Task 1R: Review of GUTHIO

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GUTHION

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Colorimetric determination of Guthion residues in plant material, IV. Application to crops containing chlorophyll, J.M. Adams, Mobay Chemical Corporation (Analyses by Analytical Biochemistry Laboratories) April 21, 1964 thru February 16, 1979, Acc. No. 099214, Tab Nos. 13534, 50419, 50420, 50421, 51968, 65579, 65580, 66248, 66267, 66398, 66399, 66524, 67243, 67244, 67245, and 67249.

Procedure/Methodology

A. Colorimetric Procedure

Frozen samples with acetone added were blended at high speed in a Waring blender. Samples were made to volume with distilled water and filtered through fluted filter paper into a separatory funnel. Azinphosmethyl was twice extracted with chloroform and filtered through paper containing Super-Cel. Combined extracts were evaporated to dryness.

A chromatographic column was prepared under constant suction with tamping to remove the air. Successive layers (from bottom) of Super-Cel, Sea Sorb 43 (MgO), Super-Cel:?:1:2, acid washed alumina, and anhydrous Na_2SO_4 were introduced into the column. Isopropyl alcohol was added to wet the column. Residue remaining from chloroform extraction was dissolved in isopropyl alcohol and poured onto an equilibrating column just before first drops of wetting alcohol were eluted. - Additional isopropyl alcohol was used to elute the column contents (many interfering compounds were removed). Effluent was collected and evaporated to dryness.

The residue was dissolved in benzene. (Preparation of standards parallels procedure from this point on.) Azinphosmethyl in the residue was hydrolyzed by the addition of a KOH-isopropyl alcohol mixture followed by HCl and distilled water additions. The solution was poured into a separatory funnel and extracted with benzene (removes carotenoid pigments). Hydrolyzed (aqueous) phase was collected and reduced by the addition of zinc dust. The sample was filtered and divided into two aliquots.

Sodium nitrite was added to each aliquot followed by ammonium sulfamate. A coupling reagent (1% aqueous solution of N-(1-naphthyl) ethylenediamine dihydrochloride) was added to one of the aliquots to correct for residual color in the extract. Both samples were diluted (with water or diluting solution) and timed for full color development.

Optical density (OD) was determined at 550 nm, subtracting the OD of the aliquot without coupling reagent from the OD of the aliquot with coupling reagent.

Recovery experiments were run in which known amounts of azinphosmethyl or its oxygen analog were added to the extracts of various plant materials.

B. Confirmatory Gas Chromatographic Analysis

Some of the reports (Tab Nos. 51968, 66398, 66399, and 67249) contained data from a gas chromatographic confirmatory procedure. Sample preparation was the same as that for the colorimetric procedure up to the step in which azinphosmethyl was hydrolyzed for KOH-isopropyl alcohol. Instead, an aliquot of the sample was dissolved in benzene and transferred to a silica gel column for cleanup. The column was washed with benzene and the eluate was discarded. Acetonitrile in benzene was used to elute azinphosmethyl; the solvent was evaporated and the residue was dissolved in acetone. Aliquots of the acetone solution were analyzed by gas chromatography.

Results

A. Colorimetric Procedure

Depending on the spectrophotometer used (not specified in reports), Evelyn or Beckman DU, the sensitivity of the procedure was reported as 0.3 or 0.1 ppm, respectively, calculated as that concentration of azinphosmethyl that produced an OD of 0.1. Recoveries are summarized in Table 1 for the various plants and rates of addition tested. For azinphosmethyl, recoveries ranged from 55% (turnip roots) to 134% (radish roots) with a mean value of 90.4 ± 36.8% (confidence limits at p = .05). The oxygen analog of azinphosmethyl had lower recovery values in general, ranging from 61% (snap bean vines) to 106% (radish roots) with a mean value of 74.6 ± 48.2% (confidence limits at p = 0.05).

B. Confirmatory Gas Chromatographic Procedure

A comparison of the gross residue concentrations found by the colorimetric and GC procedures is given in Table 2. Although the sensitivity of the GC procedure was not stated, values <0.05 ppm and in some cases <0.01 ppm were reported, generally indicating 0.01 ppm as the sensitivity of the procedure. However, the data show recovery values of the GC method ranging from 62% (wheat straw) to 134% (wheat grain), thus implying a much lower sensitivity in fact.

Table 1. Recoveries of Guthion and the oxygen analog.

Report No.	Sample	ppm added	Percent Recovery	
			Guthion	Guthion P=O
13534	Cabbage			
"	Broccoli	0.2-0.5	99.4[a]	91.8[b]
"	Cauliflower			
50419	Snap beans, beans	0.1	95	88
"	vines	0.1	96	61
67243	beans	0.1	96	--[c]
"	vines	0.1	95	--
50420	Wheat, green forage	0.2	90	67
67244	green forage	0.1	91	-
50421	Sorghum, green forage	0.2	68	72
"	grain	0.2	83	78
"	straw	0.2	73	63
67245	green forage	0.1	95	--
"	grain	0.1	94	--
"	straw	0.1	96	--
65579	Peas, in pod	0.1	98	91
"	vines	0.1	88	81
65580	Radishes, roots	0.1	89	89
"	tops	0.1	98	94
66267	roots	0.3	134	106
"	roots	2.0	90	--
66248	Turnips roots	2.0	131	--
"	roots	0.3	55	63
"	tops	2.0	73	65
66524	Corn, forage	0.3	67	64
"	kernel	0.2	80	76

[a] For cole crops percent recovery is the average of three crops, four treatment levels; the range was 85-116%.

[b] As in (a); range was 69-133%.

[c] Not analyzed.

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Table 2. Comparison of colorimetric and gas chromatographic procedures.

Sample	ppm added	Compound	Gross residues, ppm O.D.	Gross residues, ppm G.C.
Snap beans, vine	2.0	Guthion	1.77	1.88
Snap beans, vine	2.0	Guthion P=0	1.70	2.00
Snap beans, beans	0.3	Guthion	0.66	0.26
Snap beans, beans	0.3	Guthion P=0	0.66	0.26
Carrot, tops	2.0	Guthion	1.65	1.66
Carrot, tops	2.0	Guthion P=0	1.60	1.97
Apples	1.0	Guthion	0.99	1.01
Apples	1.0	Guthion P=0	0.87	0.85
Pears	1.0	Guthion	1.11	0.94
Pears	1.0	Guthion P=0	1.16	0.96
Cherries	1.0	Guthion	0.96	0.92
Cherries	1.0	Guthion P=0	0.87	1.06
Wheat, green forage	2.0	Guthion	1.83	2.0
Wheat, grain	0.2	Guthion	0.30	0.17
Wheat, straw	2.0	Guthion	1.64	1.24
Sorghum, green forage	2.0	Guthion	1.0	1.36
Sorghum, grain	0.5	Guthion	0.70	0.67

Conclusions

Whereas the sensitivity of the instruments may be 0.10 ppm, both the GC and colorimetric procedures are plagued with interferences that render them relatively insensitive in recovering azinphosmethyl or azinphosmethyl P=O residues from plant samples (±20%). This lack of sensitivity was apparent throughout the range of spiking (0.1-2.0 ppm). Recoveries of the oxygen analog and parent compound were low with mean recoveries of 75 and 90%, respectively.

Determination of Baygon, Baytex, Bolstar, Croneton, Dasanit, Di-syston, Dylox, Guthion, Hinosan, Mesurol, Metasystox-R, Monitor, Morestan, Nemacur; and Systox residues in soils, R.A. Morris, Mobay Chemical Corp., July 28, 1977, Acc. No. 099216 and 099214, Tab No. 49675.

Procedure

Field plots were treated with a mixture of Bylox, azinphosmethyl (Guthion; Mobay Chemical Corp.; formulation and purity not specified), Hinosan, Morestan, and Systox. An additional set of plots were treated with a mixture of Baygon, Bolstar, Mesurol, Metasystox-R, Monitor, and Nemacur. Soil samples were collected after treatment (length of time not specified).

Methodology

The soil samples were refluxed with methanol, filtered, and evaporated until only the aqueous layer remained. The aqueous layer was transferred to a separatory funnel, diluted, and partitioned with chloroform. The chloroform layer was removed, mineral oil in benzene was added, and the solution was evaporated to dryness. The residue was dissolved in acetone and evaporated to dryness.

The residues from samples to be analyzed for azinphosmethyl were dissolved in benzene and were then hydrolyzed, reduced, and analyzed as described in Acc. No. 099216, Tab No. 13517. The residue from samples to be analyzed for Bolstar were oxidized and analyzed as described in Acc. No. 099216, Tab No. 45356.

Results

The recovery data for azinphosmethyl and Bolstar are presented in Acc. No. 099216, Tab Nos. 66363 and 54443, respectively.

Conclusions

This method is capable of determining azinphosmethyl and Bolstar residues in soil. However, the method sensitivity and recovery data for azinphosmethyl were not presented.

Recovery of Guthion from soil, Analytical Biochemistry Laboratories, March 5, 1979, Acc. No. 099216 and 99214, Tab No. 52901.

Procedure

Silty clay loam soil samples were spiked with azinphosmethyl and extracted as described in Acc. No. 099216, Tab No. 49675.

Methodology

The dried extract was dissolved in benzene. An aliquot of the benzene solution was eluted through a silica gel chromatography column with benzene and two portions of acetonitrile in benzene. The acetonitrile-in-benzene eluates were each evaporated to dryness, dissolved in acetone and analyzed by gas chromatography (GC).

The remainder of the benzene solution was analyzed as described in Acc. No. 099216, Tab No. 13517.

Results

With the method in Tab No. 13517, the recovery rates for azinphosmethyl and its oxygen analog were 110-130% and 72-87%, respectively. The recovery rates for azinphosmethyl and its oxygen analog with the GC method were 89 and 77%, respectively.

Conclusions

This method is capable of determining azinphosmethyl residue levels in soil. However, the minimum detection limit for the method was not presented.

Recovery of Guthion from soil, Analytical Biochemistry Laboratories, July 20, 1978, Acc. No. 099214, Tab No. 66363.

Procedure

Samples of a sandy soil were treated with azinphosmethyl or the oxygen analog of azinphosmethyl at 0.20 or 0.50 ppm.

Methodology

The samples were placed in Soxhlet thimbles and extracted with petroleum ether and acetone. The extracts were analyzed with the spectrophotometric method described in Acc. No. 099216, Tab No. 49675.

Results

The method sensitivity was 0.20 ppm. The recovery rates for azinphosmethyl and its oxygen analog were 100-107% and 90-100%, respectively.

Conclusions

This method is capable of detecting and quantifying azinphosmethyl and azinphosmethyl oxygen analog residue levels equal to or greater than 0.20 ppm.

Soil persistence residue experiment VBL-707-76H, Vero Beach Laboratories and Analytical Biochemistry Laboratories, July 14, 1978, Acc. No. 099214, Tab No. 66286.

Procedure

Azinphosmethyl (Guthion 50 WP) was applied to a sandy soil (92% sand; 1% silt; 7% clay; organic matter content 0.8%; pH 5.9; CEC 1.1 meq/100 g soil) at rates of 1, 2, 4 or 8 lb ai/A. Soil samples were collected at 0-6 inches at 29, 123, 364, and 467 days after treatment.

Methodology

The samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 49675.

Results

Azinphosmethyl residue levels of <0.10, 0.14, 0.10, and 0.41 ppm were found at depths of 0-6 inches 29 days after treatment at 1, 2, 4, and 8 lb/A, respectively. These residue levels declined to levels at or below 0.11 ppm by 123 days after treatment (Table 1).

Table 1. Dissipation of azinphosmethyl in a sandy soil sampled to a depth of 0-6 inches.

Application rate (lb/A)	Days after treatment	Residues (ppm)
1	29	<0.10
	123	<0.10
	364	<0.10
	467	<0.10
2	29	0.14
	123	<0.10
	364	<0.10
	467	<0.10
4	29	0.10
	123	<0.10
	364	0.12
	467	<0.10
8	29	0.41
	123	0.11
	364	<0.10
	467	<0.10

Conclusions

Azinphosmethyl dissipates in a sandy soil, with residue levels at or below 0.11 ppm at 123 days after treatment at 1-8 lb/A. Residue decline with time is evident in sandy soil.

It is not known if some residues leach beyond the 6-inch sampling depth.

Soil persistence study STF-706-76H, Mobay Research Center and Analytical Biochemistry Laboratories, January 17, 1979, Acc. No. 099214, Tab No. 67115.

Procedure

A silty clay loam (8% sand; 62% silt; 30% clay; organic matter content 3.2%; pH 6.7; CEC 12 meq/100 g soil) field plot was treated with azinphosmethyl (Guthion 50 WP) at 1, 2, 4, and 8 lb ai/A. Soil samples were taken to a depth of 6 inches at 32, 120, and 364 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 49675.

Results

At 32 days after treatment azinphosmethyl residue levels of 0.40, 0.71, 0.67 and 1.93 ppm were found in plots treated at 1, 2, 4, and 8 lb/A, respectively. At 120 days after treatment higher residue levels were detected, and at 364 days after treatment lower residue levels were detected (Table 1).

Table 1. Dissipation of azinphosmethyl in a silty clay loam soil sampled to a depth of 6 inches.

Application rate (lb ai/A)	Days after application	Residues (ppm)
1	32	0.40
	120	0.63
	364	0.50
2	32	0.71
	120	1.09
	364	0.55
4	32	0.67
	120	0.75
	364	0.58
8	32	1.93
	120	2.25
	364	0.80

Conclusions

The residue level detected in the control sample was 0.40 ppm, indicating that either azinphosmethyl was present in the field plot prior to treatment or an error was made in the analysis of the soil samples. In either case the data are invalid.

Guthion residues in field rotational crops, R.R. Gronberg and R.A. Morris, Mobay Chemical Corporation, analyzed by Analytical Biochemistry Laboratories Feb. 19, 1979, Acc. No. 099214, Tab Nos. 67116-67179 and 67271 (procedure and results).

Procedure

Rotational field crop studies were run at two locations: Stanley, Kansas (silty clay loam: 8% sand; 62% silt; 30% clay; pH 6.7; organic matter 3.2%; CEC 12 meq/100 g) and Vero Beach, Florida (sand: 92% sand; 1% silt; 7% clay; pH 5.9; organic matter 0.8%; CEC 1.1 meq/100 g). The plots were 50 feet long, 1-4 rows/plot depending on the crop. The plots were cultivated before Guthion 50 WP was applied as a broadcast spray in water to the surface of the bare soil and the plots were fallowed until planting of rotational crops. The treatment rates were 1, 2, 4, and 8 lb ai/A.

Prior to planting, the soil was cultivated to a 6-inch depth. At approximately 30-, 60-, 90-, and 365-day intervals postapplication, typical root, grain, and leafy vegetable crops were planted. At the Stanley, Kansas, location, carrots (radishes substituted at 120- and 365-day intervals), sorghum (wheat substituted at the 120-day interval), and snap beans were planted at each interval. At the Vero Beach, Florida location, corn, turnips, and black-eyed peas were planted at each interval. Plant sampling occurred at normal maturity. Samples were immediately frozen and stored frozen until analysis.

Methodology

The analytical method used was the colorimetric one described in Tab No.13534, which determines the combined levels of azinphosmethyl and its oxygen analog. The GC method described in Tab No. 51968 was used for reanalysis of some samples, a method that distinguishes between azinphosmethyl and its oxygen analog.

Results

Results found using the colorimetric procedure are shown in Table 1 (except for 365 days postapplication). No azinphosmethyl residues were detected (<0.1 ppm) in sorghum, wheat, or corn at any application rate or interval posttreatment. Of the leafy greens, turnip tops, radish tops, peas, and snap beans showed no residues (<0.1 ppm) except at the highest application rate (8 lb/A), 0.16 ppm was found in radish tops 120 days postapplication (Tab No. 67127) and 0.15 ppm was found in pea vines 30 days postapplication (Tab No. 67168). The radish roots (grown in silty clay soil 120 and 365 days postapplication) contained residues ranging from 0.15 to 0.55 ppm (Tab Nos. 67126-67130). Values for the carrots grown at that location 30, 50, and 90 days postapplication were not reported. 'Shogoin' turnip roots showed residue levels ranging from 0.53 to 2.18 ppm (Tab Nos. 67154-67159) in no consistent pattern following application rate or time. The 'Purple top' turnip roots also contained azinphosmethyl residues, ranging from 0.18 to

0.57 ppm (no controls were available, so these values may have been lower) (Tab Nos. 67160-67167). No residues (<0.1 ppm) were detected in any of the above-mentioned samples when reanalyzed by the GC method.

Table 1. Guthion residues in field rotational crops planted 30, 60, 90, and 120 days following application of Guthion 50 WP.

Crop group	Crop type		Guthion equivalent residue (ppm)															
			1 lb ai/A (days postapplication)				2 lb ai/A (days postapplication)				4 lb ai/A (days postapplication)				8 lb ai/A (days postapplication)			
			30	60	90	120	30	60	90	120	30	60	90	120	30	60	90	1
Grain	Sorghum Corn	Grain Kernel	-[a] <0.1	- -	- <0.1	- -	- <0.1	- -	- <0.1	- -	- <0.1	<0.1 -	- <0.1	- -	<0.1 <0.1	<0.1 -	- <0.1	
	Sorghum Corn	Forage Forage	- <0.1	- -	- <0.1	- <0.1	- <0.1	- -	- <0.1	- <0.1	<0.1 <0.1	<0.1 -	- <0.1	<0.1[b] <0.1	<0.1 <0.1	<0.1 -	- <0.1	<C <C
	Sorghum	Straw	-	-	-	-	-	-	-	-	<0.1	<0.1	-	-	<0.1	<0.1	-	
Pod vegetable	Bean Pea	Bean/pod Pea/pod	- <0.1	- -	- -	- -	- <0.1	- -	- -	- -	<0.1 <0.1	<0.1 -	- <0.1	- -	<0.1 <0.1	- -	- <0.1	
	Bean Pea	Vine Vine	- <0.1	- -	- <0.1	- -	- <0.1	- -	- <0.1	- -	<0.1 <0.1	<0.1 -	- <0.1	- -	<0.1 0.14	<0.1 -	- <0.1	
Leafy vegetable	Radish Turnip	Tops Tops	- <0.1	- -	- <0.1	- <0.1	- <0.1	- -	- <0.1	- <0.1	- <0.1	- -	- <0.1	<0.1 <0.1	- <0.1	- -	- <0.1	<
Root	Radish Turnip	Root Root[d]	- 2.18	- -	- 0.72	- <0.1	- 0.96	- -	- 0.73	- <0.1	- -	- -	- 0.64	- <0.1	- -	- -	- 0.53	<

[a] Not available/not analyzed.

[b] Wheat forage.

[c] These residues were reported to be <0.05 ppm when reanalyzed by GC.

[d] All residues reported in this row were found to be <0.1 ppm by GC; values reported are for 'Shogoin' cultivar.

Conclusions

Approximately 50% of the data generated were summarized as "not available/not analyzed." No data were submitted for carrots, the relatively deep root crop grown 30, 60, and 90 days postapplication at the Kansas location. Because residues were found in the other root crops, this is a crucial omission. The potential residue hazard for rotational crops may be regarded as unlikely for grain and aboveground vegetable crops, but residues approaching 1 ppm of a combination of azinphosmethyl and its oxygen analog may accumulate in root crops planted up to 90 days after azinphosmethyl application.

A colorimetric method for the determination of Guthion residues in soil, T.J. Olson, Chemagro Corporation, April 21, 1964, Acc. No. 099216,Tab No. 13517.

Procedure

Samples of clay, sand and muck soils were spiked with azinphosmethyl (formulation and purity not specified) or with the oxygen analog of azinphosmethyl.

Methodology

Each spiked sample was placed in a jar and extracted with water and acetone by tumbling. The solution was filtered and the filter cake was washed with acetone. The combined filtrates were added to a separatory funnel containing chloroform and shaken. The chloroform layer was removed and filtered through anhydrous Na_2SO_4. The extraction of the filtrate was repeated with an additional volume of chloroform. The combined chloroform extracts were evaporated to dryness.

The residue was dissolved in benzene and mixed with KOH-isopropyl alcohol solution. To the mixture in a separatory funnel was added HCl, distilled water, and benzene. After shaking, the aqueous phase was removed and diluted.

Zinc dust was added to the aqueous phase and the solution was filtered. To each aliquot of filtrate, $NaNO_3$ was added and the solution was mixed. Then $NH_4NH_2SO_3$ reagent was added and the solutions were mixed. A coupling reagent was added to one aliquot. Each aliquot was diluted with water, mixed and allowed to stand. The absorbance was determined spectrophotometrically at 550 nm.

Results

The method sensitivity was 0.20 ppm. The recovery rates for azinphosmethyl and the oxygen analog of azinphosmethyl were 76-86% and 72-81%, respectively.

Conclusions

This analytical method is capable of detecting azinphosmethyl and azinphosmethyl oxygen analog residues at levels of $\geq$0.2 ppm in soil.

Determination of residues of DEF in soil by thermionic emission gas chromatography, J.S. Thornton, Mobay Chemical Corp., August 23, 1966, Acc. No. 099216, Tab No. 18713.

Procedure

Silt loam and clay soil samples were spiked with DEF at 0.10 ppm

Methodology

The soil was blended with chloroform and filtered. The filtrate was evaporated and the residue was dissolved in benzene. Aliquots of the benzene solution were analyzed by gas chromatography.

Results

The method sensitivity was 0.10 ppm. The recovery rates for DEF in silt loam and clay soils ranged from 101 to 118% and 99 to 122%, respectively. The average recovery was 111% for both soils.

Conclusions

This method is capable of determining DEF residue levels of 0.10 ppm in soil.

A gas chromatographic method for the determination of Bay-NTN-9306 and metabolites in soil, S.K. Kurtz and F.E. Sandie, Mobay Chemical Corp., October 20, 1975, Acc. No. 099216, Tab No. 45356.

Procedure

Soil samples (silt loam, clay loam, loam, clay, muck, and sand) were treated with Bay NTN 9306 and stored under frozen conditions.

Methodology

The soil samples were refluxed with methanol, filtered and evaporated to dryness. The residue was dissolved in methylene chloride and n-chloroperbenzoic acid in methylene chloride was added. The solution was allowed to stand, transferred to a separatory funnel containing $Ba(OH)_2$, and shaken. The partitioning with $Ba(OH)_2$ was repeated and the lower phase was eluted through a column of Na_2SO_4. The eluate was then evaporated to dryness. The residue was dissolved in acetone and analyzed by gas chromatography.

Results

The sensitivity of the method was 0.01 ppm. The recovery rates were: Bay-NTN-9306, 79-111%; sulfoxide, 104-111%; sulfone, 128%; oxygen analog, 114-124%; oxygen analog sulfoxide, 90-111%; and oxygen analog sulfone, 82-122%.

Conclusions

This method is capable of determining residues of Bay NTN 9306 and its metabolites of 0.01 ppm in soil. This method converts Bay NTN 9306 and five metabolites (the sulfoxide, sulfone, oxygen analog, and oxygen analog sulfoxide) to the oxygen analog sulfone of Bay NTN 9306.

Leaching characteristics of Guthion on aged soil, S. Atwell and C. Close, Chemagro Agricultural Division, April 30, 1976, Acc. No. 099216, Tab No. 48466.

Procedure

A silt loam soil (3.0% sand, 75% silt, 22% clay, organic matter content 2.3%, and pH 6.4) was treated at a rate of 1 ppm with ring-labeled [^{14}C] azinphosmethyl. The treated soil was aged moist under aerobic conditions for 28 days, then allowed to dry.

Dry untreated soil was packed in a column and saturated with water. The aged, treated soil was added to the top of the untreated column and leached over a period of 45 days.

Methodology

Samples of the aged soil were refluxed with methanol and filtered. Aliquots of the extract were subjected to thin-layer chromatography (TLC) and radio scanning. Samples of the extracted soil were stirred with NaOH and centrifuged. The supernatant was decanted and the extraction was repeated twice. The soil was then washed with water and dried, yielding the humin fraction.

The combined supernatant and water washes were assayed for radioactivity and then acidified to pH 1 (humic acid fraction precipitates). The supernatant (fulvic acid fraction) was decanted and assayed for radioactivity in a Tri-Carb scintillation spectrometer.

The leachate fractions were extracted with an acetone:chloroform mixture, acidified with HCL, and reextracted with chloroform. The neutral and acidic extracts were subjected to TLC and radiochromatogram scanning. Samples of the leached soil were analyzed for radioactivity in a Tri-Carb oxidizer.

Results

After aging and prior to elution, half of the extractable radioactivity was still in the form of azinphosmethyl and 62% of the radioactivity was soil-bound (Table 1).

After leaching, the top 2 inches of the silt loam soil contained 90% of the applied radioactivity (Table 2). Of the 4.4% of the applied radioactivity present in the leachate, 12.1% was organosoluble and did not contain azinphosmethyl.

Table 1. Distribution of radioactivity in a silt loam soil treated with ring-labeled [^{14}C] azinphosmethyl and aged aerobically for 28 days.

Soil fraction	Percent of applied radioactivity
Extractable	
Azinphosmethyl	19.0
Benzazimide	5.3
Unidentified	13.7
Soilbound	
Humic acid fraction	50.1
Fulvic acid fraction	10.5
Humin	1.4

Table 2. Distributuion of ring-labeled [^{14}C] azinphosmethyl in a silt loam soil column eluted with the equivalent of 14 acre-inches of water.

Soil depth (inches)	Percent of applied radioactivity
0-2	90.0
2-4	2.8
4-6	1.2
6-8	0.6
8-10	0.5
10-12	0.5
Leachate	4.4
Total	100.0

Conclusions

Soil aged residues of azinphosmethyl do not leach. About half of the applied azinphosmethyl is found in the humic acid fraction of soil after 28 days of aging.

The stability of Guthion in silt loam soil under frozen storage, C.L. Close, Mobay Chemical Corp., May 6, 1976, Acc. No. 099216, Tab No. 48473.

Procedure

A silt loam soil (3.0% sand; 75.0% silt; 22.0% clay; organic matter content 2.3%; pH 6.4) was treated with ring-labeled [^{14}C]azinphosmethyl, mixed, and stored at -10 C.

Methodology

After removal from storage the sample was refluxed with acidified methanol. The methanol fraction was concentrated and subjected to thin-layer chromatography for identification. Also, an aliquot of the concentrated extract was diluted with water, mixed with a scintillation solution, and assayed for radioactivity with a Tri-Carb scintillation spectrometer.

Results

After storage for 93 days at -10 C, 100% of the applied radioactivity was recovered with 97% recovered as azinphosmethyl.

Conclusions

Radiolabeled azinphosmethyl is stable and can be quantitatively recovered from a silt loam soil stored at -10 C for 93 days.

Soil thin-layer mobility of twenty four pesticide chemicals, J.S. Thornton J.B. Hurley, J.J. Obrist, Mobay Chemical Corp., December 15, 1976. Acc. No. 099216, Tab No. 51016.

Procedure

A total of 6 soils (Table 1) were air dried and passed through 250 or 420 µm sieves (the larger used for sandy soils). Thin layer chromatography (TLC) was used to analyze the 6 soil types to determine the leachability of 24 different radiolabeled pesticides, including azinphosmethyl. After chromatography was performed, the TLC plates were air dried and placed in contact with X-ray film for 5 days. The R_f values for the different pesticides (leached through the different soil plates) were calculated from the developed film.

Methodology

In the soil TLC method, sieved soils were individually mixed with distilled water and spread on glass TLC plates. The prepared plates were air dried at least 24 hours. Approximately 0.014 µCi of the various pesticides were spotted on the prepared plates with either methanol or benzene as a solvent. The solvent was allowed to evaporate. Water was permitted to rise 10 cm up the plates, then the plates were equilibrated for 10 minutes, air dried, and exposed to X-ray film for 5 days.

Results

The leaching R_f values of azinphosmethyl are presented in Table 2. Triplicate analyses resulted in a standard error of ± 0.02 R_f value.

Table 1. Textural characteristics of soils used in leaching studies.

Soil type	Origin	Sand %	Silt %	Clay %	Organic Matter %	pH
Agricultural sand[1]	Vero Beach, FL	92	1	7	0.8	5.9
Sandy loam[1]	Merril, OR	74	14	13	2.8	6.6
Sandy clay loam[1]	Howe, IN	56	21	23	0.6	5.5
Silt loam[2]	Concord, NE	18	57	25	5.1	7.9
Silty clay[2]	Hagerstown, MD	4	53	43	2.1	6.7
Silty clay[2]	Stanley, KS	0	41	59	0.5	6.0

[1]Passed through 420 μm screen.

[2]Passed through 250 μm screen.

Table 2. Azinphosmethyl R_f values[a]

Agric sand	Sandy loam	Sandy Clay loam	Silty loam	Silty clay (pH 6.7)	Silty clay (pH 6.0)	Average of all soils tested
0.18	0.22	0.11	0.18	0.14	0.24	0.18

[a]Frontal R_f values used. For each soil, value given is the average of at least 3 replicate determinations.

Conclusions

Azinphosmethyl exhibits a comparatively low (class 2) leachability potential in all 6 soils tested. Its average R_f value of 0.18 was lower than all but 7 of 24 pesticides tested. Azinphosmethyl was most mobile in Stanley silty clay soil (R_f 0.24) and least mobile in the sandy clay loam soil (R_f 0.11).

Effects of Guthion on nitrification and denitrification in soil. S.H. Atwell, Mobay Chemical Corp., January 27, 1978, Acc. No. 099216, Tab No. 54433.

Procedure

A loamy sand soil (76% sand; 18% silt; 6% clay; organic matter content 0.8%; pH 6.4; water holding capacity 2.9 ml/10 g soil; CEC 7.0 meq/100 g soil) was sieved to 2 mm, air dried, and placed in jars. The soil was fortified with azinphosmethyl at either 2 or 20 ppm.

The nitrification samples (control and treated) were amended with $(NH_4)_2SO_4$ and moistened to 60% of water holding capacity. The jars were then capped and incubated. The denitrification samples (control and treated) were amended with glucose and $Ca(NO_3)_2$, and flooded. The jars were then capped and incubated in a CO_2 and H_2 atmosphere.

Methodology

After incubation, duplicate samples of control and treated soils were mixed with KCl and filtered. An aliquot of the filtrate was analyzed for ammonium nitrogen and nitrate+nitrite by a steam distillation Kjeldahl method using MgO and MgO+Devarda's alloy, respectively. Each distillate was collected in boric acid indicator and titrated with H_2SO_4.

Results

Less nitrification occurred in the azinphosmethyl-treated samples than in the controls during the first 7 days after treatment. However, between 7 and 28 days after treatment the nitrification rates in treated and control samples were similar (Table 1).

Denitrification was not properly defined in this study and therefore no denitrification data are available (see Conclusions).

Table 1. Rate of nitrification in a loamy sand soil treated with azinphosmethyl.

Incubation period (days)	Nitrification[a] (NH_4^+:NO_3^- + NO_2^-)		
	Control	2 ppm	20 ppm
0	100:0	100:0	100:0
7	82:18	88:12	88:12
14	86:14	86:14	86:14
21	80:20	80:20	76:24
28	85:15	87:13	86:14

[a] Percent of recovered nitrogen.

Conclusions

Treatment of a loamy soil with azinphosmethyl at 2 or 20 ppm ai slightly inhibits nitrification under aerobic conditions during the first 7 days after treatment. After 7 days approximately normal nitrification rates occur. Since the rates used in this study were 1-10 X the field rate, nitrification will not be affected by recommended use rates of azinphosmethyl.

The data reported as denitrification were documenting the microbial process of assimilation. Denitrification is the process whereby NO_3^- is converted to gaseous N_2 or N_2O and thus lost from the soil. Assimilation is the reverse of the nitrification process (oxidized nitrogen forms are assimilated back to reduced, organic nitrogen forms).

Recovery of Bolstar in soil, Analytical Biochemistry Laboratories, February 2, 1978, Acc. No. 099216, Tab No. 54443.

Procedure and Methodology

The procedure for the treatment of the silty clay loam soil with Bolstar and the analytical method for recovery of Bolstar from soil are presented in Acc. No. 099216, Tab No. 49675.

Results

The recovery rates for Bolstar and its oxygen analog from soil were 94 and 124%, respectively. The method was capable of detecting Bolstar residues of 0.05 ppm in soil.

Conclusions

Bolstar residue levels equal to or exceeding 0.05 ppm can be accurately detected in soil.

Recovery of DEF from soil, Mobay Chemical Corp., March 27, 1978, Acc. No. 099216, Tab. No. 65369.

Procedure

Sandy loam and silt loam soil samples were spiked with DEF at 0.10 ppm.

Methodology

The soil samples were Soxhlet extracted with a methylchloride:methanol solution and filtered. The filtrate was evaporated and analyzed with the method described in Acc. No. 099216, Tab. No. 18713.

Results

The method sensitivity was 0.10 ppm. The average recovery rates for the silt loam and sandy loam soils were 90 and 94%, respectively.

Conclusions

This method is capable of determining DEF residue levels of 0.10 ppm in soil.

Effects of Guthion on nitrogen fixation, S.H. Atwell, Chemagro Agricultural Division, March 15, 1978, Acc. No. 099216, Tab No. 65613.

Procedure

Soybean seeds inoculated with Rhizobium japonicum were planted in pots containing perlite. The control pots were irrigated with a minus-nitrogen nutrient solution. The treated pots were irrigated with a minus-nitrogen nutrient solution plus 2 ppm ai azinphosmethyl (Guthion 50 WP, Mobay Chemical Corporation). All of the soybeans were grown under greenhouse conditions and harvested 4 weeks after treatment.

Growth differences between the control and treated soybeans were determined by measuring the number of plants nodulated, shoot length, plant fresh weight, and nodule fresh weight.

Methodology

Each pot of soybeans was analyzed separately with the acetylene reduction method. The plants were removed from the pot and placed in a glass container fitted with a septum stopper. Air was withdrawn from the sealed container with a syringe and an equal volume of acetylene was injected. At the end of the 30-minute incubation period, aliquots of the gas were withdrawn with syringes and injected into a gas chromatograph.

Results

There was no significant difference in the nitrogen-fixing ability of treated and control soybeans (0.05 level of confidence, Student's "t" test). (Table 1.) All of the soybean plants nodulated.

Table 1. Effect of azinphosmethyl on nitrogen fixation and the growth of soybeans.

Treatment	Average total C_2H_4 produced/plant (nmol)	Average shoot length/plant (nm)	Average fresh weight/plant (g)	Average nodule weight/plant (g)
Control	2,175±642	207±25	6.51±0.14	0.44±0.04
2.0 ppm	1,985±415	200±18	6.68±0.46	0.46±0.02

Conclusions

The treatment of soybeans (inoculated with Rhizobium japonicum) with 2 ppm ai azinphosmethyl does not inhibit nitrogen fixation or the growth of soybeans.

Effect of Guthion on isolated soil microorganisms, R.G. Minor and K.J. Strankowski, Mobay Chemical Corp., July 14, 1978, Acc. No. 009216, Tab No. 66401.

Procedure/Methodology

Bacteria and Actinomycetes

Suspensions of Bacillus subtilis, Cellulomonas flavigena, Pseudomonas aeruginosa, and Streptomyces scabies in Difco nutrient broth were incubated. Aliquots of the cell suspensions were plated with Difco nutrient agar. Paper discs treated with azinphosmethyl at 2, 10, 100, 1,000, or 10,000 ppm were placed in the petri dishes. The cultures were incubated for 16-64 hours and the zones of inhibition were measured. All analyses were run in triplicate.

Fungi

Spores of Aspergillus niger, Penicillium daleae, Trichoderma viride, and Phycomyces nitens were plated with potato dextrose agar and incubated. Fungus discs were removed and placed in potato dextrose agar treated with azinphosmethyl at 2, 10, 100, 1,000, or 10,000 ppm and incubated. Controls were run using untreated agar.

The growth of the fungus discs was measured and compared to controls. All analyses were run in duplicate.

Results

Bacteria and Actinomycetes

Azinphosmethyl at concentrations of 2-10,000 ppm did not inhibit any of the bacteria or actinomycetes tested.

Fungi

Azinphosmethyl at 2 and 10 ppm inhibited the growth of the fungi tested by 4-18%. At 100, 1,000, and 10,000 ppm, the fungi were inhibited 22-61%, 44-79% and 94-100%, respectively (Table 1).

Table 1. Effect of azinphosmethyl on the growth of various fungi.

Species	Percent inhibition at various concentrations				
	2 ppm	10 ppm	100 ppm	1,000 ppm	10,000 ppm
Aspergillus niger	0	6	22	44	94
Penicillium daleae	7	7	33	53	100
Trichoderma viride	4	8	31	69	100
Phycomyces nitens	4	18	61	79	100

Conclusions

Azinphosmethyl at 2-10,000 ppm does not inhibit Bacillus subtilis, Cellulomonas flavigena, Pseudomonas aeruginosa, and Streptomyces scabies.

Azinphosmethyl does not appreciably alter the soil fungal population at concentrations below 10 ppm, but is inhibitory at concentrations of 100 ppm or more.

Identification of Microorganisms in Sandy Loam Soil, L.E. Smedly and D.L. Heplu, ELARS Bioresearch Laboratories, Inc., Fort Collins, Colorado, October 1, 1978, Project No. 1399, Acc. No. 099216, Tab No. 66624.

Procedure

Soil samples from the study (Tab No. 68030) on metabolic fate of azinphos-methyl were analyzed to determine the families of bacteria, actimomycetes, and fungi. Aliquots of sandy loam samples were transferred to culture tubes. Sterile peptone water was added and the cultures shaken. Each sample was diluted 1:500 and 1:50,000, and each dilution plated in triplicate on rose bengal and sodium caseinate plates and then incubated at 30 C. Two Czapeck agar slants were inoculated with 1:500 dilution and incubated anaerobically. If aerial mycelia were found to be present in the sodium caseinate agar, transfer was made to a Czapeck agar slant. If colonies could not be differentiated into actinomycete or bacteria, transfers were made to both Czapeck agar and 'T' soy slants.

Methodology

Hypomycetes grown on rose bengal agar were observed for mycellium and spore appearance, color, and distribution. Family identification was made of blue stained colonies by microscopic and colonial morphology.

Actinomycetes pigments were observed on Czapeck agar slants. Colonies were gram stained and mycelial, sporophore, and hyphae characteristics identified. When necessary, Kinyoun acid fast stain was used to distinguish Nocardiaceae.

Bacteria from 'T' soy slants were gram stained. Gram reaction, microscopic morphology, and colonial morphology were used to identify family types. When necessary, other tests were used (such as oxidase, catalase, indol, and gelatin).

Results

Classification was determined by the Hughes system for Hyphomycetes. Table 1 presents the families of microbes found for hyphomycetes, actinomycetes and bacteria. For some families, more than one genus was detected.

Table 1. Identification of microorganisms in soil.

Hyphomycetes (Hughes classification)	
Aleurosporae (3)[a]	
Philiasporae (5)	
Blastosporae (2)	
Actinomycetes (Bergey's classification)	
Streptomycetacea (4)	
Nocardiaceae (2)	
Dermatophilaceae (1)	
Actinoplanaceae (2)	
Micromonosporaceae (1)	
Bacteria (Bergey's classification)	
Coryneform group (3)	
Micrococcaceae (2)	
Neisseriaceae (1)	
Bacillaceae (2)	
Nitrobacteraceae (1)	
Enterobacteraceae (1)	

[a] Numbers in ( ) indicate genera found for each family.

Conclusions

The distribution of organisms found in the soil samples is not unusual. The presence of Nocardiaceae may be explained by the presence of some roots in the soil sample.

It was not explicitly stated that sterile control samples were analyzed for soil microbes; however, results in study (Tab No. 68030) indicate that azinphosmethyl degradation proceeded much more slowly in the sterile soil samples. The normal distribution of organisms found in the azinphosmethyl-treated soils indicates that the pesticide is not completely inhibitory to the growth of any of these representative soil microbes at the 2 ppm concentration tested. Since quantitative data was not presented for untreated soil samples no conclusions can be made concerning the ability of azinphosmethyl to partially inhibit or stimulate the growth of those organisms tested.

Soil Adsorption and Desorption of Guthion, M.F. Lenz, Mobay Chemical Corp., April 11, 1979, Acc. No. 099216, Tab. No. 66848.

Procedure

Adsorption

Samples (2 gram) of sandy loam, silt loam, and silty clay soils (for characteristics see Table 1) were dried and sieved to 40 mesh. The soil samples were treated with 10 ml of either a 13, 6.5, 1.26, or 0.13 ppm solution of [^{14}C] azinphosmethyl and mixed on a mechnical shaker. Samples were removed from the shaker at 1.5, 3, 4.5, 21.5, and 24 hours after treatment and centrifuged.

Desorption

The treated soil samples were mixed with distilled water as described above for 24 hours and centrifuged. The supernatant was removed for analysis.

This process was repeated three additional times.

Methodology

The supernatants were analyzed with a liquid scintillation spectrophotometer.

Results

The adsorption properties of all three soils were linear (Figure 1). The amount adsorbed ranged from 52 to 87% with relative adsorption decreasing with increasing concentration (Table 2).

The adsorbed azinphosmethyl was desorbed four times from each soil type for each concentration with desorption of azinphosmethyl residues occurring each time. The four desorptions removed between 32 and 68% of the adsorbed residues (Table 2).

The Freundlich constants were determined by least squares linear regression analysis. The adsorption "K" values were 7.6, 9.9, and 16.8 for the sandy loam, silty clay and silt loam soils, respectively. The "1/n" values were all less than unity (Table 3). The desorption "K" values ranged from 9.0 to 15.1, 11.5 to 13.5, and 24.1 to 33.0 for the sandy loam, silty clay, and silt loam soils, respectively. The "1/n" values were all less than or equal to unity (Table 4).

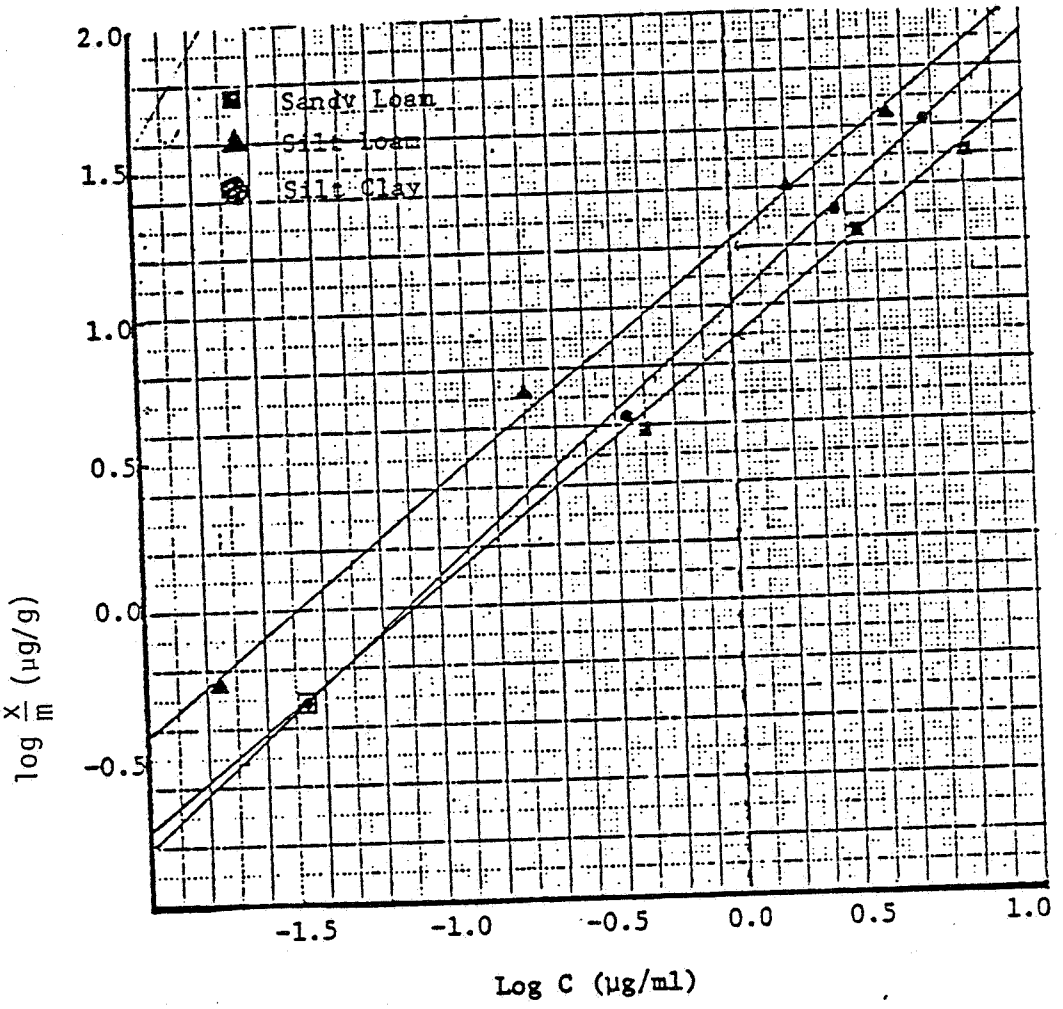

Figure 1. Freundlich isotherms for azinphosmethyl adsorption in soil.

Table 1. Soil characteristics.

Analysis	Soils		
	I	II	III
Texture	Sandy loam	Silt loam	Silty clay
% Sand	74	18	0
% Silt	14	57	41
% Clay	13	25	59
% Organic matter	2.8	5.0	0.5
pH	6.6	7.9	6.0

Table 2. Adsorption and desorption of [^{14}C]azinphosmethyl in soil.

Soil type	Initial fortification (µg)	µg adsorbed	% adsorbed	% desorbed[a]	ave % desorbed
Silt loam	1.27	1.10	87	36	10.4
	12.56	10.49	84	32	9.3
	64.77	48.78	75	39	11.5
	131.01	93.93	72	40	11.9
Sandy loam	1.27	0.93	73	47	17.3
	12.56	7.72	62	54	12.8
	64.77	37.58	58	68	27.0
	131.01	68.62	52	62	27.4
Silty clay	1.27	0.91	72	56	20.7
	12.56	8.21	65	67	-27.2
	64.77	42.55	66	62	-24.6
	131.01	84.74	66	64	25.9
Control (no soil)	250.00	--	3[b]	--	

[a]Sum of the 4 desorption extracts

[b]% lost

Table 3. Freundlich constants for the adsorption of azinphosmethyl on various soil types.[a]

Soil texture	1/n	log K	K
Silt loam	0.82	1.22	16.75
Sandy loam	0.83	0.88	7.60
Silty clay	0.93	0.99	9.85

[a] Determined by least squares linear regression of the equation

$$\log \frac{x}{m} = \frac{1}{n} \log C + \log K$$

Where

x = g azinphosmethyl adsorbed
m = g soil
C = water equilibrium concentration
1/n = slope of the line
K = distribution coefficient (0 intercept)

Table 4. Freundlich constants for the desorption of azinphosmethyl on various soil types.[a]

Soil texture	Desorption interval	1/n	log k	k
Silt loam	1	0.90	1.38	24.10
	2	0.90	1.43	26.72
	3	0.95	1.47	26.12
	4	1.01	1.52	33.04
Sandy loam	1	0.81	0.95	8.95
	2	0.86	1.09	12.23
	3	0.87	1.11	12.79
	4	0.89	1.18	15.14
Silty clay	1	0.94	1.06	11.44
	2	0.96	1.09	12.41
	3	0.96	1.13	13.52
	4	0.94	1.07	11.83

[a] For method of calculation see Table 3.

Conclusions

The % of adsorption of azinphosmethyl to soil is inversely related to the concentration of pesticide applied. The silt loam soil had the highest adsorption and lowest desorption rates , possibly because of its higher organic matter content. The soils were sieved to 40 mesh, thus removing all of the coarse sand and some of the medium sand and altering the soil texture. The removal of part of the sand fraction will increase the percentage of azinphosmethyl adsorbed.

The "K" values for azinphosmethyl indicate that adsorption of the pesticide residues to the soil will reduce the mobility of azinphosmethyl in soil.

The results of this study concur with the results of the column leaching study (Tab No. 48466).

An analytical residue method for the determination of Guthion, Guthion oxygen analog and total Guthion and metabolite residues in soils, J.P. Wargo and R.R. Gronberg, Analytical Development Corporation, June 26, 1979, Acc. No. 099216, Tab Nos. 67084, 67085, 67610, 67611, 67612, 67613, 67648, 67649, and 67650.

Procedure

Soil samples were spiked with azinphosmethyl (recrystallized Guthion) and the oxygen analog of azinphosmethyl.

Methodology

Each soil sample was refluxed with methanol and dichloromethane and filtered. The filtrate was evaporated and the residue was dissolved in hexane saturated with acetonitrile. The solution was partitioned with acetonitrile saturated with hexane, and the acetonitrile extract was divided into two equal fractions. Fraction A was analyzed for azinphosmethyl and the oxygen analog of azinphosmethyl. Fraction B was analyzed for total azinphosmethyl residues.

Analysis of Fraction A

Fraction A was evaporated to dryness and dissolved in methanol. Sodium chloride was added and the solution was partitioned with dichloromethane. The dichloromethane layer was removed and evaporated, and the residue was dissolved in dichloromethane. The solution was eluted through a silica gel chromatographic column with dichloromethane (Fraction I) and acetonitrile in dichloromethane (Fraction II).

Fraction I was evaporated and the residue was dissolved in hexane saturated with acetonitrile. The solution was partitioned with acetonitrile saturated with hexane. The acetonitrile layer was evaporated, and the residue was dissolved in ethyl acetate prior to analysis for azinphosmethyl by gas chromatography (GC).

Fraction II was evaporated and the residue was dissolved in dichloromethane. The solution was eluted with dichloromethane and an acetonitrile: dichloromethane mixture and evaporated to dryness. The residue was dissolved in acetonitrile:dichloromethane and analyzed for azinphosmethyl oxygen analog by high-pressure liquid chromatography (HPLC).

Analysis of Fraction B

Fraction B was evaporated, refluxed with KOH, acidified, and partitioned with benzene. The acidic layer was removed, buffered to pH 4, and partitioned with benzene and isopropanol. The benzene layer was removed, diluted with isopropanol, and analyzed spectrophotofluorometrically.

Results

The method sensitivity was 0.01 ppm for azinphosmethyl and the azinphosmethyl oxygen analog. The recovery rates for azinphosmethyl and the azinphosmethyl oxygen analog were 63-99% and 64-103%, respectively (Table 1). The recovery rates for the fluorescence method were 69-135% and 72-111% for azinphosmethyl and the azinphosmethyl oxygen analog, respectively (Table 2).

Table 1. Recovery of azinphosmethyl (GLC) and azinphosmethyl oxygen analog (HPLC) from soil.

Compound	Fortificiation level (ppm)	Average % recovery (range if more than one sample was analyzed)		
		Sandy loam	Sand	Silt loam (muck)
Control	--	<0.01 ppm	<0.01 ppm	<0.01 ppm
Azinphosmethyl	0.05	73 (63-79)	--	--
	0.10	88(75-99)	92	92
Azinphosmethyl oxygen analog	0.05	85 (64-99)	--	--
	0.10	93 (84-103)	76	90

Table adapted from Tab Nos. 67804, 67610. and 67611.

Table 2. Recovery for total azinphosmethyl and metabolite residues in soil (fluorescence).

Compound	Fortification level (ppm)	Average % recovery (range if more than one sample was analyzed)			
		Sandy loam	Sandy clay loam	Sand	Silt loam (muck)
Control	--	0.48 ppm (0.16-0.77)	0.10 ppm (0.00-0.10)	0.10 ppm	0.05 ppm
Azinphosmethyl	0.5	95 (69-135)	--	84	90
Azinphosmethyl oxygen analog	0.5	93 (72-111)	--	91	90
Benzazimide	0.5	95 (72-113)	--	91	84
Methyl benzazimide	0.5	--	60 (56-64)	--	--
Mercaptomethyl benzazimide	0.5	--	82 (79-82)	--	--
Hydroxymethyl benzazimide	0.5	--	88 (85-90)	--	--
Bis-methyl benzazimide sulfide	0.5	--	85 (83-86)	--	--

Adapted from Tab Nos. 67084, 67612, and 67613.

Conclusions

The GC and HPLC methods described are capable of accurately detecting azinphosmethyl and azinphosmethyl oxygen analog residue levels greater than 0.01 ppm in soil. The fluorescence method is capable of determining total azinphosmethyl residue levels in soil. However, the method sensitivity was not presented.

Recovery of Guthion from soil, R.A. Morris, Chemonics Industries, June 28, 1979, Acc. No. 099216, Tab No. 67813.

Procedure

Sandy loam and silt loam soil samples were spiked with either azinphosmethyl or the azinphosmethyl oxygen analog at 0.10 ppm.

Methodology

The samples were mixed with acetone in an Omni mixer and extracted and analyzed with the method described in Acc. No. 099216, Tab No. 13517.

Results

The recovery rates for azinphosmethyl and azinphosmethyl oxygen analog in a sandy loam soil were 140 and 130%, respectively. The recovery rates in a silt loam soil were 130% for both compounds.

Conclusions

This method is capable of determining azinphosmethyl and azinphosmethyl oxygen analog residue levels of 0.10 ppm in soil. However, the method is incorrect by 30-40%.

Stability of Guthion and Guthion oxygen analog in soil, J.P. Wargo, Mobay Chemical Corp. and Analytical Development Corp., May 29, 1979, Acc. No. 099216, Tab No. 67754.

Procedure

Soil samples (properties not reported) in jars were spiked with azinphosmethyl or the azinphosmethyl oxygen analog, at 0.5 ppm, sealed, and stored at -10 C for 4 weeks.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

After storage for 4 weeks under frozen conditions, 90-97% of the azinphosmethyl and 113-114% of the azinphosmethyl oxygen analog applied were still present.

Conclusions

Azinphosmethyl and the azinphosmethyl oxygen analog are stable in frozen soil stored for 4 weeks.

Photodegradation of Guthion in Aqueous Solution, L.C. Wilkes, J.P. Wargo, R.R. Gronberg, Analytical Development Corp., Monument, Colorado, May 21, 1979, Acc. No. 099216, Tab No. 67980.

Procedure

Ring-labeled [^{14}C]azinphosmethyl was used to prepare a fortified buffered solution at pH 4.35. The [^{14}C]azinophosmethyl concentration was 10 ppm at initiation of the photolysis experiment.

Air was pumped through the system and then through NaOH traps to capture CO_2. Aerobic conditions were maintained by CO_2 traps and pumped air dispersed throughout the photochemical reaction assembly. Temperature was maintained at 30 C. Duplicate 1 ml samples were collected at 0, 0.5, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 14.25, 24.25, 26, 27, 28, 30, 32, and 48.25 hours of light exposure. Light was provided by a 200 watt mercury lamp. Volatiles were collected and analyzed for ^{14}C. Duplicate dark controls (1 ml) were maintained at room temperature during the sample period and analyzed like the other samples after termination of the light experiments.

Methodology

Analysis was performed by radioassay and by two dimensional thin-layer chromatography (TLC). Radioactivity was measured using a scintillation system accounting for quench correction. After ^{14}C assay, aliquots were partitioned with ethyl acetate. Portions of the ethyl acetate extract were radioassayed to detect any loss of activity due to the extraction process. Two dimensional TLC with co-chromatography using reference standards was used to confirm the identity of the breakdown products. A TLC analysis of [^{14}C]anthranilic acid and polar material was also performed to identify unknown degradation products.

Results

The photodegradation products of azinphosmethyl were found to be non-volatile. Partitioning procedures did not cause radioactivity losses, and no significant losses of radioactivity occurred during the exposure period. The amount of ^{14}C residues extracted with ethyl acetate decreased with time, and the amount remaining in the aqueous layer increased with time (from 0.2 to 8.4% after 48 hours).

Photodegradation products as quantified by LSC are presented in Table 1. The dissipation of [^{14}C]azinphosmethyl is presented in Figure 1, and exhibits second order kinetics. The second order rate constant was calculated

to be 1 x $10^{-3}hr^{-1}\%^{-1}$; the half-life is 9.4 hours. Figure 2 presents graphically the photodegradation products of azinphosmethyl, the primary degradation products being benzazimide and/or hydroxymethyl benzazimide. The unknown 'x' found is suspected to be an ester of anthranilic acid and one of the benzazimide standards. In addition, polar origin material was detected (<10% of original reactivity). Dark controls exhibited about 94% of the radioactivity as azinphosmethyl after 48 hours.

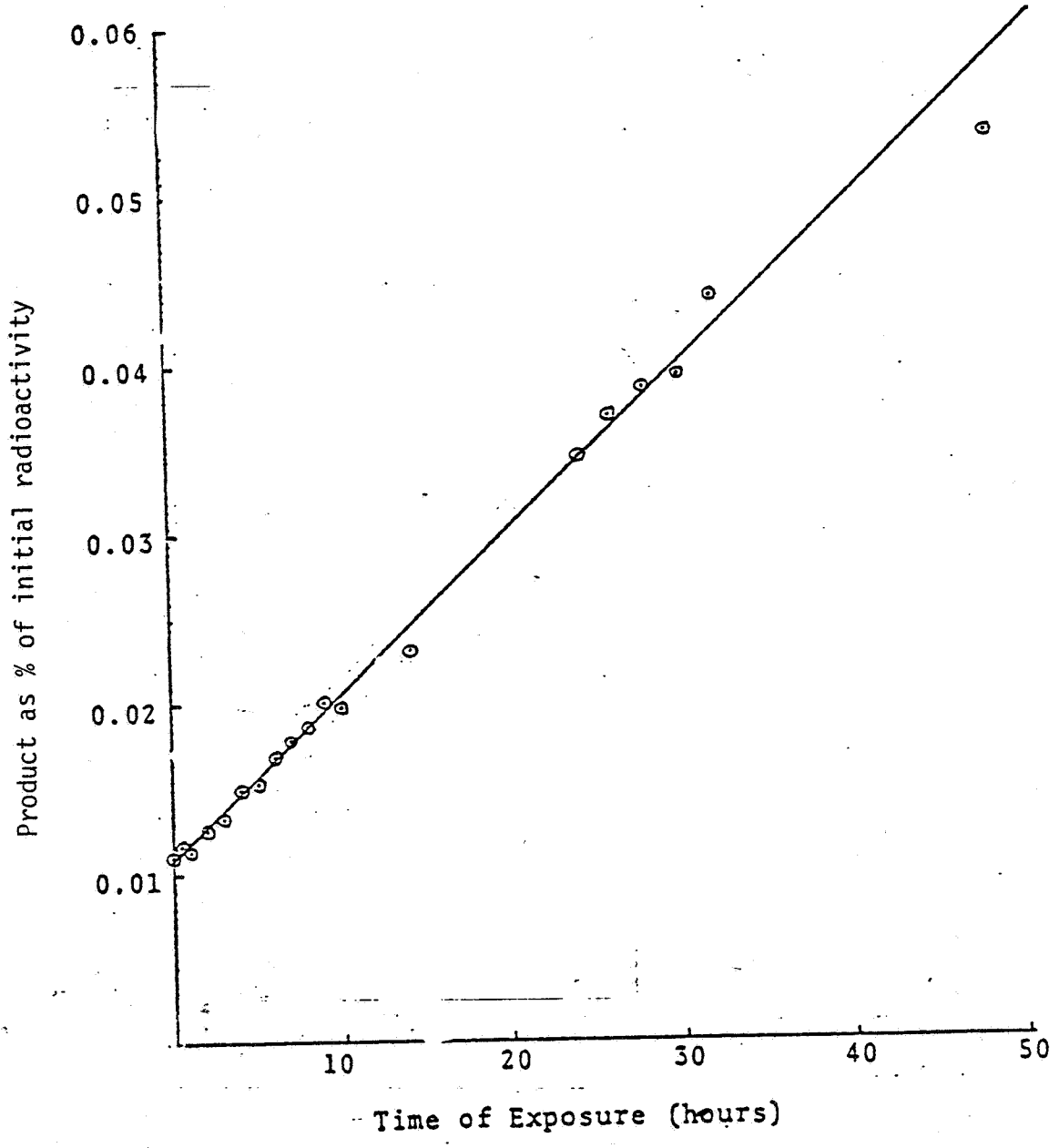

Figure 1. Photodegradation of [^{14}C]azinphosmethyl (second order plot)

65

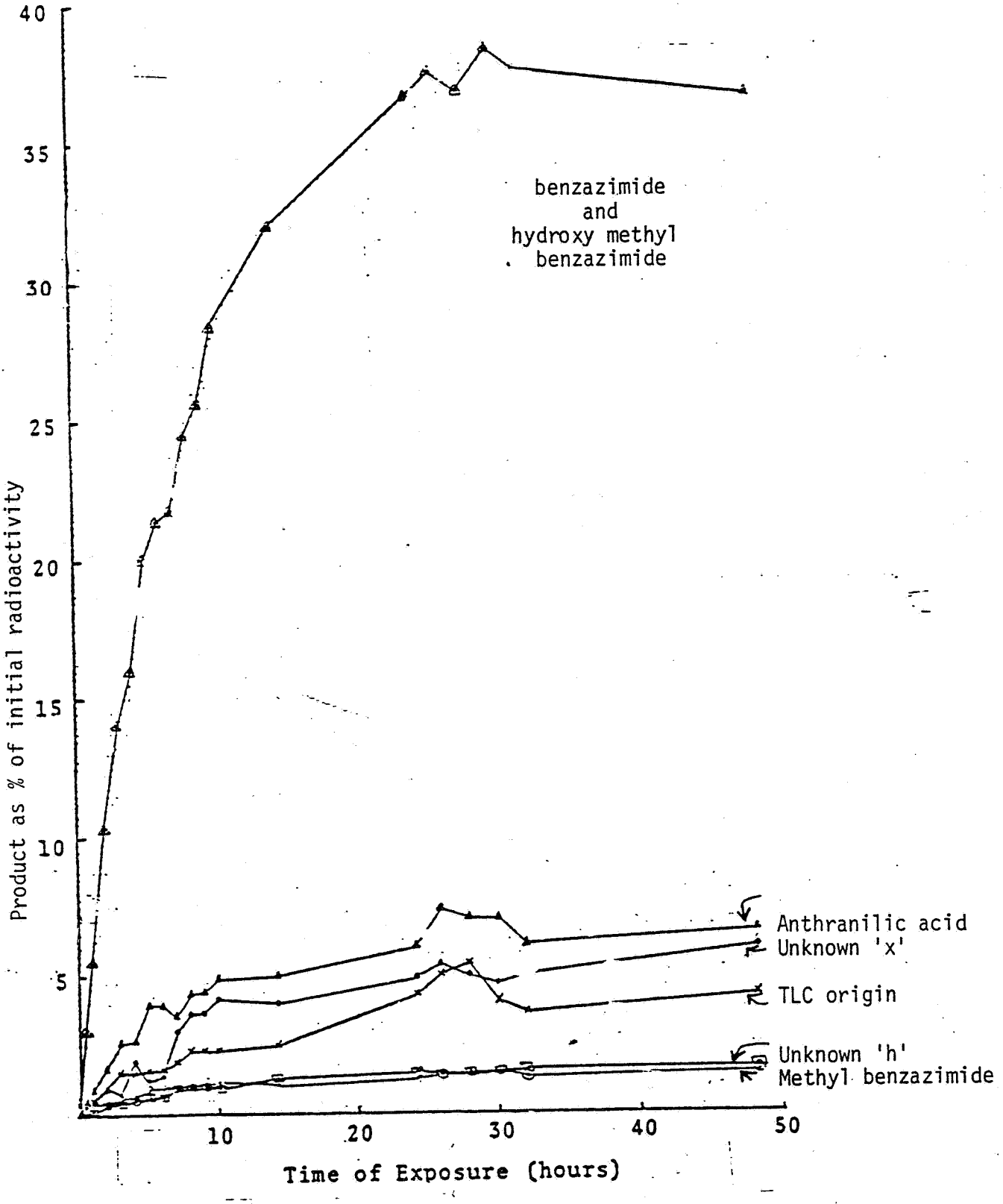

Figure 2. Photodegradation products.

Table 1. Photodegradation products.[a] Product(s) as % of initial radioactivity.[b]

Exposure time (hours)	Anthranilic acid		Azinphosmethyl		Methyl benzazimide		Hydroxymethyl benzazimide & Benzazimide		Unknown 'x'		TLC origin	
	%	ppm	%	ppm	%	ppm	%	ppm	%	ppm	%	ppm
0	0.1	0.01	90.0	9.00	0.1	0.01	0.4	0.04	0.1	0.01	0.2	0.0
48.25	6.6 (9.8)	0.66 (0.98)	18.7 (18.7)	1.87 (1.87)	1.5 (1.5)	0.15 (0.15)	36.6 (38.7)	3.66 (3.87)	6.0 (-)	0.60 (-)	4.3 (5.2)	0.4 (0.5
Dark controls	<0.1	<0.01	93.6	9.36	0.2	0.02	1.3	0.13	0.2	0.02	0.4	0.0

[a] Values in ( ) were obtained with no standards added.

[b] Average of two samples.

Conclusions

Azinphosmethyl undergoes photolysis in aqueous buffered solution (pH 4.3) with a half-life of 9.4 hours. The principle degradation product (about 40% of initial radioactivity after 48 hours) was benzazimide and/or hydroxymethyl benzazimide. No volatile degradation products were formed.

Hydrolysis

Dissipation of Guthion in Buffered Aqueous Solution, L.C. Wilkes, J.P. Wargo, and R.R. Gronberg, Analytical Development Corp., Monument, Colorado, May 21, 1979, ADC Project 378-F, notebook reference 79-R-126,127, Acc. No. 099216, Tab No. 67983.

Procedure

Ring-labeled [^{14}C] azinphosmethyl was studied in aqueous buffers at three pH's (4.2, 6.9, and 9.2), two temperatures (30 and 40 C), and two fortified concentrations (1 and 10 ppm) to determine its rate of hydrolysis and subsequent degradation products. Phosphate buffer solutions were prepared using distilled water, then sterilized by polycarbonate filtering and made into complete samples by addition of azinphosmethyl. The oxygen content and pH of the samples were determined before and after the incubation period. Duplicate tubes from each set of samples were taken for radioassay and thin-layer chromatography (TLC) analysis at 0, 1, 2, 4, 7, 14, 21, and 30 days of incubation.

Methodology

Samples were analyzed for ^{14}C using an analytic scintillation system - corrected for quench. The aqueous and nonaqueous portions were analyzed separately.

Extraction for TLC involved sample adjustment to pH 4 (if necessary) and then partitioning with ethyl acetate. Confirmation of breakdown product identity was achieved by co-chromatography of reference standards with ethyl acetate extracts. A replicate of several samples was assayed with no added standards to verify that no aleration of degradation products occurred. After TLC assaying, the plates were exposed for 1-8 weeks to X-ray film until 1% of the applied dpm was detected. Quanitification of radiolabeled spots on TLC plates was done by scraping off the spots, sonicating in scintillation vials containing the spots plus methanol, and assaying for ^{14}C. Determination of an unknown (X) was performed by mixing ^{14}C anthranilic acid with various standards and analyzing with TLC. Polar material was characterized by isolating from ethyl acetate and aqueous layers with TLC using various solvent systems.

Results

All initial and final pH and O_2 values were found to be comparable. No significant loss of radioactivity occurred during the incubation period or the partitioning procedures. Degradation of azinphosmethyl in solutions of pH 4 and 9 is presented graphically in Figures 1 and 2. Azinphosmethyl degradation appears to follow first-order kinetics. At pH 4, 30 C, and 10 ppm, azinphosmethyl exhibited the greatest stability of all pH/temperature/concentration combinations tested, resulting in a half-life of 42.2 days. The shortest half-life determined was 1.1 days at pH 9, 40 C, and 1 ppm concentration. The degradation of azinphosmethyl and the breakdown products formed at all pH/temperature/concentration combinations is presented in Table 1. Other products formed (at <10%) included bis-methyl benzazimide sulfide, unknown 'k' associated with anthranilic acid, and an unknown 'z'. Unknowns X and k were probably esters of anthranilic acid and the benzazimide standards. At pH 4 and 7 and majority of the radioactivity remained in the aqueous layer, while at pH 9 less than 10% remained in the aqueous layer after 30 days of incubation. No volatilization losses were encountered in the experiment.

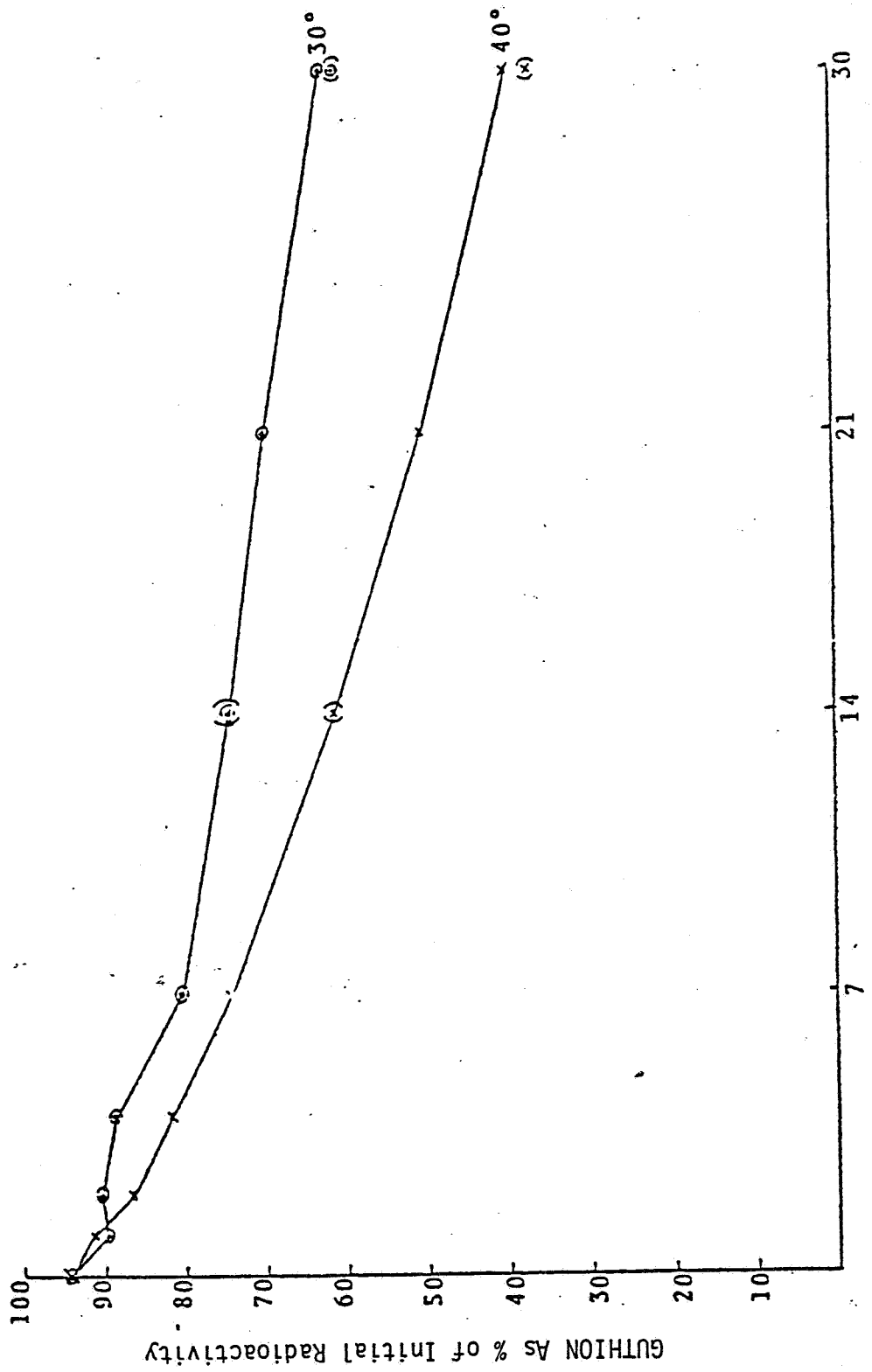

Figure 1. Dissipation of ^{14}C Azinphosmethyl (10 ppm) at pH 4 (Bracketed points have no standards added).

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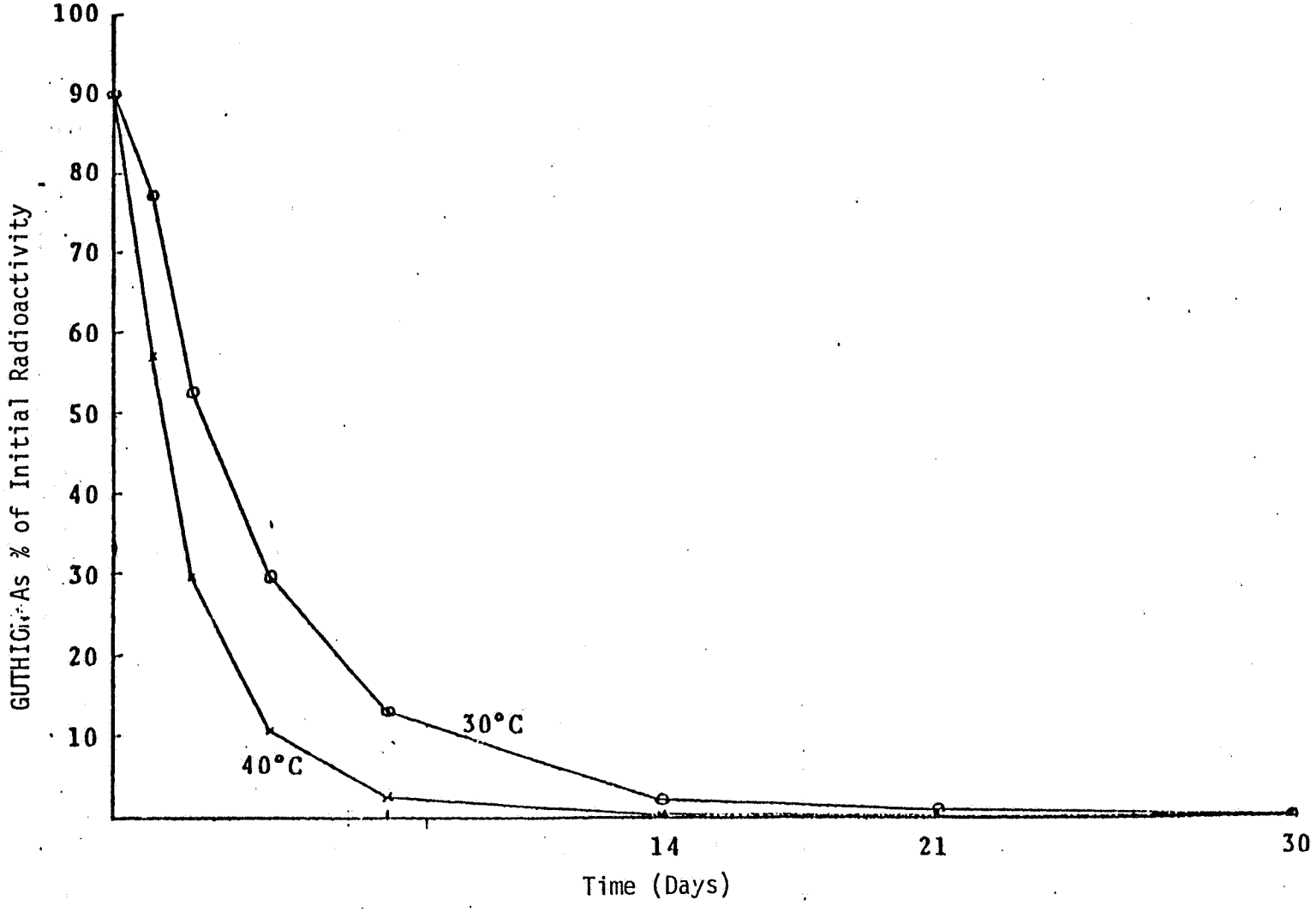

Figure 2. Dissipation of ^{14}C azinphosmethyl (1 ppm) at pH 9.

Table 1. Hydrolysis products with varying pH, temperature, and concentration.[a]

Day	Azinphosmethyl %	Azinphosmethyl ppm	Mercaptomethyl benzazimide %	Mercaptomethyl benzazimide ppm	Hydroxymethyl + benzazimide %	Hydroxymethyl + benzazimide ppm	TLC polar origin %	TLC polar origin ppm	Anthranilic acid %	Anthranilic acid ppm	Unknown X %	Unknown X ppm	Unknown X + Unknown K = Anthranilic acid %	Unknown X + Unknown K = Anthranilic acid ppm
						pH 4, 1 ppm, 30 C								
0	93.2	0.932	1.1	0.011	0.6	0.006	0.6	0.006	--	--	--	--	--	--
30	60.7	0.067	3.7	0.037	8.6	0.086	1.0	0.010	--	--	--	--	--	--
						pH 4, 1 ppm, 40 C								
0	93.2	0.932	1.1	0.011	0.6	0.006	0.6	0.006	--	--	--	--	--	--
30	36.7	0.367	5.6	0.056	13.6	0.136	1.6	0.016	--	--	--	--	--	--
						pH 4, 10 ppm, 30 C								
0	94.4		0.8	0.08	0.4	0.04	0.4	0.04	--	--	<0.1	<0.01	<0.1	<0.
30	62.1	6.21	4.5	0.45	8.1	0.81	1.1	0.11	(<0.1)	(<0.1)	<0.1	<0.01	<0.1	<0.
	(60.4)	(6.04)	(0.5)	(0.05)	(7.3)	(0.73)	(3.8)	(0.38)			(--)	(--)		
						pH 4, 10 ppm, 40 C								
0	94.4	9.04	0.8	0.08	0.4	0.04	0.4	0.04	--	--	0.1	0.01	≤0.1	<0.
30	39.5	3.95	6.8	0.68	12.2	1.22	1.3	0.13	(<0.1)	(<0.01)	<0.1	<0.01	<0.1	<0.
	(36.4)	(3.64)	(0.5)	(0.05)	(10.8)	(8.1)	(0.81)	(0.81)			--	--		
						pH 7, 1 ppm, 30 C								
0	93.5	0.935	0.8	0.008	0.4	0.004	0.4	0.004	--	--	--	--	--	--
30	42.7	0.427	4.9	0.049	6.0	0.060	2.3	0.023	--	--	0.2	0.002	0.2	0.
						pH 7, 1 ppm, 40 C								
0	93.5	0.935	0.8	0.008	0.4	0.005	0.4	0.004	--	--	--	--	--	--
30	18.8	0.188	10.4	0.104	14.2	0.142	3.5	0.035	--	--	--	--	--	--
						pH 7, 10 ppm, 30 C								
0	95.1	9.51	0.8	0.08	0.4	0.04	0.4	0.04	<0.1	<0.01	<0.1	<0.01	<0.1	<0
30	42.2	4.22	10.1	1.01	6.0	0.60	1.2	0.12	1.7	0.17	0.6	0.06	2.5	0
	(50.7)	(5.07)	(0.5)	(0.05)	(5.7)	(0.57)	(2.8)	(0.28)	(2.3)	(0.23)	(0.1)	(0.01)		
						pH 7, 10 ppm, 40 C								
0	95.1	9.51	0.8	0.08	0.4	0.04	0.4	0.04	<0.1	<0.01	<0.1	<0.01	<0.1	<0
30	18:6	1.86	10.4	1.04	8.3	0.83	5.5	0.55	2.8	0.28	1.0	0.10	4.2	0
	(18.0)	(1.80)	(0.7)	(0.07)	(6.9)	(0.69)	(9.0)	(0.90)	(3.5)	(0.35)	(0.1)	(0.01)		
						pH 9, 1 ppm, 30 C								
0	90.1	0.901	0.8	0.008	0.4	0.004	0.6	0.006	--	--	--	--	--	
30	0.3	0.003	1.0	0.010	32.4	0.324	5.6	0.056	22.8	0.228	7.4	0.074	32.8	0
						pH 9, 1 ppm, 40 C								
0	90.1	0.901	0.8	0.008	0.4	0.004	0.6	0.006	--	--	--	--	--	
30	0.2	0.002	0.5	0.005	36.7	0.367	5.3	0.053	18.1	0.181	14.5	0.145	37.5	0
						pH 9, 10 ppm, 30 C								
0	96.7	9.67	0.9	0.09	0.4	0.04	0.4	0.04	0.1	0.01	<0.1	<0.01	0.1	0
30	0.5	0.05	3.0	0.30	35.1	3.51	2.5	0.25	20.5	2.05	8.5	0.85	32.7	
	(2.0)	(0.20)	(0.4)	(0.04)	(33.1)	(3.31)	(3.8)	(0.38)	(30.1)	(3.01)	(0.5)	(0.05)		
						pH 9, 10 ppm, 40 C								
0	96.7	9.67	0.9	0.09	0.4	0.04	0.4	0.04	0.1	0.01	<0.1	<0.01	0.1	0
30	0.3	0.03	0.8	0.08	38.9	3.89	3.9	0.39	19.1	1.91	10.1	1.01	32.5	
	(2.0)	(0.20)	(0.4)	(0.04)	(35.1)	(3.51)	(3.5)	(0.35)	(31.1)	(3.11)	(1.6)	(0.16)		

[a]Values in () were determined without the addition of standards.

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Conclusions

The hydrolytic degradation of azinphosmethyl in aqueous solution was found to range from a half-life of 1 to 42 days depending upon pH, temperature, and initial concentration of the compound. The compound was found to be most stable at low pH, low temperature, and high concentrations. However, the pH of the solution appears to be the most influential factor in varying the stability of azinphosmethyl.

The major degradation products at all pH/temperature/concentration combinations tested was benzazimide and/or hydroxymethyl benzazimide. Other major degradation products (<10% each) were anthranilic acid and possibly its esters (unknown X and k), and mercaptomethyl benzazimide.

To summarize, the rate of azinphosmethyl degradation aqueous solutions depends principally on the pH of the solution. Temperature and initial concentration also effect the rate of degradation to a lesser degree. Five major breakdown products were identified as being formed by hydrolytic degradation of azinphosmethyl.

The metabolism of Guthion in sandy loam soil, R.R. Gronberg, R.J. Polluck and J.P. Wargo, Mobay Chemical Corp. and Analytical Development Corp., August 27, 1979, Acc. No. 099216, Tab No. 68030.

Procedure

Aerobic soil

A sandy loam soil (73% sand; 17% silt; 10% clay; organic matter content 1.4%; pH 7.9; CEC 4.6 meq/100 g soil) was fortified to 2 ppm azinphosmethyl (ring-labeled [^{14}C]Guthion mixed with unlabeled Guthion to form at 50 WP), moistened to 63% of field moisture capacity and mixed. Portions of the treated soil were placed in flasks wrapped with aluminum foil, attached to the aerobic soil metabolism apparatus (Figure 1), and incubated at room temperature. Samples were analyzed at 0, 1, 3, 7, 14, 30, 60, 120, 186, 242, 304, and 365 days after treatment.

Anaerobic soil

After incubation for 30 days, several flasks were removed and the soil was flooded. Anaerobic indicators were added and the flasks were purged with N_2. The flasks were sealed and incubated at room temperature. Duplicate samples were analyzed at 30 and 60 days after flooding (60 and 90 days after treatment).

Sterile soil

Untreated soil samples were placed in flasks and heated several times at 15 psi in a pressure cooker for 1 hour. Each sample was treated with 2 ppm of ring-labeled [^{14}C]azinphosmethyl and mixed. The flasks were wrapped with aluminum foil, incubated at room temperature, and sampled at 7, 14, 30, 60, and 120 days after treatment.

Methodology

All soil samples were refluxed in methanol:chloroform and filtered. The filter cake was then refluxed with methanol:HCl and filtered. The filtrates were analyzed for radioactivity with a liquid scintillation counter (LSC). The organosoluble fractions were subjected to thin-layer chromatography.

The soil was analyzed for radioactivity by combustion. The Chromosorb adsorbent was extracted with methanol, and the methanol extract was analyzed for radioactivity by LSC. The NaOH in the trap was diluted with water and analyzed for radioactivity by LSC. The cold trap was rinsed with methanol which was then analyzed for radioactivity by LSC.

Results

Aerobic soil

Total radioactive residues in aerobic soil through 365 days were relatively constant with approximately 2 ppm remaining. This suggested that no significant amount of volatile ^{14}C degradation products had formed (Table 1). The extractable residues decreased with time. Immediately after treatment, 96% of the residues were extractable and 365 days after treatment 27% of the residues were extractable. The first half-life of azinphosmethyl was 21 days and the second half-life was 62 days. At 365 days after treatment 2.0% of the residues were azinphosmethyl. Azinphosmethyl oxygen analog accounted for up to 5.3% of the residues during the experiment and the benzazimide-containing metabolites (mercaptomethyl benzazimide, hydroxymethyl benzazimide, benzazimide, and bis-methyl benzazimide sulfide) accounted for up to 12% of the residues (Table 2).

Anaerobic soil

The apparent half-life of azinphosmethyl under anaerobic conditions was 68 days. At 90 days after treatment, 23.7% of the residues remained as extractable azinphosmethyl. The azinphosmethyl metabolites were present in concentrations similar to those found in the aerobic soil (Table 3).

Sterile soil

The estimated half-life of azinphosmethyl in sterile soil was 355 days.

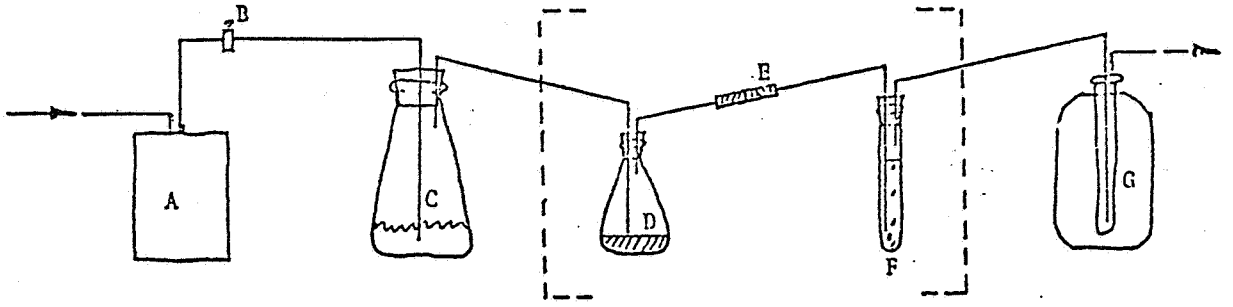

Figure 1. Aerobic soil metabolism apparatus.

A. Aquarium pump
B. Needle valve
C. Manifold jar

Independent soil systems from manifold jar

D. Soil sample jar (wrapped with aluminum foil)
E. Chromosorb 102 cartridges
F. Sodium hydroxide trap

Common trap for all soil systems

G. Dry ice acetone trap

Table 1. Azinphosmethyl residues in a sandy loam soil.

Interval (days)	Aerobic conditions Soil residues ppm	%	Volatile Residues(%)[a] NaOH trap	Anaerobic conditions Soil residues ppm	%	Sterile conditions Soil residues ppm	%
0	2.07	100.0	0.05	---	---	---	---
7	2.23	107.5	0.21	---	---	1.97	95.4
14	2.28	110.4	0.27	---	---	2.04	98.4
30	2.13	102.9	0.35	---	---	1.98	95.4
60	1.95	94.2	1.30	1.97	95.4	1.92	92.7
90	---	---	---	2.13	102.9	---	---
120	2.02	97.4	3.20	---	---	1.93	93.3
186	2.09	101.0	2.10	---	---	---	---
242	2.24	108.2	1.50	---	---	---	---
304	2.08	100.4	1.20	---	---	---	---
365	2.35	113.5	4.10	---	---	---	---

Table 2. Distribution of azinphosmethyl residues in aerobic sandy loam soil.

Interval (days)	Azinphosmethyl (%)	Azinphosmethyl oxygen analog (%)	Benzazimide metabolites (%)	Other[a] (%)	Soil bound (%)
0	92.9	0.6	1.5	1.3	3.7
7	72.7	0.8	6.6	7.8	12.1
14	59.5	1.1	5.1	13.9	20.4
30	44.3	2.8	6.0	15.7	31.2
60	26.9	0.6	9.0	12.0	51.5
120	14.4	1.1	12.0	15.9	56.6
186	4.8	5.3	11.2	17.3	61.4
242	4.0	4.4	8.3	12.8	70.5
304	2.7	4.2	5.4	16.7	70.9
365	2.0	4.0	7.0	14.3	72.7

[a]Unidentified residues.

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Table 3. Distribution of azinphosmethyl residues in anaerobic and sterile sandy loam soil.

Interval (days)	Azinphosmethyl (%)	Azinphosmethyl oxygen analog (%)	Benzazimide metabolites	Other[a] (%)	Soil bound (%)
Anaerobic					
30 (0)[b]	44.3	2.8	6.0	15.7	31.2
60 (30)[b]	33.9	1.7	9.0	13.7	41.7
90 (60)[b]	23.7	0.6	8.1	17.3	50.3
Sterile					
7	87.1	0.8	2.6	8.3	1.2
14	82.8	0.7	3.3	8.6	4.6
30	77.8	2.1	1.9	12.1	6.1
60	76.4	1.0	3.7	11.0	7.9
120	68.4	0.6	4.1	15.1	11.8

[a]Unidentified residues.

[b]Days under anaerobic conditions given in parentheses.

Conclusions

The half-lives of azinphosmethyl in aerobic and anaerobic soil were 21 and 68 days, respectively. Bound residues increased with time, with 72% becoming bound after 365 days of aerobic incubation. The estimated half-life of azinphosmethyl in sterile soil was 355 days.

The recovery rates for azinphosmethyl in soil ranged from 92 to 113% (1.92-2.35 ppm) over the course of the experiment. The reported application rate of azinphosmethyl was 2 ppm; however, 2.46 ppm (3.2 mg ai applied to 1.3 kg of soil) was actually applied, which would account for the high recovery rates throughout the experiment.

Azinphosmethyl is metabolized in soil by microorganisms.

Under aerobic conditions the benzazimide-containing metabolite residue levels reached a maximum concentration at 120 days after treatment and then declined. Azinphosmethyl oxygen analog residue levels reached a maximum at 186 days after treatment and remained constant during the remainder of the experiment.

Soil persistence study, Mobay Chemical Corp. and Analytical Biochemistry Laboratories, February 10, 1978, Acc. No. 099216, Tab Nos. 54487, 54488, 54491, and 54492.

Procedure

Silt loam field plots (8% sand; 72% silt; 20% clay; organic matter content 2.3%; pH 6.8; and CEC 32 meq/100 g soil) were treated with either 0.25 lb ai/A azinphosmethyl (Guthion 2L), 2.0 lb ai/A Bolstar or a mixture of 0.25 lb ai/A azinphosmethyl and 2.0 lb ai/A Bolstar. Soil samples were collected to a depth of 6 inches at 0, 122, and 188 days after treatment.

Methodology

The soil samples were extracted and analyzed using the methods described in Acc. No. 099216, Tab Nos. 49675 and 52901.

Results

When azinphosmethyl was applied alone residue levels of < 0.10, 0.17, and < 0.10 ppm were present at 0, 122, and 188 days after treatment, respectively. When azinphosmethyl was applied in combination with Bolstar, azinphosmethyl residue levels of 0.36, < 0.10 and 0.10 ppm were present at 0, 122, and 188 days after treatment, respectively (Table 1).

Table 1. Dissipation of azinphosmethyl and Bolstar applied alone and in combination to a silt loam soil.

Chemical	Treatment (lb ai/A)	Sampling Interval (days)	Residues (ppm)	
			Azinphosmethyl	Bolstar
Azinphosmethyl	0.25	0	<0.10	---
		122	0.17	---
		188	<0.10	---
Bolstar	2.0	0	---	5.17
		122	---	0.10
		188	---	0.60
Azinphosmethyl and Bolstar	0.25 2.0	0	0.36	2.34
		122	<0.10	0.43
		188	0.10	0.21

Conclusions

The dissipation rate and half-life of azinphosmethyl applied alone and in combination with Bolstar to a silt loam soil cannot be determined from the data presented. The application rate of 0.25 lb/A (0.125 ppm) was only slightly greater than the sensitivity of the analytical method (0.10 ppm).

Soil persistence study, Mobay Chemical Corp. and Analytical Biochemistry Laboratories, February 10, 1978, Acc. No. 099216, Tab Nos. 54489, 54490, 54493 and 54494.

Procedure

Loamy sand field plots (74% sand; 20% salt; 6% clay; organic matter content 0.6%; pH 7.3; CEC 15 meq/100 g soil) were treated with either 0.25 lb ai/A azinphosmethyl (Guthion 2L), 2 lb ai/A Bolstar or a mixture of 0.25 lb ai/A azinphosmethyl and 2 lb ai/A Bolstar. Soil samples were collected to a depth of 6 inches at 0, 122, and 188 days after treatment.

Methodology

The soil samples were extracted and analyzed using the methods described in Acc. No. 099216, Tab Nos. 49675 and 52901.

Results

When azinphosmethyl was applied alone residue levels of < 0.10 ppm were present at 0, 122, and 188 days after treatment. When azinphosmethyl was applied in combination with Bolstar, azinphosmethyl residue levels of 0.10, 0.14 and 0.13 ppm were present at 0, 122, and 188 days after treatment, respectively (Table 1).

Table 1. Dissipation of azinphosmethyl and Bolstar applied alone and in combination to a loamy sand soil.

Chemical	Treatment (lb ai/A)	Sampling interval (days)	Residues (ppm)	
			Azinphosmethyl	Bolstar
Azinphosmethyl	0.25	0	<0.10	--
		122	<0.10	--
		188	<0.10	--
Bolstar	2	0	--	3.70
		122	--	0.38
		188	--	0.15
Azinphosmethyl and Bolstar	0.25	0	0.10	3.38
	2	122	0.14	0.19
		188	0.13	0.13

Conclusions

The dissipation rate and half-life of azinphosmethyl applied alone and in combination with Bolstar to a loamy sand soil cannot be determined from the data presented. The application rate of 0.25 lb/A (0.125 ppm) was only slightly greater than the sensitivity of the analytical method (0.10 ppm).

Soil persistence study STF-4501-77D, J.S. Thornton, Mobay Chemical Corp. and Chemonics Industries, January 17, 1979, Acc. No. 099216, Tab Nos. 65614 and 67186.

Procedure

Glass pots containing silt loam soil (12% sand; 65% silt; 23% clay; organic matter 2.4%; pH 6.4; CEC 19.8 meq/100 g soil) were treated with either 2.0 lb ai/A azinphosmethyl (Guthion 2L), 2.0 lb ai/A DEF or 2.0 lb ai/A azinphosmethyl in combination with 2.0 lb ai/A DEF, and maintained in a greenhouse at field moisture capacity. Soil samples were collected to a depth of 6 inches at 0, 3, 6, 15, 21, 28, 61, 90, 120, and 152 days after treatment.

Methodology

The soil samples were extracted and analyzed for azinphosmethyl and DEF using the methods described in Acc. No. 099216, Tab Nos 13517 and 67813 and Acc. No. 099216, Tab Nos. 18713 and 65369, respectively.

Results

When azinphosmethyl was applied alone, 0.14 ppm remained at 61 days after treatment and < 0.10 ppm remained in all samples collected 90 days posttreatment. When azinphosmethyl was applied in combination with DEF a constant 0.10-0.15 ppm remained from 15 to 152 days posttreatment. The dissipation rates of azinphosmethyl and DEF applied alone were similar to the dissipation rate of the two pesticides applied in combination (Table 1).

Table 1. Dissipation of azinphosmethyl and DEF applied alone and in combination to soil.

Compound	Application rate (lb ai/A)	Sampling interval (days)	Residue levels (ppm)	
			azinphosmethyl	DEF
azinphosmethyl	2.0	0	0.31	---
		15	0.16	---
		28	<0.10	---
		61	0.14	---
		90	<0.10	---
		120	<0.10	---
		152	<0.10	---
DEF	2.0	0	---	3.33[a]
		15	---	2.37
		28	---	1.50
		61	---	0.46
		90	---	0.20
azinphosmethyl and DEF	2.0	0	0.21	3.72[a]
		15	0.13	2.54
	2.0	28	0.12	1.98
		61	0.15	0.51
		90	0.10	0.25
		120	<0.10	---
		152	0.15	---

[a]average of 2 samples.

Conclusions

The dissipation rates of azinphosmethyl and DEF applied alone to silt loam soil and the dissipation rate of the two pesticides applied in combination were similar. However, the recovery levels in posttreatment samples are low (<33% of applied).

Soil persistence study STF-4500-77D, J.S. Thornton, Mobay Chemical Corp. and Chemonics Industries, January 17, 1979, Acc. No. 099216, Tab Nos. 65615 and 67239.

Procedure

Glass pots containing sandy loam soil (73% sand; 21% silt; 6% clay; organic matter content 1.2%; pH 6.8; CEC 5.9 meq/100 g soil) were treated with either 2.0 lb ai/A azinphosmethyl (Guthion 2L), 2.0 lb ai/A DEF or 2.0 lb ai/A azinphosmethyl in combination with 2.0 lb ai/A DEF, and maintained in a greenhouse at field moisture capacity. Soil samples were collected to a depth of 6 inches at 0, 3, 6, 15, 21, 28, 61, 90, 120 and 152 days after treatment.

Methodology

The soil samples were extracted and analyzed for azinphosmethyl and DEF using the methods described in Acc. No. 099216, Tab Nos. 13517 and 67813 and Acc. No. 099216, Tab Nos. 18713 and 65369, respectively.

Results

When azinphosmethyl was applied alone or in combination with DEF, residue levels of 0.10 ppm or less were present in all samples collected 15 days posttreatment (Table 1).

Table 1. Dissipation of azinphosmethyl and DEF applied alone and in combination to a sandy loam soil.

Compound	Application rate (lb ai/A)	Sampling interval (days)	Residue levels (ppm)	
			azinphosmethyl	DEF
azinphosmethyl	2.0	0	0.25[a]	---
		15	<0.10	---
		28	<0.10	---
		61	<0.10	---
		90	<0.10	---
		120	<0.10	---
		152	<0.10	---
DEF	2.0	0	---	2.80[a]
		15	---	1.93
		28	---	1.40
		61	---	0.25
		90	---	0.13
azinphosmethyl and DEF	2.0	0	0.27[a]	3.12[a]
	2.0	15	0.10	1.66
		28	0.11	1.17
		61	<0.10	0.24
		90	0.10	0.13
		120	<0.10	---
		152	<0.10	---

[a]average of 2 samples.

Conclusions

The dissipation rates of azinphosmethyl and DEF applied alone to sandy loam soil and the dissipation rate of the two pesticides applied in combination were similar.

Soil persistence study HFI-763-77/79D, D. Doran and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 20, 1979, Acc. No. 099216, Tab No. 67803.

Procedure

A sandy loam field plot (68% sand; 23% silt; 9% clay; organic matter content 2.5%; pH 5.4; CEC 14.4 meq/100 g soil) was treated with azinphosmethyl (Guthion 2L) at 4.0 lb ai/A. Soil samples were collected to a depth of 12 inches at 0, 30, and 58 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216 Tab No. 67084.

Results

Azinphosmethyl residue levels of 0.01 ppm were present 30 days posttreatment (Table 1). Total azinphosmethyl (azinphosmethyl and metabolites) residue levels of 0.20 and 0.06 ppm were present in the 0-6 and 6-12 inch soil samples, respectively, 58 days after treatment.

Table 1. Dissipation of azinphosmethyl in a sandy loam soil treated at 4.0 lb ai/A.

Sampling interval (days)	Residue levels (ppm)			
	0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.32	0.75	0.01	0.14
30	0.01	0.31	<0.01	0.07
58	<0.01	0.20	<0.01	0.06

[a] Total of azinphosmethyl and metabolites; azinphosmethyl oxygen analog residue levels were <0.01 ppm.

Conclusions

Azinphosmethyl does not persist in a sandy loam soil. Azinphosmethyl was applied at 4.0 lb ai/A (2 ppm); however, the posttreatment 0-6 inch samples contained only 0.75 ppm. This could have been due to adsorption of the pesticide to the soil (Tab No. 66848).

Soil persistence study 661-762-77/79D, J. Warren and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 19, 1979, Acc. No. 099216, Tab No. 67804.

Procedure

A sandy loam field plot (54% sand; 32% silt; 14% clay; organic matter content 2.8%; pH 4.7; CEC 26.6 meq/100 g soil) was treated with azinphosmethyl (Guthion 2S) at 4.0 lb ai/A. Soil samples were collected to a depth of 12 inches at 0 and 61 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Total azinphosmethyl (azinphosmethyl and metabolites) residue levels of 0.60 and 0.41 ppm were present in the 0-6 and 6-12 inch soil samples, respectively, at 61 days after treatment. Undegraded azinphosmethyl residues at levels of 0.03 and 0.01 ppm were present in the respective soil layers at 61 days after treatment.

Conclusions

The half-life of total residues (azinphosmethyl and metabolites) based on the application rate of 4.0 lb/A (2 ppm), is 61 days in a sandy loam soil. During this same time interval 98% of the azinphosmethyl dissipated. However, total azinphosmethyl residue levels in the posttreatment 0-6 inch soil samples were 4.21 ppm, which was in excess of the application rate.

Azinphosmethyl does not persist in a sandy loam soil.

Soil persistence study RGV-761-77/79D, E. Rowehl and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 20, 1979, Acc. No. 099216, Tab No. 67805.

Procedure

A sandy clay loam field plot (60% sand; 17% silt, 23% clay; organic matter content 2.4%; pH 7.5; CEC 21.4 meq/100 g soil) was treated with azinphosmethyl (Guthion 2L; Mobay Chemical Corp.) at 4 lb ai/A. Soil samples were collected to a depth of 12 inches at 0, 30, 60, 119, 181, and 273 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Total azinphosmethyl residue levels of 0.41, 0.20, and 0.16 ppm were present in the 0-6 inch samples at 60, 181, and 273 days after treatment, respectively. In the 6-12 inch samples, residue levels of 0.17, 0.10, and 0.08 ppm were present at the respective time intervals. At 181 days after treatment, less than 0.10 ppm of undegraded azinphosmethyl remained in each soil layer (Table 1).

Table 1. Dissipation of azinphosmethyl in a sandy clay loam soil treated at 4 lb ai/A.

Sampling interval (days)	Residue levels (ppm)			
	0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.80	1.25	0.27	0.54
30	0.02	0.28	0.01	0.18
60	0.04	0.41	0.01	0.17
119	0.01	0.24	<0.01	0.12
181	<0.01	0.20	<0.01	0.10
273	<0.01	0.16	<0.01	0.08

[a] Total of azinphosmethyl and metabolites; azinphosmethyl oxygen analog residue levels were <0.01 ppm in all samples.

Conclusions

Combined azinphosmethyl and metabolites have a half-life of less than 30 days in a sandy clay loam soil. Greater than 90% of the azinphosmethyl was degraded by 30 days after treatment. At 273 days after treatment 99% of the azinphosmethyl and 88% of the total azinphosmethyl residues had dissipated.

Azinphosmethyl is not persistent in a sandy clay loam soil.

Soil persistence study VBL-760-77/79D, R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 20, 1979, Acc. No. 099216, Tab No. 67806.

Procedure

A silt loam field plot (32% sand; 57% silt; 11% clay; organic matter content 49%; pH 7.3; CEC 21 meq/100 g soil) was treated with azinphosmethyl (Guthion 2L) at 4.0 lb ai/A. Soil samples were collected to a depth of 12 inches at 0, 30, 61, 120, 181, and 273 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Total azinphosmethyl (azinphosmethyl and metabolites) residue levels of 0.33 and 0.02 ppm were present in the 0-6 and 6-12 inch soil samples, respectively, at 30 days after treatment. At 273 days after treatment, the respective soil samples had residue levels of 0.14 and 0.02 ppm. Azinphosmethyl residue levels of <0.01 ppm were present 60 days posttreatment (Table 1).

Table 1. Dissipation of azinphosmethyl in a silt loam soil treated at a rate of 4 lb ai/A.

Sampling interval (days)	Residue levels (ppm)			
	0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.43	0.58	0.02	0.01
30	0.05	0.33	<0.01	0.02
60	<0.01	0.16	<0.01	0.03
273	<0.01	0.14	<0.01	0.02

[a] Total of azinphosmethyl and metabolites.

Conclusions

At 273 days after treatment 99% of the azinphosmethyl and 92% of the azinphosmethyl metabolite residues had dissipated. Azinphosmethyl was applied at 4.0 lb ai/A (2 ppm); however, only 0.58 ppm were recovered in the posttreatment 0-6 inch samples.

Because of the high organic matter content of the soil (49%), it is possible that the azinphosmethyl residues were soilbound.

Azinphosmethyl does not persist in a silt loam soil.

Soil persistence study HFI-753-77/79D, D. Doran and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 19, 1979, Acc. No. 099216, Tab No. 67807.

Procedure

A sandy loam field plot (66% sand; 23% silt; 11% clay; organic matter content 2.3%; pH 5.3; CEC 14.9 meq/100 g soil) was treated with azinphosmethyl (Guthion 50 WP) at 4 lb ai/A. Soil samples were collected to a depth of 12 inches by 6-inch cores at 0, 30, and 59 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Total azinphosmethyl residue levels of 0.22 and 0.11 ppm were present in the 0-6 and 6-12 inch samples, respectively, at 58 days after treatment (Table 1).

Table 1. Dissipation of azinphosmethyl in a sandy loam soil treated at 4 lb ai/A.

Sampling interval (days)	Residue levels (ppm)			
	0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.22	0.62	0.04	0.16
30	0.02	0.40	<0.01	0.08
58	<0.01	0.22	<0.01	0.11

[a] Total azinphosmethyl and metabolites; azinphosmethyl oxygen analog residue levels were <0.01 ppm.

Conclusions

Over a 58-day period, 99% of the azinphosmethyl residues and 83% of the azinphosmethyl metabolite residues dissipated. However, less than half of the applied pesticide was recovered in the posttreatment samples. This could be due to adsorption of the pesticide by the soil (Tab No. 66848).

Azinphosmethyl does not appear to persist in a sandy loam soil.

Soil persistence study 661-752-77/79D, J. Warren and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 19, 1979, Acc. No. 099216, Tab No. 67808.

Procedure

A sandy loam field plot (53% sand; 34% silt; 19% clay; organic matter content 3.5%; pH 4.7; and CEC 29.6 meq/100 g soil) was treated with azinphosmethyl (Guthion 50 WP) at 4 lb ai/A. Soil samples were collected to a depth of 12 inches at 0 and 61 days after treatment.

Methodology

The soil samples were analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Total azinphosmethyl residue levels of 0.71 and 0.36 ppm were present in the 0-6 and 6-12 inch soil samples, respectively, at 61 days after treatment. Undegraded azinphosmethyl residue levels were 0.04 and 0.02 ppm in the respective soil layers at 61 days after treatment.

Table 1. Dissipation of azinphosmethyl in a sandy loam soil treated at 4 lb ai/A.

Sampling interval (days)	Residue levels (ppm)			
	0-6 inch sample		6-12 inch sample	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.88	1.48	0.57	1.52
61	0.04	0.71	0.02	0.36

[a]Total of azinphosmethyl and metabolites; azinphosmethyl oxygen analog residue levels were <0.01 ppm.

Conclusions

At 61 days after treatment, 97% of the azinphosmethyl and approximately 50% of the azinphosmethyl metabolite residues had dissipated. Azinphosmethyl was applied at 4 lb/A (2 ppm); however, the posttreatment soil samples contained 1.50 ppm. This would indicate that possibly there was an error in the application of the pesticide. Also, the percentages of sand, silt, and clay add up to 106%. This 6% error could change the soil texture classification to a loam.

Soil persistence study RGV-751-77/79D, E. Rowehl and R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 19, 1979, Acc. No. 099216, Tab No. 67809.

Procedure

A sandy clay loam field plot (60% sand; 17% silt; 23% clay; organic matter content 2.6%; pH 7.6; CEC 19.7 meq/100 g soil) was treated with azinphosmethyl (Guthion 50 WP) at 4 lb ai/A. Soil samples were collected to a depth of 12 inches at 0, 30, 60, 119, 181, and 273 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216 Tab No. 67084.

Results

Azinphosmethyl residue levels of 0.41, 0.29, and 0.27 ppm were present in the 0-6 inch soil samples at 30, 119, and 273 days after treatment, respectively. In the 6-12 inch samples, residue levels of 0.23, 0.15, and 0.10 ppm were present at the respective time intervals. Undegraded azinphosmethyl residue levels of less than 0.01 ppm were present in each soil layer at 181 days after treatment (Table 1).

Table 1. Dissipation of azinphosmethyl in a sandy clay loam treated at 4 lb ai/A.

Sampling interval (days)	Residue levels (ppm) 0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.53	0.89	0.62	0.95
30	0.04	0.41	0.02	0.23
60	0.02	0.29	<0.01	0.17
119	0.03	0.29	<0.01	0.15
181	<0.01	0.24	<0.01	0.15
273	<0.01	0.27	<0.01	0.10

[a]Total of azinphosmethyl and metabolites; azinphosmethyl oxygen analog residue levels were <0.01 ppm in all samples.

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Conclusions

Azinphosmethyl does not persist in a sandy clay loam soil. The half-life of azinphosmethyl and its metabolites was less than 30 days in a sandy clay loam soil, with 97% of the azinphosmethyl being degraded within 30 days after treatment.

Soil persistence study VBL-750-77/79D, R.A. Morris, Mobay Chemical Corp. and Analytical Development Corp., June 19, 1979, Acc. No. 099216, Tab No. 67810.

Procedure

A sandy soil field plot (92% sand; 1% silt; 7% clay; organic matter content 0.8%; CEC 1.1 meq/100 g soil) was treated with azinphosmethyl (Guthion 50 WP) at 4.0 lb ai/A. Soil samples were collected at 0, 30, 61, 120, 181, and 273 days after treatment.

Methodology

The soil samples were extracted and analyzed with the method described in Acc. No. 099216, Tab No. 67084.

Results

Azinphosmethyl residue levels of 0.01 ppm were present in both soil layers 120 days posttreatment (Table 1). Total azinphosmethyl (azinphosmethyl and metabolites) residue levels of 0.21 and 0.12 ppm were present in the 0-6 and 6-12 inch soil samples, respectively, at 273 days after treatment.

Table 1. Disspation of azinphosmethyl in a sandy soil treated at 4.0 lb ai/A.

Sampling (days)	Residue levels			
	0-6 inch samples		6-12 inch samples	
	Azinphosmethyl	Total[a]	Azinphosmethyl	Total[a]
0	0.82	1.19	0.04	0.05
30	0.03	0.36	<0.01	0.12
120	<0.01	0.21	<0.01	0.07
273	<0.01	0.21	<0.01	0.12

[a]Total of azinphosmethyl and metabolites.

Conclusions

Azinphosmethyl does not persist in a sandy soil.

At 120 days after treatment, over 80% of the total azinphosmethyl and 99% of the azinphosmethyl had dissipated. Azinphosmethyl was applied at 4.0 lb ai/A (2 ppm); however, only 1.19 ppm were recovered in the posttreatment 0-6 inch samples. This was possibly due to soil adsoprtion. (Tab No. 66848).

ENVIRONMENTAL FATE ANALYSIS

The hydrolytic degradation of azinphosmethyl occurs with a half-life of 1-42 days, depending on pH, temperature, and initial concentration of the compound. The compound will show greater stability to hydrolysis at low pH values and low temperatures. For example, in aqueous solution at pH 4 and 30 C, azinphosmethyl is degraded with a half-life of 42.2 days. At pH 9 and 40 C, the half-life is 1.1 days. The major degradation products formed (see Figure 1) are benzazimide and hydroxymethyl benzazimide (combined residues >10%). Other degradation products (<10% each) are mercaptomethyl benzazimide, anthranilic acid, and possibly anthranilic acid esters.

Photolysis of azinphosmethyl takes place in aqueous solution with a half-life of 9.4 hours at pH 4. The major degradation products are benzazimide and hydrozymethyl benzazimide (combined residues >10%). Other degradation products include anthranilic acid and possibly its esters (<10% each) and methyl benzazimide (<10%).

Azinphosmethyl will be metabolized in a sandy loam soil by microorganisms. In nonsterile aerobic soil, the half-life is 21 days; in nonsterile anaerobic soil, 68 days; and in sterile soil, 355 days. Soilbound residues represent approximately 70% of the applied azinphosmethyl after 1 year of aerobic incubation. The metabolites present include benzazimide metabolites, which peak at approximately 120 days (at 12%) and decline thereafter, and oxygen analog metabolites, which peak at approximately 190 days (at 4.5%) and remain at that level through 1 year.

Azinphosmethyl applied at recommended rates to sandy loam soil does not appreciably affect the rate of nitrification.

Azinphosmethyl (2 ppm) does not inhibit growth or nitrogen fixation capacity of soybeans inoculated with Rhizobium japonicum.

At concentrations of 2-10,000 ppm, azinphosmethyl does not inhibit the growth of soil bacteria and actinomyces. Growth of soil fungi is not inhibited by azinphosmethyl at <10 ppm. Inhibition of fungal growth occurs at concentrations >100 ppm of azinphosmethyl.

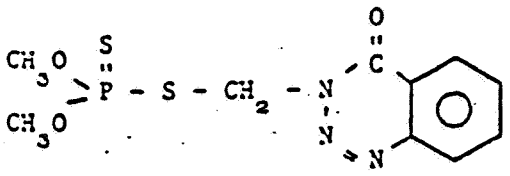
Studies show that aged residues of azinphosmethyl have a low leaching potential in soil. As shown by organic matter fractionation, radiolabeled azinphosmethyl residues and/or its metabolites are associated with the soil humic acid material after 28 days of aging.

The degradation products (Figure 1) are not volatile.

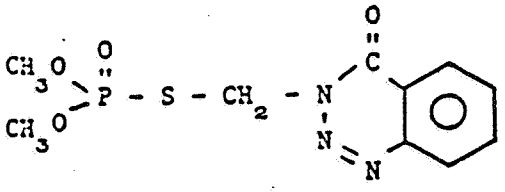
At recommended application rates, a large proportion of the pesticide (60-90%) is adsorbed onto soil. Desorption occurs at 9-27% per 24-hour desorption interval.

On the basis of available data, the half-life of azinphosmethyl in the field is less than 30 days when the 2L or 50 WP formulation is applied at 4 lb ai/A (Appendix 1). The oxygen analog of azinphosmethyl is not present in detectable quantities in the soil at days 0, 30, 60, or 90 after application. However, a total residue (azinphosmethyl and metabolites) of approximately 0.5 ppm is present in the top 6 inches of soil 30 days after a 4 lb ai/A application. The dissipation rate of either azinphosmethyl or DEF (S,S,S-tributyl phosphorotrithioate) is not affected by a combined application of azinphosmethyl and DEF.

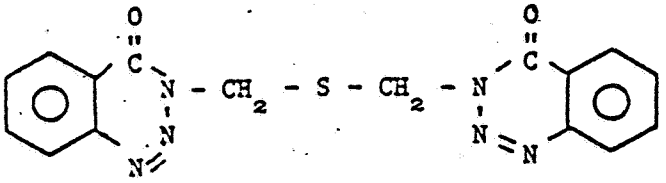
Potential residue levels in rotational crops are <0.01 ppm when they are planted 30 days or later after pesticide application at rate up to 8 lb ai/A. Root crops accumulate azinphosmethyl residues approaching 1 ppm or more if planted within 90 days after the pesticide application at rates as low as 1 lb ai/A.



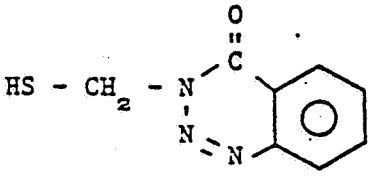
Azinphosmethyl (Guthion)



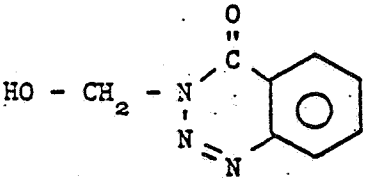
Azinphosmethyl oxygen analog



Bis-Methyl benzazimide sulfide



Mercaptomethyl benzazimide



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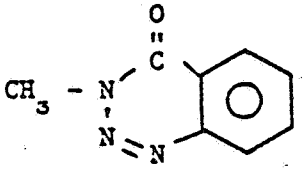
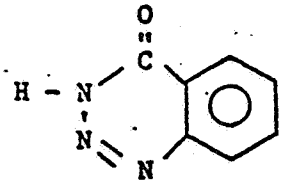
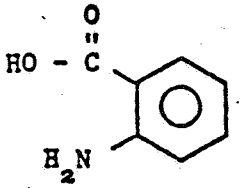


Methyl benzazimide



Benzazimide



Anthranilic acid

Figure 1. Structure and nomenclature of azinphosmethyl (Guthion) and its metabolites.

Appendix 1. Summary of soil persistence data for azinphosmethyl (Guthion) and its metabolites.[a]

Soil texture	Days after application	Residue (ppm) 0-6 inches Azinphosmethyl[b]	0-6 inches Total[c]	6-12 inches Azinphosmethyl[b]	6-12 inches Total[c]	Tab No.
Sandy loam	C[d]	<0.01	0.16	<0.01	0.05	67803
	0	0.32	0.75	0.01	0.14	
	30	0.01	0.31	<0.01	0.07	
	58	<0.01	0.20	<0.01	0.06	
Sandy loam	C	<0.01	0.31	--	--	78804
	0	4.21	5.01	0.51	1.51	
	61	0.03	0.60	0.01	0.41	
Sandy clay loam	C	<0.01	0.08	--	--	67805
	0	0.80	1.25	0.27	0.54	
	30	0.02	0.28	<0.01	0.18	
	60	0.04	0.41	<0.01	0.17	
Silt loam (muck)	C	<0.01	0.05	--	--	67806
	0	0.43	0.58	0.02	0.01	
	30	0.05	0.33	<0.01	0.02	
	61	<0.01	0.16	<0.01	0.03	
Sandy loam	C	<0.01	0.24	<0.01	0.14	67807
	0	0.22	0.62	<0.01	0.16	
	30	0.02	0.40	<0.01	0.08	
	58	<0.01	0.22	<0.01	0.11	
Sandy loam	C	<0.01	0.31	--	--	67808
	0	0.88	1.48	0.52	1.52	
	61	0.04	0.71	0.02	0.36	
Sandy clay loam	C	<0.01	0.13	--	--	67809
	0	0.53	0.89	0.62	0.95	
	30	0.04	0.41	0.02	0.23	
	60	0.02	0.29	<0.01	0.17	
Sand	C	<0.01	0.11	--	--	67810
	0	0.82	1.19	0.04	0.05	
	30	0.03	0.36	<0.01	0.12	
	61	0.02	0.35	<0.01	0.08	

[a] The rate of application was 4 lb ai/A, 2L formulation on 67803-67806 and 50WP formulation on 67807-67810.

[b] Determined by GLC (Tab No. 67084).

[c] Total azinphosmethyl and metabolite residues by fluorescence (Tab No. 67084).

[d] Control.