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Pesticides Associated with Suspended Sediments Entering San Francisco Bay Following the First Major Storm of Water Year 1996

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ABSTRACT: Estuaries receive large quantities of suspended sediments following the first major storm of the water year. The first-flush events transport the majority of suspended sediments in any given year, and because of their relative freshness in the hydrologic system, these sediments may carry a significant amount of the sediment-associated pesticide load transported into estuaries. To characterize sediment-associated pesticides during a first-flush event, water and suspended sediment samples were collected at the head of the San Francisco Bay during the peak in suspended sediment concentration that followed the first major storm of the 1996 hydrologic year. Samples were analyzed for a variety of parameters as well as 19 pesticides and degradation products that span a wide range of hydrophobicity. Tidal mixing at the head of the estuary mixed relatively fresh suspended sediment transported down the rivers with suspended sediments in estuary waters. Segregation of the samples into groups with similar degrees of mixing between river and estuary water revealed that transport of suspended sediments from the Sacramento-San Joaquin drainage basin strongly influenced the concentration and distribution of sediment-associated pesticides entering the San Francisco Bay. The less-mixed suspended sediment contained a different distribution of pesticides than the sediments exposed to greater mixing. Temporal trends were evident in pesticide content after samples were segregated according to mixing history. These results indicate sampling strategies that collect at a low frequency or do not compare samples with similar mixing histories will not elucidate basin processes. Despite the considerable influence of mixing, a large number of pesticides were found associated with the suspended sediments. Few pesticides were found in the concurrent water samples and in concentrations much lower than predicted from equilibrium partitioning between the aqueous and sedimentary phases. The observed sediment-associated pesticide concentrations may reflect disequilibria between sedimentary and aqueous phases resulting from long equilibration times at locations where pesticides were applied, and relatively short transit times over which re-equilibration may occur.

Introduction

Onset of winter storms in California after a typical prolonged summer dry period causes runoff that transports sediment from the Central Valley downstream, and eventually into the San Francisco Bay. The majority of suspended sediments are usually transported into estuaries generally, and the San Francisco Bay in particular, during the high river flow that follows the first major storm of the year (Meade 1972; Goodwin and Denton 1991). This event is often referred to as the first flush.

Pesticides associated with first-flush sediments may contain different amounts and types of pesticides than found associated with suspended sediments at other times of the hydrologic year. Equilibrium partitioning models (e.g., DiToro et al. 1991; Liljestrand and Lee 1992) and field studies (e.g., Domagalski and Kuivila 1993; Pereira et al. 1996) indicate that some pesticides may be concentrated on suspended sediments. Because suspended sediments may carry appreciable amounts of associated pesticides, sediment transport may be an important mechanism for introduction of pesticides into the San Francisco Bay (e.g., Domagalski and Kuivila 1993; Bergamaschi et al. 1997).

In the estuary, the first-flush sediments represent an important food source for filter-feeding organisms such as clams (Canuel et al. 1995). These sediments often settle in the estuary, giving them a longer residence time in estuaries than in water (Meade 1972; Schubel and Carter 1984). This temporally focused introduction of sediments into estuaries, the majority occurring immediately following the first winter storm, followed by deposition in the estuary may provide a mechanism for increased exposure to pesticides during biologically sensitive times, even if dissolved pesticide concentrations are low during these times. Pesticides associated with suspended sediments, particularly first-flush sediments, may have different environmental effects in estuaries than dissolved pesticides.

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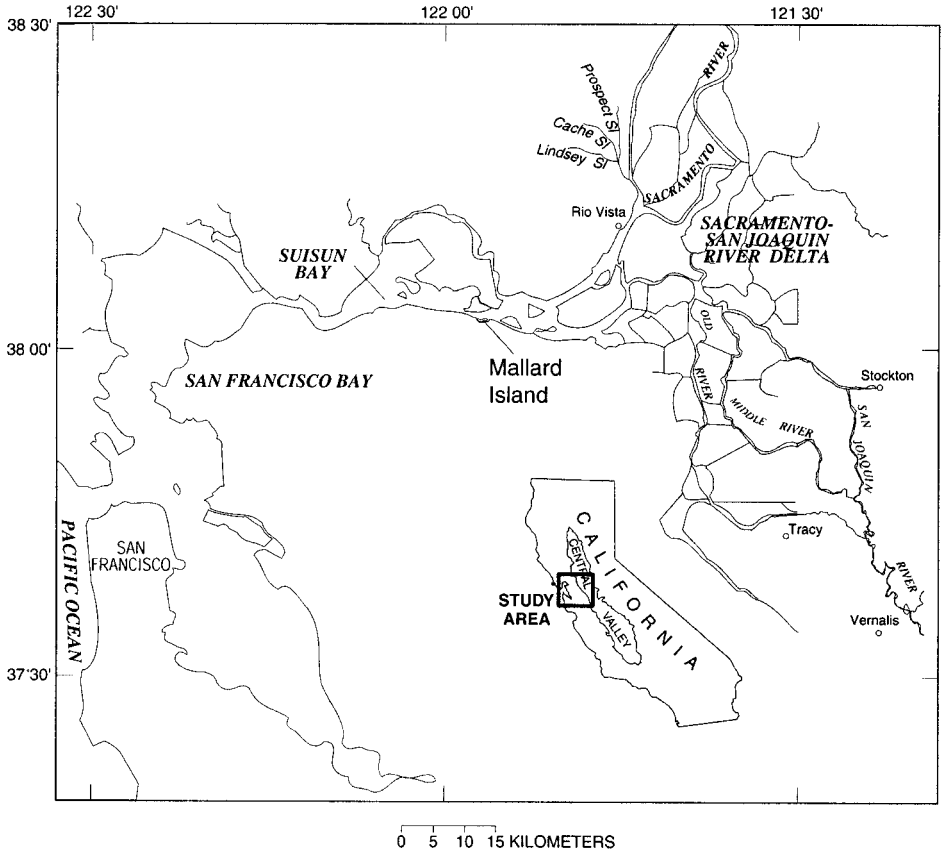

Fig. 1. Location of sampling site in relation to San Francisco Bay, and the Sacramento and San Joaquin Rivers.

Few data have been collected that address the potential for first-flush sediments to carry pesticides into estuaries and help understand the dynamics that control the transport process. This information is necessary to estimate risks of applied compounds to aquatic life, particularly benthic or epibenthic filter feeding organisms (e.g., Leight and Van Dolah 1998). We are aware of no other study that specifically focuses on this issue.

Suspended sediments pose particular challenges both for sampling and analysis (Bergamaschi et al. 1999); they must be extracted from large volumes of water and contain more interfering analytes than bed sediments. Sampling during a first-flush event poses logistical challenges as well; samples must be intensively collected as the peak in sediment transport occurs—an unpredictable event. These logistical and analytical challenges have limited examination of this mode of pesticide introduction into estuaries.

The purpose of this study was to examine temporal variability in sediment-associated pesticide (SAP) concentrations entering San Francisco Bay during the first flush in a region of the estuary subject to extensive tidal mixing. Examination of temporal variability during the peak in sediment transport permits examination of dynamic processes that may affect pesticide concentrations during transport and tidal mixing. Understanding the processes that control SAP concentrations during the time of the first flush is important to better understand interactions of the river system with the estuary, as well as to effectively design sampling and monitoring programs that capture variability within tidal environments.

Study Area

Samples were collected from a gage house on Mallard Island, California, located just south of the main shipping channel through North San Francisco Bay, 5 miles downstream of the confluence of the Sacramento and San Joaquin Rivers (Fig. 1), near the landward extent of the San Francisco Bay. The estuary receives drainage from California's Central Valley through the Delta formed by the confluence of the Sacramento and San Joaquin Rivers. Both the Central Valley and the Delta are areas of intense agricultural activity in which more than 160 different pesticides are applied (California Department of Pesticide Regulation 1996), pro-

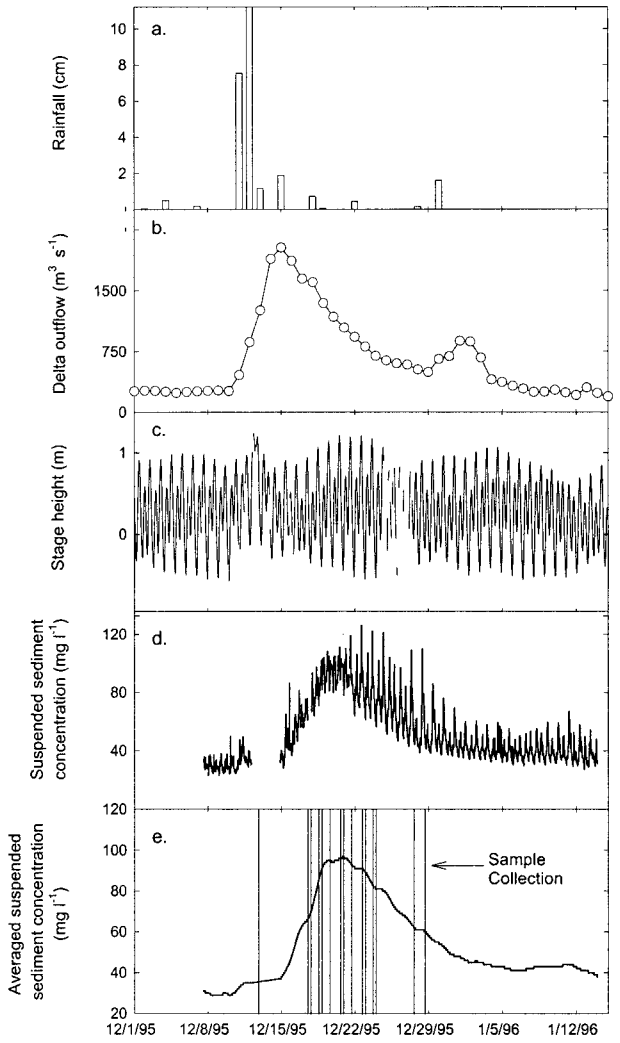

Fig. 2. Graphs of a) Mallard Island local rainfall (Fairfield, California; National Oceanic and Atmospheric Administration 1995), and b) relation to increase in daily calculated Delta outflow (California Department of Water Resources 1986). The graphs of c) stage height (California Data Exchange Center 1998) and d) suspended sediment concentration (Buchannan and Schoellhamer 1998) show the dependence of suspended sediment concentration on tidal currents. e) Tidally averaged suspended sediment concentrations (calculated with a low-pass numerical filter according to the method presented in Schoellhamer 1996) indicate when during the peak in suspended sediment concentration the samples were collected. Vertical bars are sample collection times.

viding ample opportunity for pesticides to enter the San Francisco Bay. Samples were collected following the first major storm of the season, December 11–13, 1995 (Fig. 2a), and during a period of the highest recorded suspended sediment concentrations of the 1996 water year (Schoellhamer 1997). The first major storm is defined as the first storm to cause flood runoff sufficient to double the baseline hydrograph. The California Department of Water Resources and the U.S. Geological Survey maintained a continuous record of optical backscatter data, stage height, and other ancillary parameters during the study.

Following the storm, the volume of water flowing into San Francisco Bay, i.e., Sacramento-San Joaquin River Delta outflow, rose from 250 m^3 s^{-1} on December 11 to a peak in outflow of 2,000 m^3 s^{-1} on December 15 (Fig. 2b). Delta outflow is a calculated value, and represents net non-tidal outflow, including the contribution of the tributaries of the Sacramento and San Joaquin Rivers as well as smaller and local inputs (California Department of Water Resources 1986). The peak in suspended sediment concentration (SSC) occurred on the falling limb of the peak in Delta outflow, and indicates arrival of suspended sediment transported from California's Central Valley to the San Francisco Bay (Fig. 2d). Tidally averaged SSC (Schoellhamer 1996) in Delta outflow in the Sacramento River at Mallard Island increased to 97 mg l^{-1} following the storm (Fig. 2e,f), from a base level of 30 mg l^{-1}.

Measurements of stage height at the Mallard Island site indicate it has a strong tidal influence. The stage height ranged nearly 2 m during the sampling period, with an average intertidal range of 0.4 m, and associated spring-neap variability (Fig. 2c). The SSC ranged from a high of 126 mg l^{-1} to a low of 23 mg l^{-1}, over the course of the sampling period, and there was a large change in SSC between ebb and flood tides (Fig. 2d). The intertidal change in SSC ranged from a high of 70 mg l^{-1} to a low of 15 mg l^{-1} during the sampling period, with an average intertidal change of approximately 30 mg l^{-1} (Fig. 2d).

Materials and Methods

Suspended sediment samples were collected at times of predicted slack current, either slack after ebb tide or slack after flood tide. Despite the bidirectional tidal currents, the net flow was in the direction of the estuary during the collection period, moving the peak in suspended sediment past the sampling site. The sampling strategy was developed to provide the best match to daily residual near-surface SSC obtainable from a twice-a-day sampling (Jennings et al. 1997). The daily residual sediment concentration is that concentration not attributable to tidal currents, as determined by low pass filtration of the optical backscatter data (Schoellhamer 1996). Due to mechanical and operational difficulties, it was not possible to collect every sample indicated by the strategy.

Large volume water samples (approximately 100–200 l, depending on SSC) were collected from 1 m below the surface into 37-l stainless steel con-

tainers using a high volume peristaltic pump fitted with Teflon inlet and outlet tubing, and transported to nearby facilities where 2 l were removed for dissolved pesticide analysis and 250 ml removed for analysis of sediment parameters. The remaining sample was pumped at 2 l min^{-1} into a high-speed continuous-flow centrifuge operating at 9,500 g. The water remaining in the centrifuge bowls after centrifuging was then used to wash out the isolated suspended sediments, and the resulting slurry was refrigerated for transport to the laboratory. At the laboratory, suspended sediment isolates were dewatered further by centrifuging in 50-ml Teflon tubes for 20 min at 15,000 RPM on a refrigerated centrifuge. The water was decanted and sediments were homogenized with a mortar and pestle because grain sortation was observed to occur during centrifugation. Samples were then frozen until analysis. (The use of brand names is for identification purposes only and does not constitute endorsement by the U.S. Geological Survey.)

Continuous-flow centrifugation was used for suspended sediment isolation in this study because of the large sample requirements (~5 g), and the difficulties associated with filtration of large volumes of water for suspended sediment isolation. It is our experience that suspended sediments occurring in this system during the first flush tend to be much finer grained and thus much more difficult to filter than bed sediments or suspended sediment collected from high-velocity reaches of river systems. The use of flow-through centrifugation has been extensively tested and compares favorably with other isolation techniques, including filtration (Horowitz et al. 1989; Rees et al. 1991).

Suspended sediment samples were analyzed for pesticide content according to the method described by Bergamaschi et al. (1999). Briefly, 1–10 g of wet sample were dried by grinding with sodium sulfate. Samples were extracted three times with fresh methlyene chloride in 200-ml Teflon bottles using an ultrasonic processor. The three extracts were composited and concentrated first by rotary evaporator, then under a stream of nitrogen to approximately 250 μl. Interfering co-extracted compounds were removed by passing the sample through a 1 cm internal diameter (i.d.) 6 g column of 2% deactivated Florisil. The 250-μl sample extracts were loaded onto the cleanup column and eluted with 40 ml hexane:ethyl ether (4:1 by volume), then concentrated on a rotary evaporator and dried under nitrogen to a final volume of 1 ml. Sulfur removal was accomplished by extraction against an aqueous solution of tetrabutylammonium hydrogen sulfite and the extract dried over a sodium sulfate column. Finally, internal standards (deuterated acenapthene, pyrene and phenanthrene) were added and the sample reduced to a final volume of 100 μl for chromatographic analysis. This method was developed specifically for use with ion-trap mass spectrometry (ITMS), and many spike recovery experiments and other quality assurance protocols were conducted to demonstrate its effectiveness and ensure matrix effects did not affect analytical determinations (see Bergamaschi et al. 1999). Independent analysis of replicate suspended sediment samples using this method yielded concentrations agreeing, on average, within 17%, depending on the measured value (Bergamaschi et al. 1999). Complete analysis of blanks were conducted on alternate analytical runs, approximately every 20 analyses. Other quality control procedures are described in Bergamaschi et al. (1999).

Samples collected for dissolved pesticide analysis were analyzed according to the method described in Crepeau et al. (1994). Briefly, 1 l of sample was filtered through a 142-mm diameter precombusted, 0.7-μm pore size glass fiber filter, and then extracted onto an extraction cartridge containing 3 cm^3 C-8 solid phase. Cartridges were dried under a stream of carbon dioxide and frozen until analyzed. On the day of analysis, the cartridges were thawed and sample extracts were eluted with 6 ml of a 1:1 (by volume) mixture of hexane and diethyl ether. Eluates were concentrated under a stream of nitrogen to about 200 μl, where upon internal standards (deuterated acenapthene, pyrene, and phenanthrene) were added and the mixture concentrated to a final volume of 200 μl. Blanks, replicates, and other quality control checks were routinely performed; see Crepeau et al. (1994) for details.

Chromatographic separations, identification, and quantification of dissolved pesticides and SAPs were accomplished using a gas chromatograph (GC) coupled to an ion-trap mass spectrometer. The GC was equipped with a 30 m 5%-phenyl methylpolysiloxane bonded phase 0.25-mm i.d.-fused silica capillary column, and the oven temperature programmed to provide full baseline separation of all analytes (Crepeau et al. 1994; Bergamaschi et al. 1999).

Analytes present in sample extracts were identified by the presence of at least the 3 most abundant ions of the corresponding standard in the expected ratios at the identical retention time of pure standard solutions. Analytes were quantified by comparing the relative response of each analyte to the response of a known amount of the most closely eluting internal standard spiked into the extract in the final step of preparation. The relative response of each analyte was calibrated daily by an-

alyzing a suite of standards. Calibration standards and standard solutions of known concentration were interspersed with samples during analysis to ensure analytical performance did not deteriorate during successive chromatographic runs. Duplicate chromatographic analyses of each extract were performed to verify analyte identification.

Data from below the method detection limit (MDL) of the analytical method are included in this study as these data are the best estimate of SAP content. Data from below the MDL are clearly identified in the data tables to indicate the greater statistical uncertainty of these values. Use of data from below the MDL is routine in environmental studies where the intent is to understand environmental processes because the purpose of the MDL does not necessarily support the goals of environmental studies (Clarke 1998; Childress et al. 1999). The MDL is defined as the standard deviation of 7 or more analyses divided by the student's *t* statistic. It was developed by the U.S. Environmental Protection Agency to estimate the probability of reporting a false positive test result to less than 1% when using one-dimensional detection systems, important in regulatory and compliance monitoring applications. For one-dimensional detection systems, such as electron capture and flame ionization detection, detector response at a given chromatographic retention time is the only measure of analyte concentration and identity. Use of ITMS detection provides additional dimensions (ion abundances) that improve analyte identification and significantly reduce the probability of false positive reporting (Clarke 1998; Childress et al. 1999).

In environmental studies, use of below-MDL data is warranted, and permits greater interpretation of observed temporal and spatial variations, particularly in small data sets and when sample collection and analysis are laborious and expensive (Gilliom and Helsel 1986; Helsel and Gilliom 1986; Clarke 1998; Childress et al. 1999). Although many techniques for estimating the concentration of low-level detections have been proposed for one-dimensional detection methods (Gilliom and Helsel 1986; Helsel and Gilliom 1986; Clarke 1998; Childress et al. 1999), since multidimensional detection techniques were used, the quantity reported by the analysis is the best estimate of the actual concentration (Clarke 1998; Childress et al. 1999). Evidence that this approach was warranted is the smooth variation in SAP concentrations observed in mechanistically related samples (see discussion section).

The concentrations of pesticides not observed at concentrations sufficient for quantification are assumed to be zero for the purpose of statistical calculations and are reported as not observed in the tables. All differences between means discussed below were found to be significant using the *t*-test with a degree of significance at the $\alpha = 0.05$ level (Sokal and Rolf 1981).

Results

Sediment-Associated Pesticides

The distribution of SAPs entering the San Francisco Bay for the 15 suspended sediment samples collected during the first flush ranged in total SAP concentrations from 9.8 to 43.8 ng g^{-1} dry weight sediment (Table 1). Using the SSC measured at the time of sampling (Table 2), this translates to a range of 0.7 to 3.1 ng l^{-1} SAP in whole water (unfiltered) samples. There was no significant temporal variability in the total SAP concentrations over the sampling period.

Of the 19 pesticides assayed, an average of 10 were observed in each of the 15 samples. This frequency of occurrence is higher than the number detected previously in San Joaquin River suspended sediments using the same method (Bergamaschi et al. 1997). Most samples contained chlordane (sum of α and γ), chlorpyrifos, dacthal, DDD, DDE, DDT, molinate, oxyfluorfen, pebulate, and thiobencarb. Alachlor, endosulfan, eptam, sulfotep, and trifluralin were intermittent in their occurrence (Table 1). Of the compounds analyzed, only dieldrin, ethalfluralin, fonofos, and malathion were not found in any samples. Note that thiobencarb showed a temporal variation in occurrence; it was only found in samples collected after December 19 (Table 1).

DDT together with its metabolites DDD and DDE were generally the most abundant of the commonly occurring compounds, ranging from a total of 5.1 to 11.1 ng g^{-1}. Oxyfluorfen concentrations exceeded 20 ng g^{-1} in one sample (December 18 at 2020; Table 1), but generally was much lower. Endosulfan occurred in concentrations greater than 17 ng g^{-1} in two samples (Table 1), but was not observed in other samples.

SAP concentrations of chlorpyrifos and DDE in the Mallard Island samples were similar to concentrations measured in other suspended sediments collected in San Francisco Bay after spring rains in 1991 (Domagalski and Kuivila 1993) and to measurements of SAPs in the Sacramento River (Bergamaschi et al. 1997). Concentrations of organochlorine pesticides (DDD, DDE, DDT, chlordane) were much lower in the Mallard suspended sediments than in suspended sediments from the San Joaquin River and its tributaries in other years (Bergamaschi et al. 1997; Kratzer 1999).

TABLE 1. Sediment-associated pesticide concentrations for suspended sediment samples from Mallard Island Study site, California, December 1995. Pesticide concentrations are in ng g^{-1} dry weight of sediment. Endosulfan and chlordane values are the sum of all isomers. Total is the sum of observed pesticide values; — = not observed; numbers in parentheses are concentration below method detection limit, reported value is presented as the best estimate of actual value. Suspended sediment samples also were analyzed for the following pesticides, but none were observed (method detection limit in brackets): dieldrin [2.1], ethalfluralin [0.1], fonofos [0.9], and malathion [1.2].

Sample Date and Time	Alachlor	Chlordane	Chlorpyrifos	Dacthal	DDD	DDE	DDT	Endosulfan	Eptam	Molinate	Oxyfluorfen	Pebulate	Sulfotep	Thiobencarb	Trifluralin	Total SAP
December 12, 1995 1900	—	(1.5)	1.7	(0.4)	2.1	2.6	(1.9)	—	(0.5)	(1.1)	1.2	3.3	—	—	—	16.3
December 17, 1995 1210	—	(1.2)	—	(0.4)	3.3	6.9	(0.9)	17.7	(1.1)	(1.4)	6.2	—	—	—	1.3	40.6
December 17, 1995 1900	(0.1)	(1.0)	0.6	(0.4)	1.5	3.5	(2.4)	—	—	(0.8)	(0.5)	3.4	—	—	—	14.2
December 18, 1995 1250	(0.8)	(0.3)	—	—	1.5	3.7	(2.1)	—	(0.2)	(0.2)	—	(1.0)	—	—	—	9.8
December 18, 1995 2020	—	(0.7)	(0.5)	(0.6)	1.6	3.9	(2.0)	—	—	(1.2)	21.3	3.2	—	—	—	35.0
December 19, 1995 1405	—	(1.6)	(0.4)	(0.4)	1.1	2.6	(1.4)	—	(0.6)	(0.9)	(0.8)	(1.2)	—	—	—	10.9
December 20, 1995 1430	—	(2.7)	1.0	(0.5)	2.0	4.2	(2.7)	—	—	(1.0)	—	3.3	—	1.6	—	18.9
December 20, 1995 2140	—	—	1.7	(0.6)	2.1	5.1	(2.4)	—	—	(1.4)	2.2	3.7	—	—	—	19.4
December 21, 1995 1520	—	(1.1)	0.7	(0.5)	1.6	3.8	(2.1)	—	—	(1.4)	1.2	2.3	(0.3)	(1.4)	(0.5)	17.0
December 22, 1995 1600	—	(2.0)	1.3	(0.8)	1.9	5.2	(2.5)	—	—	(2.0)	3.8	—	—	2.4	(0.6)	22.6
December 22, 1995 2310	—	(0.5)	1.4	(0.6)	2.1	6.3	(1.6)	24.6	(1.1)	(1.6)	—	—	—	3.9	—	43.8
December 23, 1995 1650	—	(0.9)	(0.3)	(0.3)	1.0	3.0	(1.1)	—	—	(0.8)	1.7	—	—	(1.3)	(0.2)	10.7
December 23, 1995 2350	—	—	2.1	(0.6)	2.4	4.5	4.4	—	—	(1.5)	—	4.7	—	2.4	—	22.6
December 27, 1995 1450	—	(1.2)	1.0	(0.3)	1.0	3.3	(1.5)	—	—	(1.3)	1.4	(1.7)	—	1.6	—	14.2
December 28, 1995 1530	—	(0.8)	0.8	(0.3)	1.3	3.9	(1.4)	—	(0.7)	(1.2)	2.1	2.6	—	1.8	—	16.9

Dissolved Pesticides

Water samples collected concurrently with the suspended sediment samples were analyzed for pesticide content. Since floodwater flow is the mechanism by which suspended sediments are transported, the corresponding water samples should provide information about equilibration during transit. Although the analytical methods were considerably different, the two methods shared 11 analytes. In comparison to the suspended sediment samples, relatively few pesticides were found in the water samples; only 3 pesticides were observed. In part this lower frequency of pesticide occurrence may be the result of the higher concentrations required for observation in the much smaller water samples (1 liter) in comparison to the suspended sediment samples (~200 l). The only dissolved pesticides detected in the water sampled between December 12–28 were diazinon, metolachlor, and simazine (Table 3). The following pesticides were not detected in the dissolved phase in any of the samples: alachlor, atrazine, carbaryl, carbofuran, chlorpyrifos, cyanazine, dacthal, diazinon-oxon, diethatylethyl, eptam, fonofos, malathion, methidation, molinate, napropamide, pebulate, sulfotep, thiobencarb, and trifluralin. Simazine concentrations ranged from 15 to 76 ng l^{-1} and the highest concentrations occurred during the peak of SSC (Fig. 2).

Discussion

A myriad of factors may affect the export of particle-associated pesticides from the area of application into the hydrologic system. At the local scale, interaction between soils and pesticides, soil type, timing, amount and technique of pesticide application, volatilization and degradation of pesticides, equilibration with sediment-associated organic material, land use, field and catchment hydrology, irreversible sorption, as well as other parameters have been found to be important under different conditions or when studying different processes within the hydrologic system (Nowell et al. 1999 and references therein). Understanding the relative importance of these processes is a complex and daunting task in a drainage basin the size of California's Central Valley, and beyond the scope of this study. Variability due to many of these factors is undoubtedly important. Scaling arguments and simple empirical analyses eliminate many of these processes as critical variables when examining tidal time-scale variations. Application site parameters such as sediment or pesticide immobilization, volatilization, soil type, and mode of pesticide application result in variability temporally disconnected from the 2-wk period of this study;

TABLE 2. Organic carbon, total nitrogen, suspended sediment concentrations, and electrical conductivity for suspended sediment samples from Mallard Island study site, California, December 1995. Organic carbon and total nitrogen concentrations are in dry weight percent. (C:N)a = atomic ratio of organic carbon to total nitrogen; SSC = sediment concentration in mg l^{-1} at time of sampling (Buchanan and Schoellhamer 1998); electrical conductivity in microsiemens cm^{-1}; na = not available.

Sample Date and Time	%C	%N	(C:N)a	SSC	Electrical Conductivity
December 12, 1995 1900	2.59	0.19	15.9	na	11,580
December 17, 1995 1210	2.63	0.17	18.0	61.2	672
December 17, 1995 1900	2.17	0.16	15.8	na	366
December 18, 1995 1250	2.33	0.16	17.0	75.5	765
December 18, 1995 2020	2.09	0.15	16.2	89.2	250
December 19, 1995 1405	2.42	0.17	16.6	87.2	732
December 20, 1995 1430	2.03	0.14	16.9	88.7	735
December 20, 1995 2140	2.37	0.17	16.3	89.3	250
December 21, 1995 1520	2.30	0.16	16.8	93.3	596
December 22, 1995 1600	2.23	0.17	15.3	101	648
December 22, 1995 2310	2.20	0.16	16.0	64.5	220
December 23, 1995 1650	2.04	0.14	17.0	107	691
December 23, 1995 2350	2.23	0.15	17.3	58.9	242
December 27, 1995 1450	2.25	0.17	15.4	46.9	241
December 28, 1995 1530	2.59	0.20	15.1	48.0	232

either expected variations are much longer or shorter than the duration of the study or would cause variation at a different time. These processes may be removed from consideration. Regional factors such as field or catchment hydrology, soil type, and land use will tend to be homogenized because of the large scale of the Central Valley system. Timing of pesticide application may be eliminated as a potential controlling variable since the period of the study follows the first winter storm, and the vast bulk of pesticide applications occur in late winter, spring, and summer (California Department of Pesticide Regulation 1996). Evidence that application rate within the Central Valley is not a controlling variable is the lack of an apparent relationship between the amount of pesticide applied in the Central Valley and Delta region that drains into the estuary (Fig. 1 and Table 4) and the SAP concentration (Table 1).

TABLE 3. Measured pesticide concentrations in water samples. Values are in ng l^{-1}. — = not observed; numbers in parentheses are concentration below method detection limit (Crepeau et al. 1994). Water samples also were analyzed for the following pesticides, but none were observed (method detection limit shown in brackets): alachlor [22], atrazine [28], butylate [19], carbaryl [31], carbofuran [28], chlorpyrifos [35], cyanazine [50], dacthal [63], eptam [129], ethalfluralin [31], fonofos [25], malathion [44], methidathion [28], molinate [53], napropamide [28], pebulate [44], thiobencarb [60], and trifluralin [60].

Sample Date and Time	Diazinon [31]	Metolachlor [47]	Simazine [63]
December 12, 1995 1330	—	—	(23)
December 12, 1995 1330	—	(1)	(33)
December 17, 1995 1210	—	—	64
December 17, 1995 1210	(2)	—	(55)
December 17, 1995 1900	(6)	—	(38)
December 18, 1995 1250	(9)	(4)	69
December 18, 1995 1250	(5)	(2)	(57)
December 18, 1995 2020	—	—	(25)
December 19, 1995 1405	(2)	(5)	68
December 19, 1995 2050	(2)	(3)	(32)
December 20, 1995 1430	(7)	(4)	76
December 20, 1995 2140	—	(4)	(53)
December 21, 1995 1520	—	(6)	(59)
December 22, 1995 1600	—	(4)	(47)
December 22, 1995 2310	—	(3)	(15)
December 22, 1995 2310	—	(4)	(29)
December 23, 1995 1650	—	(6)	(48)
December 23, 1995 2350	—	(5)	(34)
December 27, 1995 1450	—	(4)	(28)
December 28, 1995 1530	—	(5)	(24)

Equilibrium distribution between sedimentary and aqueous phase is another possible factor affecting SAP concentrations observed in this study, and one that deserves greater scrutiny because of its effects in aquatic environments. Evidence that equilibrium partitioning does not control SAP content in these samples is corroborated in several ways. The equilibrium distribution of pesticides between water and suspended sediment is commonly described by the aqueous phase-sedimentary organic carbon partition coefficient, K_{OC}, which is defined as the concentration in a system at equilibrium of sorbed pesticide in ng g^{-1} of sedimentary organic carbon divided by the concentration of dissolved pesticide in ng ml^{-1}. One way to examine if equilibrium partitioning was an important determinant of SAP concentration and distribution is to examine the relationship between K_{OC} and pesticide content. Log K_{OC} values for pesticides in this study vary from 1.97 ml g^{-1} (molinate) to 5.38 ml g^{-1} (DDD and DDT; Table 4), indicating a wide range in expected equilibrium behavior for the suite of compounds analyzed.

K_{OC} values may be used to calculate equilibrium dissolved pesticide concentrations from SAP con-

TABLE 4. Pesticide log K_{oc} and usage data for the regions of the Delta and the Sacramento and San Joaquin drainage basins that drain into the Delta. Log K_{oc} values are in ml g^{-1}. Usage values are integrated amounts applied during registered activities for the portion of the Sacramento and San Joaquin River drainage basins, and the Delta that ultimately drain into the San Francisco Bay at Mallard Island for 1995. Usage values are in kilograms active ingredient applied. — indicates there was no recorded usage.

Pesticide	Log K_{oc}	[7]Usage
Alachlor[1,2]	2.28	12,521
Chlordane[2]	5.15	538
Chlorpyrifos[1,3]	3.93	380,113
Dacthal[4]	3.70	8.968
DDD[5]	5.38	—
DDE[5]	5.29	—
DDT[1,2,6]	5.38	—
Dieldrin[3,5,6]	4.08	—
Endosulfan[5]	3.37	16,842
Eptam[1,5]	2.38	100,309
Ethafluralin[4]	3.60	15,069
Fonofos[4]	2.94	18,584
Malathion[3]	3.10	87,851
Molinate[5]	1.97	647,312
Oxyfluorfen[4]	5.00	70,632
Pebulate[1]	2.66	91,243
Sulfotep[5]	2.82	32
Thiobencarb[4]	2.95	260,624
Trifluralin[1]	3.96	230,712

[1] Kenega 1980.
[2] U.S. Environmental Protection Agency 1994.
[3] Howard 1991.
[4] Wauchope et al. 1991.
[5] Montgomery 1993.
[6] Jury et al. 1987.
[7] California Department of Pesticide Regulation 1996.

centrations (for example, Domagalski and Kuivila 1993; Kratzer 1999). Calculated dissolved pesticide concentrations for waters in equilibrium with the Mallard Island suspended sediment samples (Table 5) were very different from measured dissolved pesticide concentrations (Table 3). Calculated concentrations of alachlor, eptam, molinate, pebulate, and thiobencarb were well above the dissolved pesticide MDL, yet none of these compounds were observed in the Mallard Island water samples. Also, there was no significant correlation between individual SAP concentrations and K_{OC} in this study. Nor were there significant relations between SAP concentrations and sediment organic carbon content or the sediment carbon quality as indicated by C : N (Table 2). Taken together, these observations indicate that SAP concentrations and distributions in these samples are not controlled by equilibrium between water and sediments, in agreement with what has been observed previously for suspended sediments in this region (Domagalski and Kuivila 1993; Pereira et al. 1996; Brown 1997; Kratzer 1999).

The short transit times between areas of pesticide application and the estuary may mean SAPs do not have sufficient time to reach equilibrium during the high flows that follow winter storms (Domagalski and Kuivila 1993; Jepsen et al. 1995; Lick and Rapaka 1996). If equilibrium is not achieved, higher than expected concentrations of SAPs will result. Because of this effect, suspended sediments may carry more pesticide from areas of pesticide use into estuaries than equilibrium models suggest, and hydrologic factors may control SAP concentration. These hydrologic variables include location of land uses within the drainage, differences in the transit time between drainage and the bay, the residence time of sediments within the drainage, and the effect of tidal cycles and river discharge on sediment transport and deposition (Schubel and Carter 1984; Kratzer 1999).

The pattern of occurrence of molinate and

TABLE 5. Calculated equilibrium dissolved pesticide concentrations. Calculated from pesticide concentrations on suspended sediments using K_{oc} values in Table 1. Values are given only for pesticides analyzed both in the dissolved and particulate phases. Values are in ng l^{-1}. Numbers in parentheses are concentration below method detection limit (Crepeau et al. 1994); — = not observed associated with suspended sediment.

Sample Date and Time	Alachlor	Chlorpyrifos	Dacthal	Eptam	Molinate	Pebulate	Thiobencarb	Trifluralin
December 12, 1995 1900	—	(9)	(4)	96	531	322	—	—
December 17, 1995 1210	—	—	(3)	173	574	—	—	(6)
December 17, 1995 1900	36	(3)	(4)	—	400	341	—	—
December 18, 1995 1250	181	—	—	(37)	90	97	—	—
December 18, 1995 2020	—	(3)	(6)	—	631	332	—	—
December 19, 1995 1405	—	(2)	(3)	103	437	117	—	—
December 20, 1995 1430	—	(6)	(4)	—	507	353	88	—
December 20, 1995 2140	—	(8)	(5)	—	641	345	—	—
December 21, 1995 1520	—	(4)	(5)	—	685	222	71	(3)
December 22, 1995 1600	—	(7)	(8)	—	972	—	122	(3)
December 22, 1995 2310	—	(7)	(6)	214	762	—	198	—
December 23, 1995 1650	—	(2)	(3)	—	430	—	69	(1)
December 23, 1995 2350	—	(11)	(5)	—	700	464	123	—
December 27, 1995 1450	—	(5)	(3)	—	613	163	81	—
December 28, 1995 1530	—	(4)	(3)	127	563	256	89	—

thiobencarb provides initial evidence that hydrologic variables rather than equilibrium distribution may be controlling SAP distribution. Molinate and thiobencarb are herbicides used in the cultivation of rice. They are applied in large amounts (Table 4) to rice fields in April–June (California Department of Pesticide Regulation 1996). In comparison with the other pesticide analytes, molinate has the lowest log K_{OC} (1.97), and thiobencarb has an intermediate value (2.95; Table 4), indicating that if allowed to equilibrate in aqueous systems, these pesticides should be among the first lost from the sediments. Nevertheless, they are observed 6 mo after application on suspended sediments in the aquatic system. This suggests that sediments bearing these compounds have a short hydrologic residence time in comparison to what may be a long time required for equilibration with the aqueous phase; if, indeed, equilibration is possible. The concentrations of molinate and thiobencarb on sediments delivered to the estuary must therefore be determined outside the aqueous environment, for example, by partitioning onto the sediments during wetting and drying cycles that occur in agricultural fields. When these sediments are eroded during storms, the result may be delivery of sediments with higher-than-expected pesticide concentrations into the estuary.

The distribution of DDT and its metabolites supports the notion that these sediments are relatively fresh in the hydrologic system. Agee (1986) has proposed that the ratio of DDT to DDT+DDE+DDD may be diagnostic of recent transport into the aquatic system since DDT degrades relatively rapidly into DDD or DDE once in the water. Values above 10% DDT are thought to be relatively fresh. Samples in this study averaged near 26% DDT/ΣDDT, consistent with a relatively recent introduction of the sediments into the aquatic system.

In San Francisco Bay, the interaction between river and tidal hydrodynamics clearly plays an important role in delivering sediments (e.g., Schoellhamer 1996), but how does it affect SAP concentration and distribution? At first glance, it appeared there was no temporal trend for total SAP concentration or in the number of pesticides detected in these samples, as would be expected if SAP distribution was controlled by hydrodynamics. This was in contrast to our previous study of sediments in a non-tidal reach of the San Joaquin River (using the same methods) where total SAP concentrations were higher on the rising limb of the SSC peak than on the falling limb (Bergamaschi et al. 1997, 1999). Detailed studies of the San Joaquin River and its tributaries indicated that temporal variations in SAP concentrations in the main stem of the river were strongly influenced by the different travel times for water from various tributaries to the sampling site and differences in the SAP concentrations associated with sediment from each tributary (Kratzer and Biagtan 1996; Kratzer 1999).

Clear temporal trends in SAP concentrations and distinct compositional characteristics of riverine sediments emerged when the data analysis included consideration of the tidal mixing of suspended sediments at the Mallard Island study site. Samples from the Mallard Island site integrated inputs from the Sacramento and San Joaquin Rivers and their tributaries, as well as local inputs within the Delta. In addition, samples from the Mallard Island site may also include estuarine sediments driven landward by tidal currents. The degree of tidal mixing at a given point in an estuary can be approximated by the salinity. Because the samples were collected at the times of slack water after the flood and ebb tides at Mallard Island (the uppermost reach of the estuary), they spanned a gradient of salinities. This variation in mixing between river and estuarine waters at Mallard Island is demonstrated by the variation in electrical conductivity (EC) of the samples (Table 2).

By using the EC it is possible to discern samples that are river dominated from those that are estuarine influenced (Table 2). Prior to arrival of the storm, EC values were above 11,000 (Table 2). The freshwater brought into the sampling site by the increase in Delta outflow lowered the EC value, and the EC of the samples collected after December 17 oscillated generally with the tide between values near 260 (257 ± 49) and near 700 (690 ± 58). The samples possessing EC values near 260 can be thought of as river dominated, while those with EC values near 700 can be thought of as having some influence of estuarine water, although probably a small one. Of greater importance may be that estuary-influenced samples have been subject to greater tidal mixing. As would be expected, the river-dominated samples were found on outgoing tides and later in the flood wave caused by the storm, while estuary-influenced samples were found earlier in the storm flood wave and on incoming tides (Figs. 2 and 3). Over the course of the sampling, the river-dominated samples declined in sediment concentration as the peak in suspended sediment passed, while the SSC of the estuary-influenced samples increased as the sediments entered the estuary (Fig. 3; Table 2).

Separation of samples into river-dominated and estuary-influenced groups revealed the importance of transport of sediments from the Central Valley and Delta on the occurrence and distribution of SAP at the Mallard island site. River-dominated samples contained 27% higher total SAP than es-

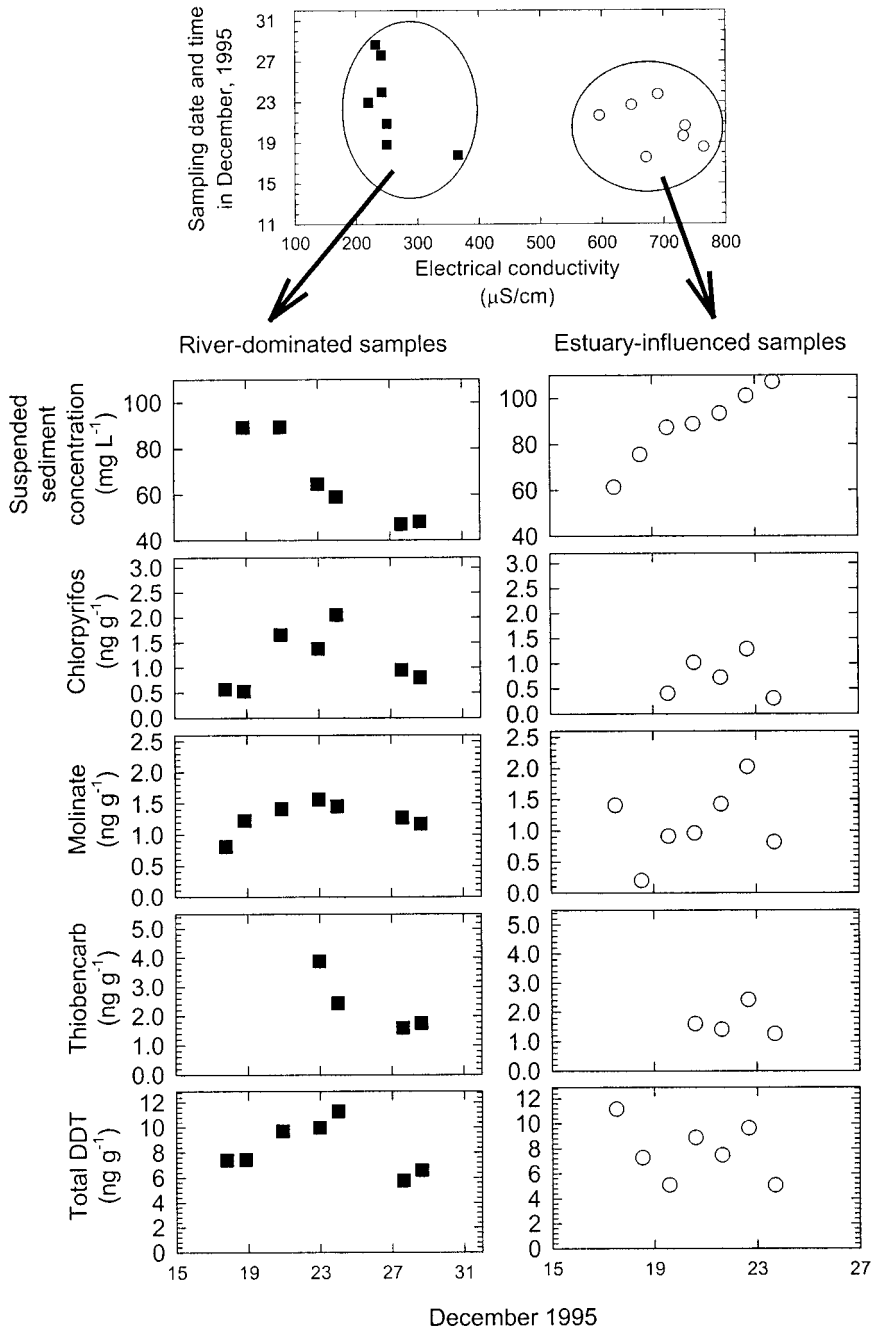

Fig. 3. Top panel indicates how Mallard Island samples after December 17 separate into two groups based on electrical conductivity. Examination of these two groups separately shows temporal trends suspended sediment concentration and selected pesticides for the river-dominated group. Suspended sediment concentration increases in the estuary-influenced group, but sediments are generally lower in pesticide content and do not show temporal trends. See text for explanation of terminology.

tuary-influenced ones. SAP concentrations of the most commonly observed modern pesticides (chlorpyrifos, dacthal, molinate, oxyfluorfen, pebulate, and thiobencarb; each occurring at least 4 times in both groups) were 46% higher in the river-dominated samples than the estuary-influenced ones (Table 1). Concentrations of pesticides also varied systematically with time in the river-dominated samples; peaking in concentration at the same time the peak in SSC passed the Mallard Island site (Figs. 2 and 3). The estuary-influenced samples did not show a similar temporal pattern.

The average concentration of the discontinued pesticide chlordane was twice as high in the estuary-influenced samples, although the average concentrations of ΣDDT in the two groups were indistinguishable (Table 1; Fig. 3). These results suggest that the sediment transported down the rivers, through the Delta, and into the estuary during the first-flush event was distinct in SAP content from suspended sediment already in the estuary mixed and carried back upstream during flood tides. The lower concentration of the modern pesticides in the estuary-influenced sediments may indicate that sediments in these samples had more time to equilibrate with surrounding waters than the more recent sediments in the river-dominated samples. The similar DDT/ΣDDT ratios in both groups indicate the equilibration time difference cannot be large.

One final consideration is whether the observed SAP concentrations will have any effect on the health of the ecosystem. U.S. Environmental Protection Agency chronic water quality criteria for protection of freshwater aquatic life contaminant chronic exposure levels have been established for 4 of the pesticides found in this study (chlordane, DDT, endosulfan, and chloropyrifos; U.S. Environmental Protection Agency 1996). Of these 4 pesticides, only the SAP concentrations of ΣDDT approached the criteria of 1 ng l^{-1}. The observed ΣDDT ranged from 0.4–1.0 ng l^{-1}. Despite these low concentrations of SAP, the SAP may still affect the estuary. Filter-feeding organisms have been shown to accumulate pesticides to levels far in excess of the associated sediment concentrations (for example, Pereira et al. 1996; Brown 1997). Also, these sediments may eventually equilibrate with their surroundings, raising aqueous concentrations of pesticides in pore waters.

Conclusions

The influence of hydrologic factors in determining the sediment pesticide content were apparent in this study, but temporal variability in pesticide concentration during the first flush was obscured by the complex tidal mixing in the region. Segregation of the samples into those having greater and lesser amounts of estuary influence revealed that transport from the Central Valley has an important influence on the concentration and distribution of SAP at the Mallard Island site and is important in the transport of pesticides into the estuary. The finding that the temporal variability is obscured by mixing but discernable when segregated by electrical conductivity is important to consider when designing studies sampling in tidally-dominated systems; electrical conductivity or a similar measurement should be used to ensure samples exposed to similar mixing histories are compared.

A large number of pesticides were found associated with suspended sediments entering the San Francisco Bay during the first storm of the water year 1996. The total amount of pesticides associated with the sediments were similar to those measured previously in Sacramento River suspended sediment, but much lower than those previously measured in San Joaquin River suspended sediment. Few pesticides were found in concurrent water samples and in concentrations much lower than may be expected from equilibrium partitioning between the aqueous and sedimentary phases. Observed sediment-associated pesticide concentrations may reflect disequilibria between sedimentary and aqueous phases resulting from long equilibration times at locations where pesticides were applied, and relatively short transit times for re-equilibration to occur. The resulting temporal trends were observed in pesticide content over the peak in suspended sediment transport when samples with similar mixing histories are segregated. This result indicates that low-frequency sampling and sampling strategies that do not compare samples with similar mixing histories will not elucidate basin processes.

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