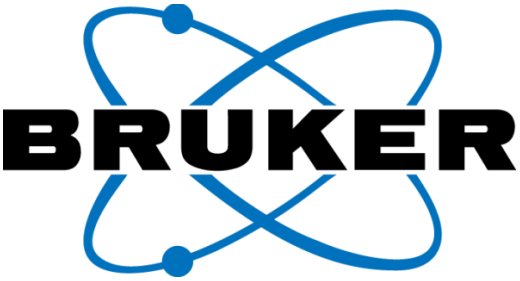
November 4, 2015 (7:30–8:30)



VENDOR SEMINAR:

Mass Spectrometric Solutions for the Analysis of Dioxins, Veterinary Drugs and Pesticides in Food and Feed

Analysis of dioxins according to Commission Regulations (EU) 589/2014 and (EU) 709/2014 by GC-Triple Quadrupole Mass Spectrometry

Gordon van't Slot, Bruker Daltonics, Germany

Already since 2012 it has been allowed to perform screening and quantitative measurements for dioxin analysis on low resolution mass spectrometers. In 2014 two new European Regulations for Food and Feed have been set into action allowing also confirmation of positive findings by TQ Mass Spectromety. 12 instead of high resolution mass spectrometry if you can show the compliance with some analytical criteria since 2012 already.

The following criteria most be fulfilled by the method:

- Each group requires at least one ^{13}C-labelled homologue per group of tetra- to octachlorinated PCDD/PCDF.
- Recovery of internal standards has to be between 30% and 140% for screening methods.
- Separation of the isomers 1,2,3,4,7,8 and 1,2,3,6,7,8-HxCDF has to be sufficient (<25% overlay peak to peak).
- The calibration curve has to cover the relevant concentrations starting from the level of detection.

Additional requirements which have been laid down with Regulations EU 589 and 709/2014 method requirements for confirmation of dioxins, and related compounds are

- Unit resolution for both analytical Quadrupoles
- Ion ratio tolerance <15%
- At least two significant precursor with one significant product ion each

Based on data from reference material in different matrices we are going to show, how modern high precision Triple Quadrupole mass spectrometers perform in these tasks. The compliance with all requirements is going to be shown on samples from food, feed and environmental with certified reference material provided by the Institute for Reference Materials and Measurements (IRMM). Pitfalls and possibilities are covered in this overview.

Screening and identification of veterinary drug and pesticide residues in food extracts by LC-QTOF Mass Spectrometry

Carsten Baessmann, Bruker Daltonics, Germany

Rapid, comprehensive screening for residues using full scan accurate mass has become a powerful tool in facilitating food safety monitoring. In addition to the high number of possible target compounds, the technique enables unknown screening and retrospective analysis. We describe the development of a solution for screening and quantitation of pesticide and veterinary drug residues in food matrices using a high-resolution LC-QTOF accurate mass system. Central parts of the solution is a newly developed software package (TASQ™: Target Analysis Screening and Quantitation) coupled with a high quality accurate mass and retention time database. TASQ software allows the simultaneous quantitation and confirmation of hundreds of pesticide residues by processing qualifier ions and subsequently applying a 'diagnostic ion' confirmation criterion to their detection. The pesticide database contains over 700 pesticides including, retention time, exact mass of precursor ions (MS mode) and broadband CID ions (MS/MS mode) that have been annotated with molecular formulae. We show the simultaneous quantitation and identification of approximately 500 pesticides in food extracts. The pesticides were matrix matched in a dilution series (0.1 µg/kg – 2000 µg/kg) using QuEChERS extracts: tomato, summer squash, potato and orange). The matrix matched calibrants were analyzed using a Bruker LC-QTOF system under a 15 minute reverse phase UHPLC gradient. Data acquisition was performed in alternating full scan and bbCID fragmentation modes. Automatic data evaluation was performed using TASQ processing software. For confident identification we use retention time, precursor accurate mass, isotopic pattern and up to 3 qualifier ions in full scan and 7 qualifier ions in bbCID acquisition. As result of this the LODs and LOQs of the 500 pesticides in the different matrices were determined. This approach can be also applied to other residues in food matrices, by changing the compounds contained in the database. As an example we present in a second study focused on the qualitative multi-residue screening for approximately 140 veterinary drugs in milk and fish.