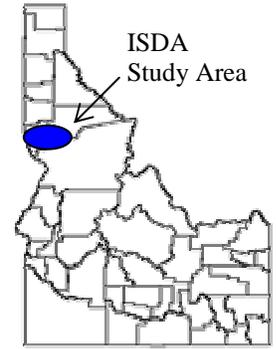




Idaho State
Department of Agriculture



Synoptic Evaluation of Pesticide Concentrations Clearwater River Basin, Central Idaho



Idaho State Department of Agriculture Water Quality Technical Report W-12

Prepared by Kirk Campbell December, 2004

In cooperation with the Environmental Protection Agency, Idaho Soil Conservation Commission, Idaho Association of Soil Conservation Districts, and the University of Idaho Analytical Science Laboratory Moscow, Idaho

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Abstract

The Idaho State Department of Agriculture (ISDA) has the responsibility under the Federal Insecticide, Fungicide, and Rodenticide Act (FIFRA) to ensure proper use of pesticides and the protection of the environment from pesticides. The use of pesticides in conjunction with agriculture and other resource concerns could have an environmental impact on water quality resources within a watershed. Environmental Protection Agency (EPA) discretionary funding allowed ISDA's surface water program to monitor water quality for the presence of pesticides within the Idaho Clearwater River Basin. Eight tributaries, of the Clearwater River, were chosen for sampling and analysis which generated a total of 64 samples over a three month period. The eight tributaries account for approximately 977,000 acres with approximately 503,000 being under some type of agricultural activity. A total of 47 detections were confirmed during this study. Of those 47 detects a total of 13 pesticides were identified with Metribuzin (11), Diuron (7), Dicamba (6) and Atrazine (5) detections leading the list. All pesticide concentrations detected during this study were below any chronic or acute levels that may cause ill effects for aquatic species.

Introduction

During the period from April 6, 2004 through July 14, 2004 the Idaho State Department of Agriculture (ISDA), Idaho Association of Soil Conservation Districts (IASCD) and the University of Idaho Analytical Science Laboratory (ASL) conducted a synoptic evaluation of pesticides on eight tributaries that discharge into the Clearwater River and the South Fork of the Clearwater River (Figure 1). The eight tributaries that were monitored are as follows: Cottonwood Creek of the South Fork Clearwater River, Cottonwood Creek of the mainstem Clearwater River, Catholic Creek, Potlatch River, Big Canyon Creek which includes Little Canyon Creek, Lapwai Creek, Jacks Creek, and Lawyer Creek. Samples were collected on a bi-weekly schedule through July 14, 2004.

The Clearwater River Basin is located in north-central Idaho and covers 9,645 square miles. The eight tributaries monitored during this study encompass approximately 977,426 acres with 503,000 of those acres involved in agricultural activities. The Clearwater area was chosen due to its salmonid fish population along with its diverse agricultural base and extensive pesticide use.

This project was made possible by discretionary funding from the Environmental Protection Agency (EPA) along with additional funding from ISDA to support labor, equipment and additional analytical costs. Additional field expertise was provided by the IASCD's water quality analyst located in Moscow, Idaho.

Idaho agriculture relies on the use of pesticides and fertilizers for crop protection and crop health. The use of pesticides, if planned and implemented properly can benefit crop production while providing minimal risk to human health and the environment. The potential for degradation of surface water from agricultural practices is a function of numerous factors including agrichemical properties, physical properties, land management at the application site, application techniques and weather. Tributaries chosen for this study, within the Clearwater River basin, were those that were known to be salmonid bearing streams and contained significant agricultural acreage where pesticide application was likely occurring (Table 1). Four of the streams chosen for this study are listed on the state of Idaho's 303(d) list for pesticides (Table 1). ISDA has the responsibility to implement FIFRA and to ensure proper use of pesticides and the protection of the environment from pesticides. This monitoring project was conducted to fulfill these responsibilities and mandates.

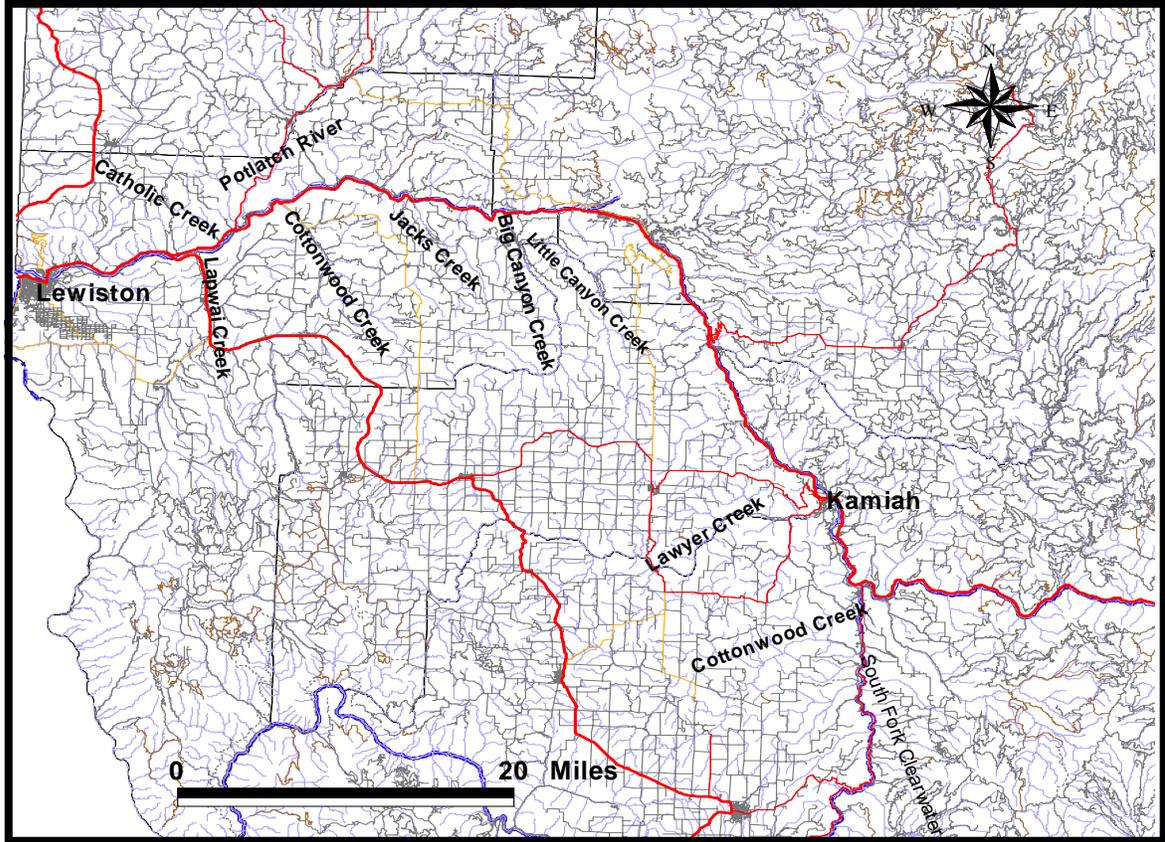


Figure 1. Clearwater River tributary sampling sites.

Table 1. Monitoring Sites Evaluation Criteria

Monitoring Sites	Total Acres	*Agricultural Acres	%Agricultural land	Salmonid Activity	303(d) listed for pesticides
Lapwai Creek	171,173	57,872	34	X	
Catholic Creek	12,415	6,391	51	X	X
Potlatch R.	378,958	156,846	41	X	X
Big Canyon Creek (includes Little Canyon Creek)	112,771	71,887	63	X	X
Lawyer Creek	137,452	90,814	66	X	X
Cottonwood River Main Clearwater	42,120	17,218	41	X	
Cottonwood River SF Clearwater	122,537	102,036	83	X	
Jacks Creek	12,157	5,650	43	X	

* Agricultural Acreage does not include rangeland

Sampling and Analytical Methods

Sampling Methods

All equipment utilized for pesticide collection was constructed of Teflon, glass or stainless steel. Samples were collected using either a US DH-95 weighted integrated sampler equipped with a Teflon sample bottle and intake nozzle or a Teflon DH-81 integrated sediment sampler. The DH-95 was used when sampling from bridges while the DH-81 was used for wadeable sites. Vertical composite samples were collected at equal spaced intervals across the stream profile. The individual vertical discrete samples were transferred into a clean 2.5 gallon glass carboy to form the composite. The resultant composite was then transferred into three 1-liter amber sample bottles and stored within a cooler on ice.

Sampling equipment (glass, stainless, and Teflon) used during this program was thoroughly cleaned between each monitoring location. The equipment was first thoroughly scrubbed with Liquinox detergent then rinsed with deionized water followed by an acetone rinse followed by deionized water and finally a thorough rinse with source water. To insure the integrity of the cleaning procedure equipment blank samples were collected periodically during this program and submitted blindly to the laboratory for analysis.

All resultant samples were placed within a cooler, on ice, for transport directly to the University of Idaho Analytical Science Laboratory (ASL) in Moscow Idaho. All samples were delivered to ASL on the same day they were collected. Chain-of-Custody forms accompanied each sample shipment to the laboratory.

Discharge was measured at 6 of the 8 locations during each monitoring event. Two sites (Lapwai Creek and Potlatch River) had United States Geological Survey (USGS) gage stations and their discharge rates were taken from the USGS web site. Discharge was measured at the 6 sites using a Marsh McBirney Flow Mate Model 2000 meter. The six-tenths depth method (0.6 of the total depth from the surface of the water surface) was used. A transect line was established at each monitoring station, across the width of the stream at an angle perpendicular to the flow, for the calculation of cross-sectional area. The discharge was computed by summing the products of the partial areas (partial sections) of the flow cross-sections and the average velocities for each of those sections. Stream discharge was reported as cubic feet per second (cfs).

Analytical Methods

EPA method 507 describes the extraction and gas chromatographic (GC) conditions for the analysis of selected organo-nitrogen and organo-phosphorus pesticides in water samples. The method is a modified version of EPA 507 that expands the number of pesticide analytes and improves the detection limits obtained for those analytes. A one-liter volume of a water sample is extracted with methylene chloride by shaking in a separatory funnel. The methylene chloride extract is isolated, dried and concentrated to a volume of 2 milliliters (mL) during a solvent exchange to methyl tert-butyl ether (MTBE). This 2 mL sample extract is divided among three instruments: gas chromatography, nitrogen, phosphorus detector (GC/NPD); large volume liquid injector, gas chromatograph, mass spectral detector (LVLI/GC/MSD); and liquid chromatograph, mass spectrometer (LC/MS). The pesticide minimum detection limits (MDL's) have been decreased to the low parts per trillion (ppt) range when compared to the original EPA 507 by incorporating a large volume injection (LVI) system (APEX ProSep 800) on the 6890/5937 GC/MSD. This LVI/GC/MSD system routinely can provide confirmation and quantification of pesticide residues at levels that are near or below the GC/NPD detection limit (0.02 to 0.005 µg/L). This modified method also describes and establishes ppt MDL's for phenyl urea pesticides that could not be routinely analyzed prior to the addition of the LC/MS instrument.

A modified 515.2 extraction method (chlorinated acid herbicides) is applied to a water sample of 250 mL that has been treated with concentrated NaOH to hydrolyze esters. The sample is then adjusted to pH 2 with phosphoric acid. The sample is subsequently run through a conditioned Oasis hydrophilic-lipophilic balance (HLB) column and eluted with MeOH/MTBE mix. The acids are converted to their methyl esters using diazomethane as the derivatizing agent. The esters are detected by capillary column/GC using an electron capture detector (ECD) and mass spectral detector (MSD). The chlorinated acid pesticide detection limits are in the low part per billion (ppb).

Preservation is unnecessary for water samples. Samples must be kept cool at 4°C and may be stored for 7 days before extraction and 40 days after extraction. Standard Operating Procedures (SOPs) for these methods can be supplied upon request.

Quality Assurance

Laboratory

The ASL quality control requires the extraction and analysis of samples to be accompanied by laboratory fortified blanks, laboratory reagent blanks, laboratory fortified sample matrix (matrix spikes), quality control samples, and performance check solutions to evaluate and document data quality. On going data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

The laboratory must demonstrate on a frequency equivalent to every analytical batch that the measurement system is in control by analyzing a laboratory fortified blank (LFB) in duplicate or a matrix spike in duplicate (MSD). Surrogate analytes must be added to all samples, blanks, and spikes to ensure the integrity of the extraction procedure. Surrogate standard recoveries must be between 70% and 130% of the true value. The concentration of analytes detected in the laboratory reagent blank must be significantly below the designated method detection limit so as to be considered nondetectable. Once a year a quality control sample from an external source will be analyzed. This is accomplished through the Water Supply (WS) performance evaluation (PE) quality assurance program.

With the exception of the samples collected on June 29 and 30, 2004 all of the analytes spikes and surrogate standard recoveries were within acceptable ranges (70-120%). Samples from June 29 and 30 met holding times and QC requirements with some exceptions. Samples from Lawyer Creek, Big Canyon Creek, Jacks Creek, and the bottle blank exhibited lower than normal surrogate recoveries upon the first extraction and analysis (58-62%). Upon repeating these samples the lab determined that the dimethoate residues detected in the first extraction/analysis had degraded before the second extraction/analysis (10 days later; DEA and atrazine detects increased at nearly the same proportion as the increase in surrogate recovery for samples Big Canyon and Jacks Creek in the second extraction). The laboratory felt it best to report the results for the samples using data from the 1st extraction/analysis due to loss of the dimethoate residues after the 7 day holding time. The results of EPA 507/508 analyses showed dimethoate in five samples with only three samples (Catholic Creek, Big Canyon Creek, and Cottonwood Creek) having detections above the method detection limit of 0.05 ppb. All of the Dimethoate detections were well below any chronic or acute threshold for aquatic species.

Field Monitoring

Three different quality control (QC) techniques were conducted during the field monitoring portion of this project. During each sampling effort one duplicate sample was collected and submitted to the laboratory. At one site, additional water was collected and composited into the clean 2.5 gallon glass carboy. The water was then mixed and transferred into six 1-liter amber bottles (3 bottles/analyses). The duplicate sample was not identified as a duplicate and submitted to the laboratory for analyses. The duplicate samples are used to determine the validity of the analytical methods, and the accuracy of laboratory and monitoring personnel. The duplicate samples collected in the field were sent to the laboratory to determine the precision of the collection method and the analytical procedures. The relative percent difference (RPD) for all duplicates ranged from 0% to 44% (Table 2).

The other two QC samples consisted of bottle blanks and equipment blanks. At least one of these blank samples was collected on alternating sampling events. The bottle blanks consist of three laboratory cleaned amber 1-liter bottles filled in the field with Type II reagent water. Bottle blanks were analyzed to insure the cleanliness of the sample containers after laboratory cleaning, during field handling and transport.

The equipment blank was collected to insure proper clean up of field sampling equipment by the field crew. During a sampling event, equipment blanks were collected from either the DH-81 sampler or the DH-94 sampler. After collecting a sample the sampling unit was thoroughly cleansed as follows: scrubbing with Liquinox soap, deionized water rinse, acetone rinse followed by a final deionized water rinse. The 2.5 gallon glass composite container was also cleansed following the previously mentioned steps. The sampling unit was then filled with deionized water and the resultant water was transferred into the glass composite basin. This procedure was continued until sufficient water was transferred into the composite container (approximately 4 liters) to fill the three 1-liter sample bottles. All of the bottle and equipment blanks submitted to the laboratory, during this study, were found to have no trace (non-detects) of pesticide residue.

All resultant quality control samples were placed on ice within a cooler and transported to the laboratory with the normal sample load. Blanks were not identified as such and entered the laboratory with the normal sample load.

Table 2. Relative percent difference for duplicate analysis.

Date	Location	Compound	Result (ppb)	RPD	Date	Location	Compound	Result (ppb)	RPD
4/7/2004	Cottonwood	Atrazine	trace	0	5/19/2004	Catholic	2-4D	0.28	11
	duplicate	Atrazine	trace			duplicate	2-4D	0.25	
	Cottonwood	Atrazine Desethyl	trace	0		Catholic	Picloram	0.7	15
	duplicate	Atrazine Desethyl	trace			duplicate	Picloram	0.6	
4/20/2004	Cottonwood	Atrazine	trace	0	6/2/2004	Catholic	Metribuzin	0.052	0
	duplicate	Atrazine	trace			duplicate	Metribuzin	0.052	
	Cottonwood	Atrazine Desethyl	trace	0	6/16/2004	Catholic	Metribuzin	0.032	13
	duplicate	Atrazine Desethyl	trace			duplicate	Metribuzin	0.028	
	Cottonwood	Diuron	0.075	9.8		Catholic	Methomyl	0.051	44
	duplicate	Diuron	0.068			duplicate	Methomyl	0.08	
5/2/2004	Lapwai	ND	ND	0	6/30/2004	Catholic	Dimethoate	0.065	23
	duplicate	ND	ND	0		duplicate	duplicate	0.082	
5/19/2004	Catholic	Metribuzin	0.14	13	7/13/2004	Potlatch	all ND	0	0
	duplicate	Metribuzin	0.16			duplicate	all ND	0	

Results and Discussions

Pesticide Detections, Precipitation, and Hydrographs

Factors that result in transport of pesticides into the environment includes timing of applications, application rates, runoff potentials, and precipitation rates prior to and after application. The one factor that is available for evaluation for this pesticide study is precipitation rates. The Clearwater River Basin precipitation records for this study period were gathered from the Lewiston City Airport and an AgriMet station located at Dworshak Dam. The Lewiston airport had approximately 56 years of historical precipitation records while the Dworshak site had approximately 38 years. This historical data allows the comparison of the precipitation during the study (April through June 2004) with overall historical trends. On average when comparing the precipitation records from this year with historical averages, this years records show an increase in overall precipitation especially in the month of May 2004 (Table 3).

Table 3. Study area precipitation data.

Lewiston	56 Year Avg.	Year 2004	Dworshak	38 Year Avg.	Year 2004
April	1.21"	1.34"	April	2.32"	2.00"
May	1.47"	3.13"	May	2.40"	5.16"
June	1.37"	1.14"	June	1.73"	0.90"
Average	1.35"	1.87"	Average	2.15"	2.68"

Table 3 indicates that May 2004 had unusually heavy precipitation with Lewiston posting a 1.66" increase over the 56 year average and Dworshak at 2.76" above the 38 year average. Although the month of May increases were sizeable the overall increase for all three months as compared to the long term average was only 0.52" of precipitation.

Although the month of May 2004 precipitation amount increased considerably from historic figures the actual number of pesticide detects remained relatively stable with April at 13 detects, May 18 detects, and June 16 detects (Figure 2 and 3).

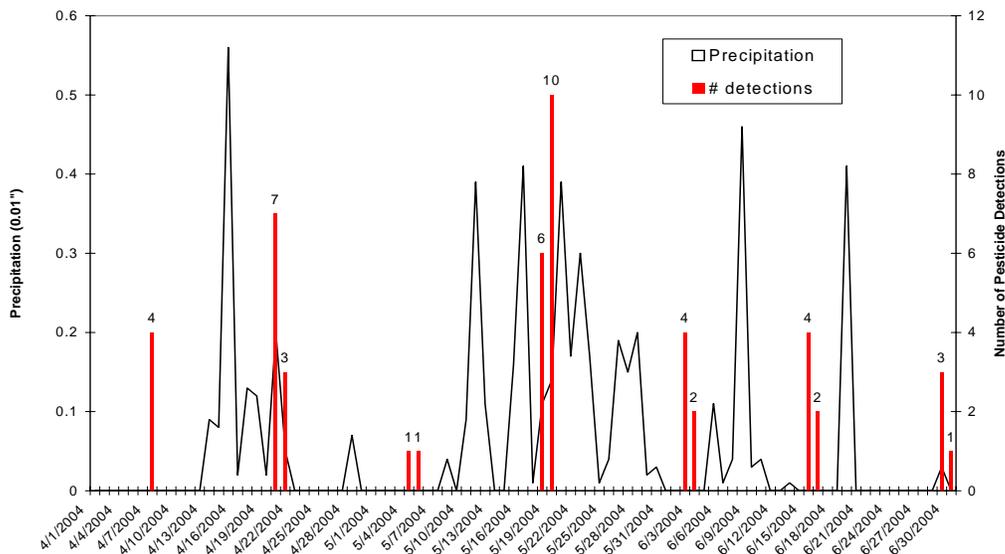


Figure 2. Lewiston Airport precipitation and pesticide detections.

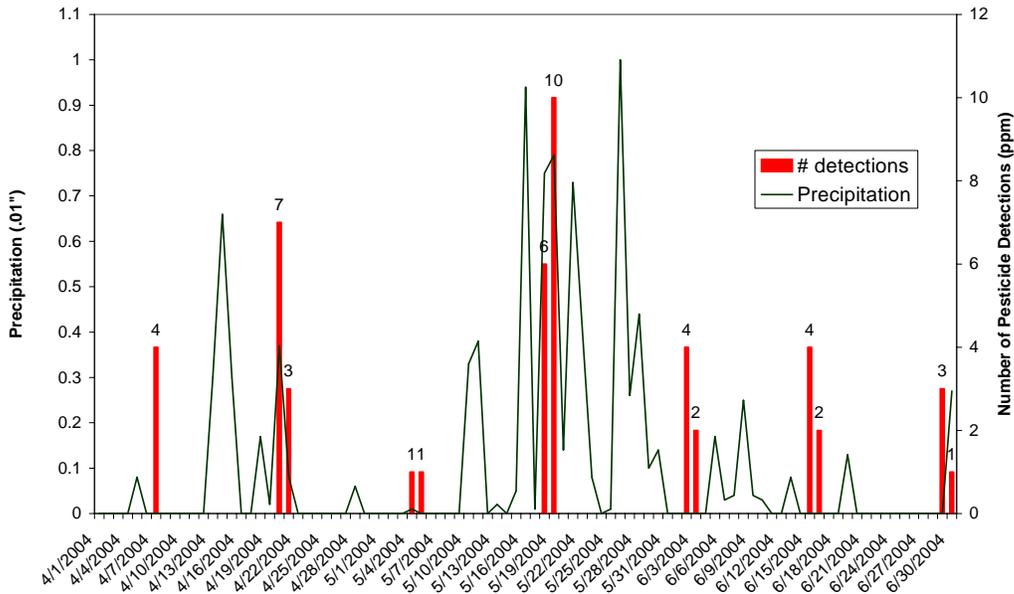


Figure 3. Dworshak AgiMet precipitation and pesticide detections.

The fact that the number of pesticide detects remained relatively equal for each month probably had more to do with the sampling schedule than the precipitation rates. With increased sampling frequency the quantity of pesticide detections probably would have increased especially in the month of May. During the month of May there were 20 out of 31 days of recorded precipitation. The large number of detections (16) recorded on May 18 and 19, 2004 occurred primarily because the sampling schedule coincided with a string of precipitation events. The number of days with precipitation in April was 10 while June had a total of 9 days.

It is difficult to predict pesticide detections based on the hydrograph of each creek. There were detections observed near the peak of the hydrograph along with detections on the rising limb of the hydrographs. There were also detections noted along the hydrograph that had nothing to do with an increase in discharge rate. Again, there may not have been sufficient collection dates to correlate positive detections with peak hydrograph activity. Figure 4 and 5 illustrate the hydrograph and pesticide detections for two of the monitoring sites that had the highest number of pesticide detections.

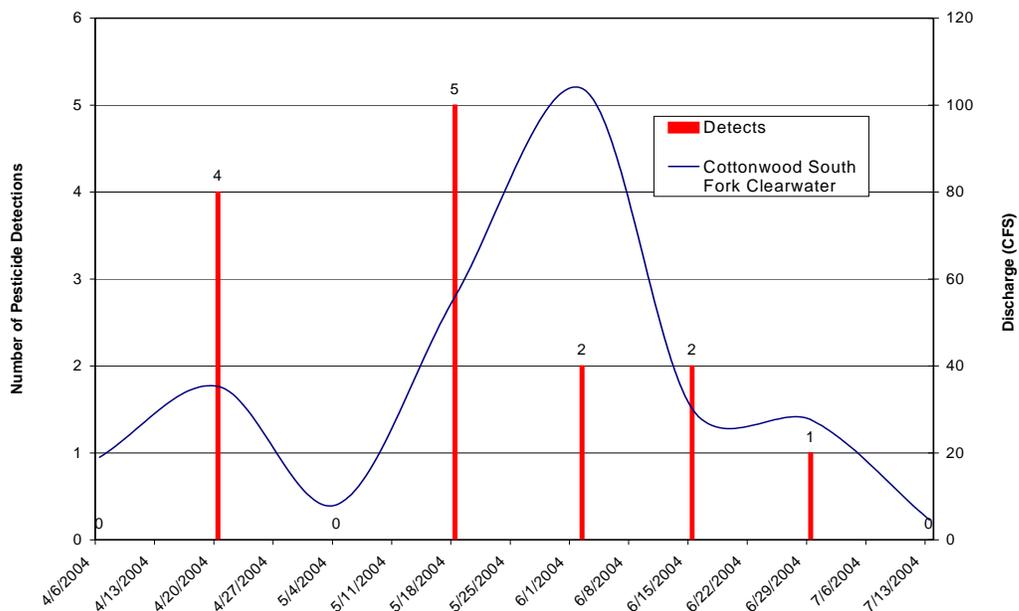


Figure 4. Cottonwood Creek South Fork Clearwater River Hydrograph and Pesticide Detections.

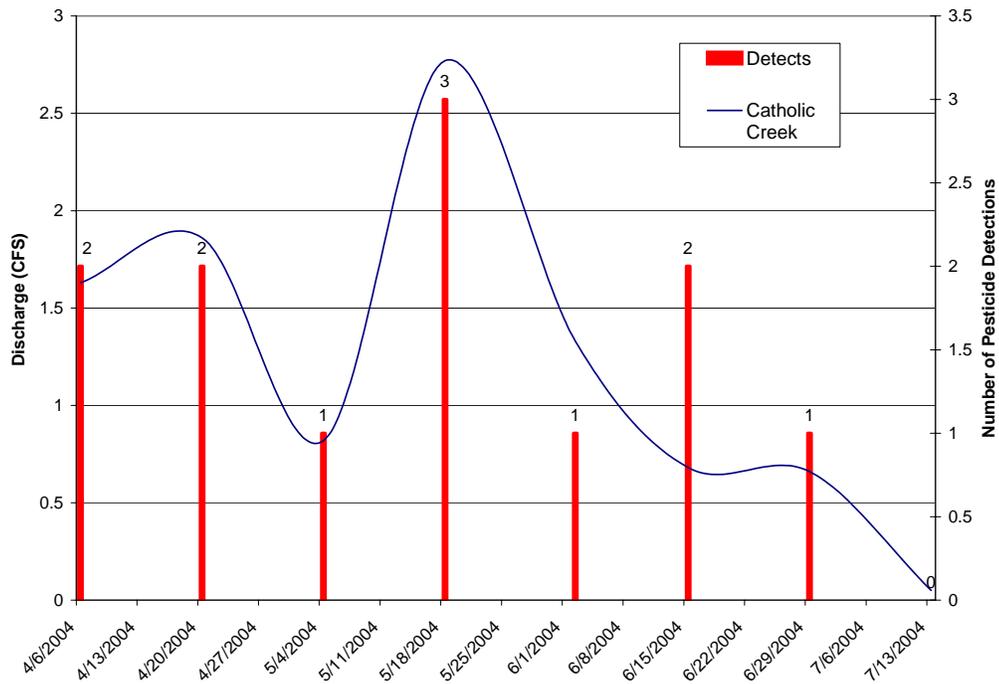


Figure 5. Catholic Creek Hydrograph and Pesticide Detections.

Pesticide Results

Metribuzin (11), Diuron (7), Dicamba (6), and Atrazine (5) were the most frequently detected pesticides in the study (Table 4). These four pesticides were detected greater than ten percent of the time (Table 4). Six of the thirteen pesticides detected occurred in two or more of the watersheds. All atrazine detections (5) were found in Big Canyon Creek. All Hexazinone detections (3) were found in the Potlatch River. The two Bromacil detections were found in Cottonwood Creek that flows into the South Fork of the Clearwater River. There were other watersheds that had multiple detections of certain pesticides. Most notable Cottonwood Creek that connects with the South Fork of the Clearwater River had four Dicamba detections, and Catholic Creek had five Metribuzin detects.

Table 4. Pesticide detections per watershed.

	Cottonwood SF Clearwater n = 8	Cottonwood Creek n = 8	Catholic Creek n = 8	Potlatch River n = 8	Big Canyon Creek n = 8	Lapwai Creek n = 8	Total	Frequency of Detects (%)
Atrazine					5		5	11
Bromacil	2						2	4
Dicamba	4		1	1			6	13
Dimethoate		1	1		1		3	6
Diuron	2	2		1		2	7	15
Hexazinone				3			3	6
Linuron			1				1	2
Methomyl			1		1		2	4
Metribuzin	3	2	5	1			11	23
Picloram	2	1	1				4	8
Simazine	1						1	2
Tralkoxydim			1				1	2
2,4-D			1				1	2
Total	14	6	12	6	7	2	47	

Table 4 also indicates which watershed received the largest number of pesticide detections during this study. The Cottonwood Creek that discharges into the South Fork of the Clearwater had 14 pesticide detections. All but one of the detections were herbicides that are used for annual and perennial broadleaf weeds and grasses. Picloram, a herbicide, used for woody plants and broad-leaved weeds was found in this drainage. The next highest pesticide detects occurred within Catholic Creek (12 detections) where there were 10 detections of herbicides and two detections of insecticides. There was a possible source observed for some of the herbicides and possibly insecticides that were detected within Catholic Creek. It was noted that a local landowner had been dumping grass clippings along the edge of the creek. These clipping could have contained some of the pesticides that are often found in lawn maintenance products. There were no pesticide detections on Lawyer or Jacks Creek during this study.

All of the pesticide concentrations reported during this study were well below any acute or chronic toxicity level for the chosen aquatic species (trout). The laboratory testing was conducted at the parts per trillion range (ppt), analytical results were reported in parts per billion (ppb). The referenced 96-hour lethal concentration (LC50) were reported in parts per million and converted to ppb for Table 5. To stay consistent, only data with available 96-hour LC50 were used for comparing toxicity levels. The 96-hour LC-50 represents the amount of chemical required to kill 50 percent of the fish in 96 hours. The data in Table 5 came from two sources: Extoxnet the Extension Toxicology Network and Toxicities of Agricultural Pesticides to Selected Aquatic Organisms, Morgan and Brunson 2002.

Table 5. Bioassay results and concentrations.

Compound	Pesticide ¹	Toxicity ²	Aquatic species ³	Bioassay type ⁴ Acute Levels	Total # of detects	Highest detect ⁵	Location of Highest Detection
Methomyl	I	MH	RT	3400 ppb	2	0.36 ppb	Big Canyon
Dimethoate	I	M	RT	6200 ppb	3	0.065 ppb	Catholic Creek
Simazine	H	PN	RT	>100000 ppb	1	0.042 ppb	Cottonwood S.F. Clearwater R.
Metribuzin	H	S	RT	64000-76000 ppb	11	0.29 ppb	Catholic Creek
Dicamba	H	L	RT	99000 ppb	6	0.76 ppb	Potlatch River
Bromacil	H	PN	RT	28000 ppb	2	0.079 ppb	Cottonwood S.F. Clearwater R.
Tralkoxydim	H	L	RT	not determined	1	0.17 ppb	Catholic Creek
Picloram	H	SM	RT	4000 ppb	4	0.75 ppb	Cottonwood S.F. Clearwater R.
Diuron	H	M	RT	4900 ppb	7	1.1 ppb	Potlatch River
Hexazinone	H	S	RT	320000 ppb	3	0.059 ppb	Potlatch River
Linuron	H	S	RT	16400 ppb	1	0.08 ppb	Catholic Creek
Atrazine	H	S	RT	4500 ppb	5	0.1 ppb	Big Canyon
2,4-D	H	S	CT	250 ppb	1	0.28 ppb	Catholic Creek

1. Pesticide type: H = herbicide; I = insecticide
 2. Toxicity: PN = practically non; MH = moderate to high; M = moderate; S = slight; L = low; SM = slight to moderate.
 3. Aquatic Species; RT = Rainbow Trout; CT = Cuthroat Trout
 4. Bioassay Type; 96 hr. LC50

Conclusions and Recommendations

Although there were detections of pesticide residue during this study none of the results indicated a serious acute or chronic toxicity concern for aquatic organisms within the study area. The analytical methods used for this study are very sensitive and are capable of detecting levels in the parts per trillion (ppt) range. The toxicity criteria (96 Hr. LC-50) used for the pesticides that were detected were all reported in the parts per million (ppm) range.

As previously mentioned the restricted scope of the sampling effort (both financial and manpower) did not allow ISDA to be present for sample collection during certain peak rain and discharge events. There is a strong possibility that a greater number of detections would have been confirmed if collection times coincided with significant precipitation events. This is not to say that higher concentrations of pesticides may have been detected but the number of detections would most likely have increased.

Although the pesticide concentrations found were very low, the fact that they were still detected in the environment should be of concern. The sublethal effects of many agricultural chemicals on aquatic life are unknown. Many factors influence the toxicity of chemicals to aquatic life and care must be exercised when utilizing pesticides. Toxicity to fish or shellfish can depend on the age of the species, size and overall health. Other contributors to toxicity include water temperature, pH, turbidity, and other physical and chemical parameters of the water. With careful management it is possible to protect crops from insects, disease, and weeds and still prevent pesticides from harming the aquatic environment.

Pesticide applicators should reduce the chance of pesticide drift and runoff entering aquatic systems by always reading and following the pesticide label directions. Other techniques to prevent pesticides from entering waterways are as follows:

- Minimize spraying when the temperature is high which may cause off site release.
- Spray when the wind speed is low and blowing away from the water body.
- If precipitation is expected delay application.
- Reduce runoff by using no-tillage or minimum tillage cropping systems.
- Use soil incorporation methods and utilize buffers to prevent product loss.
- Calibrate application equipment.
- Utilize Integrated Pest Management (IPM), scout fields and apply chemicals at the correct time and rate
- Review and implement USDA Natural Resource Conservation Service Pesticide Management (595) component practice.
- Contact ISDA, University of Idaho Extension, and Soil and Water Conservation Districts for questions, recommendations, and technical assistance.

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