

23rd Congress of Chemists and Technologists of Macedonia

BOOK of ABSTRACTS



11 October 2014

Chemical Society of Macedonia

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AC 013

IN-HOUSE VALIDATION OF UHPLC TANDEM MASS SPECTROMETRY METHOD FOR DETERMINATION OF ANTIHELMINTICS IN LIVER APPLYING MATRIX DISPERSIVE SAMPLE PREPARATION

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Helminth infections are controlled through the administration of anthelmintic agents for almost 50 years, which are essential for maintaining high weight gains and reproductive performance in livestock. The widespread availability of these veterinary medical products has led to concerns that they are often unnecessarily administered to animals leading to an increased risk of residues thereof. The EU has set maximum residue limits (MRLs) for anthelmintics and their metabolites in animal products. The MRL values range from 100–1000 $\mu\text{g kg}^{-1}$, depending on the compound.

In this work a modified Ultra-high-performance liquid chromatography coupled to triple-quadrupole detector (UHPLC-TQD) was employed for analysis of five anthelmintic compounds: albendazole, flubendazole, fenbendazole, levamisole and closantel. Sample preparation was performed applying matrix dispersive clean-up (QuEChERS) for fatty matrices. An in-house method validation study was successfully conducted according to the Decision 657/2002/EC requirements. The validation levels were in reference to the established MRL value for each compound. For a sustainable quality control of the extraction procedure each sample was spiked with isotopically labeled albendazole D_3 and flubendazole D_3 .

Matrix-matched calibration curves were with linearity better than 0.99. The obtained detection and quantification limits (LODs and LOQs) were in the range 12–25 $\mu\text{g kg}^{-1}$ and 38–75 $\mu\text{g kg}^{-1}$, respectively. Recovery of analytes was typically in the 70–95 % interval, with repeatabilities and reproducibilities less than 15 %. The decision limit ($\text{CC}\alpha$) was between $\text{MRL} + 11.9\%$ and $\text{MRL} + 16.8\%$ and the detection capability ($\text{CC}\beta$) was between $\text{MRL} + 23.7\%$ and $\text{MRL} + 33.5\%$ for the range of anthelmintics investigated.

The validation data confirmed that the modified UHPLC-TQD method has suitable performances for analysis of anthelmintics in liver samples according to the regulatory requirements.

Keywords: anthelmintics, UHPLC/MS, liver, validation, decision limit