

FLUVALINATE RESIDUES IN GREEK HONEY AND BEESWAX

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ABSTRACT

Tau-fluvalinate has been the most frequently used varroacide in Greece over the last decade. Application of the commercial apicultural product Apistan at the recommended dose does not seem to contaminate honey seriously. On the other hand, application of the non-authorized for apicultural use, agricultural formulations, using various application techniques and doses may result in the presence of residues and influence the quality of bee products.

The objective of this work was to evaluate the level of fluvalinate residues in Greek honey and beeswax. A GC-ECD analytical method was used with LOQ values of 1 µg/kg and 100 µg/kg for honey and beeswax analysis respectively. A total of 58 honey samples were analyzed over the last 7 years (1994-2001). 60% of the samples had no measurable residues (<1 µg/kg, LOQ), while concentrations up to 3.82 µg/kg were found in the rest of the samples. Fluvalinate levels in honey did not increase over the years. Beeswax samples examined referred to broodcombs (n=66), honeycombs (n=22), wax cappings (n=12), and wax foundations (n=14). Concentrations determined in beeswax were at ppm levels ranging 0.4-30.14 for broodcomb wax, 0.8-14.34 for honeycomb wax, 1.2-4.24 for wax cappings and 0.36-3.48 for foundations.

Key words: fluvalinate, residues, varroacides, honey, beeswax

INTRODUCTION

Tau-fluvalinate has been included into Annex II of Council Regulation (EEC) 2377/90, since application of the apicultural formulation (Apistan) as recommended, does not contaminate honey seriously. Repeated application of the substance or misuse, lead to residue accumulation in beeswax, which then represents a source of residues for honey. Residues in beeswax are not regulated in the European Union, while in USA an MRL of 6 mg/kg has been set. The objective of this work was to evaluate the level of fluvalinate residues in Greek honey and beeswax, as the pyrethroid has been the most frequently used varroacide in Greece over the last decade.

MATERIALS AND METHODS

SAMPLING

58 honey samples were obtained from the retail market or from various beekeepers over the years 1994 to 2001. Wax samples were provided by beekeepers between 1998-2000 and consisted of 66 broodcombs (22 beekeepers X 3 samples each), 22

honeycombs (11 X 2) and 12 wax samples produced from wax cappings (6 X 2). Finally, 14 wax foundations were purchased from the retail market.

SAMPLE PRETREATMENT

Combs were melted in a steamer without pressure (15 min); the wax was collected in a beaker, left to solidify and analyzed.

ANALYTICAL METHODS

The analytical methods used for fluralinate residues determination in honey and wax have been previously described [6, 7]. Briefly, for honey samples, extraction and cleanup was carried out using C₈ SPE cartridges with dichloromethane as the elution solvent. For wax samples, the method consisted of extraction by liquid-liquid partitioning on Extrelut® columns, followed by cleanup on a Florisil cartridge. The final extracts were analyzed by capillary gas chromatography with electron-capture detection (GC-ECD). The mean recoveries of the methods for honey and wax were 90.25 ± 0.85 and 77.50 ± 1.57 %, respectively, while the limits of detection and determination were 0.07 and 1 µg/kg for honey, and 23 and

100 µg/kg for wax, respectively. Typical chromatograms are given in Figure 1.

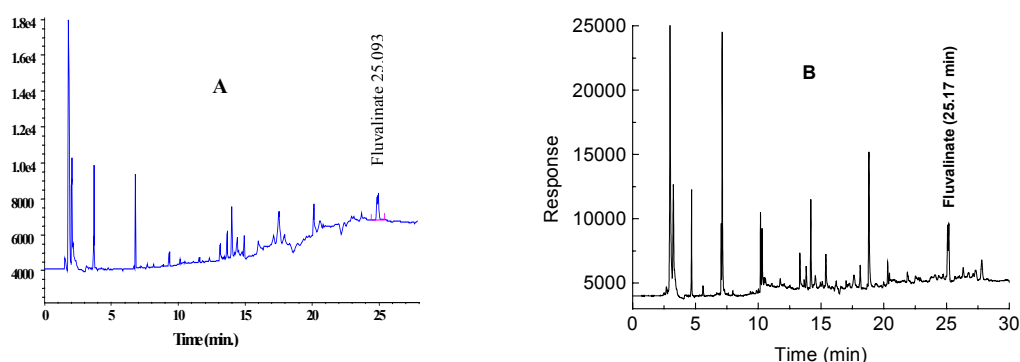


Figure 1. GC chromatograms (A) of a honey sample spiked with fluralinate at 5 µg/kg, and (B) of a wax sample spiked with fluralinate at 800 µg/kg.

RESULTS

Fluralinate residues in honey and beeswax are presented in Figure 2 and Table 1 respectively. Residue concentrations in honey were low, ranging < 0.07 – 3.82 µg/kg, with 60% of the samples having < 1µg/kg. Despite of the extensive use of the varroacide, concentrations in honey did not increase over the years 1994 to 2001, though only samples analyzed in 1994 were found with no detectable residues (< 0.07 µg/kg, LOD).

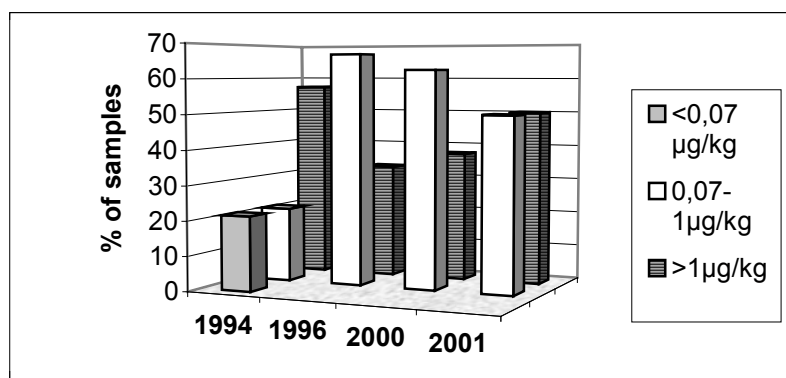


Figure 2. Fluvalinate residues in honey.

Table 1. Fluvalinate residues in beeswax.

Concentration (mg/kg)	% of samples			
	Broodcomb wax	Honeycomb wax	Wax cappings	Foundations
<1	11	5	-	43
>1-2	18	36	33	21
>2-5	30	36	67	36
>5-10	24	14	-	-
>10-15	9	9	-	-
>15-30.14	8	-	-	-

Concentrations determined in beeswax were high following the order: broodcombs > honeycombs > foundations. None of the samples analyzed was found free of residues (< 0.1 mg/kg, LOQ). Regarding wax cappings it should be noted, that, though concentrations in general were lower than the ones measured in broodcomb wax, samples provided by 3 beekeepers had concentrations similar to the ones found in broodcomb wax supplied by the same beekeepers. For the rest of the samples comparison was not possible. This was probably due to the fact that colonies were under treatment. In such cases bees both produce contaminated wax and continuously distribute the varroacide throughout the hive by their legs and bodies, causing thus high contamination to the cappings. Our results regarding the presence of fluvalinate residues in Greek honey and beeswax are similar to those from other European countries [1, 2, 3, 4, 5, 8].

CONCLUSIONS

- Fluvalinate levels found in honey were very low. However, concentrations determined in beeswax were at ppm levels. This is due to the lipophilic character of the substance and its extensive use by the beekeepers, most of who prefer to apply the agricultural formulations in various doses.
- Residue levels in wax should be controlled, since high levels influence its quality and induce the risk of honey contamination.

- Flunalinate concentrations measured in Greek honey and beeswax are similar to those reported in other European countries.

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