

**Mercury Release from Delta Wetlands:
Facilitation and Fluxes**

An amendment to existing CALFED Projects #2000-G01

**Annual (Draft Final) Report
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INTRODUCTION

Background

Mercury (Hg) contamination in the Sacramento-San Joaquin Delta is a complex mixture of elemental Hg (Hg^0) derived from gold mining operations in the Sierra Nevada and sulfidic Hg (HgS) derived from the Hg mines in Coast Ranges. Due to mining activities, more than 800,000,000 cubic yards of Hg-laden sediment has been washed into the Sacramento River and Delta and continues today (Thompson, 1959; Alpers and Hunerlach, 1999). These sediments will pose an environmental hazard if they are (1) solubilized and (2) methylated in Delta and Estuary wetlands (See conceptual model, Figure 1; Bloom, 2002; Alpers and Hunerlach, 1999).

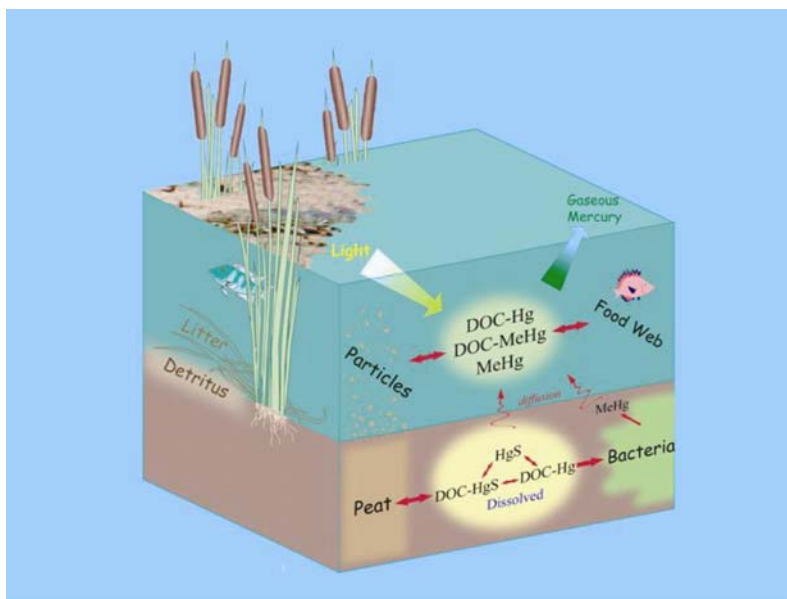


Fig. 1. Conceptual model of mercury-DOM interaction in peat sediments

Dissolved organic matter (DOM) plays an important role in mercury cycling and transport. Like mercury, DOM in the Delta is a complex mixture of different sources, with each source supplying DOM of different character and properties affecting the mercury interactions and cycling. This project focuses on evidence that tidal wetlands in the Delta convert insoluble forms of sedimentary mercury into methylmercury (MeHg) with the objectives of quantifying the capacity of DOM from representative Delta wetlands to solubilize the major sedimentary forms of mercury in the Delta – cinnabar, metacinnabar, and elemental mercury – and determining the dissolved and particulate fluxes of mercury and methylmercury at selected, representative wetland sites in the Delta.

The approach of the study is to 1) to expand the current work of the USGS on chemical composition of DOM from different sources within the Delta to include parameters related to mercury solubilization, binding, and partitioning; and 2) to expand current measurement of tidal fluxes of DOM to include mercury species by identifying correlates

measured in situ to predict real time fluxes of mercury and methylmercury in the complex hydrodynamic exchange of the tidal wetland environment.

Part 1 uses laboratory experiments to address the following:

- Binding of Hg to DOM
 - Equilibrium and binding site abundance experiments of the Delta's five main organic matter types
- Partitioning of Hg between particulate – colloidal and dissolved phases
 - Equilibrium experiments with five organic matter types and five particle types
- DOM enhanced dissolution of HgS
 - Release of mercury from mercury-rich endmember sediments (HgS and elemental Hg) upstream from Delta
- DOM-Hg photochemical reactivity
- Salinity effects on Hg geochemistry

Part 2 uses field studies consisting of:

- In-situ instrument deployment for high temporal resolution measurements of a suite of spectroscopic variables and water flux in tidal wetlands
- Intensive calibration sampling over tidal cycles
- Statistical determination of correlative relationships for accurate Hg and MeHg flux calculations
- Initial deployments identifying seasonal variability conducted at Browns Island (Figure 2), deployments identifying spatial variability after Browns Island work is completed



Figure 2. Primary site of the field study, Browns Island.

Goals and objectives

The project's primary objectives are:

OBJECTIVE 1: Quantify the capacity of DOM from representative Delta wetlands to solubilize the major sedimentary forms of mercury in the Delta, cinnabar (HgS), metacinnabar, and elemental Hg, as well as affect the partitioning of total Hg and MeHg between aqueous and particulate phases.

This part of the study will provide (1) a link between Hg binding and the environmental source of the DOM, as elucidated by the original DOM Sources study performed by the USGS; (2) a basis for evaluating the relationship between Hg binding and the chemical properties of the DOM; (3) potentially, a link to more readily measured chemical properties useful for predicting Hg binding at other sites; and (4) a means to evaluate, in conjunction with flux data, the role of DOM in Hg cycling in the Delta.

OBJECTIVE 2: Determine the fluxes of total Hg and monomethyl-Hg (MeHg) in dissolved and particulate phases at Browns Island and selected, representative wetland sites in the Delta.

This part of the study will provide (1) a means to evaluate the contribution of tidal wetlands to elevated dissolved and particulate Hg observed at various Delta locations; (2) a means to assess if process-level rates determined by other studies fully account for observed environmental fluxes; (3) a basis for estimating the likely Hg flux from future restored wetlands; and (4) demonstration of tools and correlates useful for assessing Hg fluxes in future studies.

With a better understanding of the role of wetland DOM in mobilizing Hg, provided by this study, CALFED managers will be better able to design wetlands restoration projects that minimize mobilization of Hg in the environment.

Hypotheses

- I. Tidal advective and dispersive fluxes from wetlands within the Delta export significant quantities of mercury and methylmercury to Delta channels.
- II. DOM from different environments will have significantly different rates and extents of mercury binding.
- III. DOM from different Delta environments will have significantly different capacities to solubilize mineral forms of mercury.

Management goals

Effective management strategies for mitigating mercury contamination of the Sacramento-San Joaquin Delta and San Francisco Estuary will require a more thorough understanding of the factors and processes that solubilize and transport Hg, and affect its reactivity. The purpose of the work proposed here is to help guide wetland restoration efforts by quantifying specific physical and chemical characteristics of Sacramento-San

Joaquin Delta wetlands not currently measured by other studies. The intent is to work with other ongoing studies to identify physical, chemical, and biological parameters associated with high *net* MeHg production and export, so that they may be minimized during implementation of restoration. We propose to focus our research on two aspects of Hg cycling not currently under study: 1) the role that DOM plays in the solubilization and facilitated transport of Hg species, and 2) the importance of physical processes in determining the net export of MeHg from tidal wetlands.

PROJECT TIMETABLE/PROGRESS

Starting and completion dates

Project funding was received in August 2004 and permission to begin work was granted in October 2004. The budget closeout was performed December 31, 2006. The USGS will continue to use internal matching funds awarded to the project for the completion of the products.

Project status

Lab Study

Work in the Aiken lab portion of the CALFED project is divided among three main areas: measuring the binding strength of Hg²⁺ with DOM, probing the distribution of Hg²⁺ between particulate and dissolved organic matter, and the role of DOM in the photochemical cycling of mercury. This area of work was initiated in the Aiken lab in February 2005. At this point in time, all field work and most laboratory measurements have been completed. Some additional measurements of binding site abundances are being performed to aid in a better understanding of the binding constants and partitioning experiments. We are currently in the data analyses and report writing stage for all aspects of the study.

Field Study

The field deployments related to this amendment were initiated in December 2004 and completed in February 2006. The deployments involved two optical instrumentation packages, one package located at each of the two primary channels draining the center Brown's Island (Figure 2). The package at the main channel is the primary package consisting of a CDOM fluorescence spectrophotometer, ISUS UV spectrophotometer, optical DO probe, AC9 fluorescence spectrophotometer, Seabird CTD, and an optical backscattering sensor. The package at the side channel has the same components except that it does not have an ISUS unit. An acoustic Doppler current profiler (ADCP) unit was deployed at both locations to quantify water flux.

Due to financial and time constraints, there were no deployments conducted at locations outside of Brown's Island. Instead, sub-samples were obtained from another project led by Batelle Marine Sciences and Moss Landing Marine Labs at two other locations in the Delta, Little Break and Mandeville Tip. Those samples were analyzed in the laboratory for optical characterization of DOM. Data analysis and interpretation will be performed on these samples after the other work is completed.

Complications

There were no additional noteworthy complications since the last annual report. We were only restricted by time and finances due to the complications noted in the previous report.

Milestones

Work is completed. Journal articles are currently being drafted.

HIGHLIGHTS AND RESULTS

Lab Study

In this element of the CALFED-Hg study our research group in Boulder, CO is studying the interactions of dissolved organic matter (DOM) on the distribution, speciation, and reactivity of Hg in the Sacramento River watershed and Delta. Since the last report, we have made progress on a number of fronts:

Mobilization of Hg from soils and sediments

Under conditions that exist in the Delta, the presence of DOM can cause significant dissolution of Hg from the sediment samples. Hg dissolution from soils and sediments depends on both the nature of the soils and the chemistry of the aqueous solution. For instance, the addition of increasing amounts of DOM (1 to 10 mg C L⁻¹), in the form of isolates, resulted in the mobilization of increasing amounts of Hg, in one case approaching 1000 ng Hg L⁻¹. Additionally DOM with greater aromaticity, such as DOM generated in the Delta's wetlands, is more effective at dissolving Hg than less aromatic DOM such as that found in the Delta's river channels. These results suggest that wetlands in the Sacramento River Delta, which are known to have high concentrations of DOM, with a large proportion of highly aromatic DOM, provide highly favorable conditions for the release of Hg from sediments.

Altering the water chemistry can affect both the dissolution of Hg and the stabilization of colloidal mercury. Experiments also showed that chloride, like DOM, increased dissolution of Hg from the solid fractions. The Bay-Delta region is known to have brackish regions as a result of tidal influence, and periods of low flow can result in the migration of this brackish regions upstream from the mouth. In contrast, polyvalent cations, such as calcium (Ca²⁺) or magnesium (Mg²⁺), inhibited Hg²⁺ dissolution. This might be of special importance for contaminated calcite soils as well as calcite containing tailings, which occur in the Coastal Range. Mercury release further increased with decreasing ionic strength because of the mobilization of colloidal mercury, a very mobile mercury fraction. Preliminary results indicate that a significant fraction of the mobilized Hg is in the form of colloidal particles; however, significant work remains to complete this portion of the investigation.

Mercury-DOM Binding

Hg-DOM stability constants were found to be quite large, $K_{\text{HgDOM}} = 10^{20}$ and greater, values consistent with measured stability constants in other regions. Further experimentation determined that the Hg is bound by very strong ligands that are kinetically stable (Hg is not released over the course of many hours in the presence of a

strong competing ligand, thiol). Further work on quantifying the abundance of binding sites in Delta DOM is still in progress.

Photochemistry of mercury-DOM

When photolysed with simulated solar irradiation of an intensity approaching that of summer sunlight in northern California, Hg(II) reduction half-lives ranged from 1.5 – 5 hours for the DOM isolates, depending on the nature of the added organic matter. Most fell into the range of 2 – 3 hours, with Pahokee peat humic acid being the lone organic matter yielding a half-life > 3 hours. The half-life for Hg(II) reduction with added mercaptoacetic acid (MAA) was 44 minutes. This small thiolate ligand is not capable of binding Hg(II) through multiple sites, and its half-life was much shorter than with other thiol model compounds capable of multidentate Hg(II) binding. Photoreduction half-lives of 3 hours were measured with glutathione, L-cysteine, and N-acetyl-L-cysteine as thiol ligands. The results of the kinetic studies suggest that the Hg(II) binding by DOM is more similar to that of the multidentate thiol ligands rather than the simple one-site interaction of MAA.

Analysis of the binding of Hg(II) by the model thiols using UV-vis spectroscopy indicates that while alone, neither Hg(II) nor the various thiol ligands have any overlap with the solar spectrum. Upon mixing the ligands and Hg(II), the absorbance spectrum shifts to longer wavelengths, with a significant tail > 300 nm. The maximum overlap between the Hg(II):thiol compounds and the solar spectrum is at ca. 310 nm. This is of note in determining the mechanisms by which the Hg(II) is reduced in these experiments. In experiments with these model compounds, there is no photosensitizer present capable of absorbing the radiation and producing reactive intermediates. Therefore, the quick photoreduction rates that were observed in these experiments must be due to a direct photolysis mechanism in which the Hg:thiol bond itself absorbs the radiant energy, leading to heterolytic bond cleavage and Hg(II) reduction.

The UV spectra also show very similar molar absorptivities for each of the Hg(II):thiol complexes studied. Because Hg(II):DOM interactions are dominated by thiol sites within the DOM, it is expected that the Hg(II):thiol sites present in DOM solutions would have similar absorptivities. Thus, because the light absorption behavior of the Hg(II):thiol bonds active in the photoreduction process is not expected to vary across samples, it is expected that the photochemical kinetics of the Hg(II):DOM complexes should be similar to that of the Hg:model thiol compounds in optically dilute solutions. This is seen when the photoreduction rates of Hg(II):DOM complexes are compared to that of Hg(II) complexes of simpler multidentate thiol ligands.

Field Study

The use of our *in situ* instrumentation package was able to provide us with a high frequency timeseries of THg and MeHg using a multivariate statistical model (Unscrambler, Camo Inc). One significant change since the last report was the change in single-variable correlations. The *in situ* data for spring and autumn correlated well with total mercury (turbidity, $r^2=0.90$) and filtered methylmercury (CDOM, $r^2=0.98$); however, the winter samples were relatively poorly correlated with CDOM ($r^2=0.70$) and

possessed a 30% lower slope, forcing us to use a multivariate approach. The data for unfiltered MeHg was not correlated with any single variable in any season requiring a multivariate approach as well. We decided to use the multivariate approach for all constituents because the model validations for the multivariate models were better than the single variable models (fig 3), even when correlations were very strong ($r^2 > 0.90$). The model validations are provided in Appendix A. The multivariate models are based primarily on output from the AC-9. Output from the THg and filtered MeHg models was required to help constrain the unfiltered MeHg model output.

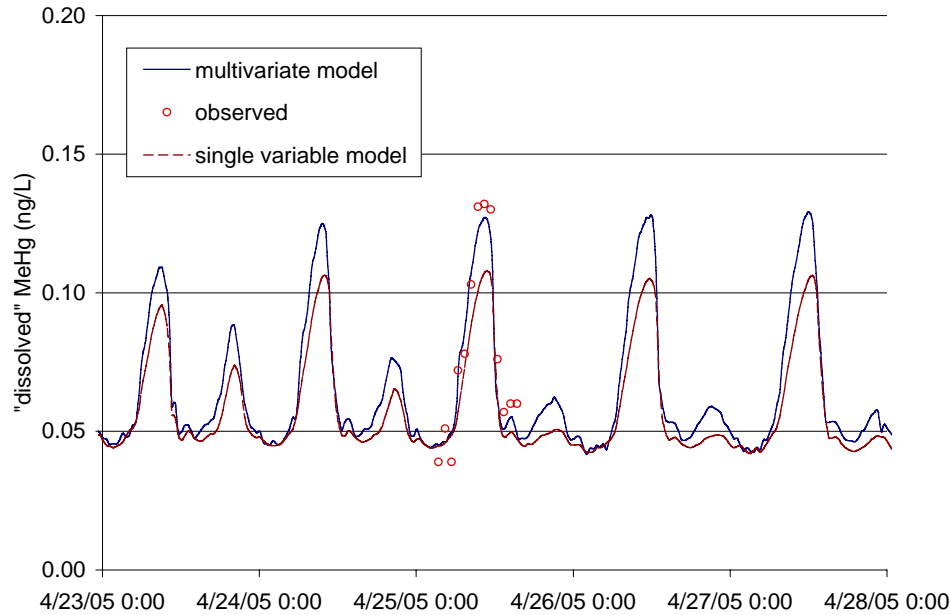


Figure 3. Comparison of multivariate and single variable models with observed data for filtered MeHg during the spring deployment in the main channel (B2).

Modeled data shows that total Hg concentrations varied greatly over several timescales. The highest concentrations in the main channel occurred during periods of strong north winds and high channel velocities (fig 4, Appendix B). These conditions reflect the strong correlation between THg and suspended particulate matter. The strong north winds suspend particles from the mudflats/shallows off the northwest side of the island outside the mouth of the main channel. Background THg concentrations in the river (high tide water) also varied over the seasons with the highest concentrations in winter and the lowest in autumn reflecting climatic forcings on the watershed's hydrology. The side channel had generally lower concentrations than the main channel most likely due to the quiescent water conditions in that channel relative to the main channel.

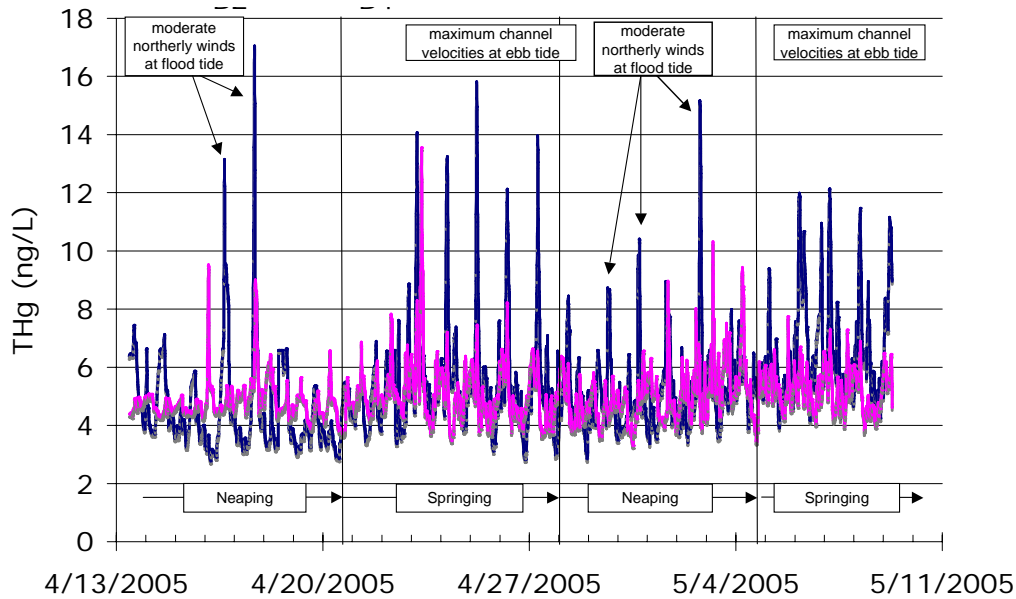


Figure 4. Variability in unfiltered THg concentrations, spring 2005. Blue line represents data from main channel (B2) and pink line represents data from side channel (B4). High concentrations occur during the highest channel velocities during springing ebb tides. High concentrations occur during flood tides when northerly winds suspend sediments in the shallows off the northwest side of the island.

The modeled concentrations of MeHg in the dissolved fraction also varied over several timescales, however, the highest concentrations of the dissolved fraction occurred at the lowest tides when the greatest proportion of island water was in the channels (fig 5, Appendix B). This effect was appeared to be amplified by barometric pressure events that can affect the island water levels thus impacting the tidal water fluctuations and thus the proportion of island water in the channels. The strong wind events that affect the particulate concentrations do not appear to greatly affect the dissolved concentrations.

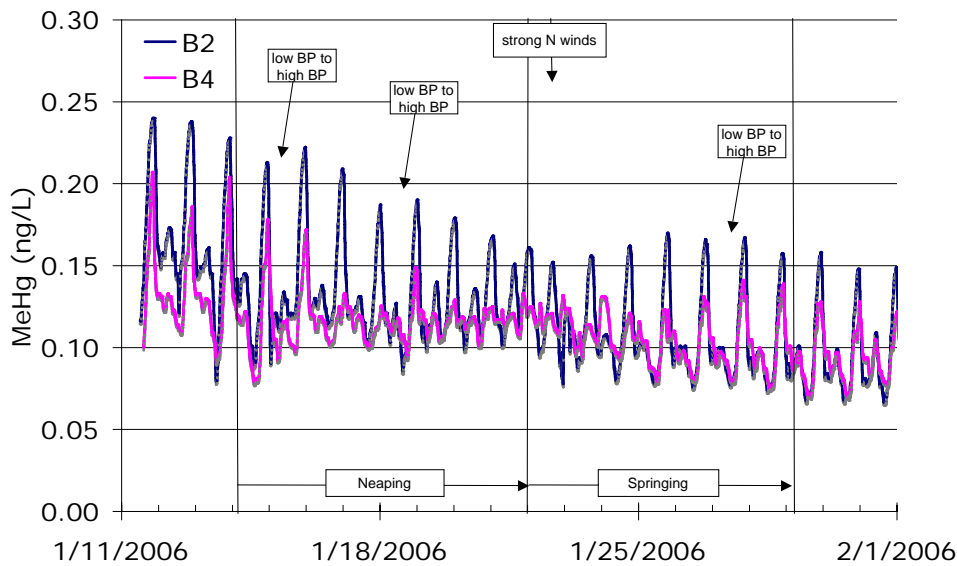


Figure 5. Variability in filtered MeHg concentrations, winter 2006. High concentrations occur during low tide during greatest contribution from island pore waters. Higher background (river) concentrations occur during high river stage, early in this record.

The concentration trends of MeHg in the unfiltered waters are the most complex. The proportion of MeHg in the dissolved fraction was highly variable with island water greater in the dissolved fraction than river water. This may be a result of simple mixing but the proportion appeared to correlate well with SUVA suggesting a chemical driver of the partitioning as well (fig 6). This agrees with results from the lab studies discussed above. Furthermore, because MeHg partitions nearly equally between particles and dissolved fractions, the driving forces for unfiltered MeHg include all of the driving forces for both the particulate and dissolved fractions (Appendix B).

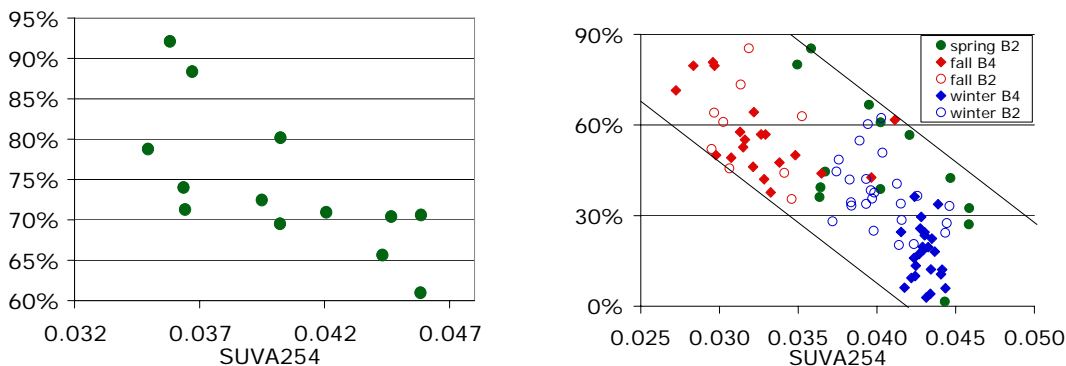


Figure 6. Relationship between $SUVA_{254}$ and the proportion of THg and MeHg in the particulate phase at Brown's Island. Island derived waters have high $SUVA_{254}$ relative to river derived water.

Constituent fluxes incorporate variability in both the constituent concentrations and water flux resulting in highly variable fluxes (fig 7). Furthermore, the net island fluxes integrate the driving forces in both channels. Using the generated high frequency timeseries, it was possible to see that fluxes of both total Hg and MeHg are variable over the same timescales as the concentrations. The timeseries of cumulative fluxes are presented in Appendix C. Supporting ancillary data defining the physical forcings are presented in Appendix D.

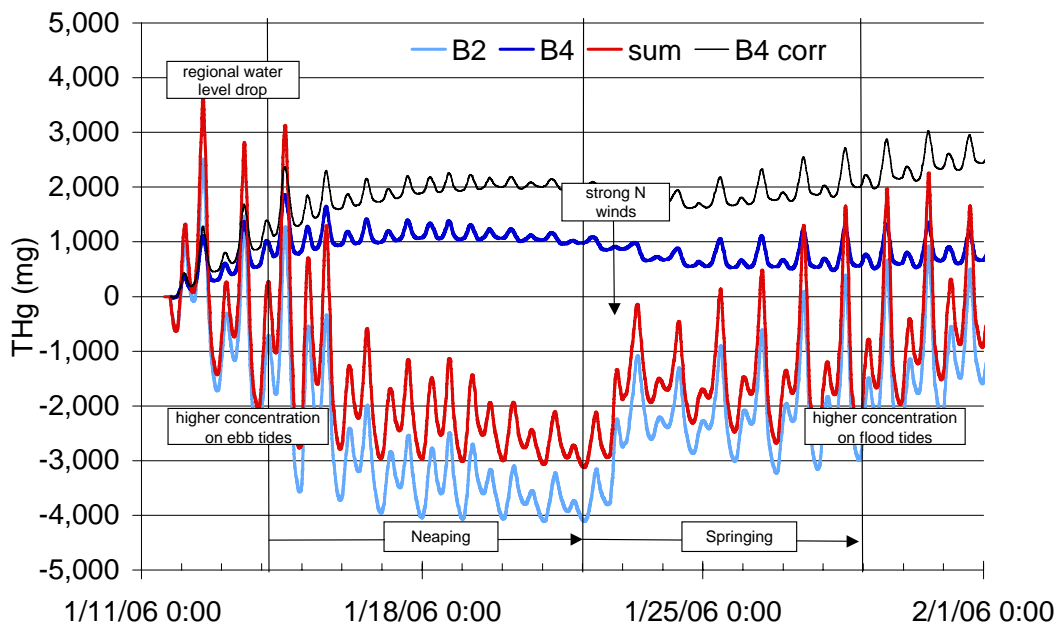


Figure 7. Variability in THg fluxes, winter 2006. The blue lines are the modeled fluxes for each channel, the black line is the corrected flux to close the water balance for the island, the red line is the net flux from the island using the corrected flux values. Forcings for variability are pointed out on the chart.

THg fluxes are largely driven by particle dynamics. In the spring, THg fluxes are dominated by hydrodynamics of the spring-neap cycle driving sediment on or off the island; whereas in autumn, winds from the north dominate particulate concentrations due to sediment resuspension which supplements the general condition when flow carries particles onto the island, leading to slow and steady net on-island flux.

The flux of the dissolved fraction of MeHg is largely driven by pressure events such as barometric and hydraulic conditions. In the spring, the fluxes of dissolved MeHg were greatest during the neap but was also the period of relatively high barometric pressure, in the autumn the quiescent flow and weather conditions led to a slow and steady off island flux of dissolved MeHg. Data for the drivers are provided in Appendix D.

POTENTIAL IMPLICATIONS OF FINDINGS TO DATE

The Bay-Delta region is a chemically and hydrologically complex system where the water chemistry variables, most notably chloride and DOM, can contribute to Hg^{2+} dissolution from the solid phase in sediments and particles. Because the primary factor controlling the rate of mercury methylation is the concentration of dissolved Hg, the ability of DOM and chloride in the Bay-Delta to facilitate the release of Hg from contaminated soils and sediments will have a direct influence on methylation in the Bay-Delta. This effect was confirmed in the field data where waters draining from Browns Island had a greater proportion of Hg in the dissolved phase than the river waters going onto the island suggesting that the interaction of these waters with the island's organic matter increased the relative proportion of Hg in the dissolved fraction.

Browns Island was found to be a source of MeHg production, but whether the island is a net source of MeHg to Bay-Delta depends on the time scale of interest because of the variability in the driving forces in tidal wetland exports. The high variability of THg and MeHg fluxes over several timescales poses a significant challenge for evaluation of the contribution of THg and MeHg to the Bay-delta system from tidal wetlands. For example, variability over harmonic cycles may lead to zero net flux over some timescales but an event driven flux can alter that balance in either direction.

To accurately quantify the flux of constituents from a tidal wetland, continuous measurements over the entire period of interest are required. The use of this type of monitoring equipment will allow future assessments of the driving factors controlling fluxes in highly dynamic systems which will aid in planning and adaptive management of dynamic systems.

PRODUCTS TO DATE

Reports/Articles

Waples, J.S., Nagy, K.L., Aiken, G.R., and Ryan, J.N., 2005, Dissolution of cinnabar (HgS) in the presence of natural organic matter, *Geochimica et Cosmochimica Acta*, v. 69, pp. 1575-1588.

Gaspar, J.D., Aiken, G.R., and Ryan, J.N., 2007, A critical review of three methods used for the measurement of mercury (Hg²⁺)-dissolved organic matter stability constants, *Applied Geochemistry*, DOI: 10.1016/j.apgeochem.2007.03.018, in press.

Suess, E., 2006, Mercury Distribution between Particulate and Dissolved States in Wetlands in California, USA, Masters Thesis, University of Freiberg, Freiberg, Germany, 155 p.

Reports in preparation (titles tentative):

Suess and others, Controls on the solubilization of mercury from contaminated soils and sediments in the Sacramento River and Sacramento-San Joaquin Delta, California, to be submitted to *Environmental Science and Technology*.

Suess and others, Dissolution of mercury from soils and sediments by dissolved organic matter and aquatic humic substances, to be submitted to *Environmental Science and Technology*.

Gerbig, Gaspar, and others, Mercury binding by dissolved organic matter in the Sacramento-San Joaquin Delta as determined by the CLE-SPE approach, to be submitted to *Environmental Science and Technology*

Latch and others, The effects of DOM on the photochemistry of mercury in Sacramento-San Joaquin Delta, to be submitted to *Environmental Science and Technology*

Downing and others, An in-situ hydro-optical sampling system to study dissolved and particulate carbon processes in a tidally forced system. To be submitted to... L+O?

Fleck and others, THg fluxes in a tidal wetland of the Sacramento-San Joaquin Delta, California. To be submitted to...L+O?

Fleck and others, MeHg fluxes in a tidal wetland of the Sacramento-San Joaquin Delta, California. To be submitted to...L+O?

Bergamaschi and others, Changes in dissolved organic carbon character over multiple timescales in a tidal wetland of the Sacramento-San Joaquin Delta, California. To be submitted to...L+O?

Pellerin, Bergamaschi, and others, MeHg fluxes in a tidal wetland of the Sacramento-San Joaquin Delta, California. To be submitted to...L+O?

Lionberger and others, Sediment fluxes in a tidal wetland of the Sacramento-San Joaquin Delta, California. To be submitted to...L+O?

Abstracts/Presentations

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- Aiken, G., 2004, Mercury and Dissolved Organic Matter in the Florida Everglades, National Conference on Ecosystem Restoration, Orlando, FL, Dec 2004.
- Aiken, G.R. and Ryan, J.N., 2005, Interactions of mercury with dissolved organic matter in the Florida, Everglades: Evidence for stabilization of colloidal mercuric sulfide, Humic Science and Technology VIII Conference, March 16-18, 2005, Boston, MA.
- Nagy, K.L., Waples, J.S., Aiken, G.R., and Ryan, J.N., 2005, Dissolution of cinnabar in the presence of dissolved organic matter, 15th Annual V.M. Goldschmidt Conference, May 20-25, 2005, Moscow, Idaho
- Aiken, G.R., Ryan, J.N., and Nagy, K.L., 2005, Interactions between dissolved organic matter and mercury in aquatic environments, 15th Annual V.M. Goldschmidt Conference, May 20-25, 2005, Moscow, Idaho
- Fleck, J.A., Bergamaschi, B.A., Downing, B.D., Lionberger, M.A., Schoellhamer, D.H., Boss, E., and Stephenson, M., 2006, A tool for assessing mercury loadings from restored tidal systems [poster]: South Bay Salt Pond Restoration Project, San Jose, California, June 6, 2006
- Bergamaschi, B.A., Fleck, J.A., Downing, B.D., Boss, E., and Aiken, G.R., 2006, Observed relationships between mercury species and properties of dissolved and particulate organic matter measured *in situ* in a tidal wetland in the San Francisco Estuary [poster]: 8th International Conference on Mercury as a Global Pollutant, Madison, Wisconsin, August 6-11 2006.
- Fleck, J.A., Bergamaschi, B.A., Downing, B.D., Lionberger, M.A., Schoellhamer, D.H., Boss, E., Stephenson, M., 2006, Measuring Hg and MeHg fluxes from dynamic systems using high resolution *in situ* monitoring, case study: the Sacramento-San Joaquin Delta tidal wetlands [poster]: 8th International Conference on Mercury as a Global Pollutant, Madison, Wisconsin, August 6-11 2006.
- Latch, D.E. and Aiken, G.R., 2006, The Influence of Dissolved Organic Matter on the Photochemical Cycling of Mercury [poster]: 8th International Conference on Mercury as a Global Pollutant, Madison, Wisconsin, August 6-11 2006.
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- Downing, B.D., Fleck, J.A., Lionberger, M.A., Ganju, N.K., Boss, E., Schoellhamer, D.H., and Bergamaschi, B.A., 2006, Seasonal changes in DOC character and flux from a tidal wetland [poster], Fourth Biennial CALFED Science Conference, Oct. 23-25, 2006, Sacramento, CA.
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- Fleck, J.A., Bergamaschi, B.A., Downing, B.D., Lionberger, M.A., Ganju, N.K., Schoellhamer, D.H., Boss, E., Heim, W., and Stephenson, M., 2006, Measuring mercury and methylmercury fluxes from dynamic systems using high resolution *in situ* monitoring – case study: the Sacramento-San Joaquin Delta [poster], Fall meeting of the American Geophysical Union, December 11-15, 2006, San Francisco, CA.
- Bergamaschi, B.A., Downing, B.D., Boss, E., Fleck, J.A., Stephenson, M., and Kalve, E., 2006, But are optical measurements good for anything practical?, [invited presentation], Fall meeting of the American Geophysical Union, December 11-15, 2006, San Francisco, CA.
- Latch, D.E. and Aiken, G.R., 2006, The Influence of Dissolved Organic Matter on the Photochemical Cycling of Mercury [poster], Fall meeting of the American Geophysical Union, December 11-15, 2006, San Francisco, CA.