

From antimicrobial residues to mycotoxins and plant toxins: A robust (U)HPLC-MS/MS multi-class method for food and feed

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Data analytics in food and agricultural

Over the past four years, I have evaluated the robustness of our fully validated in-house multi-class method. It quantifies over 900 mycotoxins and plant toxins within 40 minutes, using two injections— in positive, and negative ionization mode (20 minutes each). Our laboratory analyzes approximately 7,500 samples annually using this method, resulting in around 20,000 injections. Given this high throughput, assessing the method's robustness was of particular interest, with potential for significant gains in efficiency.

The first phase of this work focused on expanding the method's scope to include veterinary drugs. We developed and validated an HPLC-MS/MS method covering over 15 drug classes and 140 analytes for application in milk and chicken feed. The feasibility of 'dilute-and-shoot' approach was also investigated, supporting the potential for a unified method targeting both veterinary drugs and mycotoxins in feed and milk. This approach was further validated through an interlaboratory comparison with Queen's University Belfast (QUB). For most analytes, routine laboratory accuracy criteria were met—exceeding 83% in milk and ranging between 50% and 60% in chicken feed—based on apparent recoveries within the 60–140% range. Extraction efficiency criteria were met for more than 95% of analytes in milk and over 80% in chicken feed. Intermediate precision complied with the SANTE/11312/2021 guideline, with RSDR values below 20% for 80–90% of analytes in both matrices. Furthermore, for all analytes with established MRLs in milk, the achieved LOQs were below the respective regulatory limits.

The second phase focused on speeding up the method by testing alternative instrumental setups, including fast polarity switching vs. separate injections, UHPLC vs. HPLC, and shortened LC gradients (11 vs. 21 minutes). Six methods were developed and validated for cereal-based food in line with SANTE/11312/2021 criteria. All workflows showed comparable matrix effects, repeatability, and within-lab reproducibility—except for reduced repeatability at low spiking levels. LOQs increased fourfold compared to the original 2 × 21-minute method, reflecting the trade-off for a fourfold increase in throughput. The main outcome was a rapid 11-minute method for >900 analytes in only one run.

Relative matrix effects were assessed in both phases, as this is particularly important for evaluating method accuracy across different batches of the same sample type. Therefore, various samples of chicken feed, milk, and muesli were analyzed throughout the study.