# Comprehensive GCxGC(q)MS with negative chemical ionisation: PCBs in Bovine Fat

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## Introduction

The quantitative determination of PCBs in Bovine fat is shown using comprehensive GCxGC(q)MS using negative chemical ionisation [1]. Poly chlorinated Biphenyls (PCB) can be analyzed using NCI-GCMS with similar sensitivity like with electron capture detectors. In combination with comprehensive GCxGC(q)MS using a high speed quadrupol GCMS this is a highly selective tool for the quantitative determination of PCBs and Dioxins in matrices like Bovine fat. In order to produce thermal modulation the ZX1 modulator (ZOEX Corporation, USA) was used. In the first dimension a RTX-1 30 m,0.25 mm, 0.25 µm was coupled to a BPX-50 1 m, 0.15 mm, 0.15 µm. Figure 1 shows modulated fractions of EI data relative to 291.7 amu (tetra) in SIM mode.

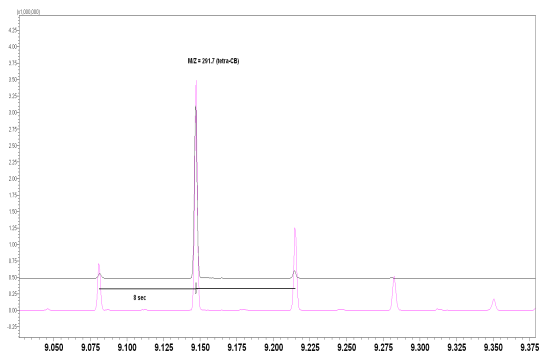


Fig. 1 modulated fractions of EI data in SIM mode

The modulation frequency was selected to be 8 sec. With matrices like Bovine fat in EI mode even in SIM operation it is difficult to locate PCB signals from the matrix. This is demonstrated with EI full scan data shown in figure 2. Here the GCimage software (ZOEX) was used to produce images from measured data. A bovine fat sample was spiked with 25 pg of PCBs. The MS was operated at 25 Hz with a mass range of 200 – 500 amu.

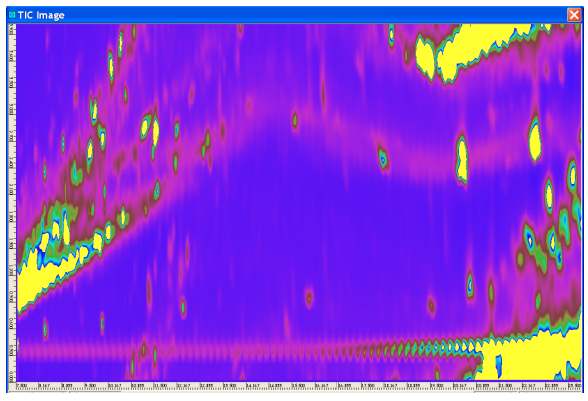


Fig. 2: Bovine fat in EI mode (full scan)

The image is dominated by matrix signals. In order to locate the signals relative to PCBs a selected ion image was produced. The corresponding mass range for the penta congeners are shown in figure 3. In this image blobs can be assigned to PCB signals while in the full scan image those blobs can hardly be identified.

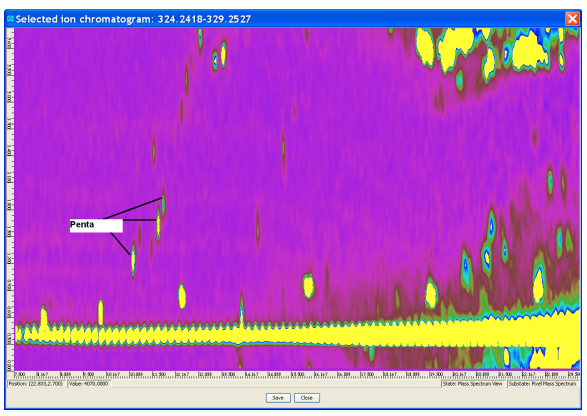


Fig. 3: Penta-PCB signal from extracted Ions

Using NCI (CH4) mode the situation is totally different. This is demonstrated with figure 4. The bottom data were recorded with the same sample of which the EI data of figure 2 were produced. For easy identification of PCB congeners a standard was measured under the same NCI conditions (top image figure 4). Except octa, nona and deca signals all PCB blobs visible in the standard can be easily identified in the fat sample. Comparing the two images from figure 2 and the one of figure 4 indicates the power of selectivity in NCI mode. As PCB28 shows only a main 35 fragment with NCI while higher chlorinated PCBs show also molecular ion response a full scan range from 34 – 500 amu was used. The data sampling was set to produce 10 data points across each modulated peak which allows quantitative determination.

## Conclusion

GCxGC(q)MS using NCI mode in combination with a fast response quadrupol MS detector allows selective and precise determination of PCBs in food related matrices.

## References

[1] Hans-Ulrich Baier* and J.-F. Focant*, (*) LCGC Europe, The Application Book March 09

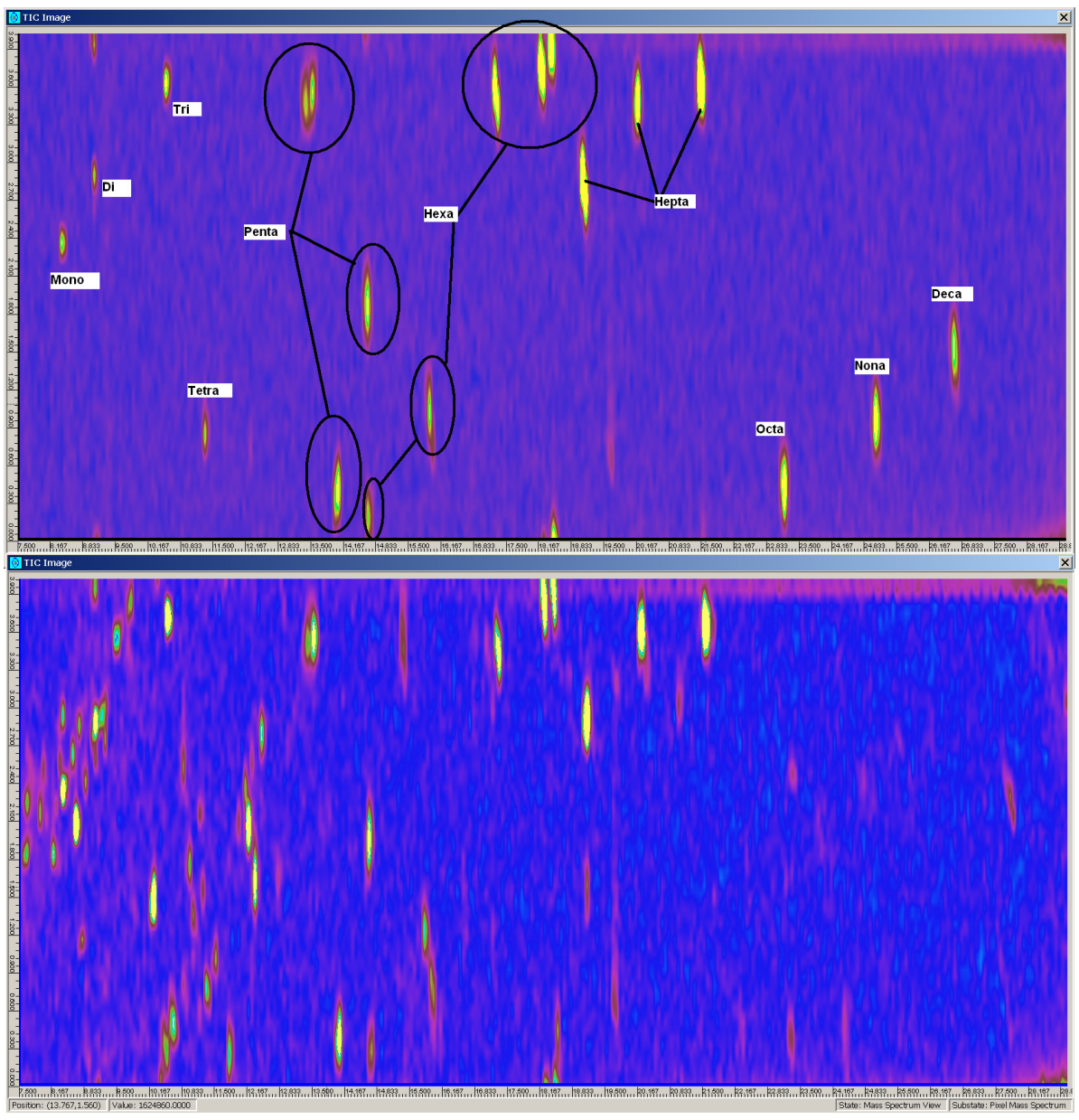


Fig. 4: Top: PCB Standard; Bottom: Bovine fat containing PCBs