Title

Revisited Sample Preparation and Analyses for Dioxin Measurements in Biological Matrices

Authors and Affiliations

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Abstract

Measuring levels of regulated dioxins and PCBs in biological samples (e.g. food and human) is challenging because of the target levels (low to sub pg range) and the complexity of matrices. Proper sample preparation requires multi-step procedures to be used[1] in conjunction with high-end mass spectrometric detection[2]. Over the last 30 years, efforts have continuously made to reduce the cost and enhance the speed and efficiency of the overall procedures.

For sample clean-up, smaller size column sets have been proposed and allow to save up to 70% of the required cycle time and from 65% to 100% of the solvent used. Such approaches are still based on fractionation between planar and non-planar fractions, but without use of any chlorinated solvents. Serum samples, containing 0,5% of lipids, can thus be cleaned-up and fractionated in 20 min. When larger fat content matrices (food) are considered, either a manual fat digestion can be performed before hand or the automated instrument column set can be extended to a 7g fat capacity (total run time of 50 min).

For measurement, next to the recent DualData approach that significantly impact sample turnover by increasing productivity of more than 60%, faster GC runs can be performed for both planar and non-planar fractions. Main advantages of Fast GC are shorter total run time and higher sensitivity related to peak squeezing. Time saving with Fast GC is relevant, around 61%: both fractions can be quantified in 31 minutes instead of 80 (19 min for “dioxin” fraction, instead of 51, and 12 min for “PCB” fraction, instead of 29). Chromatographic resolution is comparable to the classical approach. Fast GC method is more sensitive than the classical method: S/N ratio is 55 (instead of 11,5) for 12.5 fg of 2,3,7,8-TCDD on Fast GC column. The full validation of Fast GC methods is undergoing.

1. J-F. Focant, C. Pirard, E. De Pauw, Talanta 63 (2004) 1101-1113.
2. J.-F. Focant, C. Pirard, G. Eppe, E. De Pauw, J. Chromatogr. A 1067 (2005) 265-275.

Author’s Biography

Authors Biography must not exceed a word limit of more than **200** words – Please include photo

Professor Jean-François (Jef) Focant is the Head of the Chemistry Department of the University of Liège in Belgium. He is leading the Organic and Biological Analytical Chemistry group of the mass spectrometry laboratory. Main research interests are coupling of sample preparation procedures, development of new chromatography strategies in separation science, hyphenation to various types of mass spectrometric detectors through multidimensional systems, and implementation of emerging strategies under QA/QC requirements for human biomonitoring and food control. Professor Focant has been active in the field of dioxin analyses for the last 20 years. He chaired the 31st International Symposium on Halogenated Persistent Organic Pollutants and POPs (DIOXIN2011) in Brussels in 2011. Known as a dioxin expert, he is also active in other areas of Separation Science such as characterization of complex mixtures of volatile organic compounds (VOCs) for medical and forensic applications as well as metabolomics. Recent investigations include characterization of cadaveric decomposition odors, screening for biomarkers of cancer by breath analysis, and plant combustion studies. Working on the hyphenation of state-of-the-art analytical techniques to solve practical analytical issues is what he enjoys to do.

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