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TARGETED AND COMPREHENSIVE TWO-DIMENSIONAL GAS CHROMATOGRAPHY WITH TIME OF FLIGHT MASS SPECTROMETRIC DETECTION FOR ENVIRONMENTAL POLLUTANTS ANALYSIS

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This study has developed a range of new high resolution GC separation methods, based on the use of cryogenic modulation and specifically employing coupled column (multidimensional) instrumental strategies as routine screening methods for environmental pollutants analysis. The key validation tool is mass spectrometric detection to confirm the resulting molecular separations afforded by each of the developed GC technologies. The results produced by the new approaches were compared with classical GC methods. In this study, several classes of pesticides, such as organophosphorus, organochlorine and synthetic pyrethroids pesticides, were analysed using a range of GC modes, including fast sequential targeted multidimensional GC, and comprehensive two-dimensional gas chromatography interfaced to a time of flight mass spectrometer (GC×GC-TOFMS). A longitudinal modulated cryogenic system (LMCS), which cryo-focuses and rapidly re-injects the effluent from the first column (^{1}D) to second column (^{2}D) was used for GC×GC and targeted analyses, to modulate the first dimension signal. The multidimensional systems employed two column operation with different phases providing some degree of separation orthogonality: ^{1}D was a 30m x 0.25 mm I.D., 0.25 μm d_f BPX5 (5% phenyl-dimethyl siloxane phase) capillary column, and ^{2}D was a 4 m x 0.1 mm I.D., 0.1 μm d_f HT-8 (8% phenyl polycarborane-siloxane phase) capillary column. Splitless injection was used, with the oven temperature programmed from 40 ^{o}C (1 min hold) to 130 ^{o}C, at 30 ^{o}C/min (8 min hold) then 8 ^{o}C/min to 300 ^{o}C (8 min hold). Results from targeted and GC×GC analyses were compared to those from single dimension GC analysis. Using the comparatively shorter ^{2}D column, along with fast GC conditions, enabled significant peak sharpening, and improved resolution and sensitivity compared to conventional GC. The ^{2}D column reported here, whilst rather short, is still longer than that often used for GC×GC (e.g. usually 1 m in length), however the 4 m column provides for more separation capacity than achieved in the shorter column arrangement when the targeted mode is chosen. Coupled with TOFMS, these multidimensional modes will therefore enable highly sensitive and definitive analysis of organohalogen pesticides in environmental samples. Specific applications of GC×GC-TOFMS and targeted MDGC will be presented, to exemplify the power of the new analytical techniques.