November 7, 2013 (13:15-14:15)



VENDOR SEMINAR:

NEW APPROACHES FOR SAMPLE PREPARATION AND ANALYSIS BY MASS SPECTROMETRY

Determination of PCDD/F and PCB with the new automated sample preparation system DECS

T. Bernsmann¹, U. Möhlenkamp¹, P. Fürst¹, U. Aulwurm², M. Baumann²

¹Chemical and Veterinary Analytical Institute Münsterland-Emscher-Lippe (CVUA-MEL), Münster, Germany

²LCTech GmbH, Dorfen, Germany

The major PCDD/F and PCB contamination cases which have occurred in the food and feed chain during the recent years demonstrate the need for fast and high throughput methods to identify and confirm non-compliant samples and to trace back the contamination sources. For this purpose, a highly efficient clean-up procedure is required to purify raw extracts prior to the final analytical separation and quantification.

With LCTech we developed a new method which focuses on the automation of our well-proven sample clean-up procedure using the automated DECS System (LCTech). The principle of the method is based on the clean-up of the acid stable PCDD/F and PCB on silica gel coated with sulfuric acid. A separation of the PCDD/F from PCB is subsequently performed on a Florisil column. For further purification, both eluates of the Florisil column are cleaned up on two different carbon columns which contain a different active carbon. The PCB fraction can be split into a group of non-ortho PCB, and a fraction containing the mono- and di-ortho PCB. This is important because the non-ortho PCB fraction includes PCB 126 and 169 which were assigned the highest toxicity factors of the PCB (WHO 2005). If PCB 126 is measured along with the other PCB, it may cause interferences, which can lead to a substantial overestimation of the PCB 126 concentration depending on the separation column. It is therefore essential to separate the non-ortho PCB from the other PCB. The PCDD/F fraction also needs to be cleaned up on a carbon column to separate matrix substances which may potentially interfere especially with the tetra-, penta- or hexa-CDD/F traces in the mass spectrometric analysis. The advantage of the automated clean-up procedure is that the columns are easy to handle and can be bought filled and ready-to-use. The whole process of column conditioning and clean-up of the sample extracts is done automatically in 97 minutes. The recoveries for each congener determined in a proficiency test material with the automated sample preparation system are in good agreement with the legislation requirements laid down in Commission Regulation (EU) No 252/2012 and range between the requested limits of 60 % to 120 %. A comparison study was performed on quality-control samples and food samples of animal origin to evaluate the robustness of the new automated sample clean-up system and also to compare the quantification using GC-HRMS and GC-MS/MS. All results demonstrate the suitability of the automated LCTech sample preparation system and the GC-MS/MS system for a fast and reliable routine analysis of PCDD/F and PCB congeners in foodstuffs and feedstuffs that meet the requirements of European Union legislation.

Fast and Fully Automated Multi-Residue Pesticide Screening in Fruit / Vegetable Extracts using a GC-Q/TOF

Chris Sandy¹, Jennifer Gushue², Maithilee Samant², Hong Chen², Prerana Kapase², Joerg Riener³

¹ Agilent Technologies Ltd., Winnersh, UK

²Agilent Technologies Inc., Santa Clara USA

There is increasing demand to expand the scope of analytical methods employing GC/MS for food contaminants to include many hundreds of analytes. Although many laboratories have migrated their multi-residue pesticide methods from GC-MS to GC-MS/MS, as analysis suites become ever larger, concerns inevitably arise over instrument duty cycle. Another approach is to use a GC-Q/TOF system in full spectrum acquisition mode and to use the increased selectivity provided by high-resolution accurate mass measurements in order to measure, theoretically, an unlimited number of compounds. The additional sensitivity and selectivity provided by the GC-QTOF is particularly relevant for the analysis of pesticide residues in foodstuffs prepared by the QuEChERS sample preparation technique, where large amounts of sample matrix may be present in the final extract.

This presentation will introduce the All Ions workflow for the screening of pesticide residues in various foodstuffs using a GC-Q/TOF and electron impact ionization in combination with a retention time locked GC method, backflush for increased method robustness and a new accurate mass spectral database of pesticides.

The All lons workflow for accurate mass GC/Q-TOF is seamlessly integrated in to the Agilent Mass Hunter Qualitative software program and is similar to that of the All lons workflow used for screening contaminants in foodstuffs with Agilent's Q-TOF LC/MS instrument. This new, comprehensive workflow for GC/MS accurate mass data uses Agilent's proprietary Personal Compound Database and Libraries (PCDLs) to identify compounds using their precursor (reference) and fragment ions. The workflow includes the use of a new accurate mass PCDL that contains more than 700 pesticides and begins by looking for molecular evidence by isotope pattern matching, followed by examination of the co-elution profiles of fragment ions of target compounds. The introduction of a novel Co-elution Plot and Co-elution Score to visualize and express the covariance of fragment ions. Unlike competitive approaches, the Co-elution Score not only uses just the retention time, but also the entire chromatographic peak information (including peak width and symmetry) to determine covariance of fragment ions and the precursor (reference) ion.

The combination of retention time locking and accurate mass measurements by the All lons workflow provides more confidence in target compound identification and facilitates the reduction / elimination of potential false positives.

³ Agilent Technologies GmbH & Co., Waldbronn, Germany