Analysis of PCBs using N

Comprehensive GC x GC(qMS)

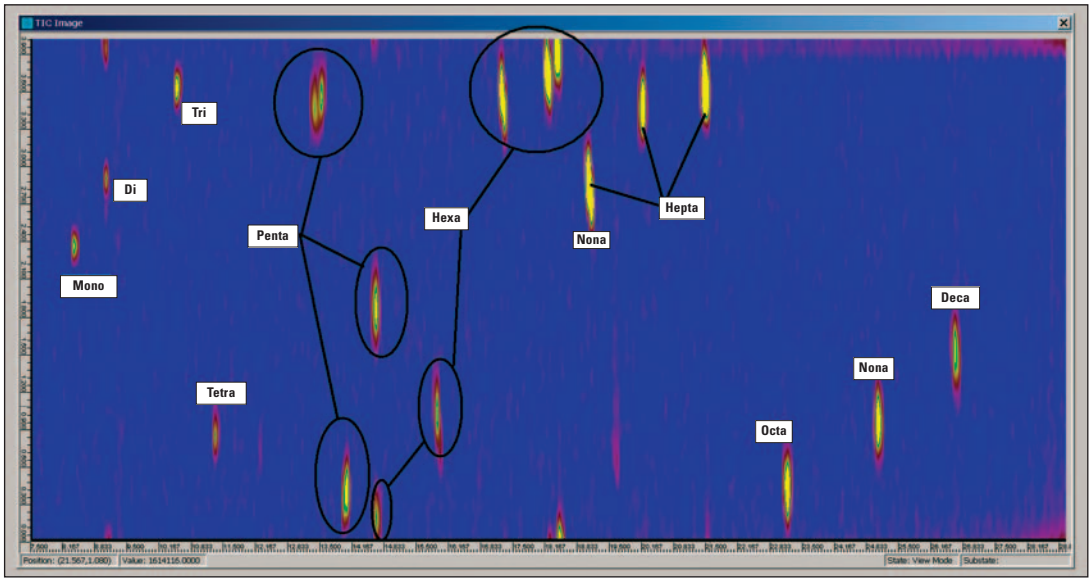

Figure 1: GC x GC(qMS) image of a PCB standard in NCI mode

Most reports are based on thermal modulation, in particular when applying MS as a detector. In this work a loop modulator (Zoex Corp.) was used where a two stage hot/cold jet modulation is achieved by only one cold and one hot jet.

In the first dimension a 30 m BPX-1 (0.25 mm ID x 0.25 µm df) was used coupled with a 1 m BPX-50 (0.15 mm ID x 0.15 µm df) in the second dimension. The modulation period was set to eight seconds. As the peak width of subsequent modulated peaks is about 250 ms at the base, the detector needs a high acquisition speed in order to supply enough data points across the peak. For this purpose, a high-speed quadrupole MS (Shimadzu GCMS-QP2010 Plus) was utilized, and the sampling frequency in full scan mode was set between 25 and 50 scans/s at a scanning speed of 10,000 amu/s.

The analysis of PCBs has been performed using EI and NCI ionization. In EI mode full scan and selected ion monitoring were performed. In Figure 1 the image of a PCB standard in full scan (NCI) is shown (34 - 500 amu).

As PCB congeners with a small number of chlorine atoms show only a fragment at 35 amu (NCl) while deca-PCB shows mainly an electron capture process (497.7 amu), this mass range was used. The concentrations were 25 pg each. Over each modulated peak (3 per substance) more than 10 data points were acquired giving enough precision for quantitative work.

Comprehensive GC x GC, invented by J. Phillips in 1989, has experienced rapidly increasing interest in the last years. The original application in the petrochemical field has been extended to many different topics. Very often coelutions with matrix peaks are observed and a high separation power is needed. Next to the enhanced GC x GC peak capacity, the hyphenation of GC x GC to mass spectrometry gives an extra dimension for easy identification of the compounds hiding under the contour plot displays used in GC x GC.

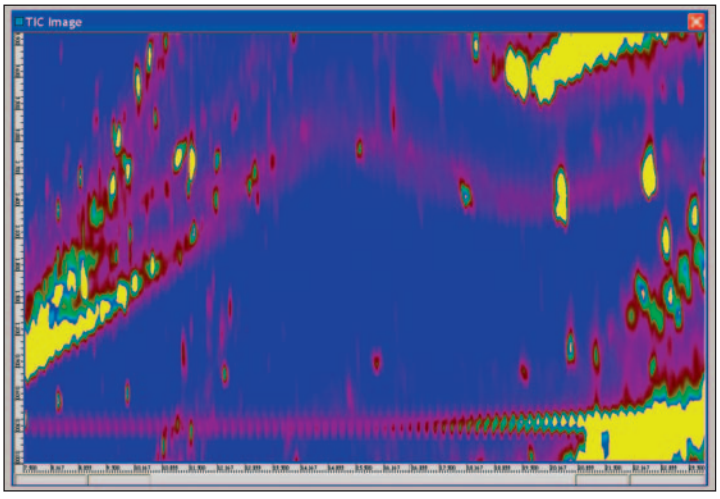

Figure 3: GC x GC(qMS) image of the bovine fat control sample in EI mode

CI and EI mode

The quantitative precision was tested by analyzing a QA standard (bovine fat extract spiked with PCB and dioxins, 10 - 250 pg (PCB)/g fat).

Outstanding selectivity

In Figure 2 the image of the QC standard is shown. Outstanding is the selectivity of the NCI mode as it shows a high response only for electrophilic compounds such as the PCBs while standard matrix signals are hardly visible. This can be seen in comparison with Figure 3 where the EI image of the bovine fat in full scan is shown. The TIC image is dominated by matrix signals.

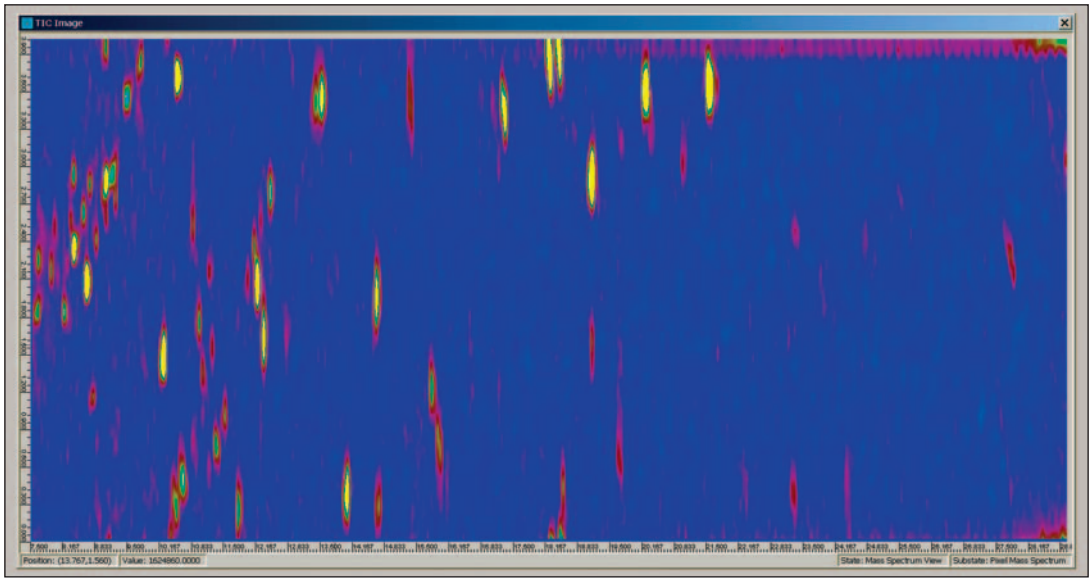

Figure 2: GC x GC(qMS) image of the bovine fat control sample in NCI mode

In order to raise PCB signal contrast to the matrix signals, extracted ion images have to be calculated. This is shown in Figure 4 for the mass range 324 - 329 amu (penta-chloro PCB). The corresponding congeners are indicated in the figure. As a result GC x GC(qMS) with a rapid scanning GCMS-QP2010 Plus instrument offers an alternative to TOF-MS, also for quantitative work, if the mass range is adjusted accordingly. Easy switching between the ionization techniques using a software switch allows high selectivity and flexibility.

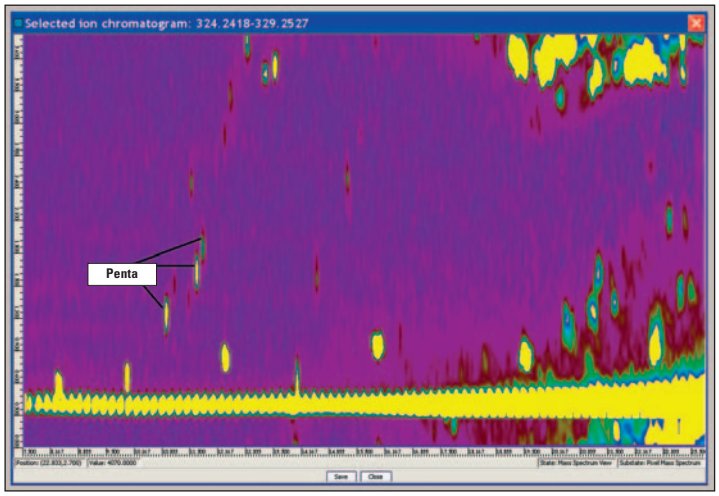

Figure 4: GC x GC(qMS) image of the bovine fat control sample in EI mode.
Ion range extracted: 324 - 329 amu.