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Residues of Fluvalinate and Permethrin on Flue-cured Tobacco*

by R. B. Leidy, T. J. Sheets and L. A. Nelson

Pesticide Residue Research Laboratory and Department of Statistics, North Carolina State University, Raleigh, North Carolina, U.S.A.

SUMMARY

High-performance liquid chromatography (HPLC) with C_{18} reverse-phase columns was used to determine residues of fluvalinate and permethrin applied in the field to flue-cured tobacco in 1980 and 1981. Methods were developed to determine residue levels in both green and cured samples. In 1980 field tests, residues of fluvalinate applied at rates of 0.1 and 0.2 kg/ha averaged 3.3 and 7.2 ppm, respectively, on green tobacco harvested immediately after application, and declined to 0.3 and 2.7 ppm by 12 days after application. In 1981, with the same application rates, residues averaged 1.7 and 3.0 ppm (0 time) and declined to 0.4 and 0.5 ppm after 16 days, respectively. A new formulation of fluvalinate, applied at 0.06 and 0.01 kg/ha, averaged 0.9 and 1.3 ppm on day 0 and declined to 0.3 and 0.6 ppm after 16 days, respectively. Flue curing reduced fluvalinate residues approximately by 61% both years. Immediately after application, residues of permethrin, applied at 0.2 kg/ha, averaged 9.6 ppm in 1980 and at application rates of 0.1 and 0.2 kg/ha averaged 3.5 and 3.8 ppm, respectively, in 1981. Residues declined to 7.0 ppm after 12 days in 1980 and to 3.2 and 3.5 ppm after 16 days in 1981. Losses of permethrin due to curing averaged 67%.

ZUSAMMENFASSUNG

Unter Einsatz von Hochleistungs-Flüssigkeitschromatographie (HPLC) mit C_{18}-Säulen zur Phasenumkehrung wurden Virginia-Tabake in den Jahren 1980 und 1981 auf Rückstände an Fluvalinat und Permethrin untersucht, nachdem sie im Feldversuch mit diesen Insektiziden behandelt worden waren. Es wurden Verfahren entwickelt, durch die sich Rückstände sowohl in grünen wie in getrockneten Proben quantitativ ermitteln ließen. Bei den Applikationsmengen von 0,1 und 0,2 kg je ha beliefen sich die Fluvalinatrückstände im Jahre 1980 in grünem Tabak, der gleich nach der Behandlung geerntet worden war, auf durchschnittlich 3,3 bzw. 7,2 ppm und gingen 12 Tage nach der Applikation auf 0,3 bzw. 2,7 ppm zurück. Im Jahr 1981 betrugen die Rückstandsmengen bei gleichen Applikationsmengen im Durchschnitt 1,7 bzw. bzw. 3,0 ppm unmittelbar nach der Applikation und verringerten sich 16 Tage danach auf 0,4 bzw. 0,5 ppm. Die Anwendung einer neuen Zubereitung von Fluvalinat in den Mengen

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von 0,06 und 0,01 kg je ha führte unmittelbar nach der Applikation zu Rückständen von durchschnittlich 0,9 bzw. 1,3 ppm, die sich 16 Tage später auf 0,3 bzw. 0,6 ppm verminderten. Durch die Trocknung (flue curing) reduzierten sich die Fluvalinatrückstände in beiden Jahren um etwa 61 %. Unmittelbar nach der Anwendung beliefen sich die Rückstände an Permethrin im Jahr 1980 bei der Applikationsmenge von 0,2 kg je ha im Durchschnitt auf 9,6 ppm und im Jahr 1981 bei Applikationsmengen von 0,1 und 0,2 kg je ha auf durchschnittlich 3,5 bzw. 3,8 ppm. Die Rückstandsmengen verminderten sich 1980 nach 12 Tagen auf 7,0 ppm und 1981 auf 3,2 bzw. 3,5 ppm nach 16 Tagen. Das Trocknungsverfahren trug zu 67 % zu der Verringerung der Permethrinrückstände bei.

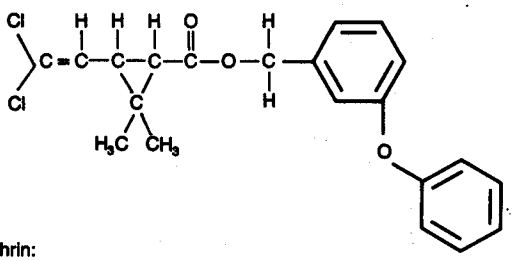
RESUME

La chromatographie en phase liquide à haute performance (HPLC) sur colonnes en C_{18} à renversement de phase a été utilisée pour déterminer les teneurs en résidus de fluvalinate et de perméthrine de tabacs de Virginie ayant été traités en plein champ par ces insecticides en 1980 et 1981. Des procédés ont été mis au point en vue du dosage des résidus contenus dans des échantillons de tabac vert et séché. Les quantités appliquées en 1980 étant de 0,1 et 0,2 kg par ha, on a trouvé dans le tabac vert cueilli immédiatement après le traitement une moyenne de 3,3 et 7,2 ppm de résidus de fluvalinate, ces valeurs passant respectivement à 0,3 et 2,7 ppm au bout de 12 jours. En 1981, les quantités de résidus observées pour les mêmes quantités d'insecticide ont été respectivement de 1,7 et 3,0 ppm immédiatement après l'application pour décroître ensuite jusqu'à 0,4 et 0,5 ppm après 16 jours. L'utilisation d'une nouvelle préparation de fluvalinate à raison de 0,06 et 0,01 kg par ha a conduit à des résidus de 0,9 et 1,3 ppm après l'application, ces valeurs se réduisant à 0,3 et 0,6 ppm 16 jours plus tard. Le «flue curing» a permis de réduire la teneur des résidus en fluvalinate d'environ 61 % pour chacune des deux années. Immédiatement après l'application de 0,2 kg par ha en 1980 et de 0,1 et 0,2 kg par ha en 1981, les résidus moyens de perméthrine ont été respectivement de 9,6 ppm et de 3,5 et 3,8 ppm. Ces quantités ont diminué ensuite jusqu'à 7,0 ppm au bout de 12 jours en 1980 et jusqu'à 3,2 et 3,5 ppm au bout de 16 jours en 1981. Le procédé de séchage a réduit en moyenne de 67 % la teneur en résidus de perméthrine.

INTRODUCTION

Pyrethroid insecticides show promise in the control of tobacco insect pest. Many advantages are listed for this class of insecticides, including low dosage for effective control, relative stability compared to natural pyrethrins, and low mammalian toxicity. One of the more

Figure 1a.
Structural formula of permethrin: contains approximately 60% *trans* and 40% *cis*-isomers).



permethrin:
3-(phenoxyphenyl)methyl (±)-*cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate

persistent pyrethroids is permethrin [3-(phenoxyphenyl)methyl (±)-*cis,trans*-3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate] (Figure 1a). This chemical is effective against a wide variety of insects with the greatest activity against lepidopterous pests. Application rates vary from 0.01 to 0.2 kg/ha depending on the crop being treated. An application rate on flue-cured tobacco of 0.22 kg/ha was recommended for permethrin in 1978 by Penick Corp. (1). A new pyrethroid, fluvalinate [*N*-(2-chloro-4-(trifluoromethyl)-phenyl)-D-valine (±)-α-cyano(3-phenoxyphenyl)methyl ester] (Figure 1b), is now in field trials. Like permethrin, it is a contact and stomach poison with residual activity. Preliminary field tests on tobacco at application rates of 0.055 to 0.22 kg/ha have shown excellent control of the tobacco budworm *(Heliothis virescens)*, tobacco hornworm *(Manduca sexta)*, the tobacco flea beetle *(Epitrix hirtipennis)*, and several species of aphids *(Aphididae)* (2).

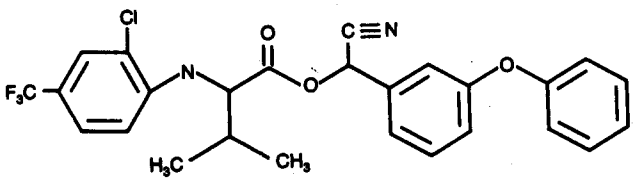
Analytical methods have been described for the determination of permethrin in fortified fiber and food crops with quantitation by gas chromatography (GLC) (3, 4, 5). Disadvantages of pyrethroid analysis by gas chromatography were discussed (6). Recently, a high-performance liquid chromatography (HPLC) system, including an infrared and an UV detector in tandem, was used to analyze formulations of three pyrethroid insecticides (7). This instrument alleviated problems associated with GLC analyses for pyrethroids. No published methods were found to determine the residues of fluvalinate on tobacco, and there are no data available on the residues of permethrin or fluvalinate on tobacco. Hence, a study was initiated to determine the residue levels of these insecticides on green and flue-cured tobacco after application at rates used for insect control.

MATERIALS AND METHODS

Insecticide Application and Sampling

In 1980, the experiment was established on a Norfolk sandy loam soil at the Central Crops Research Station

Figure 1b.
Structural formula of fluvalinate.



fluvalinate:
N-(2-chloro-4-(trifluoromethyl)phenyl)-D-valine (±)-α-cyano(3-phenoxyphenyl)methyl ester

near Clayton, N.C. Flue-cured tobacco *(Nicotiana tabacum* L. cv. McNair 944) was grown using recommended cultural practices. Permethrin (Ambush, at 0.24 kg/l a.i.) and fluvalinate (Mavrik, at 0.24 kg/l a.i.) were applied July 24, 1980 when the lower six leaves were at or near the state of normal harvest. Commercial formulations were diluted with water so that rates of 0.22 kg/ha for permethrin and 0.11 and 0.22 kg/ha for fluvalinate were applied. A single nozzle* was positioned directly above the plants in each row and delivered 234 l/ha at a spray pressure of 4.2 kg · cm^2 (8). Plots were four rows wide and 14 m long with a row spacing of 1.1 m. There were three replications. The experimental design was a split-plot in time with a randomized complete block arrangement of the whole-plot factor (rate-formulation treatments). Green samples were removed from the same stalk position of rows 1 and 4 on each of four sampling dates (0, 4, 8, and 12 days) after application. Six leaves were removed from each of eight plants at each sampling, and six discs, 5 cm in diameter, were taken (avoiding the midrib) from each leaf. The discs were placed in plastic bags and stored in insulated boxes containing solid carbon dioxide for transfer to the laboratory. Frozen discs were crushed and subsampled for analysis. Samples for curing were removed from the two center rows of each plot at the normal time for harvesting (7, 14, and 34 days after application); 48 leaves were chosen randomly and placed in bulk-curing racks for curing. Cured leaves were stemmed. The lamina was ground in a Wiley mill to pass a 50 mesh screen, and subsamples were taken for analysis. In 1981, the field test was conducted at the same location as in 1980, following standard cultural practices. Permethrin and fluvalinate were applied as described above on July 28 at the following rates: 0.11 and 0.22 kg/ha of permethrin and fluvalinate (old formulation which is a racemic mixture of four isomers) and 0.056 and 0.11 kg/ha of the new fluvalinate formulation (Mavrik Aquaflow, a half-resolved racemic mixture of two isomers in an aqueous formulation containing 0.24 kg/l a.i.). Plot size and experimental design were the same as in 1980, except that four replications were made in 1981.

Green samples were harvested from rows 1 and 4 0, 4, 8, and 16 days after application. Samples for curing were removed from the two center rows of each plot 7, 21, 35, and 43 days after application. Forty-eight leaves were chosen randomly, and four 5 cm discs were removed from each leaf for determination of the residue before curing. Cured leaves were treated as described above for the 1980 test. In addition, stems from the 0.22 kg/ha application rate were saved for analysis.

Permethrin Analysis

Twenty grams of green tobacco were tared into a 500 ml beaker and blended for 10 min with 200 ml of 2-propanol. The extract was filtered under reduced pressure through a 47 mm Whatman GF/B fiberglass filter topped with 10 g anhydrous sodium sulfate and 5 g of Celite (particle size: 26 μm) contained in a 300 ml membrane filter apparatus. The filter was rinsed with 50 ml of 2-propanol, and the combined extract was evaporated to about 3.0 ml under reduced pressure at 45 °C. Residues were transferred quantitatively to 12 ml conical tubes with *n*-hexane, and the volume was reduced to 2.0 ml under a stream of dry air. This fraction was placed on a Waters Associates Florisil Sep-Pak® and allowed to flow into the column. Four 2 ml portions of *n*-hexane were added to the Sep-Pak and allowed to flow through by gravity. Volumes were brought to 10 ml with *n*-hexane.

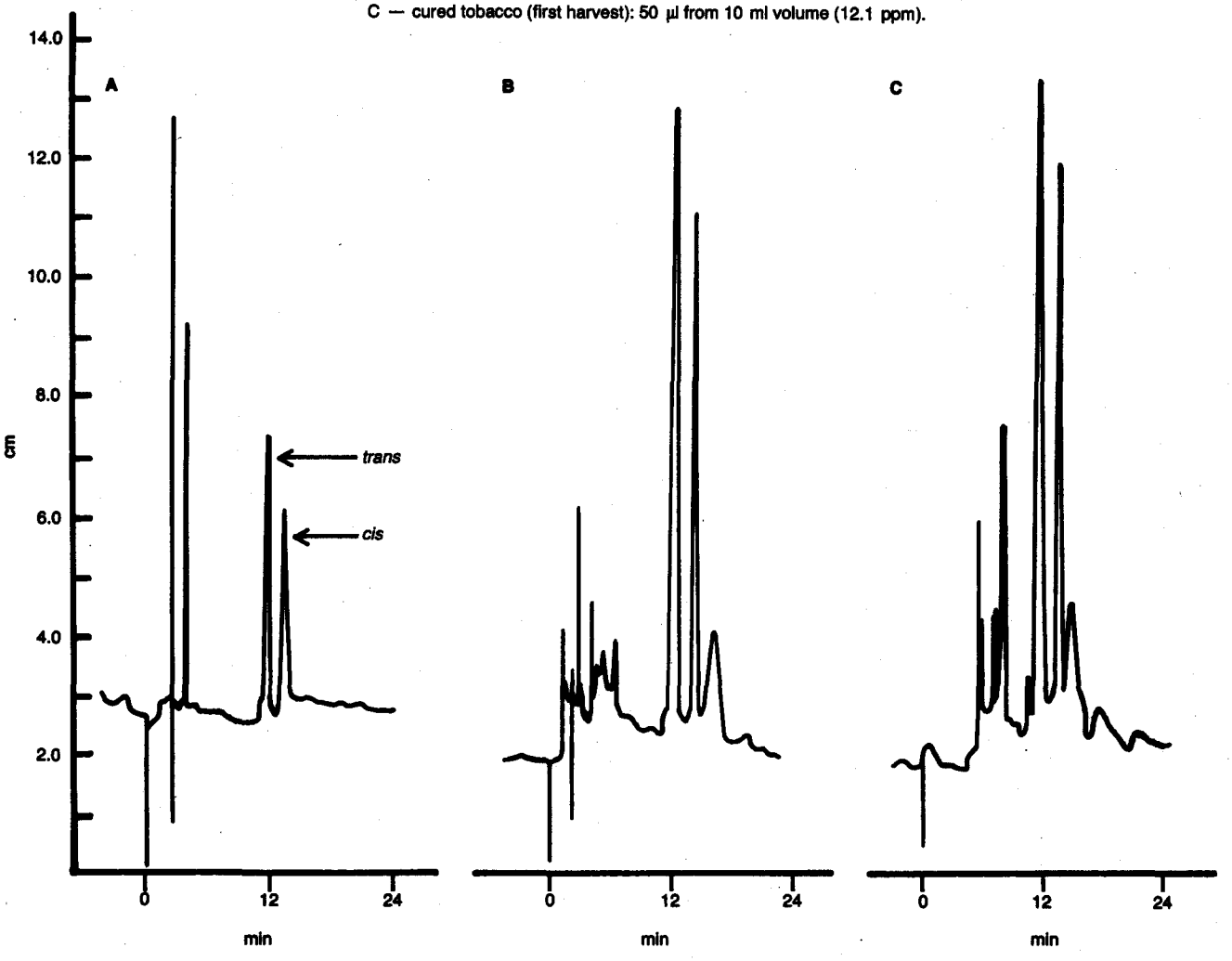
Cured samples were prepared for analysis as follows: Ten grams of cured tobacco were blended for 5 min with about 5 g of Celite and 200 ml of 2-propanol. The extract was filtered as described above. The filtrate was concentrated to 1 to 2 ml under reduced pressure at 45 °C and taken to 10 ml with *n*-hexane; the solution was transferred to a 125 ml separatory funnel using three 10 ml volumes of acetonitrile (CH_3CN) and two 10 ml volumes of *n*-hexane. The funnel was shaken gently for 30 s and the layers were allowed to separate; the CH_3CN fraction was drained into a 500 ml boiling flask. The CH_3CN extraction was done twice more, and the combined fractions were concentrated to 2 to 3 ml at 45 °C. Residues were taken up in *n*-hexane and the volume of the solvent was reduced to 2 ml under a stream of nitrogen. This extract was placed on a Florisil Sep-Pak® and permethrin was eluted as described above.

Samples were analyzed by HPLC. The pump was a Waters Associates Model 6000A. The solvent system was CH_3CN:water:2-propanol (2:1:1 (v:v:v)) with a flow rate of 1.5 ml/min. All solvents were HPLC grade and were degassed by sonication under reduced pressure. The column was a Waters Associates Radial-Pak-C_{18}® reverse phase (10 cm by 8 mm inside diameter) contained in a Waters Associates Radial Compression Module (RCM-100®). The detector was a Schoeffel SF-770 UV/vis. operated at 214 nm and 0.1 AUFS. The injector was either a Waters Associates Model U6K or a Model 710B WISP autosampler. A 10 mV recorder operated at 0.25 cm/min was used to record detector response. Data were calculated by the peak height method using standards of known concentration. Since the *cis* and

* Spraying Systems Co. (Model D2-33FC), Bellwood, Ill.

Figure 2.
Chromatograms of permethrin.

A — analytical standard: 25 µl of 10.0 µg/ml concentration.
B — green tobacco (day 0): 50 µl from 20 ml volume (9.4 ppm).
C — cured tobacco (first harvest): 50 µl from 10 ml volume (12.1 ppm).



trans-isomers were separated to the baseline, the combined heights were used to determine total residue levels of permethrin in tobacco. The efficiency of the analytical method was determined by adding known amounts of permethrin to untreated tobacco and analyzing the fortified samples by the same method. All green samples and all cured samples were extracted and analyzed at the same time. Ten recoveries were analyzed with each set of samples.

Fluvalinate Analyses

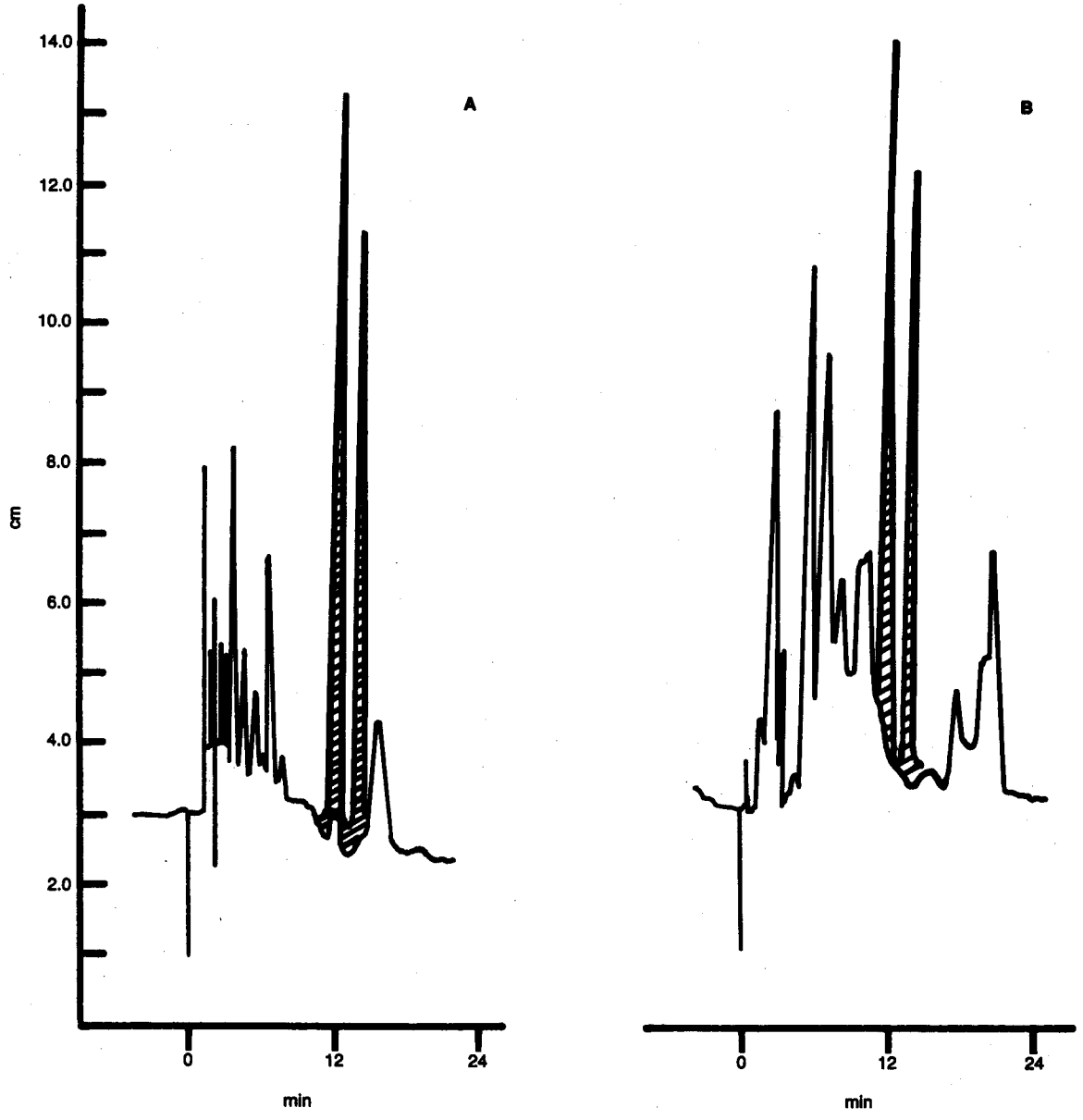
Twenty-five grams of crushed green tissue were tared into a 500 ml beaker and blended for 5 min with 5 g of Celite (particle size: 18 µm) and 200 ml of ethyl acetate. The material was filtered as described for permethrin residues. The filter was rinsed with an additional 50 ml of ethyl acetate, and the combined extract was evaporated at 40 °C under reduced pressure to 2 to 3 ml. The residue was transferred quantitatively to 12 ml tubes with *n*-hexane and brought to 10 ml for analysis. No further cleanup was required.

Ten grams of ground cured tobacco were blended for 5 min with 5 g of Celite and 150 ml of CH_3CN. The extract was filtered through 20 g of anhydrous sodium sulfate and evaporated to 2 to 3 ml at 40 °C. The remaining residue was blown to dryness with nitrogen, and 5 ml of *n*-hexane were added. The *n*-hexane fraction was transferred to a 2.5 cm by 20.0 cm glass column containing 5 g of Florisil topped with 2.5 cm of sodium sulfate. After pre-rinsing the column with 50 ml of *n*-hexane, the tobacco extract was added, and an additional 30 ml of *n*-hexane was added to the column. Fluvalinate was eluted with 100 ml of 5% 2-propanol in heptane. The eluate was concentrated to 2 to 3 ml at 40 °C and brought to volume with *n*-hexane.

Figure 3.
Chromatograms of permethrin.

A — green tobacco: untreated control with 10.0 ppm permethrin superimposed (10.0 ml volume).

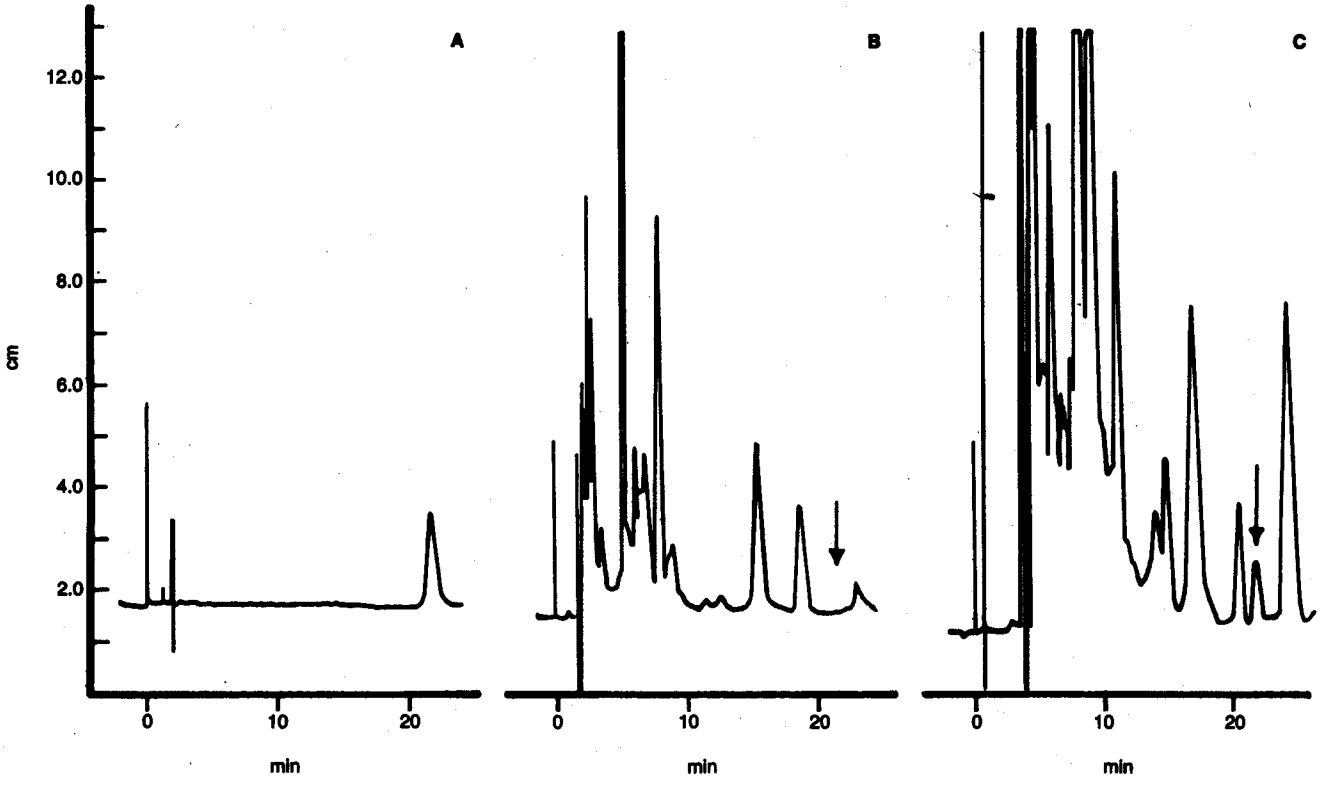
B — cured tobacco: untreated control with 10.0 ppm permethrin superimposed (5.0 ml volume).



The HPLC system used was the one described previously. The solvent was methanol:water:2-propanol (83:15:3 (v:v:v)) at flow rates of 0.9 ml/min (green tobacco) and 1.5 ml/min (cured tobacco). The detector was operated at 256 nm and 0.1 AUFS. The recorder was operated at a chart speed of 0.25 cm/min to monitor detector response. Data were quantitated by the peak height method against standards of known concentration. Two untreated control samples, fortified with known amounts of fluvalinate, were analyzed with each set of experimental samples to determine recovery values. The data were subjected to an analysis of variance.

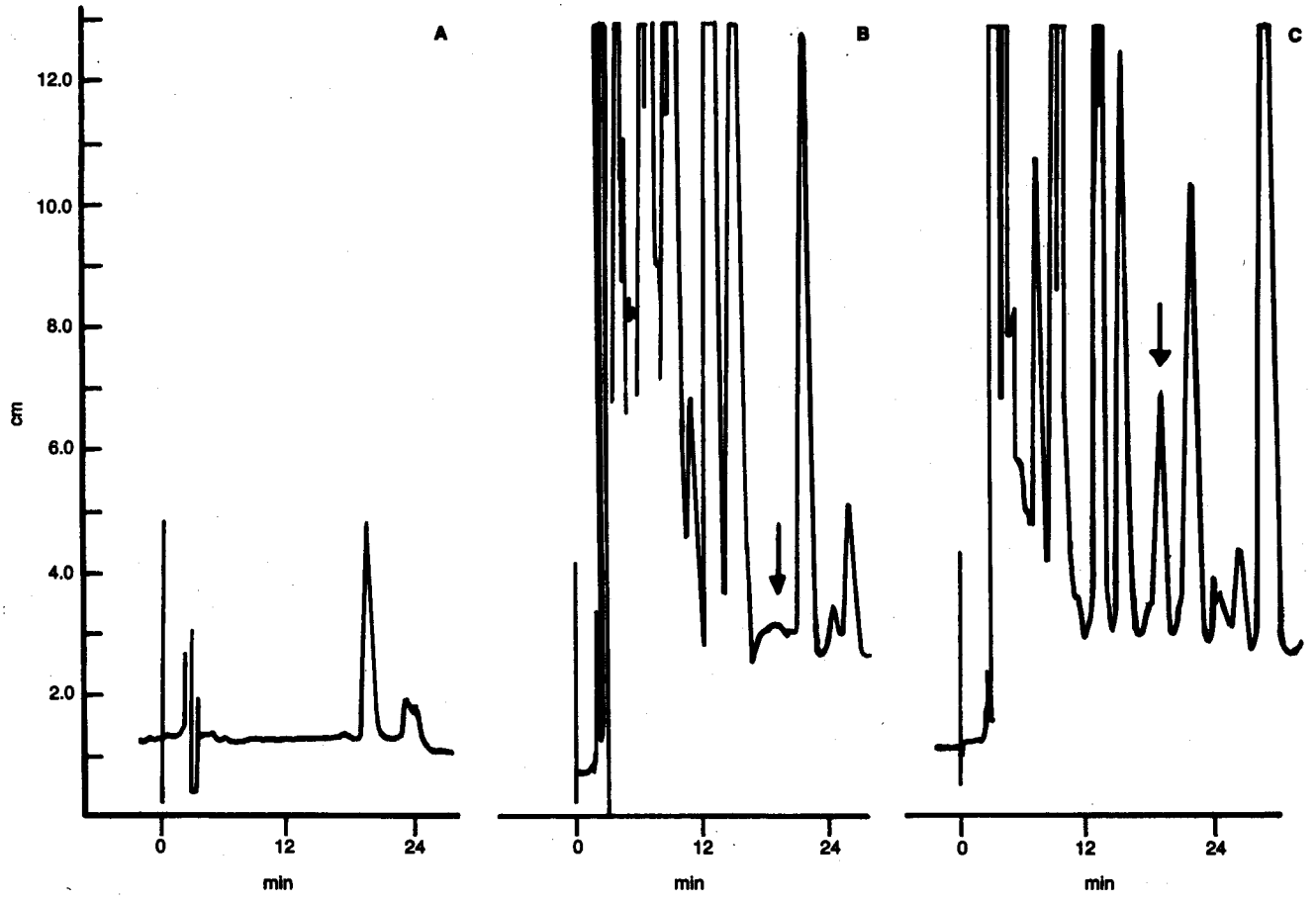
Chromatograms of permethrin from green and flue-cured tobacco showed that the solvent system gave a baseline separation of the *cis* and *trans*-isomers (Figure 2). When superimposed on chromatograms for untreated green and cured tobacco, there were no interference peaks to prevent accurate determinations (Figure 3). At 214 nm and 0.1 AUFS, linearity was maintained between 0.1 and 0.5 µg. Thus, quantities ranging from 25 to 50 µl were injected so that detected residue levels would be in this range. Chromatograms for fluvalinate on green and cured tobacco showed that green samples had fewer interferences than cured samples,

Figure 4.
Chromatograms of fluvalinate (green tobacco).



A — analytical standard: 25 µl of 10.0 µg / ml.
B — untreated control: 20.0 ml volume.
C — day 0: 25 µl from 10.0 ml volume (2.5 ppm).

Figure 5.
Chromatograms of fluvalinate (cured tobacco).



A — analytical standard: 25 µl of 10.0 µg/ml.

B — untreated control: 10 ml volume.

C — first harvest: 50 µl from 10 ml volume (5.5 ppm).

Table 2.
Recoveries of permethrin residues from untreated green and flue-cured tobacco.

Year	Tobacco type	No. of samples	Amount added* (ppm)	Amount recovered (ppm)	Range (%)	Recovered (%)
1980	green	4	0.1	0.1	95—99	96
		4	0.2	0.2	91—96	93
		4	0.5	0.5	91—96	94
		4	1.0	0.9	83—95	92
		4	5.0	4.9	95—98	97
		4	10.0	9.4	90—97	94
		4	20.0	18.6	83—99	93
	cured	4	0.1	0.1	86—93	88
		4	0.2	0.2	77—86	82
		4	1.0	1.0	89—97	95
		4	10.0	10.1	97—104	101
		4	20.0	16.8	83—85	84
1981	green	4	0.1	0.1	94—97	96
		8	1.0	0.9	82—94	93
		4	5.0	4.8	93—98	95
		4	10.0	9.2	89—95	92
	cured	4	0.1	0.1	85—92	88
		4	1.0	0.9	89—103	94
		4	10.0	9.5	88—97	95

* Amounts of permethrin added to 20 g of untreated green or flue-cured tobacco. The fortified samples were not extracted for 2 h to allow for solvent evaporation and penetration. Recoveries were based on equivalent amounts of permethrin added to 12 ml tubes, followed by dilution with *n*-hexane and chromatography.

Table 3.
Recoveries of fluvalinate residues from untreated green and flue-cured tobacco.

Year	Tobacco type	No. of samples	Amount added* (ppm)	Amount recovered (ppm)	Range (%)	Recovered (%)
1980	green	4	0.05	0.04	87—92	88
		4	0.1	0.1	87—88	88
		4	0.5	0.5	89—95	92
		4	1.0	0.9	90—98	93
		4	10.0	8.3	80—85	83
		4	25.0	24.5	97—101	98
		4	30.0	28.5	92—97	95
	cured	4	0.05	0.05	87—95	92
		4	0.1	0.1	83—95	90
		4	0.5	0.5	88—96	93
		4	1.0	0.9	83—93	89
		4	5.0	4.4	85—90	87
		4	10.0	9.3	85—101	93
1981	green	2	0.2	0.2	94—113	104
		3	0.5	0.5	66—107	90
		6	1.0	0.9	76—109	94
		3	2.0	1.9	89—106	93
		4	3.0	3.0	89—119	102
		2	10.0	9.8	97—99	98
		2	20.0	18.0	89—91	90
	cured	2	0.2	0.2	91—92	92
		7	0.5	0.5	91—94	93
		7	1.0	0.9	82—102	89
		3	5.0	4.7	89—96	93
		3	10.0	9.4	88—99	94

* Amount of fluvalinate added to 25 g of green tobacco or 10 g of cured tobacco. Fortified samples were extracted as described 2 h after fluvalinate was added. Recoveries were based on equivalent amounts of fluvalinate added to 12 ml tubes, followed by dilution with *n*-hexane and chromatography.

but flow rates were adjusted on cured tobacco samples so that the fluvalinate peak was free from interfering peaks (Figures 4 and 5). Linearity was maintained over a concentration of 0.05 to 1.25 µg at 256 nm and 0.1 AUFS. Injections of experimental samples varied between 25 and 75 µl.

The recovery data for permethrin and fluvalinate for the 2-year study are shown in Tables 2 and 3, respectively. Overall averages were as follows: permethrin — 94% from green leaf, 91% from cured leaf; fluvalinate — 93% from green leaf, 91% from cured leaf. Recovery values were consistent, for both years of the experiment when equivalent fortification levels were compared. The low detectable limit of permethrin was 0.1 ppm and that of fluvalinate was 0.05 ppm.

RESULTS AND DISCUSSION

Temperature and rainfall data are shown in Table 1. More rain fell during the 1981 test (8.5 cm total rainfall during the experimental period) than during the 1980 test (1.9 cm) although most rain in 1981 occurred 15 days after application (6.2 cm). A total of 1 cm fell on days 1 and 2 after application in the 1981 test which might have influenced residue levels.

1980 Tests

Residue levels of permethrin and fluvalinate on green tobacco in 1980 are shown in Table 4. Immediately after application, permethrin residues averaged 9.6 ppm; by 12 days residues had declined to 7.0 ppm or 73% of day 0 levels.

Green tobacco treated with fluvalinate at the rate of 0.22 kg/ha contained approximately twice the residue of that treated with 0.11 kg/ha. Day 0 levels for the 0.22 kg/ha application rate averaged 7.0 ppm whereas those for the 0.11 kg/ha rate averaged 3.1 ppm. The decrease in residues was greatest between 0 and 4 days, then levels declined gradually. Day 12 residues were 16 and 37% of day 0 levels for the 0.11 and 0.22 kg/ha application rates, respectively. The statistical analysis showed a significant difference between residues of fluvalinate (0.22 kg rate) on day 0 and the other sampling dates. Although there were no significant differences between residue levels of permethrin and fluvalinate (0.22 kg rate) on day 0, residues of fluvalinate were different from permethrin at subsequent sampling periods.

Residue levels for both insecticides on cured tobacco in 1980 are shown in Table 5. There was no difference in permethrin residues found on samples from the first and second harvests which were taken 1 week apart. The third harvest averaged 7.0 ppm of permethrin, and this level was 69% of that for the first harvest. Since green samples were not taken from the same tobacco that was cured, one cannot estimate, definitively, the losses that occurred during curing. However, if the residue levels for green tobacco converted to a 13% moisture basis are compared with residues for cured leaves that were harvested at about the same time, an indication of residue loss due to curing can be obtained. Cured leaves from the first harvest 7 days after application had residue levels 33% of green tobacco harvested on day 8; and second harvest leaves taken 14 days after application contained residues that were 42% of green leaves harvested on day 12 (Tables 4 and 5). These data suggest that losses due to curing were approximately 60% to 65%.

At the first harvest, fluvalinate residues at the 0.22 kg/ha rate averaged 62% of those for permethrin. Third harvest levels of fluvalinate were 59% of those found on the first harvest at the 0.22 kg/ha rate (Table 5). Based on residue levels on green tobacco at 13% moisture, flue curing reduced fluvalinate levels approximately 56%.

The analysis of the data from cured tobacco showed a significant difference between residues of permethrin and fluvalinate from all harvests.

Table 1.
Temperature and rainfall data for periods between application and the last harvest.*

Year	Time after application (days)	Temperature maximum (°C)	Temperature minimum (°C)	Rainfall per day (cm)
1980	0	29	21	
	1	32	20	
	2	32	20	
	3	29	19	0.1
	4	29	21	
	5	31	22	0.5
	6	34	20	
	7	36	22	
	8	38	23	
	9	36	23	
	10	34	20	1.3
	11	36	23	
	12	36	23	
1981	0	35	24	
	1	33	24	0.3
	2	26	18	0.7
	3	28	17	
	4	31	16	
	5	32	20	
	6	30	21	0.1
	7	36	21	
	8	37	22	
	9	30	23	0.2
	10	32	21	
	11	32	23	0.4
	12	32	23	0.6
	13	32	23	
	14	33	22	
	15	27	19	6.2
	16	33	22	

* Permethrin and fluvalinate were applied July 24, 1980 and July 28, 1981.

Table 4.
Residues of permethrin and fluvalinate in green tobacco, 1980.*

Compound	Rate of application (kg/ha)	Time after application (days)	Concentration** (ppm)
Permethrin	0.22	0	9.6
		4	9.1
		8	8.2
		12	7.0
Fluvalinate	0.11	0	3.1
		4	1.9
		8	1.5
		12	0.5
Fluvalinate	0.22	0	7.0
		4	4.1
		8	3.5
		12	2.6

Least significant difference (p=0.05): between treatments within a time = 4.5; between times within a treatment = 1.9

* Permethrin and fluvalinate applied July 24, 1980 at the Central Crops Research Station, Clayton, N.C.

** Residue levels based on following average moisture contents: permethrin, 76.82%; fluvalinate (0.11 kg/ha), 77.23%; fluvalinate (0.22 kg/ha), 79.39%.

1981 Tests

At the 0.22 kg/ha rate, residues of permethrin on green tobacco in 1981 were approximately 40% of the 1980 levels (Table 6). There was a 6 °C higher temperature (29 °C, 1980; 35 °C, 1981) when the insecticide was applied in 1981 which could have increased the volatility of permethrin, resulting in lower residue levels. There was little difference in residue levels of permethrin over the sampling period, and 16 days after application permethrin residue levels were equivalent to those found on day 0. On day 0, residues of fluvalinate from the 1981 test averaged about 43% of the 1980 levels. Residues declined over subsequent sampling dates and, on the 16th day both applications averaged 17% of day 0 levels. The new formulation of fluvalinate at an application rate of 0.11 kg/ha had residue levels equivalent to the old formulation for the first three harvests. On day 16, the residue level was 50% of day 0 compared to 17% for the old formulation. The data suggest that the new formulation of fluvalinate may be more persistent than the old.

Table 5.
Residues of permethrin and fluvalinate on cured tobacco, 1980.*

Compound	Rate of application (kg/ha)	Harvest No.	Concentration** (ppm)
Permethrin	0.22	1	10.2
		2	11.0
		3	7.0
Fluvalinate	0.11	1	3.3
		2	1.9
		3	2.9
Fluvalinate	0.22	1	6.3
		2	4.5
		3	3.7

Least significant difference (p=0.05): between treatments within a harvest: 2.1; between harvests within a treatment: 2.9

* Permethrin and fluvalinate applied July 24, 1980 at the Central Crops Research Station, Clayton, N.C., and harvested July 31, August 7, and August 27, 1980.

** Residue levels adjusted for a 13% moisture content.

Table 6.
Residues of permethrin and fluvalinate on green tobacco, 1981.*

Compound	Rate of application (kg/ha)	Time after application (days)	Concentration** (ppm)
Permethrin	0.11	0	3.5
		4	2.5
		8	2.5
		16	3.5
Permethrin	0.22	0	3.8
		4	3.5
		8	2.7
		16	3.7
Fluvalinate (Mavrik)	0.11	0	1.2
		4	1.8
		8	0.6
		16	0.2
Fluvalinate (Mavrik)	0.22	0	3.0
		4	2.3
		8	1.3
		16	0.5
Fluvalinate (new formulation)	0.056	0	0.4
		4	0.9
		8	0.3
		16	0.4
Fluvalinate (new formulation)	0.11	0	1.2
		4	1.3
		8	0.8
		16	0.6

Least significant difference (p=0.05): between treatments within a time = 0.9; between times within a treatment = 0.8

* Permethrin and fluvalinate applied July 28, 1981 at the Central Crops Research Station, Clayton, N.C.

** Residue levels based on following average moisture contents: permethrin, 77.1%; fluvalinate (all), 80.2%.

Table 8.
Loss of permethrin and fluvalinate due to flue curing in the 1981 test.*

Insecticide	Rate of application (kg/ha)	Kind of sample	First harvest (ppm)	First harvest (% loss)	Second harvest (ppm)	Second harvest (% loss)	Third harvest (ppm)	Third harvest (% loss)	Fourth harvest (ppm)	Fourth harvest (% loss)
Permethrin	0.11	green	9.1		11.0		13.7		12.2	
	0.11	cured	2.9	68	3.5	68	3.4	75	4.0	67
	0.22	green	10.3		9.9		13.3		8.9	
	0.22	cured	2.5	76	4.1	59	4.2	68	3.7	58
Fluvalinate (new formulation)	0.056	green	3.2		1.2		1.3		1.4	
	0.056	cured	0.9	72	0.6	50	0.7	46	0.4	71
	0.11	green**	3.8		2.3		4.1		3.0	
	0.11	cured**	1.5	61	1.1	52	1.1	73	0.8	73
Fluvalinate (Mavrik)	0.22	green	5.7		2.5		6.3		5.4	
	0.22	cured	2.2	61	1.6	32	1.8	69	1.7	69

* All residues are based on 13% moisture basis; harvest dates were August 4, August 18, September 1, September 9, 1981.

** Both formulations averaged.

Residues of permethrin and fluvalinate on cured lamina are shown in Table 7. No differences were seen in levels of permethrin among the four harvests at the 0.11 kg/ha rate. At the high rate, the residue for harvests 2, 3, and 4 are greater than that for harvest 1. These data differ from the 1980 test in that a 30% decrease in residues was seen at the third harvest in 1980. Analyses of stems from 1981 flue-cured tobacco showed low levels (<1.0 ppm) from all harvests. Lamina and stems were not separated in the 1980 tests so no comparisons could be made.

There was no significant drop in fluvalinate residues between the first and last harvest (Table 7). However, at the fourth harvest residue levels averaged 62% of those found at the first harvest. These data are similar to those from the 1980 tests (3rd harvest, Mavrik 60%). As was seen with the residues on green tobacco, there was no difference between the two formulations at the 0.11 kg/ha rate. Residues in stems averaged 0.9 ppm.

The statistical treatment of the data showed a significant difference at the 5% probability level between residues of permethrin and fluvalinate. Losses due to flue curing are shown in Table 8. Losses of permethrin residues averaged 67% and those for fluvalinate averaged 61% over all harvests and application rates. Although the loss during curing was greater for permethrin than for fluvalinate, the total residue remaining after curing was highest for permethrin.

The data indicated that permethrin is a more persistent insecticide on both green and cured tobacco than fluvalinate.

Table 7.
Residues of permethrin and fluvalinate on cured tobacco, 1981.*

Compound	Rate of application (kg/ha)	Harvest No.	Concentration** (ppm)
Permethrin	0.11	1	3.0
		2	3.5
		3	3.5
		4	4.1
Permethrin	0.22	1	2.5
		2	4.2
		3	4.1
		4	3.8
Fluvalinate (Mavrik)	0.11	1	1.4
		2	1.0
		3	1.2
		4	0.8
Fluvalinate (Mavrik)	0.22	1	2.2
		2	1.7
		3	1.8
		4	1.7
Fluvalinate (new formulation)	0.056	1	0.9
		2	0.6
		3	0.7
		4	0.4
Fluvalinate (new formulation)	0.11	1	1.5
		2	1.1
		3	0.9
		4	0.8
Least significant difference (p=0.05)		between treatments within a harvest = 1.5 between harvests within a treatment = 0.8	

* Insecticides applied July 28, 1981. Tobacco was harvested August 4, August 18, September 1, and September 9, 1981.

** Residue levels adjusted for a 13% moisture content.

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Authors' address:

Pesticide Residue Research Laboratory,
Department of Entomology,
School of Agriculture and Life Sciences,
North Carolina State University,
3709 Hillsborough Street,
Raleigh, North Carolina, 27607, U.S.A.