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Short Communication

Gas chromatographic determination of flumetralin in tobacco

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ABSTRACT

A method for the determination of flumetralin (Prime) residues in tobacco is presented. The procedure is adapted from that of Reif and Moser, which was proposed for the determination of organochlorine pesticide residues in tobacco. Improvements were made by the use of an overflow in the Soxhlet extractor and by replacing the packed column with a fused-silica capillary column. The suggested method is rapid and simple to use.

INTRODUCTION

Flumetralin [N-(2-chloro-6-fluorobenzyl)-N-ethyl-α,α,α-trifluoro-2,6-dinitro-*p*-toluidine], a substance with plant growth regulating activity [1], was introduced under the trade-mark Prime by Ciba-Geigy (Basle, Switzerland). It is applied as a topical treatment for the control of sucker growth on various types of tobacco. The product can provide full season control if applied within 24 h after topping. It has a local systemic effect, and therefore rain occurring later than 2 h after spraying does not reduce its effectiveness, in contrast to Off Shoot T (fatty alcohols). Further, as it is not translocated throughout the plant, as occurs with maleic hydrazide, less residues can be expected in tobacco leaves [2]. A comparison of sucker-control agents performed on Maryland tobacco showed that flumetralin was the most effective local systemic suckercide agent, with a persistent effect [3].

Flumetralin was registered in the U.S.A. for use on tobacco in 1983 and incorporated into the recommended sucker control program [4]. A maximum residue level of 20 ppm of flumetralin in the finished product was set in the German

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Hoechstmengenverordnung [5]. As no rapid and simple method for the determination of flumetralin residues on tobacco appears to have been published, the gas chromatographic (GC) method for organochlorine pesticides described by Reif and Moser [6] was tested for flumetralin and was found to be applicable.

EXPERIMENTAL

Chemicals

n-Hexane for pesticide analysis, used as an extraction solvent, was obtained from Fluka (Buchs, Switzerland) and Florisil (60–100 mesh) from Siber Hegner Rohstoff (Zürich, Switzerland). Flumetralin and mirex pure pesticide were supplied by Dr. Ehrenstorfer (Augsburg, F.R.G.).

Florisil conditioning

The Florisil was heated overnight in a muffle furnace at 500–550°C, then cooled in a desiccator without a desiccant. For deactivation, the Florisil was placed in a round-bottomed flask on a rotary evaporator. With constant agitation, 5 wt.-% of doubly distilled water was added and the flask rotated for 1 h. Before use, the deactivated Florisil was left to equilibrate for at least 48 h in a rightly closed glass container. After each use, the glass container must be tightly reclosed in order to prevent modification of the water content deactivated Florisil.

Gas chromatography

A Hewlett-Packard HP 5890 gas chromatograph equipped with a nickel-63 electron-capture detector, a Model 7673A automatic sampler and a Model 3393A integrator was used. A DB-5 fused-silica capillary column (30 m × 0.32 mm I.D.), film thickness 0.25 μm, was obtained from J & W Scientific (Folsom, CA, U.S.A.). The temperature programme was as follows: the initial temperature (70°C) was held for 1 min, then raised to 150°C at 20°C/min and to 270°C at 3°C/min, and then held at 270°C for 15 min. The carrier gas was helium at a flow-rate of 4 ml/min and the make-up gas was nitrogen at a flow-rate of 30 ml/min. Splitless injection was used. Under these conditions, the retention times of flumetralin and mirex were 23.5 and 34.2 min, respectively.

Extraction/clean-up

The Soxhlet extractor shown in Fig. 1 was filled with 5 g of deactivated Florisil as the lower layer and 5 g of deactivated Florisil mixed with 5 g of ground tobacco as the upper layer.

A 60-ml volume of *n*-hexane and 3 ml of a 4 μg/ml mirex internal standard solution in *n*-hexane were placed in a 250-ml round-bottomed flask. The flask was connected to the Soxhlet extractor and the *n*-hexane was heated with an electrical heater to boil the solution. The Soxhlet extractor was regulated to give a reflux rate of *ca.* 250 ml/h. The level of *n*-hexane above the tobacco had to be kept constant. The overflow prevented drying of the distillation flask. The extraction time was 4.5 h. After extraction, 0.5 μl of the extract was injected directly into the GC system. A typical chromatogram is shown in Fig. 2.

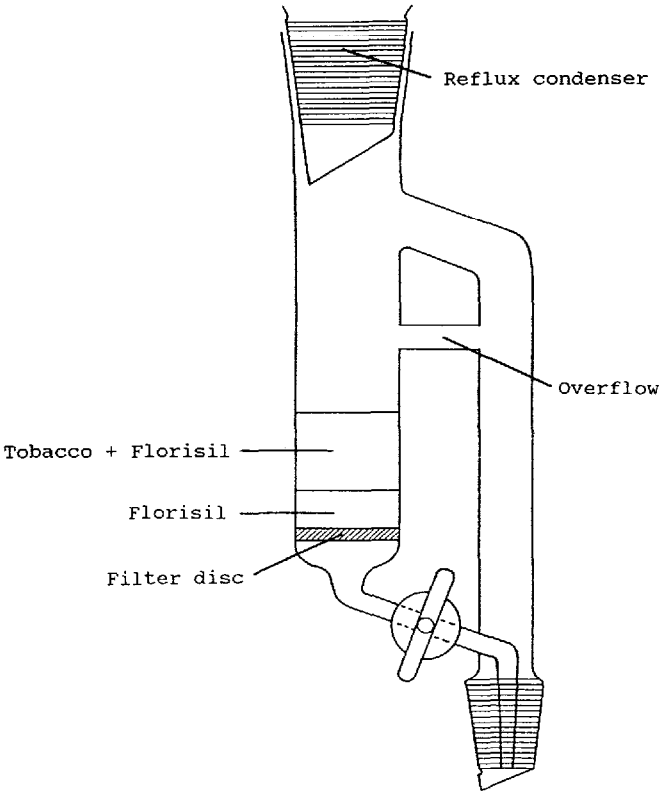

Fig. 1. Diagram of the equipment used for extraction. The filter disc is of porosity 1 and the overflow is at 85 mm. Height without standard taper joint: 170 mm. Inside diameter: 30 mm.

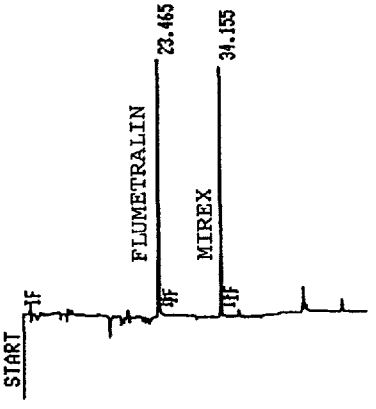

Fig. 2. Example of a chromatogram obtained after Soxhlet extraction. Sample containing 2.9 mg/kg of flumetralin.

TABLE I

FLUMETRALIN RESIDUE VALUES (mg/kg) OBTAINED BY DIFFERENT METHODS (RECOVERY CORRECTED)

The residue values are given at a 95% confidence limit level.

Sample	Coresta mean values (mg/kg)	Soxhlet extractor mean values (mg/kg)
11-L-A	2.6 ± 0.2[a]	2.8 ± 0.1[a]
11-L-B	2.5 ± 0.1	2.9 ± 0.2
11-L-C	2.6 ± 0.2	2.9 ± 0.2
21-L-A	15.0 ± 2.0	15.5 ± 0.4
21-L-B	12.4 ± 1.7	12.1 ± 0.4
21-L-C	26.4 ± 2.9	26.5 ± 0.9
22-L-A	63.1 ± 12.4	59.0 ± 2.1
22-L-B	46.1 ± 7.7	42.1 ± 1.6
22-L-C	103.0 ± 24.1	93.3 ± 4.1

[a] Mean ± standard deviation ($n = 5$).

Calibration

Owing to the non-linear response of the electron-capture detector, a multi-point calibration graph was constructed over the range of interest using standard *n*-hexane solutions containing 0.1–10 μg/ml of flumetralin corresponding to 1.3–126 mg/kg with a constant concentration of 0.2 μg/ml of mirex as an internal standard.

RESULTS

A tobacco sample spiked with 1.3 mg/kg of flumetralin gave a mean recovery of 99 ± 4% at a confidence limit level of 95% (five replicates). The detection limit was 0.1 mg/kg.

Three tobacco cultures grown in Italy and treated with different amounts of flumetralin 7 days after topping were used for the study. For each culture, three tobacco samples (leaves) were collected and the nine resulting samples were analysed for flumetralin. Table I gives the flumetralin residues found in each tobacco sample. These results were obtained by two different methods in a joint experiment of the Coresta pesticide sub-group [7,8] and with the described Soxhlet method. The results were in good agreement with each other.

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