EVALUATION OF A LLE EXTRACTION SYSTEM FOR DETERMINING PESTICIDE RESIDUES IN MILK USING DOEHLERT DESIGN

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Introduction: Among all parasites that affect livestock in Brazil, the tick is by far considered the most important being responsible for serious damage to the cattle industry. At present, the control of ectoparasites of veterinary importance still relies on the use of chemicals being insecticides pyrethroids (PYR), particularly cypermethrin, widely used for this purpose. Currently, 84 different veterinary products containing PYR as active substance are registered in Brazil for use in milk producing cows. Milk is a complex and variable matrix, containing large amounts of protein and fat. To achieve satisfactory GC analysis and improved selectivity for ECD, the previous isolation of the PYR from the milk is required in order to get almost fat-free extracts. In this way, univariate optimization may be a time-consuming and labor-intensive procedure, requiring several experiments. On the other hand, the number of experiments needed is lower in multivariate optimization, reducing costs and waste generation. Doehlert matrix designs can be used for multivariate optimization, involving two, three, or more factors (variables). By using this approach, more information is obtained, allowing the identification of critical points at each step of the optimization process.

Objective: In the present study, Doehlert matrix design is proposed for multivariate optimization and development of a method of extraction followed by clean-up using LLE-PLT(Liquid-Liquid Extraction-Precipitation at Low Temperature) for seven analites (Deltamethrin/DEL, Cyfluthrin/CYF, Cyhalothrin/CYA, Cypermethrin/CYP, Permethrin/PER and Fenvalerate/ FEN).

Material and Methods: GC analysis was achieved using a Trace Ultra gas chromatograph equipped with a ^{63}Ni electron-capture detector (GC-ECD). For separation, a OV-1701 (Ohio Valley, 30 m × 0.53 mm × 0.5 μm film thickness) was used. The injector and detector temperature were 240 °C and 340 °C, respectively. Nitrogen was used as carrier gas with the flow of 1 mL/ min. The injection volume was 1.0 μL in splitless mode for all standards and samples. Doehlert design was evaluated using a three-level factorial design with a central point involving three variables (agitation time, acetonitrile volume used in LLE and in PLT). A Doehlert design (Doehlert 1.0) developed at Laboratório de Quimiometria Teórica e Aplicada of UNICAMP/Brazil and MATLAB 7.0 were used in chemometric analysis.

Results and Discussion: Analyte recovery was used as response to evaluate the Doehlert design. For all analytes a quadratic profile for response surfaces was observed. Acetonitrile volume used in PLT procedure demonstrated not signficance. The results shown good recoveries for all analytes (between 90-97%).

Conclusion: Multivariate optimization revealed the best conditions for LLE-PLE method. The main advantages this strategy are low reagent consumption, low waste generation and robustness prevision on method proposed.

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