

Priority organic pollutant analysis of sediment cores and water samples from Los Cerritos Wetlands:
PAHs, PCBs, and Pesticides

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Project Summary

Mud core and water samples from Los Cerritos Wetlands The sum of PAH contamination ranges from (58 ± 12) ppb to (37,580 ± 7,516) ppb among eight different sample sites and/or depths. The fractional composition of all but one site was characteristic of combustion vehicle traffic sources, and gas-fired power plants. This may reflect contamination of the site, due to its different fractional composition, by the degraded wetland. PAHs that infiltrate the area will only occur during the outgoing tide, through the culvert connecting the degraded wetland to the Steam Shovel Slough, and it is therefore logical to assume that contaminants from oil drilling operations are distributed away from the Steam Shovel Slough, toward Los Alamitos Bay. Unfortunately, sample sites were not chosen from this area, and such comparisons may not be made at this time. The analysis of PCBs and pesticides did not result in their detection within the mud core or water samples within this study. The detection limit and sensitivity were determined using the selected ion signal for quantitation. The detection limit of the method was calculated (from target ion signal) to be approximately 10ppb for MWs of approximately 220g/mol and higher, and approximately 5ppb for MWs of less than 220g/mol. The sensitivity of the method was found to be approximately 200 counts ppb⁻¹ and 500 counts ppb⁻¹, respectively. The precision of the analytical results was determined to be approximately 20%, however literature suggests that environmental analyses of this type generally have precisions of 50-120% of their actual values, from sample collection to analysis (Li, et al. 2003).

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Background

The Los Cerritos Wetlands is a vital resource for southern California and the City of Long Beach. The area is classified as a tidal salt marsh, and is located on the border of Seal Beach and Long Beach, adjacent to Los Alamitos Bay (Appendix A and B). Tidal salt marshes are among the most biologically productive ecosystems in the world, and have significant biodiversity and socioeconomic value (Budzinski, et al. 1997). Los Cerritos Wetlands currently functions as an estuary and waterfowl and migratory bird habitat. The wetlands were naturally within the San Gabriel River watershed, but the river is now diverted from the wetlands, and consequently releases water directly into the ocean, southeast of Los Alamitos Bay. Los Cerritos Channel is the northernmost border of the wetlands area, and serves as a water inlet for cooling towers at the adjacent AES power station, and for the Steam Shovel Slough. The water which flows into the cooling towers is then released into the San Gabriel River. This circulation of water provides for increased influx of fresh marine waters into the Steam Shovel Slough, the only functional tidal salt marsh remaining within the wetland area. The Steam Shovel Slough is also the only area of the wetlands that is currently protected under the Clean Water Act and Public Trust Doctrine. Consequently, this is the only area that has public access. The Bryant, Bixby, and Hellman properties are all privately held and currently used for oil production. These areas are not publicly accessible and are classified as degraded wetland. The focus of the proposed study is the functional tidal salt marsh, the Steam Shovel Slough.

A significant amount of freshwater from the Los Cerritos Channel will mix with ocean waters as it enters Los Alamitos Bay. These brackish waters may also enter the Steam Shovel Slough and the ratio of fresh to ocean waters will fluctuate depending on precipitation and the hours of operation of the AES power plant cooling towers. When the cooling towers are operational, the only influx of water into the Steam Shovel Slough is tidal waters directly from Los Alamitos Bay. However, when the cooling towers are not operating, the influx of water into the Steam Shovel Slough is a brackish fresh/ocean water mixture from Los Cerritos Channel and Los Alamitos Bay. Storm water and urban run-off are the major sources of water for the Los Cerritos Channel. The Los Cerritos Channel has an inland terminus at the Long Beach Airport.

Since there is significant manufacturing and industrial activity at the Long Beach Airport and surrounding areas, it may be a significant source of Polycyclic Aromatic Hydrocarbons (PAHs) in the Steam Shovel Slough of Los Cerritos Wetlands. The incomplete combustion of fossil fuels at the AES and Haynes power plants may also contribute to airborne PAHs which may settle on mudflat and riparian environment sediments (Zhen, et al. 2002). The degraded riparian area adjacent to the AES power plant was once an unregulated burn dump site, and is contaminated with unknown combustion by-products from solid wastes (Moffatt and Nichol, 2005). These chemicals may leach into the Steam Shovel Slough over time by the action of storm water infiltration and ground water flow. The active oil pumping in the area may also contribute to sediment contamination by PAHs through interaction of the Steam Shovel Slough with the degraded area. This may occur by the action of surface waters or possible groundwater flows. The close proximity of Pacific Coast Highway may also contribute to PAH contamination (Li, et al. 2003). The high degree of industrialization in the surrounding area was mainly built prior to 1977. Consequently, there may also be significant levels of Polychlorinated Biphenyls (PCBs) within the sediments of the Los Cerritos Wetlands. Residential and commercial pesticide use may also contribute to pesticide contamination of the waters of the Los Cerritos Wetlands, especially immediately following storm events. The most significant pesticide residues may be organophosphorous pesticides, since they are currently manufactured and widely used today.

Objectives and Hypotheses

The distribution and potential sources of PAHs, PCBs, and pesticides within the Steam Shovel Slough must be determined in order to formulate effective restoration projects and management policies. The quantitation of priority PAHs, PCBs, and pesticides is essential to the development of Total Maximum Daily Loads (TMDLs) for the Los Cerritos Channel and Alamitos Bay, that will minimize the adverse impacts of these pollutants on the salt marsh ecosystem in terms of functionality, and species richness and abundance.

It is hypothesized that:

The identities and relative abundances of PAHs will depend on the source, since different types of fossil fuels are used and varying industrial processes are in place at the Long Beach Airport (jet fuel and gasoline), the AES and Haynes power plants (natural gas), and the oil drilling wells (crude oil, petroleum derivatives/contaminants).

The distribution of PAHs and/or their concentration ratios in the Steam Shovel Slough will be represented by a gradient dependent upon the distance from the source of a particular pollutant or group of pollutants. PAHs and PCBs from the power plants by aerial deposition and/or storm events may show a decreasing concentration gradient further into the Steam Shovel Slough, toward Alamitos Bay.

A decreasing concentration gradient or source footprint of PAHs will be detectable with increasing distance from the culvert connecting the Steam Shovel Slough to the degraded wetlands areas, where oil drilling wells are operational.

A decreasing concentration gradient of PAHs, PCBs, and pesticides may be shown from the inlet of the Steam Shovel Slough through the more inland mudflat areas.

The top sediments of the mudflat areas will contain lower concentrations of targeted pollutants due to increased remediation efforts and technological improvements (more efficient combustion of fossil fuels) within the past few decades.

Materials and Methods

Sample Collection

Sediment samples were collected from the Steam Shovel Slough using a stainless steel 1-inch diameter sediment core auger. The length of the sampled core is approximately 30cm. The samples were collected by boat during the incoming high tide, in the early evening in April of 2006. The sediment was separated into two fractions on site into separate Quarkpak glass jars (0-15cm and 15-30cm). Samples were collected from four different sites, mostly along the shallow areas of the Steam Shovel Slough. The samples were then stored at 0°C for approximately one week prior to analysis. Two water samples were collected at the same time at selected locations. A 1-L sample was collected in the middle of the Steam Shovel Slough using a selective depth water sampler, equipped with a 1-L glass bottle. The sample was collected at approximately 7-10ft below the surface of the water. GPS coordinates were recorded for each sample and will be included in subsequent reports. Approximately 20, 20mL glass vials were also filled with water samples from the vicinity of the culvert connecting the Steam Shovel Slough to the degraded wetland area (Steam Shovel Slough to Bixby property)

Sediment and Water Extractions, sample preparations

Sediment samples were allowed to reach room temperature (slightly frozen, then thawed completely) and were homogenized by stirring thoroughly with a plastic spoon. Excess water was drained from the jars prior to homogenization. Approximately 10g of each sample was used for analysis. Dry/wet weight ratios will be determined in order to calculate concentrations on an ng/g dry sediment basis. Separate aliquots of approximately 7g were used from each sample to accurately correct for water weight. All solvents and chemicals used are of at least pesticide grade quality, unless otherwise noted, and were purchased from Fischer Scientific, Inc.. Each sample was extracted three times by microwave extraction using 25mL of methylene chloride per extraction. A method blank was also processed, consisting of only solvent and surrogate standards, in the same batch as the analytical samples, applying the same procedures. Deuterated PAHs, and selected laboratory use PCBs were spiked into each sediment sample and method blank prior to the first addition of methylene chloride. The microwave utilizes pressure/temperature gauged Teflon vessels encased in Kevlar and operates by the following method: 25°C initial temperature, 5°C/min to 100°C (hold for 15 minutes), and then cool to approximately room temperature. The extracts were passed through pre-cleaned (baked in oven at high temp.) anhydrous sodium sulfate (pesticide grade, Fisher Scientific) and into 100mL pear-shaped flasks prior to concentration (~0.5mL) and solvent exchange into hexane (pesticide grade, Fisher Scientific) via rotary evaporation. The concentrated samples were then placed on a chromatography column packed with 12cm of prepared absorbent silica gel and 6cm of prepared absorbent alumina. The samples were quantitatively transferred with hexane and then eluted with 30mL of hexane, then 30mL of 30% methylene chloride/hexane solution. The elution was collected in solvent cleaned 100mL pearshaped flasks and subsequently concentrated by rotary evaporation to approximately 1mL. The concentrated samples were then transferred to autosampler vials and activated copper was added to remove sulfurous compounds.

Water samples were extracted into methylene chloride and dried with sodium sulfate, using the above surrogate standards for both samples and a method blank. Upon concentration by rotary evaporation, these samples were analyzed by GC/MS using internal standards. Organophosphorous pesticides are the target analytes along with possible detection of PAHs, PCBs, and other pesticide derivatives. Small amounts of activated copper were added to remove bacterial related sulfurous compounds. The method blank was processed using 500mL of nano-pure water.

Silica/Alumina Absorbent Preparation

The absorbents (pesticide grade) are prepared by placing 350g of silica gel and 500g of alumina into two separate 1-L beakers with 500mL of methanol added to each. The mixtures are sonicated for 30min. After removal of excess methanol, the absorbents are then rinsed three times with 100-200mL of methylene chloride. 500mL of methylene chloride is then added to each beaker and then they are sonicated for 30min. Once the methylene chloride is removed, the absorbent is left to dry overnight, covered with aluminum foil with small holes in it. The silica gel is then heated to 180°C and left isothermal overnight. The alumina is heated to 250°C and left isothermal

overnight. The absorbents are then placed in a jar and 3% DI water is added by weight, and the absorbents/water mixture is shaken. They are left to equilibrate overnight and then covered with hexane, then stored at room temperature in glass jars.

GC/MS Analysis/Quantitation

Immediately prior to analysis, each sample was placed under nitrogen to maintain approximately a 1mL volume. Seemingly dirtier samples, those that precipitated the most during solvent exchange and appeared dark in color, were diluted a factor of 10x prior to initial analysis. Internal standards, Anthracene-d10 and 2,2',5,5'-Tetrabromobiphenyl were added to each autosampler vial to correct for volume differences and for proper quantitation using standard curves for target analytes on a mass basis. A DB-5, 60m gas chromatography column is used to conduct analysis on an Agilent 6890N GC system coupled to an Agilent 5973 MSD, equipped with an autosampler. Characteristic GC/MS parameters are as follows: Splitless injection, injector temp.: 285°C; transfer line temp.: 285°C; oven temp. ramp: 45°C (hold 5 min), 25°C/min to 150°C, 2.5°C/min to 285°C (hold 17 min); electron ionization will be used, 70eV, MSD quad temp.: 150°C; MSD source temp.: 230°C; operated in scan mode for ions of mass 50-500g/mole. The same method will be used for sediment and water sample analysis. The method used is from IIRMES-CLP (Institute for Integrated Research in Materials, Environments, and Society- Contract Lab Program) (Gosset, et al.), and may vary from above if deemed necessary (Schubert, et al. 2003)..

Results & Discussion

The analysis of PCBs and pesticides did not result in their detection within the mud cores sampled. Although possible detection of DDT, Methoxychlor, Hepatochlor, and BHC gamma may be possible, their observed concentrations may approach the detection limit of the method used, resulting in poor accuracy of identification and quantitation. The water sample analyses did not result in detection of organophosphorous pesticides, PAHs, or PCBs. The analysis and quantitation of 25 priority PAHs was determined for four mud core sample sites, partitioned by depth (Table I). Site MC-D was by far the most contaminated site. Initial interpretations of this phenomenon reflect the close proximity of this area to high automobile traffic and to the culvert that connects the Steam Shovel Slough to the degraded wetland habitat (where oil drilling occurs) (Figure 1). The distribution of PAHs is not uniform throughout the sample locations, and may reflect partitioning effects of PAHs via sediment absorption/reactivity and aqueous transport. The timing of the tides may also effect the observed distribution of PAHs. The fractional composition of selected PAHs following EPA CMB8.2 model was also determined (Figure 2a-h).

Figure 1: The distribution of the total amount of PAHs found across each sample location. The distribution among sample sites is relatively uniform for all sites except for Site MC-D, which has significantly higher concentrations of PAHs. Another discrepancy is the increased concentration of PAHs within the top sediments of site MC-A relative to the deeper MC-A core.

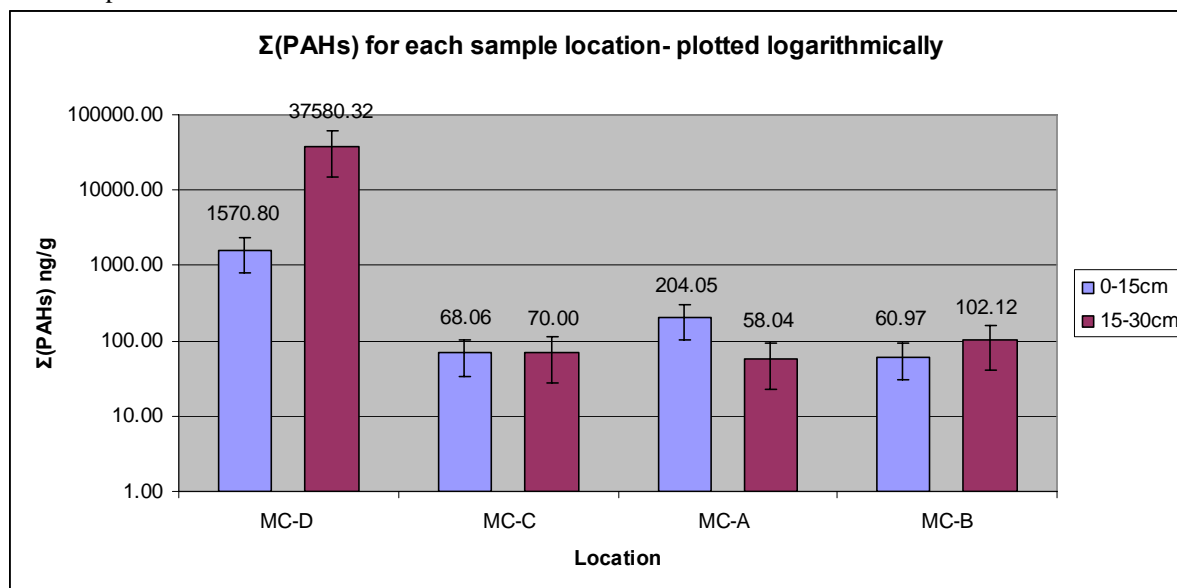


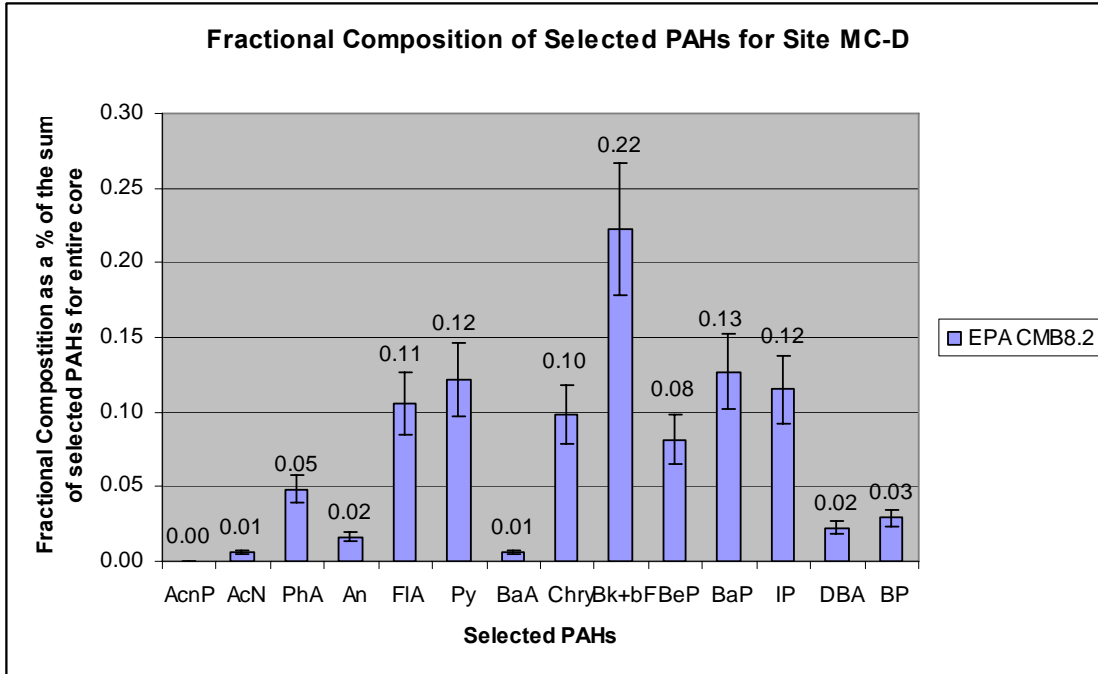
Table I: List of target PAHs and their respective concentration by location and depth of sample (estimated sample error is within 20%)

**Compound	Site MC-D [PAH] (ng/g)		Site MC-C [PAH] (ng/g)		Site MC-A [PAH] (ng/g)		Site MC-B [PAH] (ng/g)	
	(0-15cm)	(15-30cm)	(0-15cm)	(15-30cm)	(0-15cm)	(15-30cm)	(0-15cm)	(15-30cm)
Naphthalene	15.11	174.07	2.07	2.62	1.98	1.93	2.30	2.26
2-methylnaphthalene	12.45	452.90	1.97	2.87	2.60	2.23	5.15	4.00
1-methylnaphthalene	11.25	349.78	1.08	1.04	1.16	1.07	1.91	1.89
Biphenyl	1.87	52.47	1.17	1.64	1.36	0.83	1.65	1.45
2,6-Dimethylnaphthalene	13.73	451.80	1.17	1.16	3.22	2.12	5.20	3.68
Acenaphthylene*	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.94
Acenaphthene*	0.00	211.08	0.00	0.00	0.00	0.00	0.00	0.00
2,3,5-Trimethylnaphthalene*	0.00	190.42	0.00	0.00	0.00	0.00	1.97	1.43
Flourene*	13.60	262.08	0.00	0.00	2.31	0.00	1.81	1.46
Dibenzothiophene*	12.15	210.88	2.58	1.80	2.27	0.00	0.00	0.00
Phenanthrene	82.60	1644.29	4.28	2.67	6.72	3.20	3.44	5.03
Anthracene*	36.58	542.59	1.88	5.55	2.86	0.00	0.00	2.26
1-methylphenanthrene*	0.00	249.07	0.00	2.96	0.00	0.00	0.00	0.00
Flouranthene	136.64	3619.47	6.53	0.15	13.02	5.28	4.33	13.03
Pyrene	166.96	4168.63	7.60	7.38	16.64	6.22	6.92	13.34
Benz [a] anthracene	183.05	48.71	5.88	5.01	15.44	4.43	2.61	5.80
Chrysene	146.67	3345.22	2.66	5.07	9.20	3.11	2.05	6.00
Benzo [k] flouranthene	106.00	4459.59	4.54	2.66	16.59	5.26	3.74	7.74
Benzo [b] flouranthene	75.96	3278.73	4.14	4.56	13.94	3.93	3.15	6.43
Benzo [e] pyrene	109.22	2791.65	5.63	3.27	19.74	6.49	4.89	8.76
Benzo [a] pyrene	142.52	4374.76	5.08	5.46	18.15	6.17	5.81	7.95
Perylene*	88.51	979.51	0.00	6.42	9.55	0.87	0.00	4.16
Indeno [1,2,3-c,d] pyrene*	85.11	4016.70	4.08	0.36	16.85	4.90	0.00	4.52
Dibenz [a,h] anthracene*	23.86	779.87	0.00	0.00	8.07	0.00	0.00	0.00
Benzo [g, h, i] perylene*	106.95	926.06	5.71	7.35	22.38	0.00	4.04	0.00

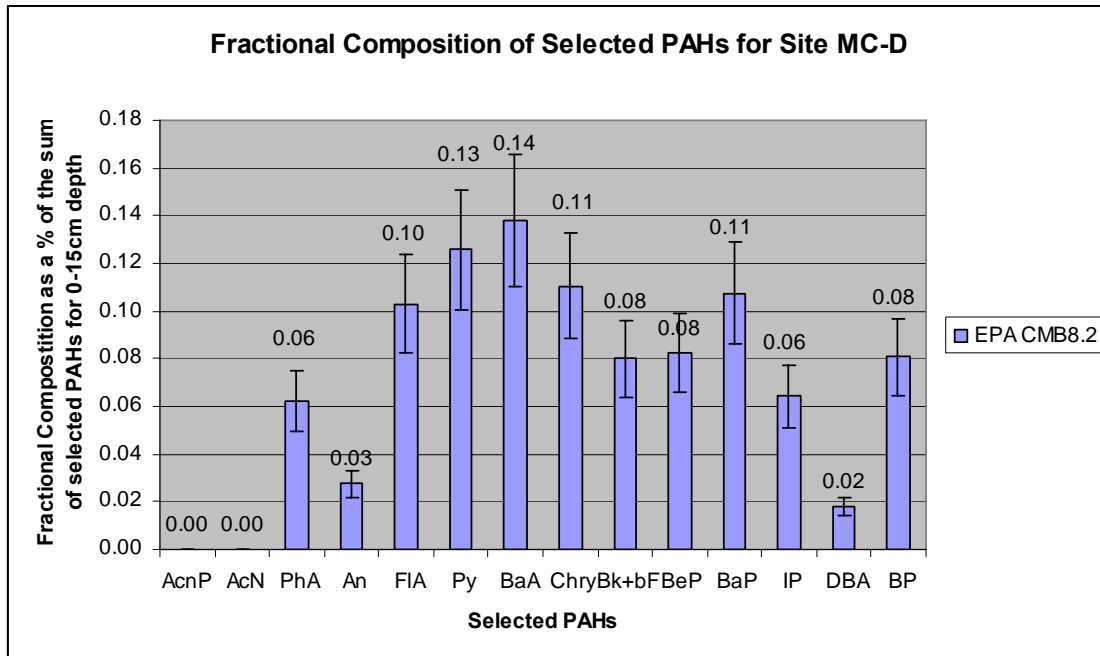
*zero level concentrations do not reflect absence of compound, but inadequate extraction, or the detection limit and sensitivity of the method.

**Compounds in bold are used for EPA CMB8.2 modeling ratios.

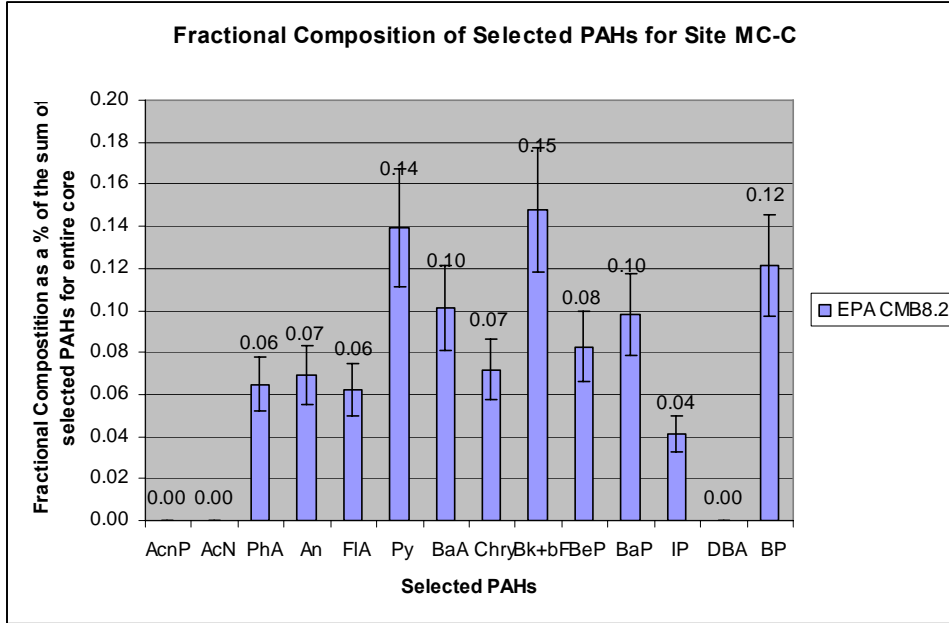
Figure 2: a) The fractional composition of individual PAHs that make up the sum of the total *selected* PAH concentration are specific to a particular source. **2b)** A high contrast between the deep core composition and the top sediments was observed for site MC-D. This reflects significant differences between the source or composition of deposition between the two depositional time frames, which must be still determined. (AcnP= Acenaphylene, AcN= Acenaphthene, PhA= Phenanthrene, An= Anthracene, FIA= Flouranthene, Py= Pyrene. BaA= Benz [a] anthracene, Chry= Chrysene, Bk+bF= Benzo [k] + [b] flouranthene, BeP= Benzo [e] pyrene, BaP= Benzo [a] pyrene, IP= Indeno [1,2,3,-c,d] pyrene, DBA= dibenzo[a,h] anthracene, BP= Benzo [g,h,i] perylene) All error bars reflect 20% analytical precision.



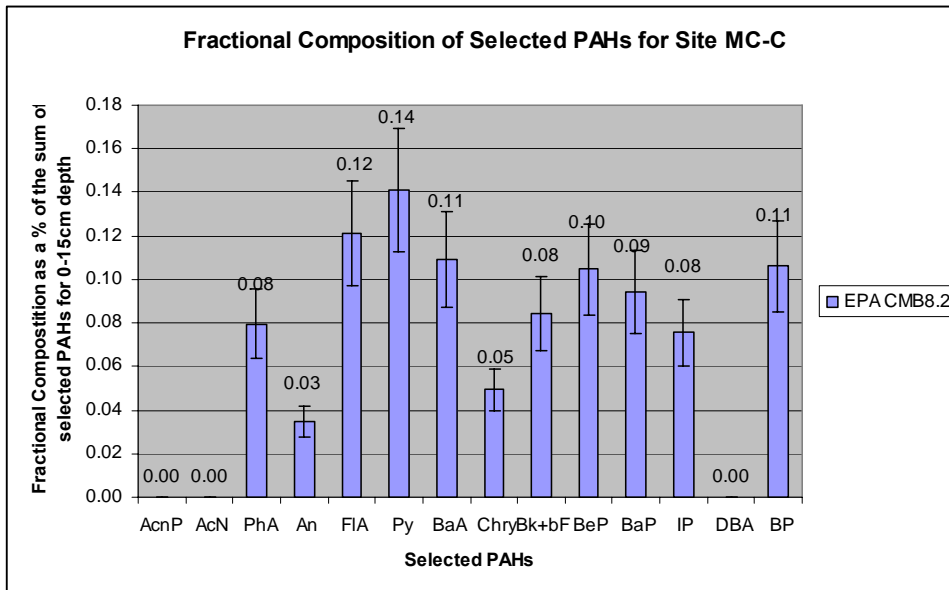
2b)



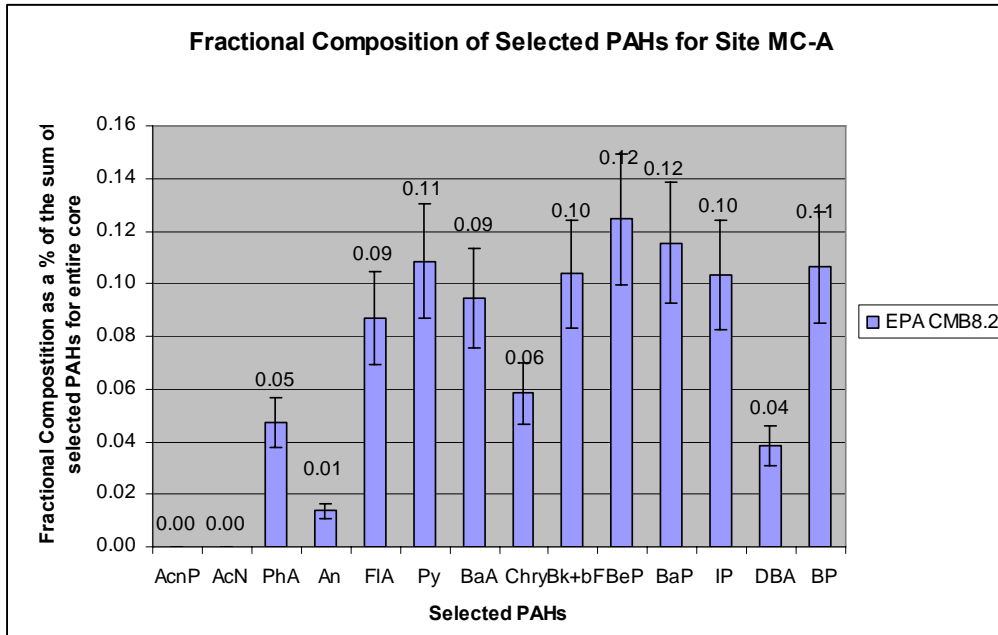
2c) The fractional composition is somewhat uniform for both top and deep sediments. However, Bk+bF and FIA are much more prevalent in deeper sediments, similar to site MC-D.



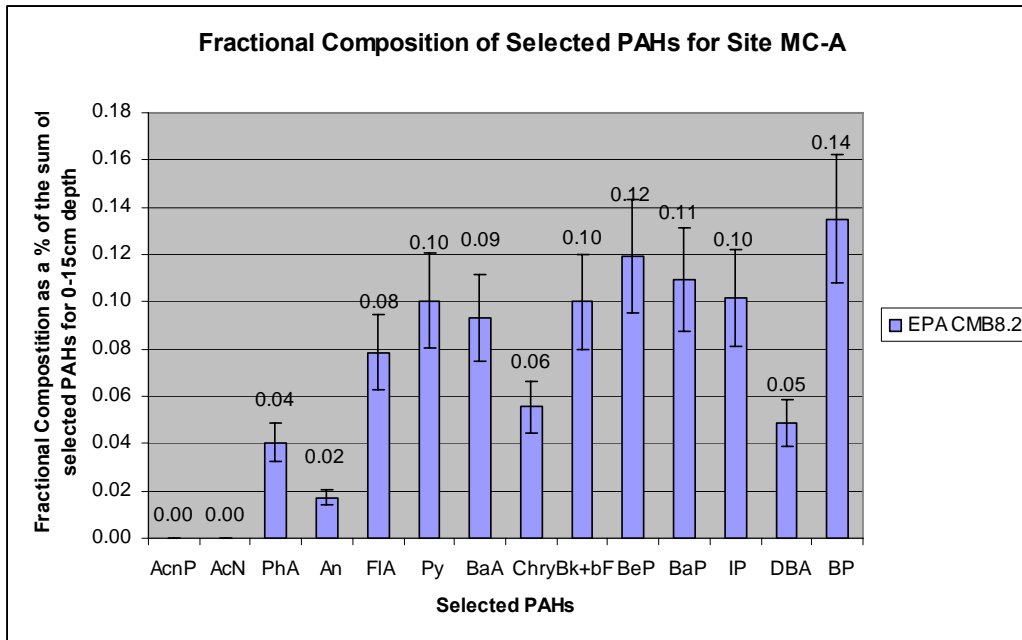
2d) A significant decrease in Bk+bF composition is evident.



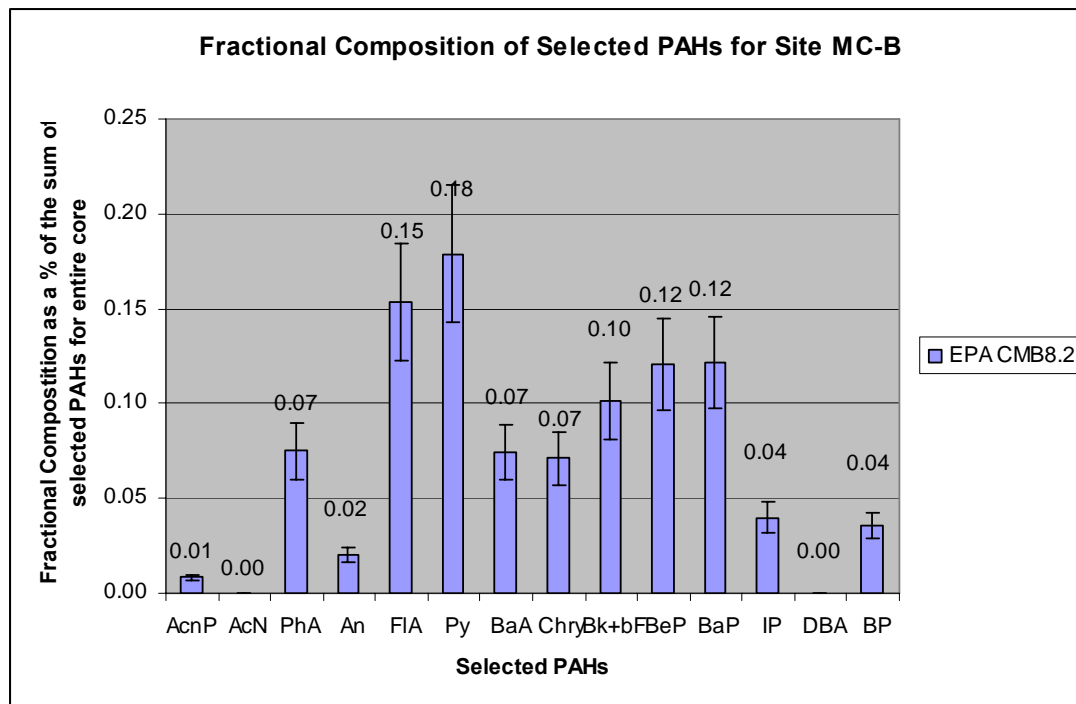
2e) The fractional composition is somewhat uniform between the top and deeper sediments.



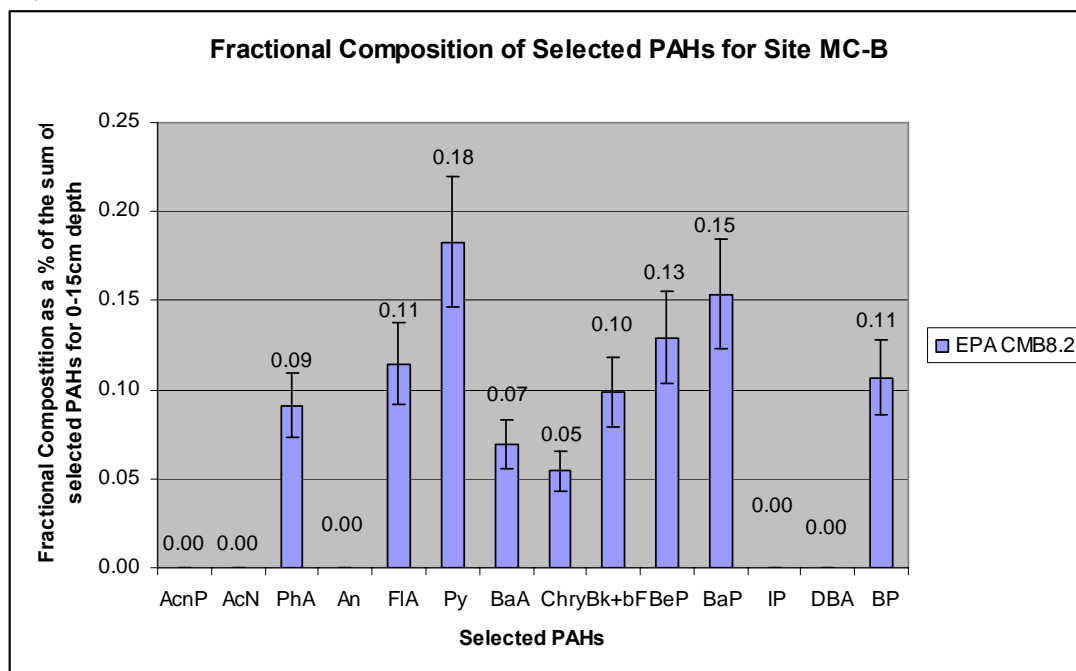
2f)



2g) Again, the fractional composition is relatively uniform among the two depths.



2h)



The sum of PAH contamination ranges from 58.4 ng/g to 37,580.32 ng/g among the four sample sites and sample depths. The detection limit of the method was calculated (from target ion signal) to be approximately 10ppb for MW of approximately 220g/mol and higher, and approximately 5ppb for MW of less than 220g/mol. The sensitivity of the method was found to be approximately 200 ppb⁻¹ and 500 ppb⁻¹, respectively. The calibration curves were linear for concentrations of up to 600ppb (beyond this the data had to be extrapolated from curve due to time constraints). The detection limit was determined by statistical analysis of three runs of the 25ppb calibration standards and three blank runs, defined as the minimum sample concentration to achieve a target ion signal equal to the average blank signal plus three times the standard deviation of the 25ppb sample signal. The sensitivity is a function of the change in target ion current (selected ion chromatogram) and the change in concentration of the sample. A sensitivity of 200 ppb⁻¹, combined with a standard deviation of approximately 500 counts (for selected ion current), results in a concentration sensitivity of approximately 3-5ppb for higher MWs, and given 500 ppb⁻¹ and a signal standard deviation of 800 counts results in a concentration sensitivity of 3-5ppb as well. Consequently, all reported concentrations below 5ppb are unreliable. The precision of the method was found to be approximately 20% from analysis of duplicate samples.

The fractional compositions of all sites except for MC-D are characteristic of combustion vehicle traffic sources, and aerial deposition from power plants. Although a statistical analysis was not performed against the literature data or apportionment model, the ratios of the fractional compositions are within their respective error limits of a uniform 20% for the GC/MS analyses (Li, et al. 2003) (Appendix C). The major discrepancy in this source apportionment is the increased Bk + bF composition, which may be due to the combination of sources (no coal fired or coke ovens have been reported in the area, known to researchers). This may reflect contamination of site MC-D by the degraded wetland. PAHs that infiltrate the area will only occur during the outgoing tide, and it is therefore logical to assume that contaminants from the oil drilling source are distributed away from the Steam Shovel Slough. Unfortunately, no sample sites were analyzed from this area, and such comparisons may not be made at this time. There is little correlation between the distribution of PAHs among site MC-D and the other locations, except for the decreased fractional composition of Benzo fluoranthenes among the top sediments of site MC-D and MC-C relative to deeper sediments. The non-detection of analytical contaminants in the water samples is thought to be due to the absence of significant storm water influences during sample collection, and the continuous operation of the AES power plant cooling towers.

Materials Cost Sheet

Funds are available or have been expended for the following:

Vendor	Catalog number	Description	Total Cost	Unit	Qty.
Category 1- Purchased Items					
Fisher	03-377-1A	Blue Autosampler Vial Caps	\$13.45	pack of 100	1
Fisher	03-391-8	2mL Autosampler Vials	\$8.35	pack of 100	1
Fisher	09-549-2A	Evap Trap- 19/22	\$67.61	each	1
Fisher	K294260-0050	50mL Pearshaped Flask	\$18.51	each	3
Fisher	K294260-0100	100mL Pearshaped Flask	\$19.21	each	10
Fisher	K420280-0213	Chromaflex Columns	\$66.38	each	3
Fisher	S415-500	Sodium Anhydrous Sulfate	\$5.23	500g	1
Fisher	06-408-10	Vial Rack	\$48.18	case of 5	1
Fisher	14-959-92B	Cotton Swabs	\$5.22	pak of 1000	1
Solvents (pesticide grade)					
Fisher	D150-4	Methylene Chloride	\$32.75	1 x 4L	1
Fisher	A40-4	Acetone	\$18.27	1 x 4L	1
Fisher	H302-4	Hexane	\$12.73	1 x 4L	1
Fisher	A450-4	Methanol	\$11.06	1 x 4L	1
Calib., Surrogate, Internal Std.					
Accustandard	B-052N	2,2',5,5'-Tetrabromobiphenyl	\$24.00	1 x 1mL	1
Accustandard	C-030S	2,4,6-Trichlorobiphenyl	\$15.00	1 x 1mL	1
Accustandard	C-112N	2,3,3',5,6-Pentachlorobiphenyl	\$55.00	1 x 1mL	1
Accustandard	S-279-5X	Tetrachloro-m-xylene	\$10.00	1 x 1mL	1
Accustandard	Z-014J	Surrogate Std Mix- PAHs	\$80.00	1 x 1mL	1
Accustandard	M-001N	Anthracene-d10	\$15.00	1 x 1mL	1
Accustandard	M-8140M-5X-PAK	Organophosphate pesticide mix	\$125.00	1 x 1mL	1
Gloves					
Fisher	11-390-13B	Med Gloves	\$16.14	pak of 100	
Fisher	11-390-13C	Large Gloves	\$16.14	pak of 100	
				Total	\$1,025.90
Category 2- Replenishment of Consumables					
Agilent	5181-3316	Injector Liners	\$28.95	each	1
Agilent	5062-3508	Ferrules 0.4mm- Injector	\$61.94	pak of 10	1
Fisher	S734-1	Silica Gel	\$87.37	1kg	140 grams
Fisher	A941-500	Alumina	\$31.45	500g	70 grams
				Actual Cost	
11 samples, 12g for each sample			Silica Gel	\$14.00	
11 samples, 6g for each sample			Alumina	\$8.00	
				Total	\$112.89

If column replacement is required as a result of analysis, an additional \$350-\$400 is available.

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Appendix A- Los Cerritos Wetlands Aerial Photo with sample sites.

Image 1: Mud Core Sample Sites; sites MC1, MC2, MC3, MC4, and MC5 were used for mineral composition studies via XRD and PIMA.



Image 2: Water Sample Sites



Appendix A: Sample Site locations data table.

MC= Mud Core WS= Water sample

<u>Sample ID</u>	<u>Core Length</u>	<u>Depth of Sample</u>	<u>Latitude</u>	<u>Longitude</u>	<u>Elevation (ft)*</u>
MCA	30cm	0-15 & 15-30cm	33°45.855'N	118°06.507'W	7
MCB	30cm	0-15 & 15-30cm	33°45.885'N	118°06.447'W	-2
MCC	30cm	0-15 & 15-30cm	33°45.846'N	118°06.648'W	20
MCD	30cm	0-15 & 15-30cm	33°45.834'N	118°06.700'W	-27
WSA	---	3 m	33°45.884'N	118°06.570'W	8
WSB	---	0.3 m	33°45.833'N	118°06.698'W	-24

*elevations are above sea level and were taken from the GPS readings, the error may be several feet

Appendix B: Aerial Photo of functioning wetland (Steam Shovel Slough) and the degraded wetland area.



Adapted from: Los Cerritos Wetlands Land Trust for Seal Beach and Long Beach, Information Brochure

Appendix C: Source fingerprints (fractional composition in sediment), adapted from Li, et al. 2005.

