

Evaluation of QuEChERS Method for Analysis of Cypermethrin Residue in Cow's Milk

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The presence of veterinary drug residues in food, especially of pesticides used for cattle disease control, can affect public health and international trading of food products if maximum residue levels are above those stipulated by FAO and WHO. Milk is one of the most complete food for humans, because it contains nutrients required for growth and development of newborns. However, milk quality can be endangered due to the presence of contaminants, especially of cypermethrin pesticide, which is widely used in Brazil to treat lactating dairy cows, mainly against ticks, flies, lice and dermatobia. Methods for monitoring veterinary drugs, for example pesticide residues, should be rapid, have small number of analytical steps, use few reagents in small amounts, be specific, and be sensitive enough to enable detection at very low levels. Anastassiades *et al.* [1] developed an approach which they dubbed quick, easy, cheap, effective, rugged, and safe (QuEChERS). The method involves extraction of the analyte with MeCN partitioned from an aqueous matrix using anhydrous MgSO₄ and NaCl, followed by a dispersive-SPE cleanup with MgSO₄ and primary secondary amine (PSA).

The aim of this study was to evaluate QuEChERS method in combination with gas chromatography-mass spectrometry (GC-MS) for the determination of cypermethrin residue in bovine milk.

The following steps were undertaken: (1) weigh a 10 g milk sample into a centrifuge tube; (2) add cypermethrin; (3) add 10 mL of MeCN, 4 g of MgSO₄ and 1 g of NaCl in each tube, and centrifuge at 3000 rpm for 1 min; (4) transfer 1 mL of MeCN extract to a 1.5 mL minicentrifuge tube for dispersive SPE using 50 mg of PSA + 50 mg of C18 + 150 mg of MgSO₄; (5) mix the extract, and centrifuge at 6000 rpm for 1 min; (6) transfer 0.5 mL of the supernatant to an autosampler vial for analysis by GC-MS. The volume analyzed was 1 μ L and the oven temperature program was set at 150°C - 15°C min⁻¹ - 270°C (7 min). The mass spectrometry was operated in the SIM mode.

The method resulted in extracts that contained the target analyte, with recovery values within the ANVISA [2] requirements, from 70% to 120%, with relative standard deviations below 20%. Detection and quantification limits were 0.025 and 0.075 mg kg⁻¹, respectively. It is possible to analyze cypermethrin in milk below the maximum residue limit of 0.10 mg kg⁻¹.

The proposed method is rapid and inexpensive, and reduces consumption of organic solvents, which are toxic to health and to the environment. The method may be used as a screening procedure for analysis of cypermethrin residue in milk.

[1] Anastassiades, M; Lehotay, S.; Stajnbaher, D; Schenk, F.J. *J. AOAC International*, **2003**, *86*, 412-431.

[2] ANVISA, **2006**. <http://e-legis.bvs.br/leisref/public/showAct.php?id=25129>

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