that of a 0.25 mm ID column with a film thickness of 0.25 µm, a film thickness of 0.1 µm has to be used for a 0.1 mm ID column. Remember, phase ratio = (column ID (µm)) / (4 x film thickness (µm)). This thin film equates with reduced sample capacities and is just another factor which needs consideration.

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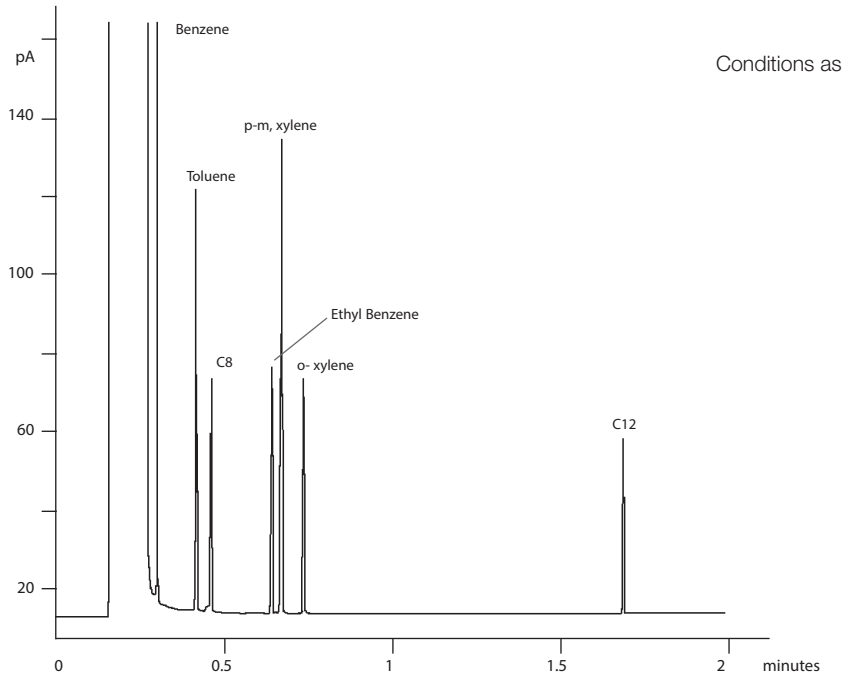

Conditions as per Figure 1.

Figure 2. Chromatogram showing elution of BTEX in under one minute