## **Accepted Manuscript**

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PII: S0731-7085(18)30195-X

DOI: https://doi.org/10.1016/j.jpba.2018.03.029

Reference: PBA 11860

To appear in: Journal of Pharmaceutical and Biomedical Analysis

Received date: 30-1-2018 Revised date: 12-3-2018 Accepted date: 14-3-2018

Please cite this article as: Angel Grande Martínez. David Moreno-González, Francisco J.Arrebola Liébanas, Antonia Garrido Frenich, Ana M.García-Campaña, **OPTIMIZATION** OF **MODIFIED QuEChERS** Α METHOD FOR THE DETERMINATION OF TETRACYCLINES IN FISH MUSCLE BY UHPLC-MS/MS, Journal of Pharmaceutical and Biomedical Analysis https://doi.org/10.1016/j.jpba.2018.03.029

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## ACCEPTED MANUSCRIPT

# 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<sup>-1</sup> 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  $|4.4~\mu g~kg^{-1}$  for the compounds in the selected samples, being in compliance with the current legislation. The precision, expressed as relative

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