REGULATED MYCOTOXINS IN CEREAL GRAIN FLOURS: A SIMPLIFIED SAMPLE PREPARATION AND LC-MS/MS METHOD

Nicola Dreolin and Sara Stead
Waters Corporation, Wilmslow, SK9 4AX, UK.

METHODS
- Powdered wheat spiked with I.S. mix and extracted with acidified MeCN:H2O
- Centrifugation at ~5300g and collect supernatant
- Dilution 1:10 with acidified water and injection
- Analysis on ACQUITY I-Class FL coupled with Xevo TQ-XS
- Validation following Reg. EU 519/2014 and SANTE/12089/2016
- Method applied to different cereal flours (oat, maize, rice, potato, tapioca)

RESULTS
- Washing solvents & mobile phase composition finely optimised to lower carryover of fumonisins whilst maintaining good separation and peak shape.
- The lowest detection capability was recorded for aflatoxins (instrumental LODs between 11 and 14 fg on column), while for others targeted analytes instrumental LOD were between 0.1 and 22.5 pg on column.
- The use of 13C-labelled internal standards showed greater analytical performance compared to the external standardisation.
- Recoveries at three spiking levels ranged from 90 to 115%, whilst RSDr were below 10% in all cases (n=3).
- A mix of mycotoxins was spiked to oat and to a mixture of different flours at the LOQ level. Recoveries met the criteria set by the European Regulation (81-114%).

DISCUSSION
Matrix effects ranged from >30% signal suppression for nivalenol, to >100% signal enhancement for ochratoxin A. This finding clearly justifies the use of the isotopically labelled internal standards to aid with quantitative accuracy, improve repeatability and to negate the effects of different matrices thus allowing the use of a calibration curve prepared using solvent standards.

CONCLUSIONS
- The method is “fit-for-purpose” for the quantitative analysis of EU regulated mycotoxins in dried cereal grain commodities such as wheat, oats, maize, rice, buckwheat-based food products.
- The excellent sensitivity of the TQ-XS and the selectivity of the MRM acquisition mode, made possible the extreme simplification of the sample treatment procedure.
- The incorporation of 13C-labelled internal standards within the analytical workflow leads to enhanced method performance and is therefore recommended as an efficient approach to correct for both matrix effects and the inevitable analyte losses during the sample preparation.
- Internal standardisation allows the analyst to avoid the use of matrix-matched calibration and work with solvent calibration curves for accurate quantitation.

References