## MIREX MONITORING IN HAWAII - 1973-74

FINAL REPORT

## Ву

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September 1974



# University of Hawaii at Manoa

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Office of the Director

September 12, 1974

#### MEMORANDUM

- TO: Robert G. Kuykendall U.S. Environmental Protection Agency Region IX 100 California Street San Francisco, California 94111
- VIA: Melvin Koizumi, Director U.S. Environmental Protection Agency Pacific Islands Basin 1000 Bishop Street, Room 601 Honolulu, Hawaii 96813
- FROM: Jerry M. Johnson Assistant Director Univ. Hawaii Environmental Center
- RE: Mirex Monitoring in Hawaii 1973-74 Final Report

#### RATIONALE

The 1973-74 Mirex Monitoring Program - Hawaii was undertaken in accordance with a U.S. Environmental Protection Agency Determination and Order dated August 31, 1973 (Attachment 1). In that D.&O., EPA stayed its prohibition against aerial application of Mirex in Hawaiian pineapple fields provided that a Mirex monitoring program was carried out according to their specifications and approval.

Pursuant to the EPA D.&O. of August 31, 1973, a meeting of State and University of Hawaii officials was held to formulate a State monitoring strategy and a specific monitoring plan. Invited to the meeting were representatives of Robert G. Kuykendall

the Governor's Office of Environmental Quality Control, the State Departments of Agriculture and Health, the Division of Fish and Game of the State Department of Land and Natural Resources and the Water Resources Research Center, Department of Agricultural Biochemistry, the Department of Zoology and the Environmental Center of the University of Hawaii. From that meeting and subsequent meetings and supportive oral and written communications, the State's monitoring strategy and detailed plan were prepared.

Program responsibilities were set out and approved as follow:

1) The State Department of Health, Vector Control Branch, provided a biologist to collect, identify, wrap and refrigerate small mammal specimens and deliver them to a centralized collection site on Maui. Dr. Henri Minette was the Department's representative on the Monitoring Program Steering Committee.

2) The State Department of Land and Natural Resources, Division of Fish and Game provided a biologist to collect, identify, wrap and refrigerate bird specimens and deliver them to a centralized collection site on Maui. Mr. David Woodside was the Division's representative on the Steering Committee.

3) The Water Resources Research Center of the University of Hawaii provided a biologist responsible for taking soil samples and for collection, identification, wrapping and refrigeration of aquatic specimens. All samples collected by the WRRC biologist were delivered directly to the analytical laboratory at the University of Hawaii. Dr. Reginald Young represented WRRC on the Steering Committee.

4) The State Department of Agriculture was responsible for mammal and bird assembly, shipment, and delivery to the analytical laboratory. The Department also provided data on Mirex usage and field specifications in the subject areas and was our liaison with the pineapple industry. Dr. Alexander M. Dollar was the Department's representative.

5) The University of Hawaii Department of Agricultural Biochemistry prepared and analyzed the samples. Dr. John Hylin was the Department's representative during this year's study.

6) The U.H. Environmental Center assumed the overall coordination role and was responsible for report preparation and review.

On September 14, 1973, the Acting Director of the Environmental Center forwarded to EPA the State's proposed 1973-74 Monitoring Plan (Attachment 2). The objectives were:

1) to determine detectability of Mirex in marine fauna in estuarine sites receiving runoff water from areas of Mirex application in Hawaii.

2) to determine surface transport of Mirex from fields by water into water courses and areas where intermittent water flows may transport soil and Mirex treated particles.

3) to determine detectability of Mirex in terrestrial small mammals and birds within or adjacent to areas of Mirex application in Hawaii.

#### MONITORING PLAN

#### Sample Sites

The Maliko Watershed (Figure 1), Maui, Hawaii was selected as the sampling area for terrestrial biota and soil. The total gross acreage of this pineapple plantation is 4962.83. Target sampling areas within the total plantation were fields 233, 234 and 235 (Figure 1). Fields 233 and 234 were newly planted and consequently open fields within which we anticipated insect feeding bird populations would be highest. Field 235 is an older field in which the pineapple plants cover the entire ground surface. Field 235 was selected as the mammal trapping location at the suggestion of a Department of Health vector control biologist who suggested a closed mature field as the best collection area for mammals. The three fields are contiguous and all border on the Maliko Gulch (Figure 1). The total acreage is 98.80 acres for field 233, 127.02 for field 234 and 213.99 for field 235.

The soil of the field 233-235 sampling area is of the Haiku series of the Pauwela-Haiku Association.<sup>1</sup> These are deep, well-drained soils having gentle to moderate slopes. Annual rainfall is from 50 to 80 inches.

Haiku soils develop in material weathered from basic igneous rock. The surface layer is a dark-brown clay of about 14" in depth. The subsoil is either yellowish-red, dark-reddish brown or dark-red clay, and has a subangular to angular blacky structure. The depth of this layer is about 31". The substratum is soft, weathered basic igneous rock.

The surface layer is "very strongly acid" in the surface layer and "extremely acidic" and "very strongly acid" in the subsoil and substratum.

Permeability in these soils is moderately rapid. Runoff is slow in the area of fields 233 and 234 and the erosion hazard, according to the Soil Conservation Service, is slight.

Aquatic specimens were collected at the Maliko (Figure 1) and Honokohau (Figure 2) Bays located on Maui, Hawaii. These two bays were selected because they were the two sampling locations where the five positive specimens, out of a total of 113 samples, were found during the 1972-73 Hawaii Monitoring Report (See Mirex Monitoring-Final Report dated August 16, 1973).

Two additional sampling sites were established after the program began. The first was a location about one mile seaward from fields 233 and 234 (Figure 1) and located on the floor of Maliko Gulch. The other location was the Wahiawa Reservoir, a freshwater reservoir lake located on the Schofield Plateau of Central Oahu (Figure 3). The freshwater reservoir was sampled during the seventh or last monitoring cycle to see whether Mirex was accumulating in freshwater fauna.

<sup>&</sup>lt;sup>1</sup>Soil Survey of Islands of Kauai, Oahu, Maui, Molokai, and Lanai, State of Hawaii 1972. U.S. Department of Agriculture, Soil Conservation Service in Cooperation with the University of Hawaii Agricultural Experiment Station.

## Sample Material

## 1. <u>Soil</u>

Samples were taken by removing topsoil to a depth of one (1) centimeter from a nine (9)-foot square. Two samples per round were taken downslope from treated fields.<sup>2</sup> One was taken adjacent to field 233 where the surface runoff converged after exiting the field. The other was taken from a similar location outside field 234. Watson Okubo, WRRC, was responsible for soil sample collection.

## 2. Birds

A minimum of five bird specimens were taken during each round, if possible, by shooting them within the confines of the fields 233-234 area. Birds entering or exiting this area were also taken. If possible, they represented those species having a food preference for insects and other invertebrates (i.e. Golden Pacific plovers, <u>Pluvialis dominica fulva</u> and the ruddy turnstone, <u>Arenaria interpres</u>). The sampling effort took place during early morning and late afternoon over two consecutive days, if necessary, to obtain the five bird minimum. We recognized from the very beginning, during our monitoring plan deliberation, that it would be entirely possible that less than five birds would be taken. Birds found dead in the sampling area still intact with no evidence of decomposition were also collected for analysis. Bird sampling was conducted under the supervision of Joseph S. Medeiros, a professional wildlife biologist employed on Maui by the Division of Fish and Game.

## 3. Rodents

Rodents, including the house mice, <u>Mus musculus</u>, Polynesian rat, <u>Rattus exulans</u> and the roof rat, <u>Rattus rattus</u> were taken with snap traps. A total of one hundred and fifty traps were set out along the margin and within field 235. These traps were not used in the open because birds would also have been caught. A minimum of five mammals per round were taken. Rodents were collected under the supervision of Joseph G. Duarte, Supervisor, Vector Control, State Department of Health, Maui Branch.

### 4. Mongooses

The Small Indian mongoose, <u>Herpestus auropunctatus</u>, was added to the sampling program during Cycle III. The rationale for its inclusion is that the mongoose fills the highest tropic level of the pineapple field food webs. It is not a true carnivore although it does eat birds and small mammals in addition to invertebrates, plants and plant material, detritus, garbage and feces. The only true carnivores that frequent Hawaiian pineapple fields are the Hawaiian or Pueo owl, <u>Asio flammeus sandwichensis</u> and the barn owl, <u>Tyto alba pratincola</u>. However, due to the limited numbers of these two birds, we decided against

 $<sup>^{2}</sup>$ Soil samples were not taken during cycles three (3) and five (5).

sacrificing them for the study. We tried to capture a minimum of five mongooses per cycle. They were taken with live traps set out along the brushy Maliko Gulch margin of Field 235. Live traps were also used to capture mongooses at the Maliko Gulch site. Mongooses were trapped under the supervision of Mr. Duarte.

## 5. Aquatic Specimens

Fish were captured by spear when the visibility was adequate. Otherwise a throw or skirt net was used. Other specimens were taken by hand. The skirt net is composed of two netting sheets, one having a larger mesh (5 inches) and the other a smaller mesh (2 and 3/4 inches). Both large and small fish were taken with this net. The throw net was used when the skirt net could not be handled effectively, for example on a surge zone. It was set perpendicular to the shore with the larger mesh on the side that moving fish would encounter. After the net was settled, the water was agitated to drive the fish into the net. Mr. Okubo, WRRC, was responsible for aquatic marine specimen collection.

#### Sample Schedule

The sampling schedule for soil, birds and mammals was established in accordance with the EPA Determination and Order. Cycle I took place approximately one week before Mirex was applied to the target area. Cycle II samples were taken during the week after application. Cycles III through VII were taken during the 4th, 12th, 18th, 24th and 36th weeks after spraying. Marine specimens were taken during Cycles I and VI. Mongooses were captured during the third through the seventh cycles. A mussel sample, <u>Isognomon californicum</u>, was taken during Cycle IV, and fresh water fishes were taken during Cycle VII at the request of local fisheries and aquatic biologists. A breakdown of the specimens collected by Cycles is provided in the following table:

Cycle	Dates	Specimens
I	Oct. 6-12, 1973	Soil, Birds, Rodents, Marine Fishes
II	Oct. 31-Nov. 1, 1973	Soil, Birds, Rodents
III	Nov. 20-21, 1973	Birds, Rodents, Mongoose
IV	Jan. 17-19, 1974	Soil, Birds, Rodents, Mongoose, Mussels
V	Feb. 27-28, 1974	Birds, Rodents, Mongoose
VI	Apr. 16-21, 1974	Soil, Birds, Rodents, Mongoose, Marine Fish, Invertebrates and Seaweed
VII	July 10-13, 1974	Soil, Birds, Rodents, Mongoose and Freshwater Fish

#### Sample Identification

Labels were affixed at the time of collection. Data provided included species name, date of collection, specific sampling location and any special information thought to be important by the biologist (i.e. the exhibiting of unusual behavior by birds shot or specimens that were dead and intact when found by the biologist).

#### Sample Handling

Soil specimens were placed in pre-baked metal cans. All biological specimens were individually wrapped in new aluminum foil and chilled until their delivery to the Department of Agricultural Biochemistry Laboratory. Soil, bird and mammal samples were assembled and shipped to Honolulu by staff of the Department of Agriculture's Maui office under the direction of Mr. Nobuo Miyahira.

### SAMPLE PREPARATION AND ANALYSES

#### 1. EQUIPMENT

#### Sample Preparation

Dissecting instruments.

One-pint Mason jars

Sorvall Omni-Mixer Homogenizer

Two speed Waring Blender with standard 1000 ml capacity removable container of heat-resistant glass and semi-micro stainless steel container having a capacity of 360 ml.

#### Sample Extraction

Soxhlet apparatus, size 23

500 ml round bottom flasks, 24/40 Joint

Briskeat heaters, 500 ml size

Glass wool (rinsed with redistilled acetone and redistilled benzene and heated at 200°C)

### Sample Cleanup

- 100 ml graduated cylinders
- 80 mm powder funnels
- 100 ml Erlenmeyer flasks
- 300 ml Round bottom flasks, 24/40 Joint
- 400 mm x 20 mm I.D. chromatographic columns with 200 ml reservoir, fitted with a Teflon Ultramax valve.

To prevent cross contamination of samples, all glassware was soaked in dichromate cleaning solution, rinsed with tap water and dried with redistilled acetone and last rinsed with redistilled hexane. The dry glassware was then heated at 200°C overnight.

## Additional Equipment

Rotary vacuum evaporator

2. REAGENTS

Sodium sulfate, anhydrous powder (J. T. Baker #3898) QUSO - G3O, Unreductionized precipitated silica (Philadelphia Quartz Company) Ethyl ether for Fat Extraction - Mallinckrodt #0844 Petroleum ether (redistilled) Acetonitrile (redistilled) Acetone (redistilled) Hexane (redistilled and nanograde) Florisil (Regular) 10% Eluting solvent (10 nl distilled water to 100 ml with acetonitrile) 6% Eluting solvent (6.0 nl ethyl ether to 100 ml with petroleum ether) Desiccant Mix (10% QUSO, 90% anhydrous sodium sulfate) Mirex Standard Solution: 1.0 ng/5ul in hexane (0.2 ppm)

### 3. SAMPLE PREPARATION

Samples were prepared as soon as possible after collection. Unprepared samples were frozen. All tissue fluids were saved and blended with the thawed samples.

#### Opihi

- 1. Weights were recorded before and after shucking.
- 2. The entire animal was removed and blended in a Waring Blender for one minute or until samples was homogeneous.
- 3. Thirty gms of the homogenate was weighed into pint-size Mason jars.
- The jars were placed in the freezer to chill about 1/2 hour DO NOT FREEZE.
- 5. The jars were removed from the freezer and exactly 4X the sample weight of the desiccant mix was added.

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- 6. The desiccant and sample were mixed thoroughly with a spatula with care to incorporate all tissue adhering to sides and bottom of jar.
- 7. The mixture was allowed to freeze (about 1 hour).
- 8. The mixture was removed from the freezer and ground on a Sorvall Omni-Mixer until it was relatively free flowing. Refreezing and regrinding were necessary to obtain a free flowing mixture.
- 9. The sample was stored in the freezer until ready for analysis.

#### Nerita

Weights were recorded. Then the entire specimens were blended in a Waring Blender for one minute or until the sample was homogeneous. The Nerita were then processed in accordance with Steps 3-9 described under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.

#### Mussels

Mussels were processed according to Steps 1-9 under "Opihi," except that 3X the sample weight of desiccant mix was added.

#### Sea Urchin, Seaweed

Samples were blotted dry and weights recorded. Preparation proceeded as with Step 2 under "<u>Opihi</u>," except that 3X the sample weight of desiccant mix was added.

#### Sea Cucumber

Samples were blotted dry and weight recorded. The animal was sliced into 1/2 inch sections. Preparation proceeded as with Step 2 under "Opihi," except that 3X the sample weight of desiccant mix was added.

## Fish

- Small fish (length less than 4cm) were blotted dry and weights recorded. Preparation proceeded as with Step 2 under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.
- 2. Small fish (total weight less than 100 gms) were blotted dry and weights recorded. Fish were scaled and head and tail discarded. If fish were small, similar species were combined to obtain sufficient sample. Preparation proceeded as with Step 3 under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.

3. Large fish (total weight more than 150 gms) were blotted dry and weights recorded. Fish were scaled and sample was taken from the fillet. Preparation proceeded as with Step 3 under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.

### Birds

Head, feathers, legs, and viscera were removed. Head and viscera were saved frozen. Breast muscle and both wing muscles were composited as one sample. Preparation proceeded as with Step 2 under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.

#### Mice

Head, skin, feet, tail and viscera (including heart, lungs and kidneys) were removed. Head and viscera were saved and frozen. Preparation proceeded as with Step 2 under "Opihi," except that 2X the sample weight of desiccant mix was added.

#### Roof Rat, Polynesian Rat

Head, skin, feet, tail and viscera (including heart, lungs and kidneys) were removed. Head and viscera were saved and frozen. Samples were taken from the tissue of the back and legs. Preparation proceeded as with Step 2 under "Opihi," except that 2X the sample weight of desic-cant mix was added.

#### Mongoose

Tissue from the two hind legs and lower back were prepared and individuals were analyzed separately. Preparation proceeded as with Step 2 under "<u>Opihi</u>," except that 2X the sample weight of desiccant mix was added.

#### Soil

Moisture determinations for each soil sample was done in the following manner: About 10 gms of soil were weighed exactly into a tared aluminum weighing dish. The sample was dried overnight at room temperature then dried at 110°C for 16 hours and cooled in a desiccator. The samples were reweighed and % Solids calculated. Sample preparation was as follows: Samples were air dried at room temperature for at least 72 hours, then blended in a Waring Blender for one minute. The soil was transferred to pint-sized Mason jars for storage and easy sampling.

## SAMPLE EXTRACTION AND CLEANUP

## Sample Extraction

- 1. A 1-inch thick plug of treated glass wool was placed in the bottom of an assembled extraction apparatus.
- The prepared sample-desiccant mixture was added and topped with a 1/2 inch thick wad of glass wool.
- 3. 250 ml petroleum ether was added to a 500 ml boiling flask. For sediments and soils the extraction was accomplished with a 1:9 acetone: petroleum ether mixture.
- 4. Samples were extracted for four hours. Heaters were adjusted to permit solvent to cycle once every 6-7 minutes.

## Sample Cleanup (Biota Only)

- 1. Extracts were concentrated to approximately 10 ml on a rotary evaporator and transferred in 3-4 ml portions to a 400 mm x 20 mm chromatographic column containing a plug of glass wool topped with 3 inches of unheated Florisil (regular). After each portion settled in the column, vacuum was applied to evaporate the solvent.
- 2. Vacuum was also applied after each of three 5 ml rinses of petroleum ether from the extraction flask.
- 3. The vacuum was disconnected after all solvent was evaporated and the residue was eluted from the column with 70 ml of a 9:1 mixture of acetonitrile:distilled water into a 300 ml round bottom flask.
- 4. The eluate was evaporated to dryness in a rotary evaporator, with care that the sample did not bump or bubble.
- 5. Biota were cleaned up on an additional column explained below.

## Florisil Cleanup

- A 400 mm x 20 mm chromatographic column was packed with four inches of Florisil (Regular, heat treated at 130°C for five hours) and topped with 1/2 inch sodium sulfate.
- 2. The column was washed with 35 ml of petroleum ether and the wash discarded.
- 3. The dried extract was transferred to the column with petroleum ether and eluted with 200 ml of 6% ethyl ether in petroleum ether and collected in a 300 ml round bottom flask.

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- 4. The eluate was evaporated to approximately one ml and transferred to a volumetric flask.
- 5. The sample was made to volume with hexane. Appropriate aliquots of sample and standards were injected on G.C. and peak heights compared to compute residues.

## Analysis of Samples

Samples were analyzed with a Hewlett-Packard Model 5750, G.C., fitted with a 1/4" o.d. x 4' glass column, 2% OV-101 on Gas Chrom Q, 100/120 mesh.

Column Temperature:	196°C
Injector Temperature:	212°C
Detector Temperature:	207°C
Nitrogen gas flow rate:	60 ml/min

### Sample Confirmation

During 1973-74 Monitoring Period, 3 samples were confirmed by M.S.-G.C. at the U.H. Agricultural Biochemistry (12/5/73)

Sample #169 - Mongoose - Maliko (Sampled 11/21/73)

Sample #171 - Rattus rattus - Maliko (Sampled 11/21/73)

Sample #180 - Rattus exulans - Maliko (Sampled 11/21/73)

The mass Spectrophotometer was a Finnegan Model 3000 G.C. Peak Identifier (Quadropole Mass Spec.). The confirmatory column was: 0.75% OV-17, 0.97% OV-210 on Gas Chrom Q 100/120 mesh. Other parameters remained the same as during the regular sample analysis.

## Standards

	Standards	<u># of Samples</u> (including fortified
lst Round	9	· 14 samples)
2nd Round	8	8
3rd Round	21	18
4th Round	7	18
5th Round	21	24
6th Round	26	41
7th Round	24	43
TOTAL	116	166

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Generally, a standard was run before and after every 2-4 samples.

## Fortified Samples

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Round	Date	Sample <u>No.</u>	Sample Character	Mirex added	Recovery percent
lst	10/6/73 to 10/11/73	151F 146F 141F	Rat, <u>rattus</u> <u>rattus</u> Labridae Maliko Soil-Field 233	0.1 0.1 0.1	77 98 91
2nd	10/31/73 to 11/5/73	161F	Soil	0.1	80
3rd	11/20/73 to 11/21/73		No Fortified Samples	×	
4th	1/17/74 to 1/19/74		No Fortified Samples		
5th	2/27/74 to 2/28/74	216F	Mongoose fetus	0.1	(spilled sample)
6th	4/16/74 to 4/21/74	267F	Nerita	0.5	76
7th	7/10/74		No Fortified Samples		

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### RESULTS AND DISCUSSION

#### A. Soil

A total of ten (10) soil samples were collected during the 1973-74 Hawaii Mirex Monitoring Program (Table I). Five were taken from the Field 233 location and the other five from the Field 234 site. Mirex was detected in six samples. The mean Mirex residual for these samples was 3.9 ppb, with a range of none detected (N.D.) to 9 ppb. The mean detected concentration for the Field 233 site was 1.4 ppb with a range of N.D. to 4 ppb. For Field 234, the mean was 6.4 ppb with a range of N.D. to 9 ppb detected.

The average residual from the two soil sampling sites represented 4.5% of the amount applied to an equivalent area (Attachment <sup>3</sup>): The average percent residual in Field 233 soil samples was 1.7% and in Field 234 was 7.6%. The two soil sampling sites were specifically selected to represent sediments transported by surface runoff. Therefore, the residual to application ratios or percentages would represent maximum rather than average Mirex transport from the fields.

#### B. Birds

A total of 20 birds were captured during the 1973-74 Hawaii Mirex Monitoring Program. Out of the twenty, two were negative for detectable Mirex (one each barred dove and lace necked dove). Due to a chemical interference, we were unable to obtain an accurate Mirex residual measurement on a third bird, a ruddy turnstone. Of the seventeen birds positive for Mirex, eleven were Golden Pacific Plovers, three were ruddy turnstones, one was a barred dove and two were common mynah birds.

The Golden Pacific plover and the ruddy turnstone are both migratory in behavior.<sup>3</sup> They reside in Hawaii from September to May and feed principally on insects and insect larvae they find in open areas especially in newly plowed and planted fields.

Of the two only one plover was captured during cycle one or before Mirex was applied to the pineapple fields. The Mirex residual measured in this bird was 118 ppb (Table I). The arithmetic mean Mirex residual for the 10 plovers taken after Mirex application was 1559 ppb. However, the variability among these samples was large: the range in residuals was from 24 to 10,400 ppb. No trends of either Mirex accumulation or excretion could be ascertained.

The three positive ruddy turnstones were collected after Mirex application. Their mean residual was 407 ppb.

<sup>&</sup>lt;sup>3</sup>From Statement of David H. Woodside, Witness for the State of Hawaii at the E.P.A. Mirex Hearing held in Honolulu, Hawaii, June, 1974.

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Barred and lace necked doves are mainly seed eaters although they also consume berries and insects.<sup>3</sup> The Mirex residual in the barred dove captured before the pesticide was applied to the pineapple fields was 6 ppb (Table I). No Mirex was detected in the doves taken during Cycles IV and VII.

The mynah bird was the only other bird species that had a detectable Mirex residual. These birds are ground feeders where they search for insects in newly plowed and immature fields.<sup>3</sup> Both specimens were taken after Mirex was applied to the fields (Table I). The Mirex residuals were 325 and 30 ppb during Cycles V and VII, respectively.

#### C. Mammals

A total of three house mice, eighteen Polynesian rats, twenty-three roof rats and twenty-two Small Indian mongoose samples were taken at the Field 235 sampling site. All contained detectable Mirex. Ten additional mongooses were trapped at the Maliko Gulch Station. Six mongoose fetuses were also analyzed: five of which were removed from females collected at the Maliko Gulch sampling site.

Of the three house mice samples taken at the Field 235 sampling site, one was taken before Mirex was applied to the field. The Mirex residual in this composite sample of four mouse specimens was 281 ppb. Mirex in the two mice samples collected after application was measured at 379 ppb in the one specimen trapped during Cycle II and 890 ppb in the composite sample of five mice collected during Cycle IV.

Rats are adaptable in their feeding behavior. They will eat any organic material available.

The feeding range of the Polynesian rat is limited to about 100 feet.<sup>4</sup> In the pineapple field, they gnaw on the stumps of the pineapple plants. Presumably, because of their limited range, they also select other food sources in the fields such as arthropods and seeds.

A composite sample of five specimens trapped during the preapplication Cycle (I) had a Mirex residual of 66 ppb (Table I). The arithmetic mean for the 17 samples collected after application was 1536 ppb. The pesticide concentration in these post-application samples ranged from 24 ppb to 9410 ppb. Residual levels appeared to drop off rapidly after the third post-application Cycle (IV). To verify this hypothesis, a t-test was applied to the two sets of data (i.e. Cycles II-IV and Cycles V-VII). The results, Attachment 4, show a high level of probability (0.05 < P < 0.02) that the pesticide residual level did decrease during the second half of the post-application monitoring program.

<sup>&</sup>lt;sup>4</sup>Kramer, R. J. 1971. <u>Hawaiian Land Mammals</u>. Charles E. Tuttle Co., Inc.: Rutland, Vermont, pp. 82-137.

Roof rats have a feeding range of about double the diameter of that of the Polynesian rat.<sup>5</sup> They apparently reside in the brushy gulch and enter the pineapple fields to find food. They eat whatever is available: this can include grass stems, fruits, birds, bird eggs, etc.

Mirex residual detected in the composite sample of four rat specimens collected prior to Mirex application was 13 ppb. The arithmetic mean for Mirex residuals in the 22 post-application samples of individual samples was 497 ppb. As with the Polynesian rats, there was an apparent decrease, with time, in the level of the residual. Again a t-test was applied, Attachment 4. The results show a high level of probability (0.001 < P) that the Mirex concentration did decrease during the second half of the post-application monitoring program.

Mongooses are opportunistic feeders, in that they eat any organic material available. These foods can include rats and mice, arthropods, crustaceans, birds, plant material, garbage and carrion. They have a fairly extensive feeding range of up to about a quarter of a mile.

All 22 mongooses trapped at the Field 235 sampling area contained detectable Mirex residuals. The arithmetic mean for these post-application samples<sup>5</sup> of individual specimens was 2455 ppb. The individual sample residuals varied from 37 ppb to 11,760 ppb. The sample Mirex concentration seemed to decrease after Cycle V. To test this hypothesis, a t-test was applied to the two sets of data (i.e. Cycles III-V and Cycles VI-VII) [See Attachment 4]. The results show a high level of probability (0.01 < P < 0.001) that the Mirex concentration did decrease during the sixth and seventh cycles.

Ten mongooses were trapped at the Maliko Gulch site during Cycles V and VI (Table II). The arithmetic mean for these 10 samples of individual specimens was 171 ppb. The range in pesticide concentrations varied from 20 ppb to 480 ppb. As a comparison, the arithmetic mean for the Cycle V-VI specimens taken from the Field 235 site was 1218 ppb or seven times larger in magnitude.

The arithmetic mean for the Mirex residuals in the five mongoose fetuses was 126 ppb.<sup>-</sup> The range was 6 ppb to 625 ppb. The arithmetic mean concentration in the mother mongoose was 1,023 ppb; thus the average fetus concentration was 12% that of their mother's level.

#### D. Aquatic Specimens

A total of 11 marine fish were captured during the 1973-74 Hawaii Monitoring Program (Table I). Three of 27% contained detectable Mirex. The positive fish were: a labrid taken from Maliko Bay, an Aholehole and a Kupipi both taken from Honokohau Bay. The Mirex residual was 3 ppb in each of these three fish.

<sup>5</sup>No mongooses were captured prior to Mirex application.

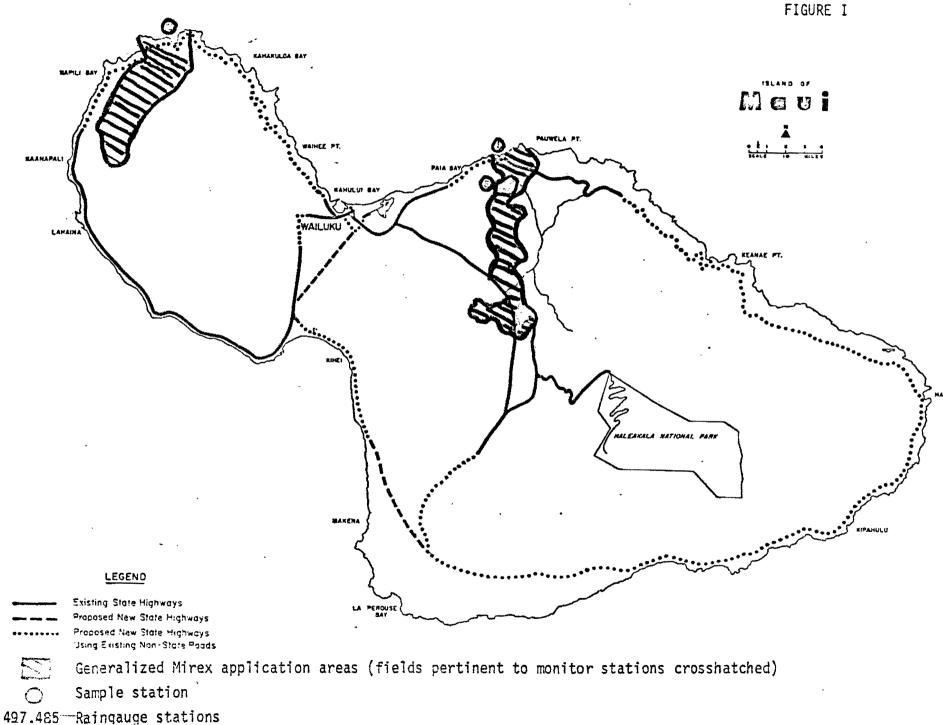
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A total of five mollusk samples, three echinoderm and two seaweed samples were collected from the Maliko and Honokohau Bay (Tables I, II). None contained detectable Mirex.

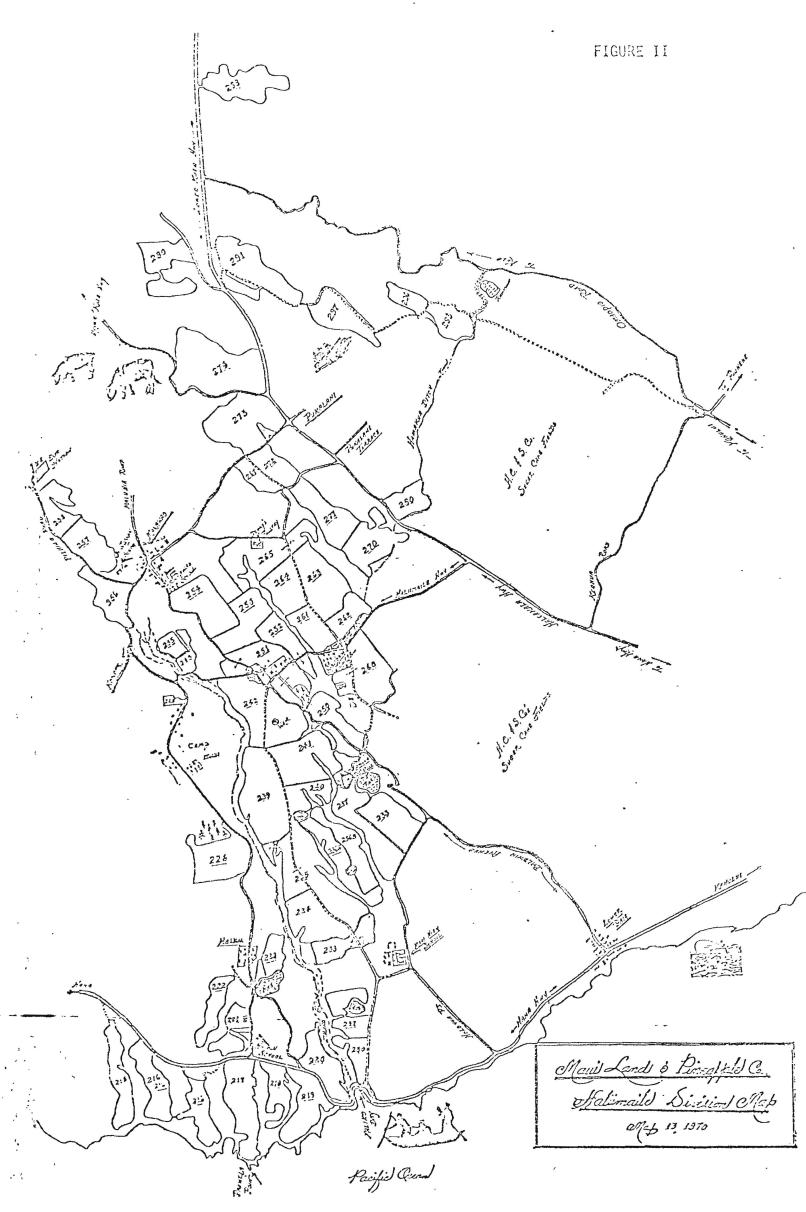
Two freshwater fish samples were collected during Cycle VII (Table II): a large mouth bass and a composite of 4 tilapias. Neither sample contained detectable Mirex.

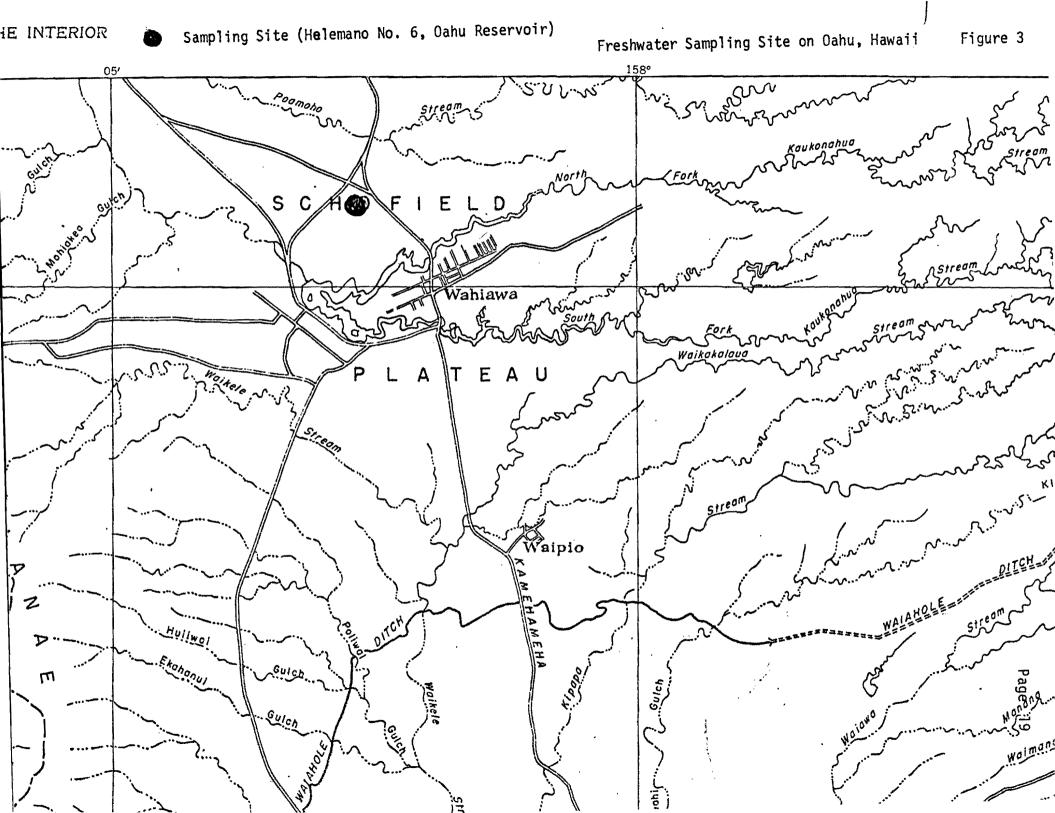
#### CONCLUSIONS

- 1. Mirex accumulated to appreciable levels in the Golden Pacific plovers, Polynesian rat, roof rat and Small Indian mongoose populations associated with the Maliko pineapple growing area. In the two rat species, the levels peaked out during the first three post-application cycles. Mongoose Mirex residuals began to drop after the fifth cycle.
- 2. There was no evidence of a Mirex buildup in the aquatic organisms sampled.









## TABLE ISamples Collected in Accordance with the Sampling Plan

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Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
141	Soil <sup>1</sup> (97% Solids)	Edge of Field 233	10/ 6/73					N.D. <sup>2</sup> , <sup>3</sup>
160	11 IF	11 11 17	11/ 3/73	11/ 3/73				3
197	" (74.5% Solids)	11 11 11	. 1/19/74	1/21/74				4
259	" (87.2% Solids)	11 II II	4/21/74	4/22/74		30		N.D.
291	" (94.9% Solids)	27 18 11	7/13/74	7/15/74				N.D.
142	" (97% Solids)	" " 234	10/ 6/73					N.D.
161	H 13	A 11 U	11/ 2/73	11/ 3/73				9
198	" (72.3% Solids)	11 11 11	1/19/74	1/21/74				5
260	" (92.3% Solids)	0 H H	4/21/74	4/22/74		30		9
292	" (90.9% Solids)	н п п	7/13/74	7/15/74				9
_ 149	House Mouse, <u>Mus</u> musculus	Edge and within Field 235	10/11/73				4	281
155	н н -	11 . 11	11/ 1/73	11/ 2/73	11			379
192	ц и	13 14	1/17/74	1/18/74	14		5	890
150	Polynesian Rat <u>Rattus exulans</u>	H 11	10/11/73				5	67
157	11 11	13 11	11/ 1/73	11/ 2/73	49/ specimen		5	8,435
176	11 13	17 11	11/21/73	11/21/73	55	. <mark>21</mark>		1,570 Page 20

TABLE I	Samples	Collected	in	Accordance	with	the	Sampling	Plan
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Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
177	Polynesian Rat <u>Rattus exulans</u>	Edge and Within Field 235	11/21/73	11/21/73	64	30		890
179	JI 11	, u n	II	R	60	24		1,470
189	и и .	a n	11	Iž	57	22		1,280
185	11 11	11 ti	1/17/74	1/18/74	57	10		9,410
186	11 11	11 II	п	81	39	7		790
187	и н	н	п	u	32	. 8		125
204	U 11	11 11	2/28/74	2/28/74	68	14		350
205	U H	17 11	11	ti.	63	12		430
229	87 B	н	4/17/74	4/17/74	60	18		890
230	11 H	11 11	11	11	53	16		50
231	11 11	JI 11	ŧ	n	53	15		150
271	41 11	. 13 IJ	7/10/74	7/10/74	62	15		133
272	11 11	n n	п	11	72	16		47
273	II 11	11 11	п	, n	22	7		68
274	17 14	11 11	11	11	51	11		24 -
151	Roof Rat Rattus rattus	11 11	10/11/73				4	13 Page
156	11 11 S	31 83	11/ 1/73	11/ 2/73	129/ specimen		5	a 1,060 <u>№</u>

## TABLE ISamples Collected in Accordance with the Sampling Plan

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
171	Roof Rat <u>Rattus</u> rattus	Edge and Within Field 235	11/21/73	11/21/73	,131	60		1,850
172	11 11	u u	н	11	139	65		110
173	11 11	u u	u	11	118	52		770
174	11 U	H LL	u	н	124	56		500
175	и с и	n n	u	31	132	56		1,670
188	11 11	п и	1/17/74	1/18/74	149	15		295
189	11 11	пп	11	U .	1.30	15	1	925
190	11 11	11 11	п	u	68	12		830
191	н н	в п	11	II.	126	15		960
206	H 11	ч	2/28/74	2/28/74	214	25		95
207	11 EI	н п	II	H	192	25		235
232	15 H	11 11	4/17/74	4/17/74	135	20		120
233	и и .	н н	н	n	74	20		85
234	<sup>.</sup> н н.	n n	11	II.	135	20		135
235	11 11	n n	11	n	145 `	23		715
236	17 14	11 U	n	"	153	20		435
275	11 H -	11 II	7/10/74	7/10/74	176	16		Page 13
276	U 11	u U	11	11	157	18		5 22

## TABLE I Samples Collected in Accordance with the Sampling Plan

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
277	Roof Rat <u>Rattus</u> rattus	Edge and Within Field 235	7/10/74	7/10/74	176	20		52
278	и п	. H H	н	LI II	55	16		21
279	11 11	11 H	u	11	137	15		49
166	Small Indian Mongoose <u>Herpestus auropunctatus</u>	Maliko Gulch edge of field 235	11/21/73	11/21/73	755	70		430
167	и и	и и	11	11	299	30		6,190
168	11 11	11 11	n		422	38		3,930
169	11 H	li ti	11	н	482	44		9,820
170	N 11	11 II	11	R	531	47		500
193	н н	12 11	1/17/74	1/18/74	445	20		11,760
194	11 11	11 11	11	81	360	20		2,080
195	11 11	11 11	. H	11	886	15		6,665
208	11 11	11 IT	2/27/74	2/28/74	468	20		2,940
209	и и	11 II	11	11	5,50	20		4,120
212	11 11	0 11	μ	11	824	25		1,730
213	II II	πы	II	11	672	20		470
237		н и	4/16/74	4/17/74	605	25		1,250 <sup>P</sup> age
238	11 11	11 11	11	u	405	<sup>.</sup> 20		a 30 23

#### TABLE I Samples Collected in Accordance with the Sampling Plan

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Sa	mple Type	Samplir	ng Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
Small I <u>Herpest</u> u	Indian Mongoose us auropunctatus	Maliko of fiel	Gulch edge Id 235	4/16/74	4/17/74	390	16		70
н	н	11	II	11	It	615	25		305
u	н	R	и ,		11	720	24		50
11	H	11	11	7/10/74	7/10/74	717	30		576
II.	H	н	11	11	H	440	20		200
11	14	11	11	ti	D	720	20		533
11	H	11	Ц	н	н	762	30		333
n	11	u	11	R	и	686	30		37
				10/11/73				2	118
11	IJ	11	II	10/31/73	11/ 2/73	104/ specimen		4	1,955
11	u	н	LI	11 ×	11	115			24 <sup>4</sup>
~11	и.	11	17 ·	11/20/73	11/21/73	113	31		440
U.	11	11	11	. 11	11	95	26		310 <sup>5</sup>
11	11	11	51	1/17/74	1/18/74	102	15		625 _
_0 _	R	11	н	2/27/74	2/28/74	127	15		10,400
11	ii s	n	11	11	11	118	15		1,250
	Small I Herpestu " " " " Golden <u>Pluvial</u> fulva " " " " "	<pre>" " " " " " " " " " " " " " " " " " " "</pre>	Small Indian Mongoose Herpestus auropunctatus       Maliko of field         """"""""""""""""""""""""""""""""""""	Small Indian Mongoose Herpestus auropunctatusMaliko Gulch edge of field 235"""""""""""""""""""""""""""""""""""	Sample TypeSampling LocationCollectedSmall Indian Mongoose Herpestus auropunctatusMaliko Gulch edge of field 2354/16/74"""""""""""""""""""""""""""""""""""	Sample Type         Sampling Location         Collected         Received           Small Indian Mongoose Herpestus auropunctatus         Maliko Gulch edge of field 235         4/16/74         4/17/74           """"""""""""""""""""""""""""""""""""	Sample Type         Sampling Location         Collected         Received         Weight (gm)           Small Indian Mongoose Herpestus auropunctatus         Maliko Gulch edge of field 235         4/16/74         4/17/74         390           """"""""""""""""""""""""""""""""""""	Sample Type         Sampling Location         Collected         Received         Weight (gm)         Analyzed (gm)           Small Indian Mongoose <u>Herpestus auropunctatus</u> Maliko Gulch edge of field 235         4/16/74         4/17/74         390         16           """"         """"         """"         615         25           """"         """"         """"         615         25           """"         """"         """"         720         24           """"         """"         710/74         7/10/74         717         30           """"         """"         """"         1"         440         20           """"         """"         """"         16         25           """"         """"         1"         10/11/74         717         30           """"         """"         """"         10/11/73         """"         686         30           Golden Pacific Plover <u>Pluvialis dominica</u> Maliko Fields 233 and 234         10/11/73         11/2 /73         104/ specimen           """"""""""""""""""""""""""""""""""""	Sample Type         Sampling Location         Date Collected         Date Received         Sample weight (gm)         Analyzed Analyzed (gm)         Compo- sited           Small Indian Mongoose Herpestus auropunctatus         Maliko Gulch edge of field 235         4/16/74         4/17/74         390         16           "         "         "         "         "         615         25           "         "         "         "         "         615         25           "         "         "         "         "         720         24           "         "         "         "         710/74         717         30           "         "         "         "         "         440         20           "         "         "         "         "         720         20           "         "         "         "         "         440         20           "         "         "         "         "         10/173         10         20           "         "         "         "         "         10/11/73         104/ specimen         2           fulva         "         "         "         10/31/73

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TABLE I Samples Collected in Accordance with the Sampling Plan	TABLE I	Samples	Collected	in	Accordance	with	the	Sampling	Plan
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Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
202	Golden Pacific Plover <u>Pluvialis dominica</u> <u>fulva</u>	Maliko Fields 233 and 234	2/27/74	2/28/74	119	15		180
203	пи	11 11	11	Ħ	138	20		330
227	n (i	11 K	4/17/74	4/17/74	165	25		80
164	Ruddy Turnstone Arenaria interpres	11 11	11/20/73	11/21/73	104	34		Inter- ference <sup>6</sup>
165	H U	11 11	FI	н	97	24		210
184	11 11	н н	1/17/74	1/18/74	80	13		810
228	u n	n u	4/17/74	4/17/74	92	10		200
153	Barred Dove <u>Geopelia</u> <u>striata</u> <u>striata</u>	11 14	10/11/73					6
182		11 11	1/17/74	1/18/74	40	12		N.D. <sup>7</sup>
183	Mynah Bird <u>Acridotheres</u> <u>tristis</u> <u>tristis</u>	11 17		11	120	19		325
199	tt tř	11 11	2/27/74	2/28/74	122	20		30
270	Lace Necked Dove <u>Stretopelia chinensis</u> chinensis	11 11	,7/10/74	7/10/74	167	20		N.D.
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## TABLE I Samples Collected in Accordance with the Sampling Plan

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mire <b>x</b> Resid <b>ual</b> (ppb)
144	Fish (Aholehole) <u>Kuhlia sandvicensis</u>	Maliko Bay	10/ 6/73					N.D.
251	н н	11	4/21/74	4/22/74	145	20	6	N.D.
145	Fish (Labridae) <u>Thalassoma</u> fuscum	11	10/ 6/73					N.D.
146	Fish (Labridae) <u>Anampses godeffroyi</u>	u	· 11					3
248	Fish (Maiko) <u>Acanthurus</u> <u>Leuco</u> - pareius	11	4/21/74	4/22/74	255	25		N.D.
250	Fish (Hinalea) <u>Thalassoma</u> umbrostigma	11	19	11	62	20		N.D.
252	Fish (Manini) <u>Acanthurus</u> <u>sandvicensis</u>	11	п	п	260	30	2	N.D.
148	Fish (Aholehole) <u>Kuhlia sandvicensis</u>	Honokohau Bay	10/ 6/73					3
261	н н	11	4/21/74	4/22/74	34	19		3
262	Fish (Kupipi) <u>Abudefduf</u> sordidus	11		If	44	24	2	N.D.
263	Fish (Maiko) <u>Acanthurus</u> <u>leuco</u> -	11	11	11	55	36		N.D.
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## TABLE I

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Samples Collected in Accordance with the Sampling Plan

174								
Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
265	Fish (Manini) <u>Acanthurus</u> <u>sandvicensus</u>	Honokohau Bay	4/21/74	4/22/74	205	25	2	N.D.
253	Mollusk (Opihi) <u>Cellana calcosa</u> and <u>C. exerata</u>	Maliko Bay	U	a	108	15 <u>.</u>	338	N.D.
254	Mollusk (Nerita) <u>Nerita picea</u>	11	H	"	250	30		N.D.
255	Mollusk (Mussel) <u>Isognomon</u> californicum	11	11		280	20		N.D.
267	Mollusk (Nerita) <u>Nerita picea</u>	Honokohau Bay	11	n	305	30		N.D.
268	Mollusk (Opihi) <u>Cellana calcosa</u> and <u>C. exerata</u>	11	u	15	40	17	17 <sup>9</sup>	<u>N.</u> D.
256	Echinoderm (Flat sea urchin) <u>Colobocentrotus</u> sp.	Maliko Bay		11	135	30	5	N.D.
257	Echinoderm (Sea Cucumber) <u>Holothuria atra</u>	'n 'n	n	п	565	30	2	N.D.
266	Echinoderm (Flat sea urchin) <u>Colobocentrotus</u> sp.	Honokohau Bay	. 11	11	140	30	4	Page 27 N.
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TABLE I

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Samples Collected in Accordance with the Sampling Plan

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residual (ppb)
258	Seaweed Sargassum sp.	Maliko Bay	4/21/74	4/22/74	190	30	10	N.D.
269	u u	Honokohau Bay	n	п	205	30	10 ן	N.D.
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Footnotes:

- 1. Soil from the vicinity of fields 233 and 234 are of the Pauwela-Haiku Association Haiku Series, 3-5% slope.
- 2. Soil analyzed on a dry weight basis.
- 3. 3ppb detectable limits.
- 4. Found dead.
- 5. Speciman appeared to be sick, reluctant to fly.
- 6. Interference due to some other substance(s) present in the sample. The peak of the unknown overlaped that of Mirex on the gas chromatograph.
- 7. 12ppb detectable limits.
- 8. With shell.
- 9. With shell.
- 10. One can of seaweed.

## TABLE II Additional Samples Collected During the 1973-74 Monitoring Year

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed `(gm)	No. Compo- sited	Mirex Residual (ppb)
196	Mussels <u>Isognomon</u> californicum	Maliko Bay	1/19/ 74	1/21/74	376	15		N.D. <sup>1</sup>
210	Small Indian Mongoose (fetus from speciman 209)	Maliko Gulch edge of Field 235	2/27/74	2/28/74	. 8	8		625 (4,120 ppb in mother)
214	Small Indian Mongoose <u>Herpestus</u> auropunctatus	Maliko Gulch ap- proximately one mile seaward of fields 233 and 234	u	u	600	25		350
215	Small Indian Mongoose (fetus from speciman 214)	н н	I	н.	49	49		15
216	11 11	41 IT	19	11	33	16		30
217	Small Indian Mongoose	n n	n	bi bi	510	20		145
218	Fetus from Mongoose No. 217	ii II		· 11	9	9		20
220	Small Indian Mongoose	31 11	11	. 11	550	25		20
221	Fetus from Mongoose No. 220	91 H	11	13	30	30		6
223	Small Indian Mongoose	11 R	11	11	4 <u>5</u> 0	18		480
224	Fetus from Mongoose No. 223	11 11	n	11	25	25		60 Page
226	Small Indian Mongoose	n n	н	11	650	25		ge 30
<sup>1</sup> 7 ppb li	it of detectability.							

## TABLE II Additional Samples Collected During the 1973-74 Monitoring Year

Sample No.	Sample Type	Sampling Location	Date Collected	Date Received	Sample Weight (gm)	Analyzed (gm)	No. Compo- sited	Mirex Residua (ppb)
242	Small Indian Mongoose	Maliko Gulch ap- proximately one mile seaward of fields 233 and 234	4/16/74	4/17/74	455	20		35
243	11 11	11 11	11	H	480	20		370
244	н `н	11 11	11	El	427	20		90
245	и и	11 11	ti	II	460	20		105
246	11 II	n u	B	u	810	20		30
287	Large Mouth Bass	Hale mano No. 6 Oahu Reservoir	7/11/74	7/11/74	3,174	30		N.D.
290	Tilapia	11 11	11	11	633	30	4	N.D.
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#### REGION IX 100 CALIFORNIA STREET SAN FRANCISCO, CALIFORNIA 94111

COPY Page 32

ATTACHMENT 1

# AUG 31 1973

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Mr. John J. Tolan Executive Vice President Pineapple Growers Association of Hawaii 1902 Financial Plaza of the Pacific Honolulu, HI 96813

Dear Mr. Tolan:

I enclose a copy of an Order of the Environmental Protection Agency in FIFRA Docket Nos. 146 and 293 (in re: Allied Chemical Corp.). This Order is a modification of previous Orders that prohibited the use of Mirex but stayed that prohibition for aerial application of Mirex in Hawaii on the condition that an approved monitoring program be instituted and carried out. This Order modified the required monitoring program, which must be approved by EPA and must contain at least the six elements included in the Order.

Any application of Mirex otherwise than as limited by this Order of August 30, 1973, is subject to a \$5,000 penalty for each offense [7 U.S.C. 1361(a)(1)]. Any knowing violation is subject to criminal penalties of \$25,000 or one year imprisonment, or both, for each offense [7 U.S.C. 1361(b)(1)].

It will be appreciated if you will bring this Order to the attention of your members and anyone else who may be affected.

The monitoring program proposed to be carried out in accordance with this Order should be submitted to EPA, Region IX, 100 California St., San Francisco 94111. If you have any questions concerning this matter, please call Mr. Robert G. Kuykendall at (415) 556-3352.

> Sincerely, Richard L. O'Connell, Director Enforcement Division

Enclosure

BEFORE THE ENVIRONMENTAL PROTECTION AGENCY

In re:

ALLIED CHEMICAL CORP.

FIFRA Docket Nos. 146 and 293

Reg. No. 218-586

#### DETERMINATION AND ORDER

)

Under authority granted by the Federal Insecticide, Fungicide and Rodenticide Act (FIFRA) (7 USC 135b[c]), the Administrator of the Environmental Protection Agency on May 3, 1972 prohibited the aerial application of pesticides containing Mirex in coastal counties or parishes and on or near rivers, streams, lakes, ponds, and other aquatic areas. (37 Fed. Reg. 10987) On June 30, 1972 the Administrator issued a Determination and Order (37 Fed. Reg. 13299) granting a stay of his May 3 prohibition with respect to aerial spraying of Hawaiian pineapple fields in th fall of 1972 to control mealybug wilt. The Administrator's stay was conditioned upon the implementation of a monitoring program approved by the Environmental Protection Agency.

Pending the completion of a public hearing on registrations of pesticides containing Mirex which this Agency has called pursuant to Section 6(b)(2) of the FIFRA, as amended in 1972, the Acting Administrator of the Environmental Protection Agency issued a Determination and Order dated May 25, 1973 which extended through the 1973 spraying season the stay approved initially on May 30, 1972. Again, however, the prohibition against aerial spraying was stayed only on condition that a monitoring program approved by this Agency would be conducted in conjunction with spraying.

In compliance with the Administrator's Order of June 30, 1972, a monitoring program was instituted to measure the effects of Mirex when used to control mealybug wilt in Hawaii. Since results of laboratory experiments and other information had indicated that Mirex is acutely toxic to certain species of aquatic biota, the monitoring program was aimed predominantly at marine biota found in waters adjacent to those islands on which Mirex was sprayed. On these islands there are no rivers or streams in which significant numbers of other aquatic biota can be found.

As reflected in the Administrator's Order of May 25, 1973, however, information gained from this monitoring program has consistently indicated that Mirex is not accumulating significantly in marine biota. I have therefore determined to modify the required monitoring program to reduce the number of samples taken in offshore waters and to require that additional samples be collected from terrestrial biota inhabiting the islands on which Mirex is sprayed. This will allow us to determine whether greater sampling in this area would indicate occurrence of any residues in inland species. Specifically, I will require that the following elements be included in a monitoring program to be implemented in conjunction with the fall 1973 spraying of Mirex in Hawaii:

Cl. Aquatic sampling to be limited to those organisms which were positive for Mirex in previous samples;

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- 2. Monitoring to include at least one terrestrial area of sufficient acreage (2500-5000 acres) to provide multiple sites where Mirex is likely accumulate, i.e., ravines, water courses, (dry) irrigation areas where soil or water movement occurs intermittently;
- 3. Terrestrial monitoring to include soils, small mammals and birds, with each of these groups yielding five samples for a total of fifteen per collection;
- 4. Baseline (pre-spraying) samples to be collected for terrestrial species monitored:
- 5. Sampling collections to be made during the 1st, 4th, 12th and 18th weeks after spraying, and also during the 24th and 36th weekds if samples containing any residues of Mirex are collected during the 12th and 18th weeks.
- 6. Monitoring areas, species to be sampled, and sampling and reporting methodology to be determined in conjunction with the Environmental Center and the Zoology Department of the University of Hawaii.

In thus revising the monitoring program, however, I do not wish to imply that the provisions of any order issued by this Agency are hereby extended to authorize spraying operations in Hawaii after the fall of 1973. Approval for further aerial spraying in Hawaii will be granted only if warranted by the results of monitoring done in coming months and by the record adduced at the impending public hearing on Mirex registrations.

August 30, 1973

Charles L. Elkins Acting Assistant Administrator for Hazardous Materials Control

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Page 36 Attachment 2

# UNIVERSITY OF HAWAII

Environmental Center

Office of the Director

September 14, 1973

Environmental Protection Agency Attn: Mr. Robert G. Kuykendall Region IX 100 California Street San Francisco, California 94111

Gentlemen:

Re: Order of the Environmental Protection Agency in FIFRA Docket No. 146 and 293

Enclosed are copies of the program for environmental monitoring of Mirex in areas of use in Hawaii during the 1973-74 season provided in response to the Determination and Order dated August 30, 1973. The original Determination and Order dated June 30, 1972 applied to the period of August through October which is the normal season for applying Mirex.

Application of Mirex was to have begun in August pursuant to the Determination and Order dated May 25, 1973 which extended the stay through the 1973 spraying season. The Determination and Order requires a monitoring program to be implemented in conjunction with the fall 1973 spraying of Mirex in Hawaii. This monitoring program limits sampling of the aquatic environment to those vertebrate species which were positive for Mirex in previous samples and requires monitoring of at least one terrigenous area for soils, small mammals and birds.

The sampling plan submitted deviates in part from the order because of physical constraints.

1. Only two soil samples will be taken; one at a point adjacent to the treated field to determine drift and the other at the point of confluence of drainage leaving the growing area. Only the Maliko watershed drainage meets the acreage requirement. Fields in crop are completely closed in by vegetation making entry difficult and severely limiting runoff and potential washout of particles. Thus sampling is limited to newly planted fields subjected to erosion and thus transport of particles by runoff.

2. It is anticipated that the bird populations will be very low in fields free of weeds. Birds in habitats near the treated fields would only transit treated areas and thus would not be exposed to Mirex treated Environmental Protection Agency

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September 14, 1973

particles. It may not be possible to obtain five samples as required. This situation is similar to the one encountered last season in taking fish samples from the easily accessible, overfished bays characteristic of the area.

The sampling plan proposed to monitor Mirex in compliance with the Determination and Order has been developed by the Environmental Center of the University of Hawaii in coordination with the Agricultural Biochemistry Department, College of Tropical Agriculture, and the Departments of Land and Natural Resources and Agriculture of the State of Hawaii. The sampling program will be coordinated by the Environmental Center; aquatic sampling by the Water Resources Research Center, University of Hawaii; and terrestrial sampling by the Department of Land and Natural Resources, Division of Fish and Game and the Department of Health, Vector Control Branch.

It must be emphasized that very little time was provided to develop this sampling program in response to the Determination and Order. Population estimates for fields are used to determine whether treatment is required. These estimates are now complete and approval of the sampling plan must be expedited to assure completion of the treatment program before the end of October when increased rainfall can be expected. The bait is less effective once the rainy season begins.

Earliest possible action is required to permit compliance with the Determination and Order.

Sincerely yours,

Jerry M. Johnson Acting Director

cc: Honolulu EPA UH WRRC UH Ag. Biochem. State DOA State DLNR State DOH State OEQC J. Tolan, PGA

### Proposed Hawaii Mirex Monitoring Program 1973-74

#### <u>Objectives:</u>

- To determine detectability of Mirex in marine vertebrates in estuarine sites receiving runoff water from areas of Mirex application in Hawaii.
- 2) To determine transport of Mirex from fields by water into watercourses and areas where intermittent water flows may transport soil and Mirex treated particles.
- To determine detectability of Mirex in terrestrial small mammals and birds within or adjacent to areas of Mirex application in Hawaii.

#### Target Analytic Sensitivity

10 ppb

## Sample Size

A minimum of 60 g per sample to be split into two sub-samples, one for analysis and one for reserve.

#### Sample Sites

<u>No.</u>	<u>Island</u>	Area	<u>Site</u>	Sample Type
1	Maui.	Maliko 2000+acres	Maliko Bay	Marine vertebrates Aholehole, Kumu and Wrasse as available
2	Maui	Maliko	Lower fields	Soils from watercourse and field discharge at 2 sites
3	Maui	Honokohau	Honokohau Bay	Same as No. 1
4	Maui	Honokohau 600+acres	Lower fields	Same as No. 2

- A. Minimum of three fish samples per site with each fish sample to be composited to meet the minimum weight requirement.
- B. Soil samples to be taken will consist of the soil removed to a depth of 1 cm from a 9 square foot area. Samples will be taken at two points, all downgrade from the treated field, one from the road or watercourse adjacent to the field, and another from the area where watercourses converge to exit from the field.

One distinct field area will be sampled; representing the newly planted crop. Two samples per site for each sampling cycle.

- C. Samples of avian species will be taken by shooting within the confines of the treated area at least two species commonly present in the fields throughout the sampling period. If possible they will represent those commonly having a food preference of insects and seed particles. Therefore the sampling effort will be conducted for 1 hour in the early morning, noon and early evening on two separate days, during the sample period. Five samples per site per sampling cycle is desired. It is entirely possible that no specimen will be obtained because of the absence of weeds and habitat in the area.
- D. Samples of small rodents, preferable field mice, will be taken by trapping within a field prior to treatment this season and at a minimum of five sampling cycles. Five samples per site per sampling cycle. Note: If any samples of birds or mammals are above 10 ppb then control samples will be taken on the next sampling round.

Sample Schedule

Marine Vertebrates

Sample	Round	Dates	3		
I		1-15	Octobe	r	1973
VI		1-15	Apri1	19	)74

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Terrigenous Areas - Soil, Birds, Mammals

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Sample Round	Dates
I	1-14 October 1973
II	15-21 October 1973
III	7-14 November 1973
IV	7 <b>-</b> 14 January 1974
V	14-28 February 197 <b>4</b>

Two additional rounds at 6 week intervals if required.

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#### Sample Material

1. Marine vertebrates taken by net, spear, or hook.

Aholehole(Kuklia sandnicensis)Kumu(Parupeneus porphyreus)Wrasse(Thalassoma deperreyi)

Each sampled separately for identification, but samples composited to obtain total mass required.

2. Avian species taken by shotgun.

Dove - Lace neck Barred	( <u>Streptopelia chinensis</u> ) ( <u>Geopelia striata</u> )				
Mynah	(Acridotheres tristis)				
Quail - California Valley	(Lophortyx californica) doubtful				
Others - for example ring-n	Others - for example ring-necked pheasant or California linnet				
Each species will be kept separate and identified.					
Any birds found dead in the field and in good condition, not decomposed,					

will be taken for analysis.

3. Small mammals (rodents) taken by trapping.

Each species will be kept separate and identified. Any mammals found dead in the field and in good condition, not decomposed, will be taken for analysis.

#### Sample Identification

Labels will be affixed at the time of sampling providing date, time, site and nature of sample material including species identification and where pertinent site conditions at time of sampling.

#### Sample Handling

Sample material to be placed in pre-baked metal cans supplied by Agr. Biochemistry Department or placed in new aluminum foil. Samples to be kept chilled until delivered to the laboratory. Samples for shipment to Honolulu will be assembled and shipped by the Department of Agriculture staff on Maui. Delivery shall be as expeditious as possible.

#### Sample Preparation

#### Fish:

The fish are generally too small for separate analysis of vital organs therefore the viscera and viscera contents will be included in the samples. Birds and Mammals:

Liver, adipose tissues and brain tissues are to be analyzed separately for larger specimens. Generally, the mammals trapped will be a species of mouse and will be treated as whole sample. Smaller birds will not be dissected. Breast muscle tissues will be dissected from the larger birds and analyzed as pooled samples for each species. Back and thigh muscles will be dissected from larger rodents combined and analyzed as pooled samples for each species.

#### Analytic Methods

It is urged that Gulf Breeze Laboratory facilities will be employed. The University of Hawaii Agricultural Biochemistry Department will not be able to commit its resources to these analyses without outside financial aid. Confirmation of presumptive positives by Gulf Breeze will be expected if analyses are completed in Hawaii.

#### <u>Usage</u>

Mirex useage in fields in drainage basins tributary to sampling sites will be verified by inspection of field logs.

#### Reviews and Reports

Preliminary reports of the results of each round of sampling, including local analyses, will be submitted to the EPA Honolulu office within 6 weeks of the end of each sampling round, or within 2 weeks of receipt of the Gulf Breeze analyses, whichever is later. If pertinent, recommendations will be made for changes in the procedure outlined above. A final report will be submitted within 3 months of the final sampling round or within 1 month of receipt of final Gulf Breeze analyses, whichever is later.

#### Responsibilities

Department of Health, Vector Control Branch (tentative):

Small mammals, sample identification and sample submittal Department of Land and Natural Resources, Wildlife Branch:

Terrigenous birds, sample identification and sample submittal Gulf Breeze Laboratory:

> Analysis of samples. If Gulf Breeze is unable to provide this service, the U.H. Agricultural Biochemistry Laboratory will do the analyses, assuming financial compensation is provided.

State Department of Agriculture:

Sample assembly, shipment and delivery to analytical laboratory. Sample preparation, local analyses and sample split holding, should check analyses be required. The Department will confirm log of Mirex usage in tributary areas.

U.H. Environmental Center:

Overall coordination preparation and review of reports U.H. Water Resources Research Center:

Aquatic vertebrate sampling and soil sampling, sample identification and sample submittal

## ATTACHMENT 3

- A. Mirex Active Ingredient Concentration in Pineapple Fields
  - 1. Application Rate: 2.5 lb. bait/acre
  - 2. 2.5 lb/acre X 1/43560 sq.ft./acre = 5.7 X  $10^{-5}$  lb/sq.ft.
  - 3. 5.7 X  $10^{-5}$  lb/sq.ft. X 456.3 gm/lb. = 2.6 X  $10^{-2}$  gm/sq.ft.
  - 4. 2.6 X  $10^{-2}$  gm/sq.ft. X 1/929 cm<sup>2</sup>/sq.ft. = 2.8 X  $10^{-5}$  gm/cm<sup>2</sup>.
  - 5. 2.8 X  $10^{-5}$  gm/cm<sup>2</sup> = 28. µg bait/cm<sup>2</sup>.
  - 6. 28.  $\mu$ g bait/cm<sup>2</sup> X 2.9 X 10<sup>-3</sup> gm active ingredient/gm bait = 8.2 X 10<sup>-2</sup>  $\mu$ g active ingredient/cm<sup>2</sup> field soil.

B. Average Mirex Residue in the Two Soil Sampling Sites

- 1. Ave. Mirex residual of the two sites = 3.9 ppb = 3.9 X  $10^{-3} \mu g/g$ .
- 2. Ave. bulk density of Haiku Clay = 1.1 g/cm<sup>3</sup>.  $^{6}$
- 3. Ave. % solids = 88.13.
- 4. Ave. Mirex residual (wet weight basis) = 3.9 X  $10^{-3} \mu g/g X$  .8813 = 3.44 X  $10^{-3} \mu g/g$
- 5. 3.44 X 10<sup>-3</sup> μg Mirex residual/g sample soil X 1.1g/cm<sup>3</sup> = 3.77 X 10<sup>-3</sup> μg Mirex residual/cm<sup>3</sup> sample soil.
- 6. Assuming sample depth was 1 cm, as per monitoring plan, and all residual was fresh Mirex applied during the 1973-74 application period, the residual would be 3.7 X  $10^{-3} \mu g/g$  Mirex residual/cm<sup>2</sup> of surface area sampled.
- 7. Ratio Mirex residual/active ingredient applied to field =  $\frac{3.7 \times 10^{-3} \mu g/cm^2}{8.2 \times 10^{-2} \mu g/cm^2} \times 100 = 4.5\%$

<sup>&</sup>lt;sup>6</sup>Soil Survey of Islands of Kauai, Oahu, Maui, Molokai, and Lanai, State of Hawaii, 1972.

## ATTACHMENT 3

- C. Average Mirex in Field 233 Soil Sampling Site
  - 1. Ave. Mirex residual in Field 233 Sampling Site = 1.4 ppb.
  - 2. Ave. bulk density of Haiku Clay = 1.1  $gm/cm^3$
  - 3. Ave. % solids = 88.4
  - 4. Ave. Mirex residual (wet weight basis) = 1.4 X  $10^{-3} \mu g/g X$  .884 = 1.24 X  $10^{-3} \mu g/g$ .
  - 5. 1.24 X  $10^{-3} \mu g$  Mirex residual/gm sample soil (wet wt.) X 1.1 g/cm<sup>3</sup> = 1.36 X  $10^{-3} \mu g$  Mirex residual/cm<sup>3</sup> soil = 1.36 X  $10^{-3} \mu g$  Mirex residual/ cm<sup>2</sup> surface area samples.
  - 6. Ratio Mirex residual/active ingredient applied to field =  $\frac{1.36 \times 10^{-3}}{8.2 \times 10^{-2}} \times 100 = 1.7\%$
- D. Average Mirex in Field 234 Soil Sampling Site
  - 1. Ave. Mirex residual in Field 234 Sampling Site = 6.4 ppb.
  - 2. Ave. % Solids = 88.1.
  - 3. Ave. Mirex residual (wet weight basis) = 6.4 X  $10^{-3} \mu g/g X$  .881 = 5.6 X  $10^{-3} \mu g/g$ .
  - 4. 5.6 X  $10^{-3} \mu g/g X 1.1 g/cm^3 = 6.2 X <math>10^{-3} \mu g/cm^3$
  - 5. Ratio Mirex residual/active ingredient applied to field = 7.6%.

A. t-test applied to Polynesian rat samples

1. 
$$\zeta = \frac{\sqrt{\Sigma(x_1 - \overline{x}_1)^2 + \Sigma(x_2 - \overline{x}_2)^2}}{n_1 + n_2 - 2}$$

2. 
$$t = \frac{\bar{x}_1 - \bar{x}_2}{\zeta} \int \frac{n_1 n_2}{n_1 + n_2}$$

- Data Set 1: Cycle 2, 3, and 4 Samples
   Data Set 2: Cycle 5, 6, and 7 Samples
- 4.  $\bar{x}_1 = 2996$ ,  $n_1 = 8$ 
  - $\overline{x}_2 = 238, n_2 = 9$
- 5. ζ = 2533
- 6. t = 2.24

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- 7. 0.05 < P < 0.02
- B. t-test applied to roof rat samples
  - Data Set 1: Cycle 2-4 Samples
     Data Set 2: Cycle 5-7 Samples
  - 2.  $\bar{x}_1 = 897, n_1 = 10$  $\bar{x}_2 = 163, n_2 = 12$
  - 3. <sub>ζ</sub> = 399.2
  - 4. t = 4.29
  - 5. 0.001 < P

## ATTACHMENT 4

- C. t-test applied to Small Indian mongoose samples
  - Data Set 1: Cycles 3-5 samples
     Data Set 2: Cycles 6-7 samples
  - 2.  $\overline{x}_1 = 4220, n_1 = 12$  $\overline{x}_2 = 338, n_2 = 10$
  - 3. ζ = 2776
  - 4. t = 3.27
  - 5. 0.01 < P < 0.001