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## INTRODUCTION

Diflubenzuron (DFB) is a pesticide that can be used in cattle breeding as an insect growth regulator. Among the flies that can be controlled by the use of this compound is the horn fly.

Diflubenzuron is usually fed to cows and is effective against larvae of Diptera breeding in manure.

## OBJECTIVES

Validate QuEChERS sample preparation method for the analyses of DFB in cattle tissues.

Evaluate the presence of DFB residue in three tissues of cattle (kidney, liver and muscle) treated orally with a commercial product containing the DFB for a 12-month period.

## METHODS

### Extraction and clean-up

Extraction and clean-up were performed based on QuEChERS sample preparation. Extraction was performed using acetonitrile, anhydrous MgSO<sub>4</sub> and NaCl. Clean-up was performed using PSA, C18 and anhydrous MgSO<sub>4</sub>.

### HPLC

Chromatographic analysis was performed by HPLC with UV detection (254nm) using a C18 column and mobile phase with 50% water, 25% acetonitrile and 25% methanol (v/v/v) at a flow rate of 1 mL/min. The injection volume was 20µL.

### Samples

Six bulls were treated with a DFB product available in the Brazilian market (Difly S3<sup>®</sup>, Champion Farmoquímico Ltda.) The product is commercialized as a powder containing 3% of DFB and was given daily to the animals for a 12-month period (daily intake of 30mg of DFB/animal).

The animals were male, crossbreeds, with ages from 20 to 36 months, weighing between 450 and 500kg. The animals were slaughtered according to Brazilian sanitary regulations and the tissue samples (kidney, liver and muscle) were collected, refrigerated and transferred to the laboratory where they were homogenized, frozen and stored until analysis.

Blank samples of kidney, liver and muscle were collected from an animal, with the same characteristics, that was not treated with DFB.

## RESULTS

The validation of the analytical method involved the following parameters: linearity, precision (repeatability), accuracy and determination of the limits of detection (LOD) and quantification (LOQ). Table 1 presents the results obtained.

**Table 1.** LOD, LOQ, recovery (R) and relative standard deviation (RSD) for DFB in cattle tissues.

Tissue	LOD (mg/kg)	LOQ (mg/kg)	Spike level (mg/kg)	R (%)	RSD (%)
Kidney	0.02	0.04	0.05	95.7*	13.1
			0.1	83.1**	11.1
			0.2	94.9**	18.6
Liver	0.03	0.05	0.05	106.0*	15.8
			0.1	88.1**	9.6
			0.2	98.6**	16.9
Muscle	0.02	0.04	0.05	98.3*	12.8
			0.1	85.1**	13.9
			0.2	100.7**	15.5

\*n=7; \*\*n=5

Recoveries obtained for the different tissues ranged from 83.1% to 106.0% and the RSD ranged from 9.6% to 18.6%. DFB showed linear response in concentrations from 0.03 to 0.60µg/mL. These results show that the analytical method used is suitable for the analysis of DFB residues in 3 cattle tissues (kidney, liver and muscle).

Diflubenzuron residue was not detected (<LOD) in any of the kidney, liver and muscle tissue samples analysed.

## CONCLUSIONS

The use of the DFB commercial product, in the manufacturer's suggested doses for achieving a proper insecticide effect, may result in cattle's liver, kidney and muscle considered safe regarding the presence of diflubenzuron residues.

The analysed samples are in accordance with the MRLs established by Codex Alimentarius for DFB in edible offal and meat fat (0.1mg/kg).

### Reference

Anastasiades, M.; Lehotay, S.J.; Stajnbaher, D.; Schenck, F.J. *Journal of AOAC International*, 86: 412-431, 2003.

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