Dioxin measurements in food and feed beyond MRL regulation using GC×GC-TOFMS

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The measurement of dioxins and dioxin-like compounds in food and feed (ingredients) is an important task within the EU food safety policy. Both gold standard sector MS and triple quadrupole MS analyzers, performing in isotope dilution (ID) mode, are used as confirmatory tools for precise and accurate measurements under the EU legislation. The required sensitivity is achieved by performing in selected ion monitoring (SIM) or selected reaction monitoring (SRM), limiting the measurements to the specific target analytes for which EU maximum levels were established. Comprehensive two-dimensional GC coupled with time-of-flight MS (GC×GC-TOFMS), acquiring all masses within a defined mass range, has been investigated in terms of target and nontarget measurements of dioxins in food/feed samples. The new generation of TOFMS allows to reach ultra-trace level (fg) detection at high acquisition frequency. As a first approach, figures of merits were obtained on the 21 dioxins and dioxin-like compounds included in EU legislation. For TCDD, 10 fg on column could be detected with a S/N of 6. Pushing the detector voltage, we were able to reach 100 ag on column detection. For all 21 compounds the degree of linear correlation (goodness of fit, R2) was 0.97 to 0.99. We also considered several real food/feed samples to estimate a potential impact of matrix effects on performance. The non-targeted screening using the TOFMS in full scan mode allowed for detection of various congeners of polybrominated dibenzofurans (PBDFs), some of which have been shown to exhibit considerable AhR-mediated dioxin-like activity and may therefore be responsible for potential high response in the assay.