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Liquid Chromatography – High Resolution Mass Spectrometry Method for Monitoring of 17 Mycotoxins in Human Plasma for Exposure Studies

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Highlights

- First LC-MS assay for 17 mycotoxins was developed and validated in human plasma.
- Liquid-liquid extraction minimized absolute matrix effects to improve quantitation.
- 0.02% acetic acid enhanced sensitivity of mycotoxin analysis in negative ESI.

Mycotoxins are secondary metabolites produced by filamentous fungi. Primary route of human exposure to mycotoxins is the intake of the contaminated food. Minimizing mycotoxin exposure is important for population health, as their chronic toxic effects have been associated with kidney and liver diseases, some types of cancer and immunosuppression. The objective of this work was to develop and validate a multi-class mycotoxin method suitable for exposure monitoring of mycotoxins in human plasma. A sensitive liquid chromatography – mass spectrometry method was developed for 17 mycotoxins: nivalenol (NIV), deoxynivalenol, fusarenon X, 3-acetyldeoxynivalenol, 15-acetyldeoxynivalenol, T-2 toxin, HT-2 toxin, aflatoxin B1, aflatoxin B2, aflatoxin G1, aflatoxin G2, zearalenone, α-zearalenol (α-ZOL), β-zearalenol, zearalanone, α-zeranoland, and β-zeranol. The method relies on three-step liquid-liquid extraction with ethyl acetate to eliminate the need for immunoaffinity extraction and minimize ionization matrix effects. Chromatographic separation of mycotoxins, including all isomers, was achieved with pentafluorophenyl column and water/methanol mobile phase. Mycotoxin detection and quantitation were performed using high-resolution mass spectrometry on LTQ Velos Orbitrap, in both positive and negative electrospray ionization (ESI(+) and (ESI(-)). The use of 0.02% acetic acid as mobile phase additive for ESI(-) resulted in significant increase in ionization efficiency ranging from 1.7 to 26 times for mycotoxins that ionize better in ESI(-). The optimized method was validated according to FDA guidance procedures. LOQs of all mycotoxins ranged from 0.1 to 0.5 ng/ml, except NIV which resulted in LOQ of 3 ng/ml because of low extraction recovery of this highly polar mycotoxin. Mean intra-day accuracy ranged from 85.8% to 116.4%, and intra-