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## HIGH-SPEED NARROW-BORE CAPILLARY GAS CHROMATOGRAPHY WITH MEDIUM-RESOLUTION MASS SPECTROMETRIC DETECTION

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Gas chromatography coupled to mass spectrometry is a powerful technique for identification and quantitation of unknown samples because it combines an efficient chromatographic separation with a universal and selective detector. During the last decade, the demand for faster analyses and increased sample throughput has increased interest in the possibilities of using narrow-bore columns. However, the use of these columns imposes stringent requirements on the instrumentation. The extra column band broadening caused by the instrumentation has to be minimized. This means that the input band width has to be very small and the acquisition rate of the detector signal sufficiently fast to enable accurate reconstruction of the chromatographic separation. For a mass spectrometer, the scan speed is limited by the physical laws governing mass analysis. Additionally, the faster a mass spectrometer is scanning, the less time is available for assessment of each mass peak in the spectrum. Hence, the sensitivity and the mass resolving power will be reduced. Another problem is that high *sensitivity* can only be obtained at the expense of *selectivity* (resolving power). Depending on the type of mass spectrometer, a compromise between scan speed, mass resolution and sensitivity has to be made. The possibilities and limitations of mass spectrometers with medium resolving power ( $1000 < R < 3000$ ) as sector instruments [1], reflectron time-of-flight (TOF) [2] and orthogonal accelerated time-of-flight (oa-TOF) in combination with narrow-bore columns have been investigated. Special emphasis was paid to scan speeds, mass resolving powers and quality of the mass spectra.

With conventional sector instruments scan speeds are limited to a few spectra per second. With the VG MasSpec, however, higher scan rates can be realized because this EBE instrument has a small, fast, fully laminated low inductance magnet. The maximum achievable scan speed in the full magnetic scan mode varied from three to ten scans over one decade per second, depending on the spectral resolving power (2000 and 3000, respectively). 20 scans per second could be acquired over a mass range of 40 to 200 Da at low resolving power. In the SIM mode, more than 50 ions could be monitored per second when the electrostatic analyser was used. However, the abundances of only 25 ions per second could be obtained by changing the magnetic field strength. The detection limits in the full scan mode are in the low pg range. In the SIM mode, detection limits as low as five to 50 fg are observed depending on the resolving power of the mass spectrometer and the sample complexity. With complex mixtures, detection limits for a given compound are limited by interferences from other compounds. By increasing the mass spectral resolution, interferences from ions of the same nominal  $m/z$  value are significantly reduced, thus enhancing the signal-to-noise ratio. An example is given in Figure 1.

The TOF mass spectrometer is inherently a very fast mass analyser. In principle, the time required to register a time-of-flight spectrum is less than 100  $\mu$ sec. In practice however, the resulting scan speed is significantly slower due to the computer time required for processing and storing the acquired data. Moreover, in order to improve the signal-to-noise ratio in the mass spectra, several transients (raw TOF-spectra) have to be accumulated. With the reflectron TOF, a scan speed up to 60 scans per second could be obtained. For reflectron

TOF mass analysers, the most successful approach to achieve mass independent focusing at the detector is obtained by using an ion mirror. With this set-up, mass resolutions up to 1500 (FWHM) could be obtained. Guilhaus *et al.* reported that by using orthogonal acceleration of the ions from a continuous ion beam, resolving powers up to 4000 (FWHM) were possible [3]. Additionally, the quality of the spectra of this instrument is much better. In contrast to reflectron TOF mass analysers, where the ions are stored in the ion source until an extraction voltage is applied, with oa-TOF the ions are extracted continuously from the ion source and gated orthogonally in the drift tube. In this way, chemical interactions between ions and molecules, which can cause distorted spectra, are avoided. Very good quality spectra are obtained. An example of a fast separation with reflectron time-of-flight mass spectrometric detection is given in Figure 2.

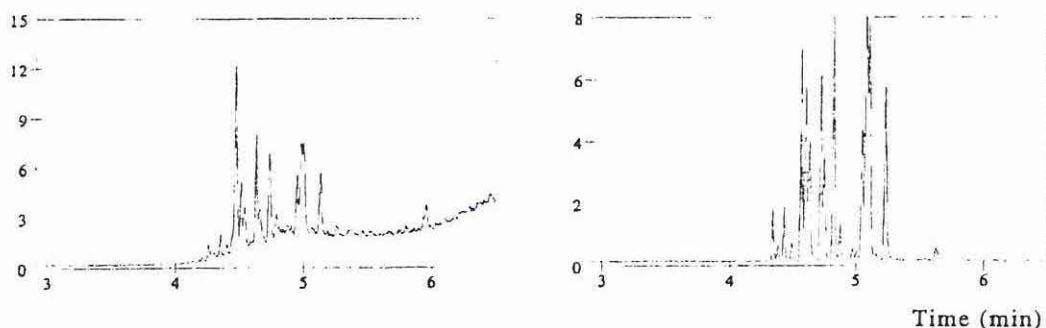


Figure 1.

Fast separation of a waste oil spiked with PCB's (Arochlor 1242). Ion current profile of  $m/z$  291.92 is shown at mass resolving power 300 (left) and 2000 (right). GC column: DB-1, L = 5 m, I.D. = 50  $\mu\text{m}$ .,  $d_f$  = 0.17  $\mu\text{m}$ .  $p_i$  = 11 atm.

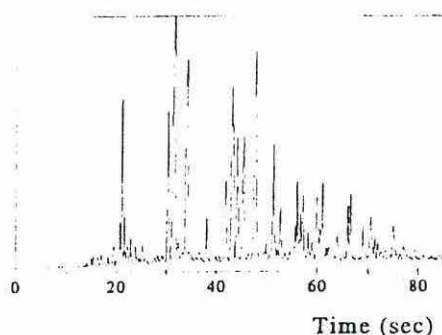


Figure 2.

Total ion current chromatogram of a gasoline sample obtained by narrow-bore GC coupled to a linear TOF mass analyser; column: OV-1, L = 6 m, I.D. = 50  $\mu\text{m}$ ,  $d_f$  = 0.05  $\mu\text{m}$ .  $T_{\text{oven}}$  = 25°C ballistically heated to 200°C,  $T_{\text{injector}}$  = 250°C,  $T_{\text{transfer line}}$  = 280°C,  $p_i$  = 12.7 bar, EI, Scan rate = 10.2 scans/sec, each scan is obtained by accumulating 128 transients.

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