UNIVERSITI TEKNOLOGI MARA

METHOD DEVELOPMENT AND VALIDATION FOR MULTI-CLASS VETERINARY DRUGS DETERMINATION IN POULTRY FEED USING QuEChERS-dSPE-ULTRA PERFORMANCE LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY (UPLC-MS/MS)

AHMAD TALHAH BIN SUHAIMI

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Chemistry)

Faculty of Applied Sciences

April 2021

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	:	Ahmad Talhah bin Suhaimi
Student I.D. No.	:	2016782091
Programme	:	Master of Science (Chemistry) – AS756
Faculty	:	Applied Sciences
Thesis Title	:	Method Development And Validation For Multi-Class Veterinary Drugs Determination In Poultry Feed Using QuEChERS-dSPE-Ultra Performance Liquid Chromatography Tandem Mass Spectrometry (UPLC-MS/MS)
Signature of Student	:	.....
Date	:	April 2021

ABSTRACT

The use of veterinary drugs has received global attention in recent years especially due to the development of antimicrobial drug resistance. It is essential that suitable methods of analysis are available to control this problem. A method for the determination of 31 veterinary drugs belonging to 9 different classes in chicken feed has been developed. The method was based on QuEChERS (quick, easy, cheap, effective, rugged, and safe) extraction equipped with d-SPE clean-up. The sample preparation included ultrasonicate the mixture of acetonitrile, methanol and McIlvaine buffer followed by phase separation with $MgSO_4$:NaCl addition. The detection and quantification were performed using single analytical run by ultra-performance liquid chromatography tandem mass spectrometry (UPLC-MS/MS) operating in both positive and negative multiple reactions monitoring (MRM). Validation was performed in accordance with the international guidelines. Acceptable results regarding linearity of the method, limit of detection (LOD) and limit of quantification (LOQ) were achieved for 23 of 31 investigated substances. The LODs ranged from 0.05 mg/kg to 1.12 mg/kg and LOQs from 0.17 mg/kg to 3.74 mg/kg. Average analyte recoveries ranged from 83.7% to 109.9%. The relative standard deviation value for repeatability ranged from 0.43% to 9.91%. Based on matrix effect analysis, only six compound which are FTD, FZD, CBR, RPM, TBL, IPZ passed for quantification with standard solution calibration. The validation results demonstrate that the described LC-MS/MS method provides sensitive, repeatable and reliable for safety monitoring and controlling veterinary drug use in chicken feed.

ACKNOWLEDGEMENT

Firstly, I want to thank Mrs. Marni Sapar, Research Officer at Veterinary Public Health Lab for her guidance during my research work. I also want to thank member of her department for their assistance and knowledge.

My appreciation also goes to my beloved Assoc. Prof. ChM. Dr. Muhd Fauzi Safian for his support and supervision. Special thanks to Nahdzatul Syima Muslim, my senior colleague for her help and encouragement on finishing my thesis writing.

Finally, this thesis is dedicated to my parents for their vision and determination to educate me. This piece of victory is dedicated to both of you.

TABLE OF CONTENTS