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OPTIMIZATION OF A MODIFIED QuEChERS METHOD FOR THE DETERMINATION OF TETRACYCLINES IN FISH MUSCLE BY UHPLC-MS/MS

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Highlights

- Tetracycline determination in fish samples by UHPLC–MS/MS in less than 4 min.
- Optimization of a modified QuEChERS procedure as sample treatment.
- Validation of the method in panga and salmon with recoveries higher than 80 %.
- LOQs were lower than 4.4 μg kg^{-1} with RSD values lower than 18.5 %.

Abstract

In this work a sample treatment based on a modified QuEChERS method combined with ultra-high-performance liquid chromatography–tandem mass spectrometry (UHPLC-MS/MS) was proposed to determine the residues of five common tetracyclines in fish muscle samples. The separation was achieved in less than 4 min and the analytes were detected in electrospray ionization in positive mode (ESI+) with multiple reaction monitoring mode. Parameters affecting the extraction step, such as the amount of sample and EDTA-McIlvaine buffer and extraction solvent volumes, were optimized by means of experimental design. In order to obtain the lowest matrix effect, parameters affecting the clean-up step by dispersive solid phase extraction (dSPE), were also studied. Under optimum conditions, matrix effect was lower than |15|% in all cases. Limits of quantification were lower than 4.4 μg kg^{-1} for the compounds in the selected samples, being in compliance with the current legislation. The precision, expressed as relative