May 4, 2009

Dear Ms. Maher:

Please accept the attached proposal titled with "PCBs, DDTs, and Flame Retardants in Sediments from South San Diego Bay" for environmental projects benefiting San Diego Bay.

The south San Diego Bay is the important part of the San Diego National Wildlife Refuge of California, which provides habitats for federally-listed endangered and threatened species and migratory birds, and maintains the biological diversity of native plants and animals. The biological ecosystem, however, is threatened by the contamination of PCBs, DDTs, and Flame Retardants according to the recent monitoring study conducted by the Fish and Wildlife Service. Sediments at south San Diego Bay are most likely to be contaminated with these organic pollutants that are supposed to contaminate food webs via their bioavailability. This proposed study will focus on PCBs, DDTs, and PBDEs in sediments from south San Diego Bay and will determine the pollution sources of these contaminants in south San Diego Bay.

At the completion of this proposed study, we expect to provide valuable information on the distribution, the concentration change, the significance of microbial degradation of these contaminants in sediments, and relationship of these contaminants in biological systems and in sediments. In particular, we expect to find out sources and pathways of the emerging contaminants, PBDEs, in the ecosystem of San Diego Bay for the first time. We strongly believe that these expected outcomes will help to set up efficient guidelines and management to mitigate the contamination and further protect the ecosystem of San Diego Bay.

The estimated project duration is two years and the total cost is $83,787. We respectfully request $55,860 from the Port of San Diego environmental fund. San Diego State University will provide $27,827 as cost-sharing. We are well experienced in environmental engineering and science, particularly in the fate and transport of organic contaminants. In addition, our laboratories are well equipped with various state-of-art instruments such as GC×GC/ToF-MS, GC/MS, and TOC analyzer which are essential to conduct this proposed project.

If you should need any additional information, please contact PI (Dr. Hoh) at 619-594-4671 or ehoh@mail.sdsu.edu.

Thank you for your consideration.

Sincerely,

Eunha Hoh, Ph.D. and Youxian Wu, Ph.D.

Table of Contents

PCBs, DDTs, and Flame Retardants in Sediments from South San Diego Bay

I. Introduction

The San Diego Bay National Wildlife Refuge is part of the National Wildlife Refuge System which is the nation's largest collection of lands and waters that has been primarily set aside and conserved for the conservation of native fish and wildlife and their habitats. The San Diego Bay is a habitat for a rich diversity of endangered, threatened, migratory, and native species and also provides the public with a great natural environmental resource in a highly urbanized area. Therefore, if possible, it is important to protect the ecosystem of the San Diego National Wildlife Refuge from pollutants by management of their sources.

A recent monitoring study conducted by United States Fish and Wildlife Service, Division of Environmental Contaminants in Carlsbad, CA reported that eggs of the sea birds nesting in south San Diego Bay contained not only PCBs and DDTs, but also the emerging contaminants, polybrominated diphenyl ethers (PBDEs), identified for the first time in the ecosystem of south San Diego Bay which is part of San Diego National Wildlife Refuge (Zeeman et al., 2008). The Service was interested in conducting a contaminant study in the ecosystem of San Diego Bay because of ongoing observations by Refuge biological staff of eggshell thinning, aberrant egg production, and poor fledgling survivorship in a number of seabird species that nest in the San Diego Bay. This study reveals that PCBs and DDTs have declined in the eggs of the sea birds but their concentrations were still associated with egg shell thinning. Some black skimmer eggs contained high concentrations of PCBs and DDTs. Additionally, the PBDE concentrations were comparable or even higher than those in the sea bird eggs in San Francisco Bay (She et al., 2008). This result indicates that the food webs at south San Diego Bay have been contaminated with not only PCBs and DDTs but also PBDEs. Sources of these organic contaminants at south San Diego Bay are not identified yet. Sediments are likely to be major sources of these organic contaminants to the birds and fishes. This proposed study will focus on PCBs, DDTs, and PBDEs in sediments from south San Diego Bay and will determine the pollution sources of these contaminants.

PCBs and DDTs are well known toxic chemicals and they were banned in the U.S. a few decades ago. However, PBDEs are emerging contaminants and not regulated federally yet. PBDEs are a major class of brominated flame retardants. PBDEs are also called "the next PCBs" due to their similarity to PCBs such as their tendency towards temporal persistence and food web bioaccumulation in the environment. Accordingly, PBDEs have received attention from experts in the field of environmental science and health and the general public. Several toxicological studies reveal that they are endocrine disruptors (leading to hormone imbalance in developing embryos) and cause adverse effects on neurodevelopment (Branchi and Alleva, 2002; Brachi et al., 2003; Stoker et al., 2004; Timmer-Laragy et al., 2006; Zhou et al., 2002). Human and wildlife's chronic exposure to PBDEs is a specific area of concern. PBDEs have been used in many consumer products due to their cheap but cost effective flame retardant properties since the1970s. Because of this heavy usage and their persistent and bioaccumulative properties, PBDEs have been detected in various environmental compartments such as sediments, soils, water, air, wildlife, and humans (Hites, 2004). PBDE environmental levels have increased since 1970s exponentially (Hites, 2004). Their spatial trends consistently show that PBDE concentrations in the samples acquired in the U.S. were highest worldwide regardless of sample types (human serum and breast milk, air, sea mammals, house dust etc) (Hites, 2004; Hoh and Hites, 2005b; Kanann et al., 2007; Petreas et al., 2003; Schecter et al., 2003; Sjödin et al., 2008).

More importantly, recent studies reveal that the body burdens of PBDEs in the California residents and the sea mammals from the California coast were highest as reported (Kanann et al., 2007; She et al., 2002; Zota et al.; 2008).

This proposed project will focus on tracking the pollution sources of PBDEs, PCBs, and DDTs to the birds and fishes in the south San Diego Bay via determining the concentrations and enantiomeric fractions (EFs) of these contaminants in the surficial and core sediments from several different sample sites. The possible sources of these contaminants are ocean deposition through the tidal sediment transport and non-point pollution via storm runoff into the bay. The Otay River is the main river to the south San Diego Bay. The south San Diego Bay includes the western end of the Otay River drainage basin, and the Otay River watershed encompasses approximately 160 square miles in southwest San Diego County. Our central hypothesis is that the Otay River is a potential source of PBDEs, PCBs, and DDTs to the environment of south San Diego Bay. Our ultimate goal is to reduce the pollution of PBDEs, PCBs, and DDTs in San Diego Bay. In most aquatic environments, sediments are major sinks for persistent organic pollutants such as PBDEs, PCBs, and DDTs. Therefore, understanding the fate of these organic pollutants in sediments is important in the assessment of their bioavailability, toxicity, and possible remediation. Our objectives are to:

1) Determine sources of PBDEs, PCBs, and DDTs to south San Diego Bay by obtaining temporal and spatial trend of these pollutants at south San Diego Bay;
2) Determine whether there is an input of fresh DDTs to south San Diego Bay and assess the significance of microbial degradation on the removal of PCBs and DDTs in the sediments.

To accomplish our objectives, we will collect and analyze two sediment cores from near the Otay River and the middle of south San Diego Bay and also collect 10 surface sediments in south San Diego Bay. The chemical analysis will be conducted by an untargeted screening analytical approach that can analyze multiple classes of organic compounds such as PBDEs, PCBs, and DDTs simultaneously (Hoh et al., 2009a; Hoh et al., 2009b). This analytical approach is superior to targeted analytical methods used in most monitoring laboratories for PBDEs in several aspects. Briefly, this untargeted approach adopts two-dimensional comprehensive gas chromatography with time-of-flight mass spectrometry (GC×GC/ToF-MS) which provides better separation and sensitivity compared to gas chromatography with quadrupole mass spectrometry (GC/MS) utilized in most monitoring laboratories to analyze PBDEs. More importantly, time-of-flight mass spectrometry (ToF-MS) provides full-scan mass spectra, which is essential to detect/identify analytes plus unknown compounds from complex of sample matrices. The Principle Investigator (PI, Dr. Hoh) developed the method for analysis of wide-range of organic compounds (including PBDEs, PCBs, and DDTs) and proved its analytical capacity in fish oils successfully (Hoh et al., 2009a; Hoh et al., 2009b). Likewise, we will analyze PBDEs, PCBs, and DDTs simultaneously in the sediment samples without extra cost. We will also determine the enantiomeric fractions (EF) of some chiral DDTs and Chiral PCBs and compare the EF values of the surficial sediments from different sites and the enantiomeric profiles of DDTs in the sediment cores. The ratio of enantiomers in sediments will be compared from different sites to determine the fate and transport of these contaminants.

We are well prepared to undertake this proposed study. The Principle Investigator (Hoh) is an expert of analyzing environmental organic pollutants; Dr. Hoh conducted several projects to investigate fates of organic pollutants such as PBDEs and other organochlorine pollutants in

environment; she discovered emerging contaminants (non-PBDE type flame retardants) in the environment for the first time; and she developed a novel analytical method to analyze multiple groups of organic compounds including PBDEs and DDTs. Co-PI (Wu) has been working professionally in the area of environmental engineering and science. He has an extensive research experience in soil/sediment remediation, water quality, and wastewater and drinking water treatment. Dr. Wu was recently involved in an EPA project on natural attenuation of organochlorines in sediments from Long Island Sound. Dr. Wu developed a laboratory technique to assess biodegradation significance on the removal of organochlorine pesticides in sediments via determining the enantiomeric fractions (EF) of chiral compounds. These experiences will be valuable to the research team for achieving the goals proposed in this project. In addition, our laboratories are well equipped to conduct this proposed study. For instance, we have a state-of-art instrument, two dimensional comprehensive gas chromatography with time-of-flight mass spectrometry (GC×GC/ToF-MS), operating in the PI's new laboratory at San Diego State University.

At the completion of this proposed study, we expect to develop metrics to elucidate the history of inputs of PBDEs to south San Diego Bay and possibly their sources and hot spots in south San Diego Bay. Also, we expect to determine whether there was an input of fresh (currently used) DDTs to south San Diego Bay and the rate of natural attenuation of these contaminants in sediments in the south San Diego Bay. These findings will help to set up guidelines to reduce inputs of the organic pollutants to the south San Diego Bay, which will help protect the ecosystem of the south San Diego Bay from unexpected adverse outcomes caused by further contamination. In addition, this proposed study will be critical to understand how the emerging contaminants, PBDEs, may be present in San Diego Bay, which will guide further work to protect the public and environmental health of San Diego's wetland ecosystems from these pollutants.

II. Project Narrative

i. Background and problems

San Diego Bay is a semi-enclosed embayment on the southwestern corner of California near the US-Mexican border. The bay is roughly 25 km along its axis and varies in width from 0.5 to 4 km. The area of this bay is approximately 43 km^2 at mean lower water with an average depth of about 6.5 m. The bay, bounded by five cities including San Diego with an estimated total population of 1.2 million, supports a large number of recreational, commercial, and naval facilities. The portion of the bay south of the Coronado Bridge is commonly referred to as south San Diego Bay. The south San Diego Bay constitutes part of the San Diego National Wildlife Refuge, CA. This refuge was established to protect, manage, and restore habitats for federally-listed endangered and threatened species and migratory birds, and to maintain and enhance the biological diversity of native plants and animals. A variety of seabirds species, including Caspian terns (*Sterna caspia*), elegant terns (*Sterna elegans*), black skimmers (*Rynchops niger*), gull-billed terns (*Sterna nilotica*), Forster's terns (*Sterna forsteri*), and California least terns (*Sterna antillarum browni*) have been nesting on the levees at the South Bay Salt Works. The South Bay Salt Works are within the boundaries of the south San Diego Bay Unit of the San Diego Bay National Wildlife Refuge. The California least tern is a federally endangered species and the elegant tern is a Fish and Wildlife Service Species of Management Concern as well as a California Department of Fish and Game Species of Special Concern.

The Bay, however, has been contaminated with various pollutants, including PCBs and DDTs. The most recent monitoring study conducted by the Fish and Wildlife Service shows that the failed eggs of sea birds collected at the South Bay Salt works (black skimmer, Caspian terns, elegant terns and federally endangered California least terns) still contained large amounts of PCBs and DDTs in spite of their stopped usage three decades ago (Zeeman et al., 2008). The average levels of PCBs and DDTs in the seabird eggs have declined overtime based on the previous monitoring data during 1980s and 1990s. However, in some black skimmer eggs, the DDT concentrations exceeded thresholds for significant eggshell thinning and reproductive failure, and the high PCB concentrations were associated with reduced hatchability. More importantly, new emerging contaminants, PBDEs, were detected in all of the seabird eggs, and the accumulated PBDE levels were comparable to those of the seabird eggs collected in San Francisco Bay (She et al., 2008). The study also examined the forage fish collected around the South Bay Salt Works. As expected, PBDEs, PCBs, and DDTs were detected in the forage fish, which suggests that these contaminants are present in sediments in south San Diego Bay and that the sediments are potential sources of these contaminants to food chains in the south San Diego Bay. In addition, higher concentrations of DDTs and PCBs were detected in the forage fish collected from the Otay River than those from the rest Salk Works. This result suggests that the Otay River is a source of these contaminants to the south San Diego Bay. Therefore, there is a critical need to know contamination and occurrences of these pollutants (PBDEs, PCBs, and DDTs) in the sediments at the south San Diego Bay. There is no available study at the south San Diego Bay. More importantly, there is not even a single study for PBDE contamination in any sediment of the San Diego Bay basin.

There are three technical products of PBDEs (penta-, octa-, and deca-BDEs). Due to their bioaccumulation potential and the toxic properties of these substances, the European Union regulated application of penta- and octa-BDE products in 2004, and California banned the penta- and octa-BDE products in 2008. Therefore, the sole producer of the penta- and octa-BDE products in the U.S., voluntarily phased out the production of the penta- and octa-BDE products in the end of 2004. However, deca-BDE product is still in use and its usage has increased. The deca-BDE product is mainly composed of fully brominated diphenyl ether (BDE-209) and its bioaccumulation is considered to be relatively low due to its large molecular size. However, recent monitoring studies reported accumulated BDE-209 in bio/human samples, and debromination of BDE-209 to smaller PBDEs was proved in various conditions (Ahn et al., 2006; Stapleton et al., 2008; Söderström et al., 2004). Although the penta- and octa-BDE products are being regulated in California and some other states (not regulated federally yet), the environmental PBDE concentrations in contemporary measurements still remain high and will likely remain high in the environment for a long time, similar to PCBs.

ii. Research Plan

Approach to Objectives

Objective #1: Determine sources of PBDEs, PCBs, and DDTs to south San Diego Bay.

As described earlier, we suspect that the sediments at south San Diego Bay must be contaminated with PBDEs, PCBs, and DDTs and the Otay River may be a potential input of these organic pollutants to south San Diego Bay. We will collect two sediment cores, one from near the Otay River and the other from the middle of the south San Diego Bay. In addition, we will collect surficial sediments at ten locations indicated in Figure 1. We selected the eight sites

where the forage fish were collected in the previous monitoring study conducted by the Fish and Wildlife Service and two more locations in the middle of south San Diego Bay (Figure 1) (Zeeman et al., 2008). The sediment cores will be sliced every certain depth (~2 cm) which represents accumulation of PBDEs during a certain time frame, so we can determine the

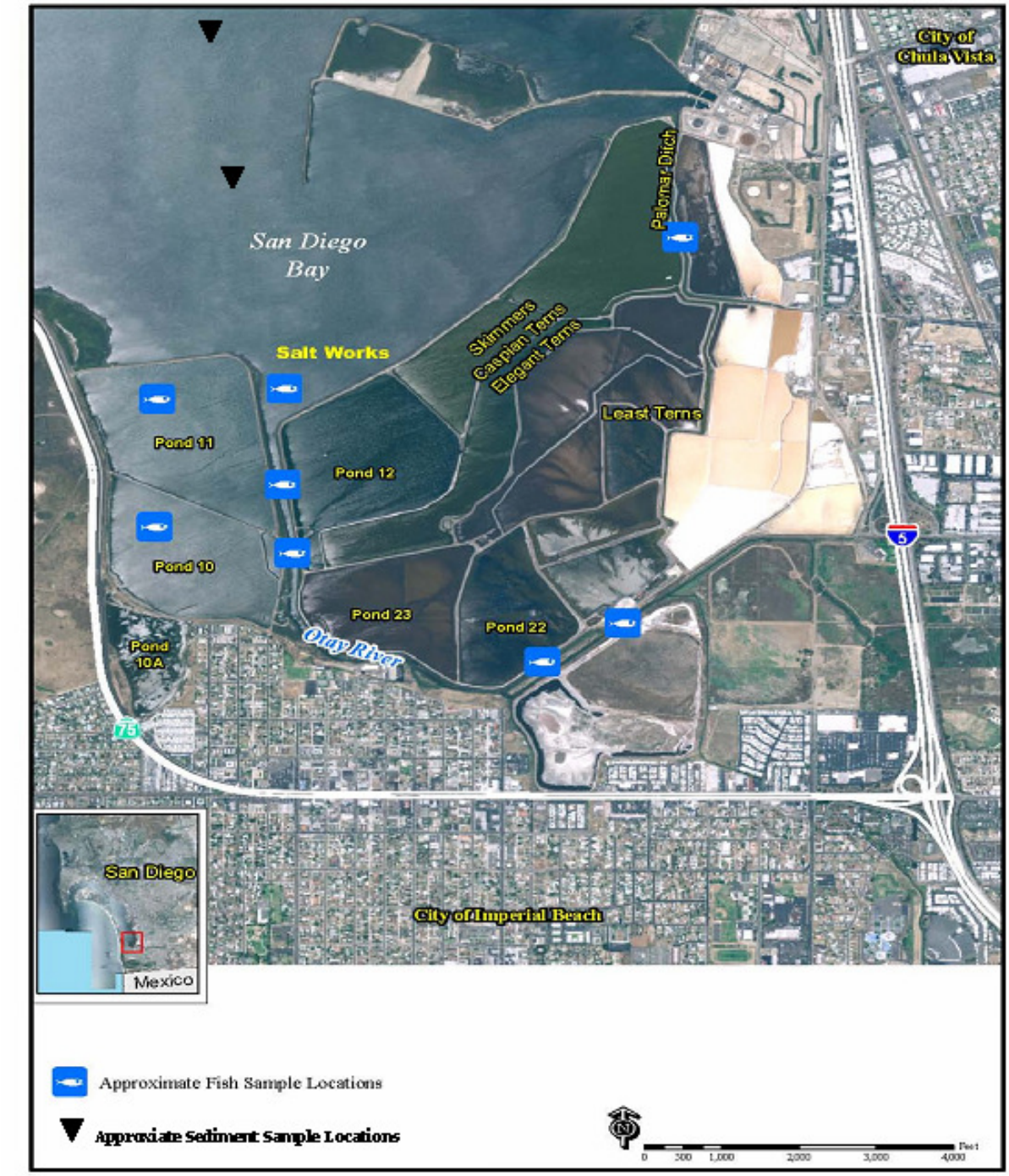

Figure 1. Proposed sediment sampling sites in San Diego Bay (modified from the report, Zeeman et al., 2008).

temporal trend of PBDEs, PCBs, and DDTs by analyzing the slices of the sediment cores. The histories of PBDEs, PCBs, and DDTs at these two sites will provide us with the contamination pathway, thus helping us to identify the pollution sources. In addition, this temporal trend is very critical to determine whether the input of these organic pollutants to south San Diego Bay has increased. Because the Otay River is a major water input to the south San Diego Bay and its watershed encompasses approximately 160 square miles in southwest San Diego County, we expect higher concentrations of the organic pollutants in the sediment core acquired from near the Otay River than the other one. Additionally, we will determine spatial trend of the PBDE concentrations at south San Diego Bay by analyzing the 10 surficial sediments samples. We also compare this spatial trend with the concentrations of the organic pollutants in the forage fish samples from the previous study (Zeeman et al., 2008), and then we will be able to determine whether the Otay River is a source of PBDEs, PCBs, and DDTs to the south San Diego Bay. To understand behaviors of emerging contaminants, PBDEs, comparing PBDEs with PCBs and DDTs in the same sample set is essential because they are different chemicals but have similar physico-chemical properties.

Objective #2: Determine whether there is an input of fresh DDTs to south San Diego Bay and assess the significance of microbial degradation on the removal of PCBs and DDTs in the sediments.

The monitoring study conducted by the Fish and Wildlife Services shows that some black skimmer eggs (collected during 2005) still contained high concentrations of DDTs. Due to close proximity of the south San Diego Bay to the US-Mexico border, heavy usage of DDTs in Mexico might contribute to total DDTs in the south San Diego Bay by atmospheric transport. After DDT was banned in the U.S. more than three decades ago, DDTs had been heavily used in Mexico for agriculture until 1990s and for malaria control until 2000 (NACEC, 2003). Another possible source of DDTs might be originated from the DDTs in the sediments in southern California Bight (coastal ocean close to Los Angeles) where the largest amounts of dumping and discharge of DDTs occurred during 1950-1970 (Zeng and Venkatesan, 1999). DDTs may have been dispersed from the hot spots to further ocean and contaminate the sediments. We expect to determine whether the source of DDTs is from the old usage or new usage by comparing the fraction of *p,p'*-DDT from the sum of *p,p'*-DDE and *p,p'*-DDT [*p,p'*-DDT/(*p,p'*-DDT+*p,p'*-DDE)] in the sediment samples with the fraction from the DDT technical product (Wong et al., 2009b). If the fraction is higher, there is likely to be input from newer usage of DDT. The fraction will be compared among the sliced sediment samples from the sediment cores and surface sediment samples.

The microbial degradation of DDTs and PCBs will be assessed via the chiral nature of several DDTs constituents (*o,p'*-DDD and *o,p'*-DDE) and PCBs (PCBs 91, 95, 136, and 149) and the enantioselective degradation of chiral compounds by microorganisms (Brandli et al., 2007; Bucheli and Brandli, 2006; Koblickova et al., 2008; Kurt-Karakus et al., 2005; Kurt-Karakus et al., 2006; Pakdeesusuk et al., 2003; Robson and Harrad, 2004; Wong et al., 2009a). Chiral compounds are generally produced as racemic mixtures that contain equal amount of (+) and (-) enantiomers. Physical processes (e.g., volatilization, leaching, and erosion) and chemical breakdown (e.g., hydrolysis and photolysis) do not change the racemic signature of chiral compounds. In contrast, microbial degradation and biological metabolism may be enantioselective and therefore change the chiral signature. Therefore, examining the enantiomeric signature of the DDTs and PCBs residues in the sediments will shed lights on the

role of microbial degradation on the removal of these contaminants (Koblickova et al., 2008; Kurt-Karakus et al., 2006; Wong et al., 2009a). Moreover, when samples with different ages (e.g., sediment cores) are available, temporal trends in enantiomer profile may yield information on the relative rates of microbial degradation over time. This will help us to take appropriate and cost-effective measures to improve the ecosystem of San Diego Bay. Recently, chiral signature was also used to identify sewage discharges into surface waters (Fono and Sedlak, 2005). Similarly, the enantiomeric analysis will help us to track the transport pathway of these chiral contaminants. The chiral signature is often described by enantiomeric fraction (EF), defined as:

$$EF = \frac{C_+}{C_+ + C_-} = \frac{C_+}{C} \quad (1)$$

where C_+ and C_- are the concentrations of the (+) and (-) forms of the enantiomers, and *C* the total concentration of the two enantiomers. When equal molar response factors are assumed, EF can be defined using peak areas of the (+) and (-) forms of the enantiomers, A_+ and A_-, rather than the actual concentrations (Harner et al., 2000):

$$EF = \frac{A_+}{A_+ + A_-} \quad (2)$$

Racemic mixtures have EF of 0.5, and a deviation of EF from 0.5 indicates enantioselective processes such as biodegradation (Bidleman et al., 1999; Harner et al., 2000; Wiberg et al., 2001). Previous studies have found that some organochlorine pesticides are enantioselectively degraded by microorganisms in soils, sewage sludge, and seawater (Bidleman et al., 2003; Bidleman et al., 1999; Fono and Sedlak, 2005; Harner et al., 2000; Wiberg et al., 2001). A significant depletion of the (+) enantiomer of *trans*-chlordane (i.e., an EF < 0.5) and a depletion of the (-) enantiomer of *DDTs and PCBs* (i.e., an EF > 0.5) have been observed in soils and water (Bidleman et al., 2003; Wong et al., 2008).

We will request some bird, egg, and fish samples from the US Fish and Wildlife Service Project collected in 2005 and determine the EFs of chiral DDTs and PCBs. The comparison between the EFs from sediment and biological samples could be indicative of the sources and the changes of these contaminants. The ratios of relatively stable forms of different enantiomers might provide the information about the pollution source of the same compound.

iii. Methodology

Sample Collection

The collection of surficial sediments and sediment cores will be conducted under the auspices of a National Wildlife Refuge System, Special Use Permit to be issued by the San Diego National Wildlife Refuge Complex. (*Brian Collins, pers comm.*). The sediment cores will be cut into 2-cm intervals and stored in pre-cleaned jars right after the cores are taken to avoid potential distortion of the sediment during delivery. All the samples (both sliced sediment core samples and surface sediment samples) will be immediately frozen in dry-ice coolers and stored in -20°C walk-in freezers in our laboratories.

Chemical Analysis

Each sediment sample will be subsampled to determine water content and concentrations of PBDEs, PCBs, and DDTs. For the chemical analysis, approximately 15 g of sediment will be mixed with 100g of precleaned anhydrous sodium sulfate (to remove water) and with 20g of precleaned granular copper (to remove elemental sulfur). After mixing, the samples will be

Soxhlet extracted for 24 h with 1:1 acetone-hexane and then acidified with sulfuric acid to digest fat and organic polymers from the extracts. Further clean up by silica and alumina will be followed. After evaporation, the final extract will be analyzed on Leco Pegasus 4D comprehensive gas chromatography with time-of-flight mass spectrometry (GC×GC/ToF-MS) operated in electron ionization. For GC×GC, DB-5-MS (20m length, 0.25 mm i.d., 0.25 μm film thickness; Agilent) will be used as a first dimensional column and DB-17MS (2 m, 0.18 mm, 0.18 μm thickness; Agilent) as a second dimensional column and the oven temperature program and the modulation condition will be optimized to maximize separation.

The final extract will be also analyzed for enantiomeric analysis of the organochlorines on a Fisons 8000 GC - Fisons Trio 1000 MS mass spectrometer operated in electron ionization. Separations will be carried out using a β-DEX 120 column (30 m, 0.25 mm i.d., 0.25 μm film thickness; Supelco). Details of the instrument conditions are described elsewhere (Aigner et al., 1998; Wu et al., 2006). Elution orders will be performed with standards of a single-enantiomer DDTs and PCBs.

Organic Carbon Content Analysis

Total organic carbon in sediments will be determined instrumentally with a TOC analyzer Shimadzu 5000A with a Solid Sample Model (SSM-5000A). Total organic carbon will be measured directly in samples from which carbonate carbon is removed by acid volatilization.

Other Analysis

Dry weight of a sediment sample will be determined by heating 10.0 g of sediment sample (discard pebbles, shells, biota and homogenize with spatula) for 24 h at 120 ˚C and cooling it in a desiccator for 30 minutes. Particle size determination will be performed using standard wet sieving techniques derived from EPA methods (Plumb, 1981). A laser diffraction particle size analyzer (Beckman Coulter) is available to determine the particle size distribution.

iv. Benefits to San Diego Bay

This proposed study will provide sources, pathways, and historical inputs of organic pollutants, PBDEs, PCBs, and DDTs in south San Diego Bay. This information is critically needed to assess current and future status of these organic pollutants in the ecosystem of south San Diego Bay further to the entire San Diego Bay. The organic pollutants are persistent and bioaccumulative, so these organic pollutants in sediments are bioavailable to organisms in the bottom level of aquatic/marine food webs. This will be continued in future unless there is no management action. These organic pollutants transport and biomagnify through food webs and threaten the ecosystem due to their high toxicity. The south San Diego Bay is a habitat for many species including endangered species. Unfortunately, the most recent monitoring study at south San Diego Bay shows the continuous burden of PCBs and DDTs and a new burden of PBDEs in the sea birds. Without setting up guidelines or management to reduce the contamination of these organic pollutants, the wildlife at south San Diego Bay will be vulnerable to exposure of the organic pollutants. We believe that our proposed study will provide critical information to set up efficient guidelines and management to mitigate the contamination and further protect the ecosystem of San Diego Bay. In addition, the sediment levels of PBDEs at south San Diego Bay will be critically important to assess the PBDE contamination in San Diego, considering the scarcity of PBDE information in San Diego.

v. Partnerships with Wildlife Refuge

The collection of surficial sediments and sediment cores will be conducted under the auspices of a National Wildlife Refuge System, Special Use Permit to be issued by the San Diego National Wildlife Refuge Complex. (*Brian Collins, pers comm.*) This proposed study follows up the monitoring study of organic pollutants in the eggs of sea birds and the forage fishes (conducted by the Fish and Wildlife Office) at south San Diego Bay to investigate sources and pathways of the organic pollutants. We have exchanged and shared our concerns and research interests in the organic pollutants in San Diego Bay with the scientists (Dr. Catherine Zeeman and her colleagues) at Carlsbad Fish and Wildlife Office conducting the monitoring study. We will share the outcomes from this proposed study with them and have meetings with them for discussion regularly. We believe that this cooperation will make our study more useful, help both parties to plan next projects, and further management officials to set up effective guidelines to protect San Diego Bay from the organic pollutants.

vi. Research Schedule

The total length of this proposed study is two years. The below Table indicates the project timeline.

PROJECT TIMELINE

Year 01

10/09	11	12	01/10	02	03	04	05	06	07	08	09
	sample collection……..										
investigation protocol development…….											
						lab study/analyses……. ……					

Year 02

10	11	12	01/11	02	03	04	05	06	07	08	09
lab study/analyses.....…….……..…….											
data analysis……											
	conference presentations/ manuscript/final report to the Port…..…….										

III. Qualifying Experience

Eunha Hoh (PI)

Dr. Eunha Hoh joined SDSU in the spring of 2009 as a tenure-track Assistant Professor in Environmental Health Division in the Graduate School of Public Health. Prior to joining SDSU, Dr. Hoh participated in several research projects closely relevant to this proposed study. During her Ph.D., Dr. Hoh had participated in an international monitoring project of persistent organic pollutants, "Integrated Atmospheric Deposition Network (IADN)", to determine atmospheric inputs and sources of the persistent organic pollutants to the Great Lakes basin. Dr. Hoh successfully determined atmospheric transport, sources, and pathways of the persistent organic pollutants (organochlorine pesticides and flame retardants) to the Great Lakes basin. Dr. Hoh also achieved chemical detective work; she discovered novel flame retardants and a chlorinated flame retardant (Dechloran Plus) for the first time in the environment. Policy-makers (as well as the media) took notice of these findings, as toxic chemical managers do not want toxic chemicals replaced with others that may still pose risks. During her postdoctoral years at U.S. Department of Agriculture, she developed novel analytical methods which included fast and efficient analyses of multiple classes of targeted chemical residues, and an untargeted analytical

approach for organic compounds of interest in food and environmental samples. Dr. Hoh's previous projects are very similar to this proposed study in terms of investigating fate of the same organic pollutants in environment, and the analytical method that she developed will be used as a main method for this proposed study. Dr. Hoh is an expert in the field of persistent organic pollutants and her works were published in top journals such as *Environmental Science and Technology, Journal of Chromatography A, and Journal of Agriculture and Food Chemistry.* She will lead and manage the proposed project. The titles and references of the projects are described below.

a. Integrated Atmospheric Deposition Network (Dr. Hoh's participation: 2001-2005)
Integrated Atmospheric Deposition Network (IADN) monitors persistent organic pollutants in the air in the Great Lakes to determine atmospheric inputs and sources of these chemicals to the Great Lake basin. This project has been operated by the United States Environmental Protection Agency (US EPA) and Environment Canada since 1990. On the U.S. side, sampling and analysis of samples is provided by the laboratory of Distinguished Professor Ronald A. Hites at Indiana University, an internationally renowned scientist in the field of environmental chemistry. P.I. (Dr. Hoh) of this proposed study was his Ph.D. student and participated in this project, sponsored by USEPA-GLNPO (Grant GL995656-05). Dr. Hoh's research focused on atmospheric transport of persistent organic pollutants in the Great Lakes basin, as well as the United States as a whole, and investigation of new, "emerging" pollutants that the scientific community has just begun to study.

1. Reference: Distinguished Professor Ronald A. Hites (PI of the project)
School of Public and Environmental Affairs (Room 410), Indiana University
1315 East Tenth Street, Bloomington, IN 47405
Phone) 812-855-0193
E-mail) hitesr@indiana.edu
2. Reference: Melissa Hulting: (former coordinator of the project)
U.S. EPA Region 5, Air and Radiation Division, Chicago, IL 60604
Phone) 312.886.2265
E-mail) hulting.melissa@epa.gov

b. Novel Technologies and Techniques for the Detection of Residues, Toxins, and Other Chemicals in Foods (Dr. Hoh's participation: 2006-2008)
This is a CRIS project being conducted for 2006-2010 in U.S. Department of Agriculture, Agricultural Research Service. Dr. Hoh was involved in this project as a postdoctoral fellow during 2006-2008. Dr. Hoh successfully achieved two important subprojects. Dr. Hoh developed a fast and cost-effective analytical method screening dioxins in foods. As a second project, Dr. Hoh developed an untargeted analytical approach that can analyze multiple classes of organic chemicals and untargeted organic compounds successfully. Using this analytical method, Dr. Hoh analyzed multiple classes of persistent organic pollutants (including PBDEs, PCBs, and DDTs) and other halogenated organic compounds simultaneously.
Reference: Dr. Steven J. Lehotay (direct supervisor): Lead Scientist
Eastern Regional Research Center (ERRC)
U.S. Department of Agriculture Agricultural Research Service (USDA ARS)

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E-mail) steven.lehotay@ars.usda.gov

ii. Youxian Wu (Co-PI)

Youxian Wu, PhD in Environmental Engineering (2002, University of Sheffield), was a soil scientist in China for many years and then worked as an environmental engineering scientist in Europe and the US. Dr. Wu is the director of Environmental Engineering Laboratories in the Department of Civil, Construction, and Environmental Engineering at SDSU. His research interests include soil/groundwater remediation, water quality, wastewater and drinking water treatment. Dr. Wu has published his research findings in premier journals, such as Environmental Science & Technology, Applied and Environmental Microbiology, Chemosphere, Journal of Geomicrobiology, and Journal of Environmental Engineering Science. He will assist Dr. Hoh to manage the project, supervise students working in laboratories, collect samples, and design laboratory research. Dr. Wu was recently involved in many projects in the area of environmental engineering and science. The examples of recent similar projects are below:

a. Evaluation of construction best management practices for sediment and water quality (2008 – 2009)
This is an ongoing research project sponsored by California State Water Control Board. Dr. Wu started to be involved in the project in 2008. He has helped the PI (Dr. Edward Beighley) and co-PI (Dr. Fatih Büyüksönmez) to collect samples, design laboratory studies, and supervise students.
Reference: Dr. Edward Beigley, PI of the project, Assistant Professor
Department of Civil, Construction, and Environmental Engineering
San Diego State University
Phone) 619-594-2284
Email) beighley@mail.sdsu.edu

b. Perchlorate removal from groundwater(2006 – 2007)
This was a research project sponsored by the U.S. Department of Defense. Dr. was the important member to conduct the project. He helped the PI, Dr. Mirat Gurol to manage the project including budget, supervise several students working in laboratory, design the experiments, conduct sample treatment and analysis, and prepare report and paper manuscripts.
Reference: Dr. Mirat Gurol, PI of the project, Professor
Department of Civil, Construction, and Environmental Engineering
San Diego State University
Phone) 619-594-0391
E-mail) mgurol@mail.sdsu.edu

c. Bioremediation for chromium removal from contaminated soils and natural attenuation of organochlorine pesticides in sediments of Long Island Sound (2002 – 2005)
Dr. Wu was involved in the projects as a postdoctoral fellow from 2002 to 2005. The projects were sponsored by NSF and the USEPA, respectively. Dr. Wu's task was to help the PI, Dr. Baolin Deng to manage the projects, supervise students, develop the relevant

laboratory methods and techniques, and prepare reports and paper manuscripts. He developed a laboratory technique to assess biodegradation significance on the removal of organochlorine pesticides in sediments via determining the enantiomeric fractions (EF) of chiral compounds.
Reference: Dr. Baolin Deng, PI of the projects, Professor
Department of Civil and Environmental Engineering
The University of Missouri
Phone) 573-882-0075
Email) DengB@missouri.edu

IV. Objectives of Grant Proposal

Objective #1: Determine sources of PBDEs, PCBs, and DDTs to south San Diego Bay.
1. Determine temporal trend of concentrations of PBDEs, PCBs, and DDTs in the sediment cores taken at south San Diego Bay (Depth vs. Concentrations of PBDEs, PCBs, and DDTs).
2. Compare concentrations of PBDEs, PCBs, and DDTs between a sediment core taken near the Otay River and a sediment core from the middle of south San Diego Bay.
3. Determine spatial trend of concentrations of PBDEs, PCBs, and DDTs among surficial sediments taken at south San Diego Bay.
4. Test correlation between the concentrations of PBDEs, PCBs, and DDTs in the surficial sediments and fish levels of these organic pollutants from the previous monitoring study (conducted by the Fish and Wildlife Service).
5. Analyze congener patterns of PBDEs in the sediment samples.
6. Compare the PBDE concentrations in the sediment samples with those of outside San Diego Bay (literature data).

Objective #2: Determine whether there is an input of fresh DDTs to south San Diego Bay and assess the significance of microbial degradation on the removal of PCBs and DDTs in the sediments.
1. Determine temporal trend of the fraction of *p,p'*-DDT from the sum of *p,p'*-DDT and *p,p'*-DDE in the sediment cores (Depth vs. the fraction of *p,p'*-DDT).
2. Compare the fraction of *p,p'*-DDT between the two sediment cores (one near the Otay River and the other from the middle of south San Diego Bay).
3. Determine spatial trend of the fraction of *p,p'*-DDT from the surficial sediment samples.
4. Determine the enantiomeric fractions (Efs) of chiral PCBs and DDTs in the sediments to assess the microbial degradation of PCBs and DDTs in sediments.

At the completion of this proposed study, we will obtain the results of the above objectives and expect to determine sources and pathways of these organic pollutants in south San Diego Bay. Especially, there is no such data for PBDE contamination in San Diego area, so the outcomes of this study will be valuable to assess the contamination of this emerging contaminant, PBDEs, in the environment of San Diego.

V. Cost Proposal

			Year 1		Year 2		Total	
PERSONNEL			**Request**	***Match***	**Request**	***Match***	**Request**	***Match***
		Base						
Salaries								
E. Hoh, PI								
5%	AY	68856		*3443*		*3546*		*6989*
Youxian Wu, Co-PI								
5%	AY	67188		*3359*		*3460*		*6819*
TBN, Graduate Asst.								
50%	AY	24960	12480		12854		25334	
	Total Salaries		**12480**	***6802***	**12854**	***7006***	**25334**	***13808***
Fringe								
Hoh @	34.80%			*1198*		*1234*		*2432*
Wu @	34.80%			*1169*		*1204*		*2373*
Grad Asst @	12.00%		1498		1542		3040	
	Total Fringe		**1498**	***2367***	**1542**	***2438***	**3040**	***4805***
	TOTAL PERSONNEL		**13978**	***9169***	**14396**	***9444***	**28374**	***18613***
SUPPLIES								
Project Supplies			11000		8000		19000	
	TOTAL SUPPLIES		**11000**		**8000**		**19000**	
OTHER DIRECT COSTS								
Sampling costs			1200				1200	
	TOTAL OTHER COSTS		**1200**				**1200**	
Total Direct Costs			**26178**	***9169***	**22396**	***9444***	**48574**	***18613***
F&A @ 15% (request)			3927		3359		7286	
F&A @ 49.5% (match)				*4539*		*4675*		*9214*
TOTAL PROJECT COSTS			**30105**	***13708***	**25755**	***14119***	**55860**	***27827***
			% match:	*45.53%*	% match:	*54.82%*	% match:	*49.82%*

Budget Justification

Personnel

Funds are requested to cover the stipend (year 1 and year 2, $16/hour) of a graduate student, who will conduct the proposed field and laboratory work. An appropriate student is necessary to ensure the success of the proposed work due to a lot of laboratory work.

Fringe Benefits

The fringe benefits for students is 12%.

Supplies

Year 1 ($11,000)
Van Venn bottom grab, gravity corer, and sample bottles: $2,500
Columns for GC separation: $1,500
Reagents: $3,000
Other miscellaneous laboratory supplies: $2,000

Gas for instruments: $2,000
Year 2 ($8,000)
Columns for GC separation: $1,500
Reagents: $3,000
Other miscellaneous laboratory supplies: $1,500
Gas for instruments: $2,000

Sampling

Year 1 ($1,200)
Vessel rental: $150/hr × 8 hr/day = $1,200

VI. Personnel

	Position	Contact information
Eunha Hoh (PI)*	Assistant Professor Graduate School of Public Health, San Diego State University	Phone: 619-594-4671 Fax: 619-594-6112 E-mail: ehoh@mail.sdsu.edu
Youxian Wu (Co-PI)	Director, Environmental Engineering Laboratories The College of Engineering San Diego State University	Phone: 619-594-0944 Fax: 619-594-8078 E-mail: ywu@mail.sdsu.edu
TBN	Research assistant	

*Primary contact

BIOGRAPHICAL SKETCH

NAME Hoh, Eunha	POSITION TITLE Assistant Professor, Environmental Health Graduate School of Public Health San Diego State University
PI	

INSTITUTION AND LOCATION	DEGREE *(if applicable)*	YEAR(s)	FIELD OF STUDY
Korea Advanced Institute of Science and Technology (Daejeon, Republic of Korea)	B.S.	1995	Chemistry
Korea Advanced Institute of Science and Technology (Daejeon, Republic of Korea)	M.S.	1997	Chemistry
Indiana University (Bloomington, IN)	M.S.	2003	Environmental Science
Indiana University (Bloomington, IN)	Ph.D.	2006	Environmental Science

A. POSITIONS

2009- Assistant Professor, Environmental Health Division, Graduate School of Public Health, San Diego State University, San Diego, CA

2006-2008 Postdoctoral Fellow, U.S. Department of Agriculture Agricultural Research Service, Eastern Regional Research Center, Wyndmoor, PA

2001-2006 Research Assistant, Indiana University, Bloomington, IN (Research Advisor: Ronald A. Hites)

2001 Teaching Assistant, Indiana University, Bloomington, IN (Professor: Ming Tai-Seale)
1997-1999 Researcher, Samsung SDI, Suwon, Kyongki-Do, Republic of Korea

B. PUBLICATIONS

1. **Hoh, E**.; Lehotay, S.J.; Mastovska, K.; Ngo, H.L.; Vetter, W.; Pangallo, J.C.; Reddy, C.M. "Capabilities of Direct Sample Introduction-Comprehensive Two-Dimensional Gas Chromatography-Time-of-Flight Mass Spectrometry to Analyze Organic Chemicals of Interest in Fish Oils" *Environmental Science and Technology*, **2009,** *43*, 3240-3247.
2. **Hoh, E**.; Lehotay, S.J.; Pangallo, J.C.; Mastovska, K.; Ngo, H.L.; Reddy, C.M.; Vetter, W. "Simultaneous Quantitation of Multiple Classes of Organohalogen Compounds in Fish Oils with Direct Sample Introduction Comprehensive Two-Dimensional Gas Chromatography and Time-of-Flight Mass Spectrometry", *Journal of Agricultural and Food Chemistry*, **2009,** *57,* 2653-2660.
3. **Hoh, E**.; Lehotay, S.J.; Mastovska, K.; Huwe, J.K. "Evaluation of automated direct sample introduction with comprehensive two-dimensional gas chromatography/time-of-flight mass spectrometry for the screening analysis of dioxins in fish oil" *Journal of Chromatography A*, **2008**, *1201*, 69-77.
4. **Hoh, E**.; Mastovska, K. "Large Volume Injection Techniques in Capillary Gas Chromatography" *Journal of Chromatography A*, **2008**, *1186*, 2-15.
5. **Hoh, E**.; Mastovska, K.; Lehotay, S.J. "Optimization of Separation and Detection Conditions for GC×GC-TOF analysis of Polychlorinated dibenzo-*p*-dioxins and dibenzofurans" *Journal of Chromatography A*, **2007**, *1145*, 210-221.
6. **Hoh, E**.; Zhu, L.Y.; Hites, R.A. "Dechlorane Plus, a Chlorinated Flame Retardant, in the Great Lakes" *Environmental Science and Technology,* **2006**, *40*, 1184-1189.
7. **Hoh, E**.; Hites, R.A. "Brominated Flame Retardants in the Atmosphere of the Eastern United States" *Environmental Science and Technology*, **2005**, *39*, 7794-7802.
8. **Hoh, E**.; Zhu, L.Y.; Hites, R.A. "Novel Flame Retardants, 1,2-*Bis*(2,4,6-tribromophenoxy)ethane and 2,3,4,5,6-Pentabromoethylbenzene, in United States' Environmental Samples" *Environmental Science and Technology*, **2005**, *39*, 2472-2477.
9. **Hoh, E**.; Hites, R.A. "Sources of Toxaphene and Other Organochlorine Pesticides in North America As Determined By Air Measurements and Potential Source Contribution Function Analysis" *Environmental Science and Technology*, **2004**, *38*, 4187-4194.

BIOGRAPHICAL SKETCH

NAME Youxian Wu			NAME Youxian Wu
Co-PI			Co-PI
INSTITUTION AND LOCATION	DEGREE	YERA	INSTITUTION AND LOCATION
Huazhong Agricultural University, P.R. China	B.S.	1986	Huazhong Agricultural University, P.R. China
Chinese Academy of Sciences, P.R. China	M.S.	1989	Chinese Academy of Sciences, P.R. China
University of Sheffield, UK	Ph.D.	2002	University of Sheffield, UK

A. POSITIONS

04/2006 – Present: Director of Environmental Engineering Laboratories, Department of Civil, Construction & Environmental Engineering, San Diego State University

01/2005 – 01/2006: Visiting Assistant Professor, Department of Earth and Atmospheric Science, The City College, City University of New York.

04/2002 – 12/2004*:* Post-Doctoral Fellow, Department of Civil and Environmental Engineering, University of Missouri-Columbia. University of New York.
01/1998 – 12/2001*:* Research Assistant, Department of Civil and Structural Engineering, University of Sheffield, UK

B. SELECTED PUBLICATIONS

Clevenger, Thomas, **Youxian Wu**, Eric DeGruson, Blaise Brazos and S. Banerji. 2007. Comparison of the inactivation of Bacillus subtilis spores and MS2 bacteriophage by MIOX, ClorTec and hypochlorite. *Journal of Applied Microbiology*, 103 (6): 2285–2290.
Wu, Youxian, David N. Lerner, Steven A. Banwart, Steven F. Thornton, and Roger W. Pickup. 2006. Persistence of Fermentative Process to Phenolic Toxicity in Groundwater. *Journal of Environmental Quality*. 35:2021–2025.
Wu, Youxian and Baolin Deng. 2006. Assessing chromium oxidation mediated by *Pseudomonas putida* in the presence of FeS. *Environmental Engineering Science*. 23(3): 552-560.
Wu, Youxian, Thomas Clevenger, and Baolin Deng. 2005. Effects of Goethite particles on UV inactivation effectiveness. *Appl. Environ. Microbiol.* 71(7):4140-4144.
Wu, Youxian, Baolin Deng, Huifang Xu, and Hiromi Kornishi. 2005. Chromium(III) oxidation coupled with microbially mediated Mn(II) oxidation. *Geomicrobiology Journal*. 22(3-4): 161-170.
Watson, I., S. Oswald, K. Mayer, **Y. Wu**, and S. Banwart. 2003. Modeling kinetic processes controlling hydrogen and acetate concentrations in an aquifer-derived microcosm. *Environmental Science and Technology* 37(17): 3910-3919.
Wu, Youxian, S. A. Banwart, S. F. Thornton and D.N. Lerner. 2002. Microbial production of fatty acids in phenol-contaminated groundwater and its significance in subsurface bioremediation. In: *Groundwater Quality: Natural and Enhanced Restoration of Groundwater Pollution*. IAHS Publ. no. 275, 2002. pp. 219–224
Anderson R; **Wu, Youxian**. 2001. Phosphorus quantity-intensity relationships and agronomic measures of P in surface layers of soil from a long-term slurry experiment. *CHEMOSPHERE* 42 (2): 161-170.

VII. Subconsultants

We do not have subconsultants for this proposed project.

VIII. Non-Profit Status

San Diego State University is a non-profit academic institution.

IX. Applicant Disclosure

No violation.

X. Agreement

We have reviewed the proposed contract and find it acceptable.

XI. Conflict of Interest

No conflict of interest

XII. Additional Information

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Bidleman TF et al. (2003). Air-soil and air-water exchange of chiral pesticides. In: J.R. Coats and H. Yamamoto (Editors), Environmental Fate and Effects of Pesticides. Acs Symposium Series. Amer Chemical Soc, Washington, pp. 196-225.

Branchi I, Alleva E, Costa LG. (2002) Effects of perinatal exposure to a polybrominated diphenyl ether (PBDE99) on Mouse neurobehavioral development. NeuroToxicology 23:375-384.

Branchi I, Capone F, Alleva E, Costa LG. (2003) Polybrominated diphenyl ethers: neurobehavioral effects following developmental exposure. NeuroToxicology 24:449-462.

Brandli RC et al. (2007). Fate of PCBs, PAHs and their source characteristic ratios during composting and digestion of source-separated organic waste in full-scale plants. Environ Pollut. 148: 520-528.

Bucheli TD, Brandli RC. (2006). Two-dimensional gas chromatography coupled to triple quadrupole mass spectrometry for the unambiguous determination of atropisomeric polychlorinated biphenyls in environmental samples. J Chromatogr A, 1110: 156-164.

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