

# MONITORING OF THE 1982 GYPSY MOTH ERADICATION GROUND SPRAY PROGRAM IN SANTA BARBARA COUNTY



ENVIRONMENTAL HAZARDS

ASSESSMENT PROGRAM

State of California  
Division of Pest Management, Environmental Protection & Worker Safety  
Unit of Environmental Monitoring & Pest Management  
1220 N Street, Sacramento, California 95814

MONITORING OF THE 1982 GYPSY MOTH ERADICATION  
GROUND SPRAY PROGRAM IN SANTA BARBARA COUNTY

by

L.A. Neher, R.T. Segawa, R.J. Oshima

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Field Monitoring: R.T. Segawa, D. Duncan,  
R. Sava, F. Zalkin,  
L. Herschberger, S. Simpson

Sampling Methods: T.M. Mischke, J.R. Franz

Chemical Analysis: CDFA Chemistry Laboratory

Programming and  
Graphics: T. Arnold

Statistics: R. Gallavan, T. Younglove

Environmental Hazards Assessment Program  
California Department of Food and Agriculture

## ABSTRACT

The environmental monitoring results for the ground spray carbaryl applications were generally very predictable. Low levels of carbaryl (ppb range) were detected in the air during periods when the spray was being applied. Post-spray monitoring indicated that once spraying was completed, airborne levels dropped quickly into the parts per trillion (ppt) range, a factor of 1000 times lower. Only a single background sample was positive for carbaryl. This value was extremely small ( $0.016 \mu\text{g}/\text{m}^3$ ) and was attributed to spraying on adjacent properties.

Monitoring results for carbaryl residues on foliage provided an unexpected trend. Essentially no carbaryl degradation occurred on foliage over a 75 day period of time. These results were replicated under controlled conditions in a greenhouse with essentially the same outcome. Both the field foliage degradation of carbaryl applications and the greenhouse controlled study refute published results in the scientific literature where carbaryl applications of the same formulation degraded rather rapidly. It is hypothesized that the pesticide was trapped within diatomaceous earth particles and somehow stabilized but no experiments were conducted to prove this. Significant drops in carbaryl residues were associated with periods of rainfall.

Carbaryl residues were detected in the soil after application in the parts per million range. Water levels monitored in a stream after rain runoff were below 47 parts per billion. The water sampling provided data indicating that Sevin 80S or some other carbaryl formulation had been applied upstream of the eradication area. The extent of the contribution to the high levels found downstream of the application area could not be accurately estimated.

#### ACKNOWLEDGMENTS

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## I. INTRODUCTION

The Environmental Hazards Assessment Program (EHAP), California Department of Food and Agriculture (CDFA) was requested in January, 1982, to design an environmental monitoring sampling protocol (available on request) which would characterize potential gypsy moth aerial eradication sprays in Santa Barbara county. Results of the study would be disseminated to the agencies and physicians responsible for the evaluation of human health and environmental exposure impacts.

A survey of the anticipated study area was initiated in January, 1982. Numbers and locations of potential monitoring sites were cataloged to determine the population distribution of hospitals, schools, natural and man-made bodies of water, and other areas of concern. Since an eradication method had not been selected, preparations were made for a potential "worst case" aerial spray over a large area. Members of the County Agricultural Commissioner's staff assisted EHAP staff in locating, inspecting, and obtaining written permission to sample (see Appendix A) from 114 private residences, all hospitals, and schools located within the 46 geographical cells in Appendix B. Universal Transverse Mercator (UTM) cell coordinates were used to facilitate site location and mapping.

Following public hearings on February 24, 1982, an eradication plan was selected incorporating mass trapping using delta traps and + enantiomer pheromone, together with aerial applications of Bacillus thuringiensis (Dipel 4L), and limited applications of carbaryl.

Monitoring was restricted to the carbaryl ground spray operation because Dipel was known to be toxic only to specific insect populations. A new protocol specifically addressing only the ground spray program was written and implemented (see Appendix C).

## II. SITE DESCRIPTION

The majority of the treatment area was located within the Montecito area of Santa Barbara County. The three specific areas designated for the ground spray program, along with the geographical boundaries for the aerial Dipel applications, are shown in Figure 1. Each ground spray area incorporated all properties within either a 1/4 mile radius of a gypsy moth eggmass find (areas 1 & 2) or an area surrounding a trap location with multiple moth finds for two consecutive years (area 3). No properties outside of these three areas were treated with carbaryl by the eradication project. EHAP

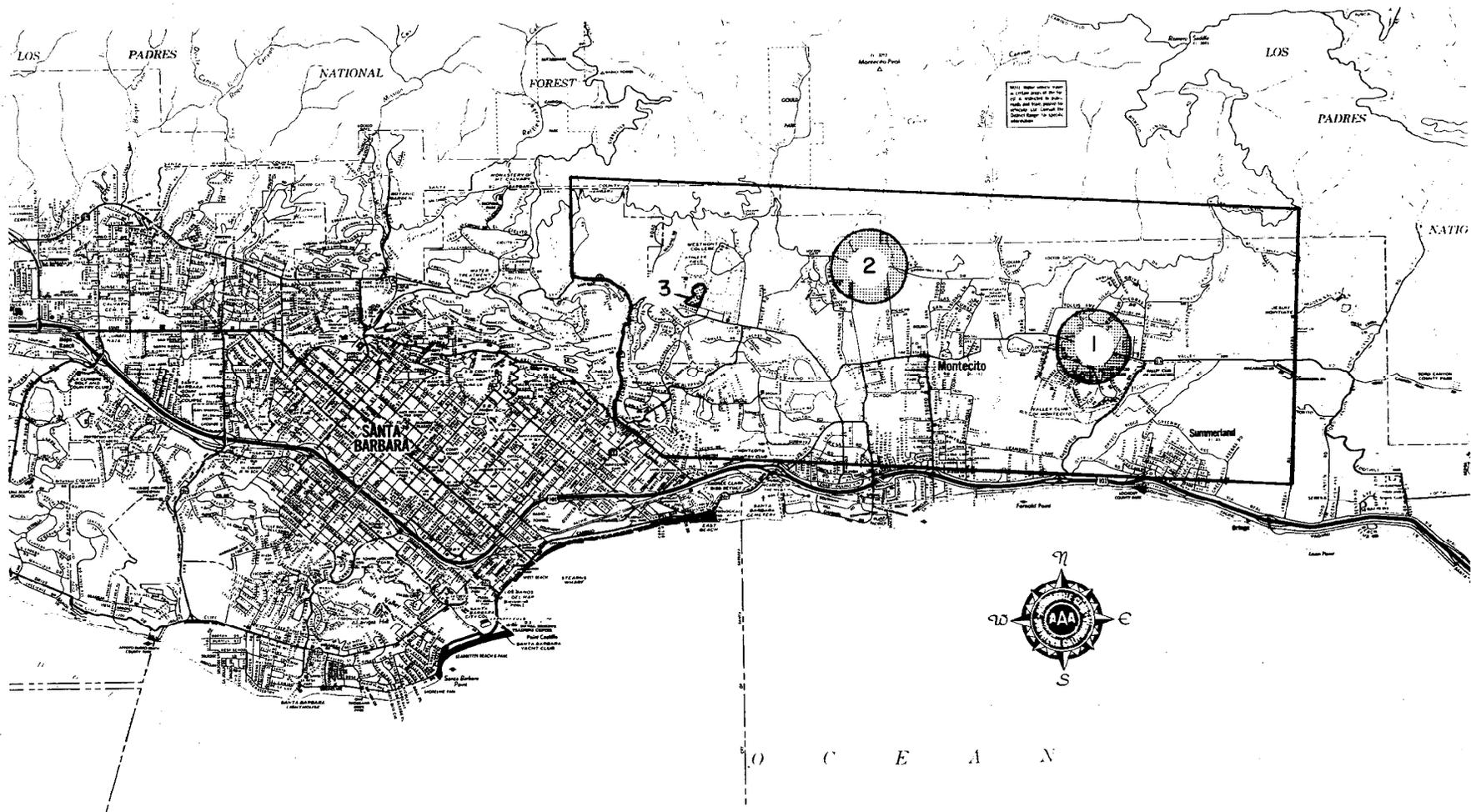


Figure 1. Geographical area covered by the ground spray protocol. The ground spray areas are numbered 1-3, surrounded by the boundaries for the Bacillus thuringiensis aerial spray area.

personnel selected one residence within each area for monitoring carbaryl levels in air, soil and foliage. Each residence was selected based on ease of access, availability of electricity, presence of suitable host foliage, and a compatible spray schedule to avoid the simultaneous spraying of the three monitored properties.

All spray areas were characterized by heavy tree and bush foliage. Altogether, the three areas contained 173 residential properties. The acreage treated within each area is shown below:

area one = 126 acres  
area two = 132 acres  
area three = 18 acres

Periods of measurable precipitation occurred in the treatment area during the study. No records of rainfall were kept in the study area itself, but the Santa Barbara airport (FAA), a distance of 10 miles, recorded the following data:

March 25 - 0.14 inches	April 1 - 0.34 inches	May 5 - trace
March 26 - 0.06 "	April 10 - 0.36 "	
March 28 - 0.22 "	April 11 - 1.41 "	
March 29 - 0.43 "		
March 31 - 0.65 "		

### III. APPLICATION TIME TABLE

The aerial application of Bacillus thuringiensis by helicopter began 24 March 1982, and continued weekly through 4 May 1982. The ground applications of carbaryl began 9 March 1982 and were completed on 4 May, 1982.

### IV. FORMULATION AND APPLICATION

The Sevin 80SP (80% active ingredient: carbaryl (1-naphthyl N-methylcarbamate) formulation contained 20% (wt.) diatomaceous earth, a highly porous, siliceous material. This formulation was mixed to a working concentration of 1.25 lb./100 gallons water; equivalent to 0.120% active ingredient or 1200 ppm. All mixing was done directly in each of the ten 100 to 500 gallon hydraulic ground spray trucks and kept under constant agitation during application. Workers operated adjustable pressure guns with #6 spray nozzles at 225-600 psi. pressure. The pressure was increased to reach the tops of trees or lowered to prevent damage to more fragile plants. A total of 163,000 gallons of the diluted formulation was applied during the 1982 carbaryl ground spray program.

Long hoses enabled applicators to walk throughout a property, spraying all sides of any designated "host" plant present on the grounds. A separate water system was then used to wash off any children's play equipment or lawn furniture. When present, non-host foliage which might be damaged by the spray or non-host personal gardens (at owner's request) would be tarped by eradication personnel.

## V. MATERIALS & METHODS

a. SAMPLE SECURITY Each sample collected by EHAP was accompanied by a chain of custody form documenting the sequence of transfers from sample medium preparation through chemical analysis (Appendix D). Every individual who handled the sample was required to sign and date the form, acknowledging receipt and relinquishment of the sample. This form was also designed for recording data and remarks to be keypunched into a computer file.

b. CHEMICAL ANALYSIS All chemical analyses were performed by the Chemistry Laboratory Services Unit of the California Department of Food and Agriculture at the main laboratory in Sacramento. The samples were kept at 4°C during transport and during storage before analysis.

Extractions were made with "Pesticide Grade" solvents. All analyses were performed using a Varian model 3700 Gas Chromatograph equipped with a Thermionic Specific Detector (TSD) operated in the nitrogen mode. The analyses utilized either a 1-1/2 ft. long, 20% OV-101 or a 1-1/2 ft long, 20% SP-2100 column. Both columns were operated at 190°C with the instrument detector operated at 220°C. Questionable results were checked using both columns. Sample chromatograms for the standard and leaf extracts are presented in Appendix E. Detailed descriptions of the extraction procedures for each sample type are given in their respective methods section.

c. QUALITY CONTROL In addition to the above, aliquots of selected extracts were sent to the Department of Health Services, Hazardous Materials Laboratory Section, for comparative analysis. The Hazardous Materials Lab utilized a high pressure liquid chromatography method as opposed to the gas chromatographic method employed by CDFA. Results of this comparison are given in Table 1. The data separates into two groups based on the large bimodal numerical separation. Those sample types with low carbaryl levels (soil and water) are not comparable with types having high carbaryl levels (leaf & air). Results for low concentrations do not represent two distinct populations (F test), indicating that the two analytical methods are comparable at this level.

However, a trend towards higher values from the CDHS laboratory is statistically significant (t test). Among the high concentration samples, a significant difference in populations does exist among the laboratories, but no significant trend was present. This last condition is less satisfactory since no significant bias exists, showing that a large amount of variability exists for high concentration analyses at both laboratories. The statistical tests used for these determinations are found in Appendix F.

TABLE 1. Results for the split sample carbaryl extracts performed by the CDFA Chemistry Lab and the CDHS Hazardous Materials laboratory.

SAMPLE TYPE	CDFA LAB <sup>a</sup>	CDHS LAB <sup>b</sup>
SOIL	0.0053 <sup>c</sup> < 0.022	0.02 < 0.05
WATER	< 0.001 < 0.0003 0.022	0.0066 0.0066 0.047
LEAF	255 <sup>d</sup> 1030	240 500
AIR	2640 153 509	1100 230 500

- a. Gas chromatography method.
- b. High pressure liquid chromatography method.
- c. Data in micrograms carbaryl per gram soil or water.
- d. Data in micrograms carbaryl.

d. TANK SAMPLING The spray mixture was sampled directly from the spray nozzle. One liter amber glass bottles with teflon lined caps were used to collect the sample immediately following the treatment of the tree used for foliage sampling. The sample was immediately packed in wet ice and kept on ice or refrigerated until analysis by the Chemistry Lab in Sacramento.

e. AIR MONITORING The number of air monitoring sites was set at three, one in each spray area. A single sampler was located at each property between the residence and treated foliage. A background sample of 24 hour duration was collected prior to the scheduled application. A second air sampling period was initiated at the start of the spray treatment, and terminated when the spray crews had completed treating the property. The length of these spray period samples could not be standardized as property sizes and the speed with which a spray crew could treat them varied. The presence of large amounts of lawn furniture or children's toys requiring removal or washing, protecting sensitive plants and/or areas to be tarped prior to spraying all added to the total spray period at any one property. A third post-spray air sample was initiated one half hour after the cessation of foliage spraying on a property. The sampling period for the post spray samples was 24 hours.

Modified General Metals Works High-Volume (HiVol) air samplers equipped with Kurz Instruments constant flow controllers were operated at 40 cubic feet per minute (CFM). Air being sampled was drawn through 4 inch diameter glass cartridges packed with pre-cleaned XAD-2 macroreticular resin. The adsorbant collection efficiency of the HiVol samplers was 100%.

All HiVol samples were immediately stored on dry ice following collection and kept frozen during shipment and prior to analysis at the Chemistry Laboratory in Sacramento. Resin was quantitatively transferred to 500 ml amber glass, wide mouth bottles and approximately 375 ml of acetone was added to each bottle. The bottles were covered with aluminum foil and placed in an ultrasonic bath (20°C) for one hour. The solvent was then decanted into a 90 ml Buchner funnel lined with sharkskin paper. Another 375 ml of acetone was added to the bottle and the contents were sonicated again for 20 minutes. The contents of the bottle were quantitatively transferred into the funnel. The bottle and the contents of the funnel were then rinsed twice with 100 ml aliquots of acetone and the collected solvent was evaporated under nitrogen to near dryness on a steam bath. The residue was picked up in hexane and brought up to a final volume of 10 ml.

The minimum detectable level for air samples was 50 micrograms of carbaryl per sample, with a recovery off the resin of 83%.

f. SOIL SAMPLING Background and one day post-spray soil samples were collected beneath the trees selected for foliage sampling. These samples were taken to verify that soil concentrations in the Montecito area would fall in the same range as previously documented values present in literature. Three replicate samples were collected from the top 5 cm of soil. Each sample was individually packed into 500 ml amber glass bottle with a teflon lined cap and immediately frozen on dry ice for transport to the State Chemistry Laboratory in Sacramento.

Stored samples were thawed and enough acetonitrile was added to the amber bottle to cover the sample (about 150 ml). The sample bottle was placed in the ultrasonic cleaner for one hour, then removed and the contents vacuum filtered through sharkskin paper. The soil remaining in the jar was rinsed twice with acetonitrile (50 ml each wash) and the washings were vacuum filtered through sharkskin paper. The combined filtrate and washings were run through anhydrous sodium sulfate to remove water and then concentrated on a rotoevaporator at 60°F to dryness. The sample was picked up in acetone, again rotoevaporated to dryness and finally picked up with hexane to a final volume of 10 ml.

g. WATER MONITORING A survey of the ground spray areas showed no exposed drinking water reservoirs or treatment plants. Only one active stream or creek could be found flowing through a treatment area: Sycamore Canyon Creek, area 3. Other named creeks were observed and found to be dry before and during rainfall periods. Replicate background samples were collected within the treatment area at Chelham Way and downstream of the treatment area at Westmont Road. These samples were collected April 5 & 6, one week prior to the first substantial rainfall. A third site upstream of the treatment area at Mountain Dr. did not have water present. Rainfall runoff samples were collected on 11 April 1982, after runoff water had begun to flow at all three sites.

Replicated samples were collected in one liter amber glass bottles, filled to capacity, and sealed with teflon lined caps. Water samples were packed in wet ice and kept on ice or refrigerated until analysis at the Chemistry Laboratory in Sacramento.

An 800 ml volume of water was extracted three times with separate 50 ml aliquots of methylene chloride. The combined extracts were then evaporated to dryness and picked up with hexane to a final volume of 5 ml.

h. FOLIAGE SAMPLING One tree was designated for leaf sampling by EHAP personnel at each of the three residences chosen for air sampling. Triplicate background leaf samples were collected the morning of the first scheduled treatment at each property. Spray samples were taken just after the spray residue had dried, post spray samples collected every other day up to the second treatment. At location two, sampling was reinitiated following the third and final ground spray with leaves being collected every fourth day for twenty days. Sampling continued at this location at 10 day intervals for an additional 30 days; extending to 12 June 1982, 50 days after spray three. All locations were also sampled on 7 July, 1982.

Whole leaves were collected at shoulder height by circling the entire tree. As time progressed, obvious new growth was avoided during collection. Triplicate samples were collected, each consisting of 15-25 leaves placed in 500 ml amber glass bottles with teflon lined caps. The bottles were immediately packed in wet ice and kept on ice or refrigerated until analysis at the Chemistry Laboratory in Sacramento. All leaves were placed in a leaf press following extraction and mailed to EHAP offices in Riverside where leaf areas and dry weights were determined.

Leaf samples were extracted with 100 ml of distilled water containing five drops of Surten soap. Samples were tumbled for one hour and the water decanted into a 500 ml separatory funnel. The leaf samples were then extracted again with 100 ml of the water and Surten solution for 20 minutes. The second extraction was decanted into the separatory funnel holding the first extraction. The leaf samples were then rinsed with 100 ml of pure distilled water and this rinse was also added to the separatory funnel. The contents of the separatory funnel was then extracted three times with 50 ml aliquots of dichloromethane. Each dichloromethane extract was run through a funnel containing anhydrous sodium sulfate. The dried dichloromethane extracts were collected in a 250 ml round bottom flask. The samples were then evaporated to dryness at 40 C on a rotoevaporator. The residue was picked up with hexane and brought to a final volume of 10 ml.

The minimum detectable level was 2 micrograms of carbaryl per sample with a recovery off the leaves of 92%.

i. DEGRADATION STUDY Initial results from foliage sampling suggested the need for a greenhouse study to document the degradation of Sevin 80SP residues under controlled conditions. Such a study was initiated in June, 1982, at Sacramento, using a quantity of the ground spray program tank mix transported from Santa Barbara.

A pipette was used to transfer 5 ml of the tank mix into each of 130 prenumbered 4 inch petri dishes. After the spray mix had dried the dishes were placed beneath cardboard boxes on benches in a greenhouse located near the State Chemistry lab. Ambient temperature and relative humidity were recorded for the duration of the study.

Five dishes were collected daily according to a random number list for days 2 through 14. Collection was made every 3rd day for days 15 through 50. The dishes were extracted immediately following collection and the extracts frozen. All extracts were then analyzed at the completion of the study. Extractions were made with the identical method described for foliage samples.

## VI. RESULTS

a. **TANK SAMPLES** Tank sample results showed a high variation (Table 2). The practice of "topping off" the spray rigs with additional spray mix at the completion of the day's application schedule may have been a source for mixer error in estimating the quantity of water and pesticide required, but no specific study was done to assess the variation observed in tank concentrations.

TABLE 2. Concentrations of carbaryl (ppm) in tank samples during the ground spray program starting 24 March, 1982 and ending 4 May, 1982.

SAMPLE TYPE	LOCATION ONE			LOCATION TWO			LOCATION THREE		
	DATE	REPLICATE		DATE	REPLICATE		DATE	REPLICATE	
		1	2		1	2		1	2
Tank Samples <sup>a</sup>	3-25	1530	1560	4-02	640	610	4-21	336	n.d. <sup>b</sup>

a. Tank samples collected immediately following the sprayings of each location.

b. n.d. = no data collected.

b. **AIR SAMPLING** The air sampling data for the first ground spray application to each property is shown in Table 3. Location three was monitored twice because the spray period sample was lost during the first series. It should be emphasized that these air sampling values represent single, unreplicated data points - not mean values. All air monitoring data represent time-weighted averages. Background and post spray periods were of equal length and are

therefore directly comparable. The one exception occurred at location 2 when a gardener unplugged a sampler to mow a lawn. Spray period samples were collected for varying lengths of time and therefore cannot be directly compared with any of the air sample data points.

TABLE 3. Concentrations of carbaryl ( $\mu\text{g}/\text{m}^3$ ), expressed as a time weighted average, in air samples collected outside of residences during the first two weeks of the ground spray program.

SAMPLING PERIOD	LOCATION ONE		LOCATION TWO		LOCATION THREE			
	DATE	CARBARYL	DATE	CARBARYL	DATE	CARBARYL	DATE	CARBARYL
Background (24hr) <sup>a</sup>	3-23	0.0	3-29	0.0	4-07	0.068	4-17	0.016
1st spray	3-24	12.00 (80 min.)	4-02	0.160 (330 min.)	4-08	n.d. <sup>b</sup>	4-21	2.318 (8 min.)
2nd spray <sup>c</sup>								
Post Spray (24hr)	3-25	0.120	4-03	0.160 (270 min.)	4-09	0.153	4-22	0.306

a. Where the sampling period was other than 24 hours, the length in minutes is shown next to the carbaryl value. Sampler flowrate was set at 40 cubic feet per minute.

b. n.d.= no data collected.

c. Second spray sampled only at location three.

It is felt that the small lot sizes in spray area 3 may account for the presence of airborne carbaryl detailed in the background samples. Nearby properties were being sprayed by project personnel during the background sampling period. The large difference in lot sizes between areas 1 & 2 and area 3 is demonstrated by the length of time required to spray the properties. Areas 1 and 2 required 80 and 330 minutes respectively, while area 3 required only 8 minutes.

c. SOIL SAMPLING Results for soil sampling are shown in Table 4. Background carbaryl levels in soil were all zero (none detected). Samples collected one day post spray showed a significant difference between locations, but a much lower variability occurred within respective samples at each location. The results seem consistent in view of the variability seen in the application concentration and that variability expected between any two locations in terms of biological activity and environmental conditions.

TABLE 4. Concentrations of carbaryl (ppm; weight:weight) found in soil before and after the first ground spray application to each of the three properties.

SAMPLING PERIOD	LOCATION ONE				LOCATION TWO				LOCATION THREE			
	DATE	REPLICATE			DATE	REPLICATE			DATE	REPLICATE		
		1	2	3		1	2	3		1	2	3
Background	3-24	0.0	0.0	0.0	3-27	0.0	0.0	0.0	4-08	0.0	0.0	0.0
1st day Post Spray	3-25	5.5	10.5	7.5	4-03	0.09	0.05	0.0	4-09	0.21	1.49	0.39

d. WATER SAMPLING Carbaryl concentrations found in Sycamore Canyon Creek, ground spray area 3, are given in Table 5. No carbaryl was detected in background samples. However, following the first significant rainfall, a single

sample of 33.0 ppb was found upstream of the spray area. A single sample of 3.1 ppb was found within spray area 3 and values of 47 and 44 ppb were found downstream. The two single sampling points reflect replicate samples lost due to glass bottle breakage. The levels upstream suggest that carbaryl had been used by persons other than the eradication project spray crews and the residue was subsequently carried to the creek by runoff.

TABLE 5. Concentrations of carbaryl (ppb) in Sycamore Canyon Creek during rain runoff period.

SAMPLE TYPE	DATE	UPSTREAM <sup>a</sup> REPLICATE		SPRAY AREA REPLICATE		DOWNSTREAM REPLICATE	
		1	2	1	2	1	2
Background	4-05	DRY <sup>b</sup>	DRY	0.0	0.0	0.0	0.0
Post-rain	4-11	33.0	n.d. <sup>c</sup>	n.d.	3.1	47.0	44.0

a. For actual locations refer to the text.

b. Stream dry at this location.

c. No data available.

Dilution from additional runoff occurred in the 1/2 mile distance between the upstream sampling site and the spray area. Drainage patterns from sprayed properties emptying into the creek are not known, but the downstream concentration would represent the combined effect of carbaryl residue washing out of the upstream drainage and the ground spray area. A large dilution factor would be expected in the 3 mile distance from the spray area to the ocean. The concentrations observed are not unexpected considering the large surface area treated 3 days previously.

e. FOLIAGE SAMPLING The results from background foliage samples verified that carbaryl was not present on any of the sampled trees prior to the gypsy moth ground spray program (Table 6). The variation seen among the three replicate spray or post-spray samples for any location-day was fairly be consistent throughout the study. The individual samples noted in Table 6 as missing leaf area measurements had chemical analysis results similar to their respective replicates. However, without the leaf area measurement, neither value can be utilized for quantitative comparison.

TABLE 6. Concentrations of carbaryl ( $\mu\text{g}/\text{cm}^2$ ) extracted from surfaces of leaves collected from three spray locations during the monitoring period: 23 March, 1982 through 7 July 1982.

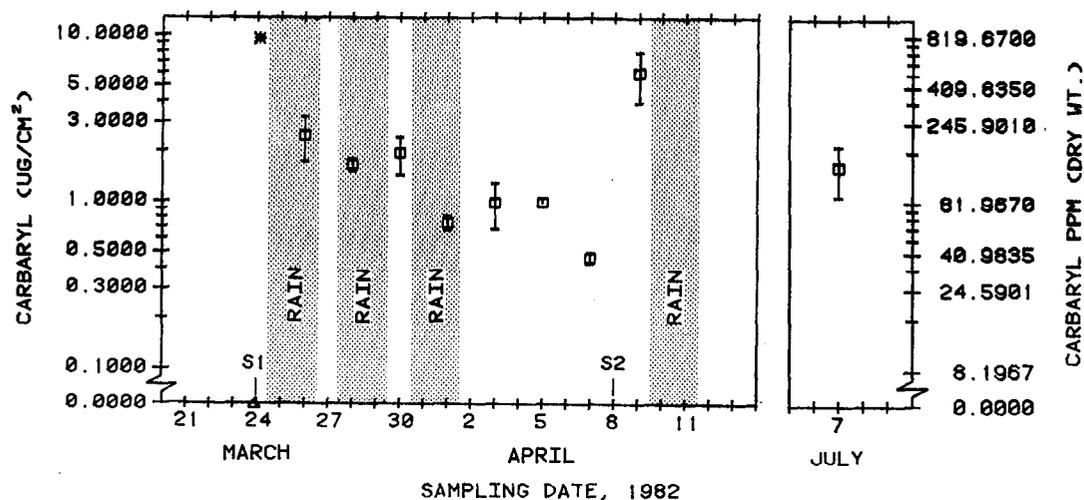
SAMPLING PERIOD	LOCATION ONE				LOCATION TWO				LOCATION THREE				
	DATE	1	2	3	DATE	1	2	3	DATE	1	2	3	
Background	3-24	0.0	0.0	0.0	3-27	0.0	0.0	0.0	4-08	0.0	0.0	0.0	
1st Spray Period	3-24	9.88	9.49	8.95	4-02	1.80	3.14	3.76	4-08	5.34	7.16	10.10	
#Days Post Spray 1:	2	3-26	3.97	1.50	1.94	4-04	2.22	3.30	3.80	4-10	3.64	3.46	3.08
	4	3-28	1.47	1.93	1.52	4-06	2.31	3.02	3.17	4-12	0.93	1.49	1.24
	6	3-30	2.82	1.88	1.07	4-08	2.27	3.82	3.48	4-14	0.23	1.09	0.67
	8	4-01	0.62	0.72	0.87	4-10	0.12	1.52	1.02	4-16	1.93	1.02	0.88
	10	4-03	1.56	0.56	0.81	4-12	0.25	0.38	0.17	4-18	1.07	0.55	1.10
	12	4-05	n.d. <sup>a</sup>	1.27	0.69					4-20	n.d.	1.66	1.27
	14	4-07	0.52	0.40	0.46								
2nd Spray Period	4-07	not sampled <sup>b</sup>			4-13				4-22	6.51	6.68	7.65	
#Days Post Spray 2:	1				4-14	7.14	2.73	2.76					
	2	4-09	2.13	8.93	6.75								
3rd Spray Period	4-19				4-23				5-04				
#Days Post Spray 3:	4				4-27	4.72	5.97	9.45					
	8				5-01	4.34	7.35	9.01					
	12				5-05	5.29	5.95	7.99					
	16				5-09	3.55	4.25	3.67					
	20				5-13	3.80	3.25	3.10					
	31				5-24	10.24	7.31	3.49					
	40				6-02	7.47	13.34	9.04					
	50				6-12	9.85	5.39	9.03					
	64				7-07	6.03	3.98	5.18	7-07	1.56	2.37	0.33	
	75												
	79	7-07	0.85	2.68	1.35								

a. Leaf area data not available.

b. Where no data is presented, locations were not sampled on that date.

Figures 2 and 3 plot the means and standard errors of the mean for the micrograms of carbaryl per square centimeter leaf area data presented in Table 6. When the standard error bars would plot inside of their respective mean symbol, the error bars were suppressed for clarity. In the

LEAF RESIDUAL - LOCATION ONE



LEAF RESIDUAL - LOCATION THREE

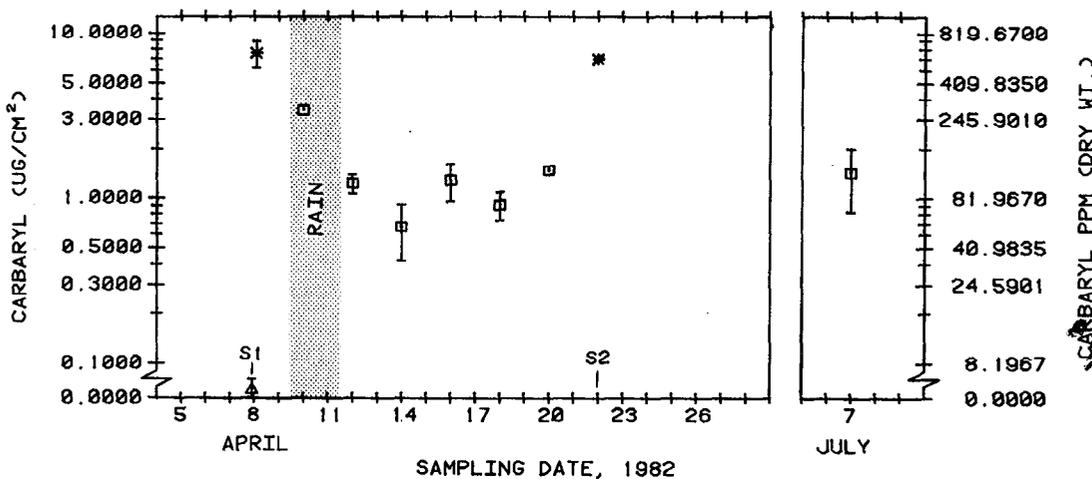


Figure 2. Dislodgable foliar residues of carbaryl for locations one and three, either  $\mu\text{g}/\text{cm}^2$  leaf area or as ppm dry wt. Each point is the mean of 3 samples except for 5 April-location one and 20 April-location three, which are the mean of two samples. The standard error for the means of three samples is given by the vertical bars. S1, S2, correspond to the dates of spray 1 and spray 2 respectively.  $\Delta$  = background samples, \* = spray samples,  $\square$  = post spray samples.

# LEAF RESIDUAL - LOCATION TWO

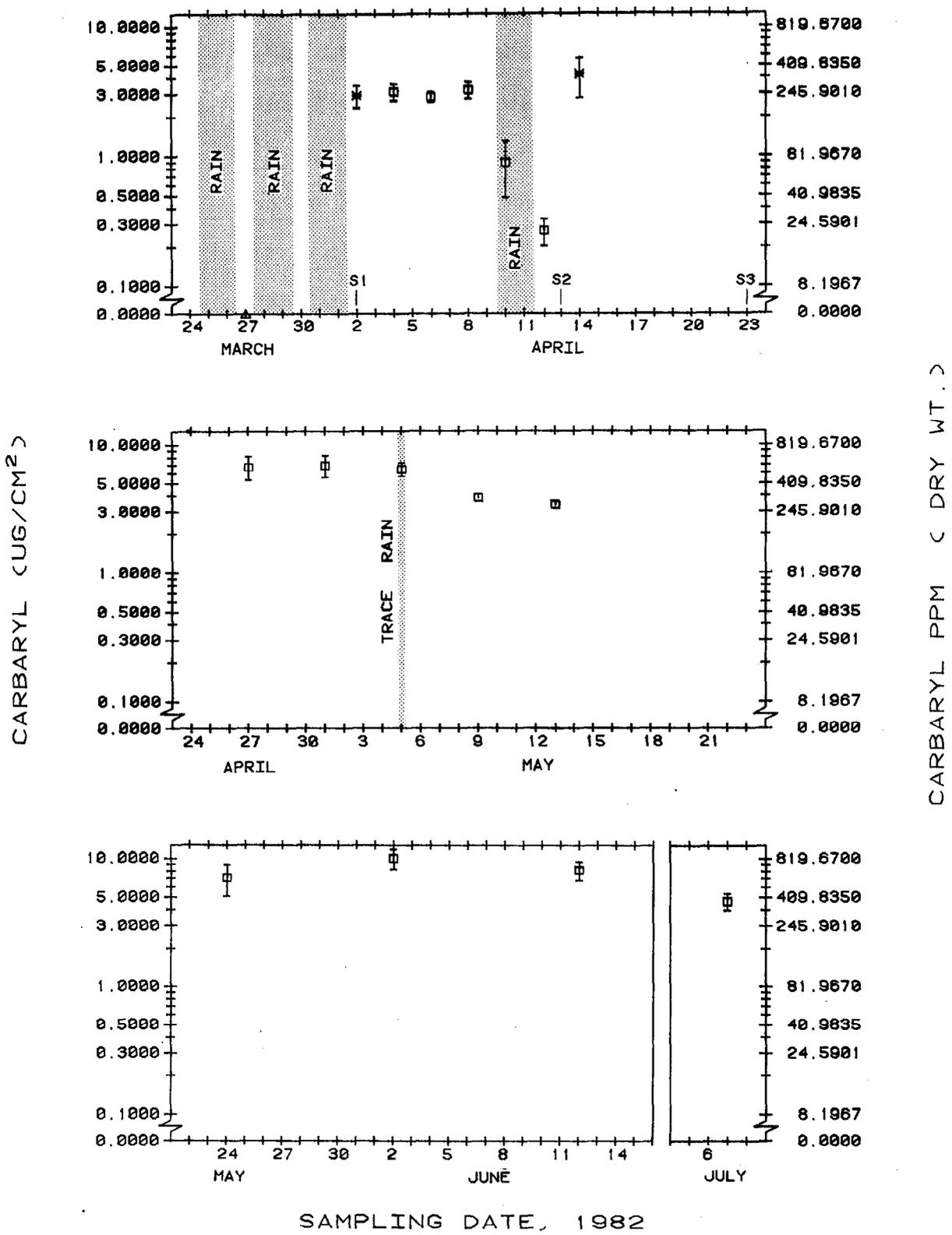


Figure 3. Dislodgable foliar residues of carbaryl for location two, shown as either ug/cm<sup>2</sup> leaf area or ppm dry wt. Each point is the mean of 3 samples. S1, S2, S3 correspond to the dates of spray 1, spray 2, and spray 3, respectively. Δ = background samples, \* = spray samples, □ = post spray samples. The standard error of the means is given by the vertical bars.

two cases discussed earlier, where only two replicate samples were available, no S.E. was calculated.

An additional Y-axis has been plotted on Figures 2 and 3 showing the estimated ppm carbaryl calculated on a dry weight basis. One overall mean of dry weight for the three locations was used for this calculation. The decision to use one overall mean was based on a study comparing individual values for each location. Data on the gram dry weight leaf tissue per  $\text{cm}^2$  leaf area collected is presented in table 7. The subsequent statistical analysis, showing no significant difference to be present among the locations, is exhibited in Appendix G. A Bartlett's test for homogeneity of variances (Chi-square) was performed first, followed by an F test on the means to determine whether significant differences in dry weight existed among locations. The equivalent analysis for mean separation shifts from a t test to an F test, as the sample size goes from 2 to 3 values. For clarity, both the Chi-square and F tests are presented: each showing no significance. Earlier spot checks of the dry wt/ $\text{cm}^2$  relationship made throughout the study all yielded data values within the range of the data in Table 7, but these earlier values were not statistically comparable data points; and as such, were not included in the analysis.

TABLE 7. Dry weights of leaf samples ( $\mu\text{g}/\text{cm}^2$ ) collected from the three monitoring locations on 7 July, 1982.

LOCATION ONE	LOCATION TWO	LOCATION THREE
0.011	0.010	0.012
0.017	0.010	0.013
0.013	0.015	0.012
	0.014	

The variation in foliar residue results between each of the three locations was less than would have been expected considering the tank sampling results. Differences between locations of leaf surface characteristics and orientation, would influence the amount of diatomaceous earth (containing carbaryl) retained following a saturation ground spray. The orientation of leaf surfaces would also have affected retention of the dried residue during the measurable rainfalls in the study area (see VI, and Figures 2 & 3). The immediate drop in residue levels associated with periods of rainfall can be seen at all three locations.

The association between precipitation and significant reductions in residues of Sevin 80S on foliage have been previously reported (Kuhr and Dorough, 1976). Further, aged residues were considerably more persistent to weathering from rainfall than fresh material. The data of Iwata et al (1979) shows a first order degradation over a 60 day study period. The authors felt that a 40 mm rainfall 25 days

after application did not appear to remove a significant amount of Sevin 80S residues. However, the periods of rainfall did coincide with large drops in residue levels for both orange and lemon leaves. Sampling in their study did not continue long enough to establish if the residue levels would have remained stable without rainfall.

The results of our study indicated that no significant degradation of the Sevin 80S occurred on trees in the Montecito environment during the 75 day period. These results differ sharply from other studies which reported a half-life of Sevin 80S of 12 days on citrus (Iwata, 1979) and 1-3 days on cotton (Ware, 1978). High temperatures exceeding 100° F were present in both these studies, but the relationship of high temperatures is unknown.

If one ignores rainfall, initial foliar residues at locations 1 and 3 seemed to indicate that carbaryl degradation was taking place. However, it would be unrealistic to summarily dismiss the influence of rainfall. Rainfall appeared to be the only factor related to significant decreases in foliar carbaryl levels. This was supported by the first results from location 2, where the effect of precipitation can be clearly seen 8 days after treatment. Sampling was continued at location 2 without the expected decrease in

dislodgable carbaryl residues. It was noteworthy that for this period no substantial rainfall occurred.

Statistical tests indicate that although residues varied among sampling dates at location 1 (F test), no significant degradation occurred (t test of regression slope). Analyses of results from locations 2 and 3 provided similar results (Appendix H). In no case was a significant downward trend established. At location 2 it is not appropriate to test for a downward trend, because the slope is positive.

The decrease of carbaryl foliar residues associated with rainfall has been previously documented. (Argauer and Webb, 1972) conducted a 16 day study that simulated precipitation on tomato leaves. Leaves exposed to rainfall had a carbaryl residue level of 386 ppm, leaves with no rainfall had a level of 940 ppm.

f. CONTROLLED DEGRADATION STUDY The results for the controlled degradation study are given in Table 8. No significant degradation of carbaryl exists in this data, corroborating the foliage sampling results. Daily mean values (mg carbaryl per ml) over time are plotted in Figure 4, with standard error of the mean bars. A significant difference exists among the means for days (F test), but the slope of

the regression line is not significantly different from zero. The tests used are shown in Appendix I. The  $R^2$  value of 51.8% indicates that the regression function is not very useful as a predictive tool since only approximately 1/2 of the variability present in the data is accounted for by the regression.

TABLE 8. Concentrations of carbaryl (PPM) extracted from petri dishes during the degradation study.

DATE	ELAPSED TIME (DAYS)	REPLICATE					MEAN VALUE
		1	2	3	4	5	
6-22	1	93	83	60	64	68	73.6
6-23	2	85	80	85	55	63	79.8
6-24	3	74	105	71	95	54	79.8
6-25	4	83	76	66	78	74	75.5
6-26	5	73	65	69	70	67	68.8
6-27	6	66	69	79	53	n.d.*	66.8
6-28	7	70	61	45	45	59	56.0
6-29	8	45	58	36	63	55	51.4
6-30	9	53	137	57	n.d.	n.d.	82.3
7-1	10	53	44	85	62	n.d.	61.0
7-2	11	55	53	53	38	n.d.	49.8
7-3	12	72	42	48	55	64	56.2
7-4	13	54	32	39	33	n.d.	39.5
7-5	14	67	91	99	108	80	89.0
7-8	17	23	55	53	41	35	41.4
7-11	20	36	9	73	39	34	38.2
7-14	23	19	46	23	47	n.d.	33.8
7-17	26	48	44	40	54	40	45.2
7-23	32	20	20	7	10	47	20.8
7-29	38	32	32	20	n.d.	n.d.	28.0
8-1	41	35	11	54	31	20	30.2
8-4	44	34	53	40	22	42	38.2
8-7	47	40	38	50	68	31	45.4
8-10	50	15	65	38	38	n.d.	39.0

\* Data not available.

# CARBARYL DEGRADATION

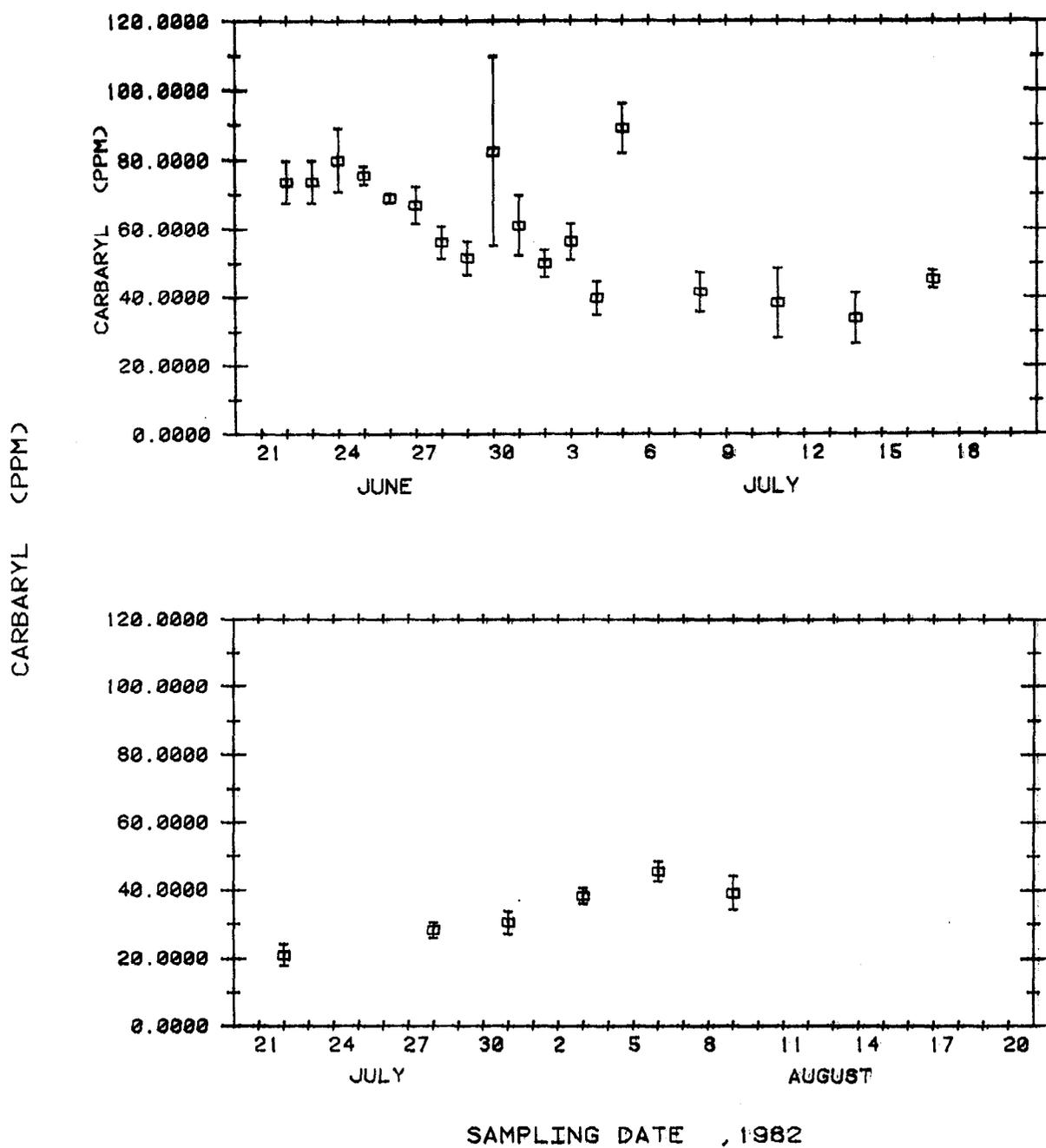


Figure 4. Carbaryl residues from controlled degradation. Each point is the mean of at least 3 samples. The standard error of the means is given by the vertical bars.

## VII. CONCLUSIONS

Carbaryl residues from the environmental monitoring of the ground spray program were generally very low. Levels of carbaryl detected in soil one day post-spray were in the ppm range. Air sampling results range from a high value of 12.0  $\mu/m^3$  (10 ppb) monitored during a spray, to a low of 0.016  $\mu/m^3$  (1.9 ppt) monitored during a background period. As expected, the highest airborne concentrations occurred during the spray period. Post spray air levels were many orders of magnitude below the spray levels. Only a single positive sample in the ppt range was detected during background air sampling.

Water samples taken immediately downstream of the ground spray areas, showed concentrations as high as 46 ppb. It was however, impossible to determine the contribution of carbaryl from the ground sprays since 33 ppb was detected at the upstream site. This site was approximately 1/2 mile upstream of the ground spray area. This value most likely is a result of the general use of carbaryl (Sevin) by landscape maintenance companies and the general public.

Foliage residues were unique in that the expected decay pattern did not occur. Caution must be used in the

interpretation of these results. The lack of significant degradation does not necessarily mean that carbaryl would continue to be lethal to gypsy moth larvae. The carbaryl may have become imbedded in the diatomaceous earth particles used in the formulation. If this was the case, the material may not have been susceptible to the normal degradative process which acts on exposed chemicals. Studies are needed to test the efficacy of aged Sevin 80S residues to gypsy moth instars.

Studies using Sevin-4-oil formulation have shown little loss of residual activity over time. Skoog (1971) showed no decrease in the % mortality to grasshoppers 12 days post-treatment even with a simulated half inch rainfall 8-9 hours after application. Markin, et al (1978) found no decrease in the % mortality to tussock moth larvae over 14 days post-treatment with a 0.3 inch rainfall 7-10 hours after application. Care must be taken in interpreting these results since the time periods differed considerably and formulation was different.

## LITERATURE CITED

Iwata, Y., M.E. Dusch, G.E. Carman, and F.A. Gunther. 1979. "Worker Environment Research: Residues from Carbaryl, Chlorobenzilate, Dimethoate, and Trichlorfon Applied to Citrus Trees," Agriculture and Food Chemistry 27(6): 1141.

Kuhr, R.J. and H.W. Dorough. 1976. Carbamate Insecticides: Chemistry, Bio-chemistry and Toxicology. CRC Press, Cleveland. 301 pp.

Ware, G.W., B. Estes, and W.P. Cahill. 1978. "Dislodgable Insecticide Residues on Cotton (1975)", Bull. Environm. Contam. Toxicol. 20: 17-19.

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Argauer, R., and R. Webb. 1972. "Rapid fluorometric evaluations of the deposition and persistence of carbaryl in the presence of an adjuvant on bean and tomato leaves," J. Agr. Food Chem. 20(3): 732-734.

## DEPARTMENT OF FOOD AND AGRICULTURE



1220 N Street  
Sacramento  
95814

During the period March to July the Department of Food and Agriculture's Environmental Hazards Assessment Program will be studying the impact of selected agricultural chemicals used to control the Gypsy Moth in this area of the State. We request your permission to enter your property and obtain samples of fallout, the ambient air inside and outside of your dwelling and of foliage on your premise. \_\_\_\_\_

\_\_\_\_\_

The exact dates and times will be arranged with you prior to obtaining the samples.

Brief description of actual location: \_\_\_\_\_

\_\_\_\_\_

Signature of Property  
Owner Granting Permission: \_\_\_\_\_ Date: \_\_\_\_\_

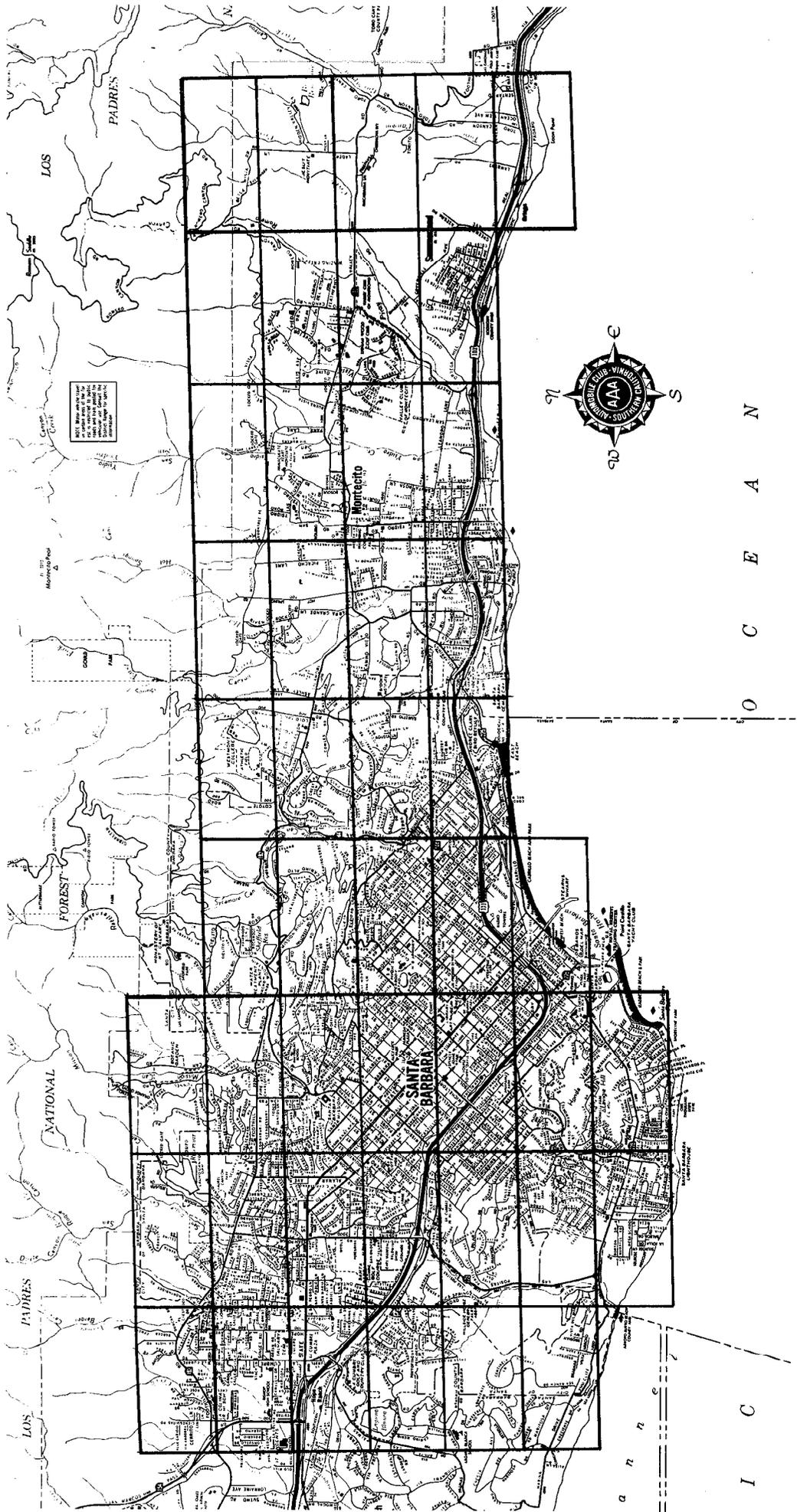
Property Address: \_\_\_\_\_

\_\_\_\_\_

Phone Number: \_\_\_\_\_

If any problems should arise, please contact:

Ron Oshima  
(916) 322-2395



Appendix B. 46 cell boundaries for the geographic area covered by the potential aerial spray protocol. Each cell's dimensions are 1km x 2km.

PROTOCOL FOR THE 1982 GYPSY MOTH ERADICATION GROUND SPRAY  
PROGRAM MONITORING IN SANTA BARBARA COUNTY

I. Objective

To monitor the environmental levels of the pesticides applied during the Gypsy Moth Eradication effort.

II. Personnel

The Gypsy Moth eradication ground spray program will be under the overall supervision of Ronald J. Oshima, Environmental Hazards Assessment Program (EHAP) (Phone 916-322-2395 or ATSS 492-2395). Key personnel participating from EHAP-CDFA are listed below:

Lee Neher - Responsible for the study design, supervision over sample collection and data processing of results. Phone (714) 787-4684 or ATSS 651-4684.

Tom Mischke - Responsible for selection of sampling methodology, field storage and transport of collected samples, and liaison to CDFA Chemistry Laboratory Services for questions concerning all aspects of the chemical analysis of collected samples. Phone (916) 322-2395 or ATSS 492-2395.

EHAP sampling personnel will consist of one crew of two people. It is understood that the Gypsy Moth Eradication Project will assist in locating the monitoring locations.

III. Study Timetable

Projected time for the outlined study is March 15 through April 15, 1982.

IV. Monitoring Plan

A maximum of three residential properties randomly selected within the total treatment zone will be sampled. Samples will determine pre-spray background levels and the post-spray levels of the pesticide applied.

V. Sampling Methods and Monitoring Timetable

Sampling will be separated into four tasks: first, to quantify the presence or absence of detectable air concentrations at treatment properties; second, to quantify the concentration per unit area on treated foliage; third, to quantify the concentration found in soil underneath treated foliage; fourth, to quantify the impact on existing water bodies. Tank samples will be collected from the application equipment to establish a baseline for all sampling.

Section I - Air Monitoring

Air monitoring equipment will sample ambient air outside of the residence for 24 hours prior to the treatment, during the actual treatment period and for 24 hours post treatment. One residential property will be selected within each defined  $\frac{1}{4}$  mile radius treatment area up to the maximum of three sites total. Should a treatment zone encompass a school for which permission to sample has been obtained, air samples will be collected using the time table for residential air monitoring. Hi-volume air samplers, utilizing an adsorbant resin bed and electronic flow controllers, will operate at a flow rate of 30 cubic feet per minutes (30 CFM).

Section II - Foliage Sampling

A representative foliage type scheduled for treatment will have a replicated leaf tissue sample collected prior to the treatment, the day of the treatment

and every other day up to and including the second pesticide application. Following the final application at one residence, foliage samples will again be collected at four day intervals, for a 20 day period. Foliage sampling will be done at each residential property selected under Section I. The actual sample will consist of 20-30 leaves, collected in glass jars and analyzed for dislodgeable pesticide residue. Each sample will also be run through a leaf area meter to obtain the  $\text{cm}^2$  area.

#### Section III - Soil Sampling

Replicate soil samples will be taken prior to the scheduled application and on the day following the application. Soil sampling will be done at each residential property selected under Section I. The top 2 inch level will be sampled, and subsequently extracted and analyzed for residues of the treatment pesticide.

#### Section IV - Impact on Existing Water Bodies

- a) Two replicate water samples will be drawn from any exposed public drinking water reservoirs or treatment plant located within the treatment area prior to pesticide release and again immediately following pesticide release in the area.
- b) Two replicate water samples will be drawn from any stream or creek flowing through a treatment area in the event that an appreciable rainfall occurs within 14 days of ground treatment. These samples will include a background sample downstream of the treatment area and post-rain samples, both above and below the treatment area.

VI. Handling and Storage of Samples

All sampling media and containers will be prepared and pre-numbered at the California Department of Food and Agriculture Laboratories in Sacramento. Each device or container will be shipped to the sampling sites with an accompanying Chain of Custody Record (see attachment). The Chain of Custody Record will be filled out by all parties handling or storing the sampling media or sample containers from the time they leave the Sacramento CDFA lab until they are returned to the lab for analysis. The Chain of Custody Record also contains an internal chain of custody record for use by the laboratory.

All samples will be collected by EHAP personnel, sealed in glass containers and stored in the following manner until and during transport to the CDFA laboratory in Sacramento.

On Dry Ice (-70°C)

air samples

foliage samples

soil samples

On Ice (4°C)

tank samples

water samples

VI. Analysis of Samples

All samples will be analyzed for the presence of the pesticide by CDFA Chemistry Laboratory Services. Quality control duplicate samples will be analyzed by CDFA and an alternate, EPA approved laboratory. If deemed necessary, selected samples may also be analyzed for other known breakdown

products of the selected pesticide. Approximately ten percent of the total number of each type of sample collected will have duplicate analysis performed as part of the quality control program. Sample analysis by the CDFA laboratories will be prioritized to allow for rapid access to critical data. Brief details of the analytical methods for each type of sample are available, if requested.

STATE OF CALIFORNIA  
DEPARTMENT OF FOOD  
AND AGRICULTURE

CHAIN OF CUSTODY RECORD  
Use Ball Point Pen Only

ENVIRONMENTAL MONITORING  
ENVIRONMENTAL HAZ. ASSESS. PROG.  
1220 N STREET, ROOM A-328  
SACRAMENTO, CA. 95814

Study #	Sample #	Sampler Date on				Sampler Date off				Collector	Cell Location Code		Spray	BSPF	Key *	Key EO	Key	Sample Type
		Mo	Day	Yr.	Time on	Mo	Day	Yr.	Time off			Site						
1	7			8	2			8	2									

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40

Companion	Bad Sample																	Chemist	Priority
UCD/CDFA																			

41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80

Remarks, Chemicals, Observations,  
Type of Biota, etc.

Partner: Rep #

Lab Results

Save Leaves

Carbaryl

Dimilin

Acephate

Monitor

Chemist

Date

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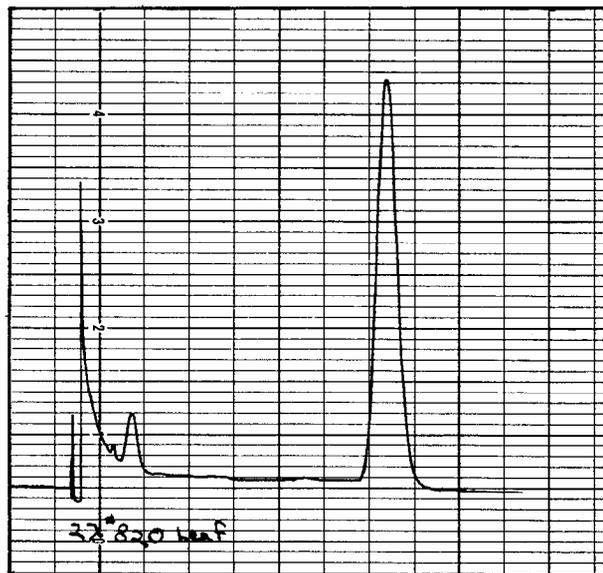
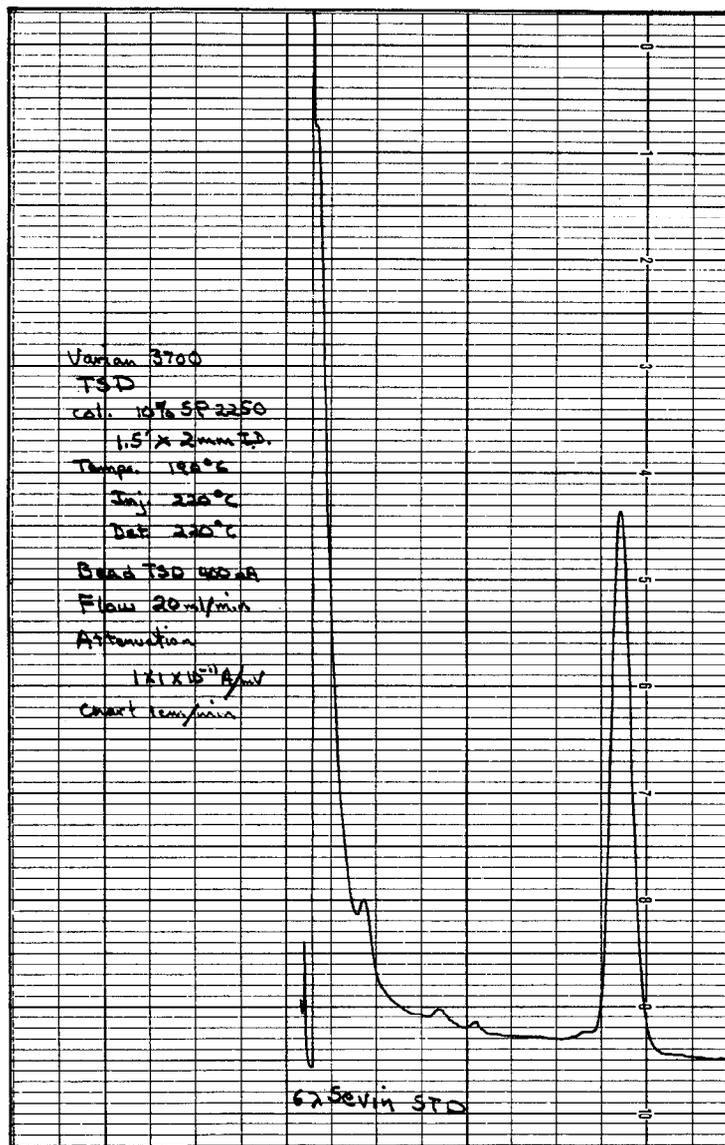
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- Key
- COL 36: B= Background TAN= Tank  
S= Spray HIV= Hi-Vol  
P= 1st Post LOV= Lo-Vol  
F= 2nd Post BIO= Biota
- COL 37: \*= Flagged FAL= Fallout CDFA  
E= Extra UCD= Fallout UCD  
O= Outside WAT= Water  
FLO= Floater  
SOI= Soil  
LEA= Leaves

Appendix 'E' Chromatograms of the Carbaryl Standard (Left) and a Leaf Extract (Right).



Appendix F: Comparison of Carbaryl Analyses Using Split Extracts

Low Concentrations

	Hazardous Materials	Dept. of Food & Agriculture		
	<u>Lab Analysis</u>	<u>Lab Analysis</u>	<u>Σ</u>	<u>Diff.</u>
1	.0053	.02	.0253	.0147
2	.022	.05	.072	.028
3	.001	.0066	.0076	.0056
4	.0003	.0066	.0069	.0063
5	.022	.047	.069	.025

$$t_4 = 3.44^*$$

$$t_4 = \frac{\bar{d} - 0}{S_{\bar{d}}}$$

$$F_4^4 = 3.72^{n.s.}$$

High Concentrations

	Hazardous Materials	Dept. of Food & Agriculture		
	<u>Lab Analysis</u>	<u>Lab Analysis</u>	<u>Σ</u>	<u>Diff.</u>
1	255	240	495	15
2	1030	500	1530	530
3	2640	1100	3740	1540
4	153	230	383	-77
5	509	500	1009	9

$$t_4 = 1.33^{n.s.}$$

$$t_4 = \frac{\bar{d} - 0}{S_{\bar{d}}}$$

$$F_4^4 = 8.35^*$$

\*5% level of significance

Appendix G : Test for Difference in Results Between Sampling Locations

Location	Sample Size	Mean	Standard Deviation	Variances <sup>a</sup>		Means <sup>b</sup>	
				$\chi^2_{\text{calc.}}$	$\chi^2_{\text{crit.}} (.05)$	$F_{\text{calc.}}$	$F_{\text{crit.}} (.05)$
1	3	0.0137	0.0031				
2	4	0.0123	0.0026				
3	3	0.0123	0.0006				
				3.46 <sup>n.s.</sup>	$\chi^2_2 = 5.99$	0.35 <sup>n.s.</sup>	$F^2_3 = 4.74$

<sup>a</sup>Bartlett's test for longevity of variances.

<sup>b</sup>Test for differences among means.

Appendix H: Test for Differences in Carbaryl Concentrations Over Time

		Location 1			Post Spray 1			
		3/26	3/28	3/30	4/01	4/03	4/05	4/07
		3.97	1.47	2.82	.62	1.56	1.27	0.52
		1.50	1.93	1.88	.72	0.56	0.69	0.40
		1.94	1.52	1.07	.87	0.81		0.46
$\bar{X}$		2.47	1.64	1.92	.74	.98	.98	.46

$F_{13}^6 = 3.39^*$

Test to determine if slope differed from 0:  $t_5 = \frac{-0.148}{0.719} = -0.206^{n.s.}$

Location 2 Post Spray 1

	4/4	4/6	4/8
	2.22	3.02	2.27
	3.39	3.17	3.82
	3.80	3.17	3.48
$\bar{X}$	3.11	3.10	3.19

$F_5^2 = 0.01^{n.s.}$

Location 2 Post Spray 3

	4/27	5/01	5/05	5/09	5/13	5/24	6/02	6/12	7/07
	4.72	4.34	5.29	3.55	3.80	10.24	7.47	9.85	6.03
	5.97	7.35	5.95	4.35	3.25	7.31	13.34	5.39	3.40
	9.45	9.01	7.99	3.67	3.10	3.49	9.04	9.03	3.98
									5.18
$\bar{X}$	6.71	6.90	6.41	3.82	3.38	7.01	9.95	8.09	4.650

$F_{19}^8 = 3.00^*$

Location 3 Post Spray 1

	4/10	4/12	4/14	4/16	4/18	4/20
	3.64	.93	.23	1.93	1.07	1.66
	3.46	1.49	1.09	1.02	.55	
	3.08	1.24	.67	.88	1.10	1.27
$\bar{X}$	3.30	1.22	.66	1.28	.91	1.47

$F_{11}^5 = 19.56^{**}$

Test to determine if slope differed from 0:  $t_4 = \frac{-0.142}{0.976} = -0.146^{n.s.}$

\* 5% level of significance

\*\* 1% level of significance

Appendix I. Test for differences in Carbaryl concentrations of degradation study<sup>1</sup>

	<u>DF</u>	<u>SS</u>	<u>MS</u>	
Factor	23	37874	1647	$F_{23}^{86} = 6.90^{**}$
Error	86	20513	239	
Total	109	58387		

\*\* Significant at the 1% level

Regression equation is  $Y = 69.8 - 0.886 x$   
 $R^2 = 51.8\%$        $t_{22} = \frac{-0.886}{19.1} = -0.046$  n.s.

1. Data from Table .