Evaluation of Efforts to Reduce Pesticide Contamination in Cranberry Bog Drainage

Abstract

During June and July of 1998, the Washington State Department of Ecology collected water samples at three sites in surface drainage ditches from cranberry growing areas near Grayland, Washington. Two locations were test sites, and the third was a control site. Water samples were analyzed for organophosphorus pesticides and general water quality parameters. Laboratory and *in situ* bioassays using *Daphnia pulex* tested the toxicity of the drainage water. These data were collected to evaluate ongoing efforts to reduce pesticide contamination discovered in cranberry bog drainage in 1994.

Three organophosphorus pesticides were detected in the water samples: chlorpyrifos, diazinon, and azinphos-methyl. All pesticide concentrations were lowest prior to pesticide application. The highest levels were measured after application.

All three pesticides detected exceeded recommended water quality criteria for most of the study period.

- Chlorpyrifos exceeded the state water quality criterion of 0.041 ug/L in 25% of the samples before pesticide application and 83% after application.
- Diazinon exceeded available water quality guidelines of 0.04 ug/L in 50% of the samples before application and 100% after application. Diazinon concentrations were the highest of the three detected pesticides.
- Azinphos-methyl exceeded the EPA water quality criterion of 0.01 ug/L throughout the study period.

When compared to data collected between 1994 and1996, the 1998 data reveal no reduction in overall pesticide levels, despite implementation of best management practices by a few of the cranberry growers.

Bioassay results show the potential exists for adverse biological effects in the drainage ditches after pesticide application occurs.

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Introduction

During June and July of 1998, the Washington State Department of Ecology (Ecology) collected water samples at three sites in surface drainage from cranberry growing areas near Grayland.

Water draining from cranberry bogs and residential property in the Grayland/North Cove area south of Westport is collected in a ditch system that discharges to the south into Willapa Bay and to the north into the south bay of Grays Harbor. Both the north and south ditches originate from a wetland area just south of the Grays Harbor/Pacific County line and west of Highway 105 in Grayland. The ditches parallel Highway 105, collecting runoff from about 900 acres of cranberry bogs. Both ditches also receive water (1) from small streams that run down from the hills east of the cranberry bogs, and (2) probably directly from shallow groundwater within the bogs (Davis et al., 1997). The north ditch is called Grays Harbor County Drainage Ditch No. 1 (GHCDD-1), and the south ditch is called Pacific County Drainage Ditch No. 1 (PCDD-1).

Drainage ditches have been designated by the state Attorney General (1969) as waters of the state. As such, these waters are subject to Washington State Water Quality Standards (Chapter 173-201A WAC).

Ecology collected water from GHCDD-1 in 1994 and 1995, and analyzed for 161 pesticides and breakdown products as part of Ecology's Washington State Pesticide Monitoring Program (WSPMP). High concentrations of numerous pesticides were detected in these samples, prompting a more intensive investigation of contamination from pesticide use on cranberries in the Grayland area.

Water samples were collected from GHCDD-1 and PCDD-1 throughout the 1996 growing season. All samples were found to contain one or more organophosphorus insecticides at concentrations above the state water quality standards (Davis et al., 1997). Many concentrations were above LC_{50} for some aquatic invertebrates, and were toxic to *Daphnia pulex* in laboratory and *in situ* bioassays (Wood, 1997).

The cranberry industry agrees that there is a significant pesticide contamination problem in water draining from cranberry bogs, and they have already developed some best management practices (BMPs) in an effort to reduce concentrations in drainage water. Field simulations in 1996, using a pesticide surrogate, indicated that some combinations of the BMPs were nearly 100% effective (Bicki et al., 1997). In addition, the industry is working to find less toxic chemicals to replace the pesticides they are currently using.

The primary objectives of this 1998 study were to:

- Determine if there had been any reduction of pesticide concentrations in cranberry bog drainage as a result of implementation of BMPs by the cranberry growers. This objective was evaluated by comparing concentrations of pesticides detected in 1998 to results from 1996.

- Verify the toxicity of insecticides used on cranberries by performing laboratory and *in situ* bioassays on drainage ditch water. The bioassay results were statistically compared to results from controls and a reference site. Comparison of the toxicity of the ditch water before and after pesticide applications was also evaluated.

Methods

Site Selection

In 1998 water samples were collected and *in situ* bioassays were conducted in the drainage ditches at the same three sites used in 1996 (Davis et al., 1997).

- The GHCDD-1 site is at the bridge on Schmid Road.
- The PCDD-1 site is at the bridge on Larkin Road.
- The reference site is a drainage ditch, north of the GHCDD-1 site, that does not receive drainage water from cranberry bogs.

Sampling locations are shown in Figure 1. A detailed list of station locations and descriptions is also included in Appendix A, Table A1.

Study Design

The focus of this study was to evaluate the effectiveness of BMPs in reducing pesticide concentrations in cranberry bog drainage. June and July were selected for sampling, since this is the most intensive application period of the growing season. Historically, this period has had the highest pesticide concentrations in ditch water (Davis et al., 1997).

Water samples were collected on five occasions to evaluate pre- and post-spray conditions. Samples were analyzed for organophosphorus pesticides only; these were the compounds most commonly detected above water quality criteria in previous years.

The pre-application bioassay and water sampling took place in late June. Honeybees were brought in for pollination in early June and remained in the bogs for most of the month. Most of the growers apply pesticides at the same time the bees are removed.

While the bees were present, no organophosphate insecticides were sprayed on the crops. Data from 1996 showed that insecticide concentrations were lowest the week before bee removal occurred (Davis et al., 1997). The pre-application bioassay and water sampling took place on June 20 and 24. However, due to a laboratory error the results for the *in situ* toxicity test for June 20 and 24 were not collected. To make up for the lost *in situ* data, additional pre-spray data were collected on June 26 and 30.

Post-spray samples were collected on July 12, 14, and 16. Post-application bioassays were also run on this schedule to represent anticipated peak pesticide concentrations. The *in situ* bioassay toxicity tests were run July 12 – 16 and July 16 – 20.

Sampling Procedures

Water samples were collected using U.S. Geological Survey depth integrated samplers, modified so sample water contacts only Teflon or glass. Water depths did not exceed four feet, so a DH-81 adapter with a D-77 cap and a wading rod was used.

Depth integrated samples were taken by slowly lowering the sampler to the bottom and immediately raising the sampler at the same rate. Samples were taken at three points (quarter-point transects) across each site. Each sample was hand split into sample containers, filling each container one-third full from each quarter point.

All samples for pesticide analyses were placed in specially pre-cleaned containers. The sampler bottles for water collection were also pre-cleaned. Sampling equipment was decontaminated prior to field work by sequential rinses with laboratory grade detergent (Liquinox), rinsing with tap water and deionized water, and rinsing with pesticide-grade acetone.

Water samples for organophosphorus pesticides were composited in one-gallon glass bottles, stored on ice, and transported to Manchester Environmental Laboratory (MEL) for analysis. Chain-of-custody was maintained throughout the study.

Analytical methods used for the cranberry bog investigation are described in Table 1.

Table 1. Analytical Methods Used for Evaluation of Cranberry Bog Drainage

Analysis	Method	Reference	Laboratory
Chemical			
pH	Orion pH meter	Ecology 1993	Field
Temperature	Hg thermometer	Ecology 1993	Field
Flow	Flow meter	Ecology 1993	Field
Organophosphorus Pesticides	EPA 8085	USEPA 1999	MEL[1]
Biological			
Daphnia pulex	*In situ*-96 hours	Wood 1997	Field
Daphnia pulex	Static-96 hours	Wood 1997	BECR[2]

[1]Manchester Environmental laboratory

[2]Biomonitoring and Environmental Compliance Resource

Water samples for laboratory bioassays were collected as duplicates (splits) with the samples for pesticide analysis, using the same methods and equipment as the water sampling. Samples were collected in one-liter glass bottles. The *in situ* bioassays were run for 96 hours. For the pre-application event, the samples were collected on the first and last day. For the post-application event, the samples were collected on the first, third, and last day (0 hours, 48 hours, and 96 hours). Specific techniques for the laboratory and *in situ* bioassays are described in the bioassay case narratives in Appendix B.

All organophosphorus pesticide target compounds are extracted simultaneously from one sample at MEL (SOP 730011, version 1.1). All target compounds were analyzed by Draft EPA Method 8085 (USEPA, 1999). This method uses a gas chromatograph with an atomic emission detector. A complete list of target compounds is included in Appendix B, table B1.

Data Quality

Standard quality assurance and quality control (QA/QC) procedures used by MEL were employed for this project and are documented in MEL's Quality Assurance Manual (Kirchmer et al., 1989). The sample from one site was duplicated (split) on the first day after pesticide applications as an estimate of overall precision (sampling and analytical). Matrix spike and matrix spike duplicate samples were analyzed from one site on the first day before applications, to evaluate potential interferences and to estimate analytical accuracy and precision.

Accuracy and precision criteria have not been established for Method 8085. Accuracy and precision for organophosphorus pesticides analyses performed for the WSPMP have typically been excellent (Davis and Johnson, 1994; Davis, 1996). Matrix spike recoveries have ranged from 69% to 110%, with an average of 89% and an average relative percent difference (RPD) between spike duplicates of 11%.

Based on historical results from the WSPMP and a desire to obtain exceptionally high quality organophosphorus insecticide data for this study, matrix spike recoveries for the organophosphorus pesticides analysis were regarded as questionable when < 50% recoveries were obtained. Data associated with spike recoveries between 30 and 50% were qualified as estimates. When matrix spike recoveries were below 30%, associated data were rejected.

For this study, recoveries of spiked target compounds were acceptable, ranging from 75% to 139%, except for dimethoate at 153% and 159%. There were relatively low recoveries for diazinon (75% and 85%) due to a subtraction of a substantial concentration of native diazinon from the spiked concentration. However, the recoveries were still acceptable and were not regarded as questionable. The data for the study can be considered acceptable and useable as qualified.

Even though not analyzed, relatively large concentrations of nitrogen-containing pesticides, dichlobenil and norflurazon – as well as a breakdown product of dichlobenil, 2,6-dichlorobenzamide – were found to be present in most samples. More importantly, a substantial concentration of chlorthalonil (a compound in the dichlobenil family) was present at a level similar to that of diazinon.

Results and Discussion

Water Sampling

A complete list of organophosphorus pesticides and associated concentrations are shown in Appendix C, Table C1. Conventional parameters measured at each site in the field are also shown. The conventional parameters include temperature, pH, and flow. For all three sites the temperature ranged from 13.8°C to 20.8°C. The pH for the sites was found to range between 6.7 and 7.3. Flow ranged between 0.24 cfs and 76.5 cfs.

Three organophosphorus pesticides applied to cranberries were detected in water samples collected from GHCDD-1 and PCDD-1 in 1998. All three of the compounds – chlorpyrifos, diazinon, and azinphos-methyl – were detected in both drainage ditches. Concentrations of the detected pesticides were generally higher in PCDD-1 than in GHCDD-1 (Table 2). This may be due to the larger portion of cranberry bogs draining into PCCD-1.

Table 2. Summary of Detected Pesticides From Grayland Cranberry Bog Drainage (ug/L)

Field ID	GHCDD-1					Water Quality Criteria
Date	6/20/98	6/24/98	7/12/98	7/14/98	7/16/98	
	Pre-Spray		*Post-Spray*			
Chlorpyrifos	0.020	0.014	0.0095 J	**0.044**	**1.8**	0.041 [1]
Diazinon	0.033	**0.230**	**0.140**	**0.870**	**4.4**	0.04 [2]
Azinphos (Guthion)	**0.017 J**	**0.038**	0.004 NJ	**0.031 U**	**1.2**	0.01 [3]
Field ID	**PCDD-1**					**Water Quality Criteria**
Date	6/20/98	6/24/98	7/12/98	7/14/98	7/16/98	
	Pre-Spray		*Post-Spray*			
Chlorpyrifos	0.019	**0.056**	**0.640**	**0.910**	**1.3**	0.041 [1]
Diazinon	0.033	**0.190**	**0.094**	**4.5**	**7.0**	0.04 [2]
Azinphos (Guthion)	**0.012 J**	**0.065**	**0.150**	**0.040**	**1.4**	0.01 [3]

[1] Washington State Water Quality Standards, WAC 173-201A.

[2] Menconi and Cox (1994), California Department of Fish and Game.

[3] USEPA (1986), Quality Criteria for Water (Gold Book).

Bold values exceed criteria.

U = Not detected at or above the reported result.

J = Estimated concentration for a positively identified pesticide.

NJ = Estimated concentration for a pesticide believed to be present.

- Chlorpyrifos was detected in all samples collected from both drainage ditches throughout the sampling period. Concentrations ranged from 0.019 ug/L to 1.8 ug/L.

- Diazinon was detected at high concentrations in all but two samples collected from both drainage ditches. Concentrations for diazinon ranged from 0.033 ug/L to 7.0 ug/L.
- Azinphos-methyl was detected in all samples from the PCDD-1, but in only three of the five samples collected from the GHCDD-1. The concentration for azinphos-methyl ranged from 0.004 ug/L to 1.4 ug/L.

For both drainage ditches, the three detected pesticides stayed fairly low and stable until the second post-application sampling on July 14 when the concentrations began to sharply increase. Results from the last sample collection date showed the highest concentrations of all three pesticides for both drainage ditches. In both ditches diazinon was present in the highest concentration. Figures 2 and 3 graphically depict the change over time in the concentrations of chlorpyrifos, diazinon, and azinphos-methyl in each of the ditches.

Comparisons to Water Quality Criteria

Detected concentrations of organophosphorus pesticides are compared to available water quality criteria in Table 2. All listed criteria were developed for the protection of freshwater aquatic species (Davis et al., 1997). The water quality criteria came from several sources:

- Washington State Administrative Code (WAC)
- U.S. Environmental Protection Agency's *Quality Criteria for Water* (Gold Book)
- California Department of Fish and Game (Meconi and Cox, 1994). The diazinon criterion from California is not used in Washington but was selected for comparison, because it represents more recent toxicology than other available criteria for diazinon.

Chlorpyrifos concentrations in GHCDD-1 exceeded the Washington State water quality criterion of 0.041 ug/L on July 14 (0.044 ug/L) and July 16 (1.8 ug/L). Diazinon exceeded the California guidelines of 0.04 ug/L from June 24 through July 16. The highest diazinon concentration was measured at 4.4 ug/L. Azinphos-methyl exceeded the EPA water quality criterion of 0.01 ug/L on the first two sample dates and the last sample date. The highest azinphos-methyl concentration was measured at 1.2 ug/L.

Chlorpyrifos and diazinon concentrations in PCDD-1 exceeded the water quality criteria on all sample dates except June 20. The highest concentration for chlorpyrifos was measured at 1.3 ug/L. The highest concentrations of diazinon were found on the last two sampling dates, and both samples exceeded the water quality criterion by more than 100 times (4.5 and 7.0 ug/L). Azinphos-methyl exceeded the water quality criteria on all sampling dates. The highest measured concentration was 1.4 ug/L.

Comparison to Previous Sampling Results

Three previous studies have assessed the quality of the surface water in the Grayland cranberry bog area. Studies were conducted in 1994, 1995, and 1996 as part of the Washington State Pesticide Monitoring Program (WSPMP). Studies in 1994 and 1995 took place only in

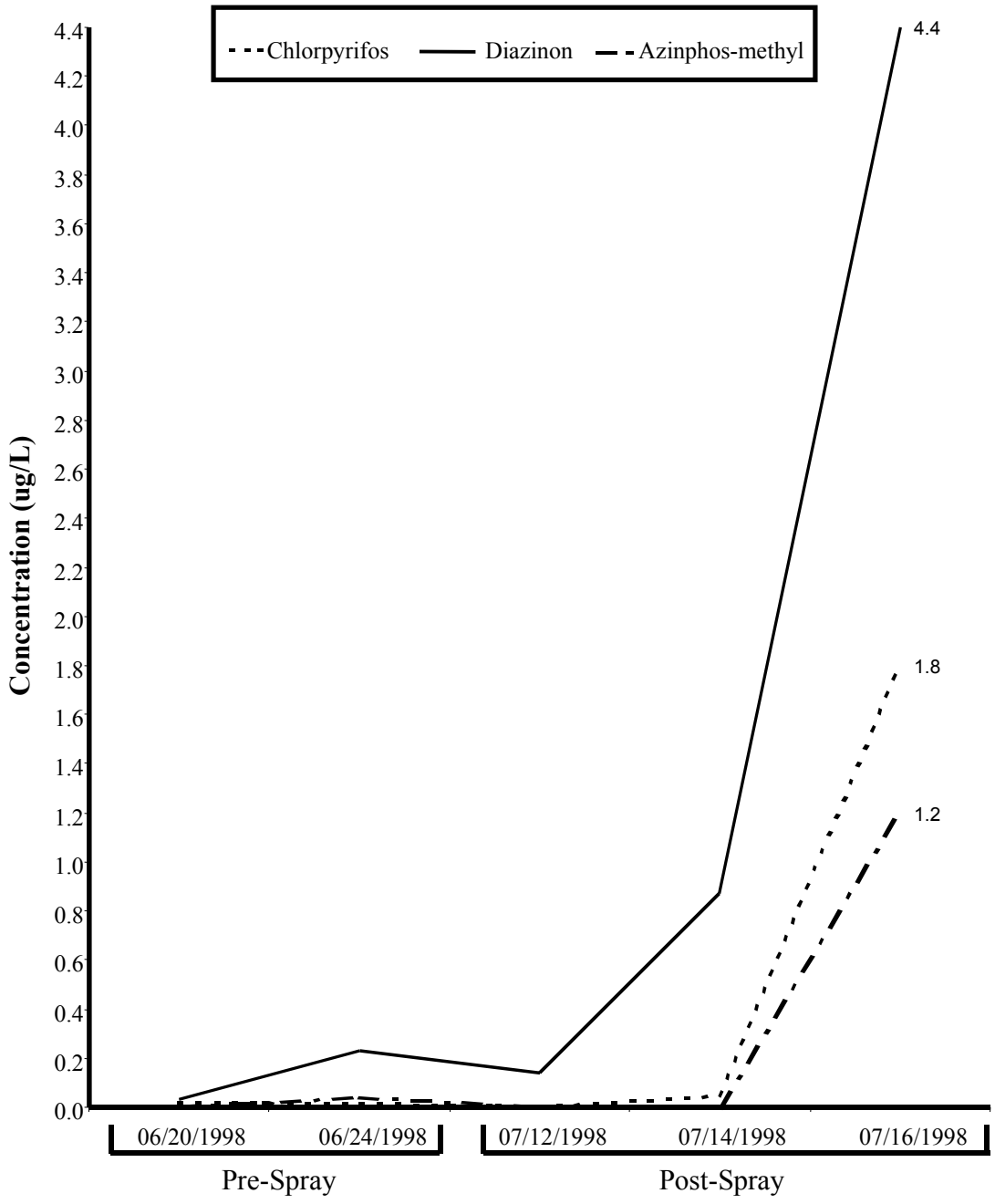

Figure 2. Pesticide Concentrations in GHCDD-1 from 6/20/98-7/16/98

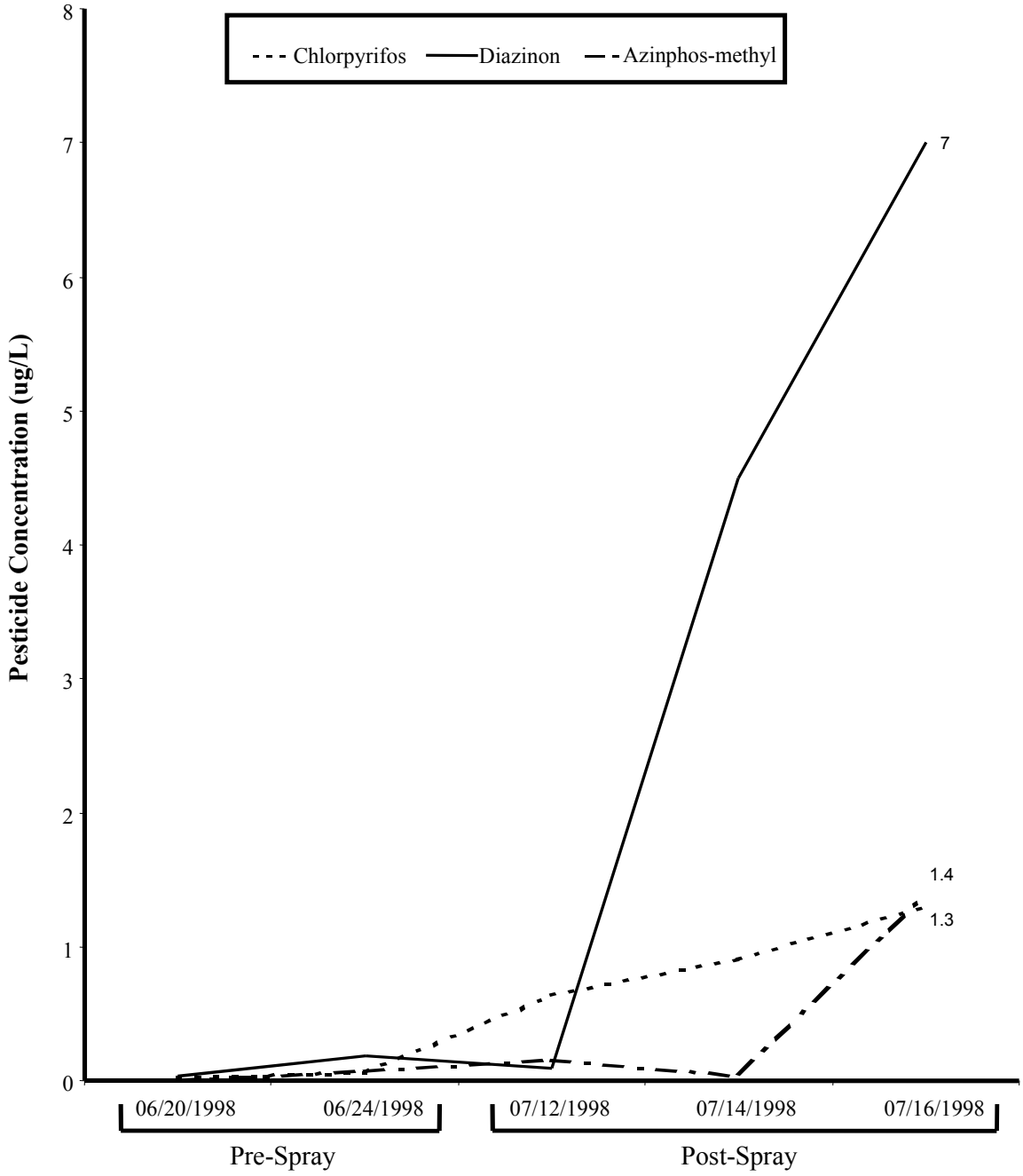

Figure 3. Pesticide Concentrations in PCDD-1 from 6/20/98 - 7/16/98

GHCDD-1 and only evaluated pesticide contamination of surface water. The 1996 study was an overall assessment of cranberry bog drainage; it included an assessment of pesticide contamination in surface water, tissue, and sediment samples in both the GHCDD-1 and PCDD-1.

In 1994, 12 pesticides were detected in the surface water of GHCDD-1. In 1995, 20 pesticides were detected in the surface water of GHCDD-1. In 1996, there were seven pesticides detected in both the GHCDD-1 and PCDD-1. The present study (1998) detected three pesticides (chlorpyrifos, diazinon, and azinphos-methyl) in the surface water of both ditches. These pesticides have been consistently detected throughout all of the studies, and all three have frequently been found to exceed water quality criteria for the protection aquatic life.

Table 3 compares results of the present study with those of the three previous studies.

- GHCDD-1 shows an increase in diazinon concentrations from 1994 to 1998. Chlorpyrifos and azinphos-methyl were detected at similar levels from 1994 to 1996, followed by a large increase in 1998.
- For PCDD-1 there is only one study for comparing results. Chlorpyrifos, diazinon, and azinphos-methyl concentrations in the PCDD-1 increased from 1996 to 1998. The highest concentrations for all pesticides were measured in 1998.

Table 3. Comparison of 1998 Pesticide Study to Previous Studies (mean and range)

		1994[1]	1995[1]	1996	1998
GHCDD-1					
Chlorpyrifos	mean	0.026	0.051	0.008	0.38
	range	0.21-0.030	0.012-0.045	0.003-0.016	0.0095-1.8
Diazinon	mean	0.20	0.24	0.86	1.13
	range	0.011-0.029	0.014-0.68	0.026-5.4	0.033-4.4
Azinphos-methyl	mean	0.014[2]	0.24	0.17	0.26
	range	--	0.018-0.48	0.010-0.73	0.004-1.2
PCCD-1					
Chlorpyrifos	mean	No Data	No Data	0.44	0.59
	range			0.003-3.7	0.019-1.3
Diazinon	mean	No Data	No Data	0.3	2.4
	range			0.008-1.7	0.033-7.0
Azinphos-methyl	mean	No Data	No Data	0.17	0.33
	range			0.006-0.74	0.012-1.4

[1]Data from Washington State Pesticide Monitoring Program - Surface Water Sampling Reports.

[2]Only one detection was recorded.

Bioassay

Pre-spray biomonitoring showed no toxicity in laboratory tests conducted with water samples from all three sites. However, 100% mortality of *Daphnia pulex* occurred during the *in situ* test at the PCDD-1 site between June 26-30. No significant mortality was measured in GHCDD-1 during this period.

Post-spray biomonitoring of the PCDD-1 detected toxicity in both laboratory and *in situ* tests between July 12-16 and between July 16-20. The mortality of the *Daphnia pulex* was 100% in both the laboratory and *in situ* tests. The GHCDD-1 biomonitoring showed no toxicity in the laboratory test but 100% mortality in the *in situ* test between July 12-16. However, between July 16-20, 100% mortality was observed for laboratory and *in situ* testing. Laboratory and *in situ* test results are shown in Table 4.

Table 4. Summary of Laboratory and *In Situ* Bioassay Test Results for Grayland Cranberry Bog Drainage.

Site	Condition	Date	Laboratory % Mortality	*In Situ* % Mortality
Pacific County	Pre-spray	6/26/98 - 6/30/98	0	100
	Post-spray	7/12/98 - 7/16/98	100	100
	Post-spray	7/16/98 - 7/20/98	100	100
Grays Harbor County	Pre-spray	6/26/98 - 6/30/98	0	10
	Post-spray	7/12/98 - 7/16/98	0	100
	Post-spray	7/16/98 - 7/20/98	100	100
Reference	Pre-spray	6/26/98 - 6/30/98	0	2
	Post-spray	7/12/98 - 7/16/98	0	5
	Post-spray	7/16/98 - 7/20/98	0	15

While less conclusive for the in-situ tests in the PCDD-1, overall these data clearly show the potential for adverse biological effects in both the PCDD-1 and GHCDD-1 after pesticide application.

Conclusions

During this 1998 study, three organophosphorus pesticides – chlorpyrifos, diazinon, and azinphos-methyl – were detected in water samples from PCDD-1 and GHCDD-1, which drain cranberry growing areas near Grayland. The lowest pesticide levels were detected prior to pesticide spraying. The highest levels were detected after spraying.

- All pesticides detected exceeded applicable water quality criteria/guidelines during most of the sampling period.

- Chlorpyrifos exceeded the state water quality criterion of 0.041 ug/L in 25% of the samples before pesticide application and 83% after application.

- Diazinon exceeded available water quality guidelines of 0.04 ug/L in 50% of the samples before application and 100% after application. Diazinon concentrations were the highest of the three detected pesticides.

- Azinphos-methyl exceeded the EPA water quality criterion of 0.01 ug/L throughout the study period.

- Comparisons among pesticide levels found in 1994, 1995, 1996, and 1998 show that the highest levels were typically measured in 1998. The data show no reduction in overall pesticide levels in the drainage ditches, despite implementation of best management practices by a few cranberry growers.

- Bioassay results indicate the potential for adverse biological effects in the cranberry drainage ditches after pesticide spraying. The high mortality rate observed for the laboratory and *in situ* bioassay is, in all probability, a direct result of the pesticide concentrations in both ditches after spraying.

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Appendix A

Station Position and Descriptions

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Table A1. Station Locations for Pesticide Sampling, Grayland Cranberry Bog Drainage Water Quality Assessment.

	Latitude			Longitude			
Station ID	Deg	Min	Sec	Deg	Min	Sec	Description
GHCDD-1	46	48	58	124	5	25	Grays Harbor County Drainage Ditch No. 1 at the Schmid Road Bridge
PCDD-1	46	44	27	124	4	21	Pacific County Drainage Ditch No. 1 at the Larkin Road Bridge
Reference	46	49	25	124	6	5	Drainage ditch (north of GHCDD-1) that receives no drainage water from the bogs

Datum = WSG84

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Appendix B

Bioassay Case Narratives - In-Situ
Bioassay Case Narratives - Laboratory
Target Pesticides List

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Bioassay Case Narratives – In-Situ

Biomonitoring and Environmental Compliance Resource
BECR Laboratory
16125 Railway Rd. Bldg. C
Yelm, WA 98597-9404
Phone: 360 458-8210 Fax: 360 458-8220

Project Description
Biological Assessment of Pesticides Entering The Grayland Drainage Ditch, Grayland, Washington.

Project Organization and Responsibility
Biomonitoring and Environmental Compliance Resource (BECR Laboratory) will conduct *in situ* and laboratory bioassay tests using the standard test organism *Daphnia pulex* to determine toxicity of the Grayland Drainage Ditch water under ambient and controlled test conditions. Bioassay testing will be conducted using two test sites and a control site during two testing events, including pre- and post-application of pesticides. Each testing event will include a two level screen laboratory bioassay, conducted concurrently with a 96-h *in situ* bioassay.

Sampling Procedures
When required, BECR Laboratory will collect grab water samples in containers supplied by Washington State Department of Ecology, Environmental Investigations Laboratory, Olympia, WA.

Sample Custody
Chain of custody is to follow sample from collection to delivery to BECR Laboratory.

Calibration Procedures and Frequency
Calibration of instruments (dissolved oxygen, pH, conductivity meter, and the Denver balance) will be conducted on a daily schedule before use following manufacturer's instructions. A NIST certified thermometer is located in each test and culture incubator for temperature recording.

Analytical Reagents - Only analytical grade reagents will be used in preparation of solutions and/or reagents. Milli-Q or equivalent water will be used to prepare solutions and MHSW. If required, distilled water will be purchased.

Temperature - Daily temperature records will be kept on each incubator (2), refrigerators (2), and the laboratory. NIST thermometers are located in each of the test and culture incubators.

pH measurement - An Accumet Basic pH meter will be used for laboratory use. A YSI 600R multi-meter is used in the field. A three-point calibration (pH 4, pH 7, pH 10) will be conducted prior to testing an unknown sample. A two-point calibration will be conducted prior to use on a sample of known pH range, i.e., a basic sample -- pH 7, followed by pH 10 or an acidic sample -- pH 7 followed by pH 4. Calibration is conducted daily prior to use. Calibration data are recorded in indelible ink on data sheets.

Dissolved Oxygen - A YSI Model 50B will be used for laboratory use. A YSI 600R multi-meter is used in the field. Storage and calibration are conducted following manufacture's instructions. Air calibration of the D.O. meter is performed daily prior to use. A monthly calibration will be conducted monthly to assure proper working condition of the probe and instrument. The D.O. membrane is visually checked regularly and replaced when necessary or after approximately 500 hours of use. Daily calibration data are recorded on data log sheets.

Conductivity - USEPA Method 120.1 (Direct measurement). An Oakton meter is used in the laboratory. A YSI 600R multi-meter is used in the field. Conductivity standards (84 [uS and 447 uS @ 25 ^{o}C) are available and used for calibration of meter daily prior to use.

Chemical Parameter Methods

BECR Laboratory will follow chemical analysis methods detailed in USEPA EPA-600/4-79-020 "Methods for Chemical Analysis of Water and Wastes." Standard Operating Procedures (SOPs) for all methods are located in Appendix A of the BECR Laboratory QA/QC manual. In addition, all SOPs are available for immediate reference in calibration and test binders.

Internal Quality Control Checks and Frequency

A reference toxicant testing will be done concurrently with test sample to assure test organism health and laboratory performance is acceptable. Reference toxicant tests run as a positive control for effluent tests can satisfy the monthly reference test.

A control chart using the last 20 reference toxicant test results for each test conducted by BECR are kept on file to assure facilities and equipment, test organisms, food quality, and technician skill are adequate. All valid reference toxicant test results will be used to construct a control chart for each test conducted by BECR. All valid reference toxicant test results must fall within the upper and lower control limits (0 - 2 SD) to be acceptable.

Laboratory Cultures

Cultures of Daphnia *(Ceriodaphnia dubia* and *Daphnia pulex)* and algae *(Selenastrum capricornium)* are maintained in the laboratory. Cultures of *Ceriodaphnia dubia* and *Daphnia pulex* were originally obtained from EPA, Duluth, MN, and thus taxonomically identified. Each species has been verified using the key "Freshwater Invertebrates of the United States; Protozoa to Mollusca (Penneck 1989). Each species is preserved on a permanent slide for reference. A sterile culture of *Selenastrum capricornium* was originally obtained from Aquatic Research Organisms.

Test Conditions
All freshwater toxicity bioassays are conducted following procedures in EPA 600/4-91/002 and EPA 600/4-90/027F.

Moderately Hard Synthetic Water (MHSW), prepared with Milli-Q equivalent grade water and reagent grade chemicals, is used for culture organism rearing and testing. Each batch (20 L) of MHSW has water quality parameters measured and recorded on the MHSW log sheets. MHSW is kept for a maximum of 14 days in the dark before being discarded.

Each Precision incubator (one test, one culture) has been programmed to run at 25 ± 1 ^{O}C for 24 hours, 7 days a week. Illumination (300 foot-candles) has been programmed to run at a 16 hour on (0600 - 2200) - 8 hour off (2200 - 0600) cycle during each 24-hour period. Temperature of incubators is recorded daily in 2 places in each incubator. During testing, temperature is recorded in each test concentration during a test to determine temperature uniformity throughout the test. Surrogate containers may be placed at each comer and in the middle of the test for the purpose of recording temperatures. All test chambers are covered with a plexiglass cover to avoid evaporation and contamination of test chambers.

Data Validation and Reporting
Test bench sheets, test calculations, and transcriptions of all electronic data will be reviewed by the QA personnel prior to the final report being issued. The anticipated test review and final report time should be less than two weeks following final test data collection. Test conditions, data analysis and report requirements listed in WDOE WQ--R-95-80 will be followed to assure acceptance by the WDOE Water Quality Program.

STANDARD OPERATING PROCEDURE: *IN SITU* TOXICITY TESTING:

IN SITU BIOASSAYS:

In situ chambers are constructed with 5.1- by 12.7 -cm clear liner tubes (cellulose acetate butyrate) capped with two polyethylene closure caps. Two long rectangular windows (6 x 2.5 cm) are covered with 74 micron mesh to contain organisms and exclude predators while allowing exposure to test media.

Daphnia pulex - 96 h

NOTE: Requires removal of neonates from stock cultures 24-h prior to test set-up.

IN SITU TEST INITIATION:

On day of test set-up, remove <24-h neonates from stock cultures. Pool neonates and feed 1:1 YTC and Selenastrum 2-h prior to use.

- Label 20 ml test tubes with a number, starting with one. Each test site requires a total of four replicates. Mark an additional four test tubes for travel control data. Generate random test positions using TOXCALC. Mark assigned position below replicate number.

- Fill test tubes half full with Moderately Hard Synthetic Water (MHSW)

- Introduce 1-2 test organisms/replicate by submerging 2 mm i.d. pipette just under water surface, avoiding any air bubbles. Continue until there are a total of ten organisms/replicate. Verify that ten organisms are in each test and control replicate using a fiber light.

- Place test tubes in order of randomized position into a test tube rack. Cover and place in ice cooler with blue ice. NOTE: Organisms should be chilled to field water temperature slowly over a minimum of two hours.

- In the field, at *in situ* test set-up, measure and record the physical and chemical parameters using the YSI 600R multi-meter; D.0. (%, mg/L), temperature (^{o}C), pH and conductivity (uS /cm). Upon arrival back to laboratory, measure and record hardness and alkalinity. Place travel control replicates into 4 ^{o}C refrigerator until test termination.

Biomonitoring and Environmental Compliance Resource, Yelm, WA.
Phone 360 459-8210 Fax 360 459-8220

*Collect a grab sample in a USEPA approved container by rinsing three times with sample water, submerging container at least 12 inches below the surface and allowing container to fill. Expel all air and seal with no headspace. Immediately chill to 4 ^{o}C. Sample holding time is a maximum of 36 hours at 4 ^{o}C. Upon arrival back to the laboratory, set-up a *D. pulex* laboratory bioassay screen (48 h) with 100% and 50% sample concentration following SOP.

IN SITU TEST TERMINATION:

- In the field, at *in situ* test termination, collect and record the physical and chemical measurements using the YSI 600R multi-meter; D.O. (%, mg/L), temperature (^{o}C), pH and conductivity (uS /cm).
- Collect *in situ* chambers and place into bucket with sample water.
- At the laboratory.
- Slowly remove one end cap from chamber. Rinse interior sides of chamber to assure all organisms are collected.
- Note and record any mortalities and abnormal behavior in test organisms collected from the control and test water sites. Record findings on test data sheet.

- Note and record any mortalities and abnormal behavior in travel control replicates that were held at 4 ^{o}C. Record findings on test data sheet.
- Analyze survival data using the statistical program TOXCALC.

TEST ACCEPTABILITY:

In situ test acceptability is no less than 80 % survival in the control test site. If no field control site was used, *in situ* test acceptability is no less than 90% in the travel controls.

* It is recommended (however, not required) that an acute laboratory bioassay be conducted concurrently with sample water from each *in situ* test site.

Bioassay Case Narratives – Laboratory

Biomonitoring and Environmental Compliance Resource, Yelm WA
Phone 360 458-8210 Fax 360 459-9220

Daphnia pulex - Acute LC_{50}{tc "*Daphnia pulex* - LC_{50}" \l 2}
48 h static - Non-renewal

EPA/600/4-90/027F Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms (pg. 45-75)

NOTE: Requires removal of neonates from stock cultures 24 -h prior to test set-up. On day of test set-up, remove <24-h neonates from stock cultures. Pool neonates and feed YTC and *Selenastrum* 2-h prior to use.

Prepare water sample for testing at 25 ± 1 ^{o}C following SOP.

Once temperature has reached equilibrium at 25 ± 1 ^{o}C record the following physical and chemical measurements following SOP; D.O., temperature, pH, conductivity, hardness, and alkalinity. Measure total chlorine and ammonia if sample is from a WWTP or a suspected effluent.

Identify and label each concentration and control with a different color marking pen. Generate random test positions using TOXCALC. Mark assigned position below replicate number. (Example: Red represents 50% concentration. Using a red marker, label replicate number (1-4), with random position number written underneath (1-24)).

Prepare 250 ml of sample for a minimum of five effluent concentrations using a 0.5 dilution factor; 100%, 50%, 25%, 12.5%, and 6.25%.

100% - 250 ml of test sample
50% - 125 ml of test sample and 125 of MHSW.
25% - 62.50 ml of test sample and 187.5 ml of MHSW
12.5% - 31.25 ml of test sample and 219.75 ml of MHSW
6.25% - 15.60 ml of test sample and 234.40 ml of MHSW

Test sample - fill 4 - 30 ml polypropylene cups with 25 ml of each test concentration.

Control - fill 4-30 ml polypropylene cups with 25 ml Moderately Hard Synthetic Water (MHSW).

Introduce 1-2 test organism/replicate by submerging a 2 mm i.d. pipette just under water surface, avoiding any air bubbles. Continue until there are a total of five organisms/

replicate. Verify that five organisms are in each test and control replicate using a lighted magnifying glass. Replace transfer pipette after each concentration. Verify that five organisms are in each test and control replicate using lighted magnifying glass.

Place test chambers on a test tray and place in 25 ± 1 ^{o}C test incubator.

If mortality is noted at one hour after test initiation, set-up an additional test concentration of 3.175%.

At 4-8 hours after test set-up, check D.O. in the control and test concentration. If necessary, aerate all test chambers with ~100 bubbles/min. Do not over aerate.

At 24-h note any mortalities and abnormal behavior in control and test water samples. Record findings on bench sheet.

At 48-h note any moralities and abnormal behavior in control and test water samples. Record findings on bench sheet. Remove *Daphnia* from each replicate and pool water samples for physical and chemical measurements; temperature, D.O., pH, and conductivity.

Test acceptability is no less than 90 % survival in the control, with all test conditions within limits for temperature, D.O., pH, etc. (see WQ-R-95-80).

Reference Toxicant Test Procedure

The current reference toxicant used is sodium chloride (NaCl).

Set-up a test following the above procedure - the 100% effluent used is 8 g/L NaCl.

Place 8g of NaCl in a 1 L volumetric flask. Fill flask 3/4 full. Add stirbar and place on magnetic stirrer until dissolved. Remove stirbar. Fill to fill line.

Prepare dilutions as described above.

Plot the result on the established control chart for this test.

Target Pesticides List

Table B1. Target List for Water Analyses - Organophosphorus Pesticides

Analyte	Quantitation Limit[1] (ug/L)		Analyte	Quantitation Limit[1] (ug/L)
acephate	0.3		fenosulfothion	0.075
azinphos-ethyl	0.12		fenthion	0.055
azinphos-methyl	0.12		fonophos	0.045
carbophenothion	0.8		imidan	0.08
chlorpyrifos	0.055		malathion	0.06
chlorpyrifos-methyl	0.055		merphos	0.12
coumaphos	0.09		methamidophos	0.3
DEF	0.11		mevinphos	0.075
demeton-O	0.055		paraoxon-methyl	0.15
demeton-S	0.06		parathion	0.06
diazinon	0.06		parathion-methyl	0.055
dichlorvos	0.06		phorate	0.055
demethoate	0.06		phosphamidan	0.18
dioxathion	0.12		propetamphos	0.15
disulfthion	0.045		ronnel	0.055
EPN	0.075		sulfotepp	0.045
ethion	0.055		sulprofos	0.055
ethoprop	0.06		temephos	0.7
fenamiphos	0.12		tertachlorvinphos	0.15
fenitrothion	0.055			

[1]Quantitation limits are approximate and are often different for each sample; these values are representative of a typical sample

Appendix C

Summary of Water Sample Analyses

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Table C1. Summary of Analysis of Water Samples From Grayland Cranberry Bog Drainage

	Pre-Spray				Post-Spray					
Field ID	**GHCDD-1**		**GHCDD-1**		**GHCDD-1**		**GHCDD-1**		**GHCDD-1**	
Sample No. 98-	258050		268050		298080		298090		298095	
Date	6/20/98		6/24/98		7/12/98		7/14/98		7/16/98	
Flow (CFS)	6.69		14.90		7.38		9.40		33.25	
Temperature (oC)	19.5		14.1		14.8		14.4		15.7	
pH	6.90		6.77		6.89		6.78		6.69	
Pesticides (ug/L)										
Fensulfothion	0.019	U	0.019	U	0.02	U	0.019	U	0.021	U
Malathion	0.015	U	0.015	U	0.016	U	0.015	U	0.140	
Fenitrothion	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Demeton-S	0.013	UJ	0.013	UJ	0.014	UJ	0.013	UJ	0.015	UJ
Ethoprop	0.015	U	0.015	U	0.016	U	0.015	U	0.017	U
Merphos (1 & 2)	0.023	U	0.023	U	0.024	U	0.023	U	0.025	U
EPN	0.019	U	0.019	U	0.020	U	0.019	U	0.021	U
Fenamiphos	0.028	UJ	0.028	UJ	0.030	UJ	0.029	UJ	0.032	UJ
Azinphos Ethyl	0.030	U	0.030	U	0.032	U	0.031	U	0.034	U
Chlorpyrifos	0.020		0.014		0.0095	J	0.044		1.8	
Phosphamidan	0.045	UJ	0.045	UJ	0.048	UJ	0.046	UJ	0.051	UJ
Methyl Parathion	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Phorate	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Demeton-O	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Disulfoton (Di-Syston)	0.011	U	0.011	U	0.012	U	0.012	U	0.013	U
Ronnel	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Propetamphos	0.038	UJ	0.038	UJ	0.04	UJ	0.038	U	0.170	UJ
Diazinon	0.033		0.230		0.140		0.870		4.4	
Abate (Temephos)	0.110	U	0.110	U	0.12	U	0.12	U	0.130	U
Bolstar (Sulprofos)	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Sulfotepp	0.011	U	0.011	U	0.012	U	0.012	U	0.011	
Fenthion	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Methyl Chlorpyrifos	0.015	U	0.015	U	0.016	U	0.015	U	0.017	U
Ethion	0.013	U	0.013	U	0.014	U	0.013	U	0.015	U
Parathion	0.015	U	0.015	U	0.016	U	0.015	U	0.017	U
Coumaphos	0.023	U	0.023	U	0.024	U	0.023	U	0.025	U
Dimethoate	0.015	U	0.015	U	0.016	U	0.015	U	0.017	U
Dichlorvos (DDVP)	0.015	U	0.015	U	0.016	U	0.015	U	0.017	U
Imidan	0.021	U	0.021	U	0.022	U	0.021	U	0.023	U
Mevinphos	0.019	U	0.019	U	0.020	U	0.019	U	0.021	U
Dioxathion	0.032	U	0.032	U	0.034	U	0.033	U	0.036	U
Tribufos (DEF)	0.027	U	0.027	U	0.028	U	0.027	U	0.03	U
Carbophenothion	0.019	U	0.019	U	0.020	U	0.019	U	0.021	U
Azinphos-methyl	0.017	J	0.038		0.004	NJ	0.031	U	1.2	
Fonofos	0.011	U	0.011	UJ	0.012	U	0.012	U	0.170	UJ
Methyl Paraoxon	0.034	U	0.034	U	0.036	U	0.035	U	0.038	U
Tetrachlorvinphos (Gardona)	0.038	U	0.038	U	0.040	U	0.038	U	0.042	U

U = Not detected at or above the reported result.

UJ = Estimated detection limit.

J = Estimated concentration for a positively identified pesticide.

NJ = Estimated concentration for a pesticide believed to be present.

Table C1. Summary of Analysis of Water Samples from Grayland Cranberry Bog Drainage

	Pre-Spray				Post-Spray					
Field ID	**PCDD-1**		**PCDD-1**		**PCDD-1**		**PCDD-1**		**PCDD-1**	
Sample No. 98-	258051		268051		298081		298091		298096	
Date	6/20/98		6/24/98		7/12/98		7/14/98		7/16/98	
Flow (CFS)	19.88		29.49		20.03		22.90		76.54	
Temperature (oC)	14.8		14.0		13.8		14.5		14.6	
pH	6.75		7.18		7.29		7.28		7.08	
Pesticides (ug/L)										
Fensulfothion	0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Malathion	0.016	U	0.015	U	0.021		0.017	U	0.017	U
Fenitrothion	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Demeton-S	0.014	UJ	0.013	UJ	0.014	UJ	0.015	UJ	0.015	UJ
Ethoprop	0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Merphos (1 & 2)	0.023	U	0.023	U	0.024	U	0.025	U	0.025	U
EPN	0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Fenamiphos	0.029	UJ	0.029	UJ	0.030	UJ	0.031	UJ	0.031	UJ
Azinphos Ethyl	0.031	U	0.031	U	0.031	U	0.033	U	0.033	U
Chlorpyrifos	0.019		0.056		0.640		0.910		1.3	
Phosphamidan	0.047	UJ	0.046	UJ	0.047	UJ	0.170	UJ	0.05	UJ
Methyl Parathion	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Phorate	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Demeton-O	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Disulfoton (Di-Syston)	0.012	U	0.012	U	0.012	U	0.013	U	0.013	U
Ronnel	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Propetamphos	0.039	UJ	0.038	UJ	0.039	UJ	0.042	UJ	0.170	UJ
Diazinon	0.033		0.190		0.094		4.5		7.0	
Abate (Temephos)	0.120	U	0.120	U	0.120	U	0.130	U	0.130	U
Bolstar (Sulprofos)	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Sulfotepp	0.012	U	0.012	U	0.009	J	0.007	J	0.009	J
Fenthion	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Methyl Chlorpyrifos	0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Ethion	0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Parathion	0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Coumaphos	0.023	U	0.023	U	0.024	U	0.025	U	0.025	U
Dimethoate	0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Dichlorvos (DDVP)	0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Imidan	0.021	U	0.021	U	0.022	U	0.023	U	0.023	U
Mevinphos	0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Dioxathion	0.033	U	0.033	U	0.033	U	0.035	U	0.035	U
Tribufos (DEF)	0.027	U	0.027	U	0.028	U	0.029	U	0.029	U
Carbophenothion	0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Azinphos-methyl	0.012	J	0.065		0.150		0.040		1.4	
Fonofos	0.012	U	0.012	U	0.012	U	0.170	UJ	0.170	UJ
Methyl Paraoxon	0.035	U	0.035	U	0.035	U	0.038	U	0.038	U
Tetrachlorvinphos (Gardona)	0.039	U	0.038	U	0.039	U	0.042	U	0.042	U

U = Not detected at or above the reported result.

UJ = Estimated detection limit.

J = Estimated concentration for a positively identified pesticide.

NJ = Estimated concentration for a pesticide believed to be present.

Table C1. Summary of Analysis of Water Samples from Grayland Cranberry Bog Drainage

		Pre-Spray				Post-Spray					
Field ID		**Reference**		**Reference**		**Reference**		**Reference**		**Reference**	
Sample No.	**98-**	258052		268052		298083		298092		298097	
Date		6/20/98		6/24/98		7/12/98		7/14/98		7/16/98	
Flow (CFS)		0.241		0.389		0.247		0.440		0.272	
Temperature (oC)		20.8		14.5		15.4		14.5		18.1	
pH		7.10		6.97		7.06		6.99		6.95	
Pesticides (ug/L)											
Fensulfothion		0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Malathion		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Fenitrothion		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Demeton-S		0.014	UJ	0.013	UJ	0.014	UJ	0.015	UJ	0.015	UJ
Ethoprop		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Merphos (1 & 2)		0.023	U	0.023	U	0.024	U	0.025	U	0.025	U
EPN		0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Fenamiphos		0.029	UJ	0.029	UJ	0.030	UJ	0.031	UJ	0.031	UJ
Azinphos Ethyl		0.031	U	0.030	U	0.032	U	0.033	U	0.033	U
Chlorpyrifos		0.016	U	0.015	U	0.016	U	0.028		0.017	U
Phosphamidan		0.047	UJ	0.046	UJ	0.048	UJ	0.050	UJ	0.050	UJ
Methyl Parathion		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Phorate		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Demeton-O		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Disulfoton (Di-Syston)		0.012	U	0.011	U	0.012	U	0.013	U	0.013	U
Ronnel		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Propetamphos		0.039	UJ	0.038	UJ	0.040	U	0.042	UJ	0.042	U
Diazinon		0.016	U	0.015	U	0.016	U	0.180		0.017	U
Abate (Temephos)		0.120	U	0.110	U	0.120	U	0.130	U	0.130	U
Bolstar (Sulprofos)		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Sulfotepp		0.012	U	0.011	U	0.012	U	0.013	U	0.013	U
Fenthion		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Methyl Chlorpyrifos		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Ethion		0.014	U	0.013	U	0.014	U	0.015	U	0.015	U
Parathion		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Coumaphos		0.023	U	0.023	U	0.024	U	0.025	U	0.025	U
Dimethoate		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Dichlorvos (DDVP)		0.016	U	0.015	U	0.016	U	0.017	U	0.017	U
Imidan		0.021	U	0.021	U	0.022	U	0.023	U	0.023	U
Mevinphos		0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Dioxathion		0.033	U	0.032	U	0.034	U	0.035	U	0.035	U
Tribufos (DEF)		0.027	U	0.027	U	0.028	U	0.029	U	0.029	U
Carbophenothion		0.019	U	0.019	U	0.020	U	0.021	U	0.021	U
Azinphos-methyl		0.031	U	0.030	U	0.032	U	0.033	U	0.033	U
Fonofos		0.012	U	0.011	U	0.012	U	0.013	UJ	0.013	U
Methyl Paraoxon		0.035	U	0.034	U	0.036	U	0.038	U	0.038	U
Tetrachlorvinphos (Gardona)		0.039	U	0.038	U	0.040	U	0.042	U	0.042	U

U = Not detected at or above the reported result.

UJ = Estimated detection limit.

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