

Preliminary Survey of  
**Pyrethroid Pesticides and Surfactants in  
San Juan County Surface Waters**



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October 2008



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## Summary

Surface water and sediments were collected from 32 streams, lakes, and ponds in San Juan County, WA, to ascertain whether household-use pesticides or surfactants have accumulated to measurable levels, and whether they may already pose a threat to Pacific salmon and other aquatic organisms in the San Juan Islands.

Pyrethroid pesticides were chosen as an indicator for total pesticides because they represent the largest chemical family found in the pesticide products currently sold in San Juan County: 18 different pyrethroid derivatives in 93 locally sold products for home and garden use. Pyrethroid pesticides can also be measured with great sensitivity by enzyme-linked immunosorbent assays (ELISA), which can be carried out with basic and relatively inexpensive laboratory equipment, and therefore pose no significant technical or financial obstacles to continued local monitoring by county government. The limit of detection for pyrethroid pesticides in the ELISA system we employed was 0.1 part per billion (ppb), or about the level at which the most toxic pyrethroid species are toxic to Pacific salmon.

Linear alkyl sulfonates (LAS) were selected as a proxy for total surfactants. They are the most common anionic surfactants found in U.S. household products, estimated to comprise as much as 40% of total household detergent use today. LAS can be measured colorimetrically using methylene blue as an indicator. This “standard method” is reliable and inexpensive. While it has some cross-reactivity with other natural and anthropogenic compounds, environmental chemists generally agree that any over-estimation of LAS by this method is comparable to its under-estimation of *total* surfactants in the environment. We adjusted the standard method to increase its sensitivity, achieving a limit of detection of 0.2 parts per million (ppm). The EPA’s national secondary drinking water standard for surfactants is currently 0.5 ppm (=500 parts per billion or ppb).

Two-thirds of the sites sampled exceeded 1.0 ppb pyrethroid pesticides in at least one sediment specimen or water specimen, and only one site had no detectable pesticides at our 0.1 ppb limit of detection. Only a single water specimen had no detectable LAS at our 0.2 ppm limit of detection. We conclude that household pesticides and detergents do currently pose a potential threat to aquatic wildlife, and that local consumption should be reduced and monitored. Neither pyrethroids nor surfactants were statistically correlated with field-measured nitrates, hence nitrates would not be a reliable proxy or indicator.

We also attempted measuring non-ionic surfactants in our specimens with another “standard method” using cobalt thiocyanate as an indicator. The best sensitivity we could achieve without using a resin column to concentrate the surfactants in our specimens was 1.0 ppm—five times the LOD for our LAS measurements. A number of water specimens exceeded even this rather high threshold. However, we do not recommend including the cobalt thiocyanate method in future monitoring on account of its cost and use of unstable, relatively toxic reagents.

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## **Purpose and focus**

The present study was undertaken for the San Juan Nature Institute as an element of a Public Participation Grant from the Washington State Department of Ecology, which has reviewed the results. Additional support by the Orcas Island Community Foundation is also acknowledged.

State and federal researchers found pesticides and herbicides in a number of Puget Sound streams more than a decade ago at levels approaching their documented toxicity to Pacific salmon (Bortleson and Davis 1997). In 2004, Seattle-based environmentalists won a federal court order overturning Environmental Protection Agency decisions to approve a number of pesticides for use on the grounds that these products posed imminent threats to Pacific salmon (*Washington Toxics Coalition v. EPA*). The National Marine Fisheries Service issued a draft biological report in July 2008 identifying the garden-use chemicals chlorpyrifos, malathion and diazinon as significant threats to the survival of endangered salmon ([http://www.nmfs.noaa.gov/pr/pdfs/pesticide\\_biological\\_opinion\\_draft.pdf](http://www.nmfs.noaa.gov/pr/pdfs/pesticide_biological_opinion_draft.pdf)), only the first step in its planned evaluation of 40 pesticides. Similar concerns have been raised with respect to the Fraser River and Georgia Strait (Johannessen and Ross 2002).

In 2007, the Northwest Straits Commission identified surfactants as an “emerging issue” in Washington’s inland marine waters. As yet, relatively little is known about the fates and effects of surfactants in the Pacific Northwest, but the fact that products such as laundry soaps and cleaners are consumed and discharged in much greater quantities than pesticides, with less regulatory oversight as toxic substances, warrants more systematic research. Managing pesticides and surfactants are linked because most pesticide products contain surfactants to serve as stabilizers, emulsifiers or spreader-stickers. Surfactants are not only toxic themselves to aquatic organisms, but also have synergistic effects when combined with pesticides and herbicides (*e.g.*, Xie et al. 2005).

San Juan County was not included in past Federal or State pesticide assessments, or in the State Department of Ecology’s ongoing Surface Water Monitoring Program for Pesticides in Salmonid-Bearing Streams. Nor have there been any systematic evaluations of surfactants in San Juan County waters. The present study is the beginning of an effort to address these data gaps, particularly in the context of salmon recovery.

## **Modes of action, fates and effects**

Persistent bioaccumulative toxics (PBTs) can have widely divergent physiological effects on different organisms (*e.g.*, Juraske et al. 2007). Our reference point here will be Pacific salmon, the genus *Oncorhynchus*, which in San Juan County includes Chinook as well as coho, chum and pink salmon in nearshore waters, and coastal cutthroat in streams. Rainbow trout, a member of the same genus, are routinely used as a laboratory model for testing the toxicity of pesticides and other contaminants on aquatic animals, hence we know a great deal more about the metabolism and effects of organic toxicants on Pacific salmon than other organisms typically found in San Juan County waters. Correlations between acute toxicity to salmon and their animal prey is good (Tremolada et al. 2004), although salmon accumulate more toxics over time so are ultimately more affected than their prey (Johnson et al. 2007).

### Pesticides

Most pesticides sold for home and garden use interfere with the nervous system of insects. Organophosphates such as malathion and carbamates such as carbaryl inhibit acetylcholinesterase, the enzyme that re-sets nerve synapses at the end of a transmission, so that insects' synapses remain "on" and cannot carry any information. Chloronicotinyls such as imadocloprid have the opposite effect: by binding to the acetylcholine receptors on neurons they turn the synapses permanently "off". Organochlorines and pyrethroids disrupt the sodium ion channel in neurons resulting in a kind of "stutter" overwhelming insect nervous systems with noise. Triazole pesticides also cause stutter, but by different means: interfering with  $\gamma$  amino butyric acid receptors on the animal's neurons.

Many pesticides designed to interfere with the nervous system of insects also have neurological effects on Pacific salmon and other aquatic animals (*e.g.* Scholz et al. 2006). Chronic sub-lethal doses may not only eventually prove fatal (Cheng and Farrell 2007), but meanwhile can disrupt chemoreception (Tierney et al. 2006a; Jarrard et al. 2004) and alarm response (Labenia et al. 2007; Tierney et al. 2006b), resulting in greater mortality from predation and failure to return to spawning riffles. Many pesticides are endocrine disrupters in fish (Monod et al. 2004), with estrogenic (Tollefsen et al. 2002 ) and other hormone-mimic effects than interfere with growth or reproduction.

The apparent specificity of pesticides to the physiology of insects is no guarantee of harmlessness to other organisms. Pesticides that mimic insect growth hormones, such as the natural tripterpenoids in neem oil, or synthetic pyridines, can also affect Pacific salmon and other aquatic animals, much as glyphosate and other herbicides that mimic plant hormones can also have toxic effects on animals.

Soaps, fats and oils, including many products advertised as "natural" alternatives, simply clog the tiny pores (spiracles) in insects' exoskeletons, causing suffocation. Many plants naturally produce soapy compounds (saponins) with the same purpose (Sparg et al. 2004), and these compounds are beginning to find their way onto store shelves as "safer" or "natural" pesticides. To the extent that these compounds function as surfactants, they pose the same potential threats to fish and other aquatic organisms as detergents.

Essential oils, such as clove, thyme and cinnamon oil, are also advertised as safer, “natural” alternatives to “chemical” pesticides, but it must be borne in mind that these oily, aromatic compounds evolved in plants as chemical weapons against herbivores. In small quantities, they have historically been used as cosmetics and flavorings, and what toxicological data exist are largely restricted to their effects on humans. Their effects on fish and aquatic animals, at this time, are poorly understood.

We chose to focus the present study on pyrethroid pesticides for several reasons. Pyrethroid products are replacing organophosphates as the dominant class of pesticides in residential and agricultural use due to their low toxicity to humans and other mammals, but they are extremely toxic to aquatic organisms including Pacific salmon (Appendix B; Wheelock et al. 2005). Pyrethroids constitute the active ingredients in 93 (48%) of the 195 pesticide products we found for sale in San Juan County in 2008, and as such they can be used as a proxy for total pesticide loads in the local environment. From a practical standpoint, there are highly sensitive and reliable laboratory methods for measuring total pyrethroid compounds employing enzyme-linked immunosorbent assays (ELISA), the same approach used for many laboratory tests on human blood and urine specimens in clinical medicine. ELISA tests can be costly—this study cost \$26 per specimen tested for supplies alone—and cannot distinguish between the various pyrethroid configurations, but more specific analytical methods would be several times costlier at the same level of sensitivity (roughly 0.1 part per billion).

Like many other pesticide compounds, pyrethroids tend to be nearly insoluble in water. Pyrethroids are synthetic derivatives of pyrethrin, a natural chemical defense in chrysanthemums, where it “stays put” in leaf tissues until it is consumed by an insect or other herbivore. From an evolutionary perspective, the relative insolubility of pyrethrin is advantageous to the plants that manufacture it, since this minimizes the likelihood that it will affect the plants’ pollinators along with herbivores. Pyrethrin also breaks down relatively quickly in soil or water. These are problems for using pyrethroids in house and garden products, however, because they must somehow be rendered more stable (shelf-life), spreadable (for instance in a water spray) and likely to stick to the plants on which they are sprayed.

By analyzing a number of common garden products for sale in San Juan County, we discovered that manufacturers have solved these problems by floating tiny particles of pyrethroids, or plastic particles coated with pyrethroids, in a water-soluble carrier fluid composed of one or more surfactants. Surfactants help keep particles suspended, prevent them from clumping together, and stick them to leaves and bark to be ingested by insects when they attack the treated plants. Rain will wash the particles and surfactants into soil, and storm water runoff will convey the particles and surfactants downstream. We do not know the extent to which plastic micro-particles or beads are used in pesticide products, nor what threats they pose to the environment, but we suggest that they may function on a very small scale like Styrofoam grains in the ocean: ingested by small animals, cannot be metabolized, and will fill and block the gut.

## Surfactants

Although the toxicity of surfactants to Pacific salmon and other aquatic organisms has been known for more than 40 years, little research has been conducted until recently on the fate of these compounds in the environment under realistic conditions (Cook and Goldman 2008; Wong et al. 2002; Buhl and Hamilton 2000).

Beginning in the 1960s, public and policy concern over cleaning products focused on the orthophosphates added to detergents as “builders”—molecules that bind with ions such as calcium and magnesium that can interfere with detergents’ action—not detergents themselves. Mounting evidence that adding phosphates to aquatic environments resulted in rapid algal growth (eutrophication) and de-oxygenation led to a nationwide movement to reduce or eliminate phosphates in the 1970s. Ironically, this led to rapid growth in the development and marketing of new, stronger surfactants and arguably also increased total surfactant consumption by implying the safety of phosphate-free products: the 1980s saw reduced consumer demand for anionic surfactants manufactured with phosphate builders, and greater demand for non-ionic products (Haupt 1983).

Surfactants have become ubiquitous in our economy as constituents of medicines, foods, colorings, inks, motor oils, plastics and rubber, disinfectants, fertilizers, pesticides, and herbicides as well as dish and laundry soaps, shampoos and conditioners, and other commercial and household cleaners. Most North American homes probably use several surfactant products every day.

Surfactants are chainlike molecules that have a hydrophilic (“water-loving”) head and a lipophilic (“fat-loving”) tail. Surfactant molecules pull water and fat/oil molecules together, and thereby help make oily substances miscible in water. This is useful in soaps and other cleaners, where the aim is pulling oils molecules into solution so that they can be flushed away. It is also used to help paints stick to surfaces; water-based pesticide and herbicide sprays stick to plants; and fertilizers penetrate soils (“wetting”). This property also makes surfactants toxic to aquatic organisms by interfering with the functioning, and potentially rupturing cellular membranes in gills and eggs that are completely exposed to water without a protective cuticle or sheath.

Most contemporary surfactants are either anionic (that is to say, polar molecules with a negative ionic charge) or non-ionic. The anionic surfactants include sodium lauryl sulfate, linear alkyl sulfonates (LAS), and salts of both naturally occurring and synthetic fatty acids. Non-ionic surfactants, a more diverse family, includes alkyl polyethylenes, polypropylenes, polyglucosides, polysorbates, cocamides, and fatty alcohols. There are also cationic (positively charged) surfactants such as benzalkonium chloride, as well as a number of amphiteric surfactants such as cocamidopropyl betaine that carry both positive and negative charges, commonly used as medical disinfectants and germicides.

LAS has been described as the “workhorse” of detergents, with over 300,000 tons consumed annually in North America, chiefly as a constituent of laundry and dish soaps, while alkylphenols, mainly used in industry and hospitals as cleaners and disinfectants, represent another 150,000 tons of annual consumption (Innovation Group 2008).

All families of surfactants cause irritation, mucus formation, and chemical stress responses in the gills of fish (*e.g.* Bolis and Rankin 2006; Stagg and Shuttleworth 2004;

Alcaraz et al. 1993) and are cytotoxic to varying degrees (Mori et al. 2002). Many of the non-ionic surfactants are estrogenic hormone disrupters (Ward et al. 2006; Sheahan et al. 2002), and others have been described as “information disrupters” (Lurling and Scheffer 2007) owing to their effects on chemical signaling between aquatic animals. Surfactants can bio-accumulate as well (*e.g.*, Martin et al. 2003), making fish and aquatic mammals especially vulnerable to toxic effects. As Brasino and Dengler (2007) have demonstrated, even the seemingly innocent car-wash effluent in storm drains can be toxic to juvenile rainbow trout.

Surfactants vary greatly in their chemical structure, and consequently in the force they apply to exposed tissues. Some generalizations can be made, however. The damage to aquatic organisms is partly a function of the *total concentration* of all surfactants, since all surfactants have similar functions. Total concentration is a function of the *persistence* of the constituent surfactant species—how slowly they break down in the environment. Most surfactant species eventually do yield to light, heat, reduction-oxidation conditions, or bacteria, but their half-lives can vary from hours to years. Using fewer surfactants, and choosing less persistent surfactants, should reduce the mean total concentration of all surfactants in the environment. Regardless of how “safe” their manufacturers claim them to be, all surfactants contribute to total concentration and toxic effects in proportion to their persistence.

### Environmental fates

Owing to their structure and low solubility in water, pyrethroids tend to adsorb to silt particles (Todd et al. 2003, 169-80). Surface water runoff conveys pyrethroid-laden particles into streams, ponds, lakes and the nearshore, where suspended sediments may settle in low-velocity areas. Although changes in acidity and temperature may release some pyrethroids from sediments into the water column, the main vector for adsorbed pyrethroids into the ecosystem is the action of detritivores that ingest silt grains and the hydrophobic molecules adsorbed to them, before they are eaten in turn by larger animals. Pesticide spraying also produces aerosols that can drift a considerable distance, before eventually settling. High concentrations of pesticides in water have usually been traced to aerosols rather than transport on suspended sediments. For the present study we examined both adsorbed (“solid phase”) and dissolved pyrethroids.

Since sediments accumulate over time, even relatively shallow sediment “grabs” may represent years of pyrethroid accumulation. Most pyrethroid pesticides also degrade over time, however, so that on balance, the pyrethroid signal in sediments should reflect no more than 3-5 years of inputs. On the other hand, dissolved pyrethroids reflect nearer-term use and even very short-term events, and always underestimate the application rate, since the solubility of most pyrethroid compounds is less than 5 percent.

By comparison, surfactants are highly soluble in water, so most of the load should be found in the water column rather than being adsorbed to silt. The hydrophobic end of surfactant molecules can adsorb to clay minerals (see, generally, van Olphen 1977; Theng 1974), however, so geology can influence the ratio of dissolved:adsorbed surfactants in a particular system. Clays are found near or at the surface in the young post-glacial soils of San Juan County, and tend to be a significant component of surface runoff. Clays may be

a significant transporter and reservoir of surfactants in San Juan County watersheds, then, but we did not investigate this possibility in the present study.

We have assumed for the purposes of this study that pyrethroids and surfactants in San Juan County freshwater systems are of local origin. It should be borne in mind that a significant level of atmospheric dispersal and deposition of PBTs such as organochlorines and organobromines has been documented in the Pacific Northwest (Demers et al. 2007; Ohyama et al. 2004). Pyrethroids are generally applied as sprays with water or petroleum distillates as carriers, and the pesticide-laden aerosols can be dispersed over considerable distances, although photolytic degradation is accelerated under these conditions (Todd et al. 2003, 169-180). Surfactants can also be dispersed in aerosols (e.g. Oono et al. 2008). Since the San Juan Islands are located mid-way between three major urban areas, Seattle, Vancouver and Victoria, it is possible that airborne deposition is significant, although we did not have the means to try to distinguish airborne from local signals. The presence of significant PCBs, PBDEs and TCDDs in resident pink salmon or kokanee (*Oncorhynchus gorbuscha*) in Mountain Lake on Orcas Island (Seiders et al. 2007) is cogent evidence of airborne deposition in the islands since there are few possible local sources of these PBTs and no industrial activity or power lines in the Mountain Lake basin.

Similarly, we found significant pyrethroids and surfactants in Summit, Mountain, and Cascade Lakes in Moran State Park, Orcas (Appendix A), whereas two of the lower-elevation undeveloped lakes in our sample (Diamond, Killebrew) were less contaminated. This is consistent with some airborne deposition, since there is a strong correlation in the San Juan Islands between elevation and precipitation; Summit Lake on average receives twice the annual precipitation as Diamond or Killebrew. Further investigation is required to estimate the strength of the atmospheric signal, and determine the level of background pyrethroid-surfactant contamination more reliably.

## Materials and methods\*

### Sampling procedure

Specimens of water and sediments were collected in July and August 2008 when there was little rainfall and water levels in streams, lakes and ponds were low, hence any pesticides or surfactants would be at their greatest seasonal concentration. Water samples for surfactant testing were collected in the barrel of a glass syringe and injected into clean (acid-washed, rinsed three times with ultra-pure Milli-Q water) glass tubes through fresh 0.22-micron syringe filters to remove suspended solids. Tubes were capped with Teflon-coated lids and kept on ice until refrigerated at our laboratory. Water for pesticide testing were mixed 1:1 with absolute analytical grade methanol in the field after syringe filtering, and kept on ice until refrigerated at our laboratory, to prevent adhesion to the walls of the glass collection tubes. Sediments were collected in a 10 cc glass cup, drained, and added to 20 mL of absolute analytical grade methanol in clean (methanol washed) glass jars. To minimize degradation of our target analytes between collection and testing, all specimens were held at 4°C for a maximum of five days.

Sediment slurries in methanol were allowed to settle overnight at 4°C before their supernatant was removed by pipette and syringe-filtered into clean glass tubes. Sediment collection jars were weighed before and after the addition of sediment specimens in order to make later adjustments in test results based on the weight of each sample.

At the time of collection, a YSI Professional Series electronic logger was used to record water temperature, oxidation-reduction potential (ORP), and nitrate concentration. Notes were also taken on riparian and aquatic vegetation, sediment characteristics, water color, flow, and aquatic animals. Field measurement of nitrates was preferred over water collection for laboratory analysis due to the rapid degradation of nitrates in environmental samples (recommended holding times at 4°C of 24 hours or less).

### Surfactant determinations

This study utilized two kinds of measurement methods: ion pair extraction for the surfactants, and enzyme-linked immunosorbent assays (ELISA) for pyrethroid pesticides. In ion pair extraction, an indicator is added to the specimen and forms a colored complex with the target analyte. The aqueous specimen is floated on top of an organic solvent (in our procedures, methylene chloride) and ordinary salt is added, which forces the colored complex from the aqueous layer into the organic solvent below. The concentration of the complex in the organic solvent can be measured spectrophotometrically; that is, by measuring the reacted specimen's absorption of an appropriate wavelength of light.

Following published standard methods, we used methylene blue as our indicator for anionic surfactants, and cobalt thiocyanate as our indicator for non-ionic surfactants. Both produce blue-violet complexes with surfactants, as well as cross-reacting with some other molecules. For example, methylene blue can complex with inorganic ions such as sulfates, chlorides, even nitrates if their concentrations in the specimen are great enough. Analytical chemists accordingly refer to "methylene blue active substances" (MBAS) and

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\* For complete details of materials and methods please refer to Appendix C.

“cobalt-thiocyanate active substances” (CTAS) when describing the results of these tests. It is also customary to report results in terms of *standard equivalents*, where the *standard* is the chemical compound used by the laboratory to calibrate its reagent system. We used the anionic detergent sodium lauryl sulfate (SLS), *aka* sodium dodecyl sulfate or SDS, to calibrate our methylene blue tests, and the common non-ionic detergent Triton X-100 (4-octylphenol polyethoxylate) to calibrate our cobalt thiocyanate tests. Hence a test result of MBAS=0.6 ppm should be understood as “equivalent to 0.6 ppm of SLS”.

Ion pair extraction methods are relatively fast (about one hour) and use relatively inexpensive reagents, but their cross-reactivity with non-target molecules means that the results are approximate, and may somewhat over-estimate actual surfactant loads. Since there are many chemical families of surfactants, however, combining MBAS and CTAS results for a single water specimen almost certainly still *underestimates* total surfactants.

The sensitivity of CTAS testing can be improved five- to tenfold by concentrating the non-ionic surfactants in specimens prior to testing. This can be achieved by dripping the specimen through a glass column filled with a specially designed resin such as ABX. The surfactants in the water cling to the resin beads, and can later be eluted into a smaller volume of an appropriate organic solvent such as methanol. Concentration using a resin column can take up to 24 hours. While this method would bring the limit of detection for CTAS down to about 0.2 ppm, comparable with our modified MBAS method, it involves considerable additional time and expense. Another consideration is the instability of the cobalt thiocyanate complex at low concentrations, which requires that standard solutions be prepared almost daily.

We do not recommend CTAS testing as part of a county water-quality monitoring scheme. The expense of achieving adequate sensitivity is not trivial, and the MBAS test, which is inexpensive and reliable, and captures phosphonate and sulfonate surfactants, is arguably a sufficient proxy for total surfactants, anionic and non-ionic.

### Pyrethroid determinations

ELISA operates on different principles than ion-exchange methods, achieving far greater sensitivity and specificity albeit at greater expense. It is considered a fundamental tool of clinical immunology. A large number of medical tests, for example for hormones, doping and toxicants in blood and urine, and growing number of commercial drinking water testing use ELISA technology. ELISA uses antibodies to react with target analytes in the specimen. The antibodies are either bonded to wells in a glass plate, or to magnetic particles or beads. The specimens to be tested are either added to the wells or mixed with the antibody-bearing particles. An enzyme-linked chemical analog to the target analyte is then added, and it binds to any antibodies in the test solution that have not already reacted with the target. The wells are then rinsed out or the magnetic particles separated from the solution and then rinsed. At this stage the proportion of the enzyme bound to the wells or particles is inversely proportional to the concentration of the target analyte in the original specimen. A solution containing a “chromogen” is then added to the wells or mixed with the magnetic particles, and cleaved by the enzymes remaining on the wells or particles to produce a colored product. The concentration of this colored product can be measured

spectrophotometrically. Color (absorbance) is inversely proportional to the concentration of the target analyte in the original specimen.

To produce antibodies for this purpose, a target analyte (such as a pyrethroid) is injected into an appropriate animal, frequently rabbits or goats, and the animals' immune systems respond by constructing defensive antibodies. Antibodies must then be purified from the blood of the animal, a complex and expensive process. Many antibodies cross-react with structurally similar molecules at varying levels of sensitivity. This can be very useful: for example, if it means that a single test can detect all of the different forms that a large chemical family such as pyrethroids may take. On the other hand, antibodies are generally not the best tools for identifying individual chemical species within a chemical family.

For the present study, we used a magnetic-particle ELISA system for the chemical family of pyrethroids developed and sold by Abraxis (Warminster, PA).

The antibodies in the Abraxis ELISA system for pyrethroid pesticides cross-react with most pyrethroids but at different levels of sensitivity. The chemical standard used to calibrate the system is permethrin, with which the test system antibodies provided react strongly. They do not react quite as strongly with other pyrethroids, such as tetramethrin or resmethrin. Test results therefore generally *underestimate* the total pyrethroid load in the specimen tested. For clarity, test results can be expressed as "equivalent to 0.65 parts per billion of permethrin". In actuality, the specimen may contain, for example, 0.41 ppb permethrin and more than 1.0 ppb other pyrethroids.

### Quality Assurance

Standards at four dilutions were included in each batch of specimens tested, along with positive and negative controls. The resulting standard curve was used to determine the limit of detection and quantitation limit for each batch tested. In several instances, we rejected test results and retested a batch of specimens based on a poor standard curve. In two instances, sites were re-sampled and retested to confirm results.

Three sets of water and sediment specimens were collected at each sampling site. Contaminants are not distributed homogeneously in relatively still water, much less so in sediments. We expected results to vary within sites, especially with regard to pyrethroid pesticides in sediments, as they did. MBAS results were more consistent within sampling sites, which is not surprising in light of the chemical behavior of surfactants in water as spreaders and emulsifiers.

CTAS results were more varied within sites than MBAS results, which may be due partly to the chemistry of non-ionic surfactants, which can begin to precipitate out of solution when water warms, producing a more patchy distribution. It may also reflect the instability we observed in the standards and reagents used for this test. We would advise greater caution in reading the CTAS results in Appendix A, than the pyrethroid or MBAS results.

## Results

A total of 32 lakes, ponds, streams, and storm sewer outfall was sampled in July-August 2008, with three replicate sets of specimens collected from each site. Please refer to Appendix A and to the attached Maps for complete results.

Pyrethroid pesticides in excess of 1.0 part per billion (ppb) were found in at least one water specimen or one sediment specimen from 22 of 32 sampling sites. More than 10 ppb pyrethroid pesticides were found in water from three sites and sediments from two sites, with some results as high as 18 ppb. Only one site had no detectable pyrethroid pesticides in either water or sediments at the 0.1 ppb limit of detection. Mean pyrethroid concentrations for all specimens tested were slightly higher for water than for sediments, but this difference was not statistically significant; variance was also greater for water (Table 1). The means of the *highest* results for each sampling site were a little greater: 2.68 ppb (standard deviation 4.06) for water and 2.28 ppb (standard deviation 3.36) for sediments. It can safely be said that pyrethroid pesticide levels of 1-2 ppb are widespread in San Juan County waters.

Anionic surfactants of 1.0 part per million (ppm) or greater were found in at least one water specimen from 8 of 32 sampling sites. The highest concentration observed was 1.6 ppm. Anionic surfactants were detected at all 32 sampling sites at our 0.2-ppm limit of detection. Indeed, only a single specimen from one site had no detectable surfactants. Variation was low between sampling sites and very low within sites as well. This family of surfactants appears to be nearly ubiquitous in San Juan County freshwater systems at a level of roughly 0.5 parts per million (500 parts per billion). This is currently the EPA’s national secondary drinking water standard for surfactants—the maximum recommended for water consumed by humans.

Non-ionic surfactants were measured in specimens from a sub-set of 24 sites, half of which produced results between 2.3 and 12.6 ppm. The higher mean result for CTAS compared with MBAS is consistent with industry estimates that non-ionic surfactants are now a majority of all surfactants in residential and commercial use. Variation within and between sampling sites was relatively high. As discussed above under Quality Assurance this variation may be partly actual environmental patchiness, and partly due to the testing system. While our limit of detection for MBAS was 0.2 ppm, we were unable to achieve a CTAS limit of detection of less than 1.0 ppm.

**Table 1. Mean test results for all specimens from all sampling sites**

	Mean pyrethroids ppb		Mean surfactants ppm		Mean NO <sub>3</sub> ppm
	Water	Sediment	MBAS	CTAS	
Specimens (N)	99	96	102	69	33
Mean test result*	1.97	1.28	0.57	1.27	13.4
Standard deviation	6.90	2.16	0.32	2.45	7.94

\*Treating results below the LOD as zeroes.

We found no statistically significant relationship between pyrethroid or surfactant concentrations and field-measured nitrates: higher nitrates did not predict higher pesticide or surfactant levels. This suggests different sources and vectors for nitrates and pesticide-surfactant contamination. Non-correlation would be consistent with a hypothesis that the main sources of pyrethroids and surfactants in the local environment are outdoor uses and runoff, as opposed to failing or seasonably overflowing septic systems. Most surfactants should be largely degraded in functioning on-site septic systems (Nielsen et al. 2002; McAvoy et al. 1994), but we cannot discount the possibility that the surfactant loads we observed are partly due to septic system leakage or incomplete degradation.

Since pyrethroids and other hydrophobic pesticide compounds tend to be particle-bound, their concentrations should be highest in sediment traps such as stream pools, dug ponds and wetlands (*e.g.*, Braskerud and Haarstad 2003). We found no clear relationship between hydrological contexts and contaminant concentrations (Table 2); however, this is not surprising given the relatively small number of specimens tested from each category. On average, pyrethroid pesticides were somewhat most concentrated in stream water and storm sewer sediments. Anionic surfactants loads were highest in storm sewer water but non-ionic surfactants were most concentrated in streams. It is difficult to interpret these differences in the absence of additional data, and unwise to rely on them.

**Table 2. Highest test results per sampling site: means by type of site**

	Total sites	Mean pyrethroids ppb		Mean surfactants ppm		Mean NO <sub>3</sub> ppm
		Water	Sediment	MBAS	CTAS	
Streams	11	4.1	2.4	0.69	3.6	13.9
Lakes	10	2.2	2.1	0.48	0.9	9.1
Dug ponds	7	2.1	1.2	0.64	1.2	15.7
Runoff/sewer	4	1.2		1.02	0.8	14.7
	32	2.7	2.3	0.66	2.5	12.9

These are preliminary results, subject to further confirmation and refinement by chromatography and mass spectrometry when and if these more sophisticated and costly techniques become available.

## Discussion and Recommendations

We found widespread total pyrethroid pesticide levels of 1-2 ppb in San Juan County's waters, with as much as 10-18 ppb at some sites. Six pyrethroid pesticides in local use are each toxic to rainbow trout at less than 2 ppb; thirteen pyrethroid pesticides in local use are each toxic to rainbow trout at 18 ppb or less (Appendix B). In brief, current accumulations of pyrethroid residues in many San Juan County waters already pose a threat to salmonids—if not also to many aquatic crustaceans and larval insects that form the base of our freshwater and nearshore food chains. Efforts to reduce pyrethroid pesticide consumption in San Juan County should be made, such as promoting less toxic alternatives and, if feasible, reducing all pesticide use.

Anionic surfactants were found at an average of 0.57 ppm (or 570 ppb) in every body of water we tested. This just exceeds the current EPA national secondary drinking water standard for surfactants, and based on the limited environmental toxicology that exists, also poses a potential threat to aquatic wildlife including salmonids.

Anionic surfactants are five hundred times more concentrated in the waters tested than pyrethroid pesticides. This is not surprising, given the fact that soaps, shampoos and other household cleaning products (not to mention garden products that use surfactants as sticker-spreaders) are consumed in much greater quantities in the average household, than pesticides. At the same time, pyrethroids tend to be much more toxic, on the whole, than most anionic or non-ionic surfactants.

County policy makers should also take into consideration the cumulative effect of pyrethroid pesticides and other pesticides in the environment (Capkin et al. 2006; Scholz et al. 2006), as well as the synergistic effect of surfactants on pesticides (Xie et al. 2005).

Our results suggest the advisability of taking the following actions:

- Reduce or eliminate the sale and use of products containing the pyrethroid pesticides most toxic to Pacific salmon;
- Encourage reduced household use of all pesticides and detergents;
- Promote better public understanding of the role of septic drain fields and surface water runoff in the transportation of pesticides and surfactants in aquatic ecosystems;
- Focus public action on outdoor uses of pesticides and surfactants including farms, gardens, washing motor vehicles and boats;
- Establish a program to monitor pyrethroid pesticides and LAS surfactants in water, sediments and appropriate indicator animal species in at least the streams and lakes of the county that currently have salmonid populations;
- Support further research to collect baseline data on the current distribution of other pesticides and herbicides in widespread use in San Juan County.

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Appendix A

**FIELD MEASUREMENTS AND TEST RESULTS, BY WATERSHED**

Map reference	ORP	NO <sub>3</sub>	Pyrethroid sediment	Pyrethroid Water	Anionic surfactants	Non-ionic surfactants
	mV	ppm	ppb	ppb	ppm	ppm
Bayhead Marina	117.8	19.5	2.10	0.59	0.80	•
			•	0.25	0.82	4.9
			•	0.61	0.85	1.1
Beaverton Valley	42.1	22.2	•	•	0.31	•
			•	•	0.30	•
			0.11	•	0.31	•
Boyce Road	80.6	24.4	•	1.50	0.22	•
			0.08	18.0	0.18	•
			0.33	2.34	0.21	3.6
Briggs Lake	83.1	5.0	1.74	1.04	0.28	
			1.56	1.20	0.26	
			1.15	1.20	0.33	
Cascade Creek	41.6	29.4	1.97	•	0.36	•
			•	•	0.26	•
			1.31	•	0.36	•
Cascade Lake	38.7	6.7	0.67	0.22	0.30	
			0.81	0.22	0.21	
			0.97	•	0.27	
Crow Valley	54.9	2.55	0.32	0.48	0.45	•
			10.71	0.51	0.40	•
			2.50	•	0.74	•
Davis Bay West	82.3	4.4	1.53	0.50	0.90	
			1.32	•	1.10	
			2.02	2.34	1.00	
Diamond Lake	33.4	10.7	•	•	0.34	
			•	•	0.33	
			0.09	•	0.36	
Doe Bay	93.1	2.78		10.6	0.35	
				3.60	0.34	
				0.93	0.33	
Doe Bay	125.4	24.3	0.28	0.55	0.93	6.2
			1.44	0.70	0.93	2.3
			4.53	0.47	1.00	4.9
Eastsound slough	22.6	13.0	•	1.80	1.6	•
			•	0.80	1.2	•
			•	0.93	0.6	•

Map reference	ORP	NO <sub>3</sub>	Pyr sed	Pyr water	Anionic	Non-ionic
False Bay	39.1	16.9	0.62	•	0.38	12.6
			0.33	•	0.37	6.2
			0.39	0.22	0.36	•
Fowler's Pond	-204.5	15.0	0.29	•	0.47	
			•	•	0.38	
			•	•	0.38	
Friday Harbor sewer	81.3	18.7		0.54	0.63	•
				•	0.74	•
				•	0.69	•
Frog Pond	10.5	23.9	0.59	0.67	0.38	
			0.45	1.15	0.39	
			0.81	0.63	0.38	
Garrison Bay	81.2	11.3	0.51		1.06	•
			0.49	3.64	1.04	•
			•	1.18	1.03	•
Hummel Lake	77.7	0.2	1.17	1.70	0.34	•
			0.63	•	0.30	•
			0.39	2.40	0.50	•
Killebrew Lake	44.4	15.7	•	•	0.40	
			•	•	0.33	
			•	•	0.34	
Lopez Village	36.8	5.1	15.50	1.46	1.22	•
			3.24	0.94	0.81	•
			2.23	2.40	0.82	3.2
Mountain Lake	146.6	3.8	0.54	0.57	0.49	4.8
			1.67	0.48	0.34	•
			1.46	1.10	0.51	7.2
Ole's Pond	78.7	15.9	0.56	1.40	0.53	
			•	3.10	0.56	
			•	1.12	0.56	
Pickett Springs	28.4	6.88	•	0.96	0.69	•
			0.45	3.00	0.58	5.0
			0.87	10.1	•	•
Richardson Pond	107.9	10.4	1.26	0.44	1.04	3.8
			•	0.51	1.18	1.0
			0.81	0.63	1.19	2.2
Roche Harbor	78.5	8.5	1.94	1.20	0.32	
			1.85	1.46	0.30	
			1.51	5.80	0.31	
Skull Creek	129.2	7.6	3.79	0.56	0.93	•
			1.15	0.39	1.06	•
			1.35	0.41	1.00	•

Map reference	ORP	NO <sub>3</sub>	Pyr sed	Pyr water	Anionic	Non-ionic
Sportsman Lake	19.8	12.0	1.66	•	0.31	•
			6.65	•	0.32	•
			1.44	1.38	0.32	•
Summit Lake	42.7	28.7	2.44	0.32	0.34	
			1.19	5.60	0.37	
			4.44	6.60	0.35	
Summit Lake	139.6	13.4	2.89	1.21	0.68	2.3
			3.33	0.74	0.56	2.3
			•	0.65	0.49	2.2
Terrell Road	-107.0	22.2	•	•	0.49	•
			0.12	•	0.50	•
			•	•	0.52	•
Trout Lake	81.9	16.1	•	0.54	0.32	•
			•	1.26	0.17	•
			•	8.10	0.44	•
Victorian Valley	50.9	1.9	0.99	•	1.2	•
			0.16	•	1.3	5.9
			0.28	•	1.3	•
West Beach	17.2	10.4	2.60	•	0.44	•
			3.25	•	0.42	5.9
			3.87	•	0.45	•
Wold Road	74.8	16.6	1.08	0.96	0.46	•
			0.80	3.06	0.50	•
			0.97	0.80	0.69	•

• Below limit of detection

Appendix B

**TOXICITY AND FATES OF PESTICIDES AND HERBICIDES  
IN COMMON USE IN SAN JUAN COUNTY**

Organized by their acute toxicity to rainbow trout (*Oncorhynchus mykiss*)  
Pyrethroid pesticides highlighted by shading

Active ingredient	Chemical Family	Acute toxicity in ppb or $\mu\text{g}/\text{bee}$			Max persistence in days		BCF
		Rainbow	Daphnia	Honeybee			
Bifenthrin	Pyrethroid	0.15	1.6	0.015	244	$\infty$	11750
Esfenvalerate	Pyrethroid	0.25	0.9	0.06	44	10	3250
Deltamethrin	Pyrethroid	0.26	0.56	0.0015	104	48	1400
Cyhalothrin	Pyrethroid	0.46	380	0.027	82	-	1950
Cyfluthrin	Pyrethroid	0.47	0.16	0.001	54	215	506
Fenbutatin	Organotin	1.1	480	200	365	1	730
Chlorpyrifos	Organophosphate	1.3	0.1	0.059	141	72	1374
Tralomethrin	Pyrethroid	1.6	0.04	0.13	27	$\infty$	1200
Rotenone	Cubé resin	1.9	4	0.24	3	1	26
Etofenprox	Pyrethroid	2.7	1.2	0.13	25	$\infty$	2000
Phenothrin	Pyrethroid	2.7	4.3	-	1	-	730
Cypermethrin	Pyrethroid	2.8	0.3	0.02	199	179	1204
Fenvalerate	Pyrethroid	3.6	0.03	0.23	40	115	1664
Prallethrin	Pyrethroid	12	6.2	-	-	-	-
Permethrin	Pyrethroid	12	6	0.029	42	31	300
Tetramethrin	Pyrethroid	16	45	0.16	3	-	-
Bifenazate	Hydrazine	17	500	8.5	5	1	-
Malathion	Organophosphate	22	0.7	0.16	1	98	103
Pyrethrin	Pyrethroid	32	2.5	0.15	12	35	-
Imiprothrin	Pyrethroid	38	5.6	-	15	59	-
Disulfoton	Organophosphate	39	1.3	4.1	30	300	460
Aminopyralid	Pyridine	100	100000	100	147	$\infty$	100
Naphthalene	PAH	110	15000	-	-	$\infty$	-
Hydramethylnon	Hydrofluorocarbon	160	1140	30	45	10	34900
Resmethrin	Pyrethroid	170	3.7	0.063	30	485	68
Captan	Phthalimide	186	7100	100	70	$\infty$	140
Pyriproxifen	Pyridine	270	400	100	25	$\infty$	1379
Neem oil	Triterpenoid	480	1160	2.5	26	$\infty$	-
Methoxychlor	Organochlorine	520	0.78	23.6	120	-	16222
<i>B. thuringensis</i>	Pathogen	656	13000	-	120	-	-
MGK264	Pyrethroid	1400	2300	-	-	-	-
Carbaryl	Carbamate	2600	6	0.14	99	12	44
Tebuconazole	Carbazate	4400	2790	83.5	610	$\infty$	78
Glyphosate	Auxin	8200	-	100	180	$\infty$	0.5
Copper sulfate	Metallic salt	13200	2300	23.5	$\infty$	$\infty$	-
Allethrin	Pyrethroid	19000	21	3.4	60	-	3281
Diquat dibromide	Organobromide	21000	1200	13	$\infty$	$\infty$	1
Spinosad	Spinosyn	30000	1400	0.0029	15	200	0.1
Metaldehyde	Cyclooctane	75000	78400	87.5	73	$\infty$	11
Pelargonic acid	Vanillylamide	91000	96000	-	-	-	-
Imazapyr	Imidazolinone	100000	100000	25	90	30	2.54
Acephate	Organophosphate	110000	67200	1.2	3	50	0.03

Trichlopyr	Pyridine	117000	132000	100	54	9	0.77
Sulfur	Non-metallic element	180000	5000000	50	1500	∞	-
Imidacloprid	Neonicotinoid	211000	85100	0.0037	341	∞	0.61
Aldehyde	Benzaldehyde	569000	17000	-	-	-	-
Triforine	Piperazine	1000000	25000	10	21	2	1

Notes:

The main source for this table is FOOTPRINT, the European Community ecotoxicology database; it is accessible online at <http://sitem.herts.ac.uk/aeru/footprint/en/index.htm>

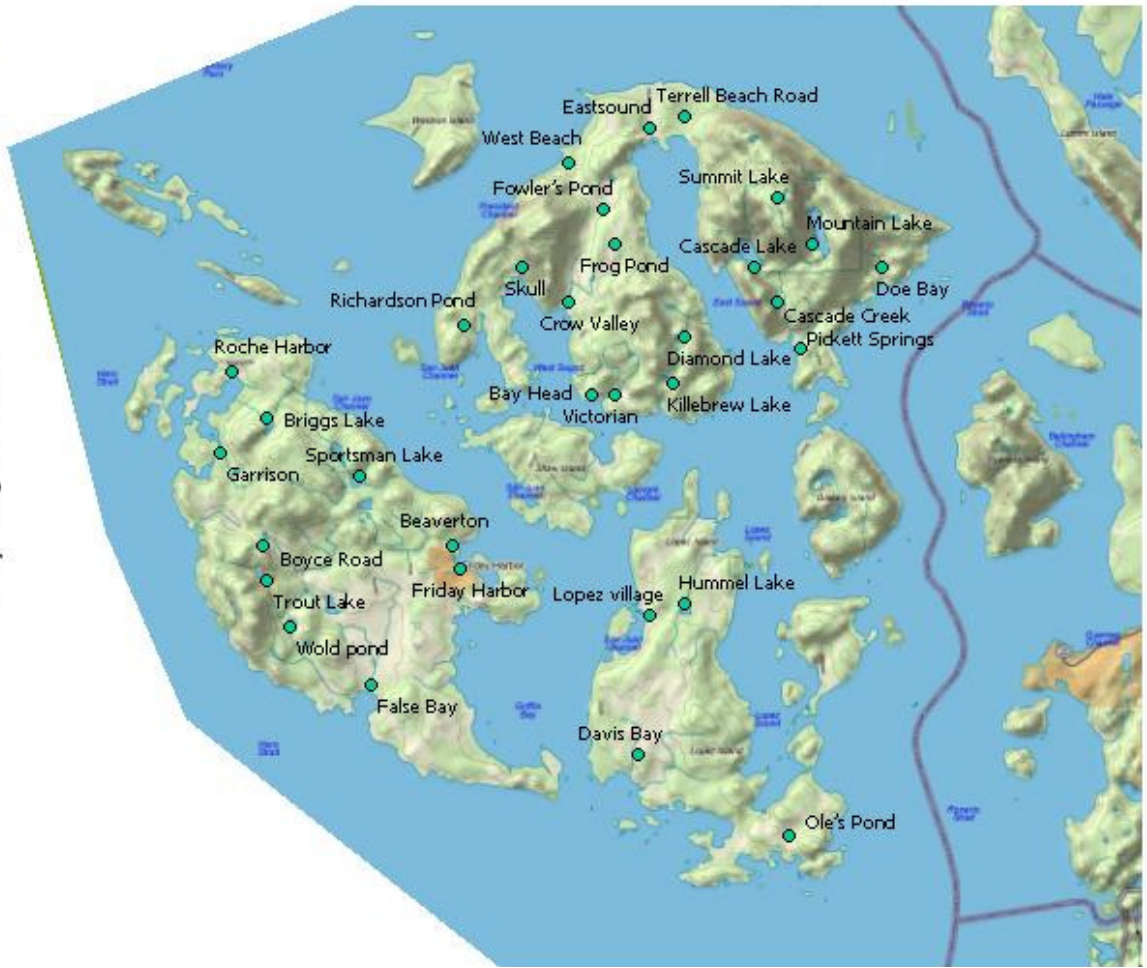
Acute toxicity is shown as LC50 (Lethal Concentration 50%) in parts per billion (micrograms per liter of water, µg/L) for rainbow trout and the crustacean *Daphnia magna*; and as LD50 (Lethal Dose 50%) in micrograms per bee for honeybees

Maximum observed persistence shown in whole days; persistence may vary greatly depending on temperature, pH (acidity), and light

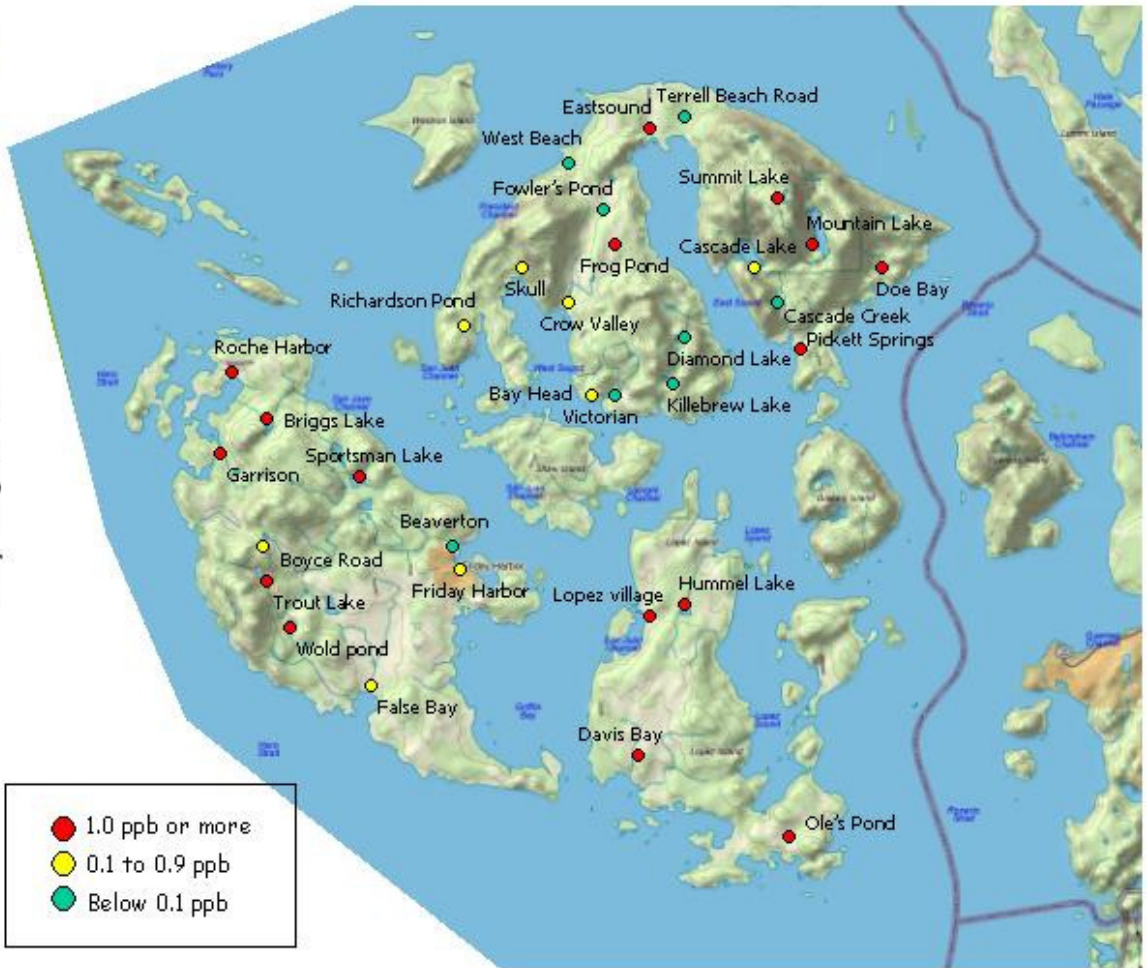
BCF = Bio-Concentration Factor (from FOOTPRINT)



Map 1  
SJC Household Toxics Study  
Surface water sampling sites  
July-August 2008

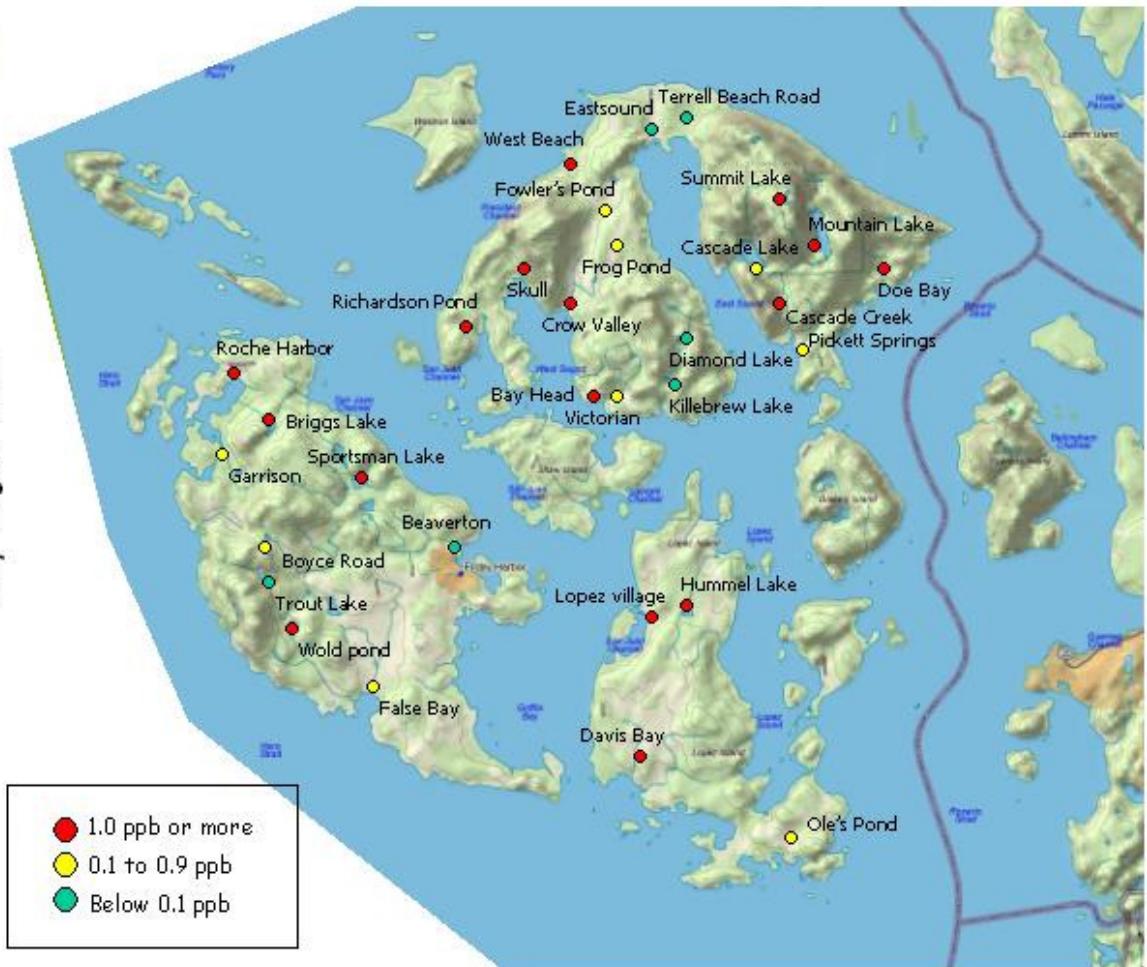


Map 2  
SJC Household Toxics Study  
Surface water pyrethroid test results  
July-August 2008

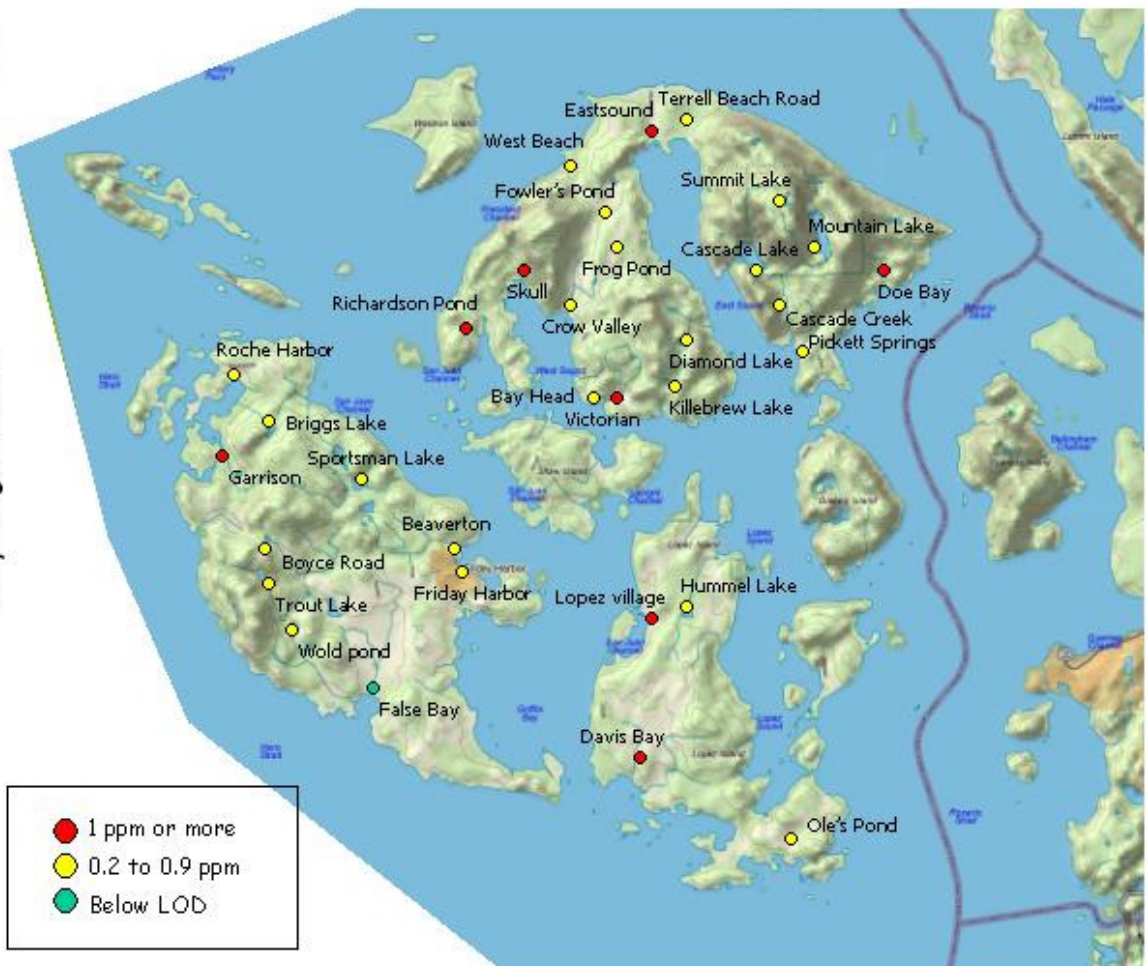




Map 3  
SJC Household Toxics Study  
Sediment pyrethroid test results  
July-August 2008



Map 4  
SJC Household Toxics Study  
Surface water anionic surfactant results  
July-August 2008





Map 5  
SJC Household Toxics Study  
Surface water non-ionic surfactant results  
July-August 2008

