

**Monitoring Diazinon  
in the Mediterranean Fruit Fly Eradication  
Soil Treatment Program,  
Los Angeles County, California, 1992**

**By**

**C. Ando, J. Leyva, and C. Gana**

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**ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM**

STATE OF CALIFORNIA  
Environmental Protection Agency  
Department of Pesticide Regulation  
Environmental Monitoring and Pest Management Branch  
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EXECUTIVE SUMMARY  
of Report EH 93-01 Entitled  
"Monitoring Diazinon in the Mediterranean Fruit Fly  
Eradication Soil Treatment Program,  
Los Angeles County, California, 1992"

Environmental Monitoring and Pest Management Branch  
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**PURPOSE**

Since no specific residue monitoring has been done for diazinon during past fruit fly eradication projects, this study was conducted to measure over time the amounts of diazinon and diazoxon (a compound that has been chemically transformed from the parent compound, diazinon) on turfgrass and surface soil during a fruit fly eradication program. This data will be used for exposure assessment by the Department of Pesticide Regulation.

**BACKGROUND**

Soil treatment with diazinon is used in exotic fruit fly eradication programs by California Department of Food and Agriculture (CDFA) to kill pre-pupal to adult emergent stages of the insect in the soil. Diazinon is sprayed on the ground under the host tree canopy from the trunk to the drip line, and then watered in to a point before puddling or runoff occurs. A treatment program consists of up to three diazinon applications made at 14-day intervals.

People may be potentially exposed to diazinon through the skin or ingestion if they contact soil and turf to which diazinon has been applied during a fruit fly eradication program. No specific residue monitoring has been done for diazinon during past fruit fly eradication projects. Residue monitoring has been done in association with diazinon applications for Japanese beetle.

This study was conducted to monitor the levels and estimate the changes in concentration over time of diazinon and diazoxon on turfgrass and surface soil under field conditions during a fruit fly eradication program in Duarte, Los Angeles County.

**STUDY METHODS**

During September and October, 1993, Environmental Hazards Assessment Program scientists selected four soil and four turf sites beneath trees for long-term monitoring in CDFA eradication project.

Two small subsites were randomly located under the tree canopy of each of the eight sites and samples were collected from the subsites during the monitoring. Samples of surface soil (soil within 1 cm of surface) and turfgrass were collected just before (background samples) and approximately one hour after (Day 0 samples) each of the three diazinon applications that comprised a treatment. Additional samples of both soil and turf were collected up to 33 days following the third, final diazinon application.

## RESULTS

Diazinon residue levels for both media were inexplicably lower for the third application than for the levels reported for either applications one or two. The pesticide deposition rate and the amount of water applied to the sites after pesticide application were not quantified during the study, and consequently, values could not be compared to identify if these or other factors were responsible for the observed residue differences between applications. Also, the diazinon quality control data associated with the third application suggested possible analytical problems. Due to the uncertainty of the data collected from the third application, it is suggested that data from the first and second applications be more appropriate for estimating exposure.

Soil: One-hundred and four samples were collected from September 27 to November 29, 1993, and were analyzed for diazinon and diazoxon residues. Diazinon was not detected in soil before the first ground application. Within approximately one hour after the first application, diazinon concentrations increased to 17.10 micrograms ( $\mu\text{g}$ ) diazinon per gram (g) soil, which is equivalent to 17.10 ppm. This value was the highest diazinon level reported in soil throughout the entire monitoring period.

After the final application, changes in concentrations over time were measured. From day zero to day three of the third (and final) application, diazinon levels decreased from 1.87  $\mu\text{g/g}$  (1.87 ppm) soil to 0.38  $\mu\text{g/g}$  (0.38 ppm) soil and remained at approximately that level to the end of the 33-day sampling period.

Of the 104 surface soil samples collected, only eight contained detectable levels of diazoxon. Seven of the eight detections were reported just after the first application. These values ranged from 0.0048 (0.0048 ppm) to a high of 0.0194  $\mu\text{g}$  diazoxon/g (0.0194 ppm) soil.

Turf: The greatest amount of dislodgeable residues of diazinon was 144.36  $\mu\text{g/g}$  (144.36 ppm) turf, which was reported for the second application. Dislodgeable residues of diazoxon were also highest at this sampling period with the diazoxon concentration reported at 0.59  $\mu\text{g/g}$  (0.59 ppm) turf.

Data collected from monitoring which followed the third application indicated a decrease in dislodgeable residues of diazinon and diazoxon over the three days following the third application with levels declining from 36.48 (36.48 ppm) to 5.12  $\mu\text{g}$  diazinon/g (5.12 ppm) turf and from 0.35 (0.35 ppm) to 0.05  $\mu\text{g}$  diazoxon/g (0.05 ppm) turf. Dislodgeable concentrations for both compounds remained at approximately these lowest levels during the rest of the 33-day sampling period.

## CONCLUSIONS

The highest diazinon residues in soil were detected immediately following the first soil application. The results from this study do not indicate any significant accumulation of diazinon in soil or turfgrass with each successive spray. Diazinon appeared to dissipate gradually following the third application, although residues remained detectable over the 33-day period following the third application. Since diazoxon detections occurred almost exclusively in samples immediately following the first application, this would indicate

that analysis in soil should be limited to diazinon since diazoxon was not frequently detected.

Dislodgeable residues of both diazinon and diazoxon were present on turf up to 33 days after the last application.

These data should be used with discretion for exposure assesement due to the inexplicable low residue values obtained after the third application and due to the laboratory quality control data, which suggested possible analytical problems during this same period. For exposure assessment, it is suggested that data from applications one and two be utilized.

It is recommended that additional monitoring of future diazinon treatment programs be conducted for both soil and turf in a sequence of three diazinon applications since the highest detectable levels of diazinon may be found in any one of the three applications. This additional data may be used in estimating exposure and determining dissipation rates.



John Sanders  
Branch Chief

12/01/93

**MONITORING DIAZINON IN THE MEDITERRANEAN FRUIT FLY ERADICATION SOIL  
TREATMENT PROGRAM, LOS ANGELES COUNTY, CALIFORNIA, 1992**

**BY**

**C. Ando, J. Leyva, and C. Gana**

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**ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM**

## ABSTRACT

The Pest Detection/Emergency Projects Branch of the California Department of Food and Agriculture (CDFA) applied diazinon pesticide as a ground treatment in Duarte, California (Los Angeles County). Applications were made under the canopy of host fruit trees to eradicate larval or adult stages of the Mediterranean fruit fly. One treatment consisted of three diazinon applications made at 14-day intervals. From September to December, 1992, the Environmental Hazards Assessment Program of the Department of Pesticide Regulation monitored concentrations of diazinon and diazoxon residue at four soil and four turf sites following each of the three applications. Furthermore, additional samples were also collected up to 33 days after the last application and analyzed for both residues. Data was logarithmically transformed and estimated means were calculated. In soil, mean diazinon levels ranged from 0.38 to 17.10  $\mu\text{g/g}$  over the 2 month monitoring period. Individual diazoxon levels measured in soil never exceeded 0.02  $\mu\text{g/g}$  and detections were reported in less than 10% of the samples analyzed. In turf, measurable mean levels of dislodgeable diazinon ranged from 0.20 to 144.36  $\mu\text{g/g}$ . Total mean diazinon (dislodgeable and internal residue) ranged from 2.83 to 190.93  $\mu\text{g/g}$ . Mean diazoxon concentrations in turf were lower than those reported for diazinon and ranged from 0.01 to 0.59  $\mu\text{g/g}$  for dislodgeable residue and 0.02 to 0.94  $\mu\text{g/g}$  for total residue. Since the diazinon residue levels in both media were inexplicably low following the third application and the quality control data suggested possible analytical problems after the third application, we suggest that data from the first and second applications would be more appropriate for estimating exposure.

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## **DISCLAIMER**

The mention of commercial products, their source or use in connection with material reported herein is not to be construed as an actual or implied endorsement of such product.

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## INTRODUCTION

The staff of the Environmental Hazards Assessment Program (EHAP) monitored the residues of diazinon insecticide applied by the Pest Detection/Emergency Projects Branch of the California Department of Food and Agriculture (CDFA). In the CDFA Mediterranean fruit fly eradication program, diazinon is used as a soil drench under the canopy of host fruit trees to kill prepupal or adult stages of the fruit fly as it enters or emerges from the soil, respectively. A treatment is composed of up to three diazinon applications made at 14-day intervals.

Diazinon (O,O-Diethyl O-(2-isopropyl-6-methyl-4-pyrimidinyl)phosphorothioate) is a non-systemic organophosphate chemical with a vapor pressure of 0.097 mPa at 20°C and a specific gravity of 1.116-1.118 at 20°C. The reported water solubility is 40 mg/L at 20°C (Biggar and Seiber, 1987). Diazinon is a non-systemic insecticide with contact stomach and respiratory actions, and it is a cholinesterase inhibitor.

In 1992, EHAP staff monitored the CDFA diazinon ground application in the city of Duarte, Los Angeles County, California. Dissipation of diazinon and diazoxon, a breakdown product, was measured in soil and turf samples collected from September to December during the eradication program.

## MATERIALS AND METHODS

### Site Selection

In September and October, 1992, EHAP staff selected four soil and four turf sites beneath host fruit trees in the city of Duarte. The average tree canopy diameter was 3 m at soil sites and 4 m at turf sites. Two subsites, each measuring 0.6 x 0.6 m, were randomly located under the canopy of each tree and sample collection was confined to the subsites. Background (day -1) soil and turf samples were collected prior to each of the diazinon treatments and then at approximately 1 hour following each spray (day 0). Intensive monitoring of both media occurred at the third diazinon application with samples collected on 1, 2, 3, 8, 12, 16, and 33 days after application.

### C DFA Diazinon Ground Treatment Program

Diazinon AG500® (emulsifiable concentrate containing 1.82 kg active ingredient (ai) per 3.79 L) was applied by C DFA staff to the dripline of host trees in three applications, each spaced fourteen days apart. The target application rate was 0.10 L of pesticide concentrate to 11.37 L of water per 92.9 m<sup>2</sup>. The water was buffered to pH 6.5 before mixing in the concentrate. Applications were made with a 11.6-L hand held Chapman sprayer equipped with a fan tip nozzle. The volume of chemical applied to the ground was estimated by examining the volume remaining in the sprayer. Shortly after application, diazinon was moved into the upper layers of soil by applying water to application sites with a garden hose.

### Soil Monitoring

Samples were collected using a stainless steel cylinder with an internal diameter of 6.03 cm. Three soil cores were randomly collected from each subsite by inserting the steel cylinder down to a 1 cm depth from the soil surface. Soil from each subsite was composited and placed into a 1-pint glass mason jar and sealed with an aluminum-lined lid. Split soil samples to be analyzed by the primary and secondary laboratories were collected on days 0, 1, 2, and 3 of the third application. On these days, 6 cores of soil were collected. Samples were thoroughly mixed, placed on dry ice for shipping, and kept frozen until extraction for chemical analysis. Diazinon and diazoxon residues were expressed on weight per weight basis ( $\mu\text{g/g}$  of soil, wet weight) and on an area basis,  $\mu\text{g}/\text{cm}^2$ .

Additional soil samples were collected from each subsite prior to the first and third applications to determine percent soil organic carbon, soil pH, and soil texture. These samples were also collected from the top 1 cm of soil, placed in pint mason jars, and kept frozen until analysis.

### Turf Monitoring

A steel cylinder with an internal diameter of 6.03 cm was used to randomly sample turf and thatch. A core consisting of turf and soil was removed from the ground at each subsite. The cylinder contents were gently removed by holding the cylinder in a vertical position (turf side up) and pushing a smaller diameter rod upward from the soil portion of the chamber. Once the core was exposed, the turf and thatch was then cut directly from the core into a glass, pint mason jar. Turf samples were composited from each subsite until 25 g of vegetative material was collected. Results were expressed on a

weight per weight basis ( $\mu\text{g/g}$  of turf and thatch, wet weight) and on a ground-surface area measurement ( $\mu\text{g/cm}^2$ ), which should not be confused with leaf blade surface area measurement, which was not determined. Samples were sealed with aluminum-lined lids, transported on wet ice, and maintained at  $4^{\circ}\text{C}$  until extraction for chemical analysis. Turf samples were extracted by CDFA Laboratory personnel to determine dislodgeable residue and total residue which is the sum of the amount in dislodgeable and internal analyses.

### Tank Samples

One tank sample was collected on each application day to measure the percent diazinon in the tank mix. A one-pint sample was collected directly from the nozzle of the hand sprayer into the sample container. Each container was sealed with an aluminum-lined lid, double bagged, and placed in an ice chest on wet ice for transportation. This ice chest contained only tank samples.

### Chemical Analyses and Quality Control

Chemical analyses for diazinon and diazoxon in soil were conducted by CDFA's Chemistry Laboratory Services in Sacramento, California (Appendix A). CDFA used ethyl acetate to extract diazinon and diazoxon in soil. Residues were analyzed using gas liquid chromatography with a flame photometric detector - phosphorus mode for both compounds. The minimum detection limit for diazinon and diazoxon was  $0.15 \mu\text{g/sample}$ .

Quality control analyses of split soil samples was conducted by Enseco Laboratory located in West Sacramento, California. Enseco's soil extraction method differed from CDFA's in that Enseco extracted diazinon from soil with methylene chloride-acetone (Appendix A) and not ethyl acetate. Diazinon was

the only compound which Enseco analyzed for. The minimum detection limit for diazinon was 0.10  $\mu\text{g/g}$  and analyses was performed using a gas chromatograph with a thermionic specific detector.

CDFA analyzed turf to determine dislodgeable and internal levels of diazinon and diazoxon residues (Appendix A). The dislodgeable fraction was removed by rotating the turf in a surfactant-water solution. Aqueous washings were repeated and the wash water combined and extracted with ethyl acetate. The minimum detection limit was 0.15  $\mu\text{g/sample}$  for dislodgeable diazinon and diazoxon. Internal diazinon and diazoxon residues were extracted from the washed turf by grinding the material in a blender containing ethyl acetate. The minimum detection limit for internal diazinon and diazoxon residue was 0.15  $\mu\text{g/sample}$ . Analyses for dislodgeable and internal residue were performed by gas-liquid chromatography using a flame photometric detector - phosphorus mode.

### Statistical Analyses

The appearance of the data was consistent with the assumption of lognormally distributed values on each day. Therefore observations were transformed to the natural logarithm (log) before analysis, and methods for estimation in lognormal populations were employed.

Mean diazinon residue for each day was estimated by the method due to Finney (1941) and described by Gilbert (1987). It provides an unbiased estimate of the mean of the lognormal (i.e., untransformed) population. The estimate,  $\hat{\mu}$ ,

is calculated as  $\hat{\mu} = \exp(m) \cdot \psi(s^2/2)$  where  $m$  is the mean of the logs, and  $\psi$  is a function of the estimated variance,  $s^2$ , and its degrees of freedom. This estimate is superior to the simple arithmetic or raw mean of the untransformed observations (which is also unbiased for  $\mu$ ) because it has smaller variance.

The calculation of  $\hat{\mu}$  requires an estimate of the variance of the log observations,  $s^2$ . Since variability on the log scale was approximately the same for all days, a common pooled estimate of within-day variance was used for all the days, instead of separate daily variance estimates. When the assumption of common variance is correct, the pooled estimate is more precise. The common within-day variance was obtained from an analysis of variance (Appendix A) on log residue by day, with repeated measures on two sizes of experimental unit, sites and subsites. Sites and subsites were treated as random factors, with subsites nested in sites. Variance components were estimated by equating each mean square to its expected value and solving the resulting system of equations (e.g., Neter et al., 1985). The estimated variance of observations within days was then calculated as the sum of the variance components for sites, subsites within sites, sites by days, and subsites by days within sites, and its degrees of freedom calculated by the Satterthwaite approximation (Neter et al., 1985).

Confidence limits for each daily mean were calculated by the method developed by Land (1971) and Land et al. (1987). The 5th and 95th quantiles of the daily distributions of individual observations were estimated as  $\hat{\theta}(q) = \exp\{m + t(q) \cdot s\}$  where  $q$  indicates the  $q$ th quantile. This simple estimator is Maximum Likelihood, although not unbiased (Shimizu, 1988).

In situations where diazinon or diazoxon was not detected in soil or turf, values were calculated using one-half the detection limit. This provided a conservative estimate of the concentration in either media since it is assumed that the residue was present but below the detection limit.

Since residue levels following applications number 1 and 2 were closer to expected levels than application number 3, dissipation rates over the 14-day periods following the first and second applications were determined by using analysis of variance to test for the presence of an application by day interaction. The analysis was performed on the data from four days: day 0 and day 14 for application 1, and day 14 and day 28 for application 2.

## RESULTS AND DISCUSSION

The original objective of the study was to develop dissipation curves for diazinon and diazoxon based on residue levels present after the third diazinon application. Diazinon residue levels in both media following the third application were inexplicably low. Neither the pesticide deposition rate or the amount of water applied to the sites following pesticide application were measured, and consequently, the relationship of these characters to the low residue levels could not be evaluated. The quality control data for diazinon also suggested possible analytical problems after the third application (See Quality Control) and cast some uncertainty on the representativeness of the data. Based on these results, dissipation curves were not determined after the third application. Instead, means, confidence intervals, and quantiles are presented for each sampling date.

The arithmetic (raw) and estimated means for diazinon and diazoxon concentrations in soil and turf are presented both as  $\mu\text{g/g}$  on a wet weight basis and as  $\mu\text{g/cm}^2$ . Reference to values in this report pertain to the estimated mean because it provided a more precise value than the raw mean. Raw data are presented in Appendix B. Tables 1 to 4 also contain the lower and upper confidence limits, which define a range in which the true mean is expected to lie, with 90% certainty, and the 5th and 95th quantiles which define the theoretical distribution of individual samples for a sampling date. The confidence limits and the quantile values are presented here because they may provide additional information which may be useful in estimating exposure.

Because the values for the third application were unexpectedly low, we suggest that the data from the first and second applications would be more appropriate for estimating exposure.

### Soil

Soil texture at the four sites was classified either as sandy loam, loamy sand, or loam. The proportion of sand, silt, and clay averaged over all sites was 63% sand, 28% silt, and 9% clay. Soil moisture ranged from 2 to 30% by weight. Organic carbon (OC) content and soil pH averaged over all sites was similar in samples taken prior to applications 1 and 3. Percent OC and soil pH were 2.3% and 7.2, respectively, at application 1, and 2.6% and 7.3, respectively, at application 3.

Diazoxon was detected in only 8 of the 104 soil samples. Seven of the eight individual detections were reported on day 0 of application 1 and one additional sample was detected on day 0 of application 3. Concentrations ranged from 0.0032 to 0.0194  $\mu\text{g/g}$ . Lack of diazoxon detection is probably due to the fact that transformation to the oxygen analog in soil is not a major pathway and that the compound is transient in nature (Biggar and Seiber, 1987). Because of the limited number of diazoxon detections only diazinon results will be discussed.

Discussion of diazinon will be on a  $\mu\text{g/g}$  basis rather than  $\mu\text{g/cm}^2$ , as there were no apparent differences in the general dissipation trend when expressed in either unit of measurement.

Diazinon was not detected in soil prior to application 1. Immediately following the first application, however, diazinon averaged 17.10  $\mu\text{g/g}$  in soil and decreased in concentration with successive applications: 9.23 at application 2 and 1.87  $\mu\text{g/g}$  for application 3 (Figure 1). Tank samples collected on each application day were 95 to 118% of the theoretical concentration, so differences in application concentration could not account for the apparent decline in measured residue levels. CDFA laboratory, however, consistently reported lower diazinon levels than the secondary laboratory in split soil samples collected on the third application day and 3 consecutive days following this application.

Diazinon's dissipation rate was similar during each of the 14-day periods following applications 1 and 2. Following application 3, diazinon residue remained at detectable levels 33 days after the third and final application. The mean concentration at the end of this period was 0.38  $\mu\text{g/g}$ . The average rainfall was 0.40 cm for the entire monitoring period as determined from precipitation data obtained from the Hollywood and Pomona weather stations located to the east and west of Duarte, respectively (IMPACT - University of California).

Table 1A. Mean, confidence limits, and quantiles of diazinon residue in soil expressed on a wet weight basis

Appl <sup>a</sup> Seq	Sample Day	Diazinon ( $\mu\text{g/g}$ wet weight)					
		Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence Limits <sup>*</sup>		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	14.54	17.10	9.14	40.41	2.73	57.45
2	-1	7.11	6.48	3.46	15.31	1.03	21.77
2	0	14.73	9.23	4.93	21.81	1.47	31.01
3	-1	0.70	0.76	0.41	1.80	0.12	2.56
3	0	1.89	1.87	1.00	4.41	0.30	6.28
3	1	1.19	1.54	0.82	3.64	0.25	5.18
3	2	0.81	0.94	0.50	2.23	0.15	3.17
3	3	0.32	0.38	0.20	0.91	0.06	1.29
3	8	0.40	0.49	0.26	1.15	0.08	1.64
3	12	0.56	0.70	0.38	1.66	0.11	2.36
3	16	0.39	0.48	0.26	1.13	0.08	1.60
3	33	0.35	0.38	0.20	0.90	0.06	1.27

Table 1B. Mean, confidence limits, and quantiles of diazinon residue in soil expressed on a surface area basis

Appl <sup>a</sup> Seq	Sample Day	Diazinon ( $\mu\text{g}/\text{cm}^2$ )					
		Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence Limits <sup>*</sup>		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	32.70	38.52	20.08	96.34	5.64	135.07
2	-1	9.14	8.60	4.48	21.51	1.26	30.16
2	0	26.71	17.77	9.26	44.44	2.60	62.30
3	-1	1.08	1.06	0.55	2.65	0.16	3.72
3	0	3.43	3.22	1.68	8.06	0.47	11.30
3	1	1.86	2.42	1.26	6.05	0.35	8.49
3	2	1.26	1.52	0.79	3.81	0.22	5.34
3	3	0.57	0.67	0.35	1.68	0.10	2.36
3	8	0.71	0.84	0.44	2.11	0.12	2.96
3	12	0.77	0.95	0.50	2.39	0.14	3.34
3	16	0.54	0.69	0.36	1.73	0.10	2.43
3	33	0.70	0.76	0.39	1.89	0.11	2.66

a = Application sequence.

b = N equals 8 (4 sites x 2 subsites per site).

c = Estimated mean of lognormal distribution - Finney (1941) method.

d = 5th and 95th percentiles of distributions of individual samples.

e = Not detected.

\* = Confidence limits for estimated mean - Land (1971) method.

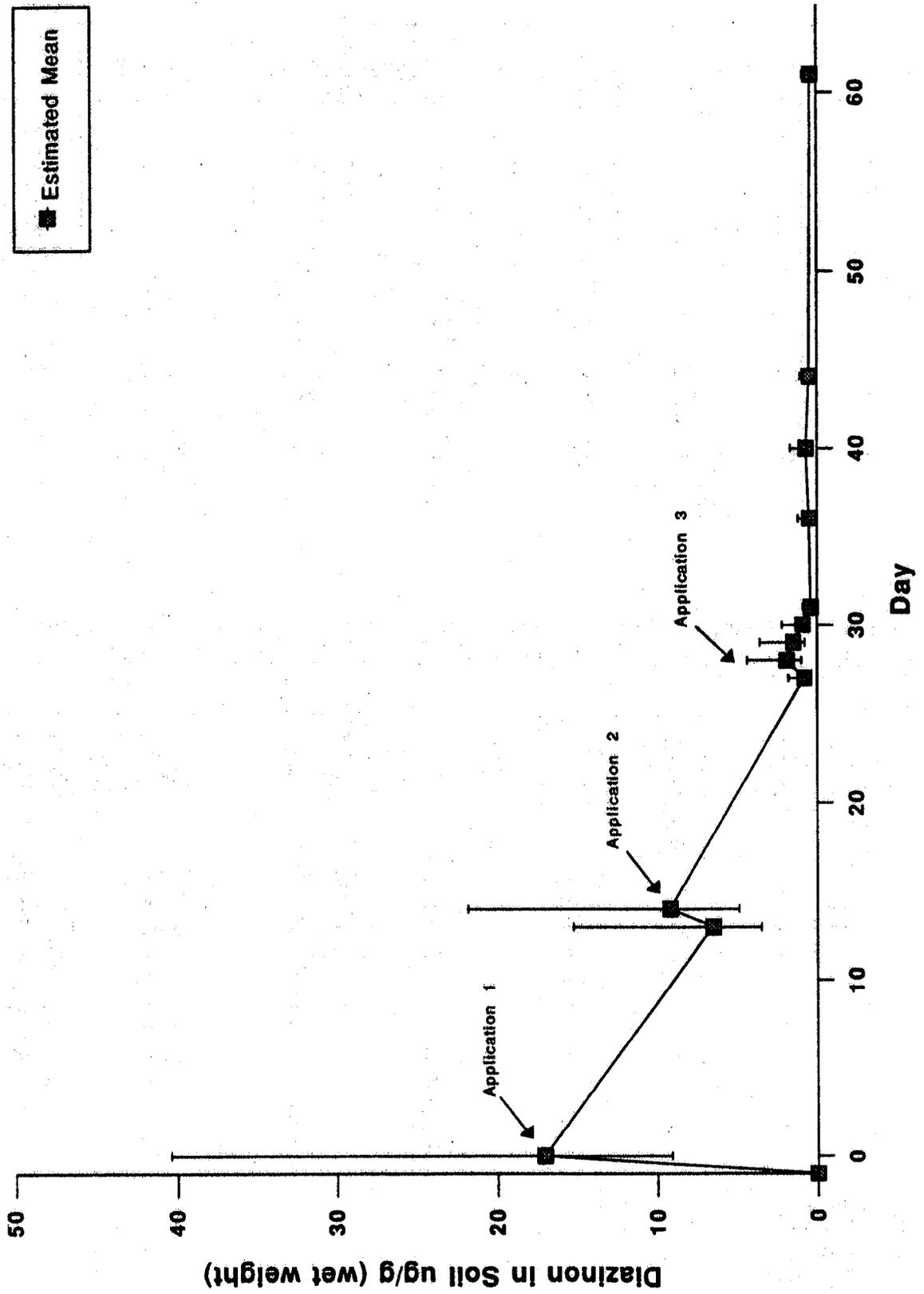


Figure 1. Diazinon dissipation in soil with time. Estimated means are connected by straight lines and the vertical bars represent lower and upper 90% confidence limits for each sampling day.

## Turf

In general, there were no appreciable differences in the pattern of results for turf when expressed as either  $\mu\text{g/g}$  or  $\mu\text{g/cm}^2$ . Consequently, discussion of results will be on a  $\mu\text{g/g}$  basis.

Diazinon was not detected in dislodgeable or total analyses of background turf samples. In contrast to soil where the highest mean diazinon level was detected at the first application, the highest mean levels for dislodgeable and total diazinon on turf were detected at the second application at 144.36 and 190.93  $\mu\text{g/g}$ , respectively (Tables 2 and 4; Figures 2 and 4).

The dissipation rate over the 14-day period following application 2 was greater than for the same 14-day interval following the first application. This was indicated by significant application by day interactions for both dislodgeable ( $F=52.9$ ;  $df=1,3$ ;  $p<0.01$ ) and total residue ( $F=36.6$ ;  $df=1,3$ ;  $p<0.01$ ).

The mean dislodgeable diazinon level was unusually low for application 3 (Figure 2). Although the tank mix for the third application was 107 and 118% of the theoretical and the quality control data during this period was acceptable, the low level remained unexplainable. The dislodgeable diazoxon level for application 3, however, was not as comparably low as the dislodgeable diazinon level for this same sampling day (Figures 2 and 3).

Following the third application, mean dislodgeable and total diazinon decreased over time, yet residue remained detectable for the remainder of the monitoring period. Thirty-three days after the last application, 0.20  $\mu\text{g/g}$

of dislodgeable diazinon and 2.83  $\mu\text{g/g}$  total diazinon residue were reported. Dislodgeable diazoxon and total diazoxon were also detected 33 days after the last application at 0.01  $\mu\text{g/g}$  and 0.02  $\mu\text{g/g}$ , respectively (Table 3; Figure 3). The average rainfall for the 61-day monitoring period was 9.04 cm.

Table 2A. Mean, confidence limits, and quantiles of dislodgeable diazinon residue on turf expressed on a wet weight basis

		Dislodgeable Diazinon ( $\mu\text{g/g}$ )					
Appl <sup>a</sup> Seq	Sample Day	Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence Limits <sup>*</sup>		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	39.78	62.30	28.79	222.64	5.29	278.29
2	-1	9.97	13.74	6.35	49.09	1.17	61.36
2	0	98.38	144.36	66.72	515.86	12.26	644.80
3	-1	2.44	2.69	1.24	9.61	0.23	12.01
3	0	29.26	36.48	16.86	130.35	3.10	162.93
3	1	30.40	38.19	17.65	136.48	3.24	170.59
3	2	14.86	13.98	6.46	49.97	1.19	62.46
3	3	5.53	5.12	2.37	18.30	0.43	22.87
3	8	1.96	1.25	0.58	4.47	0.11	5.58
3	12	1.28	1.32	0.61	4.71	0.11	5.89
3	16	0.88	0.73	0.34	2.60	0.06	3.25
3	33	0.32	0.20	0.09	0.72	0.02	0.91

Table 2B. Mean, confidence limits, and quantiles of dislodgeable diazinon residue on turf expressed on a land surface area basis

		Dislodgeable Diazinon ( $\mu\text{g/cm}^2$ )					
Appl <sup>a</sup> Seq	Sample Day	Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence Limits		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	5.89	9.26	4.02	40.74	0.62	45.91
2	-1	1.13	1.34	0.58	5.90	0.09	6.65
2	0	9.04	14.01	6.08	61.64	0.94	69.47
3	-1	0.35	0.24	0.11	1.07	0.02	1.21
3	0	2.24	3.28	1.42	14.43	0.22	16.27
3	1	2.31	2.83	1.23	12.44	0.19	14.02
3	2	1.03	1.20	0.52	5.28	0.08	5.95
3	3	0.44	0.52	0.22	2.28	0.03	2.56
3	8	0.22	0.13	0.06	0.59	0.01	0.66
3	12	0.11	0.11	0.05	0.46	0.01 <sup>f</sup>	0.52
3	16	0.07	0.05	0.02	0.23	0.00 <sup>f</sup>	0.26
3	33	0.02	0.02	0.01	0.07	0.00 <sup>f</sup>	0.08

a = Application sequence.

b = N equals 8 (4 sites x 2 subsites per site).

c = Estimated mean of lognormal distribution - Finney (1941) method.

d = 5th and 95th percentiles of distributions of individual samples.

e = Not detected.

f = Value calculated is below  $0.01 \mu\text{g/cm}^2$  or  $0.01 \mu\text{g/g}$ .

\* = Confidence limits for estimated mean - Land (1971) method.

Table 3A. Mean, confidence limits, and quantiles of dislodgeable diazoxon residue on turf expressed on a wet weight basis

Dislodgeable Diazoxon ( $\mu\text{g/g}$ wet weight)							
Appl <sup>a</sup> Seq	Sample Day	Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence <sup>*</sup> Limits		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	0.16	0.20	0.10	0.55	0.02	0.76
2	-1	0.07	0.09	0.04	0.24	0.01	0.34
2	0	0.52	0.59	0.29	1.60	0.07 <sup>f</sup>	2.23
3	-1	0.03	0.03	0.02	0.09	0.00 <sup>f</sup>	0.13
3	0	0.44	0.35	0.18	0.96	0.04	1.33
3	1	0.23	0.25	0.13	0.68	0.03	0.94
3	2	0.12	0.13	0.07	0.36	0.02	0.50
3	3	0.05	0.05	0.02	0.13	0.01 <sup>f</sup>	0.18
3	8	0.02	0.02	0.01	0.06	0.00 <sup>f</sup>	0.08
3	12	0.09	0.07	0.03	0.19	0.01 <sup>f</sup>	0.26
3	16	0.01	0.01	0.01 <sup>f</sup>	0.03	0.00 <sup>f</sup>	0.04
3	33	0.01	0.01	0.00 <sup>f</sup>	0.02	0.00 <sup>f</sup>	0.03

Table 3B. Mean, confidence limits, and quantiles of dislodgeable diazoxon residue on turf expressed on a land surface area basis

Dislodgeable Diazoxon ( $\mu\text{g/cm}^2$ )							
Appl <sup>a</sup> Seq	Sample Day	Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence <sup>*</sup> Limits		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	0.02	0.03	0.01 <sup>f</sup>	0.07	0.00 <sup>f</sup>	0.10
2	-1	0.01	0.01	0.00 <sup>f</sup>	0.02	0.00 <sup>f</sup>	0.03
2	0	0.04 <sup>f</sup>	0.05 <sup>f</sup>	0.03 <sup>f</sup>	0.13	0.01 <sup>f</sup>	0.19
3	-1	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.01	0.00 <sup>f</sup>	0.01
3	0	0.05	0.03	0.01	0.07	0.00 <sup>f</sup>	0.10
3	1	0.01	0.02	0.01	0.04	0.00 <sup>f</sup>	0.06
3	2	0.01 <sup>f</sup>	0.01 <sup>f</sup>	0.01 <sup>f</sup>	0.03	0.00 <sup>f</sup>	0.04
3	3	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.01	0.00 <sup>f</sup>	0.02
3	8	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.01	0.00 <sup>f</sup>	0.01
3	12	0.01 <sup>f</sup>	0.01 <sup>f</sup>	0.00 <sup>f</sup>	0.01 <sup>f</sup>	0.00 <sup>f</sup>	0.02 <sup>f</sup>
3	16	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>
3	33	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>	0.00 <sup>f</sup>

a = Application sequence.

b = N equals 8 (4 sites x 2 subsites per site)

c = Estimated mean of lognormal distribution - Finney (1941) method.

d = 95th and 5th percentiles of distributions of individual samples.

e = Not detected.

f = Calculated value is below 0.01  $\mu\text{g/cm}^2$  or 0.01  $\mu\text{g/g}$ .

\* = Confidence limits for estimated mean - Land (1971) method.

Table 4A. Mean, confidence limits, and quantiles of total diazinon residue (dislodgeable and internal) for turf expressed on a wet weight basis

Appl <sup>a</sup> Seq	Sample Day	Total Diazinon ( $\mu\text{g/g}$ wet weight)					
		Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence* Limits		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	69.37	87.96	49.39	198.47	15.95	284.58
2	-1	20.70	22.72	12.76	51.25	4.12	73.49
2	0	154.95	190.93	107.21	430.78	34.61	617.69
3	-1	7.36	7.77	4.37	17.54	1.41	25.15
3	0	56.54	62.44	35.06	140.88	11.32	202.01
3	1	64.81	70.48	39.58	159.02	12.78	228.02
3	2	33.17	32.05	18.00	72.32	5.81	103.70
3	3	16.17	14.62	8.21	32.98	2.65	47.28
3	8	7.61	6.64	3.73	14.98	1.20	21.49
3	12	9.15	8.54	4.80	19.27	1.55	27.63
3	16	4.97	4.28	2.40	9.65	0.78	13.84
3	33	3.96	2.83	1.59	6.39	0.51	9.16

Table 4B. Mean, confidence limits, and quantiles of total diazoxon residue (dislodgeable and internal) for turf expressed on a wet weight basis

Appl <sup>a</sup> Seq	Sample Day	Total Diazoxon ( $\mu\text{g/g}$ wet weight)					
		Raw <sup>b</sup> Mean	Est <sup>bc</sup> Mean	90% Confidence* Limits		Quantiles <sup>d</sup>	
				Lower	Upper	5th	95th
1	-1	ND <sup>e</sup>	-	-	-	-	-
1	0	0.25	0.31	0.17	0.70	0.05	1.00
2	-1	0.12	0.15	0.08	0.35	0.03	0.50
2	0	0.89	0.94	0.52	2.14	0.17	3.06
3	-1	0.07	0.08	0.05	0.19	0.01	0.27
3	0	0.58	0.50	0.27	1.12	0.09	1.60
3	1	0.46	0.48	0.26	1.09	0.08	1.56
3	2	0.23	0.25	0.14	0.56	0.04	0.81
3	3	0.11	0.10	0.06	0.23	0.02	0.33
3	8	0.07	0.06	0.03	0.14	0.01	0.21
3	12	0.14	0.14	0.08	0.32	0.02	0.45
3	16	0.08	0.07	0.04 <sup>f</sup>	0.15	0.01 <sup>f</sup>	0.22
3	33	0.02	0.02	0.00 <sup>f</sup>	0.05	0.00 <sup>f</sup>	0.08

a = Application sequence.

b = N equals 8 (4 sites x 2 subsites per site).

c = Estimated mean of lognormal distribution - Finney (1941) method.

d = 5th and 95th percentiles of distributions of individual samples.

e = Not detected.

f = Calculated value is below 0.01  $\mu\text{g/g}$ .

\* = Confidence limits for estimated mean - Land (1971) method.

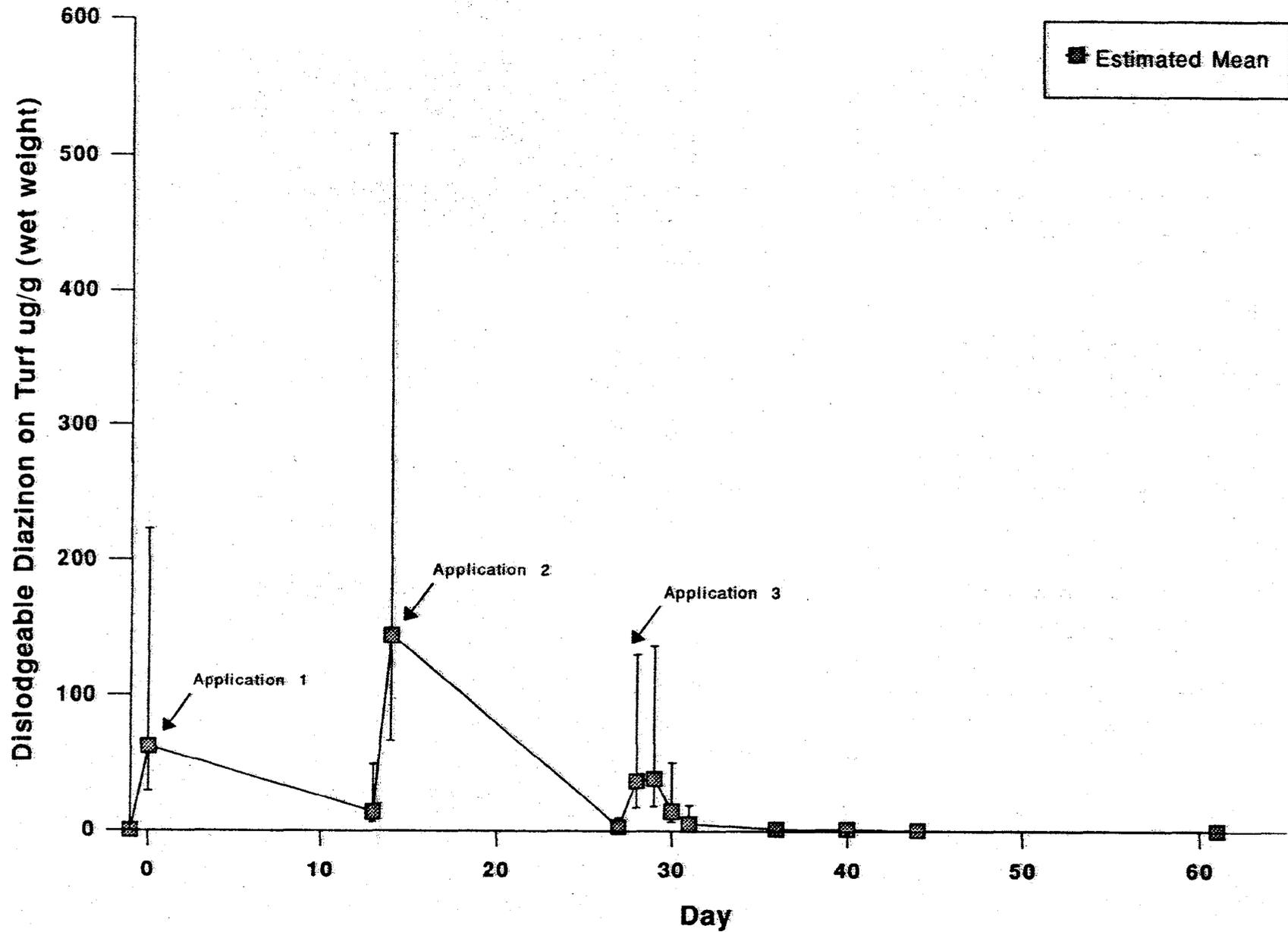


Figure 2. Dislodgeable diazinon dissipation on turf with time. Estimated means are connected by straight lines and the vertical bars represent lower and upper 90% confidence limits for each sampling day.

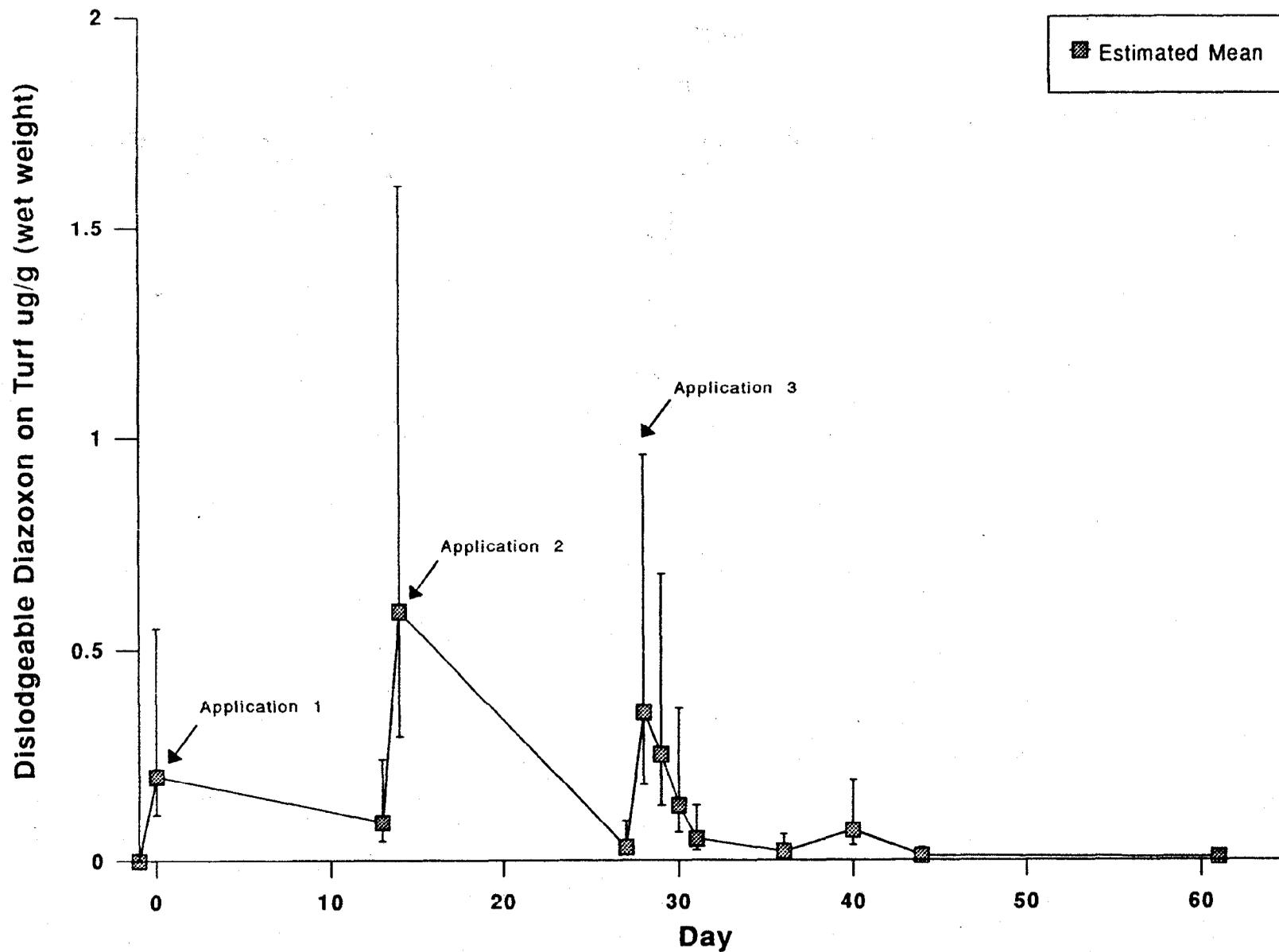


Figure 3. Dislodgeable diazoxon dissipation on turf with time. Estimated means are connected by straight lines and the vertical bars represent lower and upper 90% confidence limits for each sampling day.

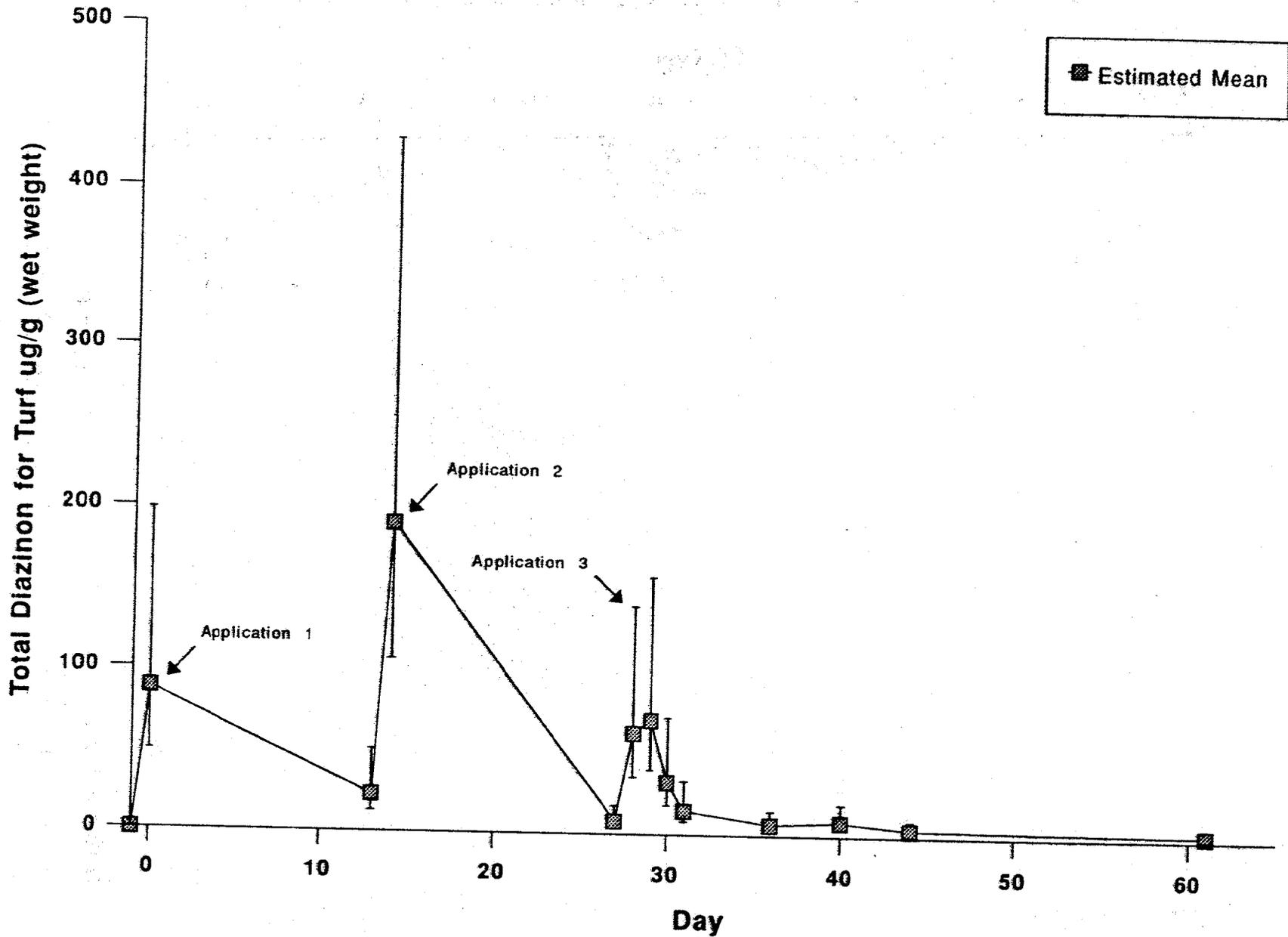


Figure 4. Total diazinon (dislodgeable and internal residue) dissipation on turf with time. Estimated means are connected by straight lines and the vertical bars represent lower and upper 90% confidence limits for each sampling day.

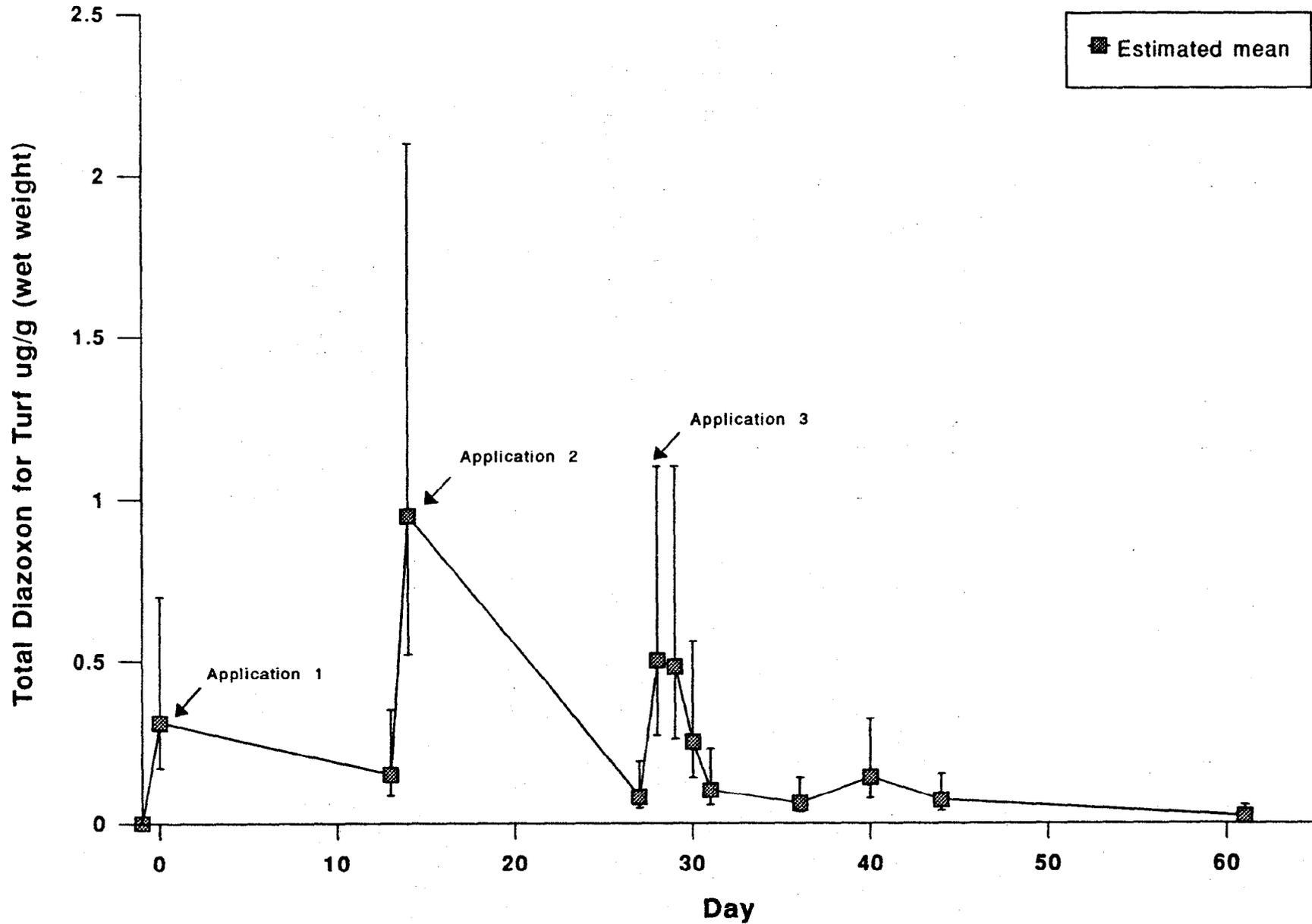


Figure 5. Total diazoxon (dislodgeable and internal residue) dissipation on turf with time. Estimated means are connected by straight lines and the vertical bars represent lower and upper 90% confidence limits for each sampling day.

### Tank Samples

The six tank samples contained diazinon concentrations which ranged from 89 to 118% of the theoretical concentration (0.45%). Diazoxon was not detected in any of the samples and the minimum detection limit for the oxygen analog was 0.3 mg/L.

### Quality Control

Method validation studies for diazinon and diazoxon in soil were completed prior to the monitoring study; diazinon mean recovery was 102% (SD=6.1 and CV=6.0%). Lower and upper control limits were set at mean recovery  $\pm$  3 times the SD (Appendix C). For continuous quality control during analyses, one matrix spike and one blank matrix sample was analyzed with each extraction set. During four extraction sets, spiked soil samples analyzed for diazinon fell below the lower control limits. Samples associated with the four extraction sets were collected primarily from the third application (day 0 to day 12). Overall continuing quality control for diazinon recovery in soil was 85% (SD=8.2, CV=9.6%) for spike matrix samples.

Method validation work for diazoxon recovery in soil was 95% (SD=8.7 and CV=9.2%). Individual spike matrix samples did not exceed the determined lower or upper control limits. Overall continuing quality control for diazoxon in soil was 97% (SD=8.3, CV=8.5%) for spike matrix samples.

Diazinon analysis of the 16 individual soil samples split between CDFA and Enseco laboratories showed that soil moisture determination between both laboratories was similar. Diazinon soil concentrations in the split samples on a dry weight basis, ranged from 0.22 to 6.60  $\mu\text{g/g}$  (Table 5). Regression

analysis of Enseco diazinon results on CDFA's, excluding the sample with the highest diazinon concentration, reflected an approximate 40% difference in concentration between laboratories with CDFA reporting lower diazinon values ( $R^2=0.84$ ). Wet weight results were virtually identical to those for dry weight.

Table 5. Inter-laboratory soil sample results

Appl <sup>a</sup> Seq	Sample Day	Diazinon ( $\mu\text{g/g}$ )			
		Wet Weight		Dry Weight	
		CDFA	Enseco	CDFA	Enseco
3	0	0.77	1.21	0.89	1.40
3	0	0.80	1.20	0.99	1.50
3	0	0.66	1.02	0.77	1.20
3	0	5.82	5.82	6.56	6.60
3	1	1.83	2.87	1.97	3.10
3	1	1.28	1.36	1.39	1.50
3	1	1.04	1.37	1.20	1.60
3	1	1.56	2.95	1.79	3.40
3	2	2.11	3.10	2.32	3.40
3	2	0.45	0.81	0.51	0.93
3	2	0.47	0.59	0.57	0.73
3	2	1.05	1.51	1.31	1.90
3	3	0.65	1.17	0.80	1.80
3	3	0.34	0.58	0.48	0.83
3	3	0.17	0.50	0.22	0.66
3	3	0.54	1.48	0.71	1.90

a = Application sequence.

CDFA completed method validation work on turf prior to monitoring; however, procedural changes in the analytical method were made during the course of the study which diminished the relevance of the original validation results. Consequently, revised method validation work was undertaken (Appendix D). Since the revised validation work was conducted after the completion of the

monitoring study, lower and upper control limits were not determined for dislodgeable and internal turf residues of diazinon and diazoxon.

For turf, continuing quality control was conducted during chemical analyses and the overall mean dislodgeable diazinon results for spike matrix recovery was 98% (SD=6.4 and CV=6.5%). The overall mean recovery for internal diazinon spike matrix samples was 80% (SD=23 and CV=28%), although in nine of nineteen extraction sets, spike matrix recoveries for internal diazinon were less than or equal to 75%. Also during analyses, four blank matrix samples contained internal diazinon residues ranging from 0.88 to 1.85  $\mu\text{g}$  per sample. Field sample extracts submitted with the positive blank matrix samples were collected primarily from the third application. There were no other positive diazinon results for blank matrix samples submitted during analyses.

Overall mean recovery for continuing quality control results was 100% (SD=7.6 and CV=7.6%) for dislodgeable diazoxon on turf and 94% (SD=14, CV=15%) for internal diazoxon. Diazoxon was not detected in any blank matrix samples.

## CONCLUSIONS AND RECOMMENDATIONS

Conclusion 1: In soil, the highest average diazinon residue was reported immediately following application 1. The mean diazinon concentrations on successive application days were lower than this value, a result which could not be explained. The quality control data from the third application also cast some uncertainty of the representativeness of the data collected during this period.

Recommendation: Additional monitoring data are needed in order to obtain accurate values for exposure assessment and for determination of diazinon dissipation rate from repeated applications. Due to the uncertainty of the data collected from the third application, it is suggested that residue levels from applications one and two be used for exposure assessment.

Conclusion 2: In soil, diazoxon was detected in less than 10% of the samples and all concentrations were below 0.02 µg/g for individual samples. This agrees with Biggar and Seiber (1987) who reported degradation to diazoxon is not a major degradation pathway in soil.

Recommendation: Diazoxon can be excluded from further soil monitoring.

Conclusion 3: In turf, the highest average levels for dislodgeable and total diazinon residues were reported at application 2. Unusually low diazinon

levels were reported at the third application and questionable quality control data was associated with samples from the third application.

**Recommendation:** Additional monitoring data are needed in order to obtain data which can be used for risk exposure and for the determination of the diazinon dissipation rate from repeated applications. Data from the first and second applications are more suitable for risk exposure assessment.

**Conclusion 4:** Diazoxon was measured in turf samples with the greatest levels for dislodgeable and total values reported at application 2.

**Recommendation:** Diazoxon should be measured in turf samples and additional monitoring data is needed in order to obtain accurate values for risk exposure and for dissipation values from repeated applications.

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**APPENDICES**

**Appendix A - Statistical and Chemical Analytical Methods**

Table 1. Analysis of variance for estimating variance components

Source of Variance	Symbol	df	Expected Mean Square
Sites	A	3	$24\sigma^2_A + 12\sigma^2_{B(A)}$
Subsites (Sites)	B(A)	4	$12\sigma^2_{B(A)}$
Days	C	11	$8\Sigma\gamma^2/11 + 2\sigma^2_{AC} + \sigma^2_{BC(A)}$
Sites x Days	AC	33	$2\sigma^2_{AC} + \sigma^2_{BC(A)}$
Subsites x Days(Sites)	BC(A)	44	$\sigma^2_{BC(A)}$

CALIFORNIA DEPT. OF FOOD & AGRIC.  
ENVIRONMENTAL MONITORING SECTION  
CHEMISTRY LABORATORY SERVICES  
3292 Meadowview Road  
Sacramento, CA 95832

Original Date: August 26, 1992  
Supercedes: New  
Current Date: October 14, 1993  
Method No.

### Diazinon and Diazinon OA Residues in Soil

#### SCOPE:

This method is for the determination of diazinon and diazinon OA in soil.

#### PRINCIPLE:

Diazinon and Diazinon OA are extracted from soil with ethyl acetate. The resulting extract is concentrated to a known volume. Diazinon and Diazinon OA are subsequently determined using gas chromatography with flame photometric detector.

#### REAGENT AND EQUIPMENT

1. Ethyl Acetate, pesticide grade.
2. Sodium Chloride, granular, reagent grade.
3. Balance, Mettler PL 1200. Mettler Instrument Corp., Highstown, NJ.
4. Qorpak amber wide-mouth jar, 500-mL.
5. Graduated cylinder, 250-mL.
6. Funnel, 60 °C short stem, 3-4 inches diameter.
7. Graduated conical centrifuge tube, 15-mL.
8. Boiling flask, 24/40 ground glass joint, 500-mL.
9. Whatman #4 or Sharkskin filter paper, 15cm.
10. Aluminum weighing dish, 57 mm. Fisher Scientific, Santa Clara, CA.
11. Lab-Line oven or equivalent.
12. Desiccator.
13. Meyers N-EVAP. Organomation Associates Incorporated, Northborough, MA.
14. G-10 Gyrotory Shaker with CE-250S clamps, New Brunswick Scientific Co. Inc., Edison, NJ.
15. Thermolyne Vortex Maxi Mixer II. Sybron Corp., Dubuque, IA.
16. Rotary evaporator, Büchi/Brinkmann, Model R110.

#### PROCEDURE

##### A. Extraction

1. Thaw soil sample at room temperature or in a refrigerator overnight and mix thoroughly.
2. Transfer about 15 g of soil sample to a tarred aluminum dish. Place the sample in an oven maintained at 105 °C for a minimum of 6 hours. Cool in a desiccator and weigh. Calculate the moisture content of the sample by weight difference.

3. Transfer 50.0  $\pm$  0.1 g of sample into a Mason jar. If soil sample is dry, add 5 mL of distilled water and mix thoroughly. Add 100 g of anhydrous sodium sulfate and mix. Then add 150 mL of ethyl acetate, cap the jar, and shake vigorously for about 10 seconds.
4. Place the jar on a gyrotory shaker for 60 minutes at 200 rpm.
5. Remove the sample from gyrotory shaker and let it stand for 5 minutes.
6. Decant extract into a 500-mL boiling flask through a funnel lined with a filter paper that is filled with 20 g of anhydrous sodium sulfate.
7. Repeat the extraction as described in steps 3-6 twice, each time using 100 mL of ethyl acetate and shaking for 30 minutes at 200 rpm.
8. Concentrate the extract by rotary evaporation at 40-45 °C and 23 inches of Hg to 4-6 mL.
9. Quantitatively transfer the concentrate into a 15-mL graduated conical centrifuge tube. Rinse the flask twice each time with 2-3 mL of ethyl acetate and transferring the rinse liquid into the graduated centrifuge tube.
10. If needed, evaporate to slightly less than 10 mL using a N-EVAP purging with nitrogen at 40-45 °C. Cool, dilute to 10.0 mL and mix thoroughly using a Vortex mixer.
11. Transfer a portion of sample into an autosampler vial for GLC analysis. If necessary, dilute or further concentrate an aliquot of the concentrated extract for determining Diazinon and Diazinon OA.
12. Refrigerate excess concentrated extract until the analysis is satisfactorily completed.

## DETERMINATION

### A. GLC Conditions:

Hewlett Packard 5890 II equipped with a Flame Photometric Detector (phosphorus mode)

Hewlett Packard 7673 Auto Liquid Sampler

Injector: Splitless, 220 °C

Detector: 250 °C

Air: 100 mL/min.

Hydrogen: 75 mL/min.

Temperature Program:

Initial Temperature: 150 °C for 1 minute

Program Rate: 10 °C/min.

Final Temperature: 200 °C for 2 minutes

Column: Hewlett Packard HP-1, 10m x 0.53 x 2.65  $\mu$ , 100% Dimethyl polysiloxane (gum)

Carrier Gas: Helium

Flow Rate: 12 mL/min.

Septum Purge: 2 mL/min.

Make-up: 5 mL/min.

Injection Volume: 2  $\mu$ L

Retention Time: Diazinon = 5.37  $\pm$ 0.10 min., Diazinon OA = 5.02  $\pm$ 0.10 min.

Linearity Checked: 0.2  $\eta$ g - 20  $\eta$ g

### Calculations

Report data in ppm or in  $\mu$ g.

$$\% \text{ Moisture} = \frac{(\text{wt. undried sample + pan}) - (\text{wt. dried sample + pan})}{(\text{wt. undried sample + pan}) - (\text{wt. pan})} \times 100$$

$$\text{PPM} = \frac{(\text{peak ht. sample}) (\eta\text{g std. injected}) (\text{sample final volume, mL}) (100)}{(\text{peak ht. std.}) (\mu\text{L sample injected}) (\text{g sample}) (100 - \% \text{ moisture})}$$

$$\mu\text{g} = \frac{(\text{peak ht. sample}) (\eta\text{g std. injected}) (\text{sample final volume, mL})}{(\text{peak ht. std.}) (\mu\text{L sample injected})}$$

### VALIDATION

Validation of the above described method was made by spiking background soil samples with 10, 100, 1000  $\mu$ g of Diazinon and Diazinon OA each. The spiked samples were tumbled at 40 rpm for 10 minutes before extracting with ethyl acetate. Recoveries are listed in the Table below:

#### Recovery of Diazinon and Diazinon OA

Spike Level (n=3)	% Recovery (Avg)		SD		CV	
	Diazinon	Diazinon OA	Diazinon	Diazinon OA	Diazinon	Diazinon OA
10 ng	100	90	0.82	0.67	8.0	5.1
100 ng	99	106	1.50	3.00	1.6	2.8
1000 ng	106	89	63	28	5.9	3.2

## DISCUSSION

For quality control purposes, a background soil (blank) and a spiked background soil sample are to be run with each set of samples.

A spike sample is prepared by adding 1.00 mL standard of Diazinon and 1.00 mL standard of Diazinon OA, each containing 10 ng/ $\lambda$ , separately to a 50.0 g of background soil. Spiked samples are treated in similar manner as a normal sample.

The minimum detectable level was 0.25  $\mu$ g for both Diazinon and Diazinon OA in 50 g playground soil, with a S/N = 4.

## REFERENCE

- 1). Quan, V., Diazinon in Soil and Turf, 1984 Worker Health and Safety Methods, California Department of Food and Agriculture.
- 2). Schlocker, P. L., Wilder Ranch, Miscellaneous Organophosphate Pesticides in Soil, 1983 Environmental Monitoring Methods, California Department of Food and Agriculture.
- 3). White, Jane. Parathion on Kimbies, 1989 Environmental Monitoring Methods, California Department of Food and Agriculture.

WRITTEN BY:

*Paul Lee*

TITLE:

*Agricultural Chemist IV*

REVIEWED BY: Catherine Cooper

*Catherine Cooper*

TITLE: Agriculture Chemist III

**CALIFORNIA DEPT. OF FOOD & AGRIC.  
ENVIRONMENTAL MONITORING SECTION  
CHEMISTRY LABORATORY SERVICES**

3292 Meadowview Road  
Sacramento, CA 95832  
(916) - 262-2080  
FAX (916) - 262-2082

Original Date: Aug. 27, 1992  
Supercedes: New  
Current Date: Sept. 21, 1993  
Method No.:

## **Diazinon and Diazinon OA Residues in Turf**

### **SCOPE**

This method is for the analysis of dislodgeable and penetrated Diazinon and Diazinon OA residues in residential turf.

### **PRINCIPLE**

Dislodgeable Diazinon and Diazinon OA residues are removed from turf with water aided by addition of a surfactant. Pesticides presented in aqueous solution are then extracted with ethyl acetate.

Penetrated Diazinon and Diazinon OA are separated from turf by blending with ethyl acetate and by filtration.

The resulting extract is concentrated to a known volume. Diazinon and Diazinon OA are subsequently determined using gas chromatography with flame photometric detector.

### **REAGENT AND EQUIPMENT**

1. Ethyl Acetate , pesticide grade.
2. Sodium Chloride, granular, reagent grade.
3. Sodium Sulfate, anhydrous, granular , for residue analysis.
4. Distilled water.
5. 2% Surten solution. Dilute 1 g of Aerosol OT 75% Aqueous with 50 mL of distilled water.  
American Cyanamid Co., Wayne, N. J..
6. Balance. Mettler PL 4400. Mettler Instrument Corp., Hightstown, N.J..
7. Waring Commerical Laboratory Blendor with stainless steel cup (1 qt). Waring Products  
Division, Dynamics Corp. of America, New Hartford, Conn..
8. Centrifuge tube with screw cap, 250-mL.
9. Separatory funnel, 1-L.
10. Graduated cylinder, 200-mL.
11. Graduated conical centrifuge tube, 15-mL.
12. Funnel, short stem, 3-4 inch diameter.
13. Whatman # 4 filter paper, 18.5-cm.

14. Stainless steel forceps, teaspoon, and spatula.
15. Roller-tumbler capable of 60 rpm.
16. Rotary evaporator. Buchi/Brinkmann, Model R110.
17. Meyers N-EVAP. Organomation Associates Incorporated, Northborough, MA.
18. Thermolyne Vortex Maxi Mixer II. Sybron Corp., Dubuque, PA.

## PROCEDURE

### A. Dislodgeable Residues

- 1). Transfer about 5 g of turf sample to a dry, tared aluminium weighing pan and weigh to  $\pm 0.001$  g. Place the sample in an oven maintained at  $105^{\circ}$  C for a minimum of 6 hours. Cool in a dessicator and weigh. Calculate the moisture content of the sample by weight difference.
- 2). Weigh jar, without lid, containing the remaining turf sample to  $\pm 0.01$ g (usually 20-25 g).
- 3). Add 150 mL of distilled water and 6 drops of Surten solution. Rotate sample for 1 hour at 30 rpm.
- 4). Decant wash solution into a 250-ml centrifuge tube and centrifuge for 10 min. at about 1300 rpm.
- 5). Decant the clear liquid through a funnel containing a plug of glass wool into a 1-L separatory funnel.
- 6). Repeat steps 3-5 twice, using 100 mL of distilled water and 4 drops of Surten solution. Rotate sample for 30 min. at 30 rpm. After decanting the third wash into the centrifuge tube, add 50 mL of distilled water to the sample jar and shake for 30 seconds. Decant and combine with third wash. Centrifuge and decant liquid into separatory funnel. Save solids in centrifuge bottle and turf in jar for "penetrated residue" extraction (Section C).
- 7). Proceed to solvent extraction of dislodgeable residues.(Section B).

### B. Solvent Extraction of Dislodgeable Residues

- 1). Add 30 g NaCl to separatory funnel and shake until the salt dissolves.
- 2). Add 200 mL of ethyl acetate and shake gently for 2 minutes.
- 3). After the two layers separate, drain the water into an 1-L brown bottle or a 600-mL beaker. Add 3-5 g of NaCl into the separatory funnel and shake gently for about 20 seconds. Let it stand for about 1 min. Pour the ethyl acetate through the top of separatory funnel onto a short stem funnel filled  $\frac{2}{3}$  full with anhydrous  $\text{Na}_2\text{SO}_4$ . Collect the liquid in a 500-mL boiling flask.
- 4). Transfer the aqueous portion from previous extraction back to the separatory funnel and repeat the extraction as described in steps 2-3 twice with 100 mL of ethyl acetate.

- 5). Concentrate the extract by rotary evaporation at 40-45° C and 23 inches of Hg to 4-6 mL..
- 6). Quantitatively transfer the concentrate into a 15-mL graduated test tube. Rinse the flask twice with 2-3 mL of ethyl acetate and transfer the rinse liquid into the graduated test tube.
- 7). If needed, evaporate to slightly less than 10 ml on a N-EVAP purging with nitrogen at 40-45° C. Cool, dilute to 10.0 mL and mix thoroughly using a Vortex mixer. Transfer a portion of sample into an autosampler vial for GLC analysis.
- 8). Refrigerate excess concentrated extract until the analysis is satisfactorily completed.

C. Penetrated Residues

- 1). Using a stainless steel spatula, transfer the entire turf sample in the sample jar to a blender cup. Rinse jar twice with 25 mL of ethyl acetate and pour rinses into blender cup. Transfer residue remaining in corresponding centrifuge tube from dislodgeable extraction to the blender cup using 50 mL of ethyl acetate.
- 2). Weigh the empty jar, without lid, to  $\pm 0.01$  g. Calculate the weight of turf used for the determination by weight difference.
- 3). Add 100 mL of ethyl acetate and 50 g of anhydrous sodium sulfate and blend at medium speed (50 - 60% maximum) for 1 min. follow by another minute of blending at high speed (80% of maximum).
- 4). Let sample stand for 1 minute.
- 5). Decant extract through a funnel lined with a filter paper containing 100 g of  $\text{Na}_2\text{SO}_4$  into a 500-mL boiling flask.
- 6). Add 100 mL of ethyl acetate and blend for 2 min. at high speed. Filter extract through  $\text{Na}_2\text{SO}_4$  into the boiling flask.
- 7). Rinse blender cup twice with 25 mL of ethyl acetate and filter. Rinse  $\text{Na}_2\text{SO}_4$ , on funnel, with 30 mL of ethyl acetate.
- 8). Rotary evaporate extract at 40-45° C and at 23-24 inches of Hg to about 4-6 mL.
- 9). Quantitatively transfer concentrated extract to a 15-mL graduated test tube. Rinse side of flask twice with 2-3 mL of ethyl acetate and transfer the rinse liquid to the test tube. If needed, evaporate to slightly less than 10 mL on a N-EVAP purging with nitrogen at 40-45° C. Cool, dilute to 10.0 mL and mix thoroughly. Transfer a portion of the sample to an autosampler vial for GLC analysis.
- 10). Refrigerate excess concentrated extract until the analysis is satisfactorily completed.

## DETERMINATION

### A. GLC Conditions

Hewlett Packard 5890 II equipped with a Flame Photometric Detector (phosphorus mode)

Hewlett Packard 7673 auto liquid sampler

Injector: Splitless; 220° C

Detector: 250° C

Temperature Program:

Initial Temperature: 150° C for 1 minute

Program Rate: 10° C/ min.

Final Temperature. 200° C for 2 minutes.

Air: 100 mL/min.

Hydrogen: 75 mL/min.

Column: Hewlett Packard HP-1 (100% dimethyl polysiloxane)

10 m x 0.53 mm x 2.65 micron.

Carrier Gas: Helium

Flow Rate: 12 mL/min.

Septum Purge: 2mL/min.

Make-up: 5mL/min.

Injector Volume: 2 µL

Linearity Check: 0.2 ng - 20 ng

### B. Calculations

Report data in ppm or in µg.

$$\% \text{ Moisture} = \frac{(\text{wt. undried sample + pan}) - (\text{wt. dried sample + pan})}{(\text{wt. undried sample + pan}) - (\text{wt. pan})} \times 100$$

$$\text{PPM} = \frac{(\text{peak ht. sample}) (\text{ng std. injected}) (\text{sample final volume, mL}) (100)}{(\text{peak ht. std}) (\mu\text{L sample injected}) (\text{g sample}) (100 - \% \text{ moisture})}$$

$$\mu\text{g} = \frac{(\text{peak ht. sample}) (\text{ng std. injected}) (\text{sample final volume, mL})}{(\text{peak ht. std.}) (\mu\text{L sample injected})}$$

## VALIDATION

Validation of the above described method was made by spiking background turf samples to contain 0.1, 0.5 and 1 ng/uL of Diazinon and Diazinon OA. Spiked samples were run at the beginning, after every 10 samples and at the end of a set of normal turf samples.

Recovery of Diazinon and Diazinon OA.

Spike Level (n = 3)	% Recovery (Avg)		SD		CV	
	Diazinon	Diazinon OA	Diazinon	Diazinon OA	Diazinon	Diazinon OA
10 ng	100	90	0.82	0.67	8.3	5.0
100 ng	99	106	1.6	3.0	1.6	2.8
1000 ng	107	90	63	28	5.9	3.2

## DISCUSSION

For quality control purposes, a blank and a spiked sample are to be run with each set of samples. A 1.00 mL standard of Diazinon and 1.00 mL of Diazinon OA, each containing 10 ng/λ, is added separately to the water wash containing dislodgeable residues and again to the washed turf for pentatrated residues. Spiked samples are treated in similar manner as a normal sample.

The sharpness of the blender blade should be checked frequently so that complete homogenation of turf is accomplished.

The minimum detectable level was 0.25 ug for both Diazinon and Diazinon OA in turf.

## REFERENCE

- 1). Quan, V., Diazinon in Soil and Turf, 1984, Worker Health and Safety Methods, California Department of Food and Agriculture.
- 2). Schlocker, P. L., Wilder Ranch - Miscellaneous Organophosphate Pesticides in Soil, 1983, Environmental Monitoring Methods, California Department of Food and Agriculture .
- 3). White, Jane, Parathion on Kimbies, 1989, Environmental Monitoring Methods, California Department of Food and Agriculture.

WRITTEN BY:

*Paul Lee*

REVIEWED BY: Catherine Cooper

*Catherine Cooper*

TITLE:

*Agricultural Chemist III*

TITLE: Agricultural Chemist III

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Subject or Title:  
**DETERMINATION OF DIAZINON IN SOIL BY GAS CHROMATOGRAPHY  
WITH THERMIONIC SPECIFIC DETECTOR**

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**LM-CAL-8009**

Revision Number  
**ORIGINAL**

Supersedes: **NONE**

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### 1. SCOPE AND APPLICATION

1.1 This method applies to the determination of diazinon in soil by gas chromatography with a thermionic specific detector (GC-TSD), a detector specifically for nitrogen- and phosphorous-containing compounds.

1.2 Linearity is established with standards between 1.0 ug/mL to 0.050 ug/mL, but this will depend on the condition of the TSD bead. With the extract's final concentration of 1 g/mL, the detection limit is approximately 50 ug/Kg (ppb) and the reporting limit is 100 ppb.

Prepared By:  
**Dennis E. Gall**

Date:  
**February 26, 1993**

Management Approval:

*Karla L. Bruehler*

Date:  
**3/1/93**

QA Officer Approval:

*Vercella J. Erickson*

Date:  
**3-1-93**

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- 1.2.1 The standard range for diazinon depends on the condition of the TSD bead and affects the linearity; therefore, the range may need to be re-established to choose the appropriate five points.
  - 1.2.2 Refer to the CDPR's Analytical Laboratory Specification for their required reporting limits and the required fortification levels for their quality control.
  - 1.2.3 For a lower reporting limit, the final extract concentration can be increased to as high as 20 g/mL but this depends on the amount of interferences from the matrix, solvents, and/or reagents.
- 1.3 Analysis can typically be achieved in under 30 minutes, depending on the column type and the amount of interferences that chromatograph around the time of the analyte of interest.
- 1.4 NAME/CAS/LD<sub>50</sub><sup>1</sup>
- Diazinon  
O,O-Diethyl-O-[2-isopropyl-4-methyl-6-pyrimidyl]phosphorothioate
- CAS: 333-41-5
- LD<sub>50</sub>: 300 mg/Kg

## 2. METHOD SUMMARY

Diazinon is extracted from soil with methylene chloride:acetone (75:25, v/v) by the 8-oz shake method. The extracts are filtered, dried over sodium sulfate, and concentrated by KD. A portion of the concentrate from the KD is aliquoted and the solvent is exchanged to toluene under gentle stream of nitrogen. The extracts are analyzed by a gas chromatograph equipped with a thermionic specific detector (GC/TSD).

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### 3. COMMENTS

- 3.1 Extraction and analysis should be conducted by trained personnel.
- 3.2 Personnel must be familiar with Enseco policies for documentation and record keeping, and general laboratory safety procedures.
- 3.3 All glassware must be rinsed with three solvents as usual (acetone, hexane, and methylene chloride).
- 3.4 The analyst is responsible for the following areas:
  - 3.4.1 Adhering to the method without deviation. Any modification to the method can only be done with the written approval of the supervisor and documented in a logbook or on a mastersheet.
  - 3.4.2 Complete and accurate documentation for a particular project.
  - 3.4.3 Documentation of any anomalies that were encountered during the extraction procedure.

### 4. SAFETY ISSUES

- 4.1 This method utilizes chemicals that are dangerous and hazardous. The appropriate protective gear such as safety glasses, lab coat, and gloves should be worn, and all lab procedures should be carried out in a fume hood to maximize protection to yourself and others.
- 4.2 A Material Safety Data Sheet (MSDS), if available for each laboratory standard and reagent chemical, must be reviewed before handling the chemical(s).
- 4.3 Personnel should be familiar with the location of solvent spill kits and with the location and use of fire extinguishers, eye wash station and emergency shower.

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## 5. **SAMPLE COLLECTION AND PRESERVATION**

- 5.1 Samples are collected in glass jars, sealed with teflon-lined screw caps. Other liners such as aluminum foil are acceptable.
- 5.2 No preservative is necessary.
- 5.3 Samples are refrigerated and stored at 2 to 6 °C until extraction. Holding time is generally fourteen days from sampling date, and the analysis due date is generally 30 days from extraction.

## 6. **APPARATUS**

NOTE: The following apparatus are suggested items used in the laboratory. Alternate items may be substituted.

- 6.1 Analytical balance, Electronic top-loading, readable to 0.0001 grams; For preparation of analytical standards.
- 6.2 Balance, Electronic top-loading, readable to 0.1 grams and a capacity up to ~ 1000 grams.
- 6.3 Bottle (or jar), 8-oz, 4-oz, and 2-oz, amber glass, with teflon-lined screw cap.
- 6.4 Bottle, 8-oz, glass, french square, with teflon-lined screw cap.
- 6.5 Filter paper, Whatman 1, 18.5 cm.
- 6.6 Fiber glass (glass wool), sliver 8 micron, Corning 3950.
- 6.7 Filtering funnel, 100-mm diameter, glass, stemless or short stem.
- 6.8 Flask, volumetric, 10-mL, 25-mL, 50-mL, 100-mL.

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6.9 Kuderna-Danish concentrator.

6.9.1 Reservoir flask, 500-mL.

6.9.2 Concentrator tube, 10-mL.

6.9.3 Synder column, 3-ball.

6.10 Nitrogen gas manifold, N-Evaporator, Organomation Analytical Associates model 112.

6.11 Pipet, pasteur type, glass, 5<sup>3</sup>/<sub>4</sub> or 9 inch long.

6.12 Shaker, orbital, Lab-Line model 3520.

6.13 Test tube, disposable clear glass, 13-mm X 100-mm (8-mL) and 16-mm X 125-mm (15-mL), with teflon-lined screw cap, 13 X 415 and 15 X 415 respectively.

6.14 Vial, 4-mL, clear glass or amber glass, with teflon-lined screw cap.

6.15 Water bath, temperature-controlled at 80-90 °C.

## 7. REAGENTS AND STANDARDS

### 7.1 REAGENTS-

7.1.1 Acetone, pesticide quality.

7.1.2 Methylene chloride, pesticide quality.

7.1.3 Methylene chloride:Acetone (75:25, v/v); prepared by mixing 3000 mL of methylene chloride with 1000 mL of acetone.

7.1.4 Sodium sulfate, anhydrous, analytical reagent grade; pre-rinse with methylene chloride before using.

7.1.4.1 Sodium sulfate can be cleaned by heating in a kiln up to 450 °C for approximately 4 hours, cooled, and bottled before using.

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7.1.5 Toluene, pesticide quality.

7.2 STANDARDS-Diazinon

7.2.1 Preparation of Stock solution from Neat.

7.2.1.1 Prepare a stock solution by weighing the analytical standard in an amber glass bottle and dilute with acetone to achieve a final concentration of 10 mg/mL (or 1.0 mg/mL or 0.10 mg/mL, whichever is appropriate). Store the solution in the amber glass bottle in a refrigerator and replace the solution every twelve months or sooner.

7.2.2 Preparation of Fortification Solutions.

7.2.2.1 Prepare a 50 ug/mL standard in acetone using the stock solution from 7.2.1.1. Store the solution in an amber glass bottle and replace the solution every six months or sooner.

7.2.2.2 From the 50 ug/mL standard in 7.2.2.1, prepare a 5.0 ug/mL standard and a 0.50 ug/mL standard in acetone. Store the solutions in amber glass bottles and replace the solutions every six months or sooner.

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### 7.2.3 Preparation of Analytical Standards.

NOTE: The standard range for diazinon depends on the condition of the TSD bead and affects the linearity; therefore, the range may need to be re-established to choose the appropriate five points.

7.2.3.1 Using the 50 ug/mL standard in 7.2.2.1, prepare 1,000 ng/mL, 800 ng/mL, and 500 ng/mL standards in toluene. Store the solutions in amber glass bottles and replace the solutions every six months or sooner.

7.2.3.2 Using the 5.0 ug/mL standard in 7.2.2.2, prepare 300 ng/mL, 200 ng/mL, 100 ng/mL, and 50 ng/mL standards in toluene. Store the solutions in amber glass bottles and replace the solutions every six months or sooner.

### 7.3 Reference Standards

7.3.1 Prepare reference standards to verify the quality of the fortification solutions and analytical standards in 7.2.2 and 7.2.3.

7.3.2 A reference standard is any standard solution made from a source other than the stock standard. Reference standards may be from the EPA, the manufacturer, or from another reliable source.

7.3.3 If the secondary source is not available, a separate intermediate stock solution will be made from the same neat by another chemist or from a neat with a different lot number.

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## 8. PROCEDURE

8.1 If applicable, determine the moisture content of the soil samples by following SOP LM-CAL-3055.

8.2 Sample Preparation.

8.2.1 Label the Cal ID with the suffix 'DIAZ-S'.

8.2.2 Weigh 20 grams of soil into an 8-oz glass jar; and record the mass to the nearest 0.1 grams.

8.2.3 If applicable, add the appropriate spiking solutions to the prepared samples to yield the appropriate fortification level:

8.2.3.1 For a 0.10 ppm level, add 400  $\mu$ L of the 5.0  $\mu$ g/mL standard to a 20 gram sample.

8.2.3.2 For a 0.50 ppm level, add 200  $\mu$ L of the 50  $\mu$ g/mL standard to a 20 gram sample.

8.2.3.3 For a 1.0 ppm level, add 400  $\mu$ L of the 50  $\mu$ g/mL standard to a 20 gram sample.

8.2.3.4 For a 10 ppm level, add 4.0 mL of the 50  $\mu$ g/mL standard to a 20 gram sample.

8.2.3.5 For other specified levels, add the appropriate amount of the fortification standard to obtain the proper level; do not add more than 5.0 mL or less than 50  $\mu$ L.

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### 8.3 Sample Extraction.

- 8.3.1 Add 100 mL of 75:25 methylene chloride-acetone and 50 grams of cleaned (or kilned) sodium sulfate, seal the jar with a teflon-lined screw cap, and briefly shake well.
- 8.3.2 Position and secure the jars horizontally on the orbital shaker, and check the samples to ensure that they do not leak. If samples are ready, then turn on the shaker to shake the samples for 60 minutes at approximately 210 to 250 rpm.
- 8.3.3 After shaken, set the jars upright and shake briefly to resettle the sediments to the bottom of the jar.
- 8.3.4 Decant the extract with minimal agitation of the sediments through Whatman 1 paper into an 8-oz amber glass bottle (or 250-ml erlenmeyer flask).
- 8.3.5 Add another 100 mL of methylene chloride-acetone to the residual soil in the jar, re-seal the jar, and shake briefly.
- 8.3.6 Repeat steps 8.3.2 and 8.3.3, and shake the samples for 30 minutes.
- 8.3.7 Decant the extract as in the above step 8.3.4.
- 8.3.8 When the final extracts have passed through the paper filter, dump the soil sample onto the filter paper. Rinse the glass jar and the remaining soil with an additional 20 to 40 mL of methylene chloride, and pass through the filter paper into the bottle.
- 8.3.9 If applicable, this is a good stopping point. Store the extracts at room temperature (or at 6°C or lower for long storage), or continue on to the next step.

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#### 8.4 Concentration

8.4.1 Pass the methylene chloride-acetone extract through approximately 50 grams (3 heaping teaspoons) of anhydrous sodium sulfate (pre-rinsed with methylene chloride) into the KD apparatus.

8.4.1.1 Rinse the glass bottle well with two 40 ml portions of methylene chloride and use this and an additional 30 mL of methylene chloride to rinse the sodium sulfate into the KD flask.

8.4.2 Add three or four small teflon boiling chips, and attach the macro-snyder column.

8.4.3 Carefully concentrate the extract to approximately 5 mL on the steam bath at 80-90° C, and allow to cool for a minimum of 15 minutes.

8.4.4 Carefully detach the concentrator tube and quantitatively transfer the extract to a 15-mL test tube with methylene chloride rinse.

8.4.5 Adjust the final volume to 10.0 mL with methylene chloride. Cap and mix well. The concentration of the extract is 20g/10mL.

#### 8.5 Aliquot

8.5.1 Aliquot the appropriate amount to a 4.0-mL vial or to an appropriate size vial or test tube.

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8.5.1.1 The appropriate amount to aliquot depends on the required reporting limit and the standard range of the analytical standards.

For example: If the low end of the range is 50 ng/mL and the required reporting limit is 0.10 ppm (the detection limit should be 50 to 80 % of the reporting limit) then the appropriate final sample concentration is 1 gram/mL (=4g/4mL). Aliquot 2.0 mL to a 4.0 mL vial, exchange the solvent and adjust the final volume to 4.0 mL.

8.5.2 Reduce the extracts under nitrogen and solvent exchange to toluene several times with the water bath at 35 degrees or lower. Adjust the final volume with toluene.

8.5.3 Organize the extracts in a rack, and store in the refrigerator at 2-6 °C until

8.6 Instrument Operating Conditions.

8.6.1 Analysis is conducted using a gas chromatograph equipped with a nitrogen-phosphorous specific detector.

8.6.1.1 Varian 3700 or 3400 gas chromatograph with a thermionic specific detector (nitrogen/phosphorous specific detector) and a Varian 402 or 654 data system, or equivalent.

8.6.2 Analytical Conditions

8.6.2.1 A 30-m X 0.53-mm i.d. J&W DB-5 megabore column (5% phenyl, 95% methyl silicone), 1.5 um film thickness. J&W Scientific P/N 125-5032.

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8.6.2.2 A confirmation column may be used (or it may be substituted as the analytical column) such as:

30-m X 0.53-mm id DB-210 megabore column (50% Trifluoropropyl-50% Methyl), 1.0  $\mu$ m thickness. J&W Scientific P/N 125-0232.

or,

30-m X 0.53-mm id DB 1701 megabore column (14% Cyanopropylphenyl-86% Methyl), 1.0  $\mu$ m thickness. J&W Scientific P/N 125-0732.

or,

30-m X 0.53-mm id DB-608 megabore column, 0.83  $\mu$ m thickness. J&W Scientific P/N 125-1730.

8.6.2.3 Temperature settings:

Injector: 220 °C.

Detector: 300 °C.

Column: (temperature programmed)

Initial Temp: 140 °C.

Initial Hold: 2 minutes

Program Rate: 10 °/minute

Final Temp: 250 °C.

Final Hold: 2 to 7 minutes

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**8.6.2.4 Gas Flows (approximate values):**

Helium Carrier: 7.5 mL/min.

Nitrogen Make-up: 22.5 mL/min.

Detector Hydrogen: 4 mL/min.

Detector Air: 170 mL/min.

**8.6.2.5 Injection Volume: 5 uL**

**8.6.2.6 Retention times (approximated):**

Diazinon on DB-5 is 9.6 minutes

Note: If a change to the GC conditions is necessary, it will be noted on the GC chromatogram.

**8.6.3 Interferences**

**8.6.3.1** The temperature program of the gas chromatograph may be modified to separate or resolve the interferences from the compound of interest (Diazinon).

**8.6.4 Instrument calibration**

**8.6.4.1** All standard concentrations should be injected at least twice in the injection set.

**8.6.4.2** The standard curve is derived from all standards injected with the analysis set using a least squares fit.

**8.6.4.3** Standards are injected after every 1 to 5 samples and at the conclusion of the day's analysis.

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## 9. REPORTING REQUIREMENTS

### 9.1 Instrumentation.

9.1.1 The standard curve is derived from all standards injected with the analysis set using a least squares fit.

### 9.2 LIMS

9.2.1 LIMS is used for entering the extraction date and release date of the prep, and the analysis date and release date of the data sheet,

### 9.3 Data package.

9.3.1 Follow client's specific requirement, or the guidelines specified in the protocol.

9.3.2 See the client manager for requirements.

## 10. QA/QC REQUIREMENTS

10.1 The method blank is mandatory and is performed for each type of matrix used and for every 20 samples, or as per protocol specifications.

10.2 The matrix spike and the matrix spike duplicate are optional and must be requested. They are performed for each matrix and for every 20 samples, or as per protocol specifications.

10.2.1 Spike the MS/MSD with the appropriate standards to give the proper levels specified in the protocol. The minimum volume required for fortification is 50 uL and the maximum volume is 5.0 mL. It may be necessary to prepare a spiking solution at an appropriate concentration to meet this criteria. See section 8.2.3 for fortification solution information.

10.3 The minimum volume for sample dilution for final quantitation is 0.10 mL.

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## 11. CALCULATIONS

11.1 Enter the data manually in Graphic Outlook. See SOP Number LP-CAL-4008 as a guideline.

11.1.1 Use file name DIAZ0392.

11.2 Resultant values on the Graphic Outlook gives the amount of diazinon found from diazinon data entered.

11.2.1 If the data is acceptable, enter the results on the mastersheet and into LIMS.

## 12. REFERENCE

- 1 Chem Service, "Pesticides and Metabolites," 1991-1992 Catalog, 660 Tower Lane, PO Box 3108, West Chester, PA 19381-3108, p. 69.

**Appendix B - Duarte, California - Diazinon Field Data Results**

Soil Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DIAZINON			DIAZOXON		
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2
Applic 1	Day -1	1	1	9/27/92	427	0.0015 ND	0.0016 ND	0.0025 ND	0.0015 ND	0.0016 ND	0.0025 ND
Applic 1	Day -1	1	2	9/27/92	428	0.0015 ND	0.0016 ND	0.0032 ND	0.0015 ND	0.0016 ND	0.0032 ND
Applic 1	Day -1	2	1	9/27/92	431	0.0015 ND	0.0018 ND	0.0026 ND	0.0015 ND	0.0018 ND	0.0026 ND
Applic 1	Day -1	2	2	9/27/92	432	0.0015 ND	0.0016 ND	0.0025 ND	0.0015 ND	0.0016 ND	0.0025 ND
Applic 1	Day -1	3	1	9/27/92	433	0.0015 ND	0.0017 ND	0.0030 ND	0.0015 ND	0.0017 ND	0.0030 ND
Applic 1	Day -1	3	2	9/27/92	434	0.0015 ND	0.0019 ND	0.0031 ND	0.0015 ND	0.0019 ND	0.0031 ND
Applic 1	Day -1	4	1	9/27/92	436	0.0015 ND	0.0017 ND	0.0028 ND	0.0015 ND	0.0017 ND	0.0028 ND
Applic 1	Day -1	4	2	9/27/92	435	0.0015 ND	0.0016 ND	0.0036 ND	0.0015 ND	0.0016 ND	0.0036 ND
Applic 1	Day 0	1	1	9/29/92	503	18.4574	20.9910	59.6271	0.0162	0.0184	0.0523
Applic 1	Day 0	1	2	9/29/92	504	13.1108	15.1063	31.8043	0.0100	0.0115	0.0243
Applic 1	Day 0	2	1	9/29/92	475	21.8632	27.5320	48.9563	0.0170	0.0214	0.0381
Applic 1	Day 0	2	2	9/29/92	476	27.7394	33.4049	49.1739	0.0194	0.0234	0.0344
Applic 1	Day 0	3	1	9/29/92	510	6.7504	7.8832	14.0921	0.0048	0.0056	0.0100
Applic 1	Day 0	3	2	9/29/92	509	15.8456	18.8750	32.5248	0.0120	0.0143	0.0246
Applic 1	Day 0	4	1	9/29/92	507	5.3232	6.0061	11.7956	0.0015 ND	0.0017 ND	0.0033 ND
Applic 1	Day 0	4	2	9/29/92	508	7.2058	8.1680	13.6142	0.0068	0.0077	0.0128
Applic 2	Day -1	1	1	10/13/92	629	4.8216	4.9376	9.6579	0.0015 ND	0.0015 ND	0.0030 ND
Applic 2	Day -1	1	2	10/13/92	630	13.3662	14.0712	23.1488	0.0015 ND	0.0016 ND	0.0026 ND
Applic 2	Day -1	2	1	10/13/92	621	10.4946	12.0172	10.9237	0.0015 ND	0.0017 ND	0.0016 ND
Applic 2	Day -1	2	2	10/13/92	622	15.1000	16.9644	14.9249	0.0015 ND	0.0017 ND	0.0015 ND
Applic 2	Day -1	3	1	10/13/92	624	3.4814	3.7422	3.9587	0.0015 ND	0.0016 ND	0.0017 ND
Applic 2	Day -1	3	2	10/13/92	623	7.6622	8.3804	7.8414	0.0015 ND	0.0016 ND	0.0015 ND
Applic 2	Day -1	4	1	10/13/92	619	0.9962	1.0409	1.1763	0.0015 ND	0.0016 ND	0.0018 ND
Applic 2	Day -1	4	2	10/13/92	620	0.9416	0.9759	1.5896	0.0015 ND	0.0016 ND	0.0025 ND
Applic 2	Day 0	1	1	10/13/92	603	26.1314	32.0042	41.5996	0.0015 ND	0.0018 ND	0.0024 ND
Applic 2	Day 0	1	2	10/13/92	604	33.4824	40.1853	44.8088	0.0015 ND	0.0018 ND	0.0020 ND
Applic 2	Day 0	2	1	10/13/92	597	19.8076	23.9425	47.3565	0.0015 ND	0.0018 ND	0.0036 ND
Applic 2	Day 0	2	2	10/13/92	598	15.1732	19.4453	35.5686	0.0015 ND	0.0019 ND	0.0035 ND
Applic 2	Day 0	3	1	10/13/92	599	13.2416	15.6133	29.3419	0.0015 ND	0.0018 ND	0.0033 ND
Applic 2	Day 0	3	2	10/13/92	600	9.2322	11.0738	13.4050	0.0015 ND	0.0018 ND	0.0022 ND
Applic 2	Day 0	4	1	10/13/92	595	0.2698	0.3519	0.5538	0.0015 ND	0.0020 ND	0.0031 ND
Applic 2	Day 0	4	2	10/13/92	596	0.5004	0.5789	1.0067	0.0015 ND	0.0017 ND	0.0030 ND
Applic 3	Day -1	1	1	10/27/92	555	0.5826	0.6085	1.5967	0.0015 ND	0.0016 ND	0.0041 ND
Applic 3	Day -1	1	2	10/27/92	556	1.8816	1.9886	3.6208	0.0015 ND	0.0016 ND	0.0029 ND
Applic 3	Day -1	2	1	10/27/92	549	0.5120	0.5769	0.3977	0.0015 ND	0.0017 ND	0.0012 ND
Applic 3	Day -1	2	2	10/27/92	550	0.8740	0.9820	0.8073	0.0015 ND	0.0017 ND	0.0014 ND
Applic 3	Day -1	3	1	10/27/92	487	0.5500	0.5732	0.5542	0.0015 ND	0.0016 ND	0.0015 ND
Applic 3	Day -1	3	2	10/27/92	552	0.7764	0.8137	1.0214	0.0015 ND	0.0016 ND	0.0020 ND
Applic 3	Day -1	4	1	10/27/92	547	0.2366	0.2675	0.3477	0.0015 ND	0.0017 ND	0.0022 ND

ND = Not detected. Data reported are based upon calculations using one-half the detection limit for diazinon and diazoxon.

Soil Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DIAZINON			DIAZOXON		
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2
Applic 1	Day -1	1	1	9/27/92	427	0.0015 ND	0.0016 ND	0.0025 ND	0.0015 ND	0.0016 ND	0.0025 ND
Applic 1	Day -1	1	2	9/27/92	428	0.0015 ND	0.0016 ND	0.0032 ND	0.0015 ND	0.0016 ND	0.0032 ND
Applic 1	Day -1	2	1	9/27/92	431	0.0015 ND	0.0018 ND	0.0026 ND	0.0015 ND	0.0018 ND	0.0026 ND
Applic 1	Day -1	2	2	9/27/92	432	0.0015 ND	0.0016 ND	0.0025 ND	0.0015 ND	0.0016 ND	0.0025 ND
Applic 1	Day -1	3	1	9/27/92	433	0.0015 ND	0.0017 ND	0.0030 ND	0.0015 ND	0.0017 ND	0.0030 ND
Applic 1	Day -1	3	2	9/27/92	434	0.0015 ND	0.0019 ND	0.0031 ND	0.0015 ND	0.0019 ND	0.0031 ND
Applic 1	Day -1	4	1	9/27/92	436	0.0015 ND	0.0017 ND	0.0028 ND	0.0015 ND	0.0017 ND	0.0028 ND
Applic 1	Day -1	4	2	9/27/92	435	0.0015 ND	0.0016 ND	0.0036 ND	0.0015 ND	0.0016 ND	0.0036 ND
Applic 1	Day 0	1	1	9/29/92	503	18.4574	20.9910	59.6271	0.0162	0.0184	0.0523
Applic 1	Day 0	1	2	9/29/92	504	13.1108	15.1063	31.8043	0.0100	0.0115	0.0243
Applic 1	Day 0	2	1	9/29/92	475	21.8632	27.5320	48.9563	0.0170	0.0214	0.0381
Applic 1	Day 0	2	2	9/29/92	476	27.7394	33.4049	49.1739	0.0194	0.0234	0.0344
Applic 1	Day 0	3	1	9/29/92	510	6.7504	7.8832	14.0921	0.0048	0.0056	0.0100
Applic 1	Day 0	3	2	9/29/92	509	15.8456	18.8750	32.5248	0.0120	0.0143	0.0246
Applic 1	Day 0	4	1	9/29/92	507	5.3232	6.0061	11.7956	0.0015 ND	0.0017 ND	0.0033 ND
Applic 1	Day 0	4	2	9/29/92	508	7.2058	8.1680	13.6142	0.0068	0.0077	0.0128
Applic 2	Day -1	1	1	10/13/92	629	4.8216	4.9376	9.6579	0.0015 ND	0.0015 ND	0.0030 ND
Applic 2	Day -1	1	2	10/13/92	630	13.3662	14.0712	23.1488	0.0015 ND	0.0016 ND	0.0026 ND
Applic 2	Day -1	2	1	10/13/92	621	10.4946	12.0172	10.9237	0.0015 ND	0.0017 ND	0.0016 ND
Applic 2	Day -1	2	2	10/13/92	622	15.1000	16.9644	14.9249	0.0015 ND	0.0017 ND	0.0015 ND
Applic 2	Day -1	3	1	10/13/92	624	3.4814	3.7422	3.9587	0.0015 ND	0.0016 ND	0.0017 ND
Applic 2	Day -1	3	2	10/13/92	623	7.6622	8.3804	7.8414	0.0015 ND	0.0016 ND	0.0015 ND
Applic 2	Day -1	4	1	10/13/92	619	0.9962	1.0409	1.1763	0.0015 ND	0.0016 ND	0.0018 ND
Applic 2	Day -1	4	2	10/13/92	620	0.9416	0.9759	1.5896	0.0015 ND	0.0016 ND	0.0025 ND
Applic 2	Day 0	1	1	10/13/92	603	26.1314	32.0042	41.5996	0.0015 ND	0.0018 ND	0.0024 ND
Applic 2	Day 0	1	2	10/13/92	604	33.4824	40.1853	44.8088	0.0015 ND	0.0018 ND	0.0020 ND
Applic 2	Day 0	2	1	10/13/92	597	19.8076	23.9425	47.3565	0.0015 ND	0.0018 ND	0.0036 ND
Applic 2	Day 0	2	2	10/13/92	598	15.1732	19.4453	35.5686	0.0015 ND	0.0019 ND	0.0035 ND
Applic 2	Day 0	3	1	10/13/92	599	13.2416	15.6133	29.3419	0.0015 ND	0.0018 ND	0.0033 ND
Applic 2	Day 0	3	2	10/13/92	600	9.2322	11.0738	13.4050	0.0015 ND	0.0018 ND	0.0022 ND
Applic 2	Day 0	4	1	10/13/92	595	0.2698	0.3519	0.5538	0.0015 ND	0.0020 ND	0.0031 ND
Applic 2	Day 0	4	2	10/13/92	596	0.5004	0.5789	1.0067	0.0015 ND	0.0017 ND	0.0030 ND
Applic 3	Day -1	1	1	10/27/92	555	0.5826	0.6085	1.5967	0.0015 ND	0.0016 ND	0.0041 ND
Applic 3	Day -1	1	2	10/27/92	556	1.8816	1.9886	3.6208	0.0015 ND	0.0016 ND	0.0029 ND
Applic 3	Day -1	2	1	10/27/92	549	0.5120	0.5769	0.3977	0.0015 ND	0.0017 ND	0.0012 ND
Applic 3	Day -1	2	2	10/27/92	550	0.8740	0.9820	0.8073	0.0015 ND	0.0017 ND	0.0014 ND
Applic 3	Day -1	3	1	10/27/92	487	0.5500	0.5732	0.5542	0.0015 ND	0.0016 ND	0.0015 ND
Applic 3	Day -1	3	2	10/27/92	552	0.7764	0.8137	1.0214	0.0015 ND	0.0016 ND	0.0020 ND
Applic 3	Day -1	4	1	10/27/92	547	0.2366	0.2675	0.3477	0.0015 ND	0.0017 ND	0.0022 ND

ND = Not detected. Data reported are based upon calculations using one-half the detection limit for diazinon and diazoxon.

Soil Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DIAZINON			DIAZOXON		
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2
Applic 3	Day -1	4	2	10/27/92	548	0.1914	0.2097	0.3036	0.0015 ND	0.0016 ND	0.0024 ND
Applic 3	Day 0	1	1	10/27/92	497	5.8194	6.5556	11.1645	0.0015 ND	0.0017 ND	0.0029 ND
Applic 3	Day 0	1	2	10/27/92	498	3.3328	3.8623	6.0636	0.0032	0.0037	0.0058
Applic 3	Day 0	2	1	10/27/92	491	0.7982	0.9897	0.9542	0.0015 ND	0.0019 ND	0.0018 ND
Applic 3	Day 0	2	2	10/27/92	492	0.9152	1.1690	1.2275	0.0015 ND	0.0019 ND	0.0020 ND
Applic 3	Day 0	3	1	10/27/92	493	0.6584	0.7722	1.3284	0.0015 ND	0.0018 ND	0.0030 ND
Applic 3	Day 0	3	2	10/27/92	494	0.8630	1.0085	1.2178	0.0015 ND	0.0018 ND	0.0021 ND
Applic 3	Day 0	4	1	10/27/92	489	0.7668	0.8896	1.6678	0.0015 ND	0.0017 ND	0.0033 ND
Applic 3	Day 0	4	2	10/27/92	490	1.9760	2.3119	3.7794	0.0015 ND	0.0018 ND	0.0029 ND
Applic 3	Day 1	1	1	10/28/92	413	1.0756	1.1938	2.2705	0.0015 ND	0.0017 ND	0.0032 ND
Applic 3	Day 1	1	2	10/28/92	414	1.5634	1.7927	2.4159	0.0015 ND	0.0017 ND	0.0023 ND
Applic 3	Day 1	2	1	10/28/92	409	0.5448	0.6350	0.8196	0.0015 ND	0.0017 ND	0.0023 ND
Applic 3	Day 1	2	2	10/28/92	410	1.0408	1.2039	1.2503	0.0015 ND	0.0017 ND	0.0018 ND
Applic 3	Day 1	3	1	10/28/92	407	0.9934	1.1129	1.6567	0.0015 ND	0.0017 ND	0.0025 ND
Applic 3	Day 1	3	2	10/28/92	408	1.2782	1.3931	1.6249	0.0015 ND	0.0016 ND	0.0019 ND
Applic 3	Day 1	4	1	10/28/92	405	1.1894	1.3103	1.7062	0.0015 ND	0.0017 ND	0.0022 ND
Applic 3	Day 1	4	2	10/28/92	406	1.8310	1.9688	3.1604	0.0015 ND	0.0016 ND	0.0026 ND
Applic 3	Day 2	1	1	10/29/92	469	1.0520	1.3089	1.7545	0.0015 ND	0.0019 ND	0.0025 ND
Applic 3	Day 2	1	2	10/29/92	470	0.7768	0.9652	1.3861	0.0015 ND	0.0019 ND	0.0027 ND
Applic 3	Day 2	2	1	10/29/92	467	0.4676	0.5710	0.7062	0.0015 ND	0.0018 ND	0.0023 ND
Applic 3	Day 2	2	2	10/29/92	468	0.4266	0.5074	0.7214	0.0015 ND	0.0018 ND	0.0025 ND
Applic 3	Day 2	3	1	10/29/92	465	0.4454	0.5122	0.6519	0.0015 ND	0.0017 ND	0.0022 ND
Applic 3	Day 2	3	2	10/29/92	466	0.5116	0.5880	0.7100	0.0015 ND	0.0017 ND	0.0021 ND
Applic 3	Day 2	4	1	10/29/92	463	2.1198	2.3152	3.0285	0.0015 ND	0.0016 ND	0.0021 ND
Applic 3	Day 2	4	2	10/29/92	464	0.6618	0.7394	1.1500	0.0015 ND	0.0017 ND	0.0026 ND
Applic 3	Day 3	1	1	10/30/92	531	0.2688	0.3612	0.5392	0.0015 ND	0.0020 ND	0.0030 ND
Applic 3	Day 3	1	2	10/30/92	532	0.5374	0.7127	0.9746	0.0015 ND	0.0020 ND	0.0027 ND
Applic 3	Day 3	2	1	10/30/92	525	0.1830	0.2562	0.2860	0.0015 ND	0.0021 ND	0.0023 ND
Applic 3	Day 3	2	2	10/30/92	526	0.3380	0.4822	0.5617	0.0015 ND	0.0021 ND	0.0025 ND
Applic 3	Day 3	3	1	10/30/92	527	0.1506	0.1894	0.2687	0.0015 ND	0.0019 ND	0.0027 ND
Applic 3	Day 3	3	2	10/30/92	528	0.1660	0.2167	0.2255	0.0015 ND	0.0020 ND	0.0020 ND
Applic 3	Day 3	4	1	10/30/92	523	0.2636	0.3164	0.4365	0.0015 ND	0.0018 ND	0.0025 ND
Applic 3	Day 3	4	2	10/30/92	524	0.6498	0.7974	1.2883	0.0015 ND	0.0018 ND	0.0030 ND
Applic 3	Day 8	1	1	11/4/92	423	0.5822	0.6430	1.0728	0.0015 ND	0.0017 ND	0.0028 ND
Applic 3	Day 8	1	2	11/4/92	424	0.6470	0.7384	1.1092	0.0015 ND	0.0017 ND	0.0026 ND
Applic 3	Day 8	2	1	11/4/92	419	0.1826	0.2255	0.2939	0.0015 ND	0.0019 ND	0.0024 ND
Applic 3	Day 8	2	2	11/4/92	420	0.2246	0.2596	0.3065	0.0015 ND	0.0017 ND	0.0020 ND
Applic 3	Day 8	3	1	11/4/92	417	0.2462	0.2625	0.4135	0.0015 ND	0.0016 ND	0.0025 ND
Applic 3	Day 8	3	2	11/4/92	418	0.2672	0.2919	0.3802	0.0015 ND	0.0016 ND	0.0021 ND

ND = Not detected. Data reported are based upon calculations using one-half the detection limit for diazinon and diazoxon.

Soil Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DIAZINON			DIAZOXON		
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2
Applic 3	Day 8	4	1	11/4/92	415	0.4538	0.4821	0.7748	0.0015 ND	0.0016 ND	0.0026 ND
Applic 3	Day 8	4	2	11/4/92	416	0.5686	0.5994	1.3528	0.0015 ND	0.0016 ND	0.0036 ND
Applic 3	Day 12	1	1	11/8/92	401	0.5426	0.5641	0.9049	0.0015 ND	0.0016 ND	0.0025 ND
Applic 3	Day 12	1	2	11/8/92	402	0.8098	0.8618	1.0767	0.0015 ND	0.0016 ND	0.0020 ND
Applic 3	Day 12	2	1	11/8/92	393	0.2938	0.3408	0.4523	0.0015 ND	0.0017 ND	0.0023 ND
Applic 3	Day 12	2	2	11/8/92	394	0.2724	0.3190	0.3304	0.0015 ND	0.0018 ND	0.0018 ND
Applic 3	Day 12	3	1	11/8/92	395	0.4746	0.4915	0.4982	0.0015 ND	0.0016 ND	0.0016 ND
Applic 3	Day 12	3	2	11/8/92	396	0.4620	0.4968	0.4162	0.0015 ND	0.0016 ND	0.0014 ND
Applic 3	Day 12	4	1	11/8/92	391	0.7758	0.8096	1.1762	0.0015 ND	0.0016 ND	0.0023 ND
Applic 3	Day 12	4	2	11/8/92	392	0.8186	0.8617	1.3366	0.0015 ND	0.0016 ND	0.0024 ND
Applic 3	Day 16	1	1	11/12/92	451	0.4700	0.4930	0.7619	0.0015 ND	0.0016 ND	0.0024 ND
Applic 3	Day 16	1	2	11/12/92	452	0.6020	0.6939	0.7442	0.0015 ND	0.0017 ND	0.0019 ND
Applic 3	Day 16	2	1	11/12/92	457	0.2280	0.2550	0.3590	0.0015 ND	0.0017 ND	0.0024 ND
Applic 3	Day 16	2	2	11/12/92	458	0.1536	0.1818	0.2132	0.0015 ND	0.0018 ND	0.0021 ND
Applic 3	Day 16	3	1	11/12/92	461	0.6076	0.6494	0.7157	0.0015 ND	0.0016 ND	0.0018 ND
Applic 3	Day 16	3	2	11/12/92	462	0.2634	0.2819	0.3164	0.0015 ND	0.0016 ND	0.0018 ND
Applic 3	Day 16	4	1	11/12/92	455	0.5010	0.5571	0.7304	0.0015 ND	0.0017 ND	0.0022 ND
Applic 3	Day 16	4	2	11/12/92	456	0.2790	0.2900	0.5076	0.0015 ND	0.0016 ND	0.0027 ND
Applic 3	Day 33	1	1	11/29/92	312	1.0812	1.1034	2.1184	0.0015 ND	0.0015 ND	0.0029 ND
Applic 3	Day 33	1	2	11/29/92	352	0.4062	0.4164	0.9190	0.0015 ND	0.0015 ND	0.0034 ND
Applic 3	Day 33	2	1	11/29/92	323	0.0996	0.1112	0.2277	0.0015 ND	0.0017 ND	0.0034 ND
Applic 3	Day 33	2	2	11/29/92	324	0.2068	0.2233	0.3449	0.0015 ND	0.0016 ND	0.0025 ND
Applic 3	Day 33	3	1	11/29/92	310	0.2922	0.3123	0.4839	0.0015 ND	0.0016 ND	0.0025 ND
Applic 3	Day 33	3	2	11/29/92	311	0.2154	0.2296	0.3994	0.0015 ND	0.0016 ND	0.0028 ND
Applic 3	Day 33	4	1	11/29/92	521	0.3224	0.3471	0.8122	0.0015 ND	0.0016 ND	0.0038 ND
Applic 3	Day 33	4	2	11/29/92	522	0.1922	0.1977	0.3138	0.0015 ND	0.0015 ND	0.0024 ND

ND = Not detected. Data reported are based upon calculations using one-half the detection limit for diazinon and diazoxon.

Turf Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DISLodgeABLE			DISLodgeABLE			TOTAL			TOTAL														
						DIAZINON			DIAZOXON			DIAZINON			DIAZOXON														
						ug/g	wet	ug/g dry	ug/cm2	ug/g	wet	ug/g dry	ug/cm2	ug/g	wet	ug/g dry	ug/cm2	ug/g	wet	ug/g dry	ug/cm2								
Applic 1	Day -1	1	1	10/7/92	343	0.0048	ND	0.0212	ND	0.0006	ND	0.0048	ND	0.0212	ND	0.0006	ND	0.0096	ND	0.0424	ND	0.0012	ND	0.0096	ND	0.0424	ND	0.0012	ND
Applic 1	Day -1	1	2	10/7/92	344	0.0048	ND	0.0168	ND	0.0006	ND	0.0048	ND	0.0168	ND	0.0006	ND	0.0096	ND	0.0337	ND	0.0012	ND	0.0096	ND	0.0337	ND	0.0012	ND
Applic 1	Day -1	2	1	10/7/92	345	0.0040	ND	0.0164	ND	0.0010	ND	0.0040	ND	0.0164	ND	0.0010	ND	0.0079	ND	0.0328	ND	0.0019	ND	0.0079	ND	0.0328	ND	0.0019	ND
Applic 1	Day -1	2	2	10/7/92	346	0.0043	ND	0.0131	ND	0.0008	ND	0.0043	ND	0.0131	ND	0.0008	ND	0.0086	ND	0.0263	ND	0.0016	ND	0.0086	ND	0.0263	ND	0.0016	ND
Applic 1	Day -1	3	1	9/27/92	429	0.0030	ND	0.0099	ND	0.0006	ND	0.0030	ND	0.0099	ND	0.0006	ND	0.0061	ND	0.0199	ND	0.0013	ND	0.0061	ND	0.0199	ND	0.0013	ND
Applic 1	Day -1	3	2	9/27/92	430	0.0041	ND	0.0128	ND	0.0006	ND	0.0041	ND	0.0128	ND	0.0006	ND	0.0082	ND	0.0256	ND	0.0012	ND	0.0082	ND	0.0256	ND	0.0012	ND
Applic 1	Day -1	4	1	9/27/92	499	0.0035	ND	0.0087	ND	0.0004	ND	0.0035	ND	0.0087	ND	0.0004	ND	0.0070	ND	0.0175	ND	0.0008	ND	0.0070	ND	0.0175	ND	0.0008	ND
Applic 1	Day -1	4	2	9/27/92	500	0.0025	ND	0.0070	ND	0.0004	ND	0.0025	ND	0.0070	ND	0.0004	ND	0.0050	ND	0.0140	ND	0.0008	ND	0.0050	ND	0.0140	ND	0.0008	ND
Applic 1	Day 0	1	1	10/7/92	349	31.3720		126.7042		2.6343		0.0748		0.3020		0.0063		48.7776		197.0015		4.0959		0.1389		0.5612		0.0117	
Applic 1	Day 0	1	2	10/7/92	350	48.9616		205.8076		3.8933		0.2027		0.8522		0.0161		86.8288		364.9800		6.9044		0.3322		1.3963		0.0264	
Applic 1	Day 0	2	1	10/7/92	347	45.5858		173.5279		8.2937		0.1064		0.4052		0.0194		67.6010		257.3315		12.2990		0.2348		0.8938		0.0427	
Applic 1	Day 0	2	2	10/7/92	348	35.3338		143.4583		4.2386		0.0697		0.2829		0.0084		55.6642		226.0018		6.6774		0.1520		0.6170		0.0182	
Applic 1	Day 0	3	1	9/29/92	505	26.8191		260.3793		10.0089		0.1073		1.0416		0.0400		60.4406		586.8021		22.5565		0.1562		1.5169		0.0583	
Applic 1	Day 0	3	2	9/29/92	506	32.9692		314.2916		8.7667		0.1107		1.0554		0.0294		76.1078		725.5276		20.2376		0.2476		2.3602		0.0658	
Applic 1	Day 0	4	1	9/29/92	479	63.1787		389.2709		5.7472		0.3829		2.3589		0.0348		103.7160		639.0386		9.4348		0.4647		2.8630		0.0423	
Applic 1	Day 0	4	2	9/29/92	480	34.0057		126.0405		3.5693		0.2264		0.8392		0.0238		55.7814		206.7510		5.8550		0.2900		1.0749		0.0304	
Applic 2	Day -1	1	1	10/21/92	511	7.2516		25.7422		0.5074		0.0532		0.1890		0.0037		15.1294		53.7076		1.0587		0.0783		0.2779		0.0055	
Applic 2	Day -1	1	2	10/21/92	512	8.2164		39.3130		0.4928		0.0815		0.3900		0.0049		16.3014		77.9970		0.9777		0.1292		0.6180		0.0077	
Applic 2	Day -1	2	1	10/21/92	513	21.1061		73.4894		1.5384		0.1637		0.5699		0.0194		44.1559		153.7461		3.2186		0.2358		0.8209		0.0172	
Applic 2	Day -1	2	2	10/21/92	514	10.6799		30.8490		0.9342		0.0564		0.1630		0.0049		23.7488		68.5985		2.0773		0.1099		0.3173		0.0096	
Applic 2	Day -1	3	1	10/13/92	625	2.7434		6.7390		0.1920		0.0500		0.1229		0.0035		6.9470		17.0646		0.4861		0.1179		0.2896		0.0082	
Applic 2	Day -1	3	2	10/13/92	626	4.8159		11.5796		0.2910		0.0551		0.1326		0.0033		6.8855		16.5557		0.4161		0.0916		0.2203		0.0055	
Applic 2	Day -1	4	1	10/13/92	627	9.7904		36.4226		2.1238		0.0407		0.1515		0.0088		23.2036		86.3229		5.0334		0.1050		0.3907		0.0228	
Applic 2	Day -1	4	2	10/13/92	628	15.1208		48.9347		2.9626		0.0382		0.1236		0.0075		29.2122		94.5378		5.7236		0.1039		0.3363		0.0204	
Applic 2	Day 0	1	1	10/21/92	516	99.0712		394.3918		5.7771		0.3855		1.5347		0.0225		173.0379		688.8450		10.0903		0.6456		2.5699		0.0376	
Applic 2	Day 0	1	2	10/21/92	517	72.6628		356.3647		4.5398		0.3818		1.8726		0.0239		126.7848		621.7991		7.9213		0.6538		3.2067		0.0409	
Applic 2	Day 0	2	1	10/21/92	518	95.9140		370.0386		9.6946		0.3202		1.2352		0.0324		174.4857		673.1700		17.6362		0.6696		2.5831		0.0677	
Applic 2	Day 0	2	2	10/21/92	519	60.2425		233.8608		3.8912		0.2004		0.7781		0.0129		101.3657		393.5003		6.5475		0.3881		1.5064		0.0251	
Applic 2	Day 0	3	1	10/13/92	602	187.0029		542.3517		11.6836		1.5492		4.4930		0.0968		237.8176		689.7261		14.8584		2.4728		7.1716		0.1545	
Applic 2	Day 0	3	2	10/13/92	601	145.4591		426.1912		8.2211		0.8285		2.4274		0.0468		201.4864		590.3497		11.3877		1.3743		4.0266		0.0777	
Applic 2	Day 0	4	1	10/13/92	605	39.6352		202.0142		10.2619		0.1537		0.7835		0.0398		75.2099		383.3330		19.4725		0.2971		1.5144		0.0769	
Applic 2	Day 0	4	2	10/13/92	606	87.0657		329.0464		18.2773		0.3268		1.2350		0.0686		149.4286		564.7341		31.3690		0.5926		2.2394		0.1244	
Applic 3	Day -1	1	1	11/4/92	585	0.6567		3.1391		0.0365		0.0144		0.0686		0.0008		3.3600		16.0614		0.1867		0.0484		0.2316		0.0027	
Applic 3	Day -1	1	2	11/4/92	586	0.5549		2.7279		0.0364		0.0121		0.0597		0.0008		1.7859		8.7803		0.1172		0.0374		0.1838		0.0025	
Applic 3	Day -1	2	1	11/4/92	587	4.6743		17.1786		0.5928		0.0419		0.1541		0.0053		13.3140		48.9305		1.6886		0.1146		0.4211		0.0145	
Applic 3	Day -1	2	2	11/4/92	588	3.7304		13.3803		0.4351		0.0374		0.1340		0.0044		8.1328		29.1706		0.9485		0.0653		0.2341		0.0076	
Applic 3	Day -1	3	1	10/27/92	553	1.4793		3.6338		0.0592		0.0332		0.0816		0.0013		6.1574		15.1251		0.2462		0.0910		0.2234		0.0036	
Applic 3	Day -1	3	2	10/27/92	554	1.1909		3.0474		0.0396		0.0261		0.0667		0.0009		6.0252		15.4177		0.2003		0.0749		0.1915		0.0025	
Applic 3	Day -1	4	1	10/27/92	557	6.4665		19.9398		1.4480		0.0380		0.1170		0.0085		15.9667		49.2343		3.5753		0.0839		0.2588		0.0188	
Applic 3	Day -1	4	2	10/27/92	558	0.7799		2.9187		0.1273		0.0097		0.0365		0.0016		4.1031		15.3560		0.6699		0.0339		0.1267		0.0055	
Applic 3	Day 0	1	1	11/4/92	590	18.8044		78.0913		1.1514		0.0866		0.3595		0.0053		34.3350		142.5873		2.1023		0.1861		0.7727		0.0114	
Applic 3	Day 0	1	2	11/4/92	591	11.8431		54.3758		0.6581		0.1044		0.4795		0.0058		24.6039		112.9653		1.3672		0.1925		0.8840		0.0107	
Applic 3	Day 0	2	1	11/4/92	592	52.2201		193.4794		5.2782		0.1798		0.6662		0.0182		94.9438		351.7742		9.5965		0.2178		0.8070		0.0220	
Applic 3	Day 0	2	2	11/4/92	593	17.6180		66.2330		2.4656		0.0614		0.2307		0.0086		32.1262		120.7751		4.4961		0.1266		0.4760		0.0177	
Applic 3	Day 0	3	1	10/27/92	495	75.3569		217.0418		2.9002		0.7725		2.2250		0.0297		133.2219		383.7035		5.1272		1.3183		3.7970		0.0507	

ND = Not detected. Values calculated using one-half the detection limit.

\* = Internal diazoxon residue was not detected.

Turf Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DISLodgeABLE DIAZINON			DISLodgeABLE DIAZOXON			TOTAL DIAZINON			TOTAL DIAZOXON		
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2
Applic 3	Day 0	3	2	10/27/92	496	32.0220	89.5721	1.5405	0.2131	0.5960	0.0103	59.0831	165.2674	2.8424	0.3587	1.0033	0.0173
Applic 3	Day 0	4	1	10/27/92	403	6.7421	23.2888	1.5333	0.3484	1.2035	0.0792	41.5560	143.5442	9.4507	0.4377	1.5120	0.0995
Applic 3	Day 0	4	2	10/27/92	404	19.4674	78.7835	2.3839	1.7148	6.9398	0.2100	32.4452	131.3039	3.9731	1.8133	7.3385	0.2221
Applic 3	Day 1	1	1	11/5/92	657	24.3879	124.9382	1.1851	0.1553	0.7956	0.0075	41.7982	214.1299	2.0311	0.3244	1.6618	0.0158
Applic 3	Day 1	1	2	11/5/92	658	16.0118	83.1783	1.1204	0.1135	0.5898	0.0079	27.5488	143.1106	1.9277	0.2643	1.3732	0.0185
Applic 3	Day 1	2	1	11/5/92	656	48.0208	175.3223	4.1240	0.2628	0.9594	0.0226	108.1488	394.8477	9.2877	0.5008	1.8286	0.0430
Applic 3	Day 1	2	2	11/5/92	655	73.5043	254.6926	7.4295	0.3268	1.1325	0.0330	138.5341	480.0210	14.0024	0.6537	2.2650	0.0661
Applic 3	Day 1	3	1	10/28/92	411	24.6719	71.5335	0.7063	0.3823	1.1083	0.0109	65.8069	190.7999	1.8838	0.7172	2.0796	0.0205
Applic 3	Day 1	3	2	10/28/92	412	37.2343	96.0636	0.9380	0.5217	1.3459	0.0131	80.0177	206.4441	2.0157	0.9213	2.3768	0.0232
Applic 3	Day 1	4	1	10/28/92	583	14.0993	65.1537	2.0554	0.0771	0.3565	0.0112	31.2785	144.5402	4.5598	0.1685	0.7785	0.0246
Applic 3	Day 1	4	2	10/28/92	584	5.2501	19.2736	0.8878	0.0275	0.1011	0.0047	25.3579	93.0905	4.2882	0.0935	0.3432	0.0158
Applic 3	Day 2	1	1	11/6/92	659	12.6570	52.0648	0.6889	0.0946	0.3892	0.0051	30.5628	125.7212	1.6634	0.1849	0.7604	0.0101
Applic 3	Day 2	1	2	11/6/92	660	5.9139	28.3230	0.2759	0.0808	0.3868	0.0038	12.8605	61.5924	0.5999	0.1620	0.7759	0.0076
Applic 3	Day 2	2	1	11/6/92	661	15.8027	53.3877	1.2901	0.1185	0.4002	0.0097	47.1306	159.2251	3.8476	0.2501	0.8450	0.0204
Applic 3	Day 2	2	2	11/6/92	662	58.0419	220.1892	4.0615	0.2640	1.0014	0.0185	104.8027	397.5822	7.3336	0.4549	1.7259	0.0318
Applic 3	Day 2	3	1	10/29/92	471	10.7016	129.4027	0.4446	0.2122	2.5656	0.0088	21.5807	260.9520	0.8966	0.3550	4.2930	0.0148
Applic 3	Day 2	3	2	10/29/92	472	12.0133	370.7813	0.4646	0.1709	5.2734	0.0066	26.6546	822.6724	1.0307	0.3180	9.8163	0.0123
Applic 3	Day 2	4	1	10/29/92	473	1.5948	8.1785	0.5022	0.0238	0.1219	0.0075	11.7195	60.1001	3.6903	0.0680	0.3485	0.0214
Applic 3	Day 2	4	2	10/29/92	474	2.1499	9.0714	0.4965	0.0194	0.0821	0.0045	10.0741	42.5070	2.3263	0.0588	0.2479	0.0136
Applic 3	Day 3	1	1	11/7/92	665	7.8796	34.5447	0.3722	0.0807	0.3537	0.0038	18.5751	81.4342	0.8774	0.1444	0.6332	0.0068
Applic 3	Day 3	1	2	11/7/92	666	8.3298	38.6354	0.4736	0.0715	0.3319	0.0041	8.4013	38.9673	0.4777	0.1431	0.6637	0.0081
Applic 3	Day 3	2	1	11/7/92	663	5.2056	19.8536	0.5282	0.0394	0.1501	0.0040	33.9051	129.3099	3.4402	0.1215	0.4634	0.0123
Applic 3	Day 3	2	2	11/7/92	664	16.3367	60.3945	1.4030	0.0920	0.3402	0.0079	42.7761	158.1370	3.6736	0.1862	0.6882	0.0160
Applic 3	Day 3	3	1	10/30/92	529	2.8990	7.2385	0.1319	0.0611	0.1527	0.0028	9.5177	23.7645	0.4329	0.1273	0.3178	0.0058
Applic 3	Day 3	3	2	10/30/92	530	2.4252	6.8643	0.1145	0.0422	0.1195	0.0020	7.0989	20.0932	0.3353	0.0921	0.2607	0.0044
Applic 3	Day 3	4	1	10/30/92	533	0.2749	1.0400	0.0769	0.0046	0.0175	0.0013	6.7128	25.3986	1.8789	0.0177	0.0671	0.0050
Applic 3	Day 3	4	2	10/30/92	534	0.9068	2.2642	0.4045	0.0055	0.0137	0.0025	2.3996	5.9916	1.0705	0.0127	0.0317	0.0057
Applic 3	Day 8	1	1	11/12/92	571	1.3216	4.9462	0.0643	0.0247	0.0923	0.0012	7.6884	28.7740	0.3743	0.0724	0.2708	0.0035
Applic 3	Day 8	1	2	11/12/92	572	1.6103	6.2535	0.1167	0.0183	0.0710	0.0013	4.8317	18.7639	0.3502	0.0472	0.1832	0.0034
Applic 3	Day 8	2	1	11/12/92	573	3.5163	12.5494	0.4042	0.0285	0.1018	0.0033	16.2567	58.0181	1.8689	0.1004	0.3584	0.0115
Applic 3	Day 8	2	2	11/12/92	574	8.2355	29.5815	1.0291	0.0525	0.1886	0.0066	22.0365	79.1540	2.7536	0.1399	0.5024	0.0175
Applic 3	Day 8	3	1	11/4/92	421	0.4636	1.0390	0.0243	0.0305	0.0684	0.0016	3.7500	8.4043	0.1968	0.0927	0.2078	0.0049
Applic 3	Day 8	3	2	11/4/92	422	0.2067	0.5299	0.0134	0.0168	0.0431	0.0011	1.9207	4.9235	0.1248	0.0529	0.1356	0.0034
Applic 3	Day 8	4	1	11/4/92	425	0.1499	0.5711	0.0378	0.0030 ND*	0.0116	0.0008	1.7998	6.8565	0.4534	0.0061	0.0232	0.0015
Applic 3	Day 8	4	2	11/4/92	426	0.1389	0.4083	0.0321	0.0025 ND	0.0072	0.0006	2.5956	7.6274	0.5994	0.0191	0.0562	0.0044
Applic 3	Day 12	1	1	11/16/92	577	1.6828	2.3001	0.1132	0.2475	0.3382	0.0166	8.6012	11.7567	0.5787	0.2995	0.4094	0.0202
Applic 3	Day 12	1	2	11/16/92	578	1.0879	1.7742	0.0825	0.1850	0.3017	0.0140	4.6365	7.5611	0.3515	0.2141	0.3492	0.0162
Applic 3	Day 12	2	1	11/16/92	575	4.2393	6.7785	0.4450	0.0505	0.0807	0.0053	21.1264	33.7807	2.2175	0.1177	0.1883	0.0124
Applic 3	Day 12	2	2	11/16/92	576	1.6897	2.8041	0.1478	0.1766	0.2931	0.0154	24.3658	40.4344	2.1313	0.2619	0.4347	0.0229
Applic 3	Day 12	3	1	11/8/92	397	0.5552	1.4622	0.0131	0.0332	0.0875	0.0008	4.3232	11.3858	0.1022	0.0940	0.2476	0.0022
Applic 3	Day 12	3	2	11/8/92	398	0.5213	1.3712	0.0137	0.0286	0.0753	0.0008	3.1833	8.3727	0.0835	0.0755	0.1986	0.0020
Applic 3	Day 12	4	1	11/8/92	399	0.3193	1.2071	0.0577	0.0076	0.0286	0.0014	3.9252	14.8403	0.7096	0.0330	0.1248	0.0060
Applic 3	Day 12	4	2	11/8/92	400	0.1565	0.5663	0.0285	0.0081	0.0293	0.0015	3.0203	10.9311	0.5495	0.0327	0.1182	0.0059
Applic 3	Day 16	1	1	11/20/92	579	0.9121	4.0756	0.0426	0.0152	0.0680	0.0007	5.4304	24.2646	0.2533	0.0890	0.3977	0.0042
Applic 3	Day 16	1	2	11/20/92	580	0.4196	1.4611	0.0352	0.0041 ND	0.0143	0.0003	1.8513	6.4462	0.1555	0.0272	0.0945	0.0023

ND = Not detected. Values calculated using one-half the detection limit.

\* = Internal diazoxon residue was not detected.

Turf Results

SPRAY EVENT	SAMPLE EVENT	SITE	SUB SITE	SAMPLE DATE	SAMPLE NUMBER	DISLODGEABLE DIAZINON			DISLODGEABLE DIAZOXON			TOTAL DIAZINON			TOTAL DIAZOXON			
						ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2	ug/g wet	ug/g dry	ug/cm2	
Applic 3	Day 16	2	1	11/20/92	581	1.9629	6.3833	0.1717	0.0206	0.0669	0.0018	2.9851	9.7078	0.2611	0.1543	0.5017	0.0135	
Applic 3	Day 16	2	2	11/20/92	520	3.0699	9.8050	0.2864	0.0257	0.0822	0.0024	20.6346	65.9041	1.9252	0.2238	0.7149	0.0209	
Applic 3	Day 16	3	1	11/12/92	459	0.2868	0.8726	0.0040	0.0058	ND	0.0178	0.0001	1.9486	5.9281	0.0270	0.0448	0.1363	0.0006
Applic 3	Day 16	3	2	11/12/92	460	0.1207	0.2997	0.0056	0.0024	ND	0.0060	0.0001	1.6264	4.0388	0.0752	0.0296	0.0735	0.0014
Applic 3	Day 16	4	1	11/12/92	453	0.1833	0.8391	0.0278	0.0037	ND	0.0170	0.0006	3.0139	13.7935	0.4569	0.0191	0.0873	0.0029
Applic 3	Day 16	4	2	11/12/92	454	0.1108	0.4921	0.0144	0.0037	ND	0.0164	0.0005	2.2393	9.9480	0.2910	0.0194	0.0864	0.0025
Applic 3	Day 33	1	1	12/7/92	639	0.1307	0.7202	0.0128	0.0027	ND	0.0146	0.0003	1.1002	6.0620	0.1080	0.0094	0.0517	0.0010
Applic 3	Day 33	1	2	12/7/92	640	0.0562	0.2441	0.0062	0.0032	ND	0.0140	0.0004	0.9794	4.2546	0.1088	0.0097	0.0419	0.0011
Applic 3	Day 33	2	1	12/7/92	641	0.5267	1.8217	0.0340	0.0126	0.0435	0.0008	6.5153	22.5364	0.4208	0.0491	0.1699	0.0032	
Applic 3	Day 33	2	2	12/7/92	642	1.5947	5.9615	0.1030	0.0110	0.0413	0.0007	15.3319	57.3154	0.9902	0.0641	0.2395	0.0041	
Applic 3	Day 33	3	1	11/29/92	363	0.0714	0.1365	0.0140	0.0041	ND	0.0078	0.0001	1.2184	2.3300	0.0359	0.0170	0.0326	0.0003
Applic 3	Day 33	3	2	11/29/92	364	0.0243	0.0505	0.0007	0.0038	ND	0.0079	0.0001	0.4684	0.9744	0.0139	0.0119	0.0247	0.0003
Applic 3	Day 33	4	1	11/29/92	365	0.1607	0.4419	0.0317	0.0036	ND	0.0099	0.0007	4.1761	11.4791	0.8241	0.0161	0.0442	0.0032
Applic 3	Day 33	4	2	11/29/92	366	0.0345	0.1202	0.0070	0.0033	ND	0.0116	0.0007	1.9000	6.6204	0.3869	0.0113	0.0393	0.0023

ND = Not detected. Values calculated using one-half the detection limit.

\* = Internal diazoxon residue was not detected.

Appendix C - Laboratory Quality Control

Table 1. CDFA's Method Validation Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.2 ppm  
 Date of Report: 5/22/92

Matrix Sample Type: Soil  
 Lab: CDFA  
 Chemist: Karen Hefner

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
4812	0.199	0.2	100	100	8.0	8.0		
	0.183	0.2	92					
	0.216	0.2	108					
4814	2.01	2.0	101	100	1.73	1.73		
	1.95	2.0	98					
	2.01	2.0	101					
4816	21.56	20	108	107	6.11	5.71		
	19.92	20	100					
	22.40	20	112					
OVERALL:				102	6.10	5.98	84	120

Table 2. CDFA's Method Validation Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazoxon  
 MDL: 0.2 ppm  
 Date of Report: 5/22/92

Matrix Sample Type: Soil  
 Lab: CDFA  
 Chemist: Karen Hefner

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
4813	0.172	0.2	86	90	4.6	5.1		
	0.190	0.2	95					
	0.179	0.2	89					
4815	2.14	2.0	107	106	2.65	2.50		
	2.05	2.0	103					
	2.16	2.0	108					
4817	17.20	20	86	89	2.9	3.3		
	18.11	20	91					
	18.26	20	91					
OVERALL:				95	8.7	9.2	69	121

LCL = lower control limit (- 3 SD)  
 UCL = upper control limit (+ 3 SD)

Table 3. Enseco-Cal's Analytical's Method Validation Data (% recoveries) for the Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.1 ppm  
 Date of Report: 4/7/92

Matrix Sample Type: Soil  
 Lab: CAL Analytical  
 Chemist: D. Gall

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
63195-1	0.089	0.1	89					
63195-2	0.1	0.1	100					
63195-3	0.1	0.1	100	96	6.4	6.7		
63195-4	1.1	1.0	110					
63195-5	1.1	1.0	110					
63195-6	1.2	1.0	120	113	5.77	5.11		
63195-7	11.0	10	110					
63195-8	11.0	10	110					
63195-9	11.0	10	110	110	0	0		
OVERALL:				107	8.90	8.32	80	134

LCL = lower control limit (- 3 SD)  
 UCL = upper control limit (+ 3 SD)

Table 3A. Enseco-Cal's Analytical's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.1 ppm  
 Date of Report: 11/19/92

Matrix Sample Type: Soil  
 Lab: CAL Analytical  
 Chemist: D. Gall

Extraction Set No.'s	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
704-19	0.58	0.50	116					
	0.57	0.50	114					
OVERALL:				115	1.41	1.22	80	134

Table 4. CDFA's Method Validation Data (% recoveries) for the Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.15 ug/sample  
 Date of Report: 1/25/93

Matrix Sample Type: Dislodgeable Turf  
 Lab: CDFA  
 Chemist: J. Hernandez

Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
9.92	10	99.20					
10.05	10	100.50					
10.20	10	102.0	100.57	1.40	1.39		
103.56	100	103.60					
100.79	100	100.80					
195.02	100	195.0	133.13	53.60	40.26		
OVERALL:			116.9	38.31	32.79	89	109

Table 5. CDFA's Method Validation Data (% recoveries) for the Medfly Study

Study: 118  
 Chemical: Diazoxon  
 MDL: 0.15 ug/sample  
 Date of Report: 1/25/93

Matrix Sample Type: Dislodgeable Turf  
 Lab: CDFA  
 Chemist: J. Hernandez

Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
9.50	10	95.0					
9.42	10	94.20					
9.31	10	93.10	94.10	0.95	1.01		
94.34	100	94.34					
90.20	100	90.20					
91.69	100	91.69	92.08	2.10	2.28		
OVERALL:			93.09	1.83	1.97	96	102

LCL = lower control limit (- 3 SD)  
 UCL = upper control limit (+ 3 SD)

Table 6. CDFA's Method Validation Data (% recoveries) for the Medfly Study.

Study: 118  
 Chemical: Diazinon  
 MDL: 0.15 ug/sample  
 Date of Report: 1/25/93  
 Sample Type: Penetrated Turf  
 Lab: CDFA  
 Chemist: J. Hernandez

Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
0.9488	1.0	94.88					
1.0415	1.0	104.15					
1.0017	1.0	100.17	99.73	4.65	4.66		
9.32	10	93.20					
9.24	10	92.40					
9.45	10	94.50	93.37	1.06	1.14		
95.60	100	95.60					
93.55	100	93.60					
93.54	100	93.50	94.23	1.18	1.26		
478.16	500	95.63					
483.77	500	96.75					
464.76	500	92.95	95.11	1.95	2.05		
OVERALL:			95.61	3.42	3.57	86	106

Table 7. CDFA's Method Validation Data (% recoveries) for the Medfly Study

Study: 118  
 Chemical: Diazoxon  
 MDL: 0.15 ug/sample  
 Date of Report: 1/25/93  
 Sample Type: Penetrated Turf  
 Lab: CDFA  
 Chemist: J. Hernandez

Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)	LCL	UCL
0.9622	1.0	96.22					
1.0065	1.0	100.65					
0.9347	1.0	93.47	96.78	3.62	3.74		
9.42	9.5	99.20					
9.53	9.5	100.30					
9.98	9.5	103.20	100.90	2.07	2.05		
97.66	95	102.80					
96.58	95	101.70					
95.80	95	100.80	101.77	1.00	0.98		
464.90	500	92.98					
490.35	500	98.07					
451.96	500	90.39	93.81	3.91	4.16		
OVERALL:			98.32	4.17	4.24	85	111

LCL = lower control limit (- 3 SD)  
 UCL = upper control limit (+ 3 SD)

Table 8. CDFA's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.15 ug/sample  
 Date of Report: 12/23/92

Matrix Sample Type: Soil  
 Lab: CDFA  
 Chemist: B. Fong  
 Sample Size: 50 grams

Extraction Set No.'s	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
427,428,431-436	21.81	25	87			
619-624,629,630	20.11*	25	80			
595-600,603,604	21.69	25	87			
487,490,492,494,498, 547-550,552,555,556	20.59*	25	82			
475,476,503,504,507, 508-510	23.86	25	95			
406,408,410,414,463,465, 467,469,489,491,493	23.24	25	93			
405,407,409,413,464,466, 468,470,524,526,528,532, 415-420,423,424,523,525, 527,531	17.83*	25	71			
391-396,401,402	7.07*	10	71			
451,452,455-458,461,462	9.36	10	94			
310,311,312,323,324 521,522	8.61	10	86			
OVERALL:				85	8.2	9.6

\* Recoveries fell below the lower control limit set for soil at 84%.

Table 9. CDFA's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazoxon  
 MDL: 0.15 ug/sample  
 Date of Report: 12/23/92

Matrix Sample Type: Soil  
 Lab: CDFA  
 Chemist: B. Fong  
 Sample Size: 50 grams

Extraction Set No.'s	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
427,428,431-436	23.36	25	93			
619-624,629,630	26.68	25	107			
595-600,603,604	21.17	25	85			
487,490,492,494,498, 547-550,552,555,556	24.57	25	98			
475,476,503,504,507, 508-510	25.31	25	101			
406,408,410,414,463 467,469,489,491,493	27.27	25	109			
405,407,409,413,464,466, 468,470,524,526,528,532, 415-420,423,424,523,525, 527,531	21.20	25	85			
391-396,401,402	25.05	25	100			
451,452,455-458,461,462	9.09	10	91			
310,311,312,323,324 521,522	9.63	10	96			
	10.45	10	105			

OVERALL: 97 8.3 8.5

Table 10. CDFA's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118

Chemical: Diazinon

MDL: 0.15 ug/sample

Date of Report: 12/23/92

Matrix Sample Type: Dislodgeable Turf

Lab: CDFA

Chemist: B. Fong

Sample Size: 25 grams

Sample Number	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
479-80,482,505-506	96.97	100	97			
343-350	97.14	100	97			
601-602,605-606,625-628	96.39	100	96			
511-514,516-519	95.03	101	94			
403-4, 495-6, 553-4, 557-8	110.14	101	109			
411-412,583-584	110.10	100	110			
429-30, 499-500	90.48	101	89			
471-474	86.74	101	86			
529-530,533-534	98.38	105	97			
659-662	107.96	101	107			
663-666	107.76	101	107			
453-454,459-460,571-574	9.36	9.5	99			
421-422,425-426,585-587	94.65	101	94			
655-658	94.59	101	94			
397-400	99.99	101	99			
575-578	9.74	9.5	103			
520,579-581	9.26	9.5	97			
588, 590-3	94.65	101	94			
639-42	9.89	10	97			
363-366	9.56	10	94			

OVERALL: 98 6.4 6.5

Table 11. CDFA's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazoxon  
 MDL: 0.15 ug/sample  
 Date of Report: 12/23/92

Matrix Sample Type: Dislodgeable Turf  
 Lab: CDFA  
 Chemist: B. Fong  
 Sample Size: 25 grams

Sample Number	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
479-80,482,505-506	106.64	100	107			
343-350	102.45	100	102			
601-602,605-606,625-628	102.17	100	102			
511-514,516-519	106.07	107	99			
403-4, 495-6, 553-4, 557-8	117.91	107	110			
411-412,583-584	117.91	100	118			
429-30, 499-500	96.56	107	90			
471-474	95.51	107	89			
529-530,533-534	107.04	107	100			
659-662	115.95	107	108			
663-666	114.26	107	107			
453-454,459-460,571-574	10.41	10	104			
421-422,425-426,585-587	101.38	107	95			
655-658	104.66	107	98			
397-400	108.08	107	100			
575-578	9.29	10	93			
520,579-581	10.11	10	101			
588, 590-93	101.38	107	95			
639-42	9.52	11	87			
363-366	10.96	11	100			

OVERALL: 100 7.62 7.61

Table 12. CDFA's Continuing Quality Control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazinon  
 MDL: 0.15 ug/sample  
 Date of Report: 12/23/92  
 Matrix Sample Type: Penetrated Turf  
 Lab: CDFA  
 Chemist: B. Fong  
 Sample Size: 25 grams

Extraction Set No.'s	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
479-80,505-506	86.2	100	86			
343-350	94.3	100	94			
601-602,605-606,625-628	112.3	100	112			
511-514,516-519	82.4	101	81			
403-4, 495-6, 553-4, 557-411-12, 583-4	67.0	101	66			
471-4	127.1	101	126			
529-30, 533-4	62.4	101	62			
421-2, 425-6, 585-7	45.5	100	46			
397-400	56.2	101	56			
659-62	5.5	9.5	58			
639-42	25.0	50	50			
663-666	9.97	10	100			
453-454,459-460,571-574	7.5	10	75			
655-658	7.9	10	79			
575-578	112.1	100	112			
520,579-581	7.2	9.5	75			
588, 599-93	8.1	9.5	85			
363-366	56.20	101	56			
	9.37	10	93			
OVERALL:				80	23	28

Table 13. CDFA's Continuing Quality control Data (% recoveries) for the 1992 Medfly Study

Study: 118  
 Chemical: Diazoxon  
 0.15 ug/sample  
 Date of Report: 12/23/92

Matrix Sample Type: Penetrated Turf  
 Lab: CDFA  
 Chemist: B. Fong  
 Sample Size: 25 grams

Extraction Set No.'s	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
379-80,505-506	103.6	100	104			
343-350	95.5	100	95			
601-602,605-606,625-628	91.3	100	91			
511-514,516-519	108.3	107	101			
403-4, 495-6, 553-4, 557-	97.6	107	91			
411-12, 583-4	135.1	107	126			
471-4	84.97	107	79			
529-30, 533-4	65.59	100	66			
421-2, 425-6, 585-7	93.89	107	88			
397-400	8.32	10	83			
659-62	38.80	50	78			
639-42	9.90	9.5	104			
663-666	8.8	10	88			
453-454,459-460,571-574	9.5	10	95			
655-658	114.1	100	114			
575-578	8.58	10	86			
520,579-581	10.1	10	101			
588, 590-93	93.9	107	88			
363-366	11.17	11	102			
OVERALL:				94	14	15