ENVIRONMENTAL PROTECTION AGENCY

OFFICE OF ENFORCEMENT

A Pesticide Residue Study

Wareham, Massachusetts

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER

DENVER, COLORADO

MARCH 1978

ENVIRONMENTAL PROTECTION AGENCY
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A PESTICIDE RESIDUE STUDY

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National Enforcement Investigations Center
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I. INTRODUCTION

Cranberries, as a commercial crop, are raised in a relatively few adaptable areas in North America. The crop is grown almost exclusively in man-made or natural bogs. The few states with extensive acreage of bogs suitable for commercial cranberry crops include Massachusetts, New Jersey, Oregon, Washington, and Wisconsin; less extensive acreages are cultivated in Connecticut and Rhode Island.[1] Of these areas, the bogs of Massachusetts serve as the major cranberry growing area in the United States. They are estimated to produce half the world supply of cranberries with a wholesale value approaching $60 million.[2]

Cranberries, like many other fruit crops, are plagued by infestations of insect pests throughout the growing season. However, the intensive use of insecticides over the past few decades is considered one of the main factors that have raised cranberry yields per acre to their present levels.

During the course of a growing season, a cranberry bog may be plagued by almost a dozen different species of insects which attack the plant from its root system to the growing berry. Some of the insecticides used in the past, and still used in integrated control methods, are carbyl, parathion, Guthion and diazinon. Parathion has been used on many Massachusetts bogs for more than 15 years. Presently, Parathion is applied 2 to 3 times on a bog during a growing season at the volume rate of 0.47 l/0.4 ha (1 pt/acre).

Flooding of a cranberry bog is a practice used to prevent damage from various elements. In the winter it is used to prevent desiccation -- a condition called winter-kill, in summer to relieve drought conditions and under certain conditions is used to control infestation of insect pests without resorting to insecticides. Nevertheless, copious amounts of water are used on each occasion. Estimates place the volume at 300,000 gallons of water per acre.[3]

These large volumes of water may be contaminated with pesticides previously applied on the cranberry crop. Since the waters eventually discharge into streams or estuarine systems, the potential for translocating pesticides is likely to occur. As a result, there is a likelihood of harm to the environment and a possible health hazard to the public that consume potentially contaminated fish or shellfish.

Evidence of pesticide contamination was found in Buzzards Bay, Massachusetts in May, 1974. Shellfish (*Mya arenaria*) collected in this estuarine system contained ethyl parathion concentrations ranging from 0.01 to 0.03 mg/g (ppm).[4] Reportedly, this contamination was caused by drainage from nearby pesticide treated cranberry bogs.

With this background, EPA Region I requested the National Enforcement Investigations Center (NEIC) to conduct a study in the most productive cranberry growing area in southeastern Massachusetts with the following objectives: determine if pesticide residues were present in cranberry bogs following a season of treatment with insecticides, and determine the extent and effect, if any, of pesticide translocation from cranberry bogs to adjacent wetlands and receiving waters.

The study site selected by EPA Region I and the University of Massachusetts Cranberry Experiment Station was located near Wareham, Massachusetts, and is crossed by Pierceville Stream. Waters from this stream join the east branch of the Sippican River and eventually discharge into Buzzards Bay [Figure 1].

A five-day study began on October 11, 1977. Samples of soil, sediment, water, and fish were collected from the bogs as well as shellfish and sediments from a nearby estuary. The chemical analyses on these samples for selected pesticides residue were performed at the NEIC laboratories in Denver, Colorado.

A second investigation will be conducted prior to and during the first pesticide application in the spring of 1978. This study will be designed to determine if the field application of pesticides on cranberry bogs are performed consistent with registered labeling, FIFRA regulations and appropriate State codes.

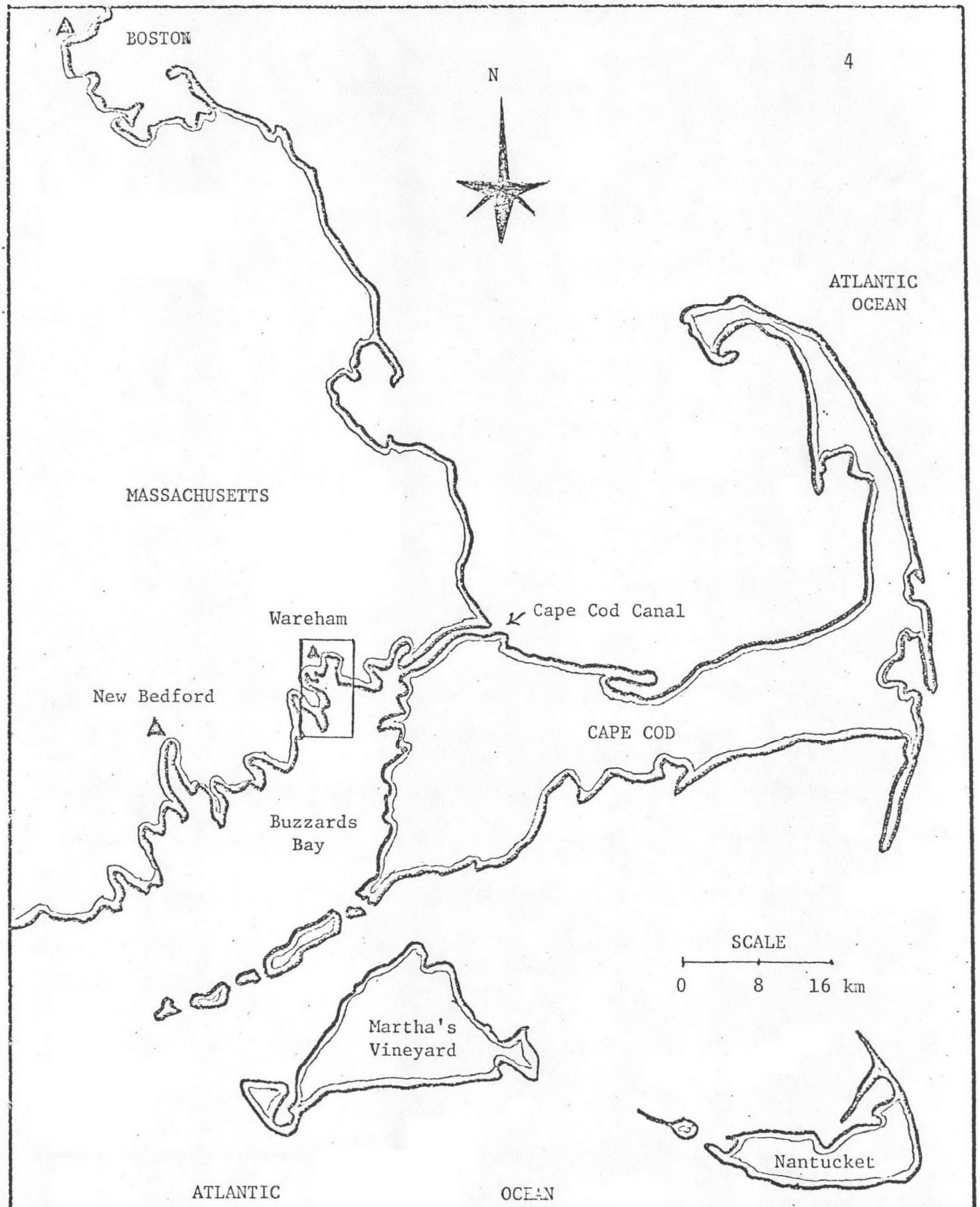

Figure 1 Cranberry Bog Study Site in Wareham, Massachusetts

II. SUMMARY AND CONCLUSIONS

During the 1977 cranberry growing season in Wareham, Massachusetts, parathion was applied on the Tremont Bog on three occasions to control insect infestations. The pesticide applications were made through a sprinkler system at the rate of 0.47 liters/hectare (1 pt/acre).

Approximately three months after the last parathion application, the NEIC conducted a field study at Tremont Bog. The 5-day study (October 11-15, 1977) was conducted before and during the cranberry harvest to determine if a parathion residue existed in the bog or if the pesticide had been translocated from the treated bog to adjacent wetlands and receiving waters.

Soil from the bog contained ethyl parathion in concentrations ranging from 0.6 to 5.6 mg/g (ppm). Water that flooded the bog during the cranberry harvest and drained into Pierceville Stream contained no measurable amounts of pesticides. Examination of sediments and fish or shellfish from the drainage ditch (Pierceville Stream) and downstream estuaries (Weweantic River and Buzzards Bay) showed no pesticide accumulations.

Based upon the sampling and analyses described in this report, it was concluded that harvest flooding did not translocate measurable amounts of parathion from the soil of Tremont Bog into receiving waters or nearby wetlands. Furthermore, if pesticide-contaminated water drained from treated cranberry bogs into Pierceville Stream or into downstream estuaries prior to the EPA study, the chemicals apparently have been diluted or dissipated sufficiently and environmental harm has not occurred.

III. DESCRIPTION OF THE STUDY

The cranberry bog selected for study is identified by the grower as the north piece of the Tremont Bog located in Wareham, Massachusetts [Figures 1 and 2]. This particular piece consisted of 9.75 hectares (19.7 acres) which was flooded during a portion of the EPA study. The Pierceville Stream flows through the middle of the bog and joins the east branch of the Sippican River, which eventually discharges into Buzzards Bay south of the Cape Cod Canal [Figures 1 and 4]. Water for flooding, sprinkler irrigation, and pesticide applications through the sprinkler system is obtained from Pierceville Stream.

The pre-harvest investigation was designed to obtain background information prior to harvesting operations which involved flooding of the upper Tremont Bog. To determine possible insecticide contamination from upstream bogs, preliminary sampling of water and sediments were obtained from the reservoir upstream of the bog (Stations 07, 06). To assess the presence of insecticide residues in the study site, water samples were taken from the Pierceville Stream and soil samples from the adjacent exposed bog (Stations 04, 05, 08). Downstream at the lower extremity of the Tremont Bog, samples of water and sediment were taken from the Pierceville Stream to determine the likelihood of pesticide translocation from the entire bog [Figure 2].

At the time of cranberry harvesting, a resampling of the study site was conducted. At the reservoir, designated as the control area, sediment and water samples were taken (Station 07, 04) to determine if pesticide residual influx from upstream harvesting operations had taken

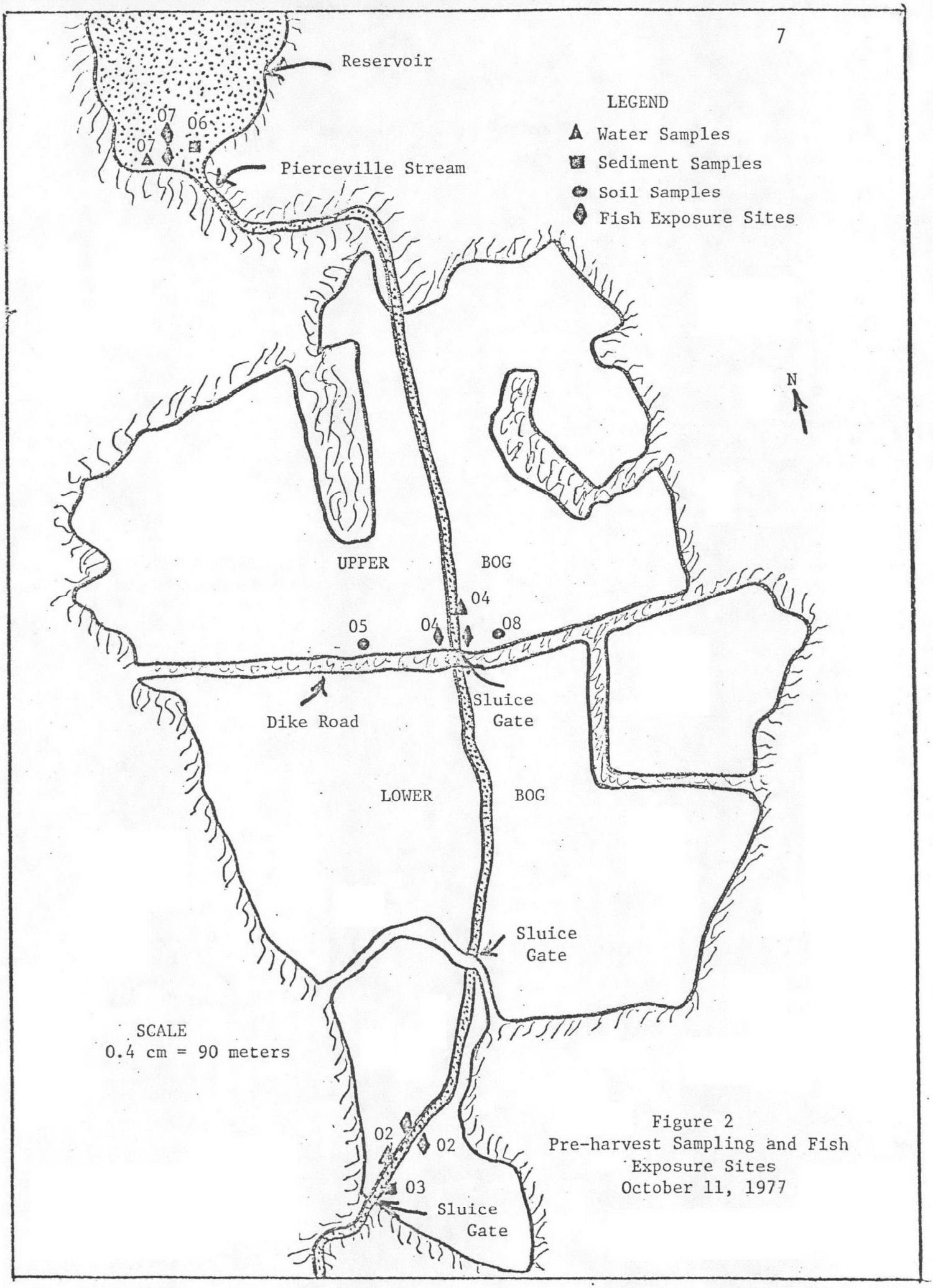

Figure 2
Pre-harvest Sampling and Fish
Exposure Sites
October 11, 1977

place. During the same period soil samples from the flooded bog were taken in close proximity to those obtained during the pre-harvest investigation. Samples of sediment and water (Stations 02, 03) were also taken in Pierceville Stream at the southern end of the Tremont Bog to assess possible runoff of insecticide contaminated water from the flooded upper bog [Figure 3].

Rainbow trout, Salmo gairdneri, were held in submerged cages in the storage reservoir (Station 07) upstream of the test cranberry bog in Pierceville Stream upstream of the first sluice gate (Station 04), and at the third sluice gate in the lower bog (Station 02) to monitor acute physiological effects of pesticide exposure [Figure 2].

To determine the chronic effects of possible pesticide translocation from cranberry bogs to adjacent wetlands and receiving waters, samples of shellfish and sediments were obtained from the estuarine area of the Weweantic River and inner Buzzards Bay [Figure 4]. A summary of the sampling effort is contained in Table 1.

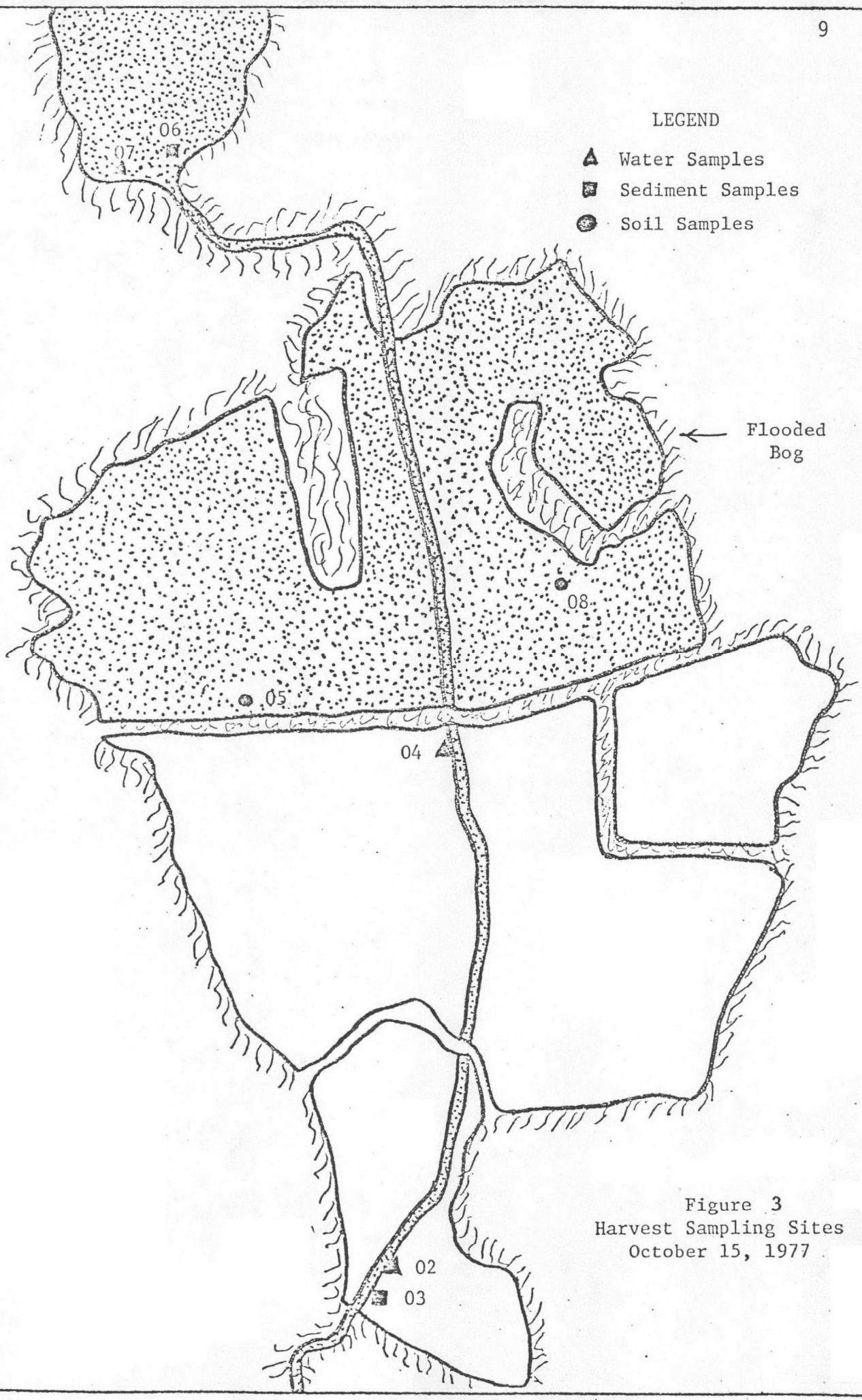

Figure 3
Harvest Sampling Sites
October 15, 1977

Table 1

SAMPLES COLLECTED FROM THE WAREHAM, MASSACHUSETTS STUDY AREA October 1977

Tremont Bog - Before Harvest - 10/11/77

Station No.	Water	Soil	Sediment
00	X+		
02	X		
04	X		
07	X		
05		X	
08		X	
03			X
06			X

Tremont Bog - During Harvest - 10/15/77

Station No.	Water	Soil	Sediment	Fish
02	X			
04	X			
07	X			
05		X		
08		X		
03			X	
06			X	
02				X
04				X
07				X

Table 1 (Cont.)

SAMPLES COLLECTED FROM THE WAREHAM, MASSACHUSETTS STUDY AREA October 1977

Weweantic River and Buzzards Bay

Station No.	Sediment	Shellfish
09		X
10		X
11	X	
12	X	
13	X	
14	X	
15	X	
16	X	
17	X	
18	X	

+From the Massachusetts Department of Fish & Wildlife Fish Hatchery

IV. DISCUSSION AND RESULTS

During the course of the 1977 cranberry growing season, several applications of chemicals were introduced into the Tremont Bog [Table 2]. In late June, when weeds were apparently hindering the flow of water in the drainage ditches, kerosene was used as a herbicide to retard further growth. Throughout the remainder of the season three applications of parathion were made at the rate of 0.47 l/ha (1 pt/acre). Carmol, which is a blend of carbyl and molasses, was applied with parathion via the sprinkler system in July and acted as a holding agent for the pesticides. When insect infestation was apparent during June and July, a total of 13.74 liters of parathion was sprayed on the 9.75 ha of the Tremont Bog.

PRE-HARVEST ASSESSMENT

Prior to harvesting operations a series of water, sediment and soil samples were collected from Tremont Bog to assess the background levels of pesticides before the bog ecosystem was disturbed by the mechanical processes used during wet harvesting. Pre-harvest sampling was completed on October 11, 1977.

All sampling stations were located at strategic points on and in the vicinity of the bog to assess the background levels of malathion*, methyl parathion, and ethyl parathion.

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* *Analyses for malathion were done because of possible application to bogs upstream of the Tremont Bog.*

Table 2

CHEMICAL APPLICATIONS
APPLIED TO TREMONT BOG, 1977

Date	Chemical	Rate	Area (ha)	Method	Total
5/25/77	Kerosene	304 1/0.4 ha	.016	Hand Spray	4.86 1
6/3/77	Parathion-Carmol*	0.47 1/0.4 ha	9.75	Sprinklers	4.58 1
		1.90 1/0.4 ha	9.75	Sprinklers	18.5 1
7/14/77	Parathion	0.47 1/0.4 ha	9.75	Sprinklers	4.58 1
7/27/77	Parathion	0.47 1/0.4 ha	9.75	Sprinklers	4.58 1
				Total Parathion	13.74 1

*Blend of Carbyl and molasses

Analysis of the water sample taken from the reservoir (Station 07) contained non-detectable levels of pesticides which indicated no apparent influx of pesticides from bogs upstream of the reservoir. A sample of water taken from Pierceville Stream (Station 04) as it flowed through Tremont Bog also proved to contain non-measurable amounts of pesticide, indicating that no leaching of water containing pesticides was taking place on the upper bog. Water obtained at Station 02, located at the southernmost point of the bog, also proved negative. It showed that runoff from the lower section of Tremont Bog contained no measureable amounts of pesticides.

Sediment samples were obtained from two locations on the study site during this period. Sediment from Station 06 in the reservoir contained non-detectable levels of pesticides indicating no translocation of pesticides was taking place from bog locations upstream. Sediment from Pierceville Stream at the lower end of Tremont Bog (Station 03) also contained non-detectable amounts of pesticides. This fact indicated that there was no apparent pesticide influence from Tremont Bog.

Two soil samples (Stations 05, 08) taken from the unflooded growing area of the upper half of the Tremont Bog contained an ethyl parathion concentration of 5.6 and 1.8 mg/g (ppm) respectively. According to Deubert and Gray the persistence of parathion (ethyl-parathion) in soil is not surprising in view of the findings that this chemical forms strong complexes with organic matter and clay.[4] The presence of this concentration of ethyl parathion bound to the soil of the cranberry bog had no influence on the water or sediments of Pierceville Stream.

DURING-HARVEST ASSESSMENT

On October 13, flooding of the cranberry bog was accomplished and harvesting begun. On October 15, when three-quarters completed, harvesting had to be suspended because of weather forecasts predicting storms accompanied by torrential rain. Wet harvesting can ordinarily be continued in a light rain, but concern of over-flooding by heavy rain and the subsequent loss of floating cranberries cause a suspension of harvest activities.* Another practice in the wet harvesting of cranberries is the conservation of water. Thus, when one bog, or section of bog, has been harvested the water is passed down to a lower bog to repeat the process. When all bogs capable of re-using the same water have been harvested, only then is the majority of water allowed to discharge into a stream or river.

Because of the impending storm and the lengthy operation involved in completing the entire harvest on the Tremont bog, a decision was made to terminate the pesticide residue investigation. It appeared that enough flood water was escaping through the sluice gate to sufficiently bathe the caged fish and lower sampling area. On October 15, the pre-harvest sampling routine was repeated and the fish retrieved for AChE and pesticide analyses.

Analysis of the water sample taken from the reservoir (Station 07) revealed no detectable traces of pesticides from the increased flow of water from the upstream bogs. The Station 04 water sample obtained at the sluice gate of the upper bog also showed no presence of a detectable concentration of pesticide which indicated little or no dissociation of pesticides from the cranberry bog soil. At the lower extremity of the

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This is method involving a machine called a "water-reel" or "egg-beater" which knocks the berries off of the vines and permits them to float to the surface of the flooded bog. The berries are then loaded into trucks by conveyor.

bog, water from Pierceville Stream at Station 02 proved also to contain non-detectable amounts of pesticide, suggesting that no leaching was taking place from the lower bog soil.

Two sediment samples were obtained at the study site during harvesting. Sediment from Station 06 in the reservoir contained non-detectable levels of pesticides, suggesting no translocation had taken place from upstream bogs. The Pierceville stream sediment taken at the lower end of Tremont Bog (Station 03) also proved to contain non-detectable amounts of pesticides, thus indicating no apparent effects from the lower Tremont Bog.

Two soil samples were obtained from the flooded portion of the upper Tremont Bog (Stations 05, 08). These samples of soil were from the same general area as pre-harvest soil samples. Again, both soil samples contained ethyl parathion in concentrations of 0.6 (Station 05) and 1.4 (Station 08). Since no detectable amounts of pesticide were found in water samples in close proximity to these soil stations, it is assumed that leaching was not taking place from the soil to the water.

FISH PHYSIOLOGICAL ASSESSMENT

Duplicate sets of ten rainbow trout, *Salmo gairdneri*, were exposed in submerged cages at three locations on the cranberry bog test site from October 11 to October 15 [Figure 2]. One set of fish from each site was analyzed for acetylcholinesterase (AChE) to determine possible enzyme inhibition related to organophosphate poisoning through contact with water borne pesticides. The remaining sets of fish were returned in a frozen condition to the NEIC laboratories and analyzed for pesticide uptake in the livers.

Field analytical results, on one set of fish from the three test locations, showed that the range in the acetylcholinesterase activity of the fish brain of exposed fish was similar to the activity in the brain of fish from a local hatchery (control fish). No inhibition, or indication of pesticide poisoning in the vicinity of the Tremont Bog could be shown.

This was further substantiated when tests on the livers of the duplicate set of fish were analyzed. No detectable concentrations of pesticides were found in any of the three groups of fish exposed in the vicinity or on the Tremont cranberry bog (Table 3).

ESTUARINE ASSESSMENT

A field study was conducted on October 13 to determine the chronic effects of possible pesticide translocation from cranberry bogs to adjacent wetlands and receiving waters.

Waters used to irrigate the Tremont Bog drain to Weweantic estuary and eventually to Buzzards Bay. If pesticide contamination of drainage waters occurs, the possibility exists that accumulation of pesticides is occurring in the waters, sediment and biota of these estuarine systems. Although most organophosphate pesticides are readily hydrolized, some have been shown to be very persistent; for example, methyl parathion can remain active in water for nearly two years, and in soils for twenty years[5,6].

During the field study three species of commercial shellfish, *Mya arenaria*, *Mercenaria mercenaria*, and *Crassostrea virginica*, were dug from two separate mud flats (Station 09, 10) on the Weweantic River (Figure 4). On the same day, several sediment samples (Stations 11-18) were collected in this river and in Buzzards Bay. Analyses of these

TABLE 3
Analytical Results
Malathion, Methyl and Ethyl Parathion

Station Number	Location	Type Sample	Residue Malathion	Methyl Parathion	Ethyl Parathion
Before Harvest	10/11/77				
00	Hatchery	Water	N.D.++	N.D.	N.D.
02	Lower bog	Water	N.D.	N.D.	N.D.
04	Upper bog	Water	N.D.	N.D.	N.D.
07	Reservoir	Water	N.D.	N.D.	N.D.
05	Upper bog	Soil	N.D.	N.D.	5.6
08	Upper bog	Soil	N.D.	N.D.	1.8
03	Lower bog	Sediment	N.D.	N.D.	N.D.
06	Reservoir	Sediment	N.D.	N.D.	N.D.
During Harvest	10/15/77				
02	Lower bog	Water	N.D.	N.D.	N.D.
04	Upper bog	Water	N.D.	N.D.	N.D.
07	Reservoir	Water	N.D.	N.D.	N.D.
05	Upper bog	Soil	N.D.	N.D.	0.6
08	Upper bog	Soil	N.D.	N.D.	1.4
03	Lower bog	Sediment	N.D.	N.D.	N.D.
06	Reservoir	Sediment	N.D.	N.D.	N.D.
02	Lower bog	Fish*	N.D.	N.D.	N.D.
04	Upper bog	Fish	N.D.	N.D.	N.D.
07	Reservoir	Fish	N.D.	N.D.	N.D.

TABLE 3 (Cont'd.)

Weweantic River and Buzzards Bay

Station Number	Location	Type Sample	Residue Malathion	Methyl Parathion	Ethyl Parathion
Weweantic River and Buzzards Bay					
09	Weweantic River	Shellfish**	a. N.D.	N.D.	N.D.
			b. N.D.	N.D.	N.D.
			c. N.D.	N.D.	N.D.
10	Weweantic River	Shellfish	a. N.D.	N.D.	N.D.
			b. N.D.	N.D.	N.D.
			c. N.D.	N.D.	N.D.
11	Weweantic River	Sediment	N.D.	N.D.	N.D.
12	Weweantic River	Sediment	N.D.	N.D.	N.D.
13	Buzzards Bay	Sediment	N.D.	N.D.	N.D.
14	Buzzards Bay	Sediment	N.D.	N.D.	N.D.
15	Buzzards Bay	Sediment	N.D.	N.D.	N.D.
16	Buzzards Bay	Sediment	N.D.	N.D.	N.D.
17	Buzzards Bay	Sediment	N.D.	N.D.	N.D.
18	Buzzards Bay	Sediment	N.D.	N.D.	N.D.

* Fish - Rainbow Trout - *Salmo gairdneri*

** Shellfish - a. Soft clam - *Mya arenaria*
b. Hard clam - *Mercenaria mercenaria*
c. Eastern Oyster - *Crassostrea virginica*

\+ Values in water expressed as mg/l; in soil, fish, shellfish and sediment as mg/g

++ None detected.

Water samples: The detection limit for both ethyl and methyl parathion was 0.1 ug/l (ppb).
The detection limit for malathion was 0.5 ug/l (ppb).

Tissue results reported on basis of wet weight; sediment samples reported on basis of dry weight.

samples were done at NEIC laboratories in Denver, Colorado using EPA standard methods [Appendix A]. The samples did not contain detectable concentrations of the type of pesticides used on cranberry bogs [Table 3]. Based upon the sampling and analyses described in this report, it was concluded that harvest flooding did not translocate measurable amounts of parathion. Furthermore, if pesticide-contaminated water drained from treated cranberry bogs in Pierceville Stream or the down stream estuaries prior to the EPA study, the chemicals apparently have been diluted or dissipated sufficiently and environmental harm did not occur.

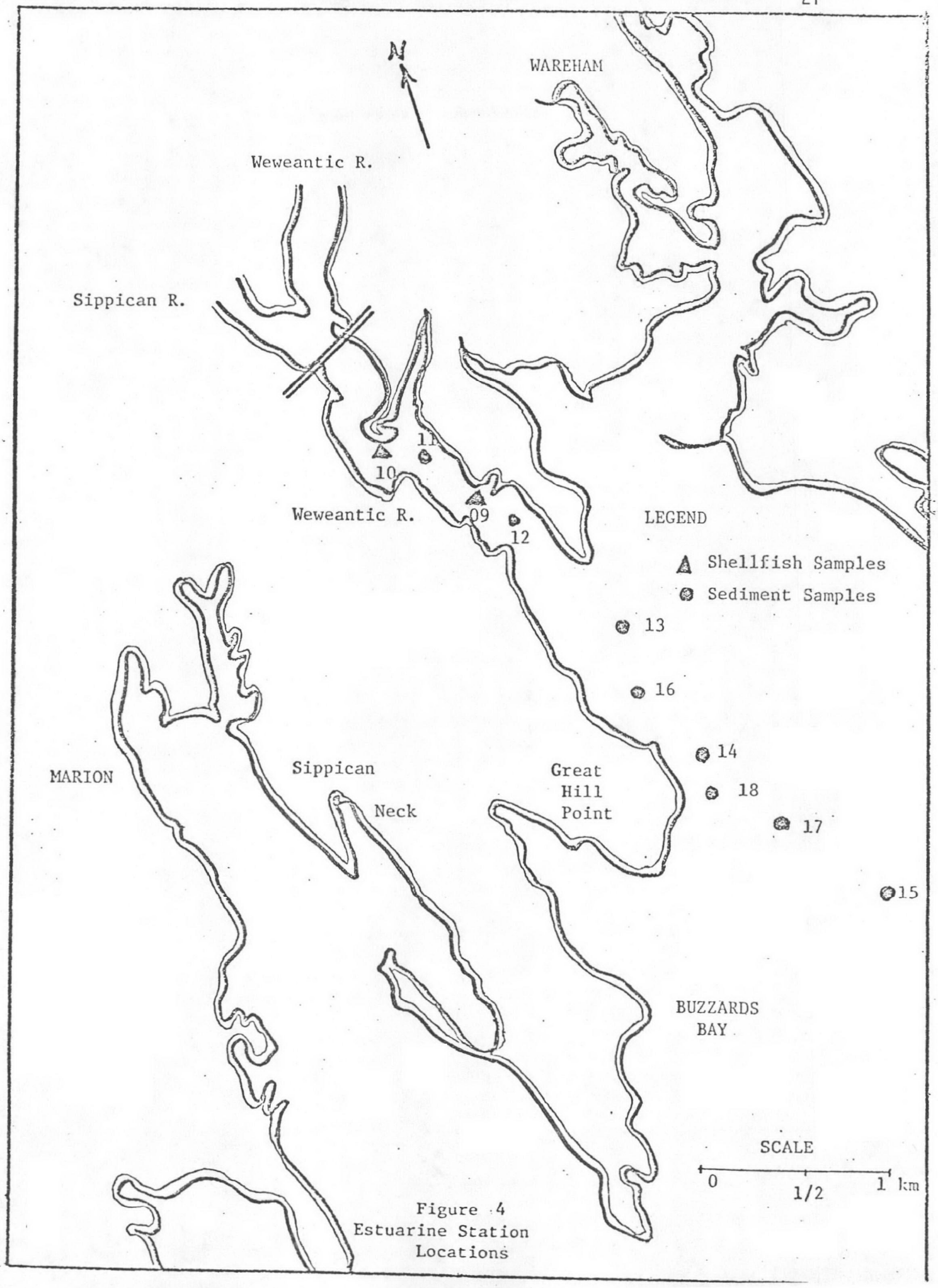

Figure 4
Estuarine Station
Locations

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APPENDIX A

ANALYTICAL METHODS

ANALYTICAL METHODS

METHODS

A. Acetylcholinesterase Inhibition Tests. Evaluation of AChE inhibition in rainbow trout (*Salmo gairdneri*) was accomplished by exposing ten fish for four day intervals at each of three stations in the reservoir, stream and pool in the vicinity of the study bog. Fish used in the study were obtained from the Massachusetts Department of Fish and Wildlife Hatchery at Sandwich and ranged in size from 10-16 cm total length.

After the exposure period, the fish were removed from the cages and transported live to the field laboratory facility where the brains were removed for AChE analysis.

The principal equipment used for AChE analyses is a recording pH-stat. Briefly, the procedure was: brains of 4-5 fish from the same exposure site were pooled, wet-weighed, and homogenized in distilled water. The brei was diluted with distilled water to a final tissue concentration of 5 mg/ml. Two ml of diluted homogenate was then mixed with 2 ml of acetylcholine iodide (a specific substrate for the enzyme AChE). The pH-stat was used to titrate the acetic acid end-product of the enzyme substrate reaction with 0.01M NaOH. The test was performed at pH 7.0 and 22°C with a nitrogen purge over the surface of the reaction vessel to prevent absorption of atmospheric carbon dioxide.

Micromiles of acetylcholine hydrolized was calculated from the micromiles of NaOH required to neutralize the free acetic acid. The AChE activity was expressed as micromiles of acetylcholine hydrolized per hour mg of brain tissue.

B. Fish and Shellfish Analysis. Fish samples were prepared for pesticide analysis in the following manner. The liver from each of the rainbow trout was removed and frozen. The frozen liver was then chopped into small pieces and extracted. The shellfish were prepared by removing the viscera from each animal, weighing the viscera into 10 gram samples, and freezing the viscera. The frozen viscera was then chopped into small pieces and extracted.

The extraction of all fish samples was done in an explosion-proof blender using 200 ml of 15% methylene chloride in cyclohexane. The samples were blended for 1-1/2 - 2 minutes. The extract was then filtered through Whatman #1 filter paper, dried with Na_2SO_4 and concentrated to 10 ml in an evaporative concentrator.

Three of the samples were cleaned to eliminate interference peeks using the standard EPA acetonitrile clean-up[7]. The remaining samples were processed using a Gel Permeation Unit packed with SX-3 resin and eluted with 15% methylene chloride in cyclohexane. The samples were analyzed on a gas chromatograph equipped with an alkali flame ionization detector.

In addition, one of the samples was processed further using a 2 x 15 cm neutral alumina column, deactivated with 3% H_2O and eluted with three 50 ml portions of benzene.

C. Sediment Analysis. Sediment samples were seived through a 2 mm mesh sieve. The percent moisture in each sample was determined by drying 10 gm of sample overnight in a 120°C oven and determining weight loss.

The samples were extracted using the USGS method for soil analysis[8] Briefly, this method involves two serial extractions with a combination of acetone and hexane using a wrist action shaker. The extract is filtered through Whatman#1 filter paper, back extracted with water, and concentrated to 10 ml in an evaporative concentrator. The extracts were then analyzed on a gas chromatograph equipped with an alkali flame ionization detector.

The presence of ethyl parathion was confirmed in two of the samples using a GC/MS. However, the other two samples suspected of containing ethyl parathion possessed too many interference peeks to analyze by GC/MS. These samples were processed using the neutral alumina column cleaning method described previously. Ethyl parathion in these two samples was identified based on gas chromatographic retention time and alumina column elution pattern.

Quality Control

A. Water Analysis. A series of tests were performed to determine the recovery and holding time for ethyl and methyl parathions in water. One liter samples of tap water were spiked with 2 ug of ethyl parathion and 2 ug of methyl parathion. The pH of the water had been adjusted to 4 before spiking. Two of the spikes were analyzed immediately using the method previously described. The others were held in a refrigerator until analyzed.

Results:

		% Recovery	
	Date Analyzed	Methyl Parathion	Ethyl Parathion
Spike 1	10/21/77**	101	99
Spike 2	10/21/77**	95	93
Spike 3	10/26/77*	93	86
Spike 4	10/31/77**	97	97

B. Sediment. A 50 gm sample from Station 06 was spiked with 20 ug each of ethyl parathion, methyl parathion and malathion. The sample was analyzed according to the procedure previously described for sediment. A solvent blank was also analyzed.

Results:

	% Recovery		
	Malathion	Methyl Parathion	Ethyl Parathion
Spike	114	129	88
Blank	---	---	--

C. Fish. Two different types of shellfish viscera were spiked with 0.5 ug of each of the following pesticides: malathion, ethyl parathion, methyl parathion. The samples were then analyzed for the method previously described.

Results: Hard clams, Station 09

% Recovery		
Malathion	Methyl Parathion	Ethyl Parathion
76	84	66

Results: Soft clams, Station 10

% Recovery		
Malathion	Methyl Parathion	Ethyl Parathion
38	47	40

* pH not adjusted to 7 before extraction
** pH adjusted to 7 before extraction

APPENDIX B

REVIEW OF CHAIN-OF-CUSTODY PROCEDURES

REVIEW OF CHAIN-OF-CUSTODY PROCEDURES

Cranberry Bog Study
Wareham, Massachusetts

Records regarding the Cranberry Bog Study were evaluated against the established NEIC chain-of-custody procedures. There was no reconnaissance of the study site made by NEIC personnel. Field data records, log books, sample tags, and chain-of-custody records were reviewed to determine if there were any deviations from the procedures. Where deviations were discovered, an assessment was made as to what effect, if any, this had on survey results. The result of this evaluation is described in the following paragraphs.

The AChE analyst on the survey did not maintain a laboratory notebook. Instead, the data for each fish brain analysis was recorded on the recording pH stat graph paper and not re-recorded into a bound notebook. Between each analysis standard calibrations of the AChE equipment was conducted, but not recorded.

Although daily calibrations for accuracy were made on the Mettler H45 analytical balance, these were not recorded in a laboratory notebook.

These above described deviations are considered minor and had no impact on the results and conclusions contained in this report.