

EFFICACY OF FIPRONIL AERIALY APPLIED IN OIL ADJUVANTS AND DRIFT
RETARDANTS AGAINST BOLL WEEVILS,
ANTHONOMUS GRANDIS BOHEMAN (COLEOPTERA:CURCULIONIDAE)

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ABSTRACT

Results of aerial application tests in the field and insecticide transfer tests in the laboratory showed that cottonseed oil was the most effective oil adjuvant to use with fipronil for controlling boll weevils under field conditions and for transferring fipronil from cotton leaf surfaces to boll weevils. The mineral oil and mineral oil + drift retardant more effectively transferred fipronil from cotton leaves to boll weevils than fipronil in water. Fipronil mixed with mineral oil was not as effective as fipronil mixed with cottonseed oil. Results of low volume aerial application of fipronil in aqueous mixtures with drift retardants showed that drift retardants slightly increased deposition of fipronil on cotton leaf surfaces compared to the standard application with water. Boll weevil mortalities from drift retardant-fipronil mixtures were slightly greater than an aqueous mixture of fipronil immediately after application. At 2 or 3 days after application, only drift retardant HM9733-A produced greater boll weevil mortality than that of fipronil in water.

INTRODUCTION

Fipronil is classified as a pyrazole insecticide that has excellent activity against insects infesting cotton. It was discovered in 1987 by Rhone Poulenc scientists and has been shown to be effective when applied to the soil, as a seed treatment, as a bait, or as a foliar spray. Fipronil interferes with the passage of chloride ions through the gamma-aminobutyric acid regulated chloride channel, disrupting activity of the central nervous system and causing death at high doses (Colliot et al. 1992).

Fipronil's effectiveness at low rates against boll weevils, *Anthonomus grandis* Boheman, (Scott et al. 1996) has drawn attention from the Animal and Plant Health Inspection Service (APHIS) Methods Development personnel involved in eradication of the boll weevil. Fipronil's low use rate (Shaw and Yang 1996) combined with its effectiveness against plant bugs (Shaw et al. 1997) make it well suited for use in Boll Weevil Eradication Programs. Although the ULV formulation of malathion is the primary insecticide presently used in Boll Weevil Eradication Programs throughout the country, fipronil has been identified as a possible alternative insecticide for use in eradication. Fipronil was shown to be effective against boll weevils at a rate of 28.0 g(AI)/ha applied in a volume of 0.58 l/ha with cottonseed oil as the adjuvant (Mulrooney et al. 1998).

The use of oils as adjuvants for insecticides increased with the increase in popularity of ultra-low-volume application of insecticides to cotton in the early 1980's. Oil diluents have been

proposed to have several advantages over water as a carrier. These include a more uniform droplet size, better coverage and canopy penetration, and greater persistence on the plant surface (McDowell et al. 1991). In a study conducted by Ochou et al. (1986), both plant (soybean and cottonseed) and petroleum oils synergized various pyrethroids against larvae of tobacco budworm, *Heliothis virescens* (Fab.), and adult house flies, *Musca domestica* L. Conversely Ochou et al. (1986) also found that these same oils were less synergistic or even antagonistic with more water-soluble organophosphate and carbamate insecticides. These authors contend that the mechanism of insecticide synergism by oils is unclear, but probably relates to polarity of the test insecticides. The more polar organophosphates and carbamate insecticides were less synergized than the less polar pyrethroids. Similarly, Treacy et al. (1986) demonstrated that the soybean oil enhanced toxicity of the pyrethroid, cyfluthrin, against the boll weevil, *Anthonomus grandis* Boheman, more than it did for selected carbamate and organophosphate insecticides. While Wolfenbarger and Guerra (1986) found permethrin/petroleum oil mixtures to be more toxic to boll weevils than a permethrin/cottonseed oil mixture, Smith and Luttrell (1987), in field efficacy tests of a pyrethroid mixed in different oils, showed that the poorest efficacy against tobacco budworms in soybeans occurred when the test insecticide was applied with petroleum oil as an adjuvant.

Optimization of the aerial application of fipronil is needed if fipronil is to be used in eradication programs. This research was conducted to evaluate several oil adjuvants for use in ultra low volume (ULV) application and to evaluate several drift retardants for use in low volume (LV) application of fipronil by aircraft.

MATERIALS AND METHODS

Insecticide Transfer Test. A series of tests were conducted to determine the ability of the oils used in these field tests to transfer fipronil from cotton leaves to boll weevils walking across the surface of a treated leaf. Leaves were collected from plants grown in the field. Fipronil (0.056 kg/ha) mixed in the different oils was applied to excised cotton leaves using a spray chamber equipped with an air-assisted spraying system (Mulrooney et al. 1997). Application was made at a 1.17 l/ha volume. The treated leaves were transported to the laboratory where boll weevils, marked with white acrylic paint, were placed on the leaves one weevil at a time. The distance traveled by the weevil over the leaf surface was measured using a VideoMex-V motion analysis system (Columbia Instruments, Columbus, Ohio). There were five leaves per treatment with five weevils per leaf. This test was repeated three times. After a weevil had walked across the leaf, it was transferred to a 35-ml plastic diet cup containing a diet plug. Mortality was recorded 48 h after exposure to the treated leaf.

Cumulative mortality was regressed on distance traveled using a Weibull function to model cumulative mortality (y):

$$F(y) = \max[1 - 1/\exp(\text{distance}/\mu)^{\text{rate}}]$$

The estimates of parameters from the Weibull function describe the following:

max - maximum cumulative mortality (%)

mu - distance (cm) at which half of the maximum mortality occurs.

rate - slope of the curve.

F-tests were used to compare estimates of these parameters for each treatment.

Oil Adjuvant Test. The efficacy of fipronil in different oil adjuvants was determined. The three oil adjuvants tested were once refined cottonseed oil from Yazoo Valley Oil Mill (Greenwood, MS), Orchem 796, and WS2908. Orchem 796 and WS2908 are horticultural mineral oils developed by Exxon Chem. Co., Baytown, TX. WS2908 is a blend of Orchem 796 and EX-100, a drift retardant.

Sprays were applied by aircraft on 7 and 20 July 1998 at Stoneville, MS. Fipronil was

applied at 0.056 kg (AI)/ha in a 1.17 l/ha volume of each of the oils using an Air Tractor 402 aircraft equipped with 18, 8002 flat-fan nozzles (Spraying Systems, Wheaton, IL). Pressure and air speed were 262 kPa and 225 km/h, respectively. Application was made parallel to the rows (east - west) in plots 27 m wide by 246 m long during mid-morning.

Leaves were collected for bioassay from the fourth node down from the terminal at 0, 1, 2, and 3 days after treatment. Thirty leaves per treatment were bioassayed in plastic petri dishes (100 mm diameter) using five boll weevils per leaf. Mortality readings were taken 48 h after placing the weevils on treated leaves.

Drift Retardant Test. Three experimental drift retardants, developed by Helena Chemical Co., were used in these tests. The drift retardants and their use rates were HM 9733-A (178 ml/378.2 l), HM9810 (1.0 % v/v), and HM9850 (454 g/378.5 l). Fipronil was mixed with each drift retardant in an aqueous mixture at a 0.019 kg/ha rate and applied in a 9.6 l/ha total volume. Applications were made with an Air-Tractor 402 equipped with Cp nozzles (Cp Products, Mesa, AZ), on 27 July, 4 August, and 25 August 1998. The aircraft was flown at 217 km/h with pressure set at 207 kPa. Application was made parallel to the rows (east - west) in plots 27 by 246 m during mid-morning.

Leaves were collected at 0, 1, 2, and 3 days after treatment for bioassay. Thirty leaves per treatment were bioassayed in petri dishes using five boll weevils per leaf. Mortality readings were taken at 48 h after placing the weevils on treated leaves.

Droplet size analyses of each fipronil/drift retardant mixture were conducted in our laboratory using a Malvern Spraytec RTS 5000 (Malvern Instruments, Inc., Southborough, MA). Mixtures were sprayed through a single TX-6 nozzle (Spraying Systems, Wheaton, IL) at a pressure of 206.8 kPa and at 18.7 l/ha volume. The nozzle was positioned 35.6 cm above the laser beam. There were six replicates of each mixture.

Each plot in the aerial application tests consisted of two swaths of the aircraft and six measurements were made within each plot. The data were analyzed as a randomized complete block with six treatments replicated in time. Measurements within each plot were subsamples. All data were subjected to an ANOVA using SAS's PROC MIXED (Littell et al. 1996). Least square means were separated using the PDIFF option.

RESULTS

Insecticide Transfer Test. Curves of the predicted percentage cumulative mortality are shown in Fig. 1. Notice that with water, Orchex 796, and WS2908, increases in distances traveled did not result in increases in mortality after maximum mortality was reached. This may indicate that within a short time frame, weevils traveling over insecticide treated cotton leaves reach a saturation point and additional contact with insecticide residues does not result in increased mortality. Salt and Ford (1984) observed that the competition for permethrin between cabbage leaf surfaces and *Spodoptera littoralis* Boisid. larvae crawling over them resulted in a steady initial accumulation of insecticide by the insect, which led to a steady state when the rate of transfer to the insect equaled the rate of detachment from the leaf.

Maximum mortality was greatest ($F=84.64$; $df=3, 159$; $P=0.05$) for cottonseed oil (38%), while mortalities for Orchex 796, WS2908, and water were 20, 16, and 8% respectively (Table 1).

Mu is defined as the distance at which half of the maximum mortality occurs. The value of Mu for the cottonseed oil treatment was 7.04 cm. This means that a level of 19 % mortality, or one half the maximum mortality of 38%, would occur if weevils traveled 7.04 cm over a cotton leaf treated with fipronil mixed in cottonseed oil. The shortest ($F=5.69$; $df=3, 159$; $P=0.05$) Mu occurred when weevils walked over cotton leaves treated with fipronil mixed in water; however, this Mu only resulted in 4% mortality.

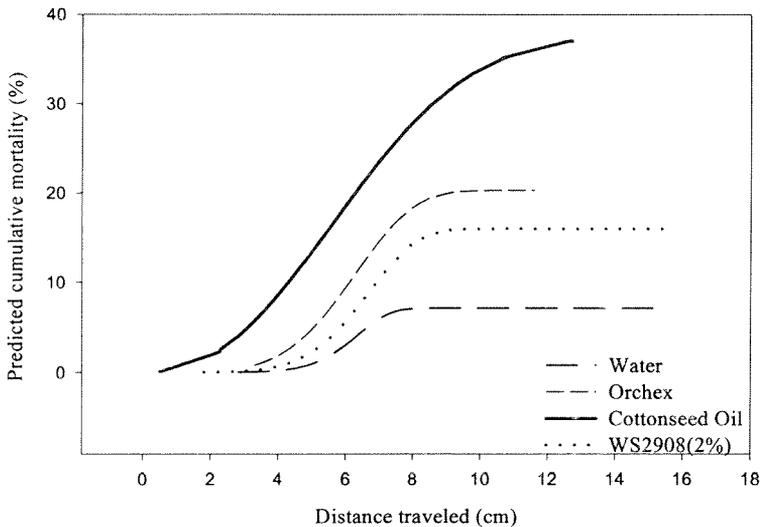


FIG. 1. Cumulative mortality of boll weevils crawling over leaves treated with fipronil in different oil adjuvants.

TABLE 1. Comparison of Parameters of Weibull Function of Cumulative Boll Weevil Mortality over Distance Traveled across Fipronil Treated Cotton Leaves.

Treatment	Max	Mu	Rate
Cottonseed Oil	38 a	7.04 a	2.39 c
Orchex 796	20 b	6.64 a	4.64 c
WS2908 (2%)	16 c	6.94 a	5.74 b
Water	8 d	6.48 b	7.58 a

Rate is the slope of the curve. The lowest ($F=83.22$; $df=3, 159$; $P=0.05$) rates among the treatments were those of cottonseed oil (2.39) and Orchex 796 (4.64), meaning that it took a greater distance for 50% of the maximum cumulative mortality (Mu) to be reached when these oils were used as adjuvants. However, cottonseed oil and Orchex 796 have higher Max's than the other treatments. If the distance where 95% of the maximum mortality occurred for each treatment is calculated and if each of these distances is divided into 95% of the Max mortality for each treatment, then the result is the percentage mortality for each centimeter traveled. When this was done, using cottonseed oil as an adjuvant resulted in 3.24% mortality for every

centimeter traveled. This was the highest among all the treatments. The next highest percentage mortality/centimeter traveled (2.3%) occurred when Orchex 796 was used as an adjuvant. When WS2908 and water were used as adjuvant and carrier, 1.8 and 1.0% mortalities were observed, respectively.

Oil Adjuvant Test. Immediately after treatment, there were no differences in percentage mortalities of boll weevils, which ranged from 98 to 100 (Table 2). At 1 day after treatment, mortality was lowest (92%) ($F=7.67$; $df=2, 123$; $P>F=0.0007$) when Orchex 796 was used as an adjuvant compared to that of cottonseed oil (98%) and WS2908 (100%). Mortality from the cottonseed oil treatment was greater than that of WS2908 for the remainder of the test and greater than that of Orchex 796 at days 2 and 4. At 0 ($F=2.67$; $df=2, 28$; $P>F=0.0239$) and 1 ($F=7.67$; $df=2, 28$; $P>F=0.0007$) day after treatment, a greater amount of fipronil was recovered from leaves treated with the fipronil and CSO mixture than when Orchex 796 and WS2908 were used as adjuvants (Table 3). At 2 and 3 days after treatment, there were no differences in the amount of fipronil recovered from leaves treated with different fipronil + adjuvant mixtures.

TABLE 2. Percentage Mortality (48 h) \pm SEM of Boll Weevils in Leaf Bioassay of Cotton Treated with Fipronil [56 g(AI)/ha] Mixed in Different Oils and Aerially Applied at 1.17 l/ha.

Adjuvant	Days after treatment				
	0	1	2	3	4
Orchex 796	98 \pm 0.7 a	92 \pm 2.2 b	51 \pm 5.9 c	51 \pm 5.3 a	38 \pm 5.7 b
Cottonseed Oil	100 \pm 0.0 a	98 \pm 0.9 a	85 \pm 3.0 a	42 \pm 4.5 a	60 \pm 6.8 a
WS2908@ 3%	99 \pm 0.7 a	100 \pm 0.0 a	70 \pm 4.8 b	20 \pm 4.3 b	18 \pm 4.0 c

TABLE 3. Fipronil Residues (ng/cm²) \pm SEM on Cotton Leaves Treated with Fipronil [56 g(AI)/ha] Mixed in Different Oils and Aerially Applied at 1.17 l/ha.

Adjuvant	Days after treatment			
	0	1	2	3
Orchex 796	47.3 \pm 5.6 b	20.6 \pm 3.9 b	9.3 \pm 4.0 a	1.7 \pm 0.6 a
Cottonseed Oil	104.5 \pm 15.6 a	40.6 \pm 5.32 a	9.4 \pm 3.7 a	9.0 \pm 5.1 a
WS2908@ 3%	60.6 \pm 18.8 b	15.9 \pm 3.7 b	9.6 \pm 3.3 a	8.7 \pm 2.6 a

Drift Retardant Test. Except for HM9810, mortalities from fipronil mixed with drift retardants were higher ($F=3.15$; $df=3, 351$; $P>F=0.0252$) than that of the standard application in water on day 0 (Table 4). If boll weevil mortality is considered an indicator of the amount of fipronil on the surface of leaves, then adding HM9733-A and HM9850 to the fipronil + water mixture resulted in an increased deposition of fipronil on the cotton plant. However at 1 day after treatment, the standard treatment with water resulted in higher ($F=12.87$; $df=3, 236$; $P>F=0.0001$) mortality than all of the drift retardants. This may be indicating that these drift retardants are binding fipronil to the plant surface. At 2 days after application there was no difference in mortalities between that of the standard water treatment and HM9733-A. Both of these treatments caused higher ($F=28.85$; $df=3, 230$; $P>F=0.0001$) mortalities than HM9850. At 3 days after treatment, boll weevils exposed to leaves treated with fipronil mixed with HM9733-A had mortalities that were higher ($F=4.75$; $df=3, 111$; $P>F=0.0037$) than all the other treatments, including the standard. This result seems to indicate that HM9733-A increased

the longevity of fipronil on the leaf surface. Greater amounts of fipronil were found on the leaf surface at 0 ($F=6.60$; $df=3, 62$; $P>F=0.0006$) and 1 ($F=5.37$; $df=3, 63$; $P>F=0.0023$) day after treatment when mixed with HM9850 and HM9733-A (Table 5). The fipronil/HM9733-A mixture had the greatest ($F=4.36$; $df=3, 63$; $P>F=0.0075$) residue among treatments at 2 days after treatment; while, fipronil mixed with HM9850 was found in the greatest ($F=7.18$; $df=3, 14$; $P>F=0.0037$) quantity at 3 days after treatment.

TABLE 4. Percentage Mortality (48 h) \pm SEM of Boll Weevils in Leaf Bioassay of Cotton Treated with Fipronil [19 g(AI)/ha] Mixed in Different Drift Retardants and Aerially Applied at 9.36 l/ha.

Adjuvant	Days after treatment			
	0	1	2	3
Water	91 \pm 1.6 b	94 \pm 1.7 a	71 \pm 3.8 a	30 \pm 5.5 b
HM9810	92 \pm 1.3 ab	65 \pm 4.9 c	54 \pm 4.3 b	29 \pm 4.3 b
HM9733-A	95 \pm 1.2 a	84 \pm 2.8 b	63 \pm 3.8 ab	50 \pm 5.4 a
HM9850	96 \pm 1.0 a	74 \pm 3.8 c	21 \pm 4.3 c	26 \pm 5.5 b

TABLE 5. Fipronil Residues (ng/cm²) \pm SEM on Cotton Leaves Treated with Fipronil (19 g(AI)/ha) Mixed in Different Drift Retardants and Aerially Applied at 9.36 l/ha.

Adjuvant	Days after treatment			
	0	1	2	3
Water	52.9 \pm 8.9 b	23.8 \pm 7.2 b	9.8 \pm 4.6 b	0.9 \pm 0.9 b
HM9810	61.9 \pm 10.6 b	9.19 \pm 2.7 b	4.7 \pm 2.6 b	3.6 \pm 2.5 b
HM9733-A	90.2 \pm 10.2 a	56.5 \pm 11.0 a	34.5 \pm 11.5 a	4.7 \pm 1.3 b
HM9850	111.2 \pm 11.6 a	37.3 \pm 11.0 a	8.5 \pm 3.0 b	15.6 \pm 5.1 a

Droplet size analyses using a Malvern Spraytec RTS 5000 showed that droplet median diameters (DV_{50}) of HM9850 and HM9733-A were 523 and 154 μ m, respectively, while droplet median diameters of HM9810 and water were 146 and 144 μ m, respectively. While droplet size may explain the greater deposition of HM9850 because larger droplets weigh more and are more subject to gravity than smaller droplets, it does not explain the increased deposition of HM9733-A.

Based on maximum mortality, CSO was shown to be the most effective oil adjuvant for transferring fipronil from the surface of cotton leaves to boll weevils in laboratory tests. In field tests, aerial application of fipronil in CSO proved to be the most effective treatment against boll weevils.

In aerial application tests, two of the drift retardants, HM9733-A and HM9850, seemed to enhance the deposition of low volume applications of fipronil. However, only HM9733-A seemed to significantly enhance fipronil's effectiveness against boll weevils.

One interesting result from these tests is the effectiveness of fipronil at 19 g(AI)/ha which is one-third the recommended rate of 56 g(AI)/ha.

Application of fipronil for boll weevil eradication would seem to be more expeditiously done with ultra-low-volume application using oil as a carrier as compared to low-volume application

of aqueous solutions. Oils have several advantages over water as a carrier and vast acreage can be treated more effectively with ultra-low-volumes of insecticide because aircraft are able to spend more time spraying and less time filling and ferrying to and from the airstrip.

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REFERENCES

- Colliot, F, K. A. Kukorowski, D. W. Hawkins, and D. A. Roberts. 1992. Fipronil: A new soil and foliar broad spectrum insecticide. Brighton Crop Prot. Conf. Pests Dis. 1: 29-34.
- Littell, R. C., G. A. Milliken, W. W. Stroup, and R. D. Wolfinger. 1996. SAS System for Mixed Models, 633 pp., SAS Institute Inc., Cary, NC.
- McDowell, L. L., G. H. Willis, L. M. Southwick, and S. Smith, Jr. 1991. Interception and persistence of parathion and fenvalerate on cotton plants as a function of application method. Pestic. Sci. 33: 271--279.
- Mulrooney, J. E., K. D. Howard, J. E. Hanks, and R. G. Jones. 1997. Application of ultra-low-volume malathion by air-assisted ground sprayer for boll weevil (Coleoptera: Curculionidae) control. J. Econ. Entomol. 90: 640 - 645.
- Mulrooney, J.E., D.A. Wolfenbarger, K.D. Howard, and D. Goli. 1998. Efficacy of ultra-low-volume and high volume applications of fipronil against the boll weevil. J. Cotton Sci. 3: 1-7.
- Ochou, G. L., S. Hester, and F. W. Plapp, Jr. 1986. Plant and mineral oils: effects as insecticide additives and direct toxicity to tobacco budworm larvae and housefly adults. Southwest. Entomol. Suppl 11: 63-68.
- Salt, D. W, and M. G. Ford. 1984. The kinetics of insecticide action. Part III: The use of stochastic modelling to investigate the pick-up of insecticides from ULV-treated surfaces by larvae of *Spodoptera littoralis* Boisid. Pestic. Sci. 15: 382-410.
- Scott, W. P., G. L. Snodgrass, and D.A. Adams. 1996. Mortality of tarnished plant bug and boll weevils to Provado and different formulations of fipronil, pp. 987-990. In Proceedings Beltwide Cotton Prod. Res. Conf. National Cotton Council, Memphis, TN.
- Shaw, R., and H. S. Yang. 1996. Performance summary of fipronil insecticide on cotton, pp. 862-865. In Proceedings Beltwide Cotton Prod. Res. Conf. National Cotton Council, Memphis, TN.
- Shaw, R. , H. S. Yang, B. K. Rowe, H. R. Smith, and B. Deeter. 1997. Summary of research results with fipronil for control of plant bugs on cotton. pp. 1043-1046. In Proceedings Beltwide Cotton Prod. Res. Conf. National Cotton Council, Memphis, TN.
- Smith, D. B., and R. G. Luttrell. 1987. Performance specifications for tobacco budworm (Lepidoptera: Noctuidae) larvae treated with vegetable oil and water sprays containing fluralinate. Jour. Econ. Entomol. 80:1314-1318.
- Treacy, M. F., J. H. Benedict and K. M. Schmidt. 1986. Toxicity of insecticide residues to the boll weevil: comparison of ultra-low-volume/oil vs. conventional/water and water-oil sprays. Southwest. Entomol. Suppl. 11: 19-24.
- Wolfenbarger, D. A., and A. A. Guerra. 1986. Toxicity and hypoxia of three petroleum hydrocarbons and cottonseed oil to adult boll weevils and larvae of tobacco budworms. Southwest. Entomol. Suppl 11: 69-74.