THE ENVIRONMENTAL MONITORING OF METHYL EUGENOL, NALED AND DICHLORVOS DURING A PEST TRAPPING AND ERADICATION PROGRAM

By

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October, 1989

Environmental Hazards Assessment Program

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EH 89-9

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Memorandum

To : John Sanders
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Date : October 4, 1989

Place : Sacramento

From : Department of Food and Agriculture — Bonnie Turner, Assoc. Environ. Haz. Scientist
Environmental Hazards Assessment Program

Subject : Executive Summary for the Report "The Environmental Monitoring of Methyl Eugenol, Naled and Dichlorvos During a Pest Trapping and Eradication Program

The California Department of Food and Agriculture (CDFA) began an oriental fruit fly (Dacus dorsalis) eradication program in Los Angeles County in October, 1988. The compounds used in this eradication program were methyl eugenol (Dorsalure ME) and naled (Dibrom 14 Concentrate). Methyl eugenol attracts male oriental fruit flies to bait stations set up during eradication programs and to traps placed in fruit trees for detection of new infestations. Naled, which degrades to dichlorvos (DDVP), is used to kill fruit flies attracted by methyl eugenol to bait stations and traps.

Because these compounds may have the potential to cause cancer and there have been no studies documenting methyl eugenol, naled or DDVP ambient air concentrations during eradication programs or near fruit fly trapping locations, CDFA began research to determine potential chemical exposure levels during such programs. In 1988, CDFA's Environmental Hazards Assessment Program (EHAP) conducted a study to determine the concentrations of methyl eugenol, naled and DDVP in ambient air and fruit during oriental fruit fly trapping and eradication programs. The study was divided into three phases.

Phase 1: The EHAP conducted a pilot study to determine the feasibility of monitoring methyl eugenol, naled and DDVP during an eradication program, and if feasible, to use these results in the design and implementation of future monitoring studies.

Phase 2: During the eradication program, the EHAP monitored ambient air concentrations of methyl eugenol, naled and DDVP in neighborhoods where oriental fruit fly eradication programs take place in Los Angeles County.

Phase 3: To detect new infestations in an area, traps are placed in fruit trees. The third phase of this study was conducted in a detection area in Sacramento County to determine if residues of methyl eugenol, naled and DDVP were present in edible portions of fruit from trees in which fruit fly traps had been placed.

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In the pilot study, air samples were collected at various time intervals and distances from treated bait stations on 0, 1 and 7 days after treatment. Methyl eugenol was unmeasurable except for one sample on day 1. Naled was not detected during any sampling, and DDVP was found on all sampling days. The pilot project results were used to design a monitoring program for an oriental fruit fly eradication project.

The EHAP monitored the oriental fruit fly eradication program during October, 1988 in Los Angeles County for ambient air concentrations of methyl eugenol, and methyl eugenol, naled and DDVP during the first and fourth applications, respectively. During the first application, methyl eugenol concentrations decreased to none detected by day 5; during the fourth application, methyl eugenol concentrations did not decrease significantly over time, but DDVP concentrations did. Naled was not detected.

Whole citrus fruit samples were collected from a detection area in September, 1988 in Sacramento County. Both methyl eugenol and DDVP were detected in several fruit from two of the four sites sampled.

A risk assessment by CDFA's Medical Toxicology Branch concluded that the potential health effects from methyl eugenol inhalation exposure are minimal since levels of exposure during an eradication program are much less than amounts currently allowed as food additives.

A literature survey was undertaken which showed methyl eugenol to be a naturally occurring compound in several fruit species inlcuding citrus fruit. However, since methyl eugenol and DDVP were found in citrus fruit samples collected from homeowners' trees, a more comprehensive monitoring program will be undertaken to test for the presence of these chemicals in common fruit tree species used in CDFA's trapping programs.

THE ENVIRONMENTAL MONITORING OF METHYL EUGENOL, NALED AND DICHLORVOS DURING A PEST TRAPPING AND ERADICATION PROGRAM

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OCTOBER 1989

ENVIRONMENTAL HAZARDS ASSESSMENT PROGRAM

ABSTRACT

The California Department of Food and Agriculture completed a three-phase study in 1988 to investigate the environmental fate of methyl eugenol, naled, and dichlorvos (DDVP), a degradation product of naled. The first phase of the study was planned to determine the feasibility of monitoring the three compounds during a routine pest eradication program. Once the feasibility of monitoring the three compounds was shown, the second-phase was planned to measure ambient air concentrations of the compounds during a fruit fly eradication. The third-phase was planned to determine if residues of methyl eugenol, naled, and DDVP were present in edible portions of fruit growing near fruit fly traps.

The first-phase was performed at a Plant Industry facility in Folsom, California. Air samplers were placed 1 m and 25 m from a treated bait station. Air samples were collected for 4, 8, and 24 hours on day 0, 1, and 7 after the bait station was treated. Methyl eugenol was unmeasurable after day 0 except for the 24 hour sample collected 1 m from the bait station on day 1. Naled was not detected. DDVP was found on all sampling days and decreased to less than 1 ng m^{-3} by day 7. Results from the first-phase were used to design the second-phase.

The second-phase was performed during the oriental fruit fly eradication program in Hacienda Heights (Los Angeles County) in the fall of 1988. Four-hour ambient air samples were collected during the first and fourth applications of bait, at four and six sites, respectively. During application one, methyl eugenol decreased significantly over days 0, 1, and 5 ($p < 0.001$) and ranged from 323 to 1050 ng m^{-3} on day 0 to none detected on day 5. During application four, there was no significant decrease in methyl eugenol concentration over days 0 through 4 but the decrease in DDVP concentration at the same sites was significant ($p < 0.001$).

The third-phase was performed in Sacramento County in September 1988. Whole citrus fruit samples were collected from four sites. Methyl eugenol was found in two samples from site 1 and one sample from site 2. DDVP was found in two samples from site 1. A literature search was undertaken which showed methyl eugenol to be a naturally occurring compound in some citrus fruits. Additional monitoring will take place in 1989 to test additional fruit tree species for the presence of both DDVP and methyl eugenol.

ACKNOWLEDGEMENTS

The authors would like to thank Plant Industry Folsom personnel for their assistance in providing a study site and storage area for our sampling equipment. We would also like to thank the Van Nuys Pest Detection/Emergency Projects crew for their help in monitoring the eradication program in Hacienda Heights. Finally, thanks go to the EHAP field group, with Karen Wiese as field coordinator, for their tireless efforts as usual, during this project.

DISCLAIMER

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

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INTRODUCTION

Objectives

In 1988, the California Department of Food and Agriculture (CDFA) began research to determine concentrations of methyl eugenol, naled and dichlorvos (DDVP), a degradation product of naled, in ambient air and fruit during oriental fruit fly trapping and eradication programs. The study was conducted by CDFA's Environmental Hazards Assessment Program (EHAP) to determine potential chemical exposure levels during such programs. The study was divided into three phases:

1. Ambient Air Monitoring Pilot Project

   Objective: To determine feasibility of monitoring methyl eugenol, naled and DDVP during an eradication program, and if feasible, to use these results in the design and implementation of such monitoring studies.

2. Ambient Air Monitoring during an Eradication Program

   Objective: To determine ambient air levels of methyl eugenol, naled and DDVP, if measurable, in neighborhoods where oriental fruit fly eradication programs take place.

3. Fruit Testing for Residues

   Objective: To determine if residues of methyl eugenol, naled and DDVP are present in fruit samples from trees in which fruit fly traps have been deployed.

Background

Methyl eugenol -- Methyl eugenol, also known as eugenyl methyl ether ($C_{11}H_{14}O_2$), is a food additive (flavoring ingredient) in non-alcoholic beverages, ice cream, jellies, and baked goods, and is used in soaps and perfumes. It is a naturally occurring compound in several fruit species including citrus. Methyl eugenol is chemically similar to safrole, a known hepatocarcinogen and is currently in the preliminary stage of evaluation for oncogenicity by the National Toxicology Program (Nelson, 1989). Under the trade name of Dorsalure ME™ (International Pheromones Inc., Hackensack, New Jersey), the compound has been used successfully for over 20 years by CDFA's Pest Detection/Emergency Projects Program. Methyl eugenol attracts male oriental fruit flies (Dacus dorsalis) to bait stations set up during eradication programs and to traps placed in fruit trees for detection of new infestations.

To date, there have been no studies documenting methyl eugenol ambient air concentrations during eradication programs or near fruit fly trapping locations. An environmental fate assessment to determine residue levels in soil, tomato leaves and water conducted in 1980 showed methyl eugenol half-lives of between 6 and 34 hr in soil and water (Shaver and Bull, 1980). Internal residues remained constant at 4 ppm for 14 days after topical application of methyl eugenol (1 mg) to tomato leaves. External residues on leaf surfaces were volatized completely by day 3 of the study.

Naled and DDVP -- Naled, employed by CDFA under the trade name, Dibrom® 14 Concentrate (Chevron Chemical Co., San Francisco, CA), is an organophosphate pesticide ($C_4H_7Br_2Cl_2O_4P$) used for insecticidal fumigation in greenhouses, animal kennels, and food-processing plants (Royal Society of Chemistry, 1988) as well as for killing fruit flies attracted to bait stations and traps by methyl eugenol. Naled, lethal to flies via direct contact, degrades to dichlorvos (DDVP) through cleavage of its bromine atoms. Of the two chemicals, DDVP is more volatile and acts as an insecticidal vapor within the traps and near bait stations. In January 1989, DDVP was added to the State of California Safe Drinking Water and Toxic Enforcement Act (Prop 65) list of chemicals known to cause cancer.

Numerous studies have measured DDVP concentrations in the air of homes, food-processing plants, dairy barns, and other enclosed structures (Gillett et al. 1972, Gold et al., 1984, Harein et al. 1970, Elgar and Steer 1972, Girish 1969, and Leary et al., 1974). Indoor concentrations of DDVP ranged from 24 $\mu g\ m^{-3}$ to 550 $\mu g\ m^{-3}$ for up to 2 h after treatment, depending upon application amount, temperature, volume of air treated, and containment time. DDVP has a Threshold Limit Value (TLV) of 1 $mg\ m^{-3}$. TLV's are time-weighted concentrations to which a person may be exposed for 8 h day^{-1}, 40 h wk^{-1}, with no adverse effects (American Conference of Governmental Industrial Hygienists, 1987).

Vegetation studies have shown that when applied to turf as an insecticidal agent, DDVP residues were below the California safe reentry concentration of 0.06 μg cm^{-2} approximately 2 h after application (Goh et al., 1986a). Safe reentry concentrations were reached after 6 h when DDVP was applied to clover and fescue (Goh et al., 1986b). Ambient air samples taken during the turf study measured DDVP concentrations of 1.9 ± 0.5 ppb immediately after application. When applied to a variety of crops in greenhouse experiments, DDVP half-life on plant sufaces ranged from 4.6 h on cotton to 6.8 h on beans and potatoes, and was 32.6 h for internal bean plant residues (Dedek et al., 1979).

MATERIALS AND METHODS

Site Description

Pilot Study -- In August 1988, EHAP initiated the methyl eugenol pilot study at the CDFA 4 ha (10 acre) Control and Eradication Storage Facility in Folsom, CA. The property contained several storage buildings and parking areas covering approximately 1.5 ha. The remaining site consisted of pasture grasses, weeds and widely spaced oak trees. Prevailing winds were from the south and southwest. Eighteen bait stations were placed in two concentric circles 50 m apart encircling most of the site (Figure 1). This was an attempt to simulate the ambient air concentrations of methyl eugenol, naled and DDVP expected to occur during an eradication program.

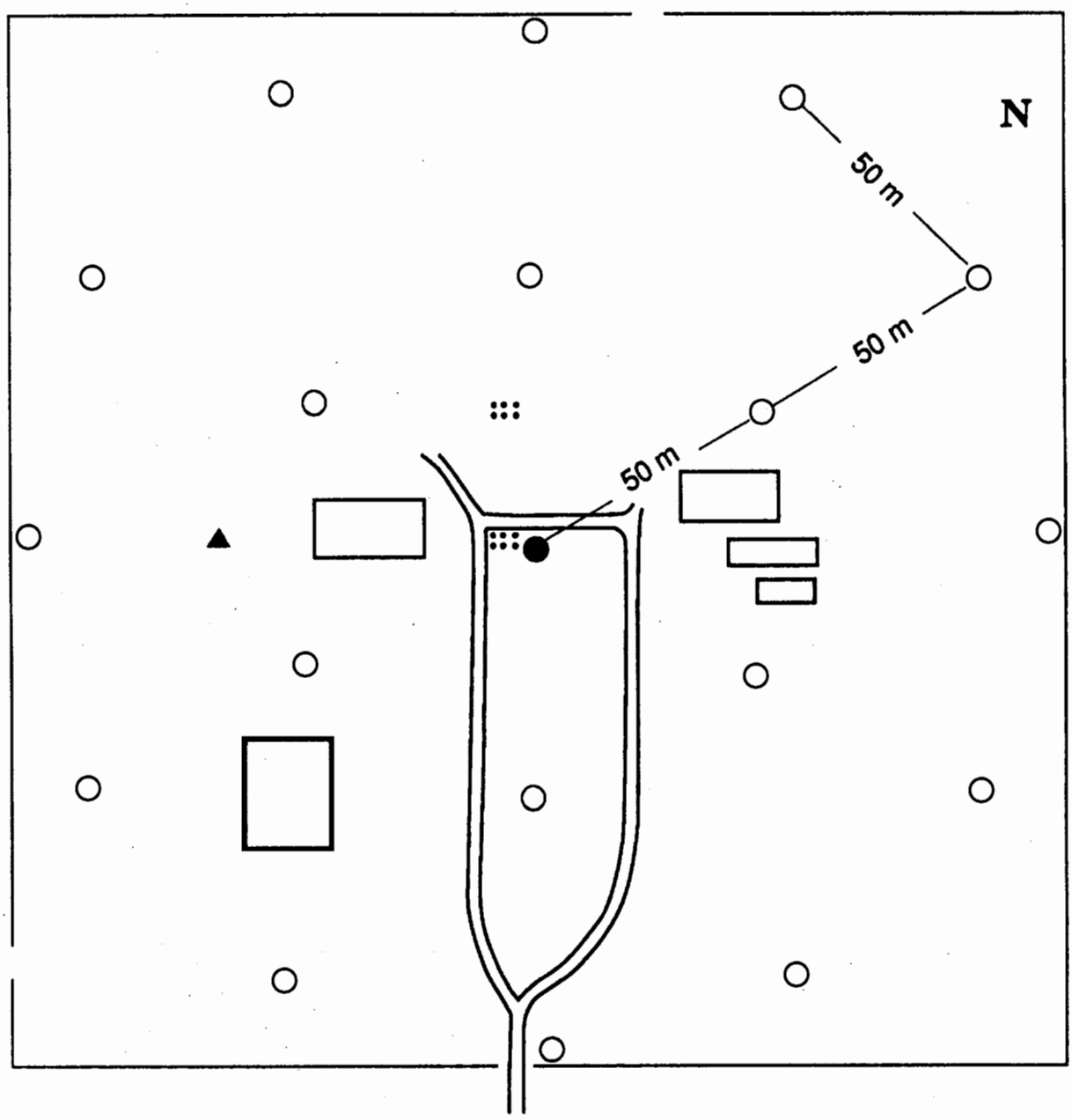

Figure 1. Sampling locations at Methyl Eugenol Pilot Study Plot, Folsom, CA, August 1988.

● Center Bait Station
○ Bait Station
▭ Building
• High Volume Air Sampler
▲ Weather Station

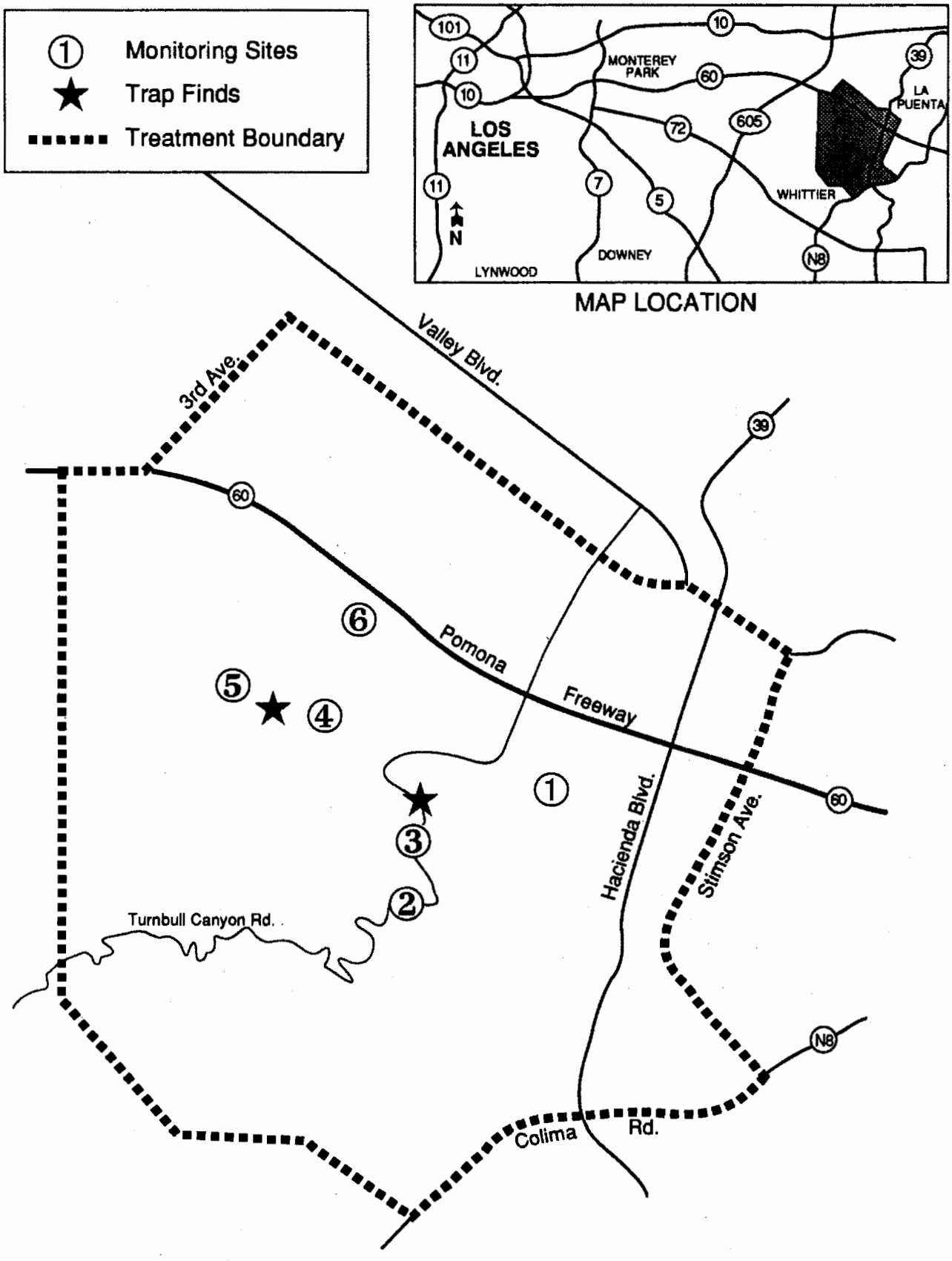

Figure 2. Air Monitoring Sites, Hacienda Heights, CA. Oriental Fruit Fly Eradication Program, Fall 1988.

Each bait station consisted of a 2.4 m (8 ft) upright 2x4 which held a 12-inch square plywood target. Six air samplers were placed within the inner circle 1 m from the center bait target and six samplers were placed 25 m away from the same target.

Eradication Program -- An oriental fruit fly eradication program began in Hacienda Heights, CA (Los Angeles County) on October 13, 1988 (Figure 2). The 14.5 km^2 (9 mi^2) treatment area contained multi-acre rural homes and moderately dense single-family developments. Topographically, the area contained several low elevation hills surrounded by valley floor. Using trees and telephone poles as bait targets, pesticide applications took place every two weeks for eight weeks. Four sites in residential areas within 1.6 km (1 mile) of the original fruit fly infestation were monitored during the first pesticide application. Two additional sites were added to the monitoring program for the final application. Whenever feasible, air samplers were situated within 5 m (16.25 ft) of treated bait stations, on city or county rights-of-way. Samplers were manned for the entire 4-hour sampling period.

Fruit Sampling -- The northern California CDFA trapping program requires one baited trap per square mile in urban areas during the months of May through November. In September 1988, whole fruit samples were collected from two lemon, one grapefruit and one orange tree at four private

residences in Sacramento, CA. Baited traps had been placed in the selected trees a minimum of 4 weeks prior to sampling.

Pesticide Application

Bait stations -- Naled (Dibrom® 14 Concentrate) was mixed with methyl eugenol (Dorsalure ME) to make a 14% solution which was then combined with Minu-Gel, an inert thickening agent. This formulation was used for both the pilot project and eradication program. Application to each target was made by a pressurized gun attached to the tank mixture transported by pickup truck.

Fruit Fly Traps -- Jackson traps are used to trap oriental fruit flies. They are made of plastic-coated cardboard and contain a cotton roll wick supported inside the trap by a wire wick holder. A sticky insert on the trap bottom captures flies. In order to activate the traps, Dibrom® 8 Concentrate was added to methyl eugenol (Dorsalure ME) to provide a 1% solution (active ingredient) used to stun the flies. Approximately 5 g was applied by eyedropper to the wick which was then inserted in each trap. When traps were re-baited at two to three week intervals, approximately 2 g were re-applied to the wick.

Sampling Methods

Pilot Project -- Kurz 310 high volume air samplers fitted with glass jars containing 125 ml XAD resin were used to sample ambient air 1 m and 25 m from the simulated central bait station at a flow rate of 1000 l min^{-1}. Separate samples were collected for methyl eugenol, naled and DDVP analysis on -1, 0, 1, 2, and 7 days after application. To determine the most efficient sampling period, samples were collected simultaneously for 4, 8 and 24 hours beginning immediately after application of bait to all stations. Four-hour samples were collected both in the morning and afternoon to compare temporal variation.

Eradication Program -- Air samples were collected for 4 hours with Kurz 310 high volume samplers located within 5 m of bait stations. Samples were taken on -1, 0, 1 and 5 days after application 1 and on 0, 1, 2, 3, and 4 days after application 4. Due to the closeness of scheduling during the emergency eradication program, only methyl eugenol was monitored during application 1. Methyl eugenol, naled and DDVP were monitored during application 4.

The jars containing resin were sealed in plastic and stored on dry ice after sampling was completed. A chain-of-custody record was compiled for each sample.

Ambient air temperatures were collected with minimum/maximum thermometers at each sampling location for all sampling periods.

Fruit Sampling -- Two lemon trees, one grapefruit and one orange tree were sampled. Whole fruits were collected 1 m and 3 to 5 m away from a trap which had been placed in the fruit tree at least 4 weeks prior to sampling. After sampling took place, it was learned that 2 sites had received re-baited traps the morning before sampling took place. The fruit was collected using a fruitpicker which had curved prongs and a basket to grab and hold each piece. Both methyl eugenol and naled analysis required 500 g for each sample. DDVP was analyzed from the same sample as naled. Samples were placed in plastic bags, kept on ice, and delivered immediately to the laboratory for extraction preparation. A chain-of-custody record was compiled for each sample.

Chemical Analysis

CDFA's Chemistry Laboratory Services, Sacramento, California, developed methods and conducted chemical analysis for methyl eugenol, naled and DDVP in resin and fruit. Extraction procedures and operating conditions for the gas chromatography (GC) method are included in Appendix I.

Methyl eugenol was quantified in resin and whole fruit by extracting with hexane and analyzed by gas chromatography using a flame ionization detector

(FID). Positive fruit samples were confirmed using 2 different columns, HP-17 and Carbowax 60M, and a high resolution gas chromatograph mass spectrometer (GC/MS).

Naled and DDVP were quantified in resin and fruit by extracting with toluene and analyzed by GC using an FID.

Quality Control Procedures

A blank matrix and blank matrix spike were analyzed with each extraction set for continuous quality control during analysis (Appendix II, Tables II-7 through II-9).

Statistical Analysis

Eradication Program -- Statistical comparison between days after each application were made using Friedman's two-way analysis of variance by ranks (Siegel, 1956). The nonparametric test was chosen because of the strong heterogeneity of variance in methyl eugenol after the first application, and the large numbers of non-detects for both methyl eugenol and DDVP after the fourth application. Only three out of the four sites (site 1, 2 and 4) were used in statistical analysis of the first application because the sampler at site 3 was placed more than 5 m away from the bait station and was not comparable to the remaining sites.

RESULTS AND DISCUSSION

Pilot Study

All sampling durations were successful in capturing methyl eugenol and DDVP. No naled was detected. The highest concentrations of methyl eugenol and DDVP were found in 4-hour samples collected on the morning of application at both the 1 m and 25 m distance from the centrally located bait station (Table 1). On 1, 2 and 7 days after application, methyl eugenol was unmeasurable except for a 24-hour sample taken at 1 m on day 1. DDVP, on the other hand, was found on all sampling days, decreasing to less than 1 ng m^{-3} by day 7.

Eradication Program

Based upon the results obtained during the pilot project, a sampling period of 4 hours was chosen for monitoring methyl eugenol during the first application and methyl eugenol plus naled and DDVP during the fourth application of the Hacienda Heights eradication program. The program began in October, 1988. Three of the four sites monitored for methyl eugenol during the first application showed concentrations ranging from 323 to 1050 ng m^{-3} on day 0 (Table 2). Methyl eugenol was below measurable detection by day 5. For the three sites at which methyl eugenol was detected, statistical analysis showed a significant decrease in methyl eugenol over days 0, 1 and 5 ($p < 0.001$, see Appendix III, Table III-1).

Table 1. Methyl eugenol and DDVP concentrations in air during pilot study, fall 1988, Folsom CA.

Days after Application	Distance from Target (m)	Sampling duration, hr: 4(am)4(pm) 8 24 — Methyl Eugenol ($ng\ m^{-3}$)				Sampling duration, hr: 4(am)4(pm) 8 24 — DDVP ($ng\ m^{-3}$)			
-1	<1	ND[1]	---[2]	---	---	ND	---	---	---
0	<1	553[3]	80	160	29	29	11	14	5
1	<1	ND	ND	ND	5	9	12	9	2
2	<1	ND	ND	ND	ND	2	3	3	2
7	<1	ND	ND	ND	ND	5	2	2	<1
-1	25	ND	--	--	--	ND	--	--	--
0	25	70	ND	40	15	11	7	9	4
1	25	ND	ND	ND	ND	11	16	9	3
2	25	ND	ND	ND	ND	2	3	2	2
7	25	ND	ND	ND	ND	1	<1	1	1

[1]ND = Not detected. Minimum detection levels were 5.0 µg and 0.1 µg per sample for methyl eugenol and DDVP, respectively. Air sampling flow rate was 1000 L min^{-1}.

[2]No sample collected.

[3]These results may be converted to ppt (vol/vol, STP) by multiplying by the following conversion factors: for methyl eugenol, use 0.137; for DDVP, use 0.111.

Table 2. Methyl eugenol concentrations found in air during first application of oriental fruit fly eradication program, fall 1988.

Days Post Application	Site 1	Site 2	Site 3[1]	Site 4
	----------(ng m^{-3})----------			
0	1050[2]	525	ND[3]	323
1	255	233	ND	233
5	ND	ND	ND	ND

[1]Site not used in statistical analysis.

[2]These results may be converted to ppt (vol/vol, STP) by multiplying by a conversion factor of 0.137.

[3]ND = Not detected. Minimum detection level = 5 μg_1 per sample = 21 ng m^{-3}. Air sampling flow rate was 1000 L min^{-1} for a 4-hr sampling period.

During the fourth application, methyl eugenol concentrations ranged from nondetectable to 544 ng m^{-3} and DDVP ranged from nondetectable to 30 ng m^{-3} (Table 3). As in the pilot project, no naled was detected during the monitoring period. After the fouth application, there was no significant change in the level of methyl eugenol over days 0, 1, 2, 3, and 4, but DDVP decreased significantly ($p < 0.001$) over the same period (Figure 3 and Appendix III, Table III-2).

There is no apparent explanation for the difference in methyl eugenol dissipation between applications 1 and 4 (Figure 4). The high variability in methyl eugenol concentration during the fourth application (Table 3) may have been due in part to application methods as well as microclimate variations at each site. DDVP results were similar to those found in our pilot project air samples.

Fruit Samples

Two of the four sites sampled in September had received freshly re-baited traps the morning that fruit samples were collected. Residues were found only in fruits which were collected from trees in which the fruit fly traps had been freshly re-baited. Results showed concentrations of both methyl eugenol and DDVP in fruit from site 1, and methyl eugenol in fruit from site 2 (Table 4). Mass spectrometry at the University of California, Davis was used to confirm the methyl eugenol positives from site 1.

Figure 3. Mean DDVP concentration in air for six sites, application 4, Hacienda Heights oriental fruit fly eradication program, fall 1988.

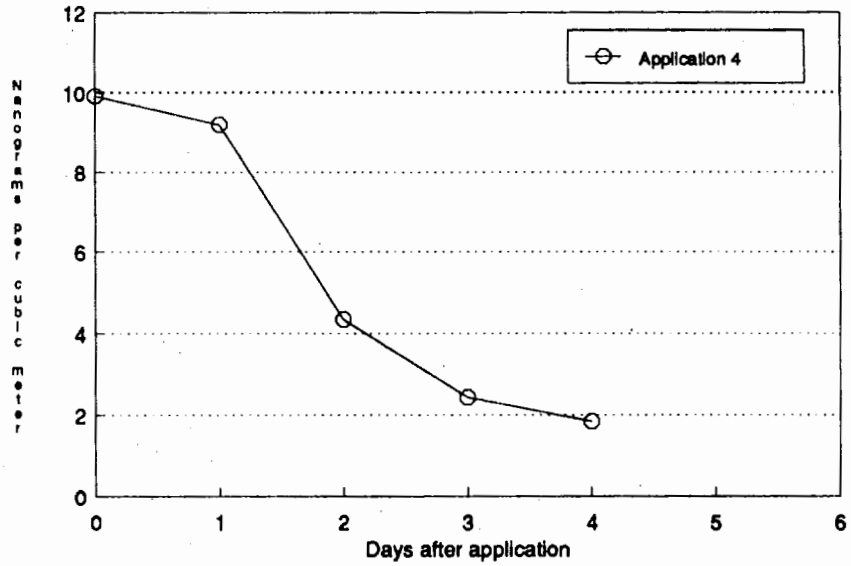

Figure 4. Mean methyl eugenol concentration in air for three sites, application 1, and six sites, application 4, Hacienda Heights oriental fruit fly eradication program, fall 1988.

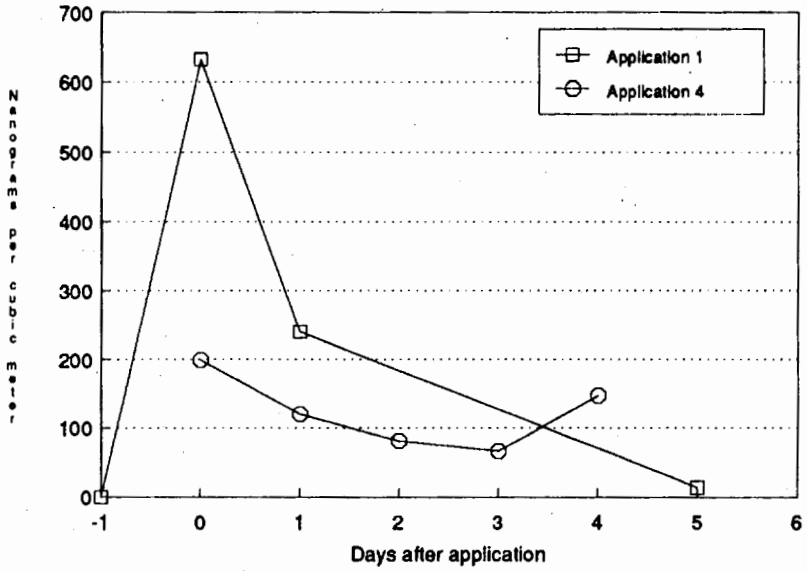

Table 3. Methyl eugenol and DDVP concentrations found in air during fourth application of oriental fruit fly eradication program, fall 1988.

Days Post Application	Site 1	Site 2	Site 3	Site 4	Site 5	Site 6
	Methyl eugenol ($ng\ m^{-3}$)					
0	ND[1]	161[2]	392	544	75	ND
1	66	ND	358	185	66	36
2	197	30	66	161	24	ND
3	131	119	21	179	ND	42
4	24	30	394	81	185	59
	DDVP ($ng\ m^{-3}$)					
0	7	3	11	30	3	5
1	6	6	11	14	10	7
2	5	2	7	8	5	ND
3	3	2	2	5	1	2
4	ND	ND	4	7	ND	ND

[1]ND = Not detected. Minimum detection levels were 5.0 µg and 0.05 µg per sample for methyl eugenol and DDVP, respectively. The air sampling flow rate was 1000 L min^{-1} for a 4-hr sampling period.

[2]These results may be converted to ppt (vol/vol, STP) by multiplying by the following conversion factors: for methyl eugenol, use 0.137; for DDVP, use 0.111.

Table 4. Methyl eugenol and DDVP residues in whole fruit sampled from citrus trees which contained baited fruit fly traps, September 1988.

Site	Distance from Trap (m)	Methyl eugenol (ppb)	DDVP (ppb)
1	1	210	1.20
	4	200	0.73
2	1	ND[1]	ND
	4	70	ND
3	1	ND	ND
	4	ND	ND
4	1	ND	ND
	4	ND	ND

[1]ND = Not detected. Minimum detection limit was 50 ppb and 0.5 ppb for methyl eugenol and DDVP, respectively.

CONCLUSIONS AND RECOMMENDATIONS

The pilot project and eradication program monitoring provided new information about methyl eugenol and DDVP ambient air concentrations during oriental fruit fly eradication programs.

- Methyl eugenol and DDVP were detected in ambient air using XAD-4 resin as the trapping medium and high volume air sampling equipment calibrated at a flow rate of 1000 L min^{-1}.

- Methyl eugenol concentrations ranged from none detected to 1050 ng m^{-3} during monitoring. During the first application, methyl eugenol concentrations decreased to none detected by day 5; during the fourth application, methyl eugenol concentrations did not decrease significantly over a 5-day monitoring period. The variability found in methyl eugenol concentrations during the fourth application is believed to be due to microclimate variation within each site and variable bait applications.

- DDVP concentrations ranged from none detected to 30 ng m^{-3} during the fourth application and declined significantly over days 0 through 4 ($p < 0.001$).

- Naled was not detected during the monitoring program.

A risk assessment by CDFA's Medical Toxicology Branch concluded that the potential health effects from methyl eugenol inhalation exposure are minimal since levels of exposure during an eradication program are much lower than amounts currently allowed as food additives (Nelson, 1989). Regarding DDVP exposure, the published TLV of 1 mg m^{-3} (American Conference of Governmental Industrial Hygienists, 1987) is more than 4 orders of magnitude greater than the highest concentration found in our air samples (30 ng m^{-3}).

A literature survey undertaken to determine the natural occurrence of methyl eugenol in various fruit species showed that methyl eugenol has been found to occur in a number of fruit species including citrus fruit. Since methyl eugenol and DDVP were found in citrus fruit samples collected from homeowners' trees, a more comprehensive monitoring program will be undertaken next year to test for the presence of these compounds in other common fruit tree species used in CDFA's trapping programs.

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APPENDIX I:

ANALYTICAL METHODS FOR METHYL EUGENOL, NALED AND DDVP

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CHEMISTRY LABORATORY SERVICES
ENVIRONMENTAL MONITORING SECTION
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Original Date:??
Supercedes: NEW
Current Date:6/8/1989
Method #:

METHYL EUGENOL

SCOPE:

This method is used to determine Methyl Eugenol in air samples.

PRINCIPLE:

XAD-2 resin is extracted with toluene.The extract is concentrated by a rotary evaporator.The residue is brought to a final volume with hexane and analyzed by GC using a flame ionazation detector.

REAGENTS AND EQUIPMENT:

Hexane,pesticide quality or equivalent
Toluene ,pesticide quality or equivalent
Sodium sulfate,ACS,granular,anhydrous
Stock standard
Working standard,prepared from stock standard
Filter funnels
Columns:19x300mm
Brown bottle:500ml
Round bottom flask:500ml
Filter paper#4
Glass wool
Rotary evaporator
Shaker
Silica sep-pak
Chloroform , pesticide quality or equivalent
Graduated test tube
GC Varian 3700 with FID

ANALYSIS:

Transfer XAD-2 resin from a hi-vol jar to a brown bottle , wash the jar with 50ml toluene .

Add 150ml toluene to the brown bottle , and shake it for 3 hours.

Transfer the resin and extract to a 19x300mm column . Filter the extract through sodium sulfate into a 500 ml round bottom flask. Wash the column two times with 100 mls toluene at a flow rate 3 ml/m.

Concentrate the extract to almost dryness by high pressure vacumn set at 65 (equivalent to 65°C.)

Transfer the residue to a graduated test tube with hexane . Reduce to a final volume of 2ml on a water bath under a stream of nitrogen.

Analyze by gas chromatography using FID.

SILICA SEP-PAK CLEAN UP (If necessary)

1.Take 1ml out of 2ml of the final volume and transfer it to a 10ml syringe which is connected to a 4ml hexane washed silica sep-pak .
2.Add 8ml of the mixture chloroform : hexane (75:25) to the 10ml syringe.
3.Force the mixture through the sep-pak at a constant flow rate(3ml/min) by pressing the plunger consistently .
4.Discard the first two milliliter of the mixture .

5.Collect the last six ml of the mixture into a graduated test tube and concentrate to a final volume of 2 mls . Mix the test tube for 10 seconds by using a vorter mixer .
6.Analyze by gas chromatography .

EQUIPMENT CONDITIONS:

Gas chromatograph : Varian 3700
- -Initial temp : 100°C
- -Initial time : 5 min
- -Prog. rate : 10°C / min
- -Final time : 0.0 min
- -Final temp : 190°C
- -Injector temp: 210°C
- -Detector temp: 260°C
- -Hydrogen flow rate : 30ml/min
- -Air flow rate : 350ml/min
- -Helium flow rate : 30ml/min

Detector type : Flame ionization detector
Column :
DB WAX 15m
Retention time : 8.5 min

CALCULATIONS:

Calculated based on peak height of the sample compared to standard
Results are reported as milligram/ sample

DISCUSSION:

Spike level : 103.46ug/125mls XAD-2 resin (without using silica clean up)

	% recovery
Spike 1	83.7
Spike 2	72.0
Spike 3	76.8
Spike 4	78.8
Spike 5	92.7
Spike 6	76.0
Spike 7	77.6

Average % recovery : 79.6
Standard deviation : 6.7

Spike 103.46 ug/125 ml XAD-2 resin (using silica sep-pak clean up)

	% recovery
Spike 1	58.2
Spike 2	61.9
Spike 3	61.9
Spike 4	63.0
spike 5	65.5

Average % recovery : 62
Standard deviation :2.6
Because methyl eugenol evaporates quickly , sample should be analyzed as soon as possible .

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Original Date:??
Supercedes: NEW
Current Date:9/14/1988
Method #:

METHYL EUGENOL ON VEGETATION

SCOPE:

This method is used to determine Methyl Eugenol on vegetation samples.

PRINCIPLE:

The vegetation is blended with dry ice, then extracted with hexaxe . The extract is concentrated to a final volume in hexane and transfered to a silica sep-pak .Elute the analyte with a mixture of chloroform and hexane.The Methyl Eugenol is analyzed by GC using a FID detector .

REAGENTS AND EQUIPMENT

Hexane , chloroform ,pesticide quality or equivalent .
Sodium sulfate , anhydrous.
Graduated test tube.
Silica sep-pak .
Working standard,prepared from stock standard
10 ml syringe.
Dry ice
Mason jars: 2 quart, 1 pint sizes
Boiling flasks: 500 ml, 250 ml
Separatory funnels: 500 ml.
Filter funnels: 90 mm
Aluminum foil
Whatman filter paper: #1, 18.5 cm
G10 gyratory shaker
Cuisinart food processor
Buchi rotovapor
Meyer N-evap analytical evaporator
GC Varian 3700 with FID

ANALYSIS :

1. Blend the entire sample with dry ice in a cuisinart, then store in a freezer to allow the carbon dioxide to evaporate. (Do not cap the container too tight.)
2. Weigh 50g of the sample into a pint size jar. Add 75ml hexane to the jar and shake for 20 minutes .
3. Filter the sample through #1 filter paper into a 500 ml separatory funnel. Rinse the jar and the filter paper several times with 70ml hexane .
4. If the water is present at the bottom of the seperatory funnel , drain the water layer into a beaker and discard . Taking care not to lose any of the solvent .
5. Drain the solvent layer through sodium sulfate into a 500 ml boiling flask.
6. Rinse the sodium sulfate well with 70ml hexane.
7. Evaporate the contents of the flask to near dryness by using a rotary evaporator .
8.Transfer the extract to a graduated test tube with 10 ml of hexane .

Evaporate the final volume to 2ml by using a nitrogen evaporator.
SILICA SEP-PAK CLEAN UP:
1.Take 1ml out of 2ml of the final volume and transfer it to a 10 ml syringe which was connected to a 4ml hexane washed silica sep-pak .
2.Add 7.0 ml of the mixture chloroform : hexane (75:25) to the 10 ml syringe.
3.Force the mixture through the sep-pak at a constant flow rate (3ml/min) by pressing the plunger consistently .
4.Discard the first two milliliters of the mixture .
5.Collect the last five milliliters of the mixture into a graduated test tube and concentrate to the final volume of 2mls.Mix the test tube for 10 seconds by using a vortex mixer.
6.Analyze by gas chromatography .

DESORPTION COEFFICIENT:

EQUIPMENT CONDITIONS:

Gas chromatograph : Varian 3700
- -Initial temp : 100°C
- -Initial time : 5 min
- -Prog. rate : 10°C/ min
- -Final time : 0.0 min
- -Final temp : 190°C
- -Injector temp: 210°C
- -Detector temp:260°C
- -Hydrogen flow rate : 30ml/min
- -Air flow rate : 350ml/min
- -Helium flow rate : 30ml/min

Detector type : Flame ionization detector
Column : DB WAX 15m
Retention time : 8.5 min

CALCULATIONS:

Results are reported as PPM

DISCUSSION:

Spike level :258.65ug/50g grapefruit

	% recovery
Spike 1	74.0
Spike 2	68.2
Spike 3	82.6
Spike 4	77.4
spike 5	61.4

Because methyl eugenol evaporates quickly, sample should be extracted and analyzed as soon as possible .

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Original Date:??
Supercedes: NEW
Current Date:2/4/88
Method #:

DDVP/NALED

SCOPE:
This method is for the determination of DDVP and NALED from resin tubes and from vegetation.

PRINCIPLE:
DDVP and NALED have been collected from the air into resin tubes, and a mixture of hexane:acetone(50:50) is used to extract DDVP and NALED from the tubes. Vegetation samples have also been collected, and hexane is used to extract DDVP and NALED from them. The concentration of DDVP and NALED is determined by GC using a TSD detector.

REAGENTS AND EQUIPMENT:
Hexane/Acetone, 1:1 mixture
Hexane
Methanol
Sodium sulfate, anhydrous
Dry ice
XAD-2 resin
Clean vegetation
1 pint Mason jars
2 quart Mason jars
500 ml brown bottles
90 mm stemmed filter funnels
500 ml separatory funnels
250 ml separatory funnels
500 ml boiling flasks
15 ml conical test tubes
Aluminum foil
Whatman filter paper, #1, 18.5 cm
Cuisinart food processor
Sonicator
Gyratory shaker
Rotary evaporator
Nitrogen evaporator
Vortex mixer
Cutting board and knife
GC (Varian 3700 TSD, Autosampler)
Stock standard
Working standard

ANALYSIS:
(I) Air Samples

1) Transfer the glass wool and/or resin from the sample hi-vol resin jar to a 500 ml brown bottle with 1:1 hexane/acetone, rinsing the resin jar. Add enough 1:1 hexane/acetone to the brown bottle to reach a final solvent

volume of 150 ml. Seal the bottle with aluminum foil and a lid.
2) Sonicate the sample for 15 minutes.
3) Shake the sample on the gyratory shaker for 1 hour at 220 rpm.
4) Pour the solvent through a 90 mm funnel containing filter paper and 70 grams of sodium sulfate. Collect the sample in a 500 ml boiling flask.
5) Add 100 ml of 1:1 hexane/acetone to the brown bottle and shake for 1 hour at 220 rpm.
6) Transfer the entire contents of the brown bottle (solvent and resin) to the filter funnel and combine the extracts in the 500 ml boiling flask. Rinse the funnel contents well with about 20ml of 1:1 hexane/acetone mixture.
7) Evaporate the contents of the boiling flask to near dryness on the rotary evaporator with the setting on 40.
8) Transfer the contents of the boiling flask to a 15 ml conical test tube with 10 ml of methanol.
9) Concentrate the sample to 4 ml on the nitrogen evaporator. Mix the contents of the tube for 10 seconds with the vortex mixer.
10)Analyze by gas chromatography.

(II) Vegetation Samples

1) Weigh the sample and cut it into small pieces with a knife.
2) Blend the sample in a Cuisinart blender with dry ice.
3) Transfer to a 2 quart Mason jar, cover lightly with aluminum foil and a lid, and place in a freezer overnight to allow the dry ice to sublime.
4) While still frozen, stir the sample and weigh out a 100 g aliquot into a 1 pint Mason jar. Add 150 ml of hexane to the jar and shake on a gyratory shaker for 30 minutes at 220 rpm.
5) Filter the sample through #1 filter paper into a 500 ml seperatory funnel. Rinse the jar and the filter paper several times with a total of 70ml of hexane.
6) If the water is present in the 500ml separatory funnel, drain the bottom water into a beaker and discard .
7) Drain the solvent layer through sodium sulfate into a 500 ml boiling flask.
8) Rinse the sodium sulfate well with 50 ml of hexane .
9) Rotoevaporate the contents of the boiling flask to near dryness.
10)Transfer the sample to a 15 ml test tube with 10 ml of methanol.
11)Reduce the volume to 4 ml on the nitrogen evaporator.
12)Mix the sample on the vortex mixer.
13)Analyze by gas chromatography.

DESORPTION COEFFICIENT:

EQUIPMENT CONDITIONS:
GC condition :

Column :Methyl Silicone .53 mm x 10M .				
Carrier gas : Helium 7psi .				
Detector : TSD .				
Bead : 5.6 , Hydrogen : 29psi .				
Tem. Progam :	Initial	:	100	5min.
	Rate	:	20	1min.
	Final	:	170	4min.
Retention time		:	DDVP approx. 4min.	
			Naled approx. 10min.	

DDVP/NALED page 3

CALCULATIONS:

$$\text{NG/CUBIC METER} = \frac{(A)(\text{ng standard})(1000)(\text{final volume mls})}{(B)(\text{total cubic meter of air})(\text{ul sample injected})}$$

A -area sample or peak heigh sample
B -area standard or peak heigh standard

$$\text{PPB} = \frac{\text{Final volume (ul)} \times \text{Amount of STD (ng)} \times \text{Peak height of sample}}{\text{Volume of sample injected(ul)} \times \text{Sample weight(g)} \times \text{Peak height of STD}}$$

DISCUSSION:
Recovery : Since Naled could be converted quickly to DDVP ,the percent recovery of Naled is proportional to the time .To determine the concentration of Naled , the sample should be analyzed within 24 hours with fresh working standards .

REFERENCES:

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APPENDIX II:

METHOD DEVELOPMENT AND QUALITY CONTROL RESULTS

METHOD DEVELOPMENT

Method validation for analysis of methyl eugenol, naled and DDVP in resin and fruit was performed by CDFA Laboratory Services in Sacramento, CA. Methyl eugenol resin and fruit spikes were prepared at levels of 103 ug and 1 ppm, respectively (Tables II-1 through II-2).

The detection limits, mean percent recoveries and SD for methyl eugenol in resin and fruit were 5 ug, 77 percent and 6.5; 0.05 ppm, 68 percent and 6.9, respectively (Tables II-1 through Tables II-2).

The detection limits, mean percent recoveries and SD for naled and DDVP in resin and fruit were 0.2 ug, 68 percent and 7.5; 0.1 ug, 89 percent and 5.1; and 1 ppb, 73 percent and 11; 1 ppb, 66 percent and 4.4, respectively (Tables II-3 through II-6).

The mean percent recovery and SD were used to calculate the warning (mean +/- SD) and control (mean +/- 2 SD) limits for accuracy.

QUALITY CONTROL RESULTS

CDFA laboratory continuing quality control spikes of methyl eugenol in resin and fruit, and DDVP in fruit showed average recovery percentages and standard deviations of 77% and 8.4, 74% and 7.6, 85% and 0, respectively (Appendix II, Tables II-7 through II-9).

One out of nine continuing quality control spike recoveries fell outside the upper control limit set for methyl eugenol in resin at 90% (Table II-7). No corrective action was taken.

Table II-1. Method Validation Blank Matrix Spikes for the Methyl Eugenol 1988 Pilot Project: XAD-2 Resin

Analyte: Methyl Eugenol
Matrix: XAD-2 Resin
Detection Limit: 5 ug/sample

Lab: CDFA
Chemist: Duc Tran
Date: 8/24/88

Lab Sample #	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
125	83.73	103.46	80			
126	71.97	103.46	70			
127	76.76	103.46	74			
128	78.76	103.46	76			
129	92.74	103.46	90			
130	75.96	103.46	73			
131	77.56	103.46	75	77	6.5	8.5

Table II-2. Method Validation Blank Matrix Spikes for the Methyl Eugenol 1988 Pilot Project: Fruit.

Analyte: Methyl Eugenol
Matrix: Fruit (Grapefruit)
Detection Limit: 0.05 ppm

Lab: CDFA
Chemist: Duc Tran
Date: 9/14/88

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
322	0.643	1	64.3			
323	0.679	1	67.9			
324	0.632	1	63.2			
325	0.857	1.073	79.9			
326	0.712	1.073	71.2			
327	0.608	1.073	60.8	67.9	6.93	10.2

Matrix	$\bar{X}$	SD	LWL	UWL	LCL	UCL
Resin *	77	6.5	71	84	64	90
Fruit *	68	6.9	61	75	54	82

* LWL and UWL = mean +/- 1 SD
** LCL and UCL = mean +/- 2 SD

Table II-3. Method Validation Blank Matrix Spikes for the Naled/DDVP Pilot Project: XAD-2 Resin.

Analyte: Naled
Matrix: XAD-2 Resin
Detection Limit: 0.2 ug/sample

Lab: CDFA
Chemist: Duc Tran
Date: 9/28/88

Lab Sample #	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
451	4.4	5.52	79.7			
450	3.90	5.52	70.7			
449	3.2	5.52	57.5			
448	3.25	5.52	58.9			
447	3.82	5.52	69.2			
446	3.75	5.52	67.9			
445	3.82	5.52	69.2	67.6	7.52	11.1

Table II-4. Method Validation Blank Matrix Spikes for the Naled/DDVP Pilot Project: XAD-2 Resin.

Analyte: DDVP
Matrix: XAD-2 Resin
Detection Limit: 0.1 ug/sample

Lab: CDFA
Chemist: Duc Tran
Date: 9/28/88

Lab Sample #	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
437	4.78	5.35	89.4			
438	4.38	5.35	81.9			
439	4.90	5.35	91.6			
440	4.73	5.35	88.4			
441	4.84	5.35	90.4			
442	5.12	5.35	95.7			
436	4.38	5.35	81.9	88.5	5.05	5.71

Matrix	$\bar{X}$	SD	LWL	UWL	LCL	UCL
Naled Resin *	68	7.5	61	76	53	83
DDVP Resin *	89	5.1	84	94	79	99

*LWL and UWL = mean +/- 1 SD, LCL and UCL = mean +/- 2 SD

Table II-5. Method Validation Blank Matrix Spikes for the Naled/DDVP Pilot Project: Fruit.

Analyte: Naled
Matrix: Fruit (Grapefruit)
Detection Limit: 1 ppb

Lab: CDFA
Chemist: Duc Tran
Date: 10/18/88

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
493	0.312	0.53	59			
494	0.452	0.53	85			
495	0.382	0.53	72			
496	0.301	0.53	57			
497	0.416	0.53	78			
498	0.428	0.53	81			
499	0.416	0.53	78	73	11	15

Table II-6. Method Validation Blank Matrix Spikes for the Naled/DDVP Pilot Project: Fruit.

Analyte: DDVP
Matrix: Fruit (Grapefruit)
Detection Limit: 1 ppb

Lab: CDFA
Chemist: Duc Tran
Date: 10/5/88

Lab Sample #	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
485	0.3749	0.5356	70.0			
486	0.3289	0.5356	61.4			
487	0.3139	0.5356	58.6			
488	0.3594	0.5356	67.1			
489	0.3594	0.5356	67.1			
490	0.3674	0.5356	68.6			
491	0.3749	0.5356	70.0	66.1	4.42	6.68

Matrix	$\bar{X}$	SD	LWL	UWL	LCL	UCL
Fruit						
Naled *	73	11	62	84	51	95
DDVP *	66	4.4	62	70	57	75

* LWL and UWL = mean +/- 1 SD, LCL and UCL = mean +/- 2 SD

Table II-7. Continuing Quality Control Data for the Methyl Eugenol 1988 Project: XAD-2 Resin

Analyte: Methyl Eugenol
Matrix: XAD-2 Resin
Detection Limit: 5ug/sample

Lab: CDFA
Chemist: Duc Tran
Date: 09/02/88

Exraction Set No.s	Lab Sample#	Results (ug)	Spike Level (ug)	Recovery %	$\bar{X}$	SD	CV (%)
333, 338, 347, 371	257	81.1	103.46	78.4			
374-81	304	74.8	103.46	72.3			
331-2, 334-5, 339-41, 344, 346, 349-53	290	81.8	103.46	79.1			
342-3, 357-8	274	75.97	103.46	73.4			
345, 348, 372-3	272	75.97	103.46	73.4			
382-6, 388-9, 392-4, 399	713	72	103.4	69.6			
391, 395-6, 398	745*	99.7	102.9	96.8			
162-3,166,189	1461	67.6	85.94	78.6			
191-3,397,409-14, 422, 425							
161-5, 190, 387 390-400, 415, 419, 421, 423-4	1519	59.17	85.94	68.9	76.7	8.43	11.0

* Sample fell outside the upper control limit set for methyl eugenol in resin at 90%

Table II-8. Continuing Quality Control Data for the Methyl Eugenol 1988 Project: Fruit

Analyte: Methyl Eugenol
Matrix: Fruit (Grapefruit)
Detection Limit: 0.05 ppm

Lab: CDFA
Chemist: Duc Tran
Date: 10/27/88

Extraction Set No.s	Lab Sample#	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
121-9	453	0.605	0.878	68.9			
195-6, 198-207	1229	4.116	5.173	79.6	74.3	7.57	10.2

Table II-9. Continuing Quality Control Data for the Methyl Eugenol 1988 Project: Fruit

Analyte: DDVP
Matrix: Fruit (Grapefruit)
Detection Limit: 1 ppb

Lab: CDFA
Chemist: Duc Tran
Date: 12/01/88

Extraction Set No.s	Lab Sample#	Results (ppm)	Spike Level (ppm)	Recovery %	$\bar{X}$	SD	CV (%)
195-6, 198-207	1229*	0.164	0.191	85.9			

* Sample fell outside the upper control limit set for DDVP in fruit at 75%.

APPENDIX III:

STATISTICAL ANALYSES TABLES

Table III-1. Analysis of variance on ranks of methyl eugenol concentration by sites for first application of oriental fruit fly eradication program, fall 1988.

Source	df	MS	F value
Days post application	2	3.0	99999*
Error	6	0	

*Significant at the 0.001 level.

Table III-2. Analysis of variance on ranks of methyl eugenol and DDVP concentrations by sites for fourth application of oriental fruit fly eradication program, fall 1988.

		Methyl Eugenol		DDVP	
Source	df	MS	F value	MS	F value
Days post application	2	1.417	0.65	11.854	24.53*
Error	6	2.173		0.483	

*Significant at the 0.001 level.