Surveillance for current-use pesticides in Maine's freshwater resources along a population gradient.

Results of 2019 environmental sampling project.

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Maine Board of Pesticides Control

Department of Agriculture, Conservation & Forestry

Executive Summary

Surface waters around urban areas are frequently degraded due to non-point source pollutants. One aspect of this contamination originates from the use of pesticides, both inside and outside of the home. While Maine is a rural state, many of our towns and cities were built alongside rivers, placing these aquatic resources at risk for contamination. Recent work elsewhere has demonstrated greater numbers of pesticide detections in areas with higher concentrations of people.

We surveyed the rivers and streams of 10 cities in Maine during the summer of 2019 for the presence of pesticides. We collected grab samples of water and sediment as well as deploying a passive sampler in the water column of each city's major river. The population of the cities spanned from 7,000 to 91,000 residents and where distributed across the state.

We found detectable levels of pesticides in each city. We found greater variety of pesticide types in areas with more people. Biddeford was the location with the greatest number of pesticides where 22 pesticide types were found. Portland, Bangor, and Lewiston-Auburn were close behind with 18 pesticide types found at each. At the other end of the spectrum Ellsworth, Farmington, and Presque Isle all had either ten or eleven pesticide types present. In the sediments the pyrethroid bifenthrin was found in eight out of ten locations.

In several locations pesticide concentrations were above threshold values, indicating negative ecological changes are predicted to start occurring at those sites. The two pesticides exceeding threshold values were bifenthrin in sediment and imidacloprid in water. The locations of these exceedances should have follow up re-sampling to verify the patterns found and more fully understand the scope of the issue.

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Introduction

Maine is a largely rural state with low relative acreage in agriculture. Current national data point to residential and agricultural areas as the two largest contributors to current-use pesticide burdens in surface water. Insecticide contamination is found to predominate in residential areas whereas herbicide contamination is predominantly found in association with agricultural areas (VanMetre et al. 2018). Due to the generally low potential for contamination, Maine is infrequently included in national-scale assessments of current-use pesticides in surface waters and its pesticide distribution patterns in the state are relatively undescribed.

Current-use pesticides tend to have short persistence in the environment which adds to the difficulty in comprehensive sampling programs being able to accurately determine pesticide contamination trends. Approaches to capture current-use pesticide occurrences include: high frequency sampling, snapshot sampling, targeted sampling, and passive sampling. This project could not rely on targeted sampling because we are attempting to capture a complete picture of all pesticide uses and thus knowing when and where pesticides are used becomes impossible. High-frequency sampling is ideal for capturing seasonal fluctuations in current-use pesticide concentrations; however, high-frequency sampling is expensive. The costs for daily or weekly samples taken across our ten locations for the summer season would add up to hundreds of thousands of dollars. The ME BPC typically relies on snapshot sampling (single samples taken in many locations at once) in order to maximize coverage per budget constraints. Passive sampling is a newer and growing technology for environmental sampling. Passive samplers are devices deployed for long periods of time, around 30 days, which collect and integrate information the entire time they are deployed. It is possible to detect the presence of chemicals that occur at lower concentrations or shorter durations because of the longer exposure for the sampling device.

In 2019, the Maine Board of Pesticides Control (BPC) surveyed ten river locations spread across the state in order to establish general patterns of current-use pesticide occurrences. Locations were selected in order to capture the differences in current-use pesticide occurrence across range of population densities. In addition to our traditional grab samples, passive samplers were deployed in the rivers for approximately one month in order to compare the passive sampling results to our traditional grab sampling program.

Objectives of this study were to:

1) Establish patterns of current-use pesticides in surface water
2) Determine usefulness of passive sampling augmentation to our sampling efforts

Methods

Site selection

Maine's larger cities and towns tend to be situated on water, mostly rivers. We categorized ten cities by population, based on capturing a range of population sizes with the intention to have locations distributed across the state. Spreading sites across the state creates a database that is more representative of the state as a whole. Figure 1 shows the ten locations selected, city population, and corresponding river names.

Specific sampling sites were selected based on access, the proximity of the passive sampler to corresponding grab and sediment sampling sites, and security of the passive sampler. Deployment of passive samplers in the Kennebec, Androscoggin, and Union Rivers required access to boats. Site conditions in Waterville, Auburn-Lewiston, and Sanford required sampling down river at the next available point of access. Sites were located below wastewater treatment plants. Some cities lacked streams or streams located above the river sample sites. A storm culvert in Ellsworth was selected for sampling because it accepts water from the downtown area, its proximity to the river, and the only input to the river with water other than Card Brook.

Surface water sampling

At each city location, a variety of samples were taken. Grab samples from two tributary streams upstream of the passive sampler location were collected. In all, grab samples of water were collected from nineteen streams, one storm drain, and each of the ten rivers halfway through the month-long sampling time used by the passive sampler. Grab samples were also collected from each river at deployment and retrieval of the passive sampler.

Surface water samples were collected into 950 ml amber, glass bottles with a Teflon-lined cap, certified as pre-cleaned for the collection pesticides. Bottles were held beneath the water's surface at the river's edge. If it was possible to collect the sample from farther out from the shore, such as wading or from a dock, samples were taken over the deepest water practicable. Glyphosate samples were taken at the same time in 120 ml polypropylene sampling bottles. Detailed notes of the location during sampling were taken and are available upon request.

Sediment sampling

Sediment samples were composites produced on-site. Briefly, the top 3 cm of sediment was collected randomly within a 50 ft segment along the water's edge. The sediment was homogenized to blend it uniformly, and a portion placed into the 1-quart metal can. A second small sediment sample was taken for organic carbon and particle size analysis.

Passive sampling

Passive sampling was conducted using POCIS (polar organic chemical integrative sampler) samplers. The POCIS sampler is a solid phase sorbent material (Oasis HLB) surrounded by thin polyethersulfone membranes. This resin predominantly absorbs polar chemicals or freely dissolved chemicals with octanol-water partitional coefficients less than three. The POCIS samplers were deployed with metal housings rented from Environmental Sampling Technologies, Inc., St. Joseph, MO 64501. Housings were emplaced to float two to three feet deep in the water column using a weighted buoy system or attached to dock supports.

Passive sampling required a tremendous input of time and collaboration with Casco Bay Estuary, the Maine Department of Environmental Protection (DEP), two Maine game wardens, a harbor master, dam owner, two municipal waste water treatment facilities, and a private citizen to deploy the samplers. The first passive sampler was placed August 6, 2019 and the last passive sampler was removed September 12, 2019.

Duplicate frequency and field blanks

For both sediment and grab samples duplicates were taken on a one in twenty basis such that every 100 samples requires the collection of 5 field blanks.

Ten passive sampling housings were deployed in the study, each housing contained three replicates. The reported concentrations are averages of the three replicates. In addition, one POCIS resin sampler was dedicated as a trip blank and one as a control which remained in an in-house freezer. At each location the canister holding the trip blank was opened and exposed to the air while the deployed sampler was emplaced or removed, then it was carefully repackaged. The laboratory was sent several blank samplers to serve as laboratory blanks and for method development.

Sample storage and transfer

Following collection, samples were placed in coolers with ice and transferred to 0°C freezers. Frozen samples were shipped, on ice, by overnight delivery to the laboratory for analysis.

Laboratory analyses

Sediment characterization

The University of Maine Analytical Laboratory, Orono, Maine, categorized the sediment samples for total organic carbon and particle size distribution. Total organic carbon was run by thermal partitioning and combustion analysis (EPA 440.0) at 1,350°C. Coarse fragments (> 2 mm) were measured gravimetrically by sieving the entire sample. Sand, silt, and clay were run after overnight dispersion in Calgon solution. Clay was determined by the hydrometer method. Sand was determined gravimetrically after wet sieving. Silt is calculated as the remainder of the sample. Particle size categories are from the USDA. Results are presented as percent in the dried sample. Sand, silt, clay, and coarse fragments constitute 100% of each sample.

Pesticide active ingredient characterization

The University of Montana Agricultural Laboratory (Helena, MT) performed four separate analyses: a multi-residue panel for 102 analytes on the surface water samples, glyphosate on the surface water samples, a multi-residue panel of 102 analytes on the POCIS resin, and a 21-analyte panel of pyrethroids on the sediment samples. The surface water and POCIS samples (after extraction as per EST Inc. protocol) were processed following the Montana Department of Agriculture's "Universal Method for the Determination of Polar Pesticides in Water Using Solid Phase Extraction and Liquid Chromatography/Mass Spectrometry/ Mass Spectrometry" procedures. POCIS results are reported as ng/POCIS and each of the three POCIS resins are averaged. Passive sampling results are reported as presence/absence. Rate sampling conversions were not attempted on the results due to the lack of available rate sampling values for many current use pesticides. The sediment samples were processed following the Montana Department of Agriculture's "Determination of Pyrethroids in Sediment Using Solid Phase Extraction and GC/MS/NCI and /or GC/MS/MS E1" PYR_SI, Revision 2: January, 2014 methodology.

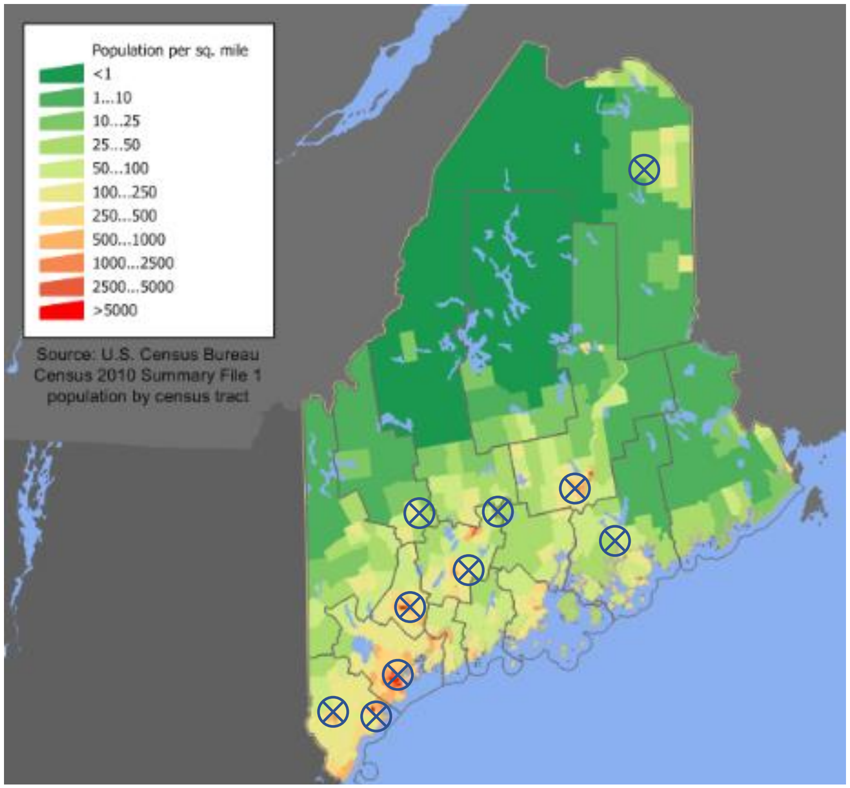
Complete lists of all the analytes, and their respective reporting limits, included in the above chemical assays are available in Appendix 1.

Budget

This work was funded by an EPA Region 1 grant that typically supports several BPC functions under the FIFRA Cooperative Agreement.

Statistics

Simple summary statistics and linear regressions were performed with Microsoft 365 Excel Data Analysis. Simple linear regression was used to test if human population significantly predicted number of pesticide detections, in addition to, the number of unique pesticide types.



The circles with the 'X' indicate locations included in this study.

Population Centers*	Waterbody	Population†
Portland / South Portland	Fore River	91,196
Lewiston-Auburn (Durham)	Androscoggin River	59,647
Bangor / Brewer / Orono (Hampden)	Penobscot River	42,521
Biddeford / Saco	Saco River	39,759
Sanford	Mousam River	20,798
Augusta	Kennebec River	19,136
Waterville (Sidney)	Kennebec River	15,722
Presque Isle	Aroostook River	9,692
Ellsworth	Union River	7,760
Farmington	Sandy River	7,741

*Locations in parentheses indicate actual sampling location.
†Population data from 2010 US Census

Figure 1. Sampling locations and associated river names. Map displays populations density across the state in terms of residents per square mile.

Results

Surface water

All locations had at least one current-use pesticide detected. Grab sampling resulted in an overall lower detection rate. POCIS data indicate atrazine, imidacloprid, metolachlor, and prometon contamination is widespread in Maine, occurring in almost every POCIS sample. Table 1 contains summary data for surface water grab sample detections, it displays the total number of occurrences and value of each detection. Table 2 contains summary data for POCIS generated current-use pesticide detections, it displays the type of pesticides found in each location. The complete suite of all analytes tested and their respective reporting limits are listed in Appendix 1.

Surface water grab samples

Forty-six surface water grab samples were sent to the laboratory and each was analyzed for 102 unique current-use pesticides and their degradation products resulting in 4,692 individual tests. From that constellation of 4,692 tests performed there were 200 unique detections. The 102 possible analytes represent a total of 77 unique pesticide product active ingredients included in these analyses. Twenty-five of the test analytes included in the analyses are the breakdown products of 77 parent active ingredients. Twenty-eight current-use pesticide product active ingredients were identified from grab sampling. The most commonly occurring analytes include (number in parentheses indicates number of times it was detected): atrazine and degradates (48), metolachlor and degradates (31), imazapyr (16), imidacloprid (14), prometon (13), and 2,4-D (13). Other active ingredients detected less frequently were: bentazon, bromacil, diuron, fipronil, hexazinone, imazamethabenz acid, imazapic, indaziflam, isoxaben, MCPA, MCPP, metalxyl, methomyl, metsulfuron methyl, propiconazole, pyrasulfotole, sulfometuron methyl, tebuconazole, tebuthiuron, terbacil, tetraconazole, and triclopyr.

Of the 28 current-use products identified by grab sampling 19 of them also appear as active ingredients on the state's Groundwater Advisory List. One product, imazamethabenz acid, does not appear to be included in any products currently or formerly registered in Maine. The majority of the 28 current-use products identified are herbicides with 3 of the detections from insecticides and 4 from fungicides.

No strong correlation can be seen between city population and the number of pesticide detections (Figure 2) There was no significant relationship between the city population size and the number of pesticide detections ($F_{(1,8)}$= 0.546, P= 0.481). The unexpectedly high number of detections in Ellsworth is likely due to a sample location that was at a storm drain, removing that one sample reduced the detections from 31 to 16.

Table 1. Current-use pesticides detected in Maine rivers with grab sampling during summer 2019. Only detections are shown. All units µg/L (ppb). 'Q' indicates a detection adequate for identification but not sufficient for quantification.

	Augusta					Bangor				
Field ID	190826MET06A	190812MET02A	190909LRS03A	190826MET05A	190826MET07A	190822MET01A	190806NJT01A	190822MET02A	190822MET03A	190904MET01A
Lab ID	AB91982	AB91809	AB92268	AB91981	AB91983	AB91934	AB91740	AB91935	AB91936	AB92118
2,4-D						Q			Q	
Atrazine	0.0026	Q		Q		0.0081		Q		
DEA		Q		Q	Q	Q			Q	
HA	Q				Q	Q			0.0064	
Bentazon										
Bromacil										
Diuron									Q	
Fipronil									Q	
Hexazinone										
Imazamethabenz acid										
Imazapic									Q	
Imazapyr	0.0038				Q	0.0035		Q	0.019	
Imidacloprid	0.0039				0.0059				0.0056	
Indaziflam										
Isoxaben						0.0049				
MCPA									0.019	
MCPP										
Metalaxyl										
Methomyl										
Metolachlor						Q				
Metolachlor ESA	Q	0.012	0.011	0.011	Q	0.049		Q		Q
Metolachlor OA						Q				
Metsulfuron methyl									Q	
Prometon	Q				0.0011				0.001	
Propiconazole										
Pyrasulfotole										
Sulfometuron methyl										
Tebuconazole									Q	
Tebuthiuron										
Terbacil										
Tetraconazole										
Triclopyr									0.047	
Total Detections	**19**					**26**				

Table 1. Continued

	Biddeford-Saco					Ellsworth				
Field ID	190823MET02A	190823MET01A	190823MET03A	190906ARP01A	190808MET01A	190820HDN03A	190806HDN01A	190820HDN01A	190820HDN02A	190905HDN01A
Lab ID	AB91941	AB91940	AB91942	AB92121	AB91807	AB91862	AB91739	AB91860	AB91861	AB92119
2,4-D		0.0094	0.016		Q			0.0098		
Atrazine								0.0074	0.0023	
DEA					Q			0.0047	0.0018	
HA		Q				Q		0.0066	0.0055	
Bentazon								0.078		
Bromacil										
Diuron										
Fipronil									Q	
Hexazinone						0.049	0.0025			0.0021
Imazamethabenz acid								Q		
Imazapic		Q	Q						0.0037	
Imazapyr		0.012	0.0037			0.0036			0.01	
Imidacloprid	0.0024	0.007	0.11		0.0021				0.0022	
Indaziflam		Q								
Isoxaben								Q		
MCPA										
MCPP		0.005	0.0049							
Metalaxyl										
Methomyl										
Metolachlor										
Metolachlor ESA	0.027			0.023	0.025			0.11	Q	
Metolachlor OA								Q		
Metsulfuron methyl										
Prometon		0.0031	0.0021					Q	0.0035	
Propiconazole								Q		
Pyrasulfotole								Q		
Sulfometuron methyl		0.004							0.0041	
Tebuconazole		Q						Q		
Tebuthiuron								0.0013		
Terbacil						Q				
Tetraconazole								Q		
Triclopyr										
Total Detections	**23**					**31**				

Table 1. Continued

	Farmington					Lewiston-Auburn				
Field ID	190815MET0 1A	190912LRS0 1A	190828MET0 1A	190828MET0 2A	190828MET0 3A	190813MET0 1A	190827MET0 3A	190911ARP0 1A	190827MET0 1A	190827MET0 2A
Lab ID	AB91853	AB92276	AB92000	AB92001	AB92002	AB91815	AB91972	AB92271	AB91984	AB91971
2,4-D									0.048	
Atrazine		Q				Q	Q		Q	
DEA						Q	Q		Q	
HA									Q	
Bentazon										
Bromacil										
Diuron										
Fipronil										
Hexazinone	0.0024									
Imazamethabenz acid										
Imazapic									Q	
Imazapyr									0.016	
Imidacloprid						Q		0.0097		
Indaziflam										
Isoxaben										
MCPA										
MCPP									0.0085	
Metalaxyl										
Methomyl										
Metolachlor										
Metolachlor ESA						0.024	0.047	0.02	Q	0.019
Metolachlor OA							Q			
Metsulfuron methyl										
Prometon					Q				0.0039	
Propiconazole										
Pyrasulfotole										
Sulfometuron methyl									0.003	
Tebuconazole									0.02	
Tebuthiuron										0.0017
Terbacil										
Tetraconazole										
Triclopyr										
Total Detections	**3**					**23**				

Table 1. Continued

	Portland					Presque Isle				
Field ID	190821ARP02A	190821ARP03A	190807MET01A	190821ARP01A	190906ARP02A	190814MET01A	190910KRB01A	190828KRB01A	190828KRB03A	190828KRB01A
Lab ID	AB91903	AB91904	AB91741	AB91902	AB92122	AB91818	AB92269	AB92003	AB92005	AB92004
2,4-D	Q	0.059								
Atrazine			Q							Q
DEA										Q
HA	Q	Q							Q	
Bentazon										
Bromacil		0.013								
Diuron	Q	0.01								
Fipronil										
Hexazinone									Q	Q
Imazamethabenz acid										
Imazapic	0.0032	Q								
Imazapyr	0.0035	0.011					Q		0.014	0.0078
Imidacloprid		0.0081								
Indaziflam		Q								
Isoxaben						Q				
MCPA		Q								
MCPP		0.02								
Metalaxyl										Q
Methomyl									0.016	
Metolachlor										
Metolachlor ESA									0.0093	0.011
Metolachlor OA										
Metsulfuron methyl										
Prometon	0.0011	0.0011							0.01	0.0019
Propiconazole										
Pyrasulfotole										
Sulfometuron methyl		Q							0.0042	
Tebuconazole	0.028									
Tebuthiuron										
Terbacil										
Tetraconazole										
Triclopyr										
Total Detections	**20**					**16**				

Table 1. Continued

	Sanford					Waterville				
Field ID	190827MET04A	190813MET03A	190827MET06A	190911ARP02A	190827MET05A	190812MET01A	190826MET04A	190909LRS01A	190826MET02A	190826MET01A
Lab ID	AB91973	AB91817	AB91975	AB92272	AB91974	AB91808	AB91980	AB92266	AB91978	AB91977
2,4-D		0.018	0.019	0.0093					0.04	
Atrazine						Q	Q	Q	Q	Q
DEA						Q	Q		Q	Q
HA									Q	Q
Bentazon										
Bromacil										
Diuron		0.0078	0.0097	0.015						
Fipronil		0.0027	0.0029	0.0029						
Hexazinone										
Imazamethabenz acid										
Imazapic					Q					
Imazapyr									Q	
Imidacloprid		0.007	0.0074		0.0026					
Indaziflam										
Isoxaben		Q		0.011						
MCPA									0.0055	
MCPP									0.0082	
Metalaxyl										
Methomyl										
Metolachlor										
Metolachlor ESA	0.01					0.012	0.011	0.016	0.0077	0.028
Metolachlor OA										
Metsulfuron methyl										
Prometon										
Propiconazole										
Pyrasulfotole										
Sulfometuron methyl										
Tebuconazole										
Tebuthiuron										
Terbacil										
Tetraconazole										
Triclopyr										
Total Detections	**16**					**20**				

Table 2. Current-use pesticides detected in Maine rivers with POCIS passive samplers. Checkmarks indicate a detection (in at least one of the three replicates). Only analytes with detections are shown; complete list of tested analytes and reporting limits are listed in Appendix 1.

Detected Analyte	**Augusta**	**Bangor**	**Biddeford -Saco**	**Ellsworth**	**Farmington**	**Lewiston-Auburn**	**Portland**	**Presque Isle**	**Sanford**	**Waterville**
2,4-D			x						x	x
Alachlor ESA			x							
Atrazine	x	x	x	x	x	x	x	x	x	x
Deethyl atrazine	x	x	x	x	x	x	x	x	x	x
Hydroxy atrazine	x	x	x	x	x	x		x	x	x
Azoxystrobin	x	x	x	x	x	x	x	x	x	x
Clothianidin						x			x	
Dimethenamid			x							
Diuron	x	x	x	x	x	x	x		x	x
Fipronil	x	x	x	x	x	x	x		x	x
Fipronil sulfone									x	
Fluroxypyr					x					
Hexazinone	x	x	x	x		x	x	x		x
Imidacloprid	x	x	x		x	x	x		x	x
Indaziflam		x	x				x			x
MCPP		x	x				x			
Metalaxyl								x		
Methomyl										
Methoxyfenozide										
Metolachlor	x	x	x			x				x
Metolachlor ESA	x	x	x		x	x	x	x	x	x
Metolachlor OA			x							
Prometon	x	x	x	x	x	x	x	x	x	x
Propiconazole	x	x	x			x	x		x	x
Simazine	x	x	x			x	x	x	x	x
Sulfometuron methyl	x	x	x	x		x	x	x		x
Tebuconazole	x	x	x	x	x	x	x		x	x
Tebuthiuron			x			x	x			x
Total Unique Analytes	**15**	**18**	**22**	**10**	**11**	**18**	**16**	**10**	**15**	**18**

Passive Sampling

Passive sampling produced data from a possible combination of 3,264 possible tests (number of analytes available versus 32 samples processed). Of the possible 102 analytes, 77 unique current-use pesticide products were represented (after combining metabolites together with their parent). There were 439 total detections from this pool of 3,264 combinations.

When metabolites are added back in with the parent compound and the total number of occurrences are combined, the passive sampling results can be described as follows: no study site major river had fewer than eight current-use pesticides; the maximum number of pesticides found in a sample was 18; the average number of current-use pesticide products in each sample was 13.

Current-use pesticide products and their metabolites occurring in 90% or more of the samples include: atrazine and its breakdown product deethyl atrazine, azoxystrobin, diuron, fipronil, metolachlor ESA, prometon, and tebuconazole. Runners up that were detected in 50% or more the samples include the above plus: hexazinone, imidacloprid, metolachlor, propiconazole, simazine, and sulfometuron methyl. Less frequently detected current-use pesticides and their breakdown products include: 2,4-D, alachlor ESA, chlothianidin, thiamethoxam, dimethenamid, fipronil sulfone, fluroxypyr, indaziflam, MCPP (mecoprop), metalaxyl, metolachlor OA, tebuthiuon, and tetraconazole.

Passive sampling data presented here only contain presence or absence data. After combining the degradates with the parents, the number of unique pesticide active ingredients can be seen for each city (Figure 3). There was a significant relationship between the city population size and the number of unique pesticides detected (R^2= 0.43, $F_{(1,8)}$= 5.9, P= 0.041).

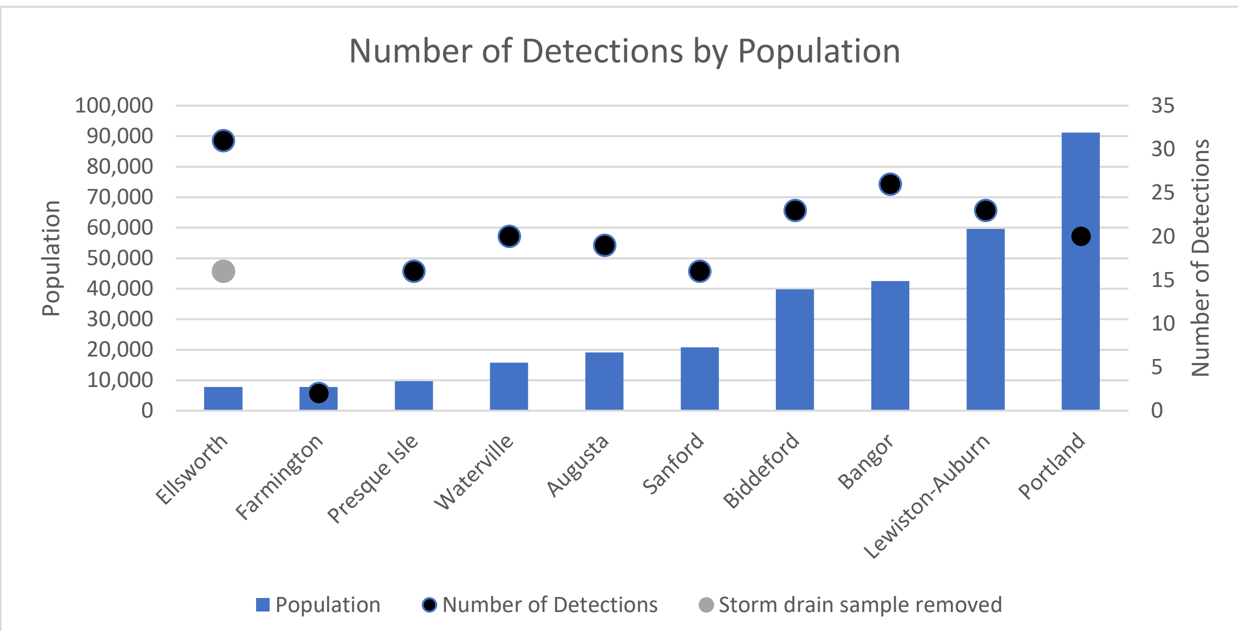


Figure 2. Number of analyte detections in surface water grab samples across the range of population centers. Bars represent the number of residents. Circles represent the number of times all of the samples from a city detected a pesticide. Five samples were taken at each city location. The gray circle represents the Ellsworth totals with a grab sample removed, see text for discussion.

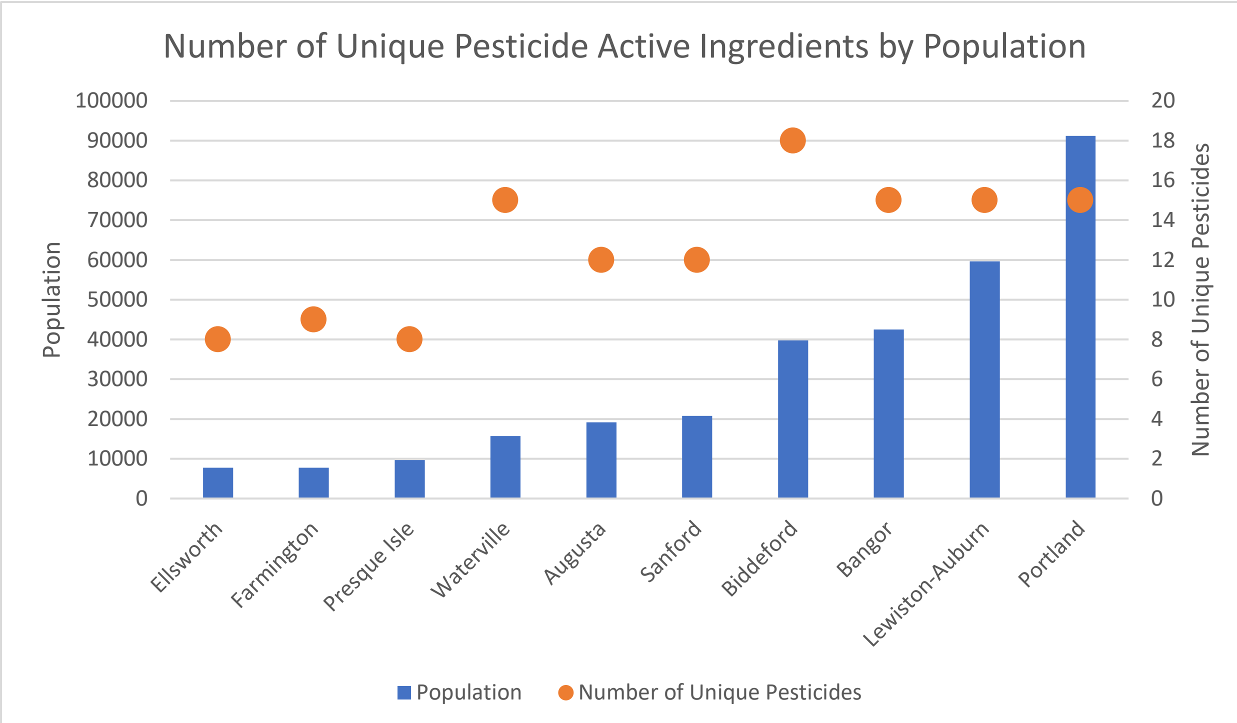


Figure 3. Number of unique pesticide products identified in surface water by passive sampling across the range of population centers. Bars represent the number of residents. Orange circles represent the number of different types of pesticides present. One POCIS sampler was used in each city, where it was deployed for one month.

Glyphosate

No glyphosate residues were detected in any of the locations. The primary breakdown product of glyphosate is AMPA; no AMPA residues were detected. Glufosinate is an herbicide similar to glyphosate requiring the same laboratory preparation as glyphosate. There were no detectable glufosinate residues. The reporting limits for these analyses are listed in Appendix 1.

Sediment

Bifenthrin was detected in eight of ten sediment samples. No other pyrethroids were detected (Table 3). Appendix 2 contains sediment characteristic data, including organic carbon concentrations.

Duplicates and blanks

No field blanks or trip blanks had detectable concentrations of the surveyed pesticides.

Table 3. Sediment sample results assessing pyrethroids and piperonyl butoxide in Maine rivers during summer 2019. Results are reported in ng/g (ppb) on a dry weight basis as well as organic carbon normalized.

	Reporting Limit (ng/g)	Augusta	Bangor	Biddeford	Ellsworth	Farmington	Lewiston-Auburn	Portland	Presque Isle	Sanford	Waterville
Percent TOC		0.73	3.58	0.31	3.93	0.25	0.09	1	0.85	5.23	0.53
Allethrin	0.20										
Bifenthrin	0.045	1.3	0.91	0.46	0.67		0.058	0.23	0.059		0.084
Bifenthrin ng/g-OC		178.1	25.4	148.4	17.0		64.4	23.0	6.9		15.8
Cyfluthrin	0.20										
Cyhalothrin	0.27										
Cypermethrin	0.20										
Deltamethrin	0.40										
Fenpropathrin	0.20										
Fenvalerate	0.13										
cis-Permethrin	0.20										
trans-Permthrin	0.20										
Phenothrin	2.0										
Piperonyl butoxide	2.0										
Prallethrin	0.20										
Resmethrin	2.0										
Tetramethrin	0.14										

Discussion

This project found current-use pesticides are present Maine waters, a pattern that is consistent with stream data throughout the United States (USGS 2020). There was significant effect of city population size on number of different types of pesticide contamination in surface water. Cities with higher population sizes typically had a greater number of different pesticides occurring in their major river. Two of the pesticide active ingredients were present at levels that could threaten aquatic organisms (EPA 2020). Specifically, bifenthrin exceeded the Threshold Effects Concentration of 0.17 ug/g-oc with a concentration of 0.18ug/g-oc in one city. Imidacloprid exceeded the invertebrate chronic Aquatic Life Benchmark of 0.01 ppb with a concentration of 0.11 ppb. These patterns should be understood relative to the limitations of the current study. Given the small number of samples it is suggested the reader recognize that other natural processes could be the cause for certain patterns observed throughout the current study.

Thresholds

EPA's Aquatic Life Benchmark (ALB) threshold values are intended to serve as a precautionary measure, prompting further investigation. When environmental values equal the ALB for a given taxon it is expected that effects could start to be seen in that population. However, marked species differences to chemicals means that the ecosystem responses cannot be completely predicted based on ALB values alone. Some species will be more or less sensitive to any given chemical, additional studies into ecosystem wide effects are often warranted following the detection of a chemical above its Aquatic Life Benchmark.

Bifenthrin was found in the sediments of eight out of ten study locations. Previous collections of sediment along the Maine coast have also indicated repeated detections of bifenthrin (BPC 2017). Bifenthrin is a commonly used mosquito and tick outdoor residential yard treatment and is additionally used in agriculture. Bifenthrin takes longer than many other current-use pesticides to degrade in the environment. Sunlight effectively degrades bifenthrin, but it is not hydrolyzed by water or biodegraded by microorganisms to any great extent, meaning it tends to be fairly stable and not breakdown when in dark locations that are either wet or dry. Bifenthrin is only slightly soluble in water and is typically only found in sediments, not the overlaying water. Predicting toxicity from chemicals displaying this pattern is not straightforward. The bulk of this chemical stays locked-up tightly bound to sediment particles. However, there exists an equilibrium that allows a small amount of the bifenthrin to enter the water column over time. EPA's Aquatic Life Benchmark is an aquatic value, based on concentrations in water, not sediment. The comparison of sediment values to aquatic benchmarks was used to identify potential issues but it does not accurately predict toxicity.

Sediment threshold values, which are similar to the Aquatic Life Benchmark values, have been developed for select pesticides including bifenthrin. Nowell et al. (2016) present an Integrated Threshold Effects Benchmark value of bifenthrin at 0.17 µg/g-oc and an Integrated Likely Effects Benchmark at 0.6 µg/g-oc. Exceeding the Likely Effects Benchmark would indicate a high probability of adverse effects and environmental concentrations below the Threshold Effects Benchmark are unlikely to result in adverse effects. After normalizing the bifenthrin values with each site's total organic carbon concentration in the sediments, our study indicates that while bifenthrin is consistently present and it can reach concerning concentrations in some Maine locations. However, the majority of the bifenthrin detections remain below levels of concern. The mean concentration across all eight sites with bifenthrin detections was 0.06 µg/g-oc, further indicating the relative lack of expected effects on aquatic organisms (values lower than 0.6 µg/g-oc are considered unlikely to result in any adverse effects). However, for one location the concentration of bifenthrin has reached the Threshold Effects Benchmark. Additional sampling in the area is warranted because these results represent only a single sample.

Imidacloprid was found in eight of the ten cities and exceeded the Aquatic Life Benchmark in one city (Biddeford). The highest measured value was 0.11 ppb exceeding the invertebrate chronic Aquatic Life Benchmark of 0.01 ppb. As a highly effective insecticide, imidacloprid is known to be highly toxic to aquatic invertebrates. Other aquatic organisms are unlikely to experience effects from these imidacloprid exposures due to its lower hazard potential to vertebrates and plants. Among samples with imidacloprid detections, the average concentration was 0.013 ppb. Similar to the issue with bifenthrin, the majority of locations would not be expected to be seeing negative effects but there are locations in Maine where the concentration was elevated to a concerning level and additional sampling is needed.

Trends

Of the pyrethroid insecticides, only the longer-lived bifenthrin was found in sediments, however, as with previous trends it is ubiquitously found in most locations. For surface waters, atrazine, imidacloprid, prometon, diuron, fipronil, and metolachlor occur almost ubiquitously. Pervasive occurrence in the environment can arise from pesticide products that degrade quickly but are often replenished or from pesticides that are very persistent by their chemical nature. Surface water testing is also more likely to detect those pesticides with the chemical properties that allow it to dissolve, move, and persist better in water.

This study is limited by the number of samples. We took roughly five surface water samples in each of the ten cities. The samples represented different tributaries as well as the major river in the city. The passive samples (POCIS) and the grab sample of each major river were located downstream of the sampled tributaries. The similarity of detections and between grab samples and POCIS samples were variable. The number of detections counted in the grab samples were varied and did not trend significantly with the city population. However, the passive sampling devices detecting only pesticide presence or absence did link the population size to the number of unique pesticides.

Passive sampling vs. grab samples

Including passive sampling as part of the surveillance increased the number of identified analytes over traditional grab sampling. There were 45 unique detections from the surface water samples between parent compounds and breakdown products. Fifteen of those analyte detections occurred in the passive samplers only. In addition to increasing the number of unique analytes detected, the passive sampling also increased the number of detections for those 30 analytes that were detected via both methods. This increase helped clarify the patterns of distribution for several of the more common analytes. For example, imidacloprid has occurred sporadically in past sampling projects. In the present study, 23% of grab samples and 80% of passive samples contained imidacloprid. The passive sampling result helped to fill gaps in data relative to the more quantitative grab sampling data. Imidacloprid is persistent in soil, the half-life in soil is just under 200 days, meaning it is likely to be detected in soil for up to two and a half years. However, in sunlit water the half-life is about five hours, meaning it could be nondetectable just over one day after application. Passive sampling can catalog chemicals that occur only briefly.

The passive sampling data are not quantitative and do not serve in establishing whether or not the accumulation of the pesticide active ingredients has reached a concentration of concern. The majority of the detections in this study found concentrations many orders below levels known to cause adverse effects; for surface water grab samples there were 2 detections over an Aquatic Life Benchmark from 6,426 tests run in this study, equaling 0.03% of all testing completed. Information on quantity is essential for scenarios where there is concern over the concentration. In Maine, there are relatively low land use burdens, where testing seldom finds concentrations violative of any thresholds, environmental surveys can use passive samplers without significant loss of information. Previous environmental surveys in Maine waters assess only the number of detections because there are seldom enough data for more quantitative analytical approaches.

Surface vs storm water

Surface water is distinct from storm water in that storm water represents only run-off while surface water represents the integration of run-off into the receiving water body. Surface water samples represent the greater portion of typical exposures to pesticides in aquatic habitats. The location of the sampling becomes important as proximity to point sources may potentially change study outcomes. In this study a storm culvert was sampled as a replacement sample site in Ellsworth because there are no streams that run through that city. The storm drain, serving the downtown and residential areas of Ellsworth, contributed to a higher number of detections in Ellsworth and complicates the interpretation of these data. As seen in Figure 2, Ellsworth had the greatest number of detections of all cities in the study despite being a relatively small city. If the culvert data are removed the comparison to other cities is compromised because there are different numbers of samples being compared between cities. With the culvert data removed there is a substantial decrease in the number of detections. If only the number of unique pesticides is tallied, this pattern largely goes away as seen in Figure 3. Storm water sampling is ideal for understanding the total suite and intensity of pollution sources. In this study, the focus was on better understanding the current status of aquatic environments not just the type of pesticides present in runoff.

Conclusions

In this study, we demonstrated that current use pesticides can be found commonly in streams and rivers in the cities of Maine. Cities with larger populations tend to have greater numbers of pesticide types. The detections of pesticides seldom reach concentrations of concern; however, it is possible in some localities. Passive sampling (POCIS) did increase the ability to detect pesticides, however, there was a trade off because of the inability to determine pesticide concentrations reliably with passive sampling. This small data set adds substantially to what is known about current use pesticide contamination of surface waters in Maine. Additional research is needed to better understand this topic.

Contributors

This study was created in collaboration between Mary Tomlinson, Pam Bryer, and Megan Patterson. Curtis Bohlen provided discussions on study design and data presentation. The Board of Pesticides Control (BPC) public policy board also approved the project. The study was organized and conducted by

Mary Tomlinson, with help of the following BPC inspectors Keith Brown, Heidi Nelson, Alex Peacock, Lucien Saucier, and Nate Thompson.

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Appendix 1. Complete lists of chemical analyses by media with their respective reporting limits.

Surface water grab samples (* indicates samples over existing thresholds):

Grab sample analyte list		Function
Analyte Name	RL ug/L (ppb)	
2,4-D	0.009	Herbicide
AMBA (mesotrione metab)	0.021	Herbicide
Acetochlor	0.14	Herbicide
Acetochlor ESA	0.02	Herbicide
Acetochlor OA	0.0084	Herbicide
Alachlor	0.11	Herbicide
Alachlor ESA	0.0068	Herbicide
Alachlor OA	0.0068	Herbicide
Aminocyclopyrachlor	0.025	Herbicide
Aminopyralid	0.03	Herbicide
Atrazine	0.0022	Herbicide
Azoxystrobin	0.0052	Fungicide
Bentazon	0.0022	Herbicide
Bromacil	0.0041	Herbicide
Bromoxynil	0.012	Herbicide
Carbaryl	0.014	Insecticide
Chlorpyrifos	0.06	Insecticide
Chlorsulfuron	0.0056	Herbicide
Clodinafop acid	0.013	Herbicide
Clopyralid	0.088	Herbicide
Clothianidin	0.016	Insecticide
Deethyl atrazine	0.0017	Herbicide
Deethyl isopropyl atrazine	0.1	Herbicide
Deisopropyl atrazine	0.04	Herbicide
Dicamba	0.88	Herbicide
Difenoconazole	0.011	Fungicide
Dimethenamid	0.006	Herbicide
Dimethenamid OA	0.0072	Herbicide
Dimethoate	0.0022	Insecticide
Disulfoton sulfone	0.0066	Insecticide
Diuron	0.0053	Herbicide
FDAT (indaziflam metab)	0.0051	Herbicide
Fipronil	0.0024	Insecticide
Fipronil desulfinyl	0.14	Insecticide
Fipronil sulfide	0.08	Insecticide
Fipronil sulfone	0.04	Insecticide
Flucarbazone	0.0024	Herbicide

Flucarbazone sulfonamide	0.0039	Herbicide
Flumetsulam	0.029	Herbicide
Flupyradifurone	0.045	Insecticide
Fluroxypyr	0.035	Herbicide
Glutaric acid	0.03	Fungicide
Hydroxy atrazine	0.004	Herbicide
Halosulfuron methyl	0.01	Herbicide
Hexazinone	0.0015	Herbicide
Imazamethabenz acid metab	0.0025	Herbicide
Imazamethabenz ester	0.001	Herbicide
Imazamox	0.0057	Herbicide
Imazapic	0.003	Herbicide
Imazapyr	0.0035	Herbicide
Imazethapyr	0.004	Herbicide
Imidacloprid	0.0018	Insecticide
Indaziflam	0.002	Herbicide
Isoxaben	0.003	Herbicide
Isoxaflutole	0.13	Herbicide
MCPA	0.0046	Herbicide
MCPP	0.0044	Herbicide
Malathion	0.028	Insecticide
Malathion oxon	0.0024	Insecticide
Metalaxyl	0.0035	Insecticide
Methomyl	0.012	Insecticide
Methoxyfenozide	0.01	Insecticide
Metolachlor	0.024	Herbicide
Metolachlor ESA	0.005	Herbicide
Metolachlor OA	0.042	Herbicide
Metsulfuron methyl	0.01	Herbicide
Pinoxaden metabolite 854	0.0052	Herbicide
Pinoxaden metabolite 204	0.02	Herbicide
Nicosulfuron	0.011	Herbicide
Norflurazon	0.02	Herbicide
Norflurazon desmethyl	0.02	Herbicide
Oxamyl	0.01	Insecticide
Parathion methyl oxon	0.012	Insecticide
Phorate sulfone	0.024	Insecticide
Phorate sulfoxide	0.003	Insecticide
Picloram	0.28	Herbicide
Picoxystrobin	0.0075	Fungicide
Prometon	0.001	Herbicide
Propiconazole	0.01	Fungicide
Prosulfuron	0.005	Herbicide
Pyrasulfotole	0.02	Herbicide
Pyroxsulam	0.013	Herbicide
Saflufenacil	0.01	Herbicide

Simazine	0.0026	Herbicide
Sulfentrazone	0.035	Herbicide
Sulfometuron methyl	0.0025	Herbicide
Sulfosulfuron	0.0054	Herbicide
Tebuconazole	0.014	Fungicide
Tebuthiuron	0.0011	Herbicide
Tembotrione	0.073	Herbicide
Terbacil	0.0048	Herbicide
Terbufos sulfone	0.011	Insecticide
Tetraconazole	0.0039	Fungicide
Thiamethoxam	0.02	Insecticide
Thiencarbazone methyl	0.04	Herbicide
Thifensulfuron	0.022	Herbicide
Tralkoxydim	0.0051	Herbicide
Tralkoxydim acid	0.005	Herbicide
Triallate	0.3	Herbicide
Triasulfuron	0.055	Herbicide
Triclopyr	0.022	Herbicide
Trifloxystrobin	0.02	Fungicide

Surface water grab sample glyphosate:

Grab Samples: Glyphosate	
Analyte Name	RL ug/L (ppb)
AMPA	1
Glyphosate	1
Glyfosinate	1

Surface water POCIS samples:

POCIS sample analyte list	
Analyte Name	RL ng/POCIS
2,4-D	0.45
Acetochlor	6.9
Acetochlor ESA	1
Acetochlor OA	0.42
Alachlor	5.5
Alachlor ESA	2.2
Alachlor OA	0.35
AMBA (mesotrione metab)	1.1
Aminocyclopyrachlor	1.3
Aminopyralid	1.5
Atrazine	0.12
Azoxystrobin	0.24

Bentazon	0.11
Bromacil	0.23
Bromoxynil	0.62
Carbaryl	0.73
Chlorpyrifos	3
Chlorsulfuron	0.3
Clodinafop-propargyl acid	0.7
Clopyralid	4.4
Clothianidin	0.84
Deethyl atrazine	0.088
Deethyl deisopropyl atrazine	5
Deisopropyl atrazine	2.1
Dicamba	44
Difenoconazole	0.49
Dimethenamid	0.31
Dimethenamid OA	0.36
Dimethoate	0.1
Disulfoton sulfone	0.34
Diuron	0.29
Iaziflam metab	0.27
Fipronil	0.12
Fipronil desulfinyl	0.12
Fipronil sulfide	7.1
Fipronil Sulfone	2
Flucarbazone	0.12
Flucarbazone sulfonamide	0.2
Flumetsulam	1.4
Flupyradifurone	3
Fluroxypyr	1.7
Glutaric acid	1.6
Hydroxy atrazine	0.2
Halosulfuron methyl	0.49
Hexazinone	0.084
Imazamethabenz acid metab	0.14
Imazamethabenz ester	0.05
Imazamox	0.31
Imazapic	0.18
Imazapyr	0.18
Imazethapyr	0.22
Imidacloprid	0.1
Iaziflam	0.095
Isoxaben	0.15
Isoxaflutole	6.5
Malathion	1.5
Malathion oxon	0.13
MCPA	0.23

MCPP	0.22
Metalaxyl	0.17
Methomyl	0.32
Methoxyfenozide	0.52
Metolachlor	1.2
Metolachlor ESA	0.25
Metolachlor OA	2.1
Metsulfuron methyl	0.51
Nicosulfuron	0.52
Pinoxaden metab 854	0.27
Pinoxaden metab 204	1
Norflurazon	1
Norflurazon desmethyl	1.1
Oxamyl	0.51
Parathion methyl oxon	0.61
Phorate sulfone	1.2
Phorate sulfoxide	0.15
Picloram	14
Picoxystrobin	0.38
Prometon	0.05
Propiconazole	0.52
Prosulfuron	0.25
Pyrasulfotole	1
Pyroxsulam	0.66
Saflufenacil	0.51
Simazine	0.14
Sulfentrazone	1.8
Sulfometuron methyl	0.13
Sulfosulfuron	0.28
Tebuconazole	0.71
Tebuthiuron	0.056
Tembotrione	3.7
Terbacil	0.24
Terbufos sulfone	0.55
Tetraconazole	0.2
Thiamethoxam	1
Thiencarbazone methyl	2
Thifensulfuron	1.1
Tralkoxydim	0.25
Tralkoxydim acid	0.25
Triallate	15
Triasulfuron	0.28
Triclopyr	1.1
Trifloxystrobin	1

Appendix 2. Study site description and sediment profiles.

	Augusta	Bangor	Biddeford	Ellsworth	Farmington	Lewiston-Auburn	Portland	Presque Isle	Sanford	Waterville
Latitude (N)	44.300052	44.762990	43.487630	44.529249	44.652030	44.016586	43.650553	46.703424	43.407291	44.428553
Longitude (W)	-69.777300	-068.800168	-070.437260	-068.423335	-070.14324	-070.167325	-070.242530	-068.007480	-070.716215	-069.701766
Sediment										
Percent Organic Carbon	0.73	3.58	0.31	3.93	0.25	0.09	1	0.85	5.23	0.53
Percent Coarse	9.9	1.2	1	21.4	0.4	0.1	16.3	8.6	0.2	49.8
Percent Sand	76	30.8	91.9	28.7	95.3	96.1	77.4	77.9	33.5	42.5
Percent Silt	8.5	53.8	3.4	38.6	1.1	0	0	6.7	43.9	4.6
Percent Clay	5.6	14.2	3.7	11.3	3.1	3.7	6.3	6.9	22.4	3.1

Appendix 3. US EPA's Aquatic Life Benchmarks and Ecological Risk Assessments for Registered Pesticides.

Bold highlighted names indicated detection within the study. Names in blue text indicates surface water grab sampling while brown text indicates sediment sampling. Numbers in parentheses is the highest value found during the study; no number indicates POCIS sample. Benchmark values exceeded during the study are noted in red.

Analyte & EPA Aquatic Benchmarks ug/L (ppb)						
Analyte Name	Fish		Invertebrate		Non-Vascular	Vascular
	Acute	Chronic	Acute	Chronic	Acute	Acute
2,4-D (0.059)			12,500			299.2
Allethrin						
AMBA						
Acetochlor						
Acetochlor ESA	>90,000		>62,500		9,900	
Acetochlor OA						
Alachlor						
Alachlor ESA						
Alachlor OA						
Aminocyclopyrachlor						
Aminopyralid						
AMPA						
Atrazine (0.0081)	2,650	5	360	60	<1	4.6
Azoxystrobin	235	147	130	44	49	3,400
Bentazon (0.078)	95,000	9,830	31,150	101,200	4,500	5,350
Bifenthrin (1.3; mean=0.47)	0.075	0.04	0.8	0.0013		
Bromacil (0.013)	18,000	3,000	60,500	8,200	6.8	45
Bromoxynil						
Carbaryl						
Chlorpyrifos						
Chlorsulfuron						
Clodinafop acid						
Clopyralid						
Clothianidin	>50,750	9,700	11	0.05	64,000	>280,000
Cyfluthrin						
Cyhalothrin, Total						
Cypermethrin						
Deethyl atrazine (0.047)	See	Parent	Compound			
Deethyl isopropyl atrazine						
Deisopropyl atrazine						
Deltamethrin						
Dicamba						
Difenoconazole						

Dimethenamid	3,150	300	6,000	1,020	14	8.9
Dimethenamid OA						
Dimethoate						
Disulfoton sulfone						
Diuron (0.015)	200	26.4	80	200	2.4	15
FDAT (indaziflam metab)						
Fenpropathrin						
Fenvalerate						
Fipronil (0.0029)	41.5	2.2	0.11	0.011	140	>100
Fipronil desulfinyl						
Fipronil sulfide						
Fipronil sulfone	12.5	0.67	0.36	0.037	140	>100
Flucarbazone						
Flucarbazone sulfonamide						
Flumetsulam						
Flupyradifurone						
Fluroxypyr	7,150		>50,000		>10,000	
Glutaric acid						
Glyphosate						
Glufosinate						
Hydroxy atrazine (0.0066)	See	Parent	Compound			
Halosulfuron methyl						
Hexazinone (0.049)	137,000	17,000	75,800	20,000	7	37.4
Imazamethabenz acid metab (Q)						
Imazamethabenz ester						
Imazamox						
Imazapic (0.0037)	>50,000	96,000	>50,000	96,000	>44.1	6.22
Imazapyr (0.037)	>50,000	43,100	>50,000	97,100	12,200	24
Imazethapyr						
Imidacloprid (0.11)	114,500	9,000	0.385	0.01		
Indaziflam (Q)						
Isoxaben (0.011)	>500	400	>650	690	922	10
Isoxaflutole						
MCPA (0.019)					630	170
MCPP (0.02)	>46,500		>45,500	50,800	14	1,300
Malathion						
Malathion oxon						
Metalaxyl (Q)	65,000	9,100	14,000	1,200		85,000
Methomyl (0.016)	250	57	4.4	0.6		
Methoxyfenozide						
Metolachlor (Q)	1,900	30	550	1	8	21
Metolachlor ESA (ethane sulfonic acid) (0.17)	24,000		>54,000		>99,450	43,000
Metolachlor OA (oxanilic acid) (Q)	>46,500		7,700		57,100	>95,400

Metsulfuron methyl (Q)	>75,000	4,500	>75,000		31	0.36
cis-Permethrin						
trans-Permthrin						
Pinoxaden metabolite 854						
Pinoxaden metabolite 204						
Nicosulfuron						
Norflurazon						
Norflurazon desmethyl						
Oxamyl						
Parathion methyl oxon						
Phenothrin						
Phorate sulfone						
Phorate sulfoxide						
Picloram						
Picoxystrobin						
Piperonyl butoxide						
Prometon (0.017)	6,000	19,700	12,850	3,450	98	
Propiconazole (Q)	425	95	650	260	21	3,500
Prosulfuron					10.6	1.22
Pyrasulfotole (Q)	>48,000	580	>47,900	12,800	8,300	28
Pyroxsulam	>43,500	10,100	>49,500	10,400	111	2.57
Resmethrin						
Saflufenacil	>54,000	997	4,250	1,330	42	87
Simazine	3,200	60	500	40	6	67
Sulfentrazone	46,900	2,950	30,200	200	31	28.8
Sulfometuron methyl (0.0042)	>74,000		>75,000	97,000	4.3	0.45
Sulfosulfuron	>46,800	100,000	>47,300	102,000	310	1
Tebuconazole (0.028)	1,135	11	1,440	120	1,450	151
Tebuthiuron (0.0017)	53,000	9,300	148,500	21,800	50	130
Tetramethrin						
Tembotrione						
Terbacil (Q)	23,100	1,200	32,500	50	11	140
Terbufos sulfone						
Tetraconazole (Q)	1,925	300	1,315	190		310
Thiamethoxam	>57,000	20,000	17.5	0.74	>99,000	>90,200
Thiencarbazone methyl						
Thifensulfuron						
Tralkoxydim						
Tralkoxydim acid						
Triallate						
Triasulfuron						
Triclopyr (0.047)	58,500		66,450		32,500	
Trifloxystrobin						