Toxicities of Insecticide Residues on Loblolly Pine Foliage to Leaffooted Pine Seed Bug Adults (Heteroptera: Coreidae)[1,2]

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ABSTRACT Fourteen commercial and four experimental formulations of insecticides, when applied to loblolly pine, *Pinus taeda* L., foliage in a simulated high volume spray, varied in their residual contact toxicities to adult leaffooted pine seed bugs, *Leptoglossus corculus* (Say). Deltamethrin emulsifiable concentrate (EC), the most toxic insecticide tested, was 7 times as toxic to adult females as the standard, azinphosmethyl EC. Azinphosmethyl wettable powder (WP) was slightly more toxic to females than the EC at LC_{90}; all other insecticides were less toxic than azinphosmethyl EC to females (half or less). Azinphosmethyl EC, deltamethrin EC, permethrin (Pounce) EC, and phosmet WP were equally toxic to both sexes, while permethrin (Ambush) EC and fenvalerate EC were both more toxic to males than females. Azinphosmethyl WP was more toxic to females than males. Several of the insecticides, including propoxur WP, malathion EC and chlorpyrifos EC, had relatively low residual contact toxicities but were very toxic to seed bug adults as fumigants. The pyrethroids permethrin, deltamethrin and fenvalerate had a relatively rapid knockdown effect, in contrast to the slower-acting organophosphorous insecticides.

KEY WORDS *Leptoglossus corculus,* leaffooted pine seed bug, *Pinus taeda,* azinphosmethyl, deltamethrin, fenvalerate, permethrin, phosmet, propoxur, malathion, methomyl.

The leaffooted pine seed bug, *Leptoglossus corculus* (Say), is a pest in pine seed orchards in the eastern United States (Ebel et al. 1980). Feeding by both nymphs and adults greatly reduces seed yields. Chemical control of seed bugs and several species of coneworms is necessary in seed orchards to prevent substantial reductions in yield of genetically improved seed used for seedling production in the southern U.S. Contact toxicities of 34 insecticides were determined for 2nd-stage nymphs of *L. corculus* by DeBarr and Nord (1978). Twelve insecticides also were tested on adults by Nord and DeBarr (1983). In those tests, solutions of technical grade insecticide were applied topically.

Here I report results of another laboratory bioassay that was designed to evaluate the residual contact toxicities of commercial insecticide formulations applied to loblolly pine (*Pinus taeda* L.) foliage in a simulated high volume spray. For this test, adult seed bugs were exposed to the residual insecticide deposits by allowing them to walk on treated foliage. Results from other tests, involving the addition of spray adjuvants and artificial weathering, will be reported in a later paper.

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[2] Mention of a commercial or proprietary product does not constitute endorsement by USDA.

Materials and Methods

Insects. Seed bugs used in the bioassays of insecticide deposits were reared in the laboratory as described by Nord and DeBarr (1983). After the last molt, adults were separated by sex and kept in containers (4.7 liters) for 2 weeks in the insectary before treatment so that mortality related to the last molt could occur. Before they were placed on treated foliage, adults (225-270) from 5-6 holding containers were placed together in a 38 liter aquarium filled with CO_2 for less than 10 min to anesthetize them. Groups of 10-20 seed bugs then were randomly selected from the aquarium and put in the test containers. Thus, the insects were thoroughly mixed to avoid any bias due to conditions in the rearing or holding containers.

Insecticides and Treatments. Eighteen insecticide formulations were tested: deltamethrin EC (emulsifiable concentrate) (=FMC 45498 0.21 EC) (FMC Corp., Philadelphia, PA); azinphosmethyl WP (wettable powder) (= Guthion 50 WP) and azinphosmethyl EC (= Guthion 2L) (Mobay Chemical Corp., Kansas City, MO); fenvalerate WP (= SD 43775 25 WP) and fenvalerate EC (= Pydrin 2.4EC) (Shell Development Co., Modesto, CA); permethrin-P EC (= Pounce 3.2EC) (FMC Corp., Philadelphia, PA); permethrin-A EC (= Ambush 2E) (ICI Americas, Inc., Richmond, CA); phosmet WP (= Imidan 50WP) and fenitrothion EC (= Sumithion 8E) (Stauffer Chemical Co., Westport, CT); propoxur WP (= Baygon 70WP), sulprofos (= BAY NTN 9306 6EC) and trichlorfon SP (soluble powder) and EC (= Dylox 80% SP and Dylox 4) (Mobay Chemcial Corp., Kansas City, MO); malathion EC (= Security Malathion 5E) (Woolfolk Chemical Works, Ft. Valley, GA); methomyl WSL (water soluble liquid) (= Lannate L 1.8 WSL) (E. I. duPont de Nemours & Co., Wilmington, DE); chlorpyrifos-methyl EC (= 4EC) and chlorpyrifos EC (= Dursban 4E) (the Dow Chemical Co., Midland, MI); and dimethoate EC (= Cygon 400-4E) (American Cyanimid Co., Wayne, NJ). All except deltamethrin EC, chlorpyrifos-methyl EC, sulprofos EC and fenvalerate WP were commercial formulations. Untreated shoots were used for controls.

A stock aqueous solution or suspension was made on a wt/wt basis and lower concentrations were prepared by serial dilution. Preliminary tests of three widely spaced concentrations, e.g. 0.01, 0.1 and 1.0% active ingredient (AI) were made with 10 adults per concentration to determine the range of concentrations to use in the final tests. Final tests consisted of 3 or more replications of 6 to 8 concentrations with 20 insects per concentration and 20 control insects for each group of adults that were mixed each day for testing. Each replication was conducted on three or more different days. Thus, about 300-500 insects were used to bioassay each insecticide. Insecticides with LC_{90}'s $> 0.5\%$ (AI) in the preliminary tests were considered to be relatively ineffective or too expensive (or both) for operational use in seed orchards and were not tested further.

Loblolly pine shoots (about 25 cm long) were submerged for about 5 sec in 100 ml of dilute insecticide in a 100 ml graduated cylinder under a fume hood to simulate a hydraulic spray to run-off. A wide-neck powder funnel set on top of the cylinder was used to compress the needles so they could be inserted into the cylinder. Some needles were removed to facilitate their passage through the funnel. Dipped shoots were inverted and clipped to a wire inside the hood until nearly dry (about 20 min). Then, the base of the shoot was cut at an angle and inserted through the straw hole in the lid of a plastic cup filled with tap water. The shoots were then allowed to dry thoroughly overnight and used for the bioassay the next day.

Bioassay and Handling after Treatment. The day after treatment, each treated shoot with its cup was put into a paper holding container (4.7 liter) with 10 or 20 adult seed bugs. Holding containers had a screen lid and screen insert in the bottom for ventilation. The tips of the foliage were clipped so that the needles spread out naturally (i.e. they were not bunched) and only needle tips came near to or just in contact with the screen. For food and moisture, slash pine, *P. elliottii* Engelm. var. *elliottii,* seed (from an untreated seed orchard) and moist pieces of rolled cotton under separate glass Petri dish covers (100 mm diameter) were put on the top screen of the holding containers in areas away from the foliage. Holding containers were put on open wire shelves in a ventilated room at 27°C and 70-80% relative humidity. The seed bugs were relatively active in the containers and were in contact with the treated foliage much of the time.

Preliminary tests showed that ventilation in the standard holding containers was inadequate to prevent fumigation by some insecticide deposits. Strong fumigants, such as propoxur, not only killed adults in standard holding containers ventilated at the bottom and top and held on open wire shelves but also killed adults on untreated foliage in control containers on shelves above and below. Therefore, to identify those insecticides that had highly volatile deposits, each formulation was checked for fumigation effects and provision was made to prevent the accumulation of toxic vapors in the holding containers (see active ventilation system below). In these fumigation tests, 0.25 liter containers were used to hold 5 *L. corculus* adults and prevent contact with treated foliage. These smaller containers were placed inside the standard holding containers with treated loblolly pine shoots (dipped in 0.5-1% (AI) concentrations of insecticide). Both small and large holding containers had screen lids and screen bottoms. If no seed bug mortality occurred after one week, I assumed that fumigation was not a problem and used the standard screened top and bottom holding containers and open wire shelving in the preliminary and final evaluations. Fumigation tests indicated that an active ventilation system was required during the tests for measuring residual contact toxicities of chlorpyrifos EC, chlorpyrifos-methyl EC, malathion EC, methomyl WSL, trichlorfon EC, propoxur WP, fenitrothion EC, and dimethoate EC. Results with sulprofos EC were not clear so the active ventilation system was used.

In a test situation, lethal toxic vapors of propoxur were eliminated with a 51-cm window fan blowing toward the holding containers at a distance of 100 cm. This led me to devise a more efficient system to draw air continuously through the holding containers and exhaust it from the holding room. This active ventilation system consisted of an elongate plywood box (0.62 × 0.65 × 2.0 m) or plenum with sixteen 18-cm circular openings in the top (into which the 4.7-liter holding containers were set with a snug fit) and with one end open (0.40 × 0.47 m opening), through which the air was drawn. The 51-cm fan in the open end of the plenum drew air through the tops of the holding containers, out of the bottoms of the holding containers, and through the plenum. A second fan exhausted the air outside of the building.

Evaluation and Data Analysis. Mortality counts were made at 24-hour intervals until all insects were dead or moribund (i.e., immobile or only able to walk with jerky, uncoordinated movements) or until 7 days had passed, whichever occurred first. Mortality at 4 days was used for the construction of the concentration-mortality regression lines because cumulative mortality leveled off at that point. Numbers of dead and moribund were combined for probit analysis.

Concentration-mortality data for the insecticides were analyzed with the probit option of POLO (Russell et al. 1977, Robertson et al. 1980). The program adjusted for the number dead and moribund by taking into consideration natural mortality occurring in control containers. Likelihood ratio tests were used to determine the significance of sex in response to 7 of the insecticide formulations (Savin et al. 1977). The hypothesis of parallelism was tested to determine whether changes in response per unit concentration (i.e., slopes) were equivalent. The hypothesis of equality was tested to ascertain whether both response thresholds (i.e., intercepts) and change in response per unit concentration were statistically equivalent. The same tests also were used to compare responses to different formulations of the same insecticide.

Results and Discussion

Deltamethrin EC was the most toxic insecticide, 7 times as toxic as azinphosmethyl EC, the only insecticide registered for use against pine seed bugs and coneworms in southern pine seed orchards when these tests were conducted (Table 1). Azinphosmethyl WP was slightly more toxic to female *L. corculus* than the EC at LC_{90} based on the toxicity index (TI) (= LC_{90} azinphosmethyl EC/LC_{90} candidate), but the difference was not significant as reported below. All other insecticides were less toxic than azinphosmethyl EC with TI's below 0.5. Malathion EC and methomyl WSL were only 0.03 and 0.02 times as toxic, respectively, as azinphosmethyl EC. Although they were quite toxic to *L. corculus* as fumigants, the more volatile insecticides, such as propoxur WP, malathion EC and chlorpyrifos EC, had quite low residual contact toxicities. The overnight drying period probably created a bias against the volatile insecticides. Preliminary tests indicated that sulprofos EC, chlorpyrifos-methyl EC, and dimethoate EC had an LC_{90} of > 0.5% (AI), while trichlorfon SP and EC, chlorpyrifos EC, and fenitrothion EC had an LC_{90} > 1.0% (AI).

The LC_{90} for azinphosmethyl EC was 0.014% (AI) for females. The registered rate of this material used as a high volume spray is 0.18% (AI), about 13 times the LC_{90}. Subsequent field tests confirmed that the registered concentration for azinphosmethyl EC is very efficacious (Nord et al. 1984, 1985). Phosmet WP had an LC_{90} for female *L. corculus* of 0.03% (AI), but like azinphosmethyl EC, effective field concentrations were much higher (about 7-10 fold) (Nord et al. 1984, 1985). The LC_{90} for permethrin as the active ingredient in Ambush EC, ranged between 0.018 and 0.061% (AI) (Table 1). In field tests, concentrations as low as 0.0125% (AI) provided good seed bug control (Nord et al. 1984, 1985). Likewise, the LC_{90} for fenvalerate EC was 0.024-0.048% (AI) for seed bug adults (Table 1), yet field tests showed good control of seed bugs at concentrations as low as 0.0125% (AI) (Nord et al. 1984, 1985). Fenvalerate EC, Ambush EC and Pounce EC are now registered at 0.025% (AI) for control of seed bugs and coneworms in southern pine seed orchards. The successful control of seed bug damage obtained with the relatively low rates of permethrin may be due to a higher contact toxicity of this insecticide to the 2nd-stage nymphs than adult females of *L. corculus*, as shown in earlier tests (DeBarr and Nord 1978, Nord and DeBarr 1983). In contrast, fenvalerate was equally toxic to 2nd-stage nymphs and adult females, while deltamethrin was more toxic to adult females than nymphs. The organophosphates phosmet and azinphosmethyl were more toxic to 2nd-stage nymphs than adults. One apparent reason that high concentrations of organophosphates, such as azinphosmethyl and phosmet, are required for successful control of seed bugs in pine seed orchards is that they are more easily washed off the foliage (Nord, unpublished). Deposits of the pyrethroids on pine foliage are much more resistant to rain washing than those of azinphosmethyl EC or phosmet WP.

Toxicity indexes of phosmet and permethrin were similar in both the residual toxicity tests and the contact toxicity tests with female seed bugs (Nord and DeBarr 1983). Deltamethrin was the most toxic in both cases, but relatively much more toxic in the contact tests than in the residual tests, (i.e., 29 versus 7 times more toxic than azinphosmethyl, respectively). Chlorpyrifos was one of the least toxic to *L. corculus* in both tests, but it appeared to be much less toxic when applied to the host foliage. Dissimilarities in results were noted in the case of fenvalerate, which was 1.4 times more toxic to females than azinphosmethyl in the contact tests, but only one third as toxic in residual tests. Malathion had a much higher TI in the contact tests than in the residual tests.

Table 1. Residual toxicities of insecticide formulations on loblolly pine foliage to leaffooted pine seed bug adults.

Insecticide	Sex	No. Insects	Slope (± SE)*	LC_{90} (95% CL)†	TI‡	Parallelism & Equality of Concentration-Mortality Lines for ♀♀ vs ♂♂§
Deltamethrin EC	F	479	1.79(0.28)	0.002(0.001-0.004)	7.00	∥ =
	M	360	1.65(0.35)	0.002(0.002-0.004)		
Azinphosmethyl WP	F	300	4.83(0.67)	0.013(0.011-0.015)	1.08	∥ ≠
	M	379	4.33(0.45)	0.016(0.014-0.018)		
Fenvalerate WP	M	300	3.02(0.42)	0.013(0.011-0.017)	-	- -
Azinphosmethyl EC	F	360	3.22(0.45)	0.014(0.012-0.020)	1.00	∥ =
	M	297	3.83(0.59)	0.015(0.013-0.019)		
Permethrin-P EC	F	399	2.01(0.31)	0.030(0.024-0.045)	0.47	∥ =
	M	480	2.24(0.25)	0.031(0.026-0.039)		
Phosmet WP	F	318	3.57(0.50)	0.030(0.027-0.038)	0.47	∥ =
	M	529	4.32(0.45)	0.032(0.029-0.036)		
Fenvalerate EC	F	359	1.80(0.28)	0.048(0.037-0.071)	0.29	∥ ≠
	M	477	1.69(0.23)	0.024(0.019-0.032)		
Permethrin-A EC	F	340	1.56(0.29)	0.061(0.040-0.131)	0.23	∥ ≠
	M	380	1.97(0.23)	0.018(0.011-0.051)		
Propoxur WP	F	380	5.61(0.67)	0.217(0.184-0.311)	0.06	- -
Malathion EC	F	379	2.85(0.47)	0.413(0.325-0.730)	0.03	- -
Methomyl WSL	F	398	3.89(0.50)	0.749(0.596-1.368)	0.02	- -

* Standard error.

† Lethal concentration (percent active ingredient) for 90% mortality with 95% confidence limits.

‡ Toxicity Index, i.e. toxicity of candidate to females relative to that of azinphosmethyl EC: TI = (LC_{90} azinphosmethyl EC) / (LC_{90} candidate).

§ ∥, lines parallel; ∦, lines not parallel; =, lines equal; ≠, lines not equal
Hyppotheses of parallelism and equality rejected with significance level p = 0.05.

Azinphosmethyl EC, deltamethrin EC, permethrin (as the active ingredient in Pounce EC), and phosmet WP were equally toxic to both sexes (Table 1). Permethrin, as the active ingredient in Ambush, and fenvalerate EC were uniformly more toxic to males than to females. Azinphosmethyl WP was more toxic to females than males. In all three cases in which differences in toxicity between sexes were found, only differences in response threshold were responsible not changes per unit concentration.

The responses to different formulations with the same active ingredient also were compared (Table 2). Azinphosmethyl EC and 50WP were equally toxic to males and females. The concentration-mortality lines of Pounce and Ambush formulations on males and females differed in response threshold only (Table 2). Pounce was more toxic than Ambush to females whereas Ambush was more toxic to males (Tables 1 and 2). In the case of the response of males to fenvalerate 25WP and EC, the wettable powder was more toxic. These formulations differed in both response threshold and changes per unit concentration.

Adult *L. corculus* on foliage treated with organophosphates showed no effects during the first 2-3 hours. However, some mortality occurred after 12-18 hrs., and it gradually increased through the 4th day. In contrast, the pyrethroids caused rapid knockdown of adults. On foliage treated with rates near the LC_{90}, most of the insects that climbed onto treated foliage fell off within 20 min. Seed bugs falling out of the treated foliage did not stand normally and did not walk and appeared unable to walk. By the next morning some treated adults recovered and

Table 2. Comparison of concentration-mortality lines for different formulations of the same insecticide.

Insecticide	Sex	Parallelism & Equality of Concentration-Mortality Lines*	
Azinphosmethyl EC vs Azinphosmethyl WP	M	∥	=
Azinphosmethyl EC vs Azinphosmethyl WP	F	∥	=
Permethrin-A EC vs Permethrin-P EC	M	∥	≠
Permethrin-A EC vs Permethrin-P EC	F	∥	≠
Fenvalerate WP vs Fenvalerate EC	M	∦	≠

* ∥, lines parallel; ∦, lines not parallel; =, lines equal; ≠, lines not equal. Hypotheses of parallelism and equality rejected with significance level p = 0.05.

walked normally again, but mortality was usually 80-90% after 24-48 hrs. Knockdown responses to pyrethroids have been reported by Chen et al. (1985). The quick knockdown action of pyrethroids on *L. corculus* adults makes these insecticides potentially useful for sampling populations by spraying tree crowns and counting insects falling on a drop cloth below (Nord and DeBarr, unpublished; DeBarr and Barber, unpublished).

The determination of LC_{90}'s of various insecticides and formulations on loblolly pine foliage for *L. corculus* adults in this study, and subsequent studies on the resistance of spray deposits to washoff by rain, enabled us to choose candidates and rates for field testing in pine seed orchards (Nord et al. 1984, 1985).

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References Cited

Chen, J. S., C. J. Lee, M. G. Yao, and C. N. Sun. 1985. Effect of pyrethroids on knockdown and lack of coordination responses of susceptible and resistant diamondback moth (Lepidoptera: Plutellidae). J. Econ. Entomol. 78: 1198-1202.

DeBarr, G. L. and J. C. Nord. 1978. Contact toxicity of 34 insecticides to second-stage nymphs of *Leptoglossus corculus* (Hemiptera: Coreidae). Can. Entomol. 110: 901-06.

Ebel, B. H., T. H. Flavell, L. E. Drake, H. O. Yates III and G. L. DeBarr. 1980. Seed and cone insects in southern pines. U. S. Dept. Agric., Forest Service. Gen. Tech. Rep. SE-8, 43 p.

Nord, J. C. and G. L. DeBarr. 1983. Contact toxicities of 12 insecticides to *Leptoglossus corculus* adults (Hemiptera: Coreidae). Can. Entomol. 115: 211-14.

Nord, J. C., G. L. DeBarr, N.A. Overgaard, W. W. Neel, R. S. Cameron and J. F. Godbee. 1984. High-volume applications of azinphosmethyl, fenvalerate, permethrin, and phosmet for control of coneworms (Lepidoptera: Pyralidae) and seed bugs (Hemiptera: Coreidae and Pentatomidae) in southern pine seed orchards. J. Econ. Entomol. 77: 1589-95.

Nord, J. C., G. L. DeBarr, L. R. Barber, J. C. Weatherby and N. A. Overgaard. 1985. Low-volume applications of azinphosmethyl, fenvalerate, and permethrin for control of coneworms (Lepidoptera: Pyralidae) and seed bugs (Hemiptera: Coreidae and Pentatomidae) in southern pine seed orchards. J. Econ. Entomol. 78: 445-50.

Robertson, J. L., R. M. Russell, and N. E. Savin. 1980. POLO: A user's guide to Probit Or LOgit analysis. U.S. Dept. Agric., Forest Service. Gen. Tech. Rep. PSW-38. 15 p.

Russell, R. M., J. L. Robertson and N. E. Savin. 1977. POLO: A new computer program for probit analysis. Bull. Entomol. Soc. Am. 23: 209-13.

Savin, N. E., J. L. Robertson and R. M. Russell. 1977. A critical evaluation of bioassay in insecticide research: likelihood ratio tests of dose-mortality regression. Bull. Entomol. Soc. Am. 23: 257-66.