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A reliable high-performance liquid chromatography with ultraviolet detection for the determination of sulfonamides in honey

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ABSTRACT

The sulfonamides are stable chemotherapeutics used against the bacterial disease affecting bees, known as American foulbrood (Bacillus larvae), so their residues could appear in the honey of treated bees. Their presence at a concentration above the limit value is a potential hazard to human health. Brazilian authorities have included in the National regulatory monitoring program, the control of the three most widely used sulfonamides in honey production, i.e., sulfathiazole, sulfamethazine and sulfadimethoxine. A method for the determination of residual sulfonamides in honey, using sulfapyridine as an internal standard has been developed, optimized and validated. Some changes were implemented on current available methodologies for the analysis of sulfonamides in honey in order to adopt such procedures to Brazilian honey samples. Sulfonamides were extracted from honey with dichloromethane after dissolution with 30% sodium chloride, and cleaned up with solid phase extraction on Florisil columns. The eluate was analyzed by high-performance liquid chromatography with ultraviolet detection. The limit of detection was determined at 3 $\mu g\,kg^{-1}$, 4 $\mu g\,kg^{-1}$ and 5 $\mu g\,kg^{-1}$ for sulfathiazole, sulfamethazine and sulfadimethoxine, respectively with average recoveries of 61.0% for sulfathiazole; 94.5% for sulfamethazine and 86.0% for sulfadimethoxine at the 100 $\mu g\,kg^{-1}$ level. As the final step of validation procedure, the analysts were submitted to a blind spiked sample prepared by the quality assurance officer which results were successfully obtained regarding recovery and deviations.

1. Introduction

Sulfonamides play an important role as effective chemotherapeutics of bacterial and protozoal diseases in veterinary medicine. In honeybees, sulfonamides are used for the prevention and treatment of a disease known as American foulbrood [1]. The drugs are administered to honeybees as a sodium salt dissolved in sucrose solution [2] in combination with dihydrofolate reductase inhibitors of the diaminopyrimidine group [3]. In bee colonies sulfonamides are used for treatment of foulbrood and this may lead to residues being present in honey. The use of sulfonamides for treatment of honeybees is illegal in many countries. Brazil has set a provisional MRL of 100 $\mu g\,kg^{-1}$ for sulfonamides in honey [4].

Based on the method described by Horie et al. [2], we modified the clean-up conditions and introduced an internal standard, sulfapyridine (SPY), with the objective to improve the recovery of the sulfathiazole (STZ). We decided to intro-

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