

Comprehensive Two-Dimensional Gas Chromatography (GCxGC) coupled to Fast Scanning Quadrupole MS for PCB and Dioxin Analysis at Ultra-Trace Level.

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The 'Quest for the Holy Grail' in the 'dioxin' analysis area is dedicated to the development of reliable procedures that can offer congener-specific results on a short time scale. Such a procedure obviously has to fulfill strict QA/QC requirements as the ones listed in Eurachem analytical guidelines and EU or other Directives, but also has to comply with ISO17025 and/or GLP procedures. Each part of such a procedure, namely extraction, clean-up, fractionation, chromatographic separation, and physico-chemical (or biological) measurement, has to be fine tuned to its optimum capabilities.

Whatever the measurement method used, either physico-chemical or biological, the sensitivity has to be at the parts-per-trillion (ppt, 10^{-12}) level. This represents an extreme case of ultra-trace analysis and a real challenge in terms of analytical chemistry. Because of the low level of target analytes, large sample sizes have to be processed and extremely large amounts of matrix-related interferences have to be removed before one can even think about measurement.

As an alternative to the expensive and complex reference GC-HRMS approach, new methods are explored. We currently evaluate the capabilities of GCxGC coupled to fast scanning low resolution qMS in this field. The basics of GCxGC technology and its application for dioxin analysis are presented elsewhere¹. The two major advantages of GCxGC are the increased peak capacity and the signal enhancement resulting from zone compression in the modulator. Because the modulation process, GC peaks are very narrow (50-200 ms) and a high sampling rate detector is required for peak characterisation.

The qMS can scan at up to 20Hz in the selected mass range. This is sufficient to perform isotope dilution quantification under good QA/QC criteria. GCxGC-qMS data show good correlation with reference GC-HRMS data. Additionally to the routinely used EI for PCBs and dioxins, NCI is available on this instrument. Although NCI has been known to be subject to reproducibility problems from the past², recent improvements in NCI hardware is supposed to significantly improve response factor stability and eventually make NCI a robust alternative to EI for GCxGC. Extremely good sensitivity can be expected for such a coupling. This can motivate the use of NCI despite the reduced information on fragmentation and the reduced flexibility in terms of quantification procedures. The development of an NCI-GCxGC-qMS method for dioxins is under consideration and the preliminary data will be presented.

1. J.-F. Focant, A. Sjödin, D.G. Patterson Jr., The Encyclopedia of Mass Spectrometry, Hyphenated Methods. In: Niessen, W.M.A. (Eds.). Elsevier, Amsterdam, The Netherlands, pp 553, 2006.
2. M.D. Erickson, 1997. In: Analytical Chemistry of PCBs. CRC Press LLC: Boca Raton, FL.