

## DEPARTMENT OF FOOD AND AGRICULTURE



EXECUTIVE SUMMARY  
Of Report EH 91-2 Entitled  
"The Influence of Dormant Spray Oil on Diazinon Deposition  
and Transfer to Non-Target Vegetation"

Environmental Monitoring and Pest Management  
Division of Pest Management, Environmental  
Protection and Worker Safety  
Department of Food and Agriculture

**PURPOSE:**

Previous studies have demonstrated that at least some pesticides move from application sites to non-target crops. The purpose of this study was to compare the effects of two different treatment methods on (1) the amount of diazinon deposited onto dormant trees, and (2) the amount of pesticide transferred to non-target vegetation after application.

**BACKGROUND:**

The California Department of Food and Agriculture completed a study in 1989 which indicated that at least four organophosphate insecticides, parathion, diazinon, chlorpyrifos and methidathion, applied to dormant trees in orchards in California's Central Valley moved onto crops to which these pesticides had not been applied. These residues may have been caused by drift during pesticide applications, or by vapor and particulate phase transport from branch and soil surfaces after application.

Insecticidal oil treatments have been used in agriculture to control mites and scale for at least 100 years. Horticultural petroleum oil can be sprayed along with organophosphate pesticides to leafless fruit trees during the dormant spray season (December through mid-February) in the Central Valley. It is unknown how oil affects the amount of pesticide deposited on tree surfaces, the amount of pesticide drifting offsite during application, and the amount of pesticide volatilizing after application and moving offsite. The following experiments were conducted during the 1990 dormant spray season to investigate some of these effects.

**STUDY METHODS:**

**Experiment 1 - Deposition on Orchard Surfaces:** Applications of diazinon were made to almond branches in a simulated orchard experiment under controlled conditions. Two treatments were used: diazinon applied with and without oil.

Mass deposition was measured on targets which consisted of small almond branches mounted on structures designed to hold the branches at tree-canopy height. After each application, almond branch

samples were collected. Surrogate branches (dowels covered with filter paper) were also used to test whether they could be used to measure pesticide deposition in future dormant spray field experiments.

Filter paper cards were used to measure the amount of pesticide which reached the ground. They were placed on the ground surrounding the experimental structures and collected after each application.

**Experiment 2 - Transfer to Non-Target Vegetation:** Two treatments of diazinon were applied by brush to the interior surfaces of wooden lattice frameworks: 1) with dormant spray oil and water, and 2) with water only. The wooden frameworks surrounded flats of parsley. Samples of parsley vegetation were collected before each application, and at regular intervals for up to five weeks after each application.

### **RESULTS:**

**Experiment 1 - Deposition on Orchard Surfaces:** The amount of diazinon deposited onto almond branches was the same, whether oil was present or not. Results from the surrogate branches were different than those from almond branches, indicating that the surrogate branch surface used in this study would not have been an appropriate substitute for almond branches in future studies.

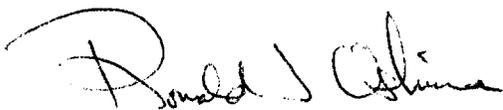
Mass deposition card sample results indicated that the average amount of diazinon deposited under the simulated orchard was similar regardless of the presence of oil.

**Experiment 2 - Transfer to Non-Target Vegetation:** Diazinon concentrations in non-target vegetation increased from day 3 to 21, then declined. Dormant spray oil had no effect on pesticide concentrations found in these samples over time.

### **CONCLUSIONS:**

**Experiment 1 - Deposition on Orchard Surfaces:** Diazinon deposition during application onto branches or onto the ground was not influenced by the addition of horticultural petroleum oil to the spray application tank mixture.

**Experiment 2 - Transfer to Non-Target Vegetation:** Horticultural petroleum oil also had no effect on pesticide transfer from lattice walls to parsley plants after application.



Ronald J. Oshima  
Branch Chief

5/28/91

**THE INFLUENCE OF DORMANT SPRAY OIL ON DIAZINON DEPOSITION  
AND TRANSFER TO NON-TARGET VEGETATION**

**By**

**B. Turner, S. Powell, D. Gonzalez and C. Ando**

**MAY, 1991**

**ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM**

## ABSTRACT

In a 1989 study, the California Department of Food and Agriculture (CDFA) determined that organophosphate pesticides used in orchard dormant spraying were being deposited on non-target row crops grown at distances less than and greater than 0.4 km from the site of pesticide application. The residues found on non-targeted crops may have been the result of drift during pesticide application or transport of pesticide in vapor or particulate phase from branch and soil surfaces after application. A field experiment was designed to determine if applying dormant spray oil in conjunction with diazinon affects mass deposition on branch surfaces. An increase in deposition on branch and soil surfaces would indicate a reduced mass available for off-site drift during an application. Experimental structures to which real and surrogate branches were attached were used to compare deposition rates between oil and non-oil applications. Fallout cards placed beneath the structures measured soil deposition. Statistical results indicated that dormant spray oil had no effect on the deposition of diazinon on branch or soil surfaces. However, the lack of difference between treatments may not be definitive because of differences in tank sample data. Deposition on surrogate branches was significantly different from that on real branches, limiting the use of surrogate branches in future studies.

A second experiment examined the effect of dormant spray oil on pesticide transfer from treated surfaces to non-target vegetation. Experimental latticework structures were coated with an aqueous solution containing diazinon either with or without oil. Parsley plants were placed within the structures and samples were collected at 8 intervals over a 36-day period. Diazinon concentrations in parsley increased from day 3 to 21, then declined. Dormant spray oil had no effect on pesticide concentrations found in these samples over time. Results suggest that oil may not be useful in reducing pesticide transfer to non-target vegetation if those pesticides volatilize at rates or have octanol:water partition coefficients similar to those of diazinon.

## ACKNOWLEDGEMENTS

We wish to express our thanks to Chico State University Farm personnel who allowed us use of their property, and the CDFA Control and Eradication Program for use of the Folsom facility.

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## INTRODUCTION

The California Department of Food and Agriculture (CDFA) completed a study in 1989 which indicated that at least four organophosphate pesticides (OPs) applied to dormant trees in Central Valley orchards were moving onto non-target vegetation (Turner et al. 1989). Parathion, diazinon, chlorpyrifos and methidathion were found in fog water and vegetation samples, and parathion was found on fallout card samples collected in fields near treated orchards. Pesticide transport was regional (greater than 0.4 km) as well as local during both foggy and clear periods. Residues found on non-targeted vegetation may have been caused by drift during pesticide applications or by vapor and particulate phase pesticide transport from branch and soil surfaces after application.

Insecticidal oil treatments have been popular in agriculture for at least 100 years, primarily for control of mites and scale (Johnson, 1980). Many California orchard growers combine the application of horticultural petroleum oil and an organophosphate pesticide to leafless fruit trees during the dormant spray season, December through mid-February. The proportion of this spray mixture impacting tree surfaces is unknown but has been estimated to be from 20 to 50 percent of the total application amount. The effect of oil on the amount of pesticide deposited on tree surfaces, the amount of pesticide drifting offsite during application, and the amount of pesticide volatilizing after application and moving offsite is also unknown. Two experiments were undertaken to relate the presence of dormant oil to 1) pesticide deposition on targeted surfaces, and 2) post-application pesticide transfer to non-target vegetation. Both experiments were performed during the 1990 dormant spray season by the Environmental Hazards Assessment Program of CDFA.

The first experiment performed at the California State University Farm in Chico tested whether dormant spray oil increased pesticide deposition on target commodities and, conversely, reduced pesticide drift during application. Drift is one possible mechanism by which pesticides may enter fog or air.

The second experiment, performed at the CDFA Eradication Facility in Folsom, California, examined whether dormant spray oil affected post-application pesticide transport and deposition on non-targeted surfaces.

## MATERIALS AND METHODS

### Experiment 1: Deposition

Diazinon, with and without oil, was applied to almond branches and surrogate targets which were attached to structures simulating tree scaffold limbs (Figure 1). Five applications (replications) were made with 91 g diazinon active ingredient (Diazinon 50 WP) mixed with 76 l water. An additional five applications consisted of 91 g diazinon mixed with 1.9 l Omni Supreme Spray Oil plus 74.1 l water. Pesticide was applied by a tractor-powered Turbo Mist Model C24P3-144 stainless steel airblast sprayer operated at 1540 kPa. Tractor speed was maintained at  $47 \text{ m min}^{-1}$  to apply diazinon at the rate of  $2.27 \text{ kg in } 1871 \text{ l water ha}^{-1}$ . New tank mixtures were prepared for each application. Pesticide treatments with oil and without oil were alternated and tank samples were collected from a bypass valve before and after each application.

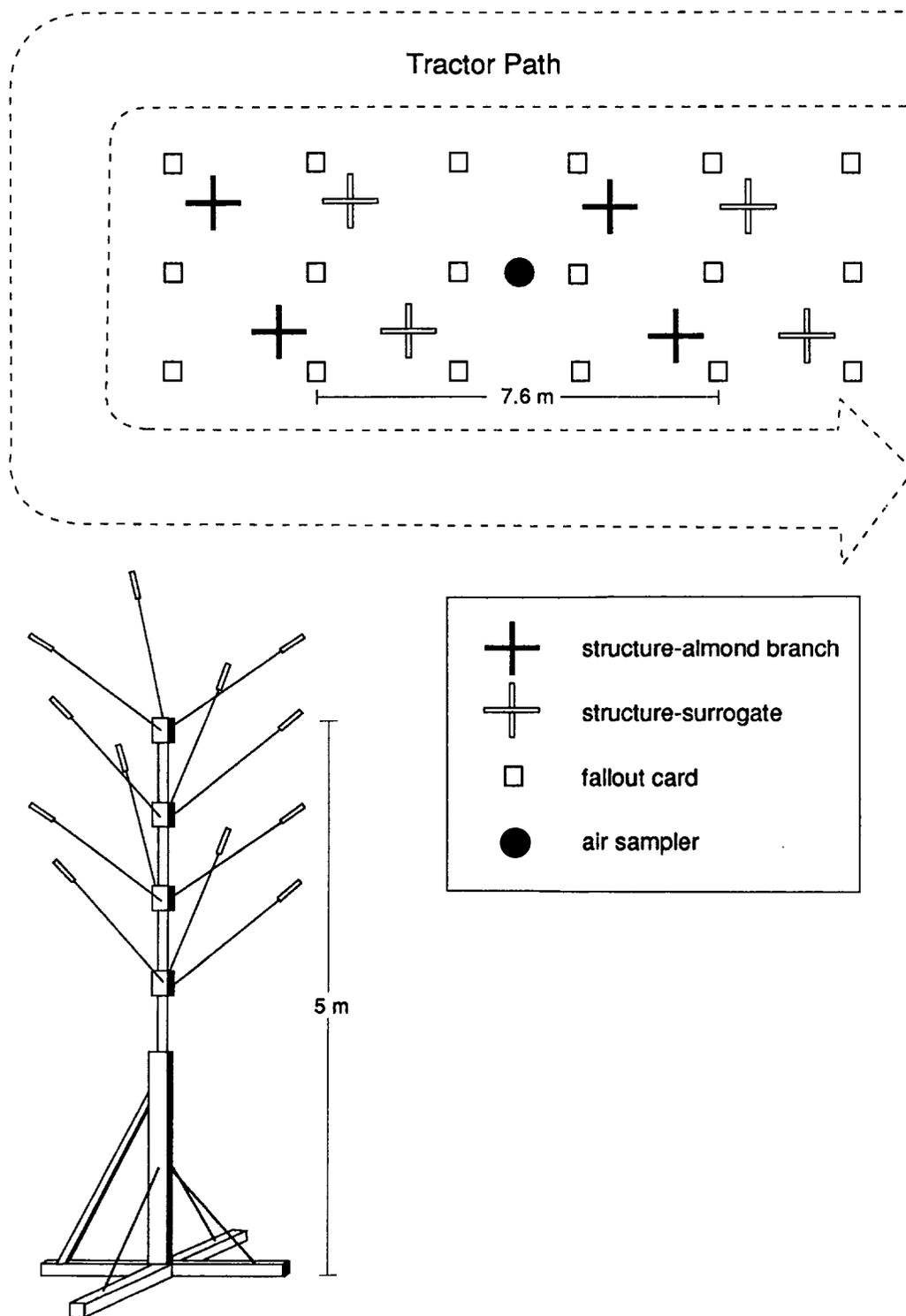


Figure 1. Diagram of experimental set-up for one application and detail of branch-holding structure for the diazinon deposition study.

Eight experimental structures were sprayed during each treatment application (Figure 1). Diameter of each real branch sample was measured with calipers and surface area calculated while surrogate branch area was calculated from premeasured filter paper rectangles. Almond branches were attached to four of the structures while surrogate branches were attached to the remaining four structures. Each branch sample consisted of either 12 lengths of cut dormant almond branches or surrogate branches (filter paper-covered dowels) which were attached at four heights and three angles to the experimental structure. An application consisted of two passes of the tractor, one in front and one behind the structures. Eighty percent of the spray solution was targeted through the upper two-thirds of the scaffold to approximate a typical orchard application. After each application, branch and surrogate samples were collected and sealed in glass jars and frozen until analysis.

Eighteen filter paper (Whatman No. 1) fallout cards were placed on the ground surrounding the experimental structures. After application, two sets of nine cards were composited into two samples, sealed in glass jars and frozen until analysis.

One high-volume air sampler ( $1000 \text{ l min}^{-1}$ ) containing 125 ml XAD-2<sup>®</sup> resin as the sampling medium was placed in the center of the experimental structures. Thirty-minute air samples were collected before and after each application.

Experimental structures were cleaned after each application using a Karcher Hot Water Cleaner with detergent and clean water rinse. The tractor and spray tank were also rinsed with clean water. Each application took place at a different location within the study area to reduce sample contamination (total

experimental area was 200 x 12 m). Temperature, wind speed and wind direction measurements were collected for each application.

Sample analysis was performed by CDFA Chemistry Laboratory Services, using acetone as the extractant for branches, filter paper and resin. Extracts were analysed using a Varian 3700 gas chromatograph equipped with a flame photometric detector (phosphorus mode) and DB-210 column (15 m x 0.537 mm x 1.0  $\mu$ m). Complete analytical methods are presented in Appendix A. Method validation and quality control results are presented in Appendix B.

The effects of oil and surface type were statistically analyzed in a treatment (oil vs. no oil) x surface (branch or surrogate) analysis of variance (ANOVA), with surface as a repeated measure.

#### **Experiment 2: Transfer to Non-Target Vegetation**

Treatments of diazinon, applied to wood both with and without dormant spray oil, were replicated five times to test whether dormant oil influences the transfer of diazinon from treated surfaces to non-target vegetation. Individually prepared solutions of either 2% (w/w) diazinon in water (250 ml) or 2% diazinon plus 2.5% (v/v) dormant oil in water were applied by brush to the interior surfaces of untreated redwood lattice walls (Figure 2). The latticework allowed free movement of air through the walls and an oversized roof raised 20 cm above the top of the structure prevented precipitation from removing diazinon from the lattice walls. The structures were located at least 30 m apart and were anchored to the ground by tent stakes.

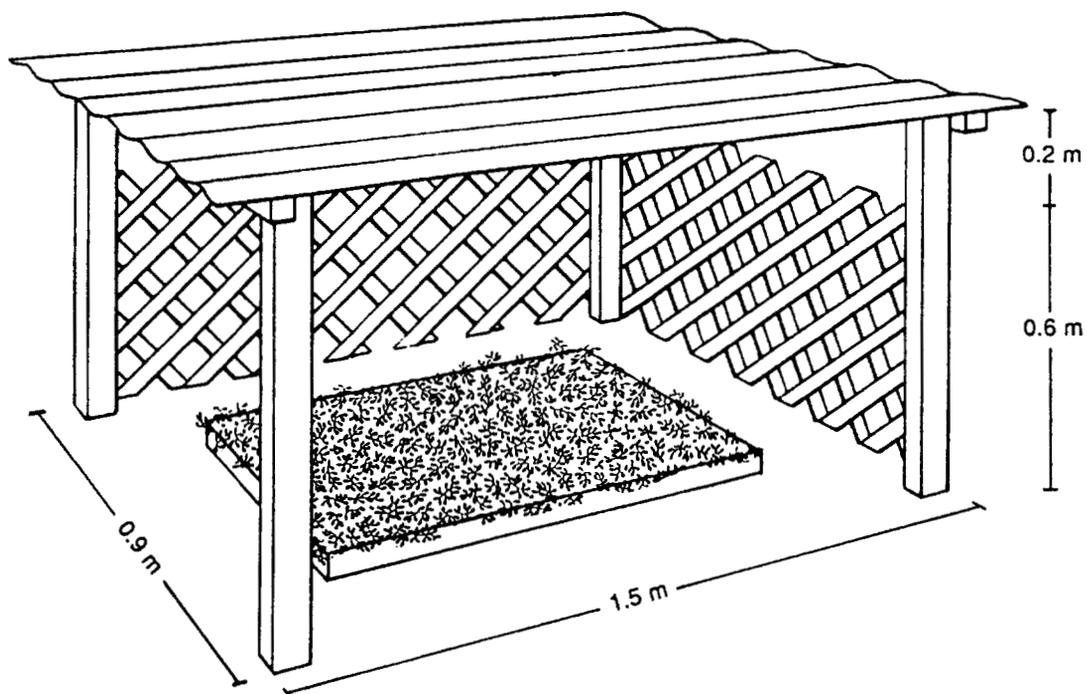


Figure 2. View of lattice structure with front and side removed to show parsley flat, diazinon transfer experiment, Folsom CA, 1990.

Background parsley samples were collected to determine possible pre-existing diazinon contamination. Following pesticide application to the lattice walls, flats of parsley were placed on the ground inside the structures with approximately 20 cm of free space between plants and walls. Parsley samples (50 g) were collected every third day beginning on day 3 after application for two weeks and then three additional samples were collected at one week intervals. Parsley was clipped at soil level, placed in jars, and frozen until analysis. Meteorological data which included ambient temperature minima and maxima, relative humidity, wind direction, and precipitation were collected for each sampling interval.

Residues of diazinon were extracted from parsley samples with acetonitrile. Extracts were filtered and the aqueous layer developed with the addition of sodium chloride. The organic layer was evaporated to dryness, redissolved in acetone to a final volume (< 1.0 ml) and analyzed using a Varian 3700 gas chromatograph equipped with a flame photometric detector (phosphorus mode) and DB-210 column (15 m x 0.537 mm x 1.0  $\mu$ m).

Data were statistically analyzed with a repeated measures ANOVA with days as the repeated measure and dormant oil versus no dormant oil as the treatment factor.

## RESULTS AND DISCUSSION

### Experiment 1: Deposition

**Tank samples:** Mean diazinon concentration was higher overall for non-oil tank samples, but the greater variability in tank samples with oil precluded statistical comparison (Table 1).

TABLE 1. Average diazinon tank mix concentrations during the dormant spray oil deposition study.

Treatment/Statistic	Before Spray	After Spray
	Tank Concentration	Tank Concentration
	-----mg kg <sup>-1</sup> <sup>a</sup> -----	
Diazinon with oil		
Mean (n=5)	408	940
Standard Deviation	246	221
Minimum	210	690
Maximum	750	1150
Diazinon without oil		
Mean (n=5)	1155	1203
Standard Deviation	39	75
Minimum	1120	1120
Maximum	1214	1304

<sup>a</sup>Theoretical tank concentration was 1200 mg kg<sup>-1</sup>.

There was no apparent cause for this difference since product amounts were measured precisely before each tank mix was prepared. Possible explanations, based on the fact that diazinon is more readily soluble in oil than in water, are: 1) Oil added to the pesticide solution in the spray tank reduced the mass of pesticide dissolved in water while increasing the amount dissolved in oil. When the initial tank samples were collected after 5 minutes of agitation, thorough mixing had not yet occurred and samples contained less oil proportionately than those with longer mixing. Therefore, samples which contained less oil also had lower concentrations of diazinon due to the partitioning effect of diazinon in oil over water. This may also explain why the after-spray oil concentration was higher than the before-spray oil concentration: the after-spray oil samples exhibited higher concentrations due to better mixing. 2) The aliquots withdrawn from each sample for chemical analysis had proportionately less oil because the samples were not well mixed. 3) It is also possible that the extraction of diazinon from oil was less efficient than its extraction from water, thereby reducing the concentration found in the oil samples.

**Deposition on real and surrogate branches:** Average diazinon deposition, with and without oil, for both surfaces is graphed in Figure 3. There was a significant main effect of surface type (Table 2), while the treatment (oil vs. no oil) main effect was not significant. However, there was a significant treatment by surface interaction, indicating that the effect of treatment could be different for the two surfaces. This possibility was tested using

Bonferroni multiple comparisons ( $\alpha = 0.05$ ; Milliken and Johnson, 1984) which showed that the oil effect was not significant for either surface, while deposition was significantly greater on surrogates both with and without oil.

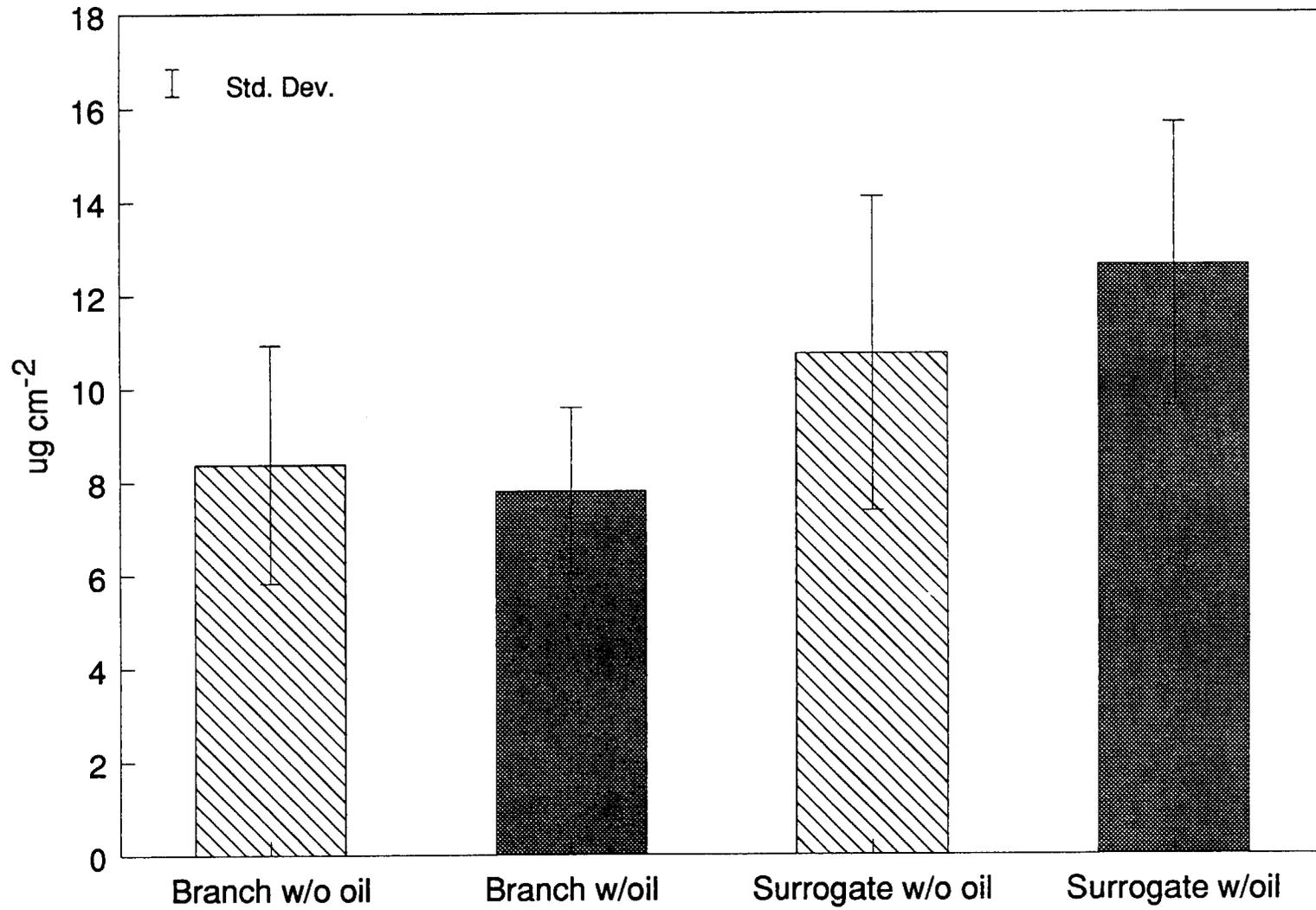


Figure 3. Average deposition of diazinon applied with and without oil to almond branches and surrogates.

Thus, the multiple comparisons failed to identify the source of the interaction. The existence of a significant crossing interaction (that is, the mean treatment differences for branches and surrogates have opposite signs) means that the branches and surrogates are not affected in the same way by the oil and non-oil treatments, so the surrogates should not be used in future experiments. The complete data set is presented in Appendix C.

**Fallout card samples:** Fallout samples were analyzed separately from branch and surrogate surfaces because variability in pesticide deposition on cards was considerably less than that seen on branches and surrogate surfaces (Table 3). A one-way repeated measures ANOVA showed no significant difference between treatments ( $p = 0.54$ ).

Fallout card concentrations substantiated branch deposition results. The lack of difference between oil and non-oil treatments suggests that oil did not affect pesticide deposition or transport offsite.

**Air concentrations:** Air concentrations of diazinon before applications tended to increase over the course of the experiment (Table 4).

The increase in concentration between pre- and post-application was much greater for the non-oil (mean = 0.904) than the oil applications (mean = 0.198). Variability in air concentrations associated with non-oil applications was also greater. These data are difficult to interpret, however, because of varying wind speed and direction, and because nearby applications of diazinon were known to occur during the study. Diazinon was applied to orchards northwest of the experimental site on January 17 through

TABLE 2. Analysis of variance table for the effects of oil treatment and surface type on diazinon deposition.

Source	Degrees of Freedom	Type III Mean Square	F
Treatment	1	0.00000214	0.37
Rep (Treatment)	8	0.00000575	
Surface	1	0.00006631	195.96**
Treatment x Surface	1	0.00000762	22.51**
Surface x Rep (Treatment)	8	0.00000034	

\*\*Significant at the 0.01 level.

TABLE 3. Mean diazinon concentrations on fallout cards during the dormant spray oil deposition study.

Statistic	Treatment	
	With Oil	Without Oil
	-----ug cm <sup>-2</sup> -----	
Mean (n=5)	2.1654	2.4784
Standard Deviation	0.6770	0.8778
Minimum	1.3813	1.3761
Maximum	2.9427	3.4008

TABLE 4. Air concentrations of diazinon before and after applications during the dormant spray oil deposition study.

Date	Rep	Treatment	Time of Spray	Wind Direction and Speed	Air Concentration	
					Before Spray	After Spray
					-----ug m <sup>-3</sup> -----	
1/11	1	No Oil	11:28 am	SSW <2 mph	ND <sup>a</sup>	2.23
1/17		Oil	3:25 pm	NNW 4-8 mph	ND	0.15
1/18	2	No Oil	10:40 am	NNW 4-5 mph	ND	0.15
1/18		Oil	3:30 pm	NNW 5 mph	0.04	0.14
1/19	3	No Oil	9:58 am	NW 4 mph	0.06	0.12
1/23		Oil	9:30 am	NW <2 mph	0.29	0.84
1/23	4	No Oil	2:30 pm	WSW <2 mph	0.35	1.67
1/24		Oil	9:12 am	WNW 4-7 mph	0.08	0.27
1/24	5	No Oil	2:34 pm	ESE 2-6 mph	0.23	0.99
1/25		Oil	9:00 am	SE 1-2 mph	0.40	0.40

<sup>a</sup>Not detected. Minimum detection limit was 0.01 ug m<sup>-3</sup>.

the 20th, and spraying of unknown pesticides continued nearby from January 23 to the end of the study.

The relationship of diazinon air concentrations to our experimental applications remains unclear but the data above show that diazinon air concentrations may vary greatly near local application sites during and up to several days after spraying.

### **Experiment 2: Pesticide Transfer to Non-Target Vegetation**

Parsley sampled prior to treatment was free of pesticide residues but movement of diazinon from lattice walls to parsley occurred rapidly after treatment. When samples were collected on day 3, residue levels were already in the milligrams per kilogram range, averaging 0.97 and 1.14 mg kg<sup>-1</sup> for no oil and oil treatments, respectively. The concentrated formulation (20 g l<sup>-1</sup>) provided an artificially large source of diazinon and reduced the time required for off-target movement. Unlike dormant spray airblast applications in orchards, the experimental application by brush caused no drift and residues on parsley could only have resulted from either vapor or particle-phase transfer of pesticide from the lattice surfaces.

An ANOVA with oil vs. no oil as the treatment factor and days as a repeated measure (partitioned into the linear, quadratic and cubic components) found no significant effect of treatment ( $p = 0.99$ ). Neither were there any significant interactions of treatment with days ( $p = 0.51, 0.96$  and  $0.34$  for the interactions with the linear, quadratic and cubic components, respectively). Only the linear ( $p = 0.003$ ) and quadratic ( $p = 0.005$ ) effects of day were significant. The curve in Figure 4 represents the concentration

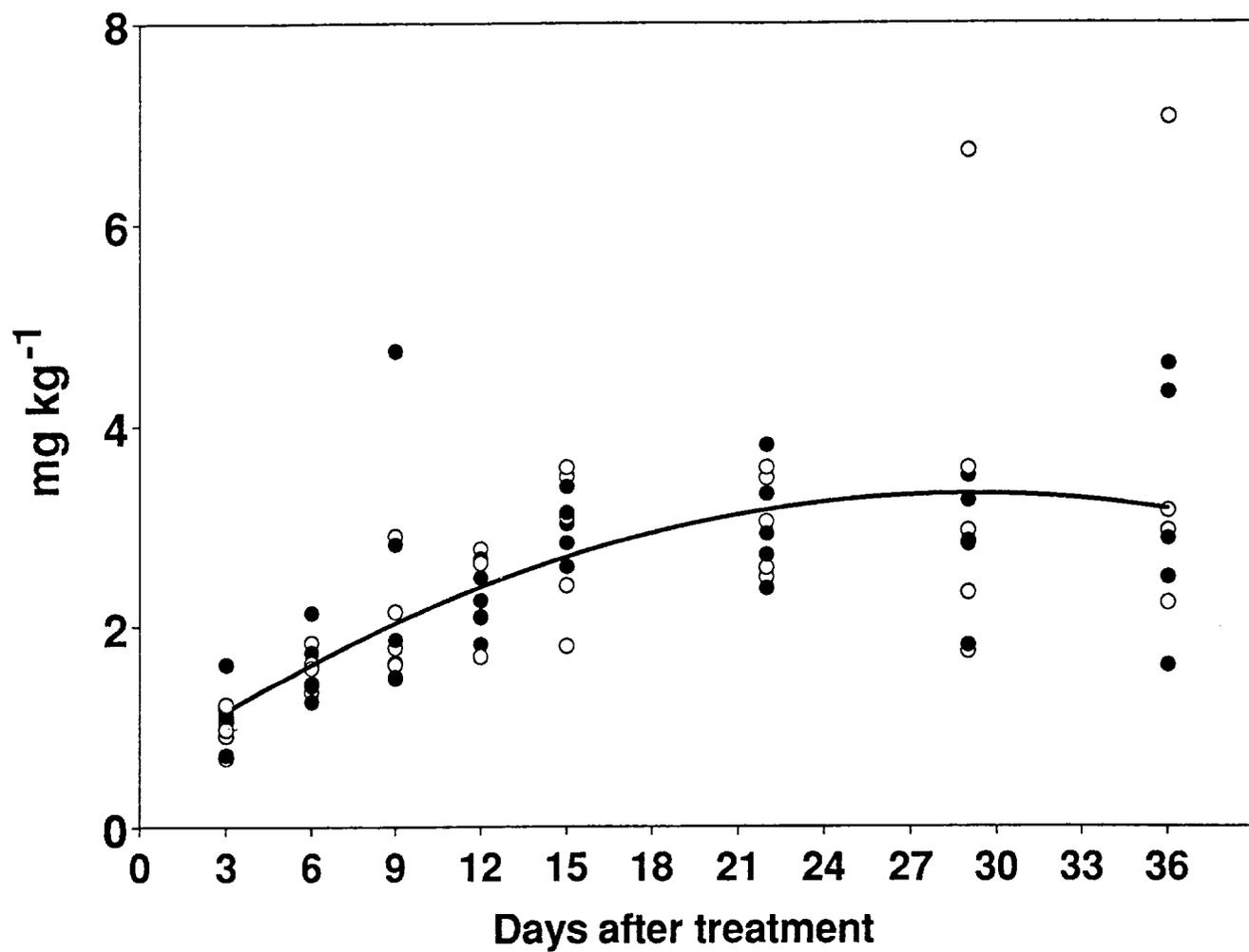


Figure 4. Predicted concentration (—) and measured residues of diazinon with oil (o) and without oil (●) found on parsley over a 36-day period.

predicted by the fitted model. The predicted concentration increases until day 29 and then begins to decline. Oil had no influence on the transfer of diazinon to parsley.

Weather appeared to exert very little influence on pesticide movement. Temperatures ranged from 0 to 30°C during the study, and 112 mm precipitation fell at the site. Rainfall occurred during 6 of the 8 sampling intervals but the data did not suggest any relationship to changes in residue accumulation on parsley.

## CONCLUSIONS AND RESEARCH NEEDS

### Experiment 1: Deposition

An aqueous solution of diazinon with dormant spray oil was compared to one without oil. It was hypothesized that dormant spray oil might increase deposition on targeted surfaces while at the same time reduce drift during applications. Statistical results indicated that diazinon deposition on almond branches and on the ground around "trees" was not influenced by the addition of oil to the tank mixture. However, the lack of an oil effect may not be definitive because of analytical differences in tank concentrations of diazinon. Further research is necessary to determine whether differences were real or spurious. No correlation was found between tank mix concentrations and diazinon deposition on branches or surrogates. This tends to support the theory that diazinon concentrations in the tank samples were actually similar even though measured concentrations were lower in before-treatment samples with oil. Although other dormant spray oil brands may affect pesticide deposition on targeted surfaces differently, our experiment provides

preliminary evidence that oil may not effectively reduce drift problems during dormant spray pesticide applications.

Air concentrations of diazinon measured immediately after application showed no apparent pattern related to the presence of oil during application. Air samples collected before applications indicated that either prior test sprays or ongoing local spraying operations resulted in measurable ambient concentrations of diazinon in 7 out of 10 samples. Because of the difficulty in discriminating between diazinon air concentrations resulting from these tests and applications of other orchard growers, the air samples collected were of no value in discerning differences between oil and non-oil applications of diazinon. Therefore, subsequent research should consider the use of a tracer to determine the true source of pesticide .

#### **Experiment 2: Transfer to Non-Target Vegetation**

In the second experiment, it was hypothesized that oil present in a pesticide solution might reduce volatilization from the target surface, thereby reducing the potential for off-target transfer of pesticide after application and its subsequent deposition on non-targeted commodities. Oil had no effect on pesticide transfer from lattice walls to parsley plants. The use of dormant oil sprays to reduce off-target movement of OPs after an application will probably fail if those OPs volatilize at rates or have octanol:water partition coefficients similar to those of diazinon.

Further study is needed to determine the mass of pesticide moving off-target both during and after applications. Gradient analysis using a tracer could help to define the limit of pesticide dispersal.

Another area of of potential interest is whether organophosphate pesticides are sources of inadvertent residues when: 1) they are applied during the summer growing season which includes drier and warmer climatic conditions; 2) they are applied to crop surfaces which may have different physico-chemical characteristics; and 3) different agricultural practices and application methods are used. Future studies will likely include research in one or more of these areas.

## REFERENCES

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Johnson, W. T. 1980. Spray oils as insecticides. Journal of Arboriculture 6(7):169-174.

Milliken, G. A. and D. E. Johnson. 1984. Analysis of Messy Data, Vol. I: Designed Experiments. Van Nostrand Reinhold Company, New York.

APPENDIX A

Analytical Methods

CALIFORNIA DEPT. OF FOOD & AGRIC.  
ENVIRONMENTAL MONITORING SECTION  
CHEMISTRY LABORATORY SERVICES  
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Sacramento, CA 95832  
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Original Date: 08/02/90  
Supercedes: NEW  
Current Date: 08/29/90  
Method #:

### **DIAZINON ON ALMOND BRANCH TWIGS**

#### **SCOPE:**

This method is for the determination of Diazinon on almond branch twigs.

#### **PRINCIPLE:**

Diazinon is extracted from almond branch twigs with acetone. The extract is then analyzed using a gas chromatograph equipped with a flame photometric detector (FPD).

#### **REAGENTS AND EQUIPMENT:**

Acetone (pesticide residue grade)  
Ultrasonic bath (Branson B72)  
Balance Mettler PC 4400)  
Wide-mouth mason jars (quart size)

#### **ANALYSIS:**

- 1) Remove sample from freezer.
- 2) Remove 12 twigs from sample and place in a tared wide-mouth mason jar. Record the weight of the twigs.
- 3) Add enough acetone to cover the twigs. Record the volume of acetone used (~ 800 mL is sufficient).
- 4) Let twigs soak for 24 hours at ambient room temperature.
- 5) Sonicate sample for 30 minutes.
- 6) Submit sample for gas chromatographic analysis.

#### **Spiking Procedure**

- 1) Spiking solution - made from 50% Diazinon wettable powder. Weigh 0.5 g of formulated product and dissolve into 500 mL distilled water.

Spiking Procedure continued

- 2) Remove 0.5 mL of the Diazinon spiking solution while it is being constantly stirred and spike it on the twigs in the sample jar by drops so that if any run-off occurs it will be caught by the jar. Allow 15 minutes drying time before spiking the other 0.5 mL (2 x 0.5 mL = 1 mL) on the twigs. The twigs are allowed to dry for 30 minutes before extraction.

RECOVERIES:

\* Recoveries of Diazinon

Levels	Diazinon(mean)
0.5 mg (n=4)	94
2.0 mg (n=4)	90
10.0 mg (n=4)	94

EQUIPMENT CONDITIONS:

Varian 3700 GC with FPD (Phosphorus mode)  
Column: DB-210 (50% tri-fluoropropyl methyl polysiloxane) 15 m x 0.537 mm  
x 1.0 um  
Carrier gas: Helium, flow rate: 17 mL/min  
Injector: 220°C  
Detector: 260°C  
Temperature: 120°C isothermal  
Injection volume: 2uL  
Retention time: Diazinon 1.29 ± 0.10 min. Diazinon OA 2.70 ± 0.10 min.  
Linearity checked 0.2 ng - 20 ng

CALCULATIONS:

Micrograms Diazinon and Diazinon Oxygen Analog

$$\text{ug in sample} = \frac{(\text{peak ht sample})(\text{ng/ul std})(\text{ul std injected}) (\text{sample volume ml})}{(\text{peak height standard}) (\text{ul sample injected})}$$

MINIMUM DETECTABLE LEVEL:

The minimum detectable level was 0.20 mg for both Diazinon and Diazinon OA (12 twigs/sample jar) with a S/N = 4.

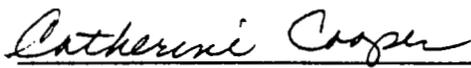
**DISCUSSION:**

All samples were checked for Diazinon OA, but no recovery study was done.

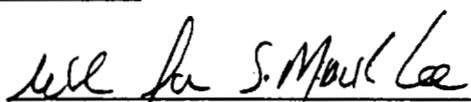
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Original Date: 08/06/90  
Supercedes: NEW  
Current Date: 08/27/90  
Method #:

**DIAZINON ON SURROGATE TWIGS AND FALLOUT CARDS  
MADE OF FILTER PAPER WITH FOIL BACKING**

**SCOPE:**

This is a method for determining Diazinon on foil backed surrogate twigs and fallout cards made of filter paper.

**PRINCIPLE:**

Diazinon is extracted from the filter paper and the protective foil with acetone. The extract is then analyzed using a gas chromatograph equipped with a flame photometric detector (FPD).

**REAGENTS AND EQUIPMENT:**

Acetone (pesticide residue grade)  
Ultrasonic bath (Branson B72)  
Balance (Mettler PC 4400)  
Wide-mouth mason jars (quart size)  
Filter paper (Whatman #1)  
Aluminum foil

**ANALYSIS:**

- 1) Remove sample from freezer.
- 2) Add 875 mL acetone to sample jar to cover filter paper and foil.
- 3) Cap jar and sonicate for 30 minutes.
- 4) Sample is then ready for gas chromatographic analysis.

**Spiking Procedure**

- 1) Spiking solution - made from 50% Diazinon wettable powder. Weigh 0.5 g of formulated product and dissolve into 500 mL distilled water.
- 2) Spike 1 mL of the above 0.5 mg/mL Diazinon solution on each set of filter papers (10 - 7 x 15 mm whatman #1/set) while on a double layer of foil. Make the upper sheet of foil just larger than the filter paper strips. Remove spiking solution while it is being constantly stirred and place by drops on the filter paper strips.

Spiking Procedure continued

Allow 15 minutes drying time before placing in mason jar with top layer of foil for extraction.

RECOVERIES:

\* Recoveries of Diazinon

Levels	Diazinon(mean)
0.5 mg (n=4)	98
2.0 mg (n=4)	92
10.0 mg (n=4)	106

EQUIPMENT CONDITIONS:

Varian 3700 GC with FPD (Phosphorus mode)  
 Column: DB-210 (50% tri-flouoropropyl methyl polysiloxane) 15 m x 0.537 mm  
 x 1.0 um  
 Carrier gas: Helium, flow rate: 17 mL/min  
 Injector: 220°C  
 Detector: 260°C  
 Temperature: 120°C isothermal  
 Injection volume: 2uL  
 Retention time: Diazinon 1.29 ± 0.10min. Diazinon OA 2.70 ± 0.10 min  
 Linearity checked 0.2 ng - 20 ng

CALCULATIONS:

Micrograms Diazinon and Diazinon Oxygen Analog

$$\text{ug in sample} = \frac{(\text{peak ht sample})(\text{ng/ul std})(\text{ul std injected}) (\text{sample volume ml})}{(\text{peak height standard}) (\text{ul sample injected})}$$

MINIMUM DETECTABLE LEVEL:

The minimum detectable level was 0.20 mg for both Diazinon and Diazinon OA on filter paper, with a S/N=4.

DISCUSSION:

Surrogate twigs received by the lab consist of a filter paper strip and foil.

DISCUSSION: continued

Fallout cards consist of filter paper on top of a layer of foil placed on the ground around the surrogate trees to simulate the orchard floor for mass deposition assessment.

All samples were checked for the breakdown product of Diazinon - Diazinon OA, but no recovery study was done for Diazinon OA.

WRITTEN BY: Jane White

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APPROVED BY: Terry Jackson

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Original Date: 01/21/90  
Supercedes: New  
Current Date: 08/28/90  
Method #:

### **DIAZINON IN HIGH VOLUME AIR SAMPLER RESIN**

#### **SCOPE:**

This method is for the determination of Diazinon and Diazinon oxygen analog in high volume air samplers containing XAD-2<sup>®</sup> resin.

#### **PRINCIPLE:**

Diazinon and Diazinon OA were extracted from XAD-2<sup>®</sup> resin with acetone. The solvent was rotary evaporated to dryness and the residues were brought back up to a final volume with acetone. The extract was analyzed using gas chromatography with a flame photometric detector (FPD).

#### **REAGENTS AND EQUIPMENT:**

Acetone; (pesticide residue grade)  
Ultrasonic bath (Branson B72)  
Chromatographic columns (19 mm by 500 mm Kimble)  
Boiling flasks, flat bottom with ground glass joint 24/40 (500 mL)  
Wide-mouth mason jars (pint size)  
Rotary evaporator (Büchi/Brinkmann, R110)  
Graduated test tubes (15 mL)  
Nitrogen evaporator (Organomation Model # 12)  
Vortex mixer for test tubes  
XAD-2<sup>®</sup> (Rohm and Haas); hexane-acetone soxhlet washed

#### **ANALYSIS:**

- 1) Remove sample from freezer and empty resin from the high volume air sampler into a wide-mouth mason jar.
- 2) Add 200 mL of acetone to the mason jar. Cover the jar with foil and cap. Place it into an ultrasonic bath for 30 minutes.
- 3) Remove a 50 mL aliquot and rotary evaporate the extract to 3-5 mL at 35°C and approximately 20 mm Hg vacuum.
- 4) Transfer the extract to a graduated test tube. Wash the flask 3 times each with 2 mL of acetone. Transfer each wash to the same graduated test tube.
- 5) Place extract on a nitrogen blow down evaporator with waterbath set at 35°C and evaporate just to dryness under a gentle stream of nitrogen.

- 6) Pipet 0.5 mL of acetone into the test tube. Stopper the graduated test tube and mix the contents by placing on a vortex mixer for about 15 seconds. Submit sample for gas chromatographic analysis.

RECOVERIES:

% Recoveries of Diazinon

Levels	Diazinon(mean)
1.0 ug (n=3)	82
5.0 ug (n=3)	84
10.0 ug (n=3)	83

EQUIPMENT CONDITIONS:

Varian 3700 GC with FPD (Phosphorus mode)  
 Column: DB-210 (50% tri-fluoropropyl methyl polysiloxane) 15 m x 0.537 mm  
 x 1.0 um  
 Carrier gas: Helium, flow rate: 17 mL/min  
 Injection: 220°C  
 Detector: 260°C  
 Temperature: 120°C isothermal  
 Injection volume: 2 uL  
 Retention times: Diazinon 1.29 ± 0.10 min. Diazinon OA 2.70 ± 0.10 min.  
 Linearity checked: 0.2 ng - 20 ng

CALCULATIONS:

Micrograms Diazinon and Diazinon Oxygen Analog

$$\text{ug in sample} = \frac{(\text{peak height sample})(\text{ng/uL std})(\text{uL injected std})(200 \text{ mL})(\text{final volume mL})}{(\text{peak height std})(\text{uL sample injected})(\text{aliquot volume})}$$

MINIMUM DETECTABLE LEVEL:

The minimum detectable level in XAD-2<sup>®</sup> resin is 0.43 ug for Diazinon and 0.60 ug for Diazinon OA (125 mL resin in high volume air sampler) with a S/N=4.

DISCUSSION:

All samples were checked for the breakdown product of Diazinon - Diazinon OA, but no recovery study was done for Diazinon OA.

REFERENCE:

- 1.) Echelberry, Jim., *Organophosphate Pesticides In High Volume Air Samples*, 1989 Environmental Monitoring Methods, California Department of Food and Agriculture.
- 2.) Schlocker, Peter L., *Wilder Ranch - Miscellaneous Organophosphate Pesticides in Low Volume Air Sampler Resin Samples*, 1983 Environmental Monitoring Methods, California Department of Food and Agriculture.

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APPENDIX B

Analytical Method Validation and  
Quality Control Results

Table 1. Method validation results (% recoveries) for the almond branch deposition study.

Study: 91				Sample Type: Twigs		
Chemical: Diazinon				Lab: CDFA		
MDL: 0.2 mg/sample				Chemist: Jane White		
Date of Report: 1/10/90						
Lab Sample #	Amount Found (mg)	Amount Added (mg)	Recovery %	$\bar{X}$	SD	CV (%)
1691	0.47	0.5	94			
1693	0.53	0.5	106			
1731	0.42	0.5	84			
1735	0.45	0.5	90	94	9.3	9.9
1691	1.98	2.0	99			
1693	1.86	2.0	93			
1731	1.49	2.0	75			
1735	1.82	2.0	91	90	10	11
1691	8.51	10.0	85			
1693	10.11	10.0	101			
1731	9.96	10.0	100			
1735	8.8	10.0	88	94	8.2	8.7
OVERALL				92	8.6	9.4
$\bar{X}$	SD	LWL	UWL	LCL	UCL	
92	8.6	83	101	75	109	

Table 2. Method validation results (% recoveries) for the almond branch deposition study.

Study: 91				Sample Type: Filter paper with foil		
Chemical: Diazinon				Lab: CDFA		
MDL: 0.2 mg/sample				Chemist: Jane White		
Date of Report: 1/10/90						
Lab Sample #	Amount Found (mg)	Amount Added (mg)	Recovery %	$\bar{X}$	SD	CV (%)
1690	0.51	0.5	102			
1694	0.54	0.5	108			
1733	0.44	0.5	88			
1737	0.46	0.5	92	98	9.2	9.4
1690	2.36	2.0	118			
1694	1.55	2.0	78			
1733	1.58	2.0	79			
1737	1.87	2.0	94	92	19	21
1690	10.24	10.0	102			
1694	11.11	10.0	111			
1733	10.79	10.0	108			
1737	10.42	10.0	104	106	4.03	3.80
OVERALL				99	13	13
$\bar{X}$	SD	LWL	UWL	LCL	UCL	
99	13	86	112	73	125	

LWL/UWL = mean +/- 1SD

LCL/UCL = mean +/- 2SD

Table 3. Storage dissipation analyses for the almond branch deposition study.

Study: 91	Sample Type: Twig
Analyte: Diazinon	Lab: CDFA
MDL: 0.2 mg/sample	Chemist: Jane White
Date of Report: 1/5/90	Date Prepared: 12/29/89

Lab Sample #	Hour	Date Extracted	Date Analyzed	Results * (mg)	Spike Level (mg)	Recovery %	$\bar{X}$	SD	CV (%)
1673	96	1/3/90	1/3/90	0.41	0.5	82			
1674	96	1/3/90	1/3/90	0.46	0.5	92			
1675	96	1/3/90	1/3/90	0.45	0.5	90			
1676	96	1/3/90	1/3/90	0.47	0.5	94			
1677	96	1/3/90	1/3/90	0.46	0.5	92	90	4.7	5.2
1679	48	1/5/89	1/5/89	0.45	0.5	90			
1680	48	1/5/89	1/5/89	0.48	0.5	96			
1681	48	1/5/89	1/5/89	0.43	0.5	86			
1682	48	1/5/89	1/5/89	0.43	0.5	86			
1683	48	1/5/89	1/5/89	0.45	0.5	90	90	4.1	4.6

OVERALL: 90      4.2      4.7

\* Samples were spiked with 0.5 mg of Diazinon, held in freezer for specified time, soaked in acetone for 24 hours, sonicated for 30 minutes then shot into GC.

Table 4. Continuing quality control data for the almond branch deposition study.

Study: 91  
 Analyte: Diazinon  
 MDL: 0.2 mg  
 Date of Report: 1/19/90

Sample Type: Twig  
 Lab: CDFA  
 Chemist: Jane White

Extraction Set #	Lab Sample #	Results (mg)	Spike Level (mg)	Recovery %	$\bar{X}$	SD	CV (%)
3, 4, 5, 6	1751	1.51	2.0	76			
13-16, 23-26, 33-36, 43-46	1814	1.37*	2.0	69			
53-56, 63-66, 73, 76, 83-86, 93-96	1820	1.37*	2.0	69			
OVERALL:					71	4.0	5.7

\* Result fell below the LCL set for diazinon at 75%.

Table 5. Continuing quality control data for the almond branch deposition study.

Study: 91  
 Analyte: Diazinon  
 MDL: 0.2 mg  
 Date of Report: 1/19/90

Sample Type: Filter Paper with foil  
 Lab: CDFA  
 Chemist: Jane White

Extraction Set #	Lab Sample #	Results (mg)	Spike Level (mg)	Recovery %	$\bar{X}$	SD	CV (%)
7, 8, 9, 10	1753	1.78	2.0	89			
1, 2	1756	1.34*	2.0	67			
17-20, 27-30, 37-40, 47-50	1811	2.18	2.0	109			
57-60, 67-70, 77-80, 87-90, 97-100	1845	1.55	2.0	78			
11-12, 21-22, 31-32, 41-42	1816	1.75	2.0	88			
51-2, 61-2, 71-2, 81-2, 91-2	1942	1.80	2.0	90			
OVERALL:					87	14	16

\* Result fell below the LCL set for diazinon at 73%.

Table 6. Continuing quality control data for the almond branch deposition study.

Study: 91  
 Analyte: Diazinon  
 MDL: 0.5 ug/sample  
 Date of Report: 1/19/90

Sample Type: XAD-2 Resin  
 Lab: CDFA  
 Chemist: Jane White

Extraction Set #	Lab Sample #	Results (mg)	Spike Level (mg)	Recovery %	$\bar{X}$	SD	CV (%)
226, 227	1755	1.23	2.0	62			
228-35	1817	1.45	2.0	73			
236-45	1818	1.74	2.0	87			
236-45	1866	1.55	2.0	78			
OVERALL:					75	10	14

Table 1. Continuing quality control data for the 1990 Off-Target Vegetation Deposition Study.

Analyte: Diazinon					Lab: CDFA		
Sample Type: Parsley					Chemist: Jane White		
Detection Limit: 0.01 ppm					Date: 3/13/90		
Extraction Set #	Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
24-5, 27-30, 62, 64, 86, 89-91	2569	0.090	0.10	90			
5-7, 12, 17-23, 26,32, 36-7, 40-1, 43-4, 47, 48, 51-6, 63, 65, 71-3, 76, 82-4, 87	2593	0.097	0.10	97			
11, 14-6, 57-60, 66, 69, 74, 81	2591	0.098	0.10	98			
51-6, 67-8, 70, 78-80, 85	2577	0.094	0.10	94			
1-4, 8-10, 31, 33-5, 38-9, 42, 45-6, 48, 77	2575	0.072	0.10	72			
	2595	0.105	0.10	105			
OVERALL:					93	11	12

Table 2. Continuing quality control data for the 1990 Off-Target Vegetation Deposition Study.

Analyte: Diazoxon					Lab: CDFA		
Sample Type: Parsley					Chemist: Jane White		
Detection Limit: 0.02 ppm					Date: 3/13/90		
Extraction Set #	Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
24-5, 27-30, 62, 64, 86, 89-91	2569	0.088	0.10	88			
5-7, 12, 17-23, 26,32, 36-7, 40-1, 43-4, 47, 48, 51-6, 63, 65, 71-3, 76, 82-4, 87	2593	0.106	0.10	106			
11, 14-6, 57-60, 66, 69, 74, 81	2591	0.103	0.10	103			
51-6, 67-8, 70, 78-80, 85	2577	0.084	0.10	84			
1-4, 8-10, 31, 33-5, 38-9, 42, 45-6, 48, 77	2575	0.092	0.10	92			
	2595	0.133	0.10	113			
OVERALL:					98	11	12

Table IV-17. Method validation blank matrix spikes for the 1989 dormant spray study: dill.

Analyte: Diazinon  
 Matrix: Dill  
 Detection limit: 0.015 ppm

Lab: Cal Labs  
 Chemist: Kris Murbach  
 Date: 2/8/89

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
45203-11	0.015	0.03	50			
45203-12	0.015	0.03	50			
45203-13	0.018	0.03	60			
45203-14	0.018	0.03	60	55	5.8	10
45203-17	3.9	5.0	78			
45203-18	3.5	5.0	70			
45203-19	2.8	5.0	56			
45203-20	3.4	5.0	68	68	9.1	13
OVERALL=				62	9.9	16
$\bar{X}$	SD	LWL	UWL	LCL	UCL	
62	9.9	52	72	42	82	

Table IV-18. Method validation blank matrix spikes for the 1989 dormant spray study: dill.

Analyte: Parathion  
 Matrix: Dill  
 Detection limit: 0.015 ppm

Lab: Cal Labs  
 Chemist: Kris Murbach  
 Date: 2/8/89

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
45203-11	0.03	0.03	100			
45203-12	0.03	0.03	100			
45203-13	0.038	0.03	127			
45203-14	0.038	0.03	127	114	15.6	13.7
45203-17	4.2	5.0	84			
45203-18	3.7	5.0	74			
45203-19	3.0	5.0	60			
45203-20	3.5	5.0	70	72	9.93	13.8
OVERALL=				93	25	27
$\bar{X}$	SD	LWL	UWL	LCL	UCL	
93	25	68	118	43	143	

LWL and UWL = mean +/- SD

LCL/UCL = mean +/- 2SD

APPENDIX C

Branch and Surrogate Data Set  
Parsley Data Set

Key:

Treatment 1 = With Oil

Treatment 2 = Without Oil

Dormant Spray - Study #91  
 Effect of oil on spray deposition  
 Comparison of branches and surrogates

3

14:01 Monday, December 17, 1990

----- Treatment=1 Replicate=1 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0055408	0.0016879
	SURDEP2	mg/cm2 on surr	0.0077797	0.0027130

----- Treatment=1 Replicate=2 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0070094	0.0014107
	SURDEP2	mg/cm2 on surr	0.0096987	0.0036573

----- Treatment=1 Replicate=3 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0109975	0.0020673
	SURDEP2	mg/cm2 on surr	0.0119599	0.0011877

----- Treatment=1 Replicate=4 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0080506	0.0020719
	SURDEP2	mg/cm2 on surr	0.0109216	0.0048430

----- Treatment=1 Replicate=5 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0102711	0.000759596
	SURDEP2	mg/cm2 on surr	0.0132866	0.0014475

Dormant Spray - Study #91  
 Effect of oil on spray deposition  
 Comparison of branches and surrogates

14:01 Monday, December 17, 1990

----- Treatment=2 Replicate=1 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0095326	0.0019481
	SURDEP2	mg/cm2 on surr	0.0141557	0.000915861

----- Treatment=2 Replicate=2 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0066592	0.000902341
	SURDEP2	mg/cm2 on surr	0.0113138	0.0051287

----- Treatment=2 Replicate=3 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0067750	0.0019858
	SURDEP2	mg/cm2 on surr	0.0120560	0.0038217

----- Treatment=2 Replicate=4 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0086880	0.000338231
	SURDEP2	mg/cm2 on surr	0.0123752	0.0017952

----- Treatment=2 Replicate=5 -----

N Obs	Variable	Label	Mean	Std Dev
4	BRADEP2	mg/cm2 on br	0.0073418	0.0017119
	SURDEP2	mg/cm2 on surr	0.0132136	0.0026784

PARSLEY STUDY RAW DATA

14:01 Monday, December 17, 199

----- TRT=1 DAY=3 -----

OBS	DEP
1	0.91
2	1.07
3	0.97
4	1.22
5	0.69

----- TRT=1 DAY=6 -----

OBS	DEP
6	1.84
7	1.35
8	1.25
9	1.63
10	1.59

----- TRT=1 DAY=9 -----

OBS	DEP
11	2.90
12	1.48
13	2.15
14	1.62
15	1.79

----- TRT=1 DAY=12 -----

OBS	DEP
16	2.77
17	2.10
18	2.09
19	2.63
20	1.70

PARSLEY STUDY RAW DATA

1

14:01 Monday, December 17, 199

----- TRT=1 DAY=15 -----

OBS	DEP
21	3.49
22	1.80
23	2.40
24	3.59
25	3.09

----- TRT=1 DAY=22 -----

OBS	DEP
26	3.48
27	2.48
28	3.04
29	3.59
30	2.58

----- TRT=1 DAY=29 -----

OBS	DEP
31	6.73
32	1.75
33	2.95
34	3.58
35	2.33

----- TRT=1 DAY=36 -----

OBS	DEP
36	7.06
37	1.61
38	2.95
39	3.15
40	2.23

PARSLEY STUDY RAW DATA

1

14:01 Monday, December 17, 199

----- TRT=2 DAY=3 -----

OBS	DEP
41	1.05
42	1.18
43	1.11
44	1.62
45	0.72

----- TRT=2 DAY=6 -----

OBS	DEP
46	1.44
47	1.74
48	2.14
49	1.41
50	1.25

----- TRT=2 DAY=9 -----

OBS	DEP
51	4.75
52	1.64
53	2.82
54	1.50
55	1.87

----- TRT=2 DAY=12 -----

OBS	DEP
56	1.82
57	2.48
58	2.67
59	2.10
60	2.26

PARSLEY STUDY RAW DATA

1  
14:01 Monday, December 17, 199

----- TRT=2 DAY=15 -----

OBS	DEP
61	3.39
62	3.02
63	2.59
64	2.83
65	3.13

----- TRT=2 DAY=22 -----

OBS	DEP
66	3.32
67	2.37
68	3.81
69	2.92
70	2.71

----- TRT=2 DAY=29 -----

OBS	DEP
71	3.50
72	1.81
73	3.25
74	2.81
75	2.85

----- TRT=2 DAY=36 -----

OBS	DEP
76	4.34
77	4.62
78	2.48
79	2.87
80	1.61