Dislodgeable Azinphosmethyl Residues on Treated Pear Foliage

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Summary

Azinphosmethyl is an organophosphorus insecticide and cholinesterase inhibiting compound. This insecticide has been in use for decades to control codling moth in pear orchards. In 1994, the Department of Pesticide Regulation, Worker Health and Safety Branch conducted dislodgeable foliar residue (DFR) sampling of row and tree crops at time of harvest (1). During this sampling some pear orchards in the Sacramento valley had azinphosmethyl residues of 5.3 ug/cm^2. This study was conducted to confirm the higher DFR levels found in 1994 and to supplement results from previous dissipation studies. Dislodgeable foliar residue sampling was conducted in the Sacramento Delta Region. The four fields monitored at the beginning of the study did not approach the higher levels found in the 1994 survey. At no time did residues exceed 1.65 ug/cm^2 for these four fields. Additional fields were then obtained for sampling and residues up to 4 ug/cm^2 were reported. These additional fields had application rates of six pounds azinphosmethyl for the season as opposed to the two pounds per season of the first fields monitored. Azinphosmethyl oxon was detected in only six samples and the highest level was 0.0175 ug/cm^2. Previous studies suggest the possibility of similar residues in the past. Linear decay models were comparable to previous branch studies. From the data it could not be concluded that azinphosmethyl accumulates on tree foliage with subsequent applications. No history of systemic illnesses to field workers harvesting pears has been observed. Research in stone fruits (21, 29), where DFR levels ranged from 0.5 - 1.75 ug/cm^2, has shown field workers developed depressed cholinesterase levels during the harvest season without clinical symptoms. The harvest season for stone fruits is anywhere from twice to six times that of the pear harvest that is usually less than four weeks. Since DFR residues are variable among fields, workers are likely not exposed to consistently high residues, and accounts for the fact there are no reported cases of systemic fieldworker illnesses while harvesting in pears.

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Introduction

In 1994, the Department of Pesticide Regulation conducted dislodgeable foliar residue (DFR) sampling of row and tree crops (1). This 1994 study was designed to survey DFR in a wide variety of crops under hand cultivation and hand picking operations. While spray history or application dates were not usually known this information provided a current index of the identity and magnitude of pesticide residues to which field workers are exposed. During this sampling some pear orchards in the Sacramento valley had azinphosmethyl residues up to 5.3 ug/cm^2. In 1985, a similar survey at time of harvest was conducted for pears in Solano and Sacramento counties and residues of 1-2 ug/cm^2 were reported (2). In 1986, a follow-up degradation study was conducted on pears and residue levels for azinphosmethyl ranged from 0.5-1.5 ug/cm^2 two days after application (3). Additional studies were also conducted for DFR levels of azinphosmethyl on pome or stone fruits (4-22). One study (10) reported sampling results as high as 3.59 ug/cm^2 thirteen days after application. No half life could be calculated from the data because residues did not degrade over the 30 days the field was monitored. From the reviewed studies, foliar dissipation for azinphosmethyl appears to be slow; it was first introduced in 1953 it as a nonsystemic insecticide and acaricide of long persistence (23). Current labeling requires each 48-hour reentry be increased to 72 hours in areas where rainfall is less than 25 inches a year. Pears also have a required pre-harvest interval of seven days after the last application. Additionally, only six pounds of azinphosmethyl may be applied per season and a seven day waiting period is required between applications. While the exact days post application are not known for the 1994 study the minimum would be seven days due to the label requirements for the pre-harvest interval. It is also unlikely that the residues are from a single application because multiple application are a common practice in the area. Also data from past studies indicate that slow degradation and repeat applications could increase the leaf residues.

Azinphosmethyl is an organophosphorus insecticide and cholinesterase inhibiting compound. During the period 1982-1990 there were 98 cases that involved azinphosmethyl as the sole contributor to systemic illness and another 56 cases where more than one cholinesterase inhibitor was present (24). Data extracted from the California Pesticide Illness and Surveillance Program for the years 1982-1993 revealed thirteen worker illnesses in pear orchards. Of the thirteen reported cases ten occurred during the application of the insecticide to pear orchards (eight ground applicators, one while mixing and loading and one person was exposed to spray drift). Two cases involved fieldworkers developing skin rashes in fields that were also treated with other insecticides. One irrigator developed symptoms of systemic poisoning after entering an orchard that was just sprayed. No incidents involved a worker entering a field to harvest pears and developing a systemic illness.

This study was conducted to confirm the higher DFR levels found in 1994 and to supplement results from previous dissipation studies.

Methods

Fields were located with the assistance of staff from the County Agricultural Commissioners, growers and farm managers. Dislodgeable foliar residue sampling was conducted according to established procedures (25,26). Leaf punches were taken from a single row starting with the fifth tree at four punches per tree from each quadrant. Samples were taken from a height of 1.5-2 meters from the older leaves. A 2.54 cm diameter Birkestrand(R) leaf punch was used to take the sample. Each sample consisted of 40 punches with three samples per field. At the beginning of the study four fields were monitored. These four fields were treated by air blast sprayer at the rate of 2 pounds Gowan Azinphos-M 50 per acre (EPA# 10163-78 AA) in 100 gallons of water (1 pound per acre active

ingredient). The label requires seven days between repeat applications. Initially the fields were to be sampled pre-application, 1, 3, 6, 12 and 24 days or a follow-up sample before a second application. If a follow-up application took place sampling would begin again or if harvest occurred sampling was terminated.

It was anticipated that at least three treatments would occur but insect pressure was lighter than expected and only one additional application took place in the initial plots. Since the observed residues at the two pound per season application rate did not approach those residues found in 1994, additional fields were located with more than two treatments. Also, five fields from the 1994 survey were resampled and an additional seven fields were sampled at harvest. Sampling of all fields is outlined in Table 1. Sampling of the later fields was terminated at harvest. Fields 3 and 4 were sampled at all the intervals after the second application but fields 1 and 2 were sampled at only three selected intervals because of available resources used to sample additional fields.

Table I Field sampling for azinphosmethyl foliar residues

Field No.	Initial date sampled	Days sampled post application 1st & 2nd or last treatment	Active ingredient lbs/ac/season	Rate/ac/treatment & No. treatments
1	6/10/95	1, 3, 6 ,12, 20 & 19	2	1# - 2
2	6/10/95	1, 3, 6, 12, 20 & 5, 14	2	1# - 2
3	6/10/95	1, 3, 6, 12, 20 & 1, 5, 14, 19	2	1# - 2
4	6/10/95	1, 3, 6, 12, 20 & 1, 5, 14, 19	2	1# - 2
5	7/6/95	1, 5, 8, 13	2	1# - 2
6	7/11/95	2, 4, 9	6	1.5# - 4
7	7/11/95	4, 5, 7, 12	6	1.5# - 4
8	7/11/95	1, 8	6	1.5# - 4
9	7/11/95	2, 9	3	1# - 3
10	7/11/95	6, 15	6	1.5# - 4
11	7/11/95	1, 8	3	1# - 3
12	7/11/95	5	6	1.5# - 4
13,14,15,21,22	6/29/95	resample of 1994 fields	unable to verify	
16,17,18,19,20,	7/20/95	sampled at harvest	unable to verify	
23,24	7/10/95	sampled at harvest	unable to verify	

Each sample jar was capped with a Teflon-lined lid, labeled, bagged, and placed on ice in an insulated chest. A Request for Analysis form was completed and submitted to the laboratory with each sample. Samples were transported to the California Department of Food and Agriculture, Center for Analytical Chemistry (CDFA) laboratory the day of collection and extracted within 24 hours. Laboratory analysis was for azinphosmethyl and the oxon. Samples were analyzed by the CDFA. Leaf disks were shaken three times with 50 mL 0.05% sodium dioctyl sulfosuccinate solution, which was then extracted three times using 50 mL ethyl acetate. The organic extract was then dried by anhydrous sodium sulfate. The samples were analyzed by gas liquid chromatography on a Hewlett-Packard 5880A chromatograph equipped with a phosphorus detector. The chromatographic conditions were: column, 10 m x 0.53 mm 50% phenyl methyl silicone; carrier gas (He), 20 mL/min; H_2, 4 mL/min; air, 90 mL/min; injector and detector temperature, 250 ^{o}C; oven temperature, 240 ^{o}C, isothermal. Using these conditions, the retention time was 6.00 minutes for azinphosmethyl and 4.89 minutes for azinphosmethyl oxon. Standards were introduced periodically during the analysis. The minimum detectable level for azinphosmethyl was 2 ug per sample and 5 ug per sample for azinphosmethyl oxon.

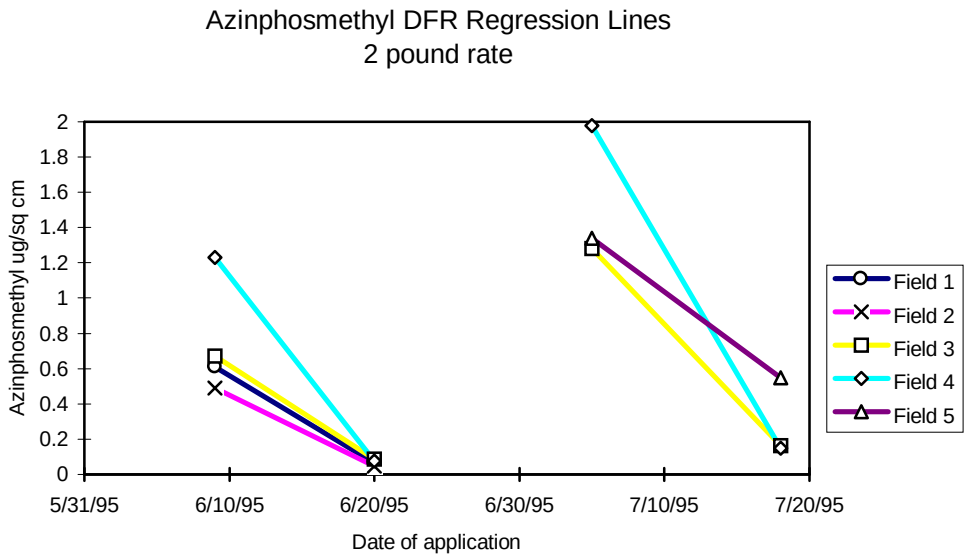
Laboratory spikes of the extract were within established parameters and no corrections of the data were performed. Azinphosmethyl oxon analysis was completed for all samples above 1000 ug azinphosmethyl but only 10% of those below. The analysis for the oxon was not completed for all samples because the oxon was detected in only six samples and these levels were at or slightly above the limit of detection when the parent compound was above 1200 ug/sample.

Data analysis used Microsoft Excel version 5.0c analysis tool pak to perform linear regression and develop a decay model.

Results

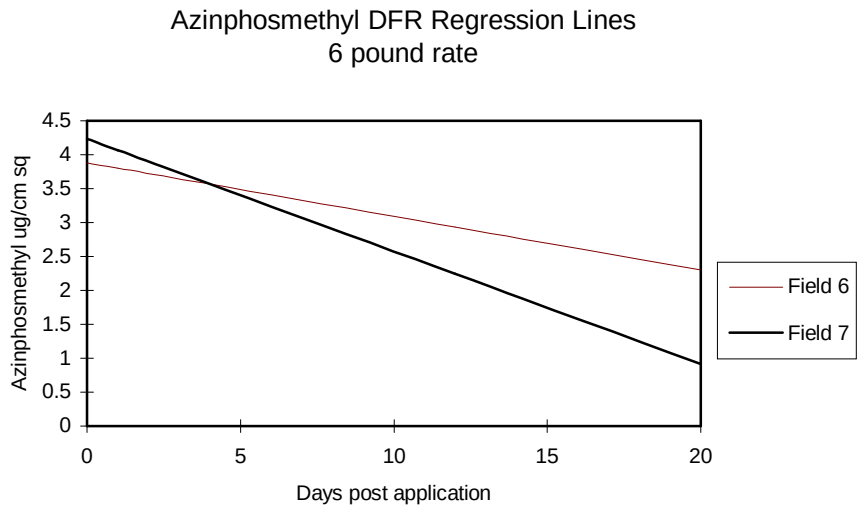
Fields monitored with a seasonal application rate of two pounds of azinphosmethyl did not approach the higher levels found in the 1994 survey. Figure 1 shows the theoretical decay rate of DFR for fields one through five. Figure 1 includes the second treatments for fields three, four and five where field five was monitored only after the second treatment. The highest residue measured for the five fields was 1.65 $\mu g/cm^2$ one day post application for the second application to field four. When the regressions were performed this gave a theoretical initial deposition of 1.98 ug/cm^2 for field four. These fields had similar degradation half-lives of 3 to 4 days except field 5 with a half life of ten days. The information on half-lives and the regressions performed to develop the figures are found in Table II.

Figure 1



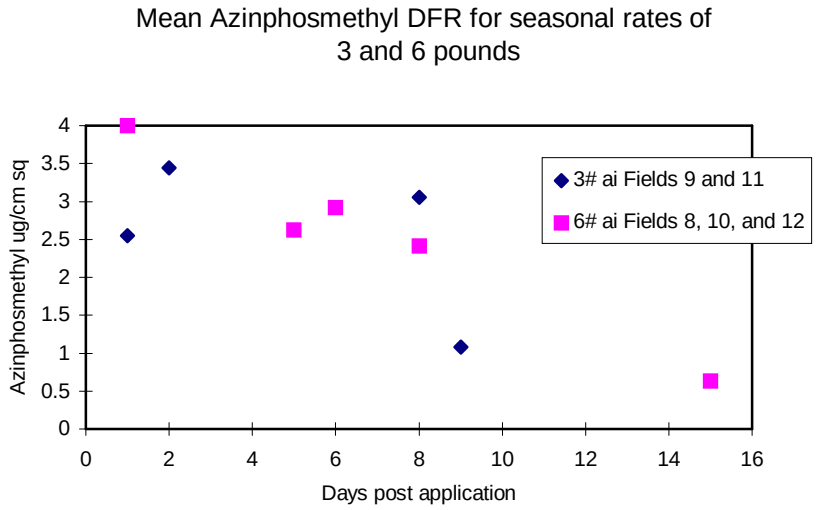
When the amount of azinphosmethyl applied is at the six pound rate per season as presented in Figure 2 the residue levels calculated at time zero after the last seasonal application are similar to those found in 1994. For the data in Figures 2 monitoring began after the last application so it is unknown how much each of the three previous applications contributed to the overall residues. Half-lives for fields six and seven were 24 and 11 days respectively.

Figure 2



Regressions were not performed on fields 8, 9, 10, 11and 12 because there were not sufficient data points to perform the calculation. Data for fields 8, 10 and 12 at the six pound application rate were combined and a regression was performed on the combined data. This regression gave a half-life of nine days with an initail depostion of 4.21 ug/cm^2 (r^2 0.52). Fields 9 and 11 showed DFR levels in the range of 3 to 4 ug/cm^2 where the seasonal application rate was three pounds per season.These levels are comparable to those found at the six pound application rate and the samples collected in 1994. Combining fields 9 and 11 to perform a regression yielded a poor r^2 of 0.22. The daily combined means for the 3# and 6# application rates for these five fields are plotted in Figure 3.

Figure 3



As stated earlier azinphosmethyl oxon was detected in only six samples and the highest level was 0.0175 ug/cm^2 or 7 ug/sample with the limit of detection at 5 ug/sample. Appendix 1 reports the data from all fields sampled. Of the five fields sampled in 1994 that had high levels, four had levels ranging from 2.8-4.4 ug/cm^2 and the fifth field was below 1 ug/cm^2. Another resampling during the harvest season showed levels dropped slightly but no confirmation of the last application date could be obtained. Of the seven fields sampled at harvest two had DFR levels that averaged 3 ug/cm^2 while the

other five had DFR levels ranging from 0.2 - 0.6 ug/cm^2. A summary of these analyses showing theoretical initial deposition, decay rate and respective half-lives is presented in Table II below.

Table II: Summary of regression analyses for azinphosmethyl

Field[a]	Theoretical initial deposition µg/cm^2	Half life Days	Decay rate ln (µg/cm^2)	r^2
1	0.61	3.7	-0.187	0.90
2	0.49	3.6	-0.194	0.98
3	0.67	4	-0.171	0.97
3[b]	1.28	4.4	-0.157	0.81
4	1.23	3	-0.230	0.98
4[b]	1.98	3.5	-0.199	0.93
5	1.34	10	-0.069	0.84
6	4.2	24	-0.029	0.87
7	4.74	11	-0.060	0.61

[a] Log-linear regressions performed only on fields with sufficient data points.
[b] Regressions performed separately for applications one and two.

Discussion

Dislodgeable foliar residue (DFR) levels from previous branch studies on pome fruit (3, 9, 11, 12, 17) ranged from 0.47 - 4.5 ug/cm^2 one day post application (n=15 fields) and are comparable to dislodgeable foliar residue levels found in this study. The 4.5 ug/cm^2 (9) was from only one of five fields with the mean level of all fields at 2.39 ug/cm^2. Previous data collected by this branch did not reveal sufficient evidence to conclude that azinphosmethyl accumulates on tree foliage with subsequent applications (3, 14). This present study shows a theoretical increase in initial deposition from the second application for fields 3 and 4 with a half day increase in the half-life. One earlier study conducted by this branch (2) did report on three fields with residues ranging from 1.0 - 1.98 ug/cm^2 at harvest with the highest level found 30 days post application. This earlier DFR data compares favorably with the linear decay model developed from the data of field six where initial residues were above 4 ug/cm^2. Half life data was also similar and showed a wide range of results from 2.8 - 26 days (3, 9, 11, 12, 17). Other studies on stone fruits and citrus (10, 21,27) have shown azinphosmethyl residues go through an initial decay then can stay at a given level for several weeks. In two studies involving peach harvesters (19, 20) where the same fields were monitored for consecutive years and application rates remained the same, results from year to year were markedly different. In the first study (19) the second years DFR results were five times higher then the previous year on day 14 at 2.2 ug/cm^2 vs 0.43 ug/cm^2, respectively. In the second study (20) the same orchards were monitored for three years and in the third year the levels were twice those found the previous two years. Averaging 1.3 ug/cm^2 for the three week exposure in the third year with an average of 0.5 ug/cm^2 in the preceding two years.

Knaak (28) developed a model using dermal dose response curves for cholinesterase inhibition, composed from animal models, and field exposure data to calculate a safe level of 3.1 ug/cm^2. This "safe level" was calculated without the presence of oxon residues that were found in only a few samples in this study. Almost all residues were at or below this "safe level" seven days post application. While this "safe level" gave an index of when pesticide exposures might become a

problem they were not developed to be work task or crop specific. During investigations of fieldworker illnesses involving phosalone (29) and methomyl (30) signs and symptoms of exposure occurred at levels below this calculated safe level and the work being performed in a specific crop played the much larger role in pesticide exposures. In one early azinphosmethyl episode (31) there were signs and symptoms of illness to harvesters picking peaches. At that time it was believed azinphosmethyl caused the worker illness but phosalone was also used on some of the orchards and likely contributed to the illness episode. Researchers (18) have observed urinary dialkylphosphate levels of harvesters in peach orchards at less than half those measured earlier in the season. This occurred when trees harvested three weeks before had denser foliage and the later variety grew more vaselike, with an open canopy. This occurred even though workers were exposed to similar leaf residues and exposure days.

Azinphosmethyl has been in use for decades to control the codling moth in pear orchards. No history of systemic illnesses to field workers harvesting pears has been observed. Previous studies (2, 9) have shown that DFR levels could have been as high as those found in this study. Research in stone fruits (21, 32), where DFR levels ranged from 0.5 - 1.75 ug/cm^2, has shown field workers developed depressed cholinesterase levels during the harvest season without clinical symptoms. The harvest season for stone fruits is anywhere from two to six times that of the pear harvest, which is usually less than four weeks. Since DFR residues are variable among fields, workers are likely not exposed to consistently high residues. This may account for the fact there are no reported cases of systemic fieldworker illnesses while harvesting in pears.

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Appendix 1

Azinphosmethyl results

			micrograms/sample		Azinphomethyl DFR
Sample Number	Date	Day	Parent	Oxon	(ug/cm2)
AM01-1005	6/10/95	1	265	*	0.6625
AM01-1006	6/10/95	1	250		0.625
AM01-1007	6/10/95	1	242		0.605
AM01-1017	6/12/95	3	139		0.3475
AM01-1018	6/12/95	3	127		0.3175
AM01-1019	6/12/95	3	92		0.23
AM01-1033	6/15/95	6	86		0.215
AM01-1034	6/15/95	6	46		0.115
AM01-1035	6/15/95	6	84		0.21
AM01-1047	6/21/95	12	32		0.08
AM01-1048	6/21/95	12	23		0.0575
AM01-1049	6/21/95	12	31		0.0775
AM01-1059	6/29/95	20	Analysis not performed, sample checked for presence of oil.		
AM01-1133	7/18/95	19	94		0.235
AM01-1008	6/10/95	1	180		0.45
AM01-1009	6/10/95	1	174		0.435
AM01-1010	6/10/95	1	178		0.445
AM01-1020	6/12/95	3	109		0.2725
AM01-1021	6/12/95	3	121		0.3025
AM01-1022	6/12/95	3	94		0.235
AM01-1036	6/15/95	6	57		0.1425
AM01-1037	6/15/95	6	54		0.135
AM01-1038	6/15/95	6	64		0.16
AM01-1050	6/21/95	12	18		0.045
AM01-1051	6/21/95	12	17		0.0425
AM01-1052	6/21/95	12	17		0.0425
AM01-1060	6/29/95	20	5		0.0125
AM01-1092	7/10/95	5	389		0.9725
AM01-1093	7/10/95	5	427		1.0675
AM01-1094	7/10/95	5	427		1.0675
AM01-1120	7/13/95	14	205		0.5125
AM01-1121	7/13/95	14	193		0.4825
AM01-1011	6/10/95	1	251		0.6275
AM01-1012	6/10/95	1	222		0.555
AM01-1013	6/10/95	1	231		0.5775
AM01-1023	6/12/95	3	174		0.435
AM01-1024	6/12/95	3	163		0.4075
AM01-1025	6/12/95	3	129		0.3225
AM01-1039	6/15/95	6	97		0.2425
AM01-1040	6/15/95	6	85		0.2125
AM01-1041	6/15/95	6	78		0.195
AM01-1053	6/21/95	12	48		0.12
AM01-1054	6/21/95	12	42		0.105
AM01-1055	6/21/95	12	34		0.085
AM01-1061	6/29/95	20	7		0.0175
AM01-1071	7/6/95	1	507	ND	1.2675
AM01-1072	7/6/95	1	469	ND	1.1725
AM01-1073	7/6/95	1	442	ND	1.105
AM01-1085	7/10/95	5	224		0.56
AM01-1086	7/10/95	5	240		0.6

*Blank cell for oxon level indicates no analysis performed
ND = none detected, limit of detection for the oxon was 5 micrograms per sample.

Appendix 1

Azinphosmethyl results

			micrograms/sample		Azinphomethyl DFR
Sample Number	Date	Day	Parent	Oxon	(ug/cm2)
AM01-1087	7/10/95	5	296		0.74
AM01-1118	7/13/95	14	189		0.4725
AM01-1119	7/13/95	14	130		0.325
AM01-1131	7/18/95	19	74		0.185
AM01-1132	7/18/95	19	84		0.21
AM01-1014	6/10/95	1	369		0.9225
AM01-1015	6/10/95	1	408		1.02
AM01-1016	6/10/95	1	347		0.8675
AM01-1026	6/12/95	3	232		0.58
AM01-1027	6/12/95	3	265		0.6625
AM01-1028	6/12/95	3	202		0.505
AM01-1042	6/15/95	6	134		0.335
AM01-1043	6/15/95	6	131		0.3275
AM01-1044	6/15/95	6	138		0.345
AM01-1056	6/21/95	12	38		0.095
AM01-1057	6/21/95	12	40		0.1
AM01-1058	6/21/95	12	25		0.0625
AM01-1062	6/29/95	20	4		0.01
AM01-1068	7/6/95	1	656	ND	1.64
AM01-1069	7/6/95	1	579	ND	1.4475
AM01-1070	7/6/95	1	659	ND	1.6475
AM01-1082	7/10/95	5	212		0.53
AM01-1083	7/10/95	5	288		0.72
AM01-1084	7/10/95	5	273		0.6825
AM01-1115	7/13/95	14	240		0.6
AM01-1116	7/13/95	14	235		0.5875
AM01-1117	7/13/95	14	176		0.44
AM01-1129	7/18/95	19	52		0.13
AM01-1130	7/18/95	19	47		0.1175
AM01-1074	7/6/95	1	529	ND	1.3225
AM01-1075	7/6/95	1	530	ND	1.325
AM01-1076	7/6/95	1	461		1.1525
AM01-1079	7/10/95	5	327		0.8175
AM01-1080	7/10/95	5	332		0.83
AM01-1081	7/10/95	5	372		0.93
AM01-1124	7/13/95	8	343		0.8575
AM01-1125	7/13/95	8	400		1
AM01-1126	7/13/95	8	344		0.86
AM01-1127	7/18/95	13	213		0.5325
AM01-1128	7/18/95	13	192		0.48
AM01-1095	7/11/95	2	1613	ND	4.0325
AM01-1096	7/11/95	2	1536	ND	3.84
AM01-1113	7/13/95	4	1410	ND	3.525
AM01-1114	7/13/95	4	1559	ND	3.8975
AM01-1139	7/18/95	9	1271	ND	3.1775
AM01-1140	7/18/95	9	1292	5	3.2425
AM01-1107	7/11/95	4	1391	ND	3.4775
AM01-1108	7/11/95	4	1391	ND	3.4775
AM01-1109	7/11/95	5	1324	ND	3.31
AM01-1110	7/11/95	5	1549	ND	3.8725
AM01-1111	7/13/95	7	989	ND	2.4725

*Blank cell for oxon level indicates no analysis performed

ND = none detected, limit of detection for the oxon was 5 micrograms per sample.

Appendix 1

Azinphosmethyl results

			micrograms/sample		Azinphomethyl DFR
Sample Number	Date	Day	Parent	Oxon	(ug/cm2)
AM01-1112	7/13/95	7	1624	ND	4.06
AM01-1137	7/18/95	12	836	ND	2.09
AM01-1138	7/18/95	12	877	ND	2.1925
AM01-1105	7/11/95	1	1613	ND	4.0325
AM01-1106	7/11/95	1	1585	ND	3.9625
AM01-1134	7/18/95	8	1179	ND	2.9475
AM01-1135	7/18/95	8	625	ND	1.5625
AM01-1136	7/18/95	8	1091	ND	2.7275
AM01-1099	7/11/95	2	1081	ND	2.7025
AM01-1100	7/11/95	2	1671	ND	4.1775
AM01-1142	7/18/95	9	433		1.0825
AM01-1101	7/11/95	6	1045	ND	2.6125
AM01-1102	7/11/95	6	1289	ND	3.2225
AM01-1158	7/20/95	15	253	ND	0.6325
AM01-1159	7/20/95	15	253	ND	0.6325
AM01-1097	7/11/95	1	679	ND	1.6975
AM01-1098	7/11/95	1	1360	ND	3.4
AM01-1141	7/18/95	8	1222	ND	3.055
AM01-1103	7/11/95	5	1089	ND	2.7225
AM01-1104	7/11/95	5	1008	ND	2.52
AM01-1065	6/29/95		1748	8	4.39
AM01-1155	7/20/95		1563	7	3.925
AM01-1156	7/20/95		1211	7	3.045
AM01-1063	6/29/95		1409	ND	3.5225
AM01-1157	7/20/95		1693	7	4.25
AM01-1064	6/29/95		1256	ND	3.14
AM01-1151	7/20/95		973	ND	2.4325
AM01-1152	7/20/95		1156	ND	2.89
AM01-1143	7/20/95		123		0.3075
AM01-1144	7/20/95		83		0.2075
AM01-1145	7/20/95		123		0.3075
AM01-1146	7/20/95		96		0.24
AM01-1147	7/20/95		225		0.5625
AM01-1148	7/20/95		246	ND	0.615
AM01-1149	7/20/95		145	ND	0.3625
AM01-1150	7/20/95		231	ND	0.5775
AM01-1153	7/20/95		1319	5	3.31
AM01-1154	7/20/95		1157	ND	2.8925
AM01-1066	6/29/95		1119	ND	2.7975
AM01-1067	6/29/95		388		0.97
AM01-1088	7/10/95		1304	ND	3.26
AM01-1089	7/10/95		184		0.46

*Blank cell for oxon level indicates no analysis performed

ND = none detected, limit of detection for the oxon was 5 micrograms per sample.