Determination of Fenvalerate, a Synthetic Pyrethroid, in Grapes, Peppers, Apples, and Cottonseeds by Gas-Liquid Chromatography

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Two methods are described for the determination of fenvalerate (α-cyano-*m*-phenoxybenzyl α-isopropyl-*p*-chlorophenylacetate) residues in grapes, peppers, apples, and cottonseeds. In grapes, peppers, and apples, acetone extraction was followed by partitioning with petroleum ether (40–60 °C). In cottonseeds, Soxhlet extraction (after blending) was followed by partitioning with propylene carbonate. The same cleanup method (Florisil) was used in each case. In grapes, initially fortified with 0.005–0.5 ppm of fenvalerate, the average recovery was 96–98%; in peppers fortified with 0.005–1.0 ppm, the recovery was 94–99%; in apples (0.01–1.0 ppm) it was 97–99%; in cottonseeds (0.01–0.2 ppm) it was 80–93%. The limit of detection was 0.005 ppm for grapes and peppers and 0.01 ppm for apples and cottonseed. Values obtained for fenvalerate residues in field-treated samples of grapes, peppers, apples, and cottonseeds are also presented.

Fenvalerate (α-cyano-*m*-phenoxybenzyl α-isopropyl-*p*-chlorophenylacetate) (also known as sumicidin, S 5602, WL 43775, and pydrin) belongs to a new group of synthetic pyrethroid esters which do not contain cyclopropanecarboxylates.

Although the cyclopropanecarboxylate group common to the naturally occurring (Crombie et al., 1976) and to many synthetic pyrethroids was generally thought to be essential for the insecticidal activity, studies on the influence of structural variation on insecticide activity have shown that the high insecticidal activity depends on the overall shape of the molecule, indicating that fenvalerate should be considered a member of the same class of synthetic pyrethroids (Ohno et al., 1976; Elliot and Janes, 1978).

Fenvalerate is used to control insect pests which are resistant to chlorinated hydrocarbons, organophosphates, and carbamates; it is especially effective against lepidoptera, hemitera, and diptera larvae. Its high efficacy allows the use of a very low dosage in agricultural treatments ("The Pesticide Manual", 1979).

Previous studies on fenvalerate residues have been reported. In the method of Talekar (1977), the insecticide was extracted from cabbage with hexane–acetone by Soxhlet or Polytron extraction. After partitioning with hexane, the extract was cleaned up by using Florisil (20 g). Benzene–ethyl acetate was used as the eluting solvent and gas liquid chromatography (GLC) [employing electron capture detection (EC)] was used for quantitative determination of the residue. In the method of Lee et al. (1978), acetonitrile was used for extraction of the compound from cabbage and lettuce, and two separate columns (Florisil and silica gel) were used for cleanup. The compound was eluted in the second fraction of each of the columns, and ~600 mL of solvent was used for cleanup of each sample. A method for the determination of several pyrethroids (including fenvalerate) by GLC equipped with an electron capture detector has been described by Chapman and Simmons (1977). Studies on the photodecomposition of fenvalerate have been reported by Holmstead and Fullmer (1977) and by Holmstead et al. (1978).

The aim of the present work was to devise a simple, quick, and efficient method for the routine analysis of fenvalerate in several crops, using a less expensive and less toxic solvents for extraction and cleanup.

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In both procedures described below, GLC (EC) was used for the quantitative determination of fenvalerate. Cleanup with Florisil followed partitioning with petroleum ether for the green plant extracts and with propylene carbonate–petroleum ether (Schnorbus and Phillips, 1964) for the cottonseed extracts.

The practical application of the method in the analysis of field-treated samples is also reported.

MATERIALS AND METHODS

Reagents. Acetone (distilled in glass), petroleum ether (40–60 °C), chloroform, analytical grade (Frutarom Chemical Co., Haifa, Israel), and propylene carbonate, chemically pure (Fluka, Switzerland), were used. Florisil (Floridin Co., Hancock, WV; 60–100 mesh) was heated for 48 h at 130 °C, cooled, and transferred to a flask. Water (4%) was added with shaking. The adsorbent was left to equilibrate for 24 h.

Anhydrous granular sodium sulfate was obtained from Merck Chemical Co. (Darmstadt). A saturated solution of sodium chloride was prepared from analytical-grade sodium chloride (Frutarom). Fenvalerate, analytical grade (94.5%), was supplied by the Agan Co., Ashdod, Israel. The stock solution of fenvalerate was 1 mg/mL of acetone, and working solutions of 1.0, 0.2, and 0.1 ng/mL of acetone were prepared.

Gas–Liquid Chromatography. A Becker Model 400 gas chromatograph equipped with a tritium (12 V) electron capture dtector was used. Analyses were carried out by using a glass column (1.1 m × 2 mm i.d) containing a column packing of 2% XE_{60} on 80–100-mesh Supelcoport. The temperatures of the column, detector, and injection port during the analyses were maintained at 215, 230, and 240 °C, respectively. Nitrogen was used as carrier gas at a flow rate of 30 cm^3/min. The electrometer setting was 2.5×10^{-10} a.f.s. A Westronics recorder, 1 mV, chart speed 7.5 in./h, was used.

Centrifuge tubes (100 and 200 mL) with ground-glass stoppers were used (in the analysis of cottonseeds).

Column Chromatography. For the cleanup step, a glass tube (0.75 × 30 cm) with a capillary opening and a funnel at the top was used.

Spray Application. All samples (grapes, peppers, apples, and cottonseeds) used in this work were provided by the Agan Co. and stored under refrigeration (approximately −15 °C) prior to analysis. The samples were treated by spraying with fenvalerate, 20% formulation, as follows.

The *grapes* were sprayed with 37.5 g of a.i./ha and with 75 g of a.i./ha. The sprayings were repeated 10 days later.

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Starting on the day of the second application, fruit samples were collected 4 times at 7-day intervals.

The *peppers* similarly received two sprayings of 150 g of a.i./ha and 300 g of a.i./ha at 10-day intervals. The samples were collected 3 times, starting on the day of the second application.

The *apples* were sprayed 5 times at 20-day intervales at 450 g of a.i./ha. Samples were collected on the day of the last spraying, 8 days later, and 1 month after the last spraying.

The *cottonseeds* received two sprayings of 300 g of a.i./ha and 600 g of a.i./ha at 10-day intervals. Samples were collected 77 days after the last spraying. Application in the trials of grapes, peppers, and cottonseeds were done with a knapsack sprayer. The apples were sprayed to the point of "runoff" with an air-blast sprayer.

Sample Extraction and Solvent Partitioning for Grapes, Peppers, and Apples. A representative sample of 50 g of plant material was placed in a Waring blender and macerated together with 100 mL of acetone for 2 min at high speed, and the resulting mixture filtered by suction through Whatman No. 1 paper. The extraction was repeated with 50 mL of acetone, and the acetone extract was then transferred to a 0.5-L separatory funnel. Saturated sodium chloride solution (10 mL) was added, and the pesticide was extracted by shaking vigorously 3 times with petroleum ether (100, 50, and 50 mL). Layers were allowed to separate (~10 min) after each shaking. The petroleum ether extract was filtered through anhydrous granular sodium sulfate and evaporated almost to dryness on a rotary evaporator (40 °C). About 20 mL of petroleum ether was then added to the residue, and the solvent was evaporated again to eliminate the traces of acetone. A few milliliters (~5 mL) of petroleum ether were added to dissolve the residue for the Florisil cleanup step.

For the apple samples only half of the petroleum ether extract corresponding to 25 g of plant material was taken to the cleanup step, since it was found that insoluble material was left in the concentrated petroleum extract subjected to the cleanup when 50-g samples were used.

Florisil Column Chromatography. The chromotograph tube was packed (dry) with a small plug of glass wool and 4 g of Florisil and topped with anhydrous granular sodium sulfate (2 cm). The column was tapped to settle the adsorbent. The plant extract was transferred to the column, petroleum ether (~10 mL) was used for rinsings of the flask containing the extract. The pesticide was eluted with 1% acetone in petroleum ether (120 mL) at a rate of 2–3 mL/min. The eluate was evaporated almost to dryness with a rotary evaporator (~40 °C), and the residue was dissolved in a suitable amount of acetone for the gas chromatographic determination.

Sample Extraction and Solvent Partitioning for Cottonseeds. A representative sample (120 g each) of cottonseeds was blended for 15 min with 60 mL of chloroform, quantitatively transferred to thimbles, and subjected to continuous extraction with chloroform for 18 h. The chloroform was evaporated with a rotary evaporator (~40 °C), and the few remaining milliliters of chloroform in the resulting oily extract were evaporated in a weighed small centrifuge tube. After all traces of chloroform had been removed from the extract, the centrifuge tube was weighed again, and the weight of the extract was recorded (in order to calculate the percentage of oil in the sample). Propylene carbonate (25 mL) was added to a 20-g aliquot of oily extract, and the mixture was shaken vigorously for 2 min and centrifuged at 1500 rpm for 10 min. The propylene carbonate phase (the lower layer) was transferred by pipet into a small separatory funnel, and the oil in the centrifuge tube was again extracted with propylene carbonate (15 mL). After further centrifugation, the propylene carbonate extract was added to the first portion in the separatory funnel (for the removal of traces of oil). The two propylene carbonate extracts were combined, and three 10-mL aliquots of the (40 mL) combined extract were transferred into large centrifuge tubes, 50 mL of water was added to each, and the mixture in each tube was partitioned with petroleum ether (50 mL). After shaking vigorously for 2 min and centrifugation, the content of the tubes (~150 mL) was passed through a layer of anhydrous sodium sulfate (to remove water). The petroleum ether extract was then reduced to a few milliliters (rotary evaporator, 40 °C) and subjected to the same Florisil cleanup procedure described above. The resulting eluate was evaporated almost to dryness, and acetone (minimum 10 mL) was added for quantitative determination by GLC.

The amount of fenvalerate in the whole sample was calculated by using the recorded weight of oil obtained from the extraction. Results are reported in ppm of fenvalerate in cottonseeds.

Gas Chromatographic Analysis. Under the GLC conditions used in this study, fenvalerate gives two peaks due to the two diastereoisomeric pairs (*RR*, *SS*, *RS*, and *SR*) (Holmstead et al., 1978). The retention time was ~7 min. The peak height method was used for quantitation and the first peak was used for measurement.

Field treated and fortified sample extracts were compared with appropriate standards under identical conditions. In some cases the samples were chromatographed under slightly different conditions and the retention times and responses were not directly comparable.

Table I. Recoveries

crop	added, ppm	no. of samples	recovery, %[a]
grapes	0.5	3	97 ± 3
	0.1	3	96 ± 4
	0.02	3	98 ± 4
	0.005	3	96 ± 2
pepper	1.0	3	99 ± 2
	0.1	2	94 ± 2
	0.02	3	97 ± 2
	0.01	3	98 ± 0
	0.005	3	97 ± 4
apples	1.0	3	99 ± 2
	0.1	3	99 ± 2
	0.01	3	97 ± 4
cottonseeds	0.2	3	93 ± 3
	0.1	2	88 ± 2
	0.02	3	83 ± 8
	0.01	3	78 ± 1

[a] Mean ± standard deviation.

RESULTS AND DISCUSSION

The recovery of fenvalerate added to untreated plants in the first extraction step was investigated for all the plant material studied. Table I records the average recovery of fenvalerate in the plant materials tested for different levels of initial fortification with fenvalerate. The average recovery of fenvalerate for grapes fortified at levels of 0.005–0.5 ppm was 96–98% and 94–99% for peppers fortified at 0.005–1.0 ppm. For apples fortified at levels of 0.01–1.0 ppm, the average recovery was 97–99% (Table I). For grapes, peppers, and apples, typical chromatograms of standards, untreated products, and samples fortified with fenvalerate at different levels and field-treated samples are shown in Figures 1–3.

For grapes and peppers, the minimum detectable level was 0.005 ppm. A limit of detection of ~0.07 ppm can be

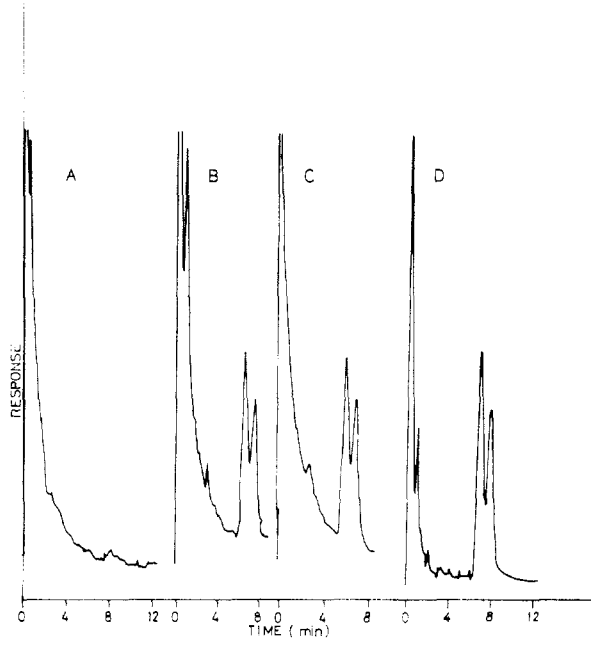

Figure 1. Chromatograms of grapes. (A) Extract equivalent to grapes (50 mg); (B) extract equivalent to grapes (8 mg) fortified with 0.1 ppm of fenvalerate; (C) standard fenvalerate (0.9 ng); (D) extract equivalent to grapes (2.5 mg) from field-treated sample, taken from agricultural trial (application: 75 g of a.i./ha, 13 days after the last application).

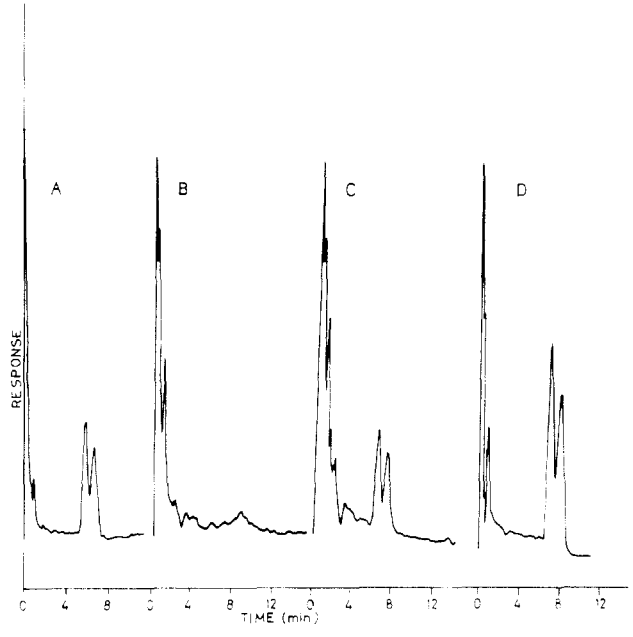

Figure 2. Chromatograms of peppers. (A) Standard fenvalerate (0.5 ng); (B) extract equivalent to pepper (40 mg); (C) extract equivalent to pepper (40 mg) fortified with 0.01 ppm of fenvalerate; (D) extract equivalent to pepper (20 mg) from field-treated sample taken from agricultural trial (application: 150 g of a.i./ha, day of the last application).

expected for the apple samples.

For the cottonseed samples, fenvalerate was added at levels of 0.01–0.2 ppm in the extraction step. Average recoveries were 83–93% at 0.02–0.2 ppm and 78% at 0.01 ppm (Table I).

Chromatograms of an untreated cottonseed sample, a sample fortified with 0.01 ppm of fenvalerate, and a field treated sample extract (600 g of a.i./ha) are shown in Figure 4.

The efficiency of the Soxhlet extraction for cottonseed

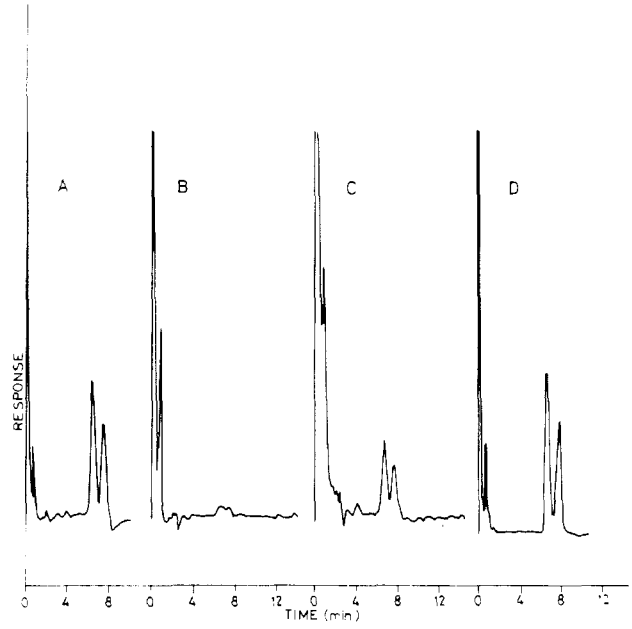

Figure 3. Chromatograms of apples. (A) Standard fenvalerate (0.2 ng); (B) extract equivalent to apples (7.5 mg); (C) extract equivalent to apples (7.5 mg) fortified with 0.01 ppm of fenvalerate; (D) extract equivalent to apples (0.25 mg) from field-treated samples (application: 450 g of a.i./ha, day of the last application).

was checked in two 80-g field-treated samples by subjecting the Soxhlet-extracted seeds to an additional extraction by blending twice with acetone. Water was added to the acetone extract and the mixture partitioned with petroleum ether. Since oil (~2 g) still remained in the concentrated petroleum ether extract, the rest of the procedure was carried out according to the principles described for the cottonseeds method.

The residues of fenvalerate found in these extracts were at the limit of detection of acetone-blending method, i.e., 0.006 ppm in both samples (recovery at 0.01 ppm was ~80%). The residues found by Soxhlet extraction alone were 0.065 and 0.059 ppm. The very low values obtained in the double-extracted seed samples indicate that most of the fenvalerate residue (~90%) had been recovered by the Soxhlet extraction.

In the partitioning step for cottonseeds, the same centrifuge tube should be used for shaking and centrifugation in order to avoid losses and to save time. Special care must be taken to removal all traces of oil and propylene carbonate from the petroleum ether extract (before the Florisil cleanup) as this can affect the quality of the chromatograms.

A parallel set of experiments on cottonseeds was carried out by using smaller samples with correspondingly smaller amounts of solvents and reagents in the extraction and partitioning steps. Approximately the same values were obtained for fenvalerate recovery, but the sensitivity of the method was somewhat lower.

The Soxhlet method described in this paper appears to be applicable to other synthetic pyrethroids, for example, residues of cypermetrin [1(*RS*)-*cis*,*trans*-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate], another commercial synthetic pyrethroid determined in samples of cottonseeds taken from field trials. The recovery values and the limit of detection were about the same as those obtained for fenvalerate.

For demonstration of the practical application of the method and to provide information about the behavior of fenvalerate in field-treated plants, samples of grapes, peppers, and apples and cottonseeds were obtained from

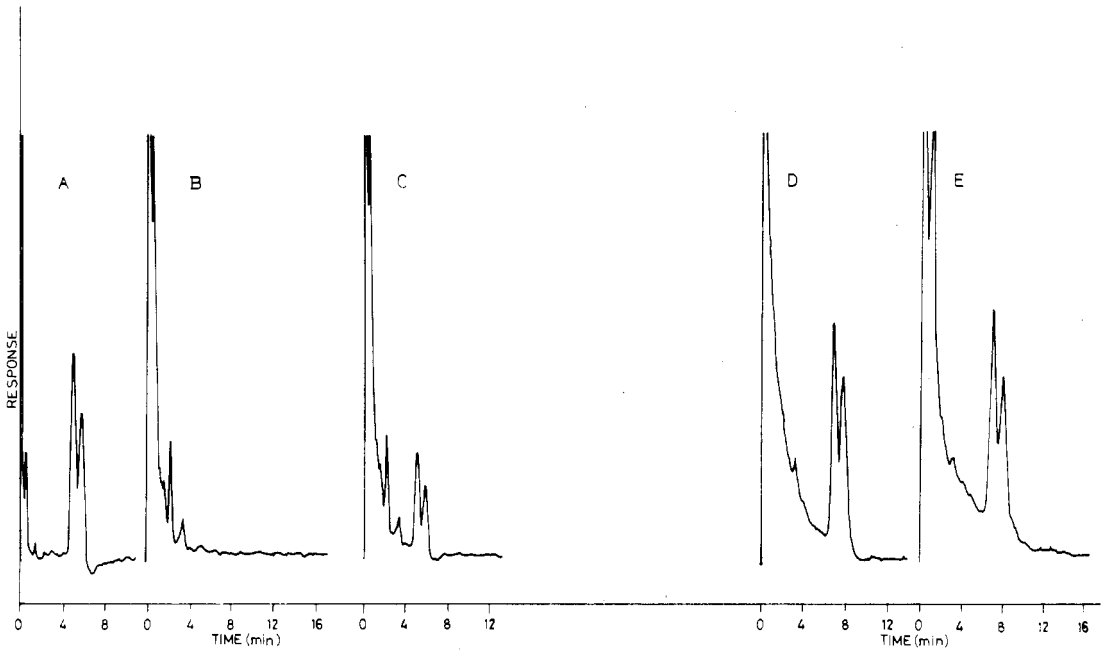

Figure 4. Chromatograms of cottonseeds. (A) Standard fenvalerate (0.2 ng); (B) extract equivalent to cottonseed (24.3 mg); (C) extract equivalent to cottonseed (17.8 mg) fortified with 0.01 ppm of fenvalerate; (D) standard fenvalerate (1.0 ng); (E) extract equivalent to cottonseed (2.5 mg) from field-treated sample (application: 600 g of a.i./ha).

Table II. Residues of Fenvalerate in Grapes (Field Trials)

days after last application	residues,[a] ppm, after application of 37.5 g of a.i./ha (25 L)	75 g of a.i./ha (25 L)
0[b]	0.08 ± 0.02	0.24 ± 0.036
6	0.08 ± 0.02	0.20 ± 0.14
13	0.02 ± 0.05	0.33 ± 0.14
20	0.025 ± 0.01	0.28 ± 0.06

[a] Average of three replicates ± standard deviation. Date of the last application: Aug 15, 1979. [b] Day of last application.

Table III. Residues of Fenvalerate in Peppers (Field Trials)

days after last application	residues,[a] ppm, after application of 150 g of a.i./ha	300 g of a.i./ha
0[b]	0.15 ± 0.13	0.08 ± 0.03
7	0.12 ± 0.075	0.12 ± 0.0
14	0.06 ± 0.015	0.11 ± 0.05

[a] Average of three replicates ± standard deviation. Date of the last application: Oct 29, 1979. [b] Day of last application.

agricultural trials carried out by Agan Co. and analyzed. The results obtained are shown in Tables II–V.

The level of fenvalerate residue in grapes (37.5 g of a.i./ha) decreased from an average of 0.08 ppm on the day of application to 0.025 ppm 20 days later. Doubling the application rate (75 g of a.i./ha) resulted in a higher residue (0.20–0.33 ppm) which did not decrease with time (Table II).

Residues in pepper sprayed with 150 g of a.i./ha decreased from an average of 0.15 ppm on the day of application to 0.06 ppm 14 days later. Doubling the application did not significantly change the residue level (Table III).

The level of fenvalerate residue in apples sprayed with 450 g of a.i./ha decreased from an average of 1.1 ppm on the day of application to 0.45 ppm 1 month later (Table IV).

Table IV. Residues of Fenvalerate in Apples (Field Trials)

days after last application	residues,[a] ppm, after application of 450 g of a.i./ha
0[b]	1.1 ± 0.3
8	0.6 ± 0.3
30	0.45 ± 0.6

[a] Average of three replicates ± standard deviation. Date of last application: Aug 9, 1979. [b] Day of last application.

Table V. Residues of Fenvalerate in Cottonseeds (Field Trials)

residues,[a] ppm, after application of 300 g of a.i./ha[b]	600 g of a.i./ha[c]
0.012 ± 0.01	0.053 ± 0.008

[a] Date of the last application: Sept 2, 1979. Date of sampling: Nov 19, 1979. [b] Average of five replicates ± standard deviation. [c] Average of eight replicates ± standard deviation.

The average values for fenvalerate residue in cottonseeds as shown in Table V were 0.012 ppm for the lower and 0.053 ppm for the higher rate of application at 77 days after the last application.

Although the residue levels for the grapes, peppers, and apples treated with the lower application rate decreased with time in each case, additional samples will have to be analyzed in order to fully understand the influence of the higher application rate on residue levels.

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Gas Chromatographic Quantitative Analysis and Persistence of Dimethoate and Dimethoxon Residues on and in Wheat Plants

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Wheat plants fortified with dimethoate and dimethoxon were blended and extracted with chloroform. The concentrated extract was passed through a silica gel column, evaporated, and injected into a gas chromatograph equipped with a flame photometric detector and a column containing 12% OV-101 and 1.2% Carbowax 20M on Chromosorb W. Recoveries of dimethoate and dimethoxon were about 92% and 89%, respectively, in the range of 0.03–10 ppm. Dimethoate was applied to wheat plants seeded on three dates. Samples were collected 0–17 days after application. The dimethoate residues were 63.2, 41.5, and 17.4 ppm immediately after application and were 0.02, 0.36, and 0.62 ppm by 17 days after application. Dimethoate concentration decreased more rapidly on the younger wheat plants. Dimethoxon concentrations increased and then decreased more rapidly on the younger wheat plants.

Dimethoate, *O,O*-dimethyl *S*-(*N*-methylcarbamoylmethyl)phosphorodithioate, is an organophosphorous insecticide that is registered in Canada for insect control in cereal, oilseeds, commercial and home gardens, forage crops, trees, shelterbelts and shrubs (Agriculture Canada, 1979). In the province of Saskatchewan one of the main uses of dimethoate is for grasshopper control in wheat.

A number of analytical methods for dimethoate and its oxygen analogue, dimethoxon [*O,O*-dimethyl *S*-(*N*-methylcarbamoylmethyl)phosphorothioate] residues have appeared in the literature based on gas chromatography. Storherr and Watts (1969) described a method for the gas chromatographic (GC) analysis of dimethoate and dimethoxon in field-sprayed kale using a 2% diethylene glycol succinate column, and Woodham et al. (1974a,b) determined dimethoate and dimethoxon residues in citrus crops by using a DC-200 column. Steller and Pasarela (1972) used a column based on a light loading Versamid 900 and a heavier loading of DC-200. Also small quantities of poly(ethylene glycol) needed to be added to every injection to maintain satisfactory performance. MacNeil (1975) described the use of OV-17 for determing dimethoate and dimethoxon residues on cherries.

The following method was developed to determine dimethoate and dimethoxon on and in wheat plants by using a column of OV-101 and Carbowax 20M double coating, and this method was used for the determination of the rate of disappearance of dimethoate and dimethoxon residues on and in field-sprayed wheat plants.

EXPERIMENTAL SECTION

Apparatus. A Beckman GC-5 gas chromatograph fitted with a Melpar flame photometric detector and phosphorous filter (526 nm) was used. The chromatographic column was a 1.8-m glass column, 4-mm inside diameter. The column packing was prepared by first coating Chromosorb W (100–200 mesh; acid wash; dimethylchlorosilane treated) with 1.2% Carbowax 20M and then applying a second coating of 12% OV-101.

The operating conditions were column, injector, and detector temperatures of 180, 210, and 160 °C, respectively. The flow rate of carrier gas, helium, was 100 mL/min, whereas those of hydrogen, air, and oxygen were 190, 90, and 30 mL/min, respectively.

Reagents. All solvents were glass-distilled residue-free grade. Analytical standards of dimethoate (99.8% purity), dimethoxon (analytical standard), and malathion (99.1% purity) were obtained from American Cyanamid Co., and stock solutions were prepared in ethyl acetate. Malathion was used as the internal standard. Hyflo Super-Cel (Fischer Scientific Co.) and silica gel, Davison grade 12 (Fischer Scientific Co.), were used as received.

Wheat Treatments. Field experiments were carried out at Saskatoon, Saskatchewan, Canada, where wheat was seeded on May 1, May 15, and June 1. The experimental plots were 20 × 20 m, and there were four plots for each seeding date. On June 28, the date of application of dimethoate, the wheat was in three stages of development: the "boot" stage, second node visible, and tillering for the May 1, May 15, and June 1 plots, respectively. Dimethoate was applied by a self-propelled boom sprayer at the recommended rate of 420 g of active ingredient/ha (6 oz/acre). The first samples were taken ~2 h after aplication, with further samples being taken at several intervals over the next 3 weeks. Samples of wheat foliage were cut off at ground level, transferred to plastic bags, sealed, and frozen at −20 °C until analyzed.

Extraction. Whole wheat plants were chopped into pieces less than 1 cm long and mixed thoroughly. A subsample (20 g) was blended with chloroform (150 mL) for 5 min at high speed in Virtis homognizer. Hyflo Super-Cel (7 g) was added to the homogenized sample and mixed. The sample was filtered through a pad of Hyflo Super-Cel (3 g) on a filter paper on a Buchner funnel with vacuum. The filter residue was scraped back into the homogenizer

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