# Analysis of contaminants

## Fast GC with narrow-bore columns maintaining

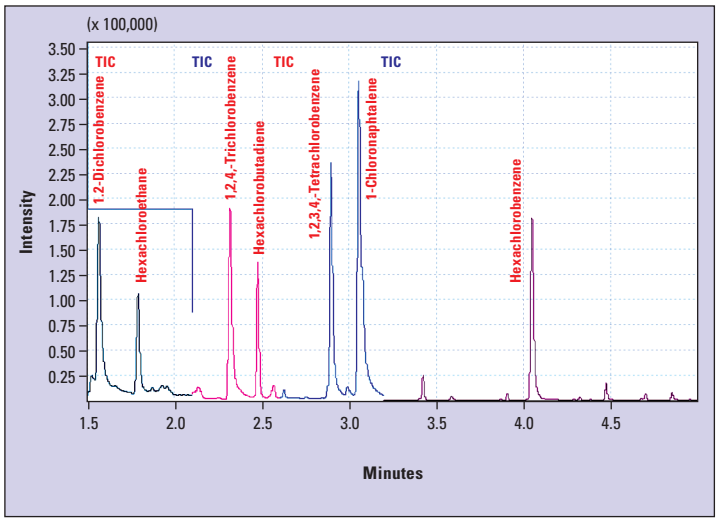


Figure 1: Fast GC-MS of chlorinated hydrocarbons; column Equity-5 5 m x 0.1 mm x 0.1 µm SPME fiber 100 µm PDMS

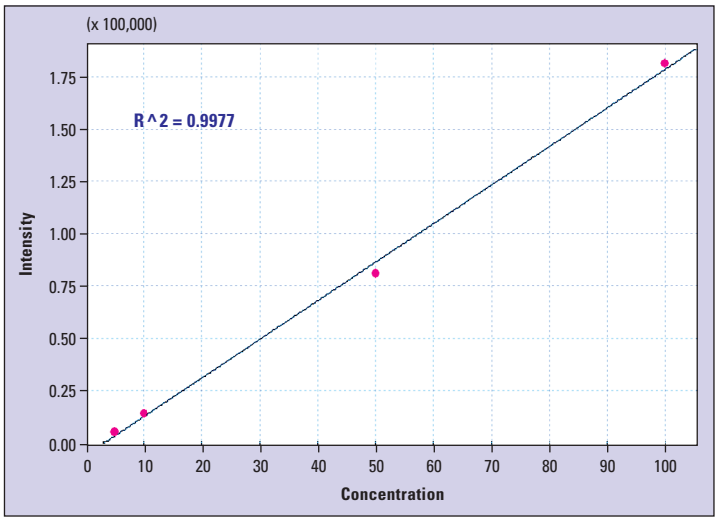


Figure 2: 1.2-Dichlorobenzene calibration (ng/L)

Rino Calori, A.R.P.A. Agenzia Regionale Prevenzione e Ambiente Regione Emilia-Romagna Sez. Provinciale di Bologna, Italy

Gas chromatography is an important method for the analysis of organic compounds. GC separates complex mixtures with high resolution, giving reliable qualitative and quantitative results. These need to be obtained in the shortest possible time in order to be cost effective for any laboratory.

A very good way of achieving high resolution separation while reducing analysis time is the use of narrow-bore columns with small inner diameters. In "conventional" GC, columns with 25 m length, an inner diameter of 0.25 mm and a film thickness of 0.25 µm are commonly used. If smaller inner diameters are used it is referred to as “Fast GC”. A decrease in column internal diameter reduces resistance to mass transfer in the gaseous phase. In this study a column with a length of 5 m, an inner diameter of 0.1 mm and a film thickness of 0.1 µm was applied.

Although the use of Fast GC columns has been possible for some time, their routine use is quite recent. This is because Fast GC requires not only special columns but also specific requirements for the GC equipment. Modern GC systems are now capable of supplying the extreme experimental conditions that narrow-bore columns necessitate: high inlet pressures, highly controlled split flows, rapid oven temperature heating/cooling and fast electronics for detection. When using a mass spectrometer for detection, the electronics need to be fast enough to have a high scan speed so as to obtain correct mass spectra. On the other hand the "inter scan set up time", a kind of "dead time", must be short, in order that sufficient data

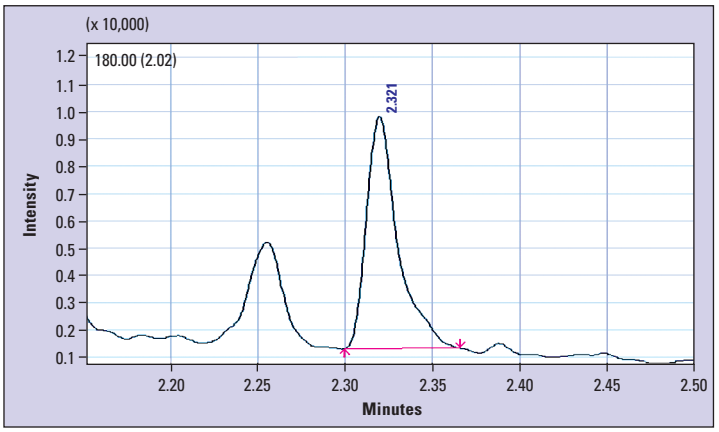


Figure 3: 1,2,4,-Trichlorobenzene m/z 180; 5 ng/L; S/N = 182.39

# in water samples

## resolution

points can be acquired for accurate quantitative analysis of a chromatographic peak.

### Solid Phase Micro Extraction (SPME)

Another limitation of an analytical method in terms of speed is often the sample preparation. In many cases the compounds of interest have to be extracted from the matrix, and further steps involve some cleaning or preconcentration. In this study for sample preparation the SPME (Solid Phase Micro Extraction) was used applying an autosampler with a special syringe. Inside the syringe the mounted fiber is coated with a material to adsorb the contaminants. The fiber is then exposed for a certain time to the sample (directly or to the headspace). The contaminants adsorb on the fiber. Afterwards the fiber is transferred to the hot injector of the GC and desorption takes place.

### Instrumentation

The instrument used in this study was a GCMS-QP2010. The GCMS-QP2010 contains the GC-2010, the first GC designed particularly for Fast GC with a high pressure electronic flow control, high split ratios as well as fast heating and cooling of the column oven. The mass spectrometer has a fast scan speed and a short inter scan set up time, making it possible to obtain acquisiton rates in scan mode of up to 50 Hz.

### Analysis of water contaminants

In this study the analysis of different contaminants in water samples with SPME and Fast GCMS was shown, ranging from volatile compounds (e.g. chlorinated hydrocarbons) to molecules with high boiling points, e.g. polycyclic aromatic hydrocarbons (PAHs). The applied method could be used successfully for all of these contaminants. Figure 1 shows an example of analysis of chlorinated hydrocarbons.

Figure 2 shows the calibration curve for 1.2-Dichlorobenzene from 5 to 100 ng/L. The fiber used for the extraction was a 100 µm PDMS 23GA; extraction time was 15 min.

Figure 3 demonstrates the excellent sensitivity of the method with a chromatogram of 1 ng/L of 1,2,4,-Trichlorobenzene in water. For good and reproducible quantitative results of fast separations like this, a high data acquisition rate is necessary.

Figure 4 shows the peak of hexachlorobenzene at approx. 4 min with a peak width at half height of approx. 1.3 sec, recorded with a data acquisition rate of 50 Hz (50 scans/sec).

Figure 5 shows the analysis of PAHs from water in less than 6 min with Fast GCMS and SPME. Even when using this complex separation, the resolving power of the 5 m column is sufficient.

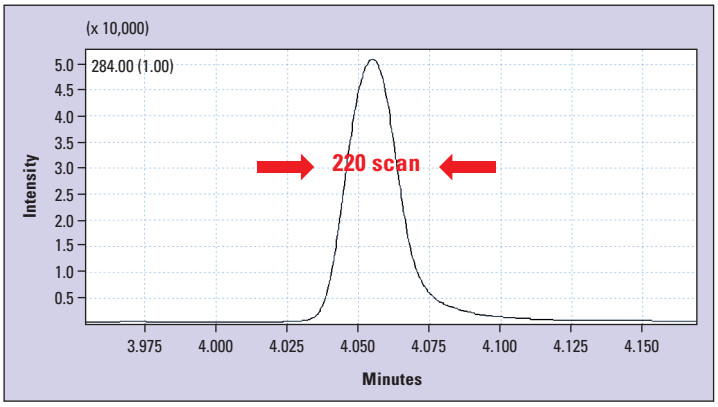


Figure 4: Hexachlorobenzene 50 ng/L; 50 scans/sec (50 Hz)

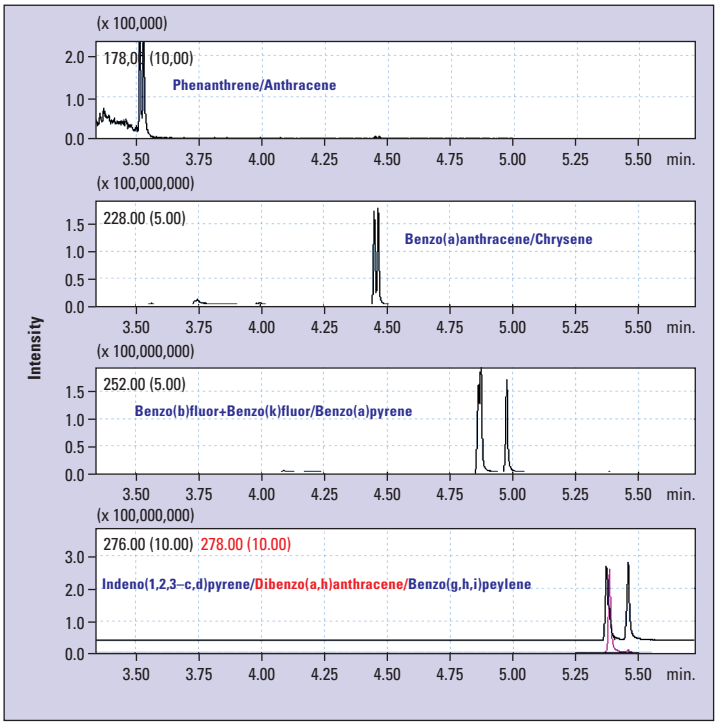


Figure 5: Analysis of PAHs according to EPA 525 Equity-5; column 5 m x 0.1 mm x 0.1 µm