

Ss. CYRIL AND METHODIUS UNIVERSITY IN SKOPJE
FACULTY OF VETERINARY MEDICINE - SKOPJE



BOOK OF ABSTRACTS

*9th International Scientific Meeting
Days of Veterinary Medicine 2022*

22 - 25 September 2022
R. of North Macedonia

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**VALIDATION OF UHPLC-MS/MS METHOD AFTER MODIFIED QUECHERS
SAMPLE PREPARATION FOR MONITORING OF PESTICIDE RESIDUES
IN BOVINE MILK ACCORDING TO EC/2018/555**

Elizabeta Dimitrieska-Stojkovikj*¹, Aleksandra Angeleska¹, Dushica Koceva¹,
Biljana Stojanovska-Dimzoska¹, Goran Stojkovic², Gordana Ilievska¹,
Risto Uzunov¹, Zehra Hajrulai-Musliu¹

¹Ss Cyril and Methodius University from Skopje, Faculty of veterinary medicine-Skopje,
Institute for food, Lazar Pop-Trajkov 5-7, 1000 Skopje, North Macedonia

²Ss Cyril and Methodius University from Skopje, Faculty of natural sciences and
mathematics, Institute for chemistry, Arhimedova br. 5, 1000 Skopje, North Macedonia

The EU Regulation EC/555/2018 has foreseen implementation of monitoring program for selected pesticide residues in bovine milk, aimed for further dietary exposure assessment. Therefore, since 2019 the required pesticide substances were introduced into the National Residue Control Program for animal origin food in North Macedonia. For this purpose, UHPLC-MS/MS method was optimized and validated after modified QuEChERS (quick, easy, cheap, effective, rugged, and safe) sample preparation. The MS/MS method was optimized for 19 pesticide substances and metabolites: carbaryl, carbofuran, fenvalerate, indoxacarb (carbamate pesticides), cypermethrin, deltamethrin, bifenthrin permethrin (pyrethroids), malathion, parathion, diazinone, dichlorvos, chlorpyrifos, chlorpyrifos-methyl, pyrimiphos-methyl (organophosphates), famoxadone, fipronil, including metabolites fipronil sulfone, fipronil sulfide. For each compound the two most sensitive MRM transitions were selected for further determination. Analysis was performed on Waters UHPLC-MS/MS system consisted of H-class UHPLC and Xevo TQ-S micro triple quadrupole detector, in ESI+ and ESI- mode. Chromatographic separation was performed on C18 column with 100 mm length and 1.7 μm particle diameter, using gradient elution with water and methanol, both modified with formic acid and ammonium formate (0.1 % v/v and 5 mmol/L, respectively), at flow rate of 0.45 mL/min. The optimized method was validated according to DG SANTE Document 2017/11813. Validation parameters were linearity, limit of quantification (LOQ), precision, accuracy, and matrix effect. The determined LOQs from the concentration level at which S/N ratio was higher than 1:10, were in the range 1.0 – 8.3 $\mu\text{g/L}$, and were equal or below the MRL values for the pesticides of interest. Linearity was determined in the range 1-100 $\mu\text{g/L}$, obtaining five-level calibration curves with correlation coefficients ≥ 0.99 for all tested analytes. Method precision was tested at two concentration levels - 10 and 50 $\mu\text{g/kg}$, except for carbofuran, for which the target levels were 1 and 10 $\mu\text{g/kg}$. The calculated relative standard deviation (RSD) ranged from 6.3 to 15.5 %. The method accuracy, estimated from the recovery at two concentration levels, was in the range 73.8 – 107.3 %. Matrix effect did not have significant influence on the analyte signal suppression or enhancement, thus solvent

standards could be used for calibration. The method performance characteristics were satisfactory according to the SANTE document requirements (LOQ \leq MRL, precision \leq 20 %, and recovery 70-120 %). With this, it was proven that the proposed method was suitable for analysis of the defined scope of substances, even for pyrethroids, for which gas chromatography was considered to be a more appropriate analysis technique.

Key words: pesticide residues, monitoring, bovine milk, UHPLC-MS/MS, validation