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Waters

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VENDOR SEMINAR:

Approaching Routine Exhaustive Organic Contaminant Screening with Innovative LC/MS, GC/MS and Ion Mobility Technologies

A fast, quantitative and qualitative QECHERS based commodity independent multi residue analysis on a APGC-XEVO TQ-S (micro)

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A fast Quantitative and Confirmative Multi Residue Pesticide on a GCMS in different commodities in one generic method is often a challenge. At Nofalab we developed a commodity independent method for over 300 pesticides on a XEVO TQ-S in combination with a APGC. At Nofalab we employ the XEVO TQ-S in combination with a APGC (Atmospheric Pressure Gas Chromatography) already for over four years successfully. We developed a standard extraction method for all food and feed commodities based on QUECHERS. Using the benefits of the soft ionisation of an Atmospheric Pressure Chemical Ionisation technic (APCI) such as the APGC where M+ and MH+ ions are abundant with minor fragmentation. A high sensitivity is reached by using this ionisation technic. Defining MRM's on EI-ionisation is often based on fragments which are not specific for one compound. In contrast to EI-technics with APCI MRM's can be based upon the M+ and/or MH+ ions giving a better specificity. Based upon this excellent performance gave the opportunity to modify the QUECHERS-extraction in such a way that a stable chromatographic separation is obtained and matrix interference is minimized. This results in a commodity independent method for the determination of pesticides. Using a XEVO TQ-S this method was validated for 300 pesticides in a 30 minute injection to injection method. Recently Nofalab acquired a XEVO TQ-S Micro. Compared with the TQ-S this instrument has a significantly faster scanning rate. In ESI-mode the sensitivity of the instrument is significantly lower, but is APGC-mode the sensitivity is comparable since in contrast to the ESI all the effluent of the GC-column is extracted into the Mass Spectrometer. In this presentation the optimization of the APGC is discussed based on 25 pesticides reflecting several behaviours such as injection stability, resolution, M+, MH+ and fragment formation on à XEVO TQ-S and the new XEVO TQ-S micro. The presentation concludes with the results of a validation of a quantitative and qualitative method for over 500 pesticides (all with 2 or more MRM's) on à XEVO TQ-S Micro within à 30 minute injection to injection run. For most pesticides the LOD is less than one fifth of the required MRL's are reached, for some even lower than 0.001 mg/kg with acceptable recoveries of 70-120% and a RSD below 20% as required by SANCO

Using a dynamic trio to empower your screening assays

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A novel new screening strategy for monitoring pesticides in foodstuffs has been developed. Increasingly, laboratories are performing preliminary low-cost, high sample throughput screening methods, using high-resolution liquid chromatography hyphenated to a full scan mass analyser (e.g. Time-of-Flight). Typically the list of targeted compounds is significant. After detection, the identification process starts and relies on different criteria with associated tolerances, to ensure low false detection rates. It is known, many factors can influence mass spectra for LC-based methods. With analysis of complex samples, reliable identification can be unreachable. Ion mobility can reduce sample complexity, whilst increasing specificity. We will present a novel way to use ion mobility features routinely and illustrate how robust CCS values can efficiently reduce false detection rates.

UPLC ion mobility data covering a mass range from m/z 50 to 1200, was initially acquired for a series of solvent standard mixtures. These were utilized to generate mobility separated single component precursor/fragmentation spectra for the M+H+ or adducted species. An in-house CCS library was built and inserted into the scientific library, used for automatic software screening. Initial feasibility studies ware performed on five different instruments to assess the robustness of the CCS values generated. Further experiments were repeated on different days, during 10 months. Matrix and concentration studies were performed using blank and fortified matrices, processed with a methanolic extraction method, without any purification step. The overall CCS screening performance was evaluated using previous proficiency tests.

CCS values for 150 pesticides were measured by UPLC-HDMSE experiments using solutions in solvents at 100 ng/mL.These values were inserted in an in-house library with associated retention times, accurate mass and diagnostic fragments. Impact of analyte concentration on CCS was assessed by analyzing standards solutions from 1 ng/mL to 100 ng/mL. These results show that at any concentration there is no more than 2% difference with the values in the library. The same protocol was applied to samples with increasing matrix content (10 g, 40 g and 60 g of sample take) and the CCS generated, demonstrate no more than 2% error. These observation clearly indicate that targeted compound and sample concentration do not impact CCS, contrary to retention times and mass accuracy in LC-MS based methods. Furthermore, the reproducibility of CCS values were studied by comparing values obtained on different days, before and after a large batch of analyses, also data acquired for a period time of ten months. The RSDs calculated for these CCS are lower than 1%. In addition, CCS values generated from five different analytical platforms were of the order of 2% compared the library values. These findings raised the possibility that CCS can be used to help the identification process of targeted compounds, and were inserted in a routine workflow for pesticide screening. To test the method, a previous proficiency test was analysed and we will demonstrate how CCS used as an identification parameter, decrease false positive and most importantly avoids false negatives, even with less stringent screening parameters. We will also discuss, detection based on monoisotopic peak information, where CCS values within 2% tolerance confirm identification and avoid false negative reporting. The robustness of CCS is further illustrated when comparing a new travelling wave IMS-QToF ion mobility platform.

Novel Aspect:

CCS used as a new point of identification in routine full scan screening assays using travelling wave ion mobility platforms.