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





Joint meeting 5<sup>th</sup> International Vet-Istanbul Group Congress & 8<sup>th</sup> International Scientific Meeting Days of veterinary medicine - 2018

# BOOK OF Abstracts

23-27 September 2018 Ohrid, R. of Macedonia Book of Abstracts

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5<sup>th</sup> International Vet-Istanbul Group Congress 8<sup>th</sup> International Scientific Meeting Days of veterinary medicine - 2018 23-27 September 2018, Ohrid, Republic of Macedonia Dear Colleagues,

It is my grea 5<sup>th</sup> International V Days of veterinary 23<sup>th</sup> to 27<sup>th</sup> Septem

This year, in c University, we'll b academic cooperat (VEE) partners, by the public good.

The Faculty o together outstanding experts in the field or researchers, veterina doctoral students, off in the veterinary educ following topics: anin and food safety.

The program for l retain its usual popular student papers and pos authors, whose researc of the International Sci electing 117 papers fro followed by two poster invited speakers for the talks.

A successful conferplace, where ideas about debated, which will ope enjoyment.

On the behalf of the ( in Macedonia.

We look forward to w

8<sup>th</sup> Internation 23-27

12.50-13.00 Effects of Black Cumin addition to rations on the performance parameters in laying hens Tahir Balevi, Uçkun Sait Uçan, Oğuzhan Kahraman, Abdullah Özbilgin	
	10.30-10.4
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Moderators: Prof. Zehra Hajrulai-Musliu and Prof. Ali Aydin	
Scientific papers – Oral presentations	10.50-11.00
14.00-14.10Evaluation Of The Quechers Method And Gas Chromatography–Mass Spectrometry For The Analysis Fipronil And Its Metabolites In Egg Products Aleksandra Tasic, Ksenija Nesic, Tijana Mitrovic, Nebojsa Vukovic	11.00-11.10
<ul> <li>14.10-14.20 Optimization And Validation Of Lc-Ms/Ms Method After Quechers Sample Preparation For Determination Of Fipronil And Its Metabolites In Eggs And Chicken Muscle</li> <li>Elizabeta Dimitrieska-Stojkovikj, Dushica Koceva, Aleksandra Angeleska, Biljana Stojanovska-Dimzoska, Gordana Ilievska, Risto Uzunov, Stefan Jovanov, Zehra Hajrulai-Musliu, Dean Jankuloski</li> </ul>	11.10-11.20
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14.30-14.40 The Evaluation Of Histamine Levels In Carp (Cyprinus Carpio) Samples Collected From Afyonkarahisar, Turkey Acaroz Ulas	11.30-11.40
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**Oral Presentation** 

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Oral Presentation

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### OPTIMIZATION AND VALIDATION OF LC-MS/MS METHOD AFTER QUECHERS SAMPLE PREPARATION FOR DETERMINATION OF FIPRONIL AND ITS METABOLITES IN EGGS AND CHICKEN MUSCLE

Elizabeta Dimitrieska-Stojkovikj\*, Dushica Koceva, Aleksandra Angeleska, Biljana Stojanovska-Dimzoska, Gordana Ilievska, Risto Uzunov, Stefan Jovanov, Zehra Hajrulai-Musliu, Dean Jankuloski

> Food Institute, Faculty of Veterinary Medicine – Skopje, Ss Cyril and Methodius University in Skopje, Lazar Pop-Trajkov 5-7, Skopje, Republic of Macedonia

Fipronil is high-effective insecticide authorized for use in plant protection practices with limited application to some plants. However, the 2017 fipronil crisis in Europe revealed its unauthorized application at food producing animal, i.e. laying hens, whereas the detected concentrations in eggs exceeded the maximum residue level (MRL) of 5 µg/kg. The food safety authorities in European Union and food business operators have implemented significant monitoring of eggs for residues of fipronil. When ingested by animal organism fipronil metabolizes mainly in fipronil sulfone, and to less extent in fipronil desulfinil. In order to control the fipronil abuse and assure safety of the produced food, it is very urgent to develop a simple, sensitive, and reliable method for determining residues of fipronil and its metabolites in chicken eggs and muscle. The proposed method within this study involves the QuEChERS sample preparation, followed by reversephase ultra high – performance liquid chromatography - tandem mass spectrometry with electro-spray ionization in negative mode, for detection and quantification of fipronil and its metabolites fipronil sulfone and fipronil desulfinil in eggs and chicken muscle. Method performances were determined through the validation procedure following the SANTE recommendations. The method was validated for the linearity, sensitivity, precision, reproducibility, and expanded measurement uncertainty. The method linearity was obtained within the range 1-50  $\mu$ g/L with correlation coefficients >0.99 for all three compounds studied. The limits of quantification were determined form 2.3 to 4.8  $\mu$ g/kg, meaning that the analytes could be quantitatively determined below the assigned MRL value. The method precision and accuracy were assessed at levels of 5 and 25  $\mu$ g/kg in five replicates, fulfilling the SANTE criteria for relative standard deviation ( $\leq 20$  %) and recovery between 70 and 120 %. The expanded measurement uncertainty (k=2) around the level of interest (5  $\mu$ g/kg) was estimated with values ranging between 9.1 and 14.4 % for egg matrix and 8.6 and 14.0 % for muscle. Additionally the method reliability was confirmed by successful participation in proficiency testing scheme PT - 502/2018organized by RIKILT – Wageningen. The within-laboratory validation and the external quality control provided evidence that the developed method was fit for the purpose of control illegal treatment of chicken with fipronil insecticide.

Key words: fipronil, fipronil sulfone, LC-MS/MS, eggs, chicken muscle

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