METALS QA/QC

PROGRAM: SPAWAR, Task 16
PARAMETER: Metals
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Stormwater

QA/QC DATA QUALITY OBJECTIVES

	Reference Method	Range of Recovery	SRM Accuracy	Relative Precision	Target Detection Limit (µg/L)
Aluminum	ICP/OES	50-150%	±20%	±50%	50.0
Iron	ICP/OES	50-150%	±20%	±50%	10.0
Manganese	ICP/OES	50-150%	±20%	±30%	0.5
Chromium	ICP/MS	50-150%	±20%	±30%	1.0
Nickel	ICP/MS	50-150%	±20%	±30%	0.05
Copper	ICP/MS	50-150%	±20%	±30%	0.05
Zinc	ICP/MS	50-150%	±20%	±30%	0.5
Arsenic	FIAS	50-150%	±20%	±30%	0.5
Selenium	FIAS	50-150%	±20%	±30%	0.2
Silver	GFAA	50-150%	±20%	±30%	0.5
Cadmium	ICP/MS	50-150%	±20%	±30%	0.05
Tin	ICP/MS	50-150%	±20%	±30%	0.5
Lead	ICP/MS	50-150%	±20%	±30%	0.05
Mercury	CVAF	50-150%	±25%	±30%	0.01

METHOD

Five (5) samples were analyzed: ten (10) metals; chromium (Cr), nickel (Ni), copper, (Cu), zinc (Zn), arsenic (As), selenium (Se), silver (Ag), cadmium (Cd), tin (Sn) and lead (Pb) by inductively coupled plasma mass spectroscopy (ICP/MS) following EPA Method 1638m, aluminum (Al), iron (Fe), and manganese (Mn) by inductively coupled plasma optic emission spectroscopy following EPA Method 200.7 and mercury (Hg) by cold vapor atomic fluorescence (CVAF) following EPA Method 1631e.

Samples were preserved with nitric acid prior to arrival at MSL. Samples were analyzed by EPA Method 1638m. Samples analyzed for Hg by CVAF were pre-treated with bromine chloride and stannous chloride to oxidize and convert all Hg compounds to volatile Hg, which is subsequently trapped onto a gold-coated sand trap.

HOLDING TIMES

Five (5) samples were received on 10/29/2004 and were logged into Battelle's sample tracking system. Five samples were analyzed within the six month holding time for metals and 90 days for Hg. The following list summarizes all analysis dates:

QA/QC SUMMARY/METALS - PRISM Task 16 (continued)

Task	Date Performed
Hg	11/16/04
ICP-MS	11/29/04 & 12/8/04
ICP-OES	11/21/04

DETECTION LIMITS

The target detection limit was met for all metals. The method detection limit was met for all metals. An MDL is determined by multiplying the standard deviation of the results of a minimum of 7 replicate low level spikes by the Student's t value at the 99th percentile.

METHOD BLANKS

One method blank was analyzed with this batch of samples. Results were less than 5 times the MDL for all metals.

BLANK SPIKES

One sample of reagent water was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

MATRIX SPIKES

One sample was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

REPLICATES

One sample was analyzed in duplicate. All results were within the project criteria.

SRM

Two matrix-appropriate standard reference materials (SRM) were analyzed for each method; 1641d, river water, and 1640, natural water, obtained from the National Institute of Science and Technology.

SRM 1640 has 22 certified metals. Recovery for all metals reported were within the control limit of ±20% of the certified value, except Se that had a % difference of 21%. All other QC for this metal were within acceptable criteria. No corrective action was taken. Tin and Hg are not certified in 1640. SRM 1641d is certified for Hg. Recovery for Hg was within the control limit of ±25% of the certified value.

REFERENCES

EPA. 1991. Methods for the Determination of Metals in Environmental Samples. EPA-600/4- 91-010. Environmental Services Division, Monitoring Management Branch.

METALS QA/QC (CONT.)

MSL Code	Rep	Sponsor I.D.	Al (µg/L) ICP-OES		Fe (µg/L) ICP-OES		Cr (µg/L) ICP-MS		Mn (µg/L) ICP-OES		Ni (µg/L) ICP-MS		Cu (µg/L) ICP-MS	
PROCEDURAL BLANK			3.36	U	2.51	U	0.018	U	0.025	U	0.009	U	0.008	U
METHOD DETECTION LIMIT			**3.36**		**2.51**		**0.018**		**0.025**		**0.009**		**0.008**	
Project Target Detection Limit			**50.0**		**10.0**		**1.00**		0.50		**0.05**		**0.05**	
STANDARD REFERENCE MATERIAL														
1640			50.5		36.0		39.9		121		28.5		88.8	
1640		certified value	52.0		34.3		38.6		122		27.4		85.2	
1640		range	±1.5		±1.6		±1.6		±1.1		±0.8		±1.2	
		% difference	**3%**		5%		**3%**		1%		**4%**		**4%**	
1641d			NA		NA		NA		NA		NA		NA	
1641d		certified value	NC		NC		NC		NC		NC		NC	
1641d		range	NC		NC		NC		NC		NC		NC	
		% difference	**N/A**		N/A		**N/A**		N/A		**N/A**		**N/A**	
ICV,CCV RESULTS														
ICV			**99%**		**104%**		**104%**		100%		**103%**		**104%**	
CCV			**98%**		**102%**		**103%**		95%		**102%**		**103%**	
CCV			**99%**		103%		**106%**		95%		**101%**		**104%**	
CCV			**NA**		**NA**		**107%**		NA		**103%**		**104%**	
BLANK SPIKE RESULTS														
		Amount Spiked	100		50.0		10.0		50.0		10.0		10.0	
		Blank	3.36	U	2.51	U	0.018	U	0.03	U	0.01	U	0.008	U
		Blank + Spike	98.6		53.1		10.9		51.8		9.64		9.83	
		Amount Recovered	98.6		53.1		10.9		51.8		9.64		9.83	
		Percent Recovery	**99%**		106%		**109%**		104%		**96%**		**98%**	
MATRIX SPIKE RESULTS														
		Amount Spiked	250		200		NS		50.0		NS		NS	
		NAV-OF14-SD45-FF (F)	14.7		26.4		N/A		29.2		N/A		N/A	
		NAV-OF14-SD45-FF (F) + Spike	258		238		NA		79.5		NA		NA	
		Amount Recovered	243		212		N/A		50.3		N/A		N/A	
		Percent Recovery	**97%**		**106%**		**N/A**		101%		**N/A**		**N/A**	
		Amount Spiked	NS		NS		50.0		NS		10.0		50.0	
		NAV-OF14-SD45-COMP	N/A		N/A		12.9		N/A		4.81		38.0	
		NAV-OF14-SD45-COMP + Spike	NA		NA		65.4		NA		15.0		88.7	
		Amount Recovered	N/A		N/A		52.5		N/A		10.2		50.7	
		Percent Recovery	**N/A**		**N/A**		**105%**		**N/A**		**102%**		**101%**	
		Amount Spiked	NS		NS		NS		NS		NS		NS	
		NAV-OF14-SD45-COMP (F)	N/A		N/A		N/A		N/A		N/A		N/A	
		NAV-OF14-SD45-COMP (F) + Spike	NA		NA		NA		NA		NA		NA	
		Amount Recovered	N/A		**N/A**		N/A		N/A		N/A		N/A	
		Percent Recovery	**N/A**		**N/A**		**N/A**		**N/A**		**N/A**		**N/A**	
MSL Code	**Rep**	**Sponsor I.D.**	**Al (µg/L) ICP-OES**		**Fe (µg/L) ICP-OES**		**Cr (µg/L) ICP-MS**		**Mn (µg/L) ICP-OES**		**Ni (µg/L) ICP-MS**		**Cu (µg/L) ICP-MS**	
REPLICATE RESULTS														
2173*27	1	NAV-OF14-SD45-FF	1322		2138		6.93		66.2		7.19		45.3	
2173*27	2	NAV-OF14-SD45-FF	NA		NA		7.22		NA		7.14		44.9	
		RPD	**N/A**		**N/A**		**4%**		**N/A**		**1%**		**1%**	
2173*28	1	NAV-OF14-SD45-FF (F)	14.7		26.4		2.22		29.2		3.67		18.9	
2173*28	2	NAV-OF14-SD45-FF (F)	14.9		31.2		NA		29.4		NA		NA	
		RPD	**1%**		**17%**		**N/A**		**1%**		**N/A**		**N/A**	

METALS QA/QC (CONT.)

MSL Code	Rep	Sponsor I.D.	Zn (µg/L) ICP-MS		As (µg/L) ICP-MS		Se (µg/L) ICP-MS		Ag (µg/L) ICP-MS		Cd (µg/L) ICP-MS		Sn (µg/L) ICP-MS		Pb (µg/L) ICP-MS		Hg (µg/L) CVAF	
PROCEDURAL BLANK			0.028	U	0.015	U	0.101	U	0.002	U	0.002	U	0.50	U	0.001	U	0.00012	U
METHOD DETECTION LIMIT			0.028		0.015		0.101		0.002		0.002		NA		0.001		0.00012	
Project Target Detection Limit			0.50		0.50		0.20		0.50		0.05		0.50		0.05		0.01	
STANDARD REFERENCE MATERIAL																		
1640			61.2		29.8		26.6		7.78		24.5		1.68		29.4		NA	
1640		certified value	53.2		26.7		22.0		7.62		22.8		NC		27.9		NC	
1640		range	±1.1		±0.73		±0.51		±0.25		±0.96		NC		±0.14		NC	
		% difference	15%		12%		21%	#	2%		7%		N/A		5%		N/A	
1641d			NA		NA		NA		NA		NA		NA		NA		1641	
1641d		certified value	NC		NC		NC		NC		NC		NC		NC		1590	
1641d		range	NC		NC		NC		NC		NC		NC		NC		±4.00	
		% difference	N/A		N/A		N/A		N/A		N/A		N/A		N/A		3%	
ICV,CCV RESULTS																		
ICV			103%		104%		102%		103%		103%		100%		105%		99%	
CCV			101%		104%		103%		104%		104%		105%		107%		100%	
CCV			101%		103%		103%		102%		102%		104%		106%		NA	
CCV			102%		103%		103%		102%		100%		103%		106%		NA	
BLANK SPIKE RESULTS																		
		Amount Spiked	10		10.0		10.0		10.0		10.0		0.100		10.0		0.00506	
		Blank	0.028	U	0.015	U	0.101	U	0.002	U	0.002	U	0.50	U	0.001	U	0.00012	U
		Blank + Spike	8.66		9.22		8.87		9.71		9.34		0.111		10.3		0.00534	
		Amount Recovered	8.66		9.22		8.87		9.71		9.34		0.111		10.3		0.00522	
		Percent Recovery	87%		92%		89%		97%		93%		111%		103%		103%	
MATRIX SPIKE RESULTS																		
		Amount Spiked	NS		NS		NS		NS		NS		NS		NS		NS	
		NAV-OF14-SD45-FF (F)	N/A		N/A		N/A		N/A		N/A		N/A		N/A		N/A	
		NAV-OF14-SD45-FF (F) + Spike	NA		NA		NA		NA		NA		NA		NA		NA	
		Amount Recovered	N/A		N/A		N/A		N/A		N/A		N/A		N/A		N/A	
		Percent Recovery	N/A		N/A		N/A		N/A		N/A		N/A		N/A		N/A	
		Amount Spiked	50.0		10.0		10.0		10.0		10.0		1.00		50.0		NS	
		NAV-OF14-SD45-COMP	220		2.39		0.530		0.0632		0.871		0.536		21.6		N/A	
		NAV-OF14-SD45-COMP + Spike	270		12.9		10.8		10.0		10.9		1.43		73.5		NA	
		Amount Recovered	50.0		10.5		10.3		9.9		10.0		0.894		51.9		N/A	
		Percent Recovery	100%		105%		103%		99%		100%		89%		104%		N/A	
		Amount Spiked	NS		NS		NS		NS		NS		NS		NS		0.0298	
		NAV-OF14-SD45-COMP (F)	N/A		N/A		N/A		N/A		N/A		N/A		N/A		0.00331	
		NAV-OF14-SD45-COMP (F) + Spike	NA		NA		NA		NA		NA		NA		NA		0.0349	
		Amount Recovered	N/A		N/A		N/A		N/A		N/A		N/A		N/A		0.0316	
		Percent Recovery	N/A		N/A		N/A		N/A		N/A		N/A		N/A		106%	
MSL Code	Rep	Sponsor I.D.	Zn (µg/L) ICP-MS		As (µg/L) ICP-MS		Se (µg/L) ICP-MS		Ag (µg/L) ICP-MS		Cd (µg/L) ICP-MS		Sn (µg/L) ICP-MS		Pb (µg/L) ICP-MS		Hg (µg/L) CVAF	
REPLICATE RESULTS																		
2173*27	1	NAV-OF14-SD45-FF	362		3.20		1.30		0.0741		1.18		0.663		21.7		0.0629	
2173*27	2	NAV-OF14-SD45-FF	362		3.32		1.27		0.0743		1.15		0.631		21.3		NA	
		RPD	0%		4%		2%		0%		3%		5%		2%		N/A	
2173*28	1	NAV-OF14-SD45-FF (F)	175		2.04		0.848		0.00601		0.492		0.0371		0.493		0.00597	
2173*28	2	NAV-OF14-SD45-FF (F)	NA		NA		NA		NA		NA		NA		NA		0.00572	
		RPD	N/A		N/A		N/A		N/A		N/A		N/A		N/A		4%	

PAHs

CLIENT ID	NAV-OF14-SD45-FF		NAV-OF14-SD45-COMP	
Battelle ID	S5983-P		S5984-P	
Sample Type	SA		SA	
Collection Date	10/27/04		10/27/04	
Extraction Date	11/02/04		11/02/04	
Analysis Date	01/04/05		11/17/04	
Analytical Instrument	MS		MS	
% Moisture	NA		NA	
% Lipid	NA		NA	
Matrix	WATER		WATER	
Sample Size	1.64		2.63	
Size Unit-Basis	L_LIQUID		L_LIQUID	
Units	NG/L_LIQUID		NG/L_LIQUID	
Naphthalene	11.28	T	9.23	
C1-Naphthalenes	7.37	T	7.37	
C2-Naphthalenes	16.31	T	0.5	U
C3-Naphthalenes	158.2	T	0.5	U
C4-Naphthalenes	21.76	T	0.5	U
2-Methylnaphthalene	6.93	T	5.32	
1-Methynaphthalene	4.34	JT	3.62	
Biphenyl	4.46	JT	2.5	J
2,6-dimethylnaphthalene	4.36	JT	0.63	U
Acenaphthylene	3.23	JT	2	J
Acenaphthene	4.19	JT	1.67	J
2,3,5-trimethylnaphthalene	5.44	JT	0.44	U
Fluorene	5.97	JT	3.03	J
C1-Fluorenes	9.55	T	0.52	U
C2-Fluorenes	115.68	T	0.52	U
C3-Fluorenes	68.19	T	0.52	U
Anthracene	6.17	JT	0.38	U
Phenanthrene	64.76	T	41.97	
C1-Phenanthrenes/Anthracen	45.92	T	36.23	
C2-Phenanthrenes/Anthracen	105.52	T	76.88	
C3-Phenanthrenes/Anthracen	54.03	T	65.96	
C4-Phenanthrenes/Anthracen	32.13	T	0.82	U
1-Methylphenanthrene	12.92	T	8.5	
Dibenzothiophene	11.03	T	7.54	
C1-Dibenzothiophenes	78.5	T	0.38	U
C2-Dibenzothiophenes	54.81	T	54.93	
C3-Dibenzothiophenes	62.53	T	72.57	
C4-Dibenzothiophenes	41.57	T	0.38	U
Fluoranthene	95.74	T	63.01	
Pyrene	97.66	T	60.55	
C1-Fluoranthenes/Pyrenes	41.41	T	40.49	
C2-Fluoranthenes/Pyrenes	59.33	T	0.68	U
C3-Fluoranthenes/Pyrenes	57.53	T	0.68	U
Benzo(a)anthracene	23.09	T	12.25	
Chrysene	71.52	T	48.34	
C1-Chrysenes	56.41	T	38.79	
C2-Chrysenes	73.5	T	54.85	
C3-Chrysenes	74.63	T	0.45	U
C4-Chrysenes	1.79	UT	0.45	U
Benzo(b)fluoranthene	43.83	T	30.59	
Benzo(k)fluoranthene	34.62	T	26.34	
Benzo(e)pyrene	48.6	T	33.67	
Benzo(a)pyrene	31.31	T	17.58	
Perylene	12.59	T	9.35	
Indeno(1,2,3-cd)pyrene	33.58	T	22.7	
Dibenz(a,h)anthracene	7.22	T	3.93	
Benzo(g,h,i)perylene	62.28	T	43.59	
Total Priority Pollutant PAHs	596.45		387.16	
Naphthalene-d8	66		60	
Phenanthrene-d10	85		84	
Chrysene-d12	82		78	

PAHs QA/QC

PROJECT: Task Order TO0016 – YO817 Stormwater FY04
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 10/27/04. The samples were received at Battelle Duxbury on 10/29/04. Upon arrival, the cooler temperature was recorded at 1.7°C. No custody issues were noted. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0432, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PAH	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.47 – 1.93

METHOD: Water samples were extracted for PAH following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds. Initial analysis of sample S5983 yielded low surrogate recoveries. This was due to a concentration issue as noted in the sample preparation records. The archived non-fractionated extract for this sample was re-processed through the HPLC, concentrated, fortified with RIS and sent to GC/MS for PAH analysis only. Results from the second analysis have been reported.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection. All extracts were analyzed within the 40-day holding time, except for S5983. The data that was reported for this sample came from the second analysis, as noted above, which occurred outside of the 40-day holding time.

Batch	Extraction Date	Analysis Date
04-0432	11/2/04	11/16/04 – 11/17/04; reanalysis 1/4/05

BLANK: One procedural blank (PB) sample was prepared with the analytical batch. The procedural blank was analyzed to ensure the sample extraction and analysis methods were free of contamination.

04-0432 – No exceedences noted.

Comments – No target analytes were detected in the procedural blank except for Naphthalene. Naphthalene was detected at a concentration greater than the MDL, yet less than the RL. The data was qualified with an "J". Any field concentration for this target analyte, that was not greater than five times the concentration detected in the PB, was qualified with a "B". This resulted in Naphthalene data for sample S5991 (Duxbury Bay Water) being qualified with a "B".

LABORATORY CONTROL SAMPLE:

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits.

Comments – None.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE:

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair was prepared with each analytical batch. The percent recoveries of target PAH and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0432 – All target analytes were recovered within the laboratory control limits. All RPDs were within the laboratory control limits.
Comments – None.

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Note: At the time of extraction, no certified second source material was available. In lieu of a certified second source, the SRM sample was generated by spiking target analyte solution into a clean seawater sample from Duxbury Bay. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).
Comments – None

SURROGATES:

Three surrogate compounds were added prior to extraction, including Naphthalene-d8, Phenanthrene-d10, and Chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0432 – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (40 – 120% recovery).

Comments – After initial analysis Naphthalene-d8 was under-recovered in sample S5983 (OF14-SD45-FF). In the sample preparation records, an issue was noted regarding the concentration step after HPLC clean-up. It was determined that this sample was blow down to quickly on the N-evaporator. The archived portion of the extract was re-fractionated and re-analyzed. Since all SIS recoveries were acceptable in the second analysis, these results are reported.

PAHs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE			MATRIX SPIKE-NAV-OF14-SD45 FF			MATRIX SPIKE DUPLICATE-NAV-OF14-SD45-FF			PROCEDURAL BLANK	
Battelle ID	BF359LCS-P			S5983MS-P			S5983MSD-P			BF358PB-P	
Sample Type	LCS			MS			MSD			PB	
Collection Date	11/02/04			10/27/2004			10/27/2004			11/02/04	
Extraction Date	11/02/04			11/2/2004			11/2/2004			11/02/04	
Analysis Date	11/16/04			11/17/2004			11/17/2004			11/16/04	
Analytical Instrument	MS			MS			MS			MS	
% Moisture	NA			NA			NA			NA	
% Lipid	NA			NA			NA			NA	
Matrix	LIQUID			LIQUID			WATER			LIQUID	
Sample Size	2.00			0.5			0.5			2.00	
Size Unit-Basis	L_LIQUID			L_LIQUID			L_LIQUID			L_LIQUID	
Units	NG/L_LIQUID		% Recovery	NG/L_LIQUID		% Recovery	NG/L_LIQUID		% Recovery	NG/L_LIQUID	
Naphthalene	635.23		63	2426.22		60	2348.43		58	1.84	J
C1-Naphthalenes	0.66	U		3672.35			3591.42			0.66	U
C2-Naphthalenes	0.66	U		2.65	U		2.65	U		0.66	U
C3-Naphthalenes	0.66	U		2.65	U		2.65	U		0.66	U
C4-Naphthalenes	0.66	U		2.65	U		2.65	U		0.66	U
2-Methylnaphthalene	706.72		71	2767.73		69	2715.61		68	0.47	U
1-Methynaphthalene	626.85		63	2507.63		63	2441.57		61	0.5	U
Biphenyl	673.04		67	2704.17		67	2645.34		66	0.62	U
2,6-dimethylnaphthalene	715.93		72	2879.07		72	2845.32		71	0.83	U
Acenaphthylene	674.62		67	2745.32		69	2689.21		67	0.7	U
Acenaphthene	679.43		68	2808.53		70	2751.59		69	0.75	U
2,3,5-trimethylnaphthalene	734.65		73	3058.87		76	3003.23		75	0.58	U
Fluorene	712.63		71	3026.87		75	3016.1		75	0.68	U
C1-Fluorenes	0.68	U		2.72	U		2.72	U		0.68	U
C2-Fluorenes	0.68	U		2.72	U		2.72	U		0.68	U
C3-Fluorenes	0.68	U		2.72	U		2.72	U		0.68	U
Anthracene	807.28		81	3399.95		85	3402.93		85	0.51	U
Phenanthrene	774.53		77	3340.67		82	3296.59		81	1.08	U
C1-Phenanthrenes/Anthracen	1.08	U		4.32	U		4.32	U		1.08	U
C2-Phenanthrenes/Anthracen	1.08	U		4.32	U		4.32	U		1.08	U
C3-Phenanthrenes/Anthracen	1.08	U		4.32	U		4.32	U		1.08	U
C4-Phenanthrenes/Anthracen	1.08	U		4.32	U		4.32	U		1.08	U
1-Methylphenanthrene	834.72		83	3505.36		87	3490.79		87	0.61	U
Dibenzothiophene	0.5	U		57.47			55.56			0.5	U
C1-Dibenzothiophenes	0.5	U		2.01	U		2.01	U		0.5	U
C2-Dibenzothiophenes	0.5	U		134.99			116.79			0.5	U
C3-Dibenzothiophenes	0.5	U		138.25			122.43			0.5	U
C4-Dibenzothiophenes	0.5	U		2.01	U		2.01	U		0.5	U
Fluoranthene	866.46		87	3567.22		87	3516.93		85	0.77	U
Pyrene	878.52		88	3591.63		87	3584.54		87	0.9	U
C1-Fluoranthenes/Pyrenes	0.9	U		60.32			55.66			0.9	U
C2-Fluoranthenes/Pyrenes	0.9	U		3.59	U		3.59	U		0.9	U
C3-Fluoranthenes/Pyrenes	0.9	U		3.59	U		3.59	U		0.9	U
Benzo(a)anthracene	948.49		95	3474.22		86	3527.93		88	1.36	U
Chrysene	900.23		90	3393.36		83	3409.71		83	0.59	U
C1-Chrysenes	0.59	U		64.31			61.27			0.59	U
C2-Chrysenes	0.59	U		95.62			81.94			0.59	U
C3-Chrysenes	0.59	U		2.36	U		2.36	U		0.59	U
C4-Chrysenes	0.59	U		2.36	U		2.36	U		0.59	U
Benzo(b)fluoranthene	931.17		93	3566.1		**88**	3600.42		89	1.16	U
Benzo(k)fluoranthene	528.84		53	4035.04		**100**	4090.67		101	1.31	U
Benzo(e)pyrene	955.65		96	3761.95		93	3764.49		93	0.51	U
Benzo(a)pyrene	918.28		92	3621.84		90	3606.61		89	1	U
Perylene	912.89		91	3636.06		91	3677.71		92	1.93	U
Indeno(1,2,3-cd)pyrene	964.78		96	3732.44		92	3795.29		94	0.99	U
Dibenz(a,h)anthracene	969.69		97	3850.92		96	3797.73		95	0.84	U
Benzo(g,h,i)perylene	929.97		93	3781.88		93	3821.02		94	0.99	U
Surrogate Recoveries (%)											
Naphthalene-d8	67			60			59			78	
Phenanthrene-d10	78			83			83			87	

PCBs

CLIENT ID	NAV-OF14-SD45-FF		NAV-OF14-SD45-COMP	
Battelle ID	S5983-P		S5984-P	
Sample Type	SA		SA	
Collection Date	10/27/04		10/27/04	
Extraction Date	11/02/04		11/02/04	
Analysis Date	12/13/04		12/14/04	
Analytical Instrument	MS		MS	
% Moisture	NA		NA	
% Lipid	NA		NA	
Matrix	WATER		WATER	
Sample Size	1.64		2.63	
Size Unit-Basis	L_LIQUID		L_LIQUID	
Units	NG/L_LIQUID		NG/L_LIQUID	
Cl2(8)	0.11	UT	0.07	UT
Cl3(18)	0.13	UT	0.08	UT
Cl3(28)	0.13	UT	0.08	UT
Cl4(44)	3.63	JT	0.15	UT
Cl4(49)	0.23	UT	0.15	UT
Cl4(52)	6.26	T	2.37	JT
Cl4(66)	1.56	JT	0.69	JT
Cl4(77)	0.23	UT	0.14	UT
Cl5(87)	7.29	T	2.29	JT
Cl5(101)	11.76	T	4.58	T
Cl5(105)	5.4	T	1.97	JT
Cl5(114)	0.37	UT	0.23	UT
Cl5(118)	7.05	T	2.67	JT
Cl5(123)	0.13	UT	0.08	UT
Cl5(126)	0.19	UT	0.12	UT
Cl6(128)	0.43	UT	0.27	UT
Cl6(138)	8.92	T	4.03	T
Cl6(153)	10.3	T	4.96	T
Cl6(156)	0.12	UT	0.08	UT
Cl6(157)	0.23	UT	0.15	UT
Cl6(167)	0.43	UT	0.27	UT
Cl6(169)	0.18	UT	0.11	UT
Cl7(170)	1.88	JT	1.05	JT
Cl7(180)	2.23	JT	2.33	JT
Cl7(183)	0.71	JT	0.58	JT
Cl7(184)	0.3	UT	0.19	UT
Cl7(187)	1.13	JT	1.02	JT
Cl7(189)	0.13	UT	0.08	UT
Cl8(195)	0.58	UT	0.36	UT
Cl9(206)	0.54	UT	0.34	UT
Cl10(209)	0.65	UT	0.41	UT
Surrogate Recoveries (%)				
Cl2(14)	86		82	
Cl3(34)	90		82	

PCBs QA/QC (CONT.)

PROJECT: Task Order TO0016 – YO817 Stormwater FY04
PARAMETER: PCB
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 10/27/04. The samples were received at Battelle Duxbury on 10/29/04. Upon arrival, the cooler temperature was recorded at 1.7°C. No custody issues were noted. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0432, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PCB	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.09 – 0.53

METHOD: Water samples were extracted for PCB following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS). The method is based on key components of the PCB congener analysis approach described in EPA Method 1668A. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection. However, extracts were not analyzed within the 40-day holding time.

Batch	Extraction Date	Analysis Date
04-0432	11/2/04	12/13/04 – 12/14/04

BLANK: A procedural blank (PB) was prepared with the analytical batch. Blanks are analyzed to ensure the sample extraction and analysis methods were free of contamination.

04-0432 – No exceedences noted.

Comments – No target analytes were detected in sample BF358PB.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE:

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target PCB and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0432 – Eight percent recovery exceedences noted. All RPDs were within the laboratory limits specified by the client.

Comments – In sample S5983MS (background OF14-SD45-FF), PCB 126, PCB 169, PCB 180, PCB 206, and PCB 209 were all over-recovered at 127%, 121%, 125%, 129%, and 129%, respectively. In sample S5983MSD (same background), PCB 126, PCB 206, and PCB 209 were all over-recovered at 121%, 123%, and 124%, respectively. Chromatography and calculations were reviewed. No discrepancies were found. The exceedences have been qualified with an "N".

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Note: At the time of extraction, no certified second source material was available. In lieu of a certified second source, the SRM sample was generated by spiking target analyte solution into a clean seawater sample from Duxbury Bay. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None

SURROGATES:

Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0432 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

PCBs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE			MATRIX SPIKE-NAV-OF14-SD45-FF			MATRIX SPIKE DUPLICATE-NAV-OF14-SD45-FF			PROCEDURAL BLANK		STANDARD REFERENCE MATERIAL-041102-01: DUXBURY BAY WATER	
Battelle ID	BF359LCS-P			S5983MS-P			S5983MSD-P			BF358PB-P		BF360SRM-P	
Sample Type	LCS			MS			MSD			PB		LCS	
Collection Date	11/02/04			10/27/2004			10/27/2004			11/02/04		11/2/2004	
Extraction Date	11/02/04			11/2/2004			11/2/2004			11/02/04		11/2/2004	
Analysis Date	12/13/04			12/13/2004			12/14/2004			12/13/04		12/13/2004	
Analytical Instrument	MS			MS			MS			MS		MS	
% Moisture	NA			NA			NA			NA		NA	
% Lipid	NA			NA			NA			NA		NA	
Matrix	LIQUID			LIQUID			WATER			LIQUID		LIQUID	
Sample Size	2.00			0.5			0.5			2.00		2	
Size Unit-Basis	L_LIQUID			L_LIQUID			L_LIQUID			L_LIQUID		L_LIQUID	
Units	NG/L_LIQUID		% Recovery	NG/L_LIQUID		% Recovery	NG/L_LIQUID		% Recovery	NG/L_LIQUID		NG/L_LIQUID	
Cl2(8)	20.49	T	69	97.51	T	82	94.59	T	80	0.09	UT	22.4	T
Cl3(18)	22.04	T	73	102.38	T	85	100.85	T	84	0.11	UT	24.2	T
Cl3(28)	24.09	T	81	108.62	T	91	106.31	T	89	0.11	UT	26.88	T
Cl4(44)	23.45	T	79	119.95	T	98	116.81	T	95	0.19	UT	26.13	T
Cl4(49)	26.7	T	89	117.08	T	97	114.22	T	95	0.19	UT	29.56	T
Cl4(52)	20.68	T	70	109.85	T	87	105.83	T	84	0.19	UT	23.38	T
Cl4(66)	15.27	T	51	92.31	T	76	88.55	T	72	0.19	UT	17.41	T
Cl4(77)	16.25	T	54	106.02	T	88	102.53	T	85	0.18	UT	18.5	T
Cl5(87)	24.55	T	82	142.5	T	113	137.64	T	109	0.31	UT	27.32	T
Cl5(101)	22.85	T	76	130.88	T	99	125.23	T	95	0.31	UT	25.85	T
Cl5(105)	20.01	T	67	139.89	T	113	132.87	T	107	0.14	UT	23.1	T
Cl5(114)	0.31	UT		1.23	UT		1.23	UT		0.31	UT	0.31	UT
Cl5(118)	13.72	T	46	93.47	T	73	89.9	T	70	0.1	UT	15.75	T
Cl5(123)	0.11	UT		0.43	UT		0.43	UT		0.11	UT	0.11	UT
Cl5(126)	23.41	T	78	152.1	T	127	144.81	T	121	0.16	UT	26.58	T
Cl6(128)	19.08	T	64	120.73	T	101	118.1	T	99	0.35	UT	21.65	T
Cl6(138)	22.65	T	76	145.4	T	114	139.45	T	109	0.35	UT	25.61	T
Cl6(153)	21.22	T	71	142.62	T	111	136.45	T	106	0.35	UT	24.64	T
Cl6(156)	0.1	UT		0.4	UT		0.4	UT		0.1	UT	0.1	UT
Cl6(157)	0.19	UT		0.76	UT		0.76	UT		0.19	UT	0.19	UT
Cl6(167)	0.35	UT		1.42	UT		1.42	UT		0.35	UT	0.35	UT
Cl6(169)	21	T	70	145.22	T	121	136.65	T	113	0.15	UT	24.12	T
Cl7(170)	19.2	T	65	134.29	T	111	127.26	T	105	0.25	UT	21.2	T
Cl7(180)	27.25	T	91	152.23	T	125	140.92	T	115	0.14	UT	30.08	T
Cl7(183)	21.78	T	73	141.01	T	117	134.65	T	112	0.25	UT	25.63	T
Cl7(184)	21.41	T	71	131.49	T	109	123.6	T	103	0.25	UT	25	T
Cl7(187)	20.72	T	70	131.33	T	109	127.82	T	107	0.25	UT	23.03	T
Cl7(189)	0.11	UT		0.42	UT		0.42	UT		0.11	UT	0.11	UT
Cl8(195)	17.12	T	57	120.89	T	101	112.53	T	94	0.48	UT	19.35	T
Cl9(206)	22.47	T	76	153.55	T	129	146.21	T	123	0.44	UT	26.26	T
Cl10(209)	27.88	T	93	154.24	T	129	148.54	T	124	0.53	UT	31.68	T
Surrogate Recoveries (%)													
Cl2(14)	69			82			80			72		76	
Cl3(34)	68			82			80			68		77	

PESTICIDEs

CLIENT ID	NAV-OF14-SD45-FF		NAV-OF14-SD45-COMP	
Battelle ID	S5983-P		S5984-P	
Sample Type	SA		SA	
Collection Date	10/27/04		10/27/04	
Extraction Date	11/02/04		11/02/04	
Analysis Date	11/12/04		11/12/04	
Analytical Instrument	ECD		ECD	
% Moisture	NA		NA	
% Lipid	NA		NA	
Matrix	WATER		WATER	
Sample Size	1.64		2.63	
Size Unit-Basis	L_LIQUID		L_LIQUID	
Units	NG/L_LIQUID		NG/L_LIQUID	
2,4'-DDD	0.99	U	0.62	U
2,4'-DDE	0.84	U	0.52	U
2,4'-DDT	0.59	U	0.37	U
4,4'-DDD	1.16	U	1.49	
4,4'-DDE	1.62		1.1	
4,4'-DDT	4.12		0.45	U
TOTAL DDT MDL	9.32		4.55	
aldrin	0.48	U	0.3	U
a-chlordane	2.16		1.67	
g-chlordane	0.49	U	0.31	U
a-BHC	0.42	U	0.26	U
b-BHC	0.58	U	0.36	U
d-BHC	0.47	U	0.3	U
Lindane	0.6	U	1.49	
cis-nonachlor	0.79	U	0.49	U
trans-nonachlor	2.03		1.44	
oxychlordane	0.48	U	0.3	U
TCHLOR	2.65		1.98	
dieldrin	0.93	U	0.58	U
endosulfan I	0.33	U	0.21	U
endosulfan II	0.84	U	0.53	U
endosulfan sulfate	0.79	U	0.49	U
endrin	0.92	U	0.57	U
endrin aldehyde	1.03	U	0.65	U
endrin ketone	1.08	U	0.68	U
heptachlor	0.72	U	0.45	U
heptachlor epoxide	1.92	U	1.2	U
Hexachlorobenzene	1.01	U	0.63	U
methoxychlor	1.19	U	0.74	U
Mirex	0.75	U	0.47	U
Surrogate Recoveries *(%)*				
Cl2(14)	73		98	
Cl3(34)	75		86	
Cl5(104)	89		89	
Cl5(112)	100		94	

PESTICIDEs QA/QC

PROJECT: Task Order TO0016 – YO817 Stormwater FY04
PARAMETER: Pesticides
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 10/27/04. The samples were received at Battelle Duxbury on 10/29/04. Upon arrival, the cooler temperature was recorded at 1.7°C. No custody issues were noted. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0432, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PESTICIDE	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.27– 1.58

METHOD: Water samples were extracted for pesticide following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for pesticide analysis were solvent exchanged into hexane and analyzed using a gas chromatography/electron capture detector (GC/ECD). Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
04-0432	11/2/04	11/11/04 – 11/12/04

BLANK: A procedural blank (PB) was prepared with the analytical batch. Blanks are analyzed to ensure the sample extraction and analysis methods were free of contamination.

04-0432 – No exceedences noted.

Comments – No target analytes were detected in sample BF358PB.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with the analytical batch. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE:

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target pesticides and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq$ 30%).

Comments – None

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Note: At the time of extraction, no certified second source material was available. In lieu of a certified second source, the SRM sample was generated by spiking target analyte solution into a clean seawater sample from Duxbury Bay. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

04-0432 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None

SURROGATES:

Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0432 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

PESTICIDEs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE		MATRIX SPIKE-OF14-SD45-FF		MATRIX SPIKE DUPLICATE-OF14-SD45-FF		PROCEDURAL BLANK	
Battelle ID	BF359LCS-P		S5983MS-P		S5983MSD-P		BF358PB-P	
Sample Type	LCS		MS		MSD		PB	
Collection Date	11/02/04		10/27/2004		10/27/2004		11/02/04	
Extraction Date	11/02/04		11/2/2004		11/2/2004		11/02/04	
Analysis Date	11/11/04		11/12/2004		11/12/2004		11/11/04	
Analytical Instrument	ECD		ECD		ECD		ECD	
% Moisture	NA		NA		NA		NA	
% Lipid	NA		NA		NA		NA	
Matrix	LIQUID		LIQUID		WATER		LIQUID	
Sample Size	2.00		0.5		0.5		2.00	
Size Unit-Basis	L_LIQUID		L_LIQUID		L_LIQUID		L_LIQUID	
Units	NG/L_LIQUID	% Recovery	NG/L_LIQUID	% Recovery	NG/L_LIQUID	% Recovery	NG/L_LIQUID	
2,4'-DDD	28.43	95	121.16	101	121.34	101	0.81	U
2,4'-DDE	23.21	77	99.26	82	90.73	75	0.69	U
2,4'-DDT	22.43	75	83.59	70	95.01	79	0.48	U
4,4'-DDD	29.17	97	121.61	101	121.33	101	0.95	U
4,4'-DDE	26.95	90	113.95	94	115.18	95	0.68	U
4,4'-DDT	30.23	101	135.23	109	133.98	108	0.59	U
aldrin	27.37	91	110.02	92	110.67	92	0.4	U
a-chlordane	27.84	92	112.32	91	110.3	90	0.38	U
g-chlordane	25.73	86	103.51	86	104.21	87	0.4	U
a-BHC	16.43	55	72.56	60	72.04	60	0.34	U
b-BHC	28.2	94	113.26	94	114.96	96	0.47	U
d-BHC	28.61	95	116.88	97	118.22	98	0.39	U
Lindane	27.72	92	115.43	96	113.94	95	0.49	U
cis-nonachlor	28.59	95	118.57	99	119.95	100	0.65	U
trans-nonachlor	28.11	94	112.6	92	112.73	92	0.4	U
oxychlordane	29.09	97	117.48	98	118.69	99	0.39	U
dieldrin	28.97	97	116.9	97	121.34	101	0.76	U
endosulfan I	27.44	91	115.78	96	114.19	95	0.27	U
endosulfan II	23.95	80	108.3	90	113.41	94	0.69	U
endosulfan sulfate	28.91	96	128.84	107	127.41	106	0.65	U
endrin	29.12	97	127.89	107	131.53	110	0.75	U
endrin aldehyde	21.79	73	80.78	67	75.85	63	0.85	U
endrin ketone	28.78	96	120.09	100	119.23	99	0.89	U
heptachlor	25.63	85	109.66	91	107.2	89	0.59	U
heptachlor epoxide	27.44	91	107	89	109.04	91	1.58	U
Hexachlorobenzene	24.75	82	108.4	90	109.34	91	0.83	U
methoxychlor	30.38	101	134.06	112	129.9	108	0.98	U
Mirex	28.14	94	117.15	98	116.76	97	0.62	U
Surrogate Recoveries (%)								
Cl2(14)	76		83		95		86	
Cl3(34)	85		82		82		83	
Cl5(104)	81		85		81		88	
Cl5(112)	83		88		91		91	

TSS

SAMPLE ID	TSS (mg/L)
NAV-OF14-SD45-FF	61.24
NAV-OF14-SD45-COMP	78.73
NAV-OF14-SD45-COMP/BTL1	60.69
NAV-OF14-SD45-COMP/BTL2	44.97
NAV-OF14-SD45-COMP/BTL5	162.88
NAV-OF14-SD45-COMP/BTL11	46.78
NAV-BAY14-SD45-PRE	1.40
NAV- BAY14-SD45-DUR1	3.97
NAV-BAY14-SD45-DUR2	6.50
NAV-BAY14-SD45-DUR3	1.89
NAV-BAY14-SD45-DUR4	2.87
NAV-BAY14-SD45-AFT1	2.49
NAV-BAY14-SD45-AFT2	1.16
NAV-BAY14-SD45-AFT3	2.92

DOC

Sample ID	MEAN DOC (mg/L)
NAV-OF14-SD45-FF	11.73
NAV-OF14-SD45-COMP	6.00
NAV-BAY14-SD45-PRE	0.91
NAV-BAY14-SD45-DUR1	0.62
NAV-BAY14-SD45-DUR2	1.63
NAV-BAY14-SD45-DUR3	1.73
NAV-BAY14-SD45-DUR4	0.95
NAV-BAY14-SD45-AFT1	1.34
NAV-BAY14-SD45-AFT2	0.74
NAV-BAY14-SD45-AFT3	0.62

Appendix D2

SUB

SDB2- 2/24/2003
SDB3- 2/2/2004
SDB4- 10/17/2004

SDB2- 2/24/2003

METALS

MSL		Instrument:	GFAA		ICP-MS		FIAS		ICP-MS		ICP-MS		ICP-MS		CVAF		ICP-MS		ICP-MS		ICP-MS		FIAS		ICP-MS		ICP-MS	
Code	Rep	Sponsor I.D.	Ag		Al		As		Cd		Cr		Cu		Fe		Hg		Mn		Ni		Pb		Se		Sn		Zn	
1979-14		SUB-OF11B-SDB2-FF (T)	0.0563	J	3040		1.46		0.556		5.60		130		5770		0.0253		306		12.5		43.5		0.237		0.686		588	
1979-29		SUB-OF11B-SDB2-FF (D)	0.010	U	25.2	J	0.448	J	0.165		0.511	J	27.2		53.6		0.00979	J	44.8		7.49		0.575		0.276		0.136	J	139	

MSL		Instrument:	ICP-MS		ICP-MS		ICP-MS		ICP-MS		ICP-MS		ICP-MS		CVAF		ICP-MS		ICP-MS		ICP-MS		ICP-MS		ICP-MS		ICP-MS	
Code	Rep	Sponsor I.D.	Ag		Al		As		Cd		Cr		Cu		Fe		Hg		Mn		Ni		Pb		Se		Sn		Zn	
1979-15		SUB-OF24-SDB2-FF (T)	0.0949	J	453		1.24		1.26		3.44		129		751		0.00679	J	22.6		6.58		9.85		0.301		0.521		267	
1979-30		SUB-OF24-SDB2-FF (D)	0.0165	J	32.9	J	1.13		0.645		1.16		75.1		33.6		0.00342	J	11.0		3.30		0.370		0.255		0.0646	J	179	
1979-13		SUB-OF26-SDB2-FF (T)	0.152	J	459		1.23		1.08		6.23		116		750		0.00666	J	30.7		16.6		14.3		0.261		0.444	J	248	
1979-28		SUB-OF26-SDB2-FF (D)	0.0140	J	18.6	J	1.14		0.472		1.59		61.9		15.3		0.00355	J	12.4		11.8		0.184		0.0991	U	0.0386	J	88.2	

METALS QA/QC

PROJECT: SPAWARS Task 11, San Diego Bay Stormwater
PARAMETER: Metals
LABORATORY: Battelle Marine Sciences Laboratory, Sequim, Washington
MATRIX: Seawater and Freshwater

SAMPLE CUSTODY AND PROCESSING: Eighteen seawater and twelve freshwater samples were received in on 03/03/03. All samples were received in good condition (i.e., all sample containers were intact). Samples were assigned a Battelle Central File (CF) identification number (1979) and were entered into Battelle's sample tracking system.

The following lists information on sample receipt and processing activities:

Chemistry Lab ID	1979-1 through –30
Collection dates	02/25/03
Laboratory arrival dates	03/03/03
Cooler temperatures, on arrival	NA – Samples arrived preserved
Fe/Pd Preconcentration (seawater)	03/14/03
FIAS (As – seawater)	03/14/03
FIAS (Se – seawater)	03/17/03
GFAA (Ag – seawater)	03/20/03
CVAA analyses (Hg)	03/13/03, 03/14/03, 03/18/03
ICP-MS analyses:	
Fe/Pd Seawater (Cd, Cr, Cu, Ni, Pb)	03/18/03
Direct Seawater (Al, Fe, Mn, Sn, Zn)	03/27/03
Freshwater (Ag, Al, As, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Se, Sn, Zn)	03/24/03
Rerun Freshwater (Al, Fe)	04/11/03

QA/QC DATA QUALITY OBJECTIVES:

Analyte	Analytical Method Seawater	Analytical Method Freshwater	Range of Recovery	SRM Accuracy	Relative Precision	Detection Limits (µg/L)		
						Target MDL [1]	Achieved MDL Seawater[2]	Achieved MDL Freshwater [2]
Silver	GFAA	ICP-MS	50-150%	≤20%	≤50%	0.50	0.010	0.0038
Aluminum	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	50.0	0.823	0.823
Arsenic	FIAS	ICP-MS	50-150%	≤20%	≤30%	0.50	0.0275	0.0087
Cadmium	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.05	0.0094	0.0008
Chromium	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	1.00	1.00	0.024
Copper	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.05	0.05	0.0029
Iron	ICP-MS	ICP-MS	50-150%	≤20%	≤50%	10.0	0.983	0.983
Mercury	CVAA	CVAA	50-150%	≤25%	≤30%	0.01	0.00014	0.00014
Manganese	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.50	0.50	0.003
Nickel	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.05	0.05	0.0114
Lead	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.05	0.0035	0.0044
Selenium	FIAS	ICP-MS	50-150%	≤20%	≤30%	0.20	0.0352	0.0991
Tin	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.50	0.0024	0.0024
Zinc	ICP-MS	ICP-MS	50-150%	≤20%	≤30%	0.50	0.50	0.0493

(1) As stated in the Statement of Work for Chemical Analysis of Marine and Estuarine Samples 15 May 2001.
(2) Reported from the 2003 MDL study.

METHODS:

Battelle MSL analyzed both seawater and freshwater samples for fourteen metals: silver (Ag), aluminum (Al), arsenic (As), cadmium (Cd), chromium (Cr) copper (Cu), iron (Fe), mercury (Hg), manganese (Mn), nickel (Ni), lead (Pb), selenium (Se), tin (Sn) and zinc (Zn). The samples were submitted for analyses by four analytical methods: GFAA, ICP-MS, FIAS and CVAA.

Seawater samples were preconcentrated using iron (Fe) and palladium (Pd) in accordance with the Battelle SOP MSL-I-025, *Methods of Sample Preconcentration*, which is derived from EPA Method 1640. The sample preconcentration was submitted for analysis by ICP-MS and GFAA.

Seawater samples were analyzed by inductively coupled plasma-mass spectrometry (ICP-MS) in accordance with Battelle SOP MSL-I-022, *Determination of Elements in Aqueous and Digestate Samples by ICP-MS.* This method is based on two EPA Methods: 200.8 and 1638. Analytes reported from the preconcentrated seawater samples include: Cd, Cr, Cu, Ni, and Pb. Analytes reported from the direct analysis of the seawater samples include: Al, Fe, Mn, Sn, and Zn. Freshwater samples were analyzed directly by ICP-MS for all analytes, except Hg.

Ag was analyzed in the Fe-Pd preconcentrate by graphite furnace atomic absorption (GFAA) following Battelle SOP MSL-I-029, *Determination of Metals in Aqueous and Digestate Samples by GFAA*, which is derived from EPA Method 200.9.

Seawater samples were analyzed by hydride generation flow injection atomic spectroscopy (FIAS) for As and Se according to Battelle SOP MSL-I-030 *Determination of Metals in Aqueous and Digestate Samples by HGAA-FIAS.*

Seawater and freshwater samples were analyzed by cold-vapor atomic fluorescence spectroscopy (CVAF) for Hg according to Battelle SOP MSL-I-013, *Total Mercury in Aqueous Samples by CVAF*, which is derived from EPA Method 1631.

All results are reported in units of µg/L.

HOLDING TIMES:

The holding times for metals analyses are 90 days from sample collection for Hg analysis, and 6 months from sample collection for analysis of all other metals. The holding times for all metals were achieved.

DETECTION LIMITS:

Target detection limits (TDL) were achieved for all analytes. Achieved method detection limits are reported from the 2003 MDL study. Sample concentrations were evaluated and flagged to the following criteria:

- U Analyte not detected at or above the detection limit, MDL reported
- J Analyte detected above MDL, but below TDL
- * Duplicate out of QC criteria
- e SRM recovery out of QC criteria
- w Spike recovery out of QC criteria due to inappropriate spiking level
- # Continuing calibration recovered outside of acceptable method criteria

NOTE ON Hg QA/QC SAMPLES:

Seawater and freshwater samples were analyzed concurrently for Hg. The QC samples are reported in both the seawater and freshwater tables.

METHOD BLANKS:

Seawater: A minimum of one method blank was analyzed with each analysis batch. Metals concentrations in the method blanks were below the TDL, with the exception of one method blank for Ni and Cu. All sample concentrations for Ni and Cu are greater than five times the detected blank. No corrective action was required. The data were not blank-corrected.

Freshwater: A minimum of one method blank was analyzed with each analysis batch. All metals concentrations in the method blanks were below the TDL. The data were not blank-corrected.

BLANK SPIKE or OPR ACCURACY:

Seawater: A minimum of one blank spike or on-going precision and recovery (OPR) sample was analyzed with each analysis batch. Recoveries were reported for spikes at approximate concentrations of 0.005 µg/L for Hg; 5 µg/L for As and Se; and 10 µg/L for Ag, Cd, Cr, Cu, Ni, and Pb. BS recoveries among all metals analyzed ranged from 82% to 107% and were within the QC acceptance criteria of 50% to 150% for all metals.

Freshwater: A minimum of one blank spike or on-going precision and recovery (OPR) sample was analyzed with each analysis batch. Recoveries were reported for spikes at approximate concentrations of 0.005 µg/L for Hg; 10 µg/L for Cr, Mn, Ni, Cu, Zn, As, Se, Ag, Cd, Sn, and Pb; and 100 µg/L for Al and Fe. BS recoveries among all metals analyzed ranged from 91% to 119% and were within the QC acceptance criteria of 50% to 150% for all metals.

MATRIX SPIKE ACCURACY:

Seawater: A minimum of one matrix spike was analyzed with each analysis batch. Recoveries were reported for spikes at approximate concentrations of 0.01 µg/L for Hg; 5 µg/L for As and Se; 10 µg/L for Cr, Ni, Cu, Ag, Cd, Sn, and Pb; and 100 µg/L for Al, Fe, Mn and Zn. Matrix spike recoveries among all metals analyzed ranged from 83% to 117% and were within the QC acceptance criteria of 50% to 150% for all metals, with the exception of one MS for Al (240%) and two replicates for Fe (0%, 220%). Low recoveries for the matrix spikes are due to an inappropriate spiking level relative to the native sample concentration. Spiking levels were less than 10% of the native sample concentration, therefore not appropriate for evaluating matrix spike accuracy. Acceptable MS accuracy for Al and Fe was demonstrated in the alternate matrix spike samples.

Freshwater: A minimum of one matrix spike was analyzed with each analysis batch. Recoveries were reported for spikes at approximate concentrations of 0.01 µg/L for Hg; 10 µg/L for Cr, Mn, Ni, Cu, As, Se, Ag, Cd, Sn, and Pb; and 100 µg/L for Al, Fe and Zn. Matrix spike recoveries among all metals analyzed ranged from 94% to 118% and were within the QC acceptance criteria of 50% to 150% for all metals.

REPLICATE PRECISION:

Analytical precision for each analysis batch was evaluated by the analysis of laboratory duplicates and expressed as the relative percent deviation (RPD) of duplicate results.

Seawater: A minimum of one set of laboratory duplicates was analyzed with each analysis batch. Precision for all metals, except Fe, ranged from 0% to 18% RPD and were within the QC limits of ≤ 30%. RPD values for Fe were 9% and 32% and were within the QC limits of ≤ 50%.

Freshwater: A minimum of one set of laboratory duplicates was analyzed with each analysis batch. Precision for all metals ranged from 1% to 19% RPD and

were within the QC limits of ≤ 30%.

STANDARD REFERENCE MATERIAL ACCURACY:

Accuracy of recovery of SRM analytes was expressed as the percent difference (PD) between the measured and certified SRM concentrations. The target QC criterion is ≤20% PD.

Seawater: Standard reference material analyzed for seawater samples include: SRM 1640, SRM CASS-4, and SRM 1641 for Hg. The SRM 1640 is not certified for Sn and the certified value for Fe in not at a level appropriate for data quality evaluation. Percent differences for SRM 1640 and SRM 1641 ranged from 0% to 17% and were within the QC criterion.

The SRM CASS-4 is a low-level seawater reference material. Analytes of interest certified in CASS-4 are less than 10 times the laboratory achieved MDL for all metals except Cu. Currently, there is not seawater SRM certified at a practical quantification level for all analytes of interest. The SRM CASS-4 was analyzed with the preconcentrated seawater samples, and applies only to the metals obtained from this method (Ag, Cr, Ni, Cu, Cd, Pb). Percent differences for analytes within the QC criteria for CASS-4 include As (9%) and Cd (15%). The required preconcentration procedure for low level seawater samples includes the addition of chelating agents to induce precipitation of metals under specific conditions. Subsequently, reagents added to the samples should be of the purest quality to result in zero addition of metals to the samples. The current reagents available contain traces of Cr, Cu and Ni. Correcting CASS-4 results for reagent contributions provide PD values within the QC criterion for Cr (9%), Ni (2%), and Cu (1%). Since CASS-4 is not certified for Ag or Hg and is not certified at practical levels for a majority of the analytes of interest, the alternate SRM (1640 or 1641, respectively) should be used to evaluate the accuracy of this data set. The data were not blank corrected, as the sample concentrations are greater than five times the detected blank for these analytes.

Freshwater: Standard reference material analyzed for freshwater samples include: SRM 1640, SLRS-3 for Fe, and SRM 1641 for Hg. The SRM 1640 is not certified for Sn and the certified value for Fe in not at a level appropriate for data quality evaluation. Percent differences for all SRMs ranged from 0% to 19% and were within the QC acceptance criterion for all metals, with the following exceptions. One replicate of 1640 for Se (28%) and one replicate of 1640 for Zn (21%). In both cases, an alternate replicate of SRM 1640 was analyzed within the batch, which demonstrated acceptable accuracy for Se (0% PD) and Zn (3% PD).

METALS QA/QC (CONT.)

MSL		Instrument:	GFAA	ICP-MS	FIAS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF	ICP-MS	ICP-MS	ICP-MS	FIAS	ICP-MS	ICP-MS
Code	Rep	Sponsor I.D.	Ag	Al	As	Cd	Cr	Cu	Fe	Hg	Mn	Ni	Pb	Se	Sn	Zn
METHOD BLANK																
blk TRM r1		ICP-MS Direct	NA	0.823 U	NA	NA	NA	NA	0.983 U	NA	0.003 U	NA	NA	NA	0.00578 J	0.5 U
blk TRM r2		ICP-MS Direct	NA	0.823 U	NA	NA	NA	NA	0.983 U	NA	0.003 U	NA	NA	NA	0.00754 J	0.5 U
1979-blk		Fe/Pd ICP-MS or GFAA-Ag	0.0174 J	NA	NA	0.0094 U	0.0913 J	0.151	NA	NA	NA	0.105	0.0203 J	NA	NA	NA
Method Blank		Hg- 03/13/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
Method Blank		Hg- 03/14/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
Method Blank		Hg- 03/18/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
BLANK		FIAS - As	NA	NA	0.0275 U	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
BLANK		FIAS - Se	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.0352 U	NA	NA
METHOD DETECTION LIMIT [1]			0.010	0.823	0.0275	0.0094	0.0218	0.0540	0.983	0.00014	0.003	0.0286	0.0035	0.0352	0.0024	0.5
Project Target Detection Limit			0.50	50.0	0.50	0.05	1.00	0.05	10.0	0.01	0.50	0.05	0.05	0.20	0.50	0.50
STANDARD REFERENCE MATERIAL																
1640 Direct		ICP-MS Direct	NA	51.8	NA	NA	NA	NA	N/A	NA	123	NA	NA	NA	1.58	62.3
1640 TRM		ICP-MS Direct	NA	48.8	NA	NA	NA	NA	N/A	NA	117	NA	NA	NA	1.52	50.6
1640 Direct		Fe/Pd ICP-MS or GFAA-Ag	6.96	NA	NA	24.7	39.4	87.2	N/A	NA	NA	28.2	28.0	NA	NA	NA
1640 Direct		Fe/Pd ICP-MS or GFAA-Ag	NA	NA	NA	24.5	37.1	83.0	N/A	NA	NA	26.7	27.3	NA	NA	NA
1641 Direct		FIAS- Se	NA	NA	NA	NA	NA	NA	N/A	NA	NA	NA	NA	21.0	NA	NA
1640		certified value	7.6	52.0	26.7	22.8	38.6	85.2	34.3	NC	122	27.4	27.9	22.0	NC	53.2
1640		range	±0.25	±1.5	±0.73	±0.96	±1.6	±1.2	±1.6	NC	±1.1	±0.8	±0.14	±0.51	NC	±1.1
		% difference	NA	0%	NA	NA	NA	NA	N/A	NA	1%	NA	NA	NA	NA	17%
		% difference	NA	6%	NA	NA	NA	NA	N/A	NA	4%	NA	NA	NA	NA	5%
			9%	NA	NA	8%	2%	2%	N/A	NA	NA	3%	0%	NA	NA	NA
			NA	NA	NA	8%	4%	3%	N/A	NA	NA	3%	2%	NA	NA	NA
			NA	NA	NA	NA	NA	NA	N/A	NA	NA	NA	NA	4%	NA	NA
1979-cass4		Fe/Pd ICP-MS or GFAA-Ag	0.0369	N/A	N/A	0.0299	0.222	0.749	N/A	N/A	N/A	0.425	0.0265	N/A	N/A	N/A
CASS-4		FIAS - As	N/A	N/A	1.01	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
CASS-4		certified value	NC	NC	1.11	0.026	0.144	0.592	0.71	N/A	2.78	0.314	0.0098	NC	NC	0.381
CASS-4		range	NC	NC	±0.16	±0.003	±0.029	±0.055	±0.058	N/A	±0.19	±0.030	±0.0036	NC	NC	±0.057
		% difference	N/A	N/A	N/A	15%	54% e	27% e	N/A	N/A	N/A	35% e	170% e	N/A	N/A	N/A
		% difference	N/A	N/A	9%	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
1641d031203		Hg	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1565	N/A	N/A	N/A	N/A	N/A	N/A
1641d031303		Hg	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1466	N/A	N/A	N/A	N/A	N/A	N/A
1641d031703		Hg	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1573	N/A	N/A	N/A	N/A	N/A	N/A
1641d		certified value	NC	NC	NC	NC	NC	NC	NC	1590	NC	NC	NC	NC	NC	NC
1641d		range	NC	NC	NC	NC	NC	NC	NC	±4.00	NC	NC	NC	NC	NC	NC
		% difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	2%	N/A	N/A	N/A	N/A	N/A	N/A
		% difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	8%	N/A	N/A	N/A	N/A	N/A	N/A
		% difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1%	N/A	N/A	N/A	N/A	N/A	N/A
ICV,CCV RESULTS																
ICV		ICP-MS Direct or Hg		101%					109%	93%	104%				108%	105%
CCV		ICP-MS Direct or Hg		98%					99%	100%	105%				104%	105%
CCV		ICP-MS Direct or Hg		106%					113%	98%	104%				105%	107%
CCV		ICP-MS Direct or Hg		101%					101%	101%	104%				101%	100%
CCV		ICP-MS Direct or Hg		107%					112%	94%	100%				101%	98%
ICV		Fe/Pd ICP-MS or Hg	102%			102%	103%	102%		94%		102%	100%			
CCV		Fe/Pd ICP-MS or Hg	104%			100%	99%	98%		92%		97%	102%			
CCV		Fe/Pd ICP-MS or Hg	101%			101%	98%	96%		94%		97%	105%			
CCV		Fe/Pd ICP-MS or Hg	101%			100%	98%	96%		97%		96%	102%			
CCV		Fe/Pd ICP-MS	N/A			99%	96%	95%		NA		94%	100%			
ICV		FIAS-As or Hg			103%					100%						
CCV		FIAS-As or Hg			100%					101%						
CCV		FIAS-As or Hg			98%					103%						
CCV		FIAS-As or Hg			99%					98%						
ICV		FIAS-Se												104%		
CCV		FIAS-Se												100%		
CCV		FIAS-Se												99%		
CCV		FIAS-Se												95%		

METALS QA/QC (CONT.)

MSL		Instrument:	GFAA	ICP-MS	FIAS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF	ICP-MS	ICP-MS	ICP-MS	FIAS	ICP-MS	ICP-MS
Code	Rep	Sponsor I.D.	Ag	Al	As	Cd	Cr	Cu	Fe	Hg	Mn	Ni	Pb	Se	Sn	Zn
BLANK SPIKE RESULTS																
		Amount Spiked	10	NS	NS	10	10	10	NS	0.00497	NS	10	10	NS	NS	NS
1979-SB Blk or			0.0246 J			0.0721	0.180 J	0.488		0.000419 J		0.475	0.0279 J			
1979-SB LCS or			9.30			8.94	9.54	9.21		0.00569		9.19	8.26			
		Amount Recovered	9.28	N/A	N/A	8.94	9.36	8.72	N/A	0.00527	N/A	8.72	8.23	N/A	N/A	N/A
		Percent Recovery	93%	N/A	N/A	89%	94%	87%	N/A	106%	N/A	87%	82%	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.00497	NS	NS	NS	NS	NS	NS
BLANK031203										0.000419 J						
OPR031203run2										0.00545						
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.00503	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	101%	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.00487	NS	NS	NS	NS	NS	NS
BLANK031303										0.000172 J						
OPR031303run1										0.00490						
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.00473	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	97%	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.00487	NS	NS	NS	NS	NS	NS
BLANK031303										0.000172 J						
OPR031303run2										0.00502						
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.00485	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	100%	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	5	NS	NS	NS	NS	0.00491	NS	NS	NS	NS	NS	NS
BLANK (FIAS					0.0275 U					0.000202 J						
LCS or					5.14					0.00528						
		Amount Recovered	N/A	N/A	5.14	N/A	N/A	N/A	N/A	0.00508	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	103%	N/A	N/A	N/A	N/A	103%	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.00491	NS	NS	NS	5.0	NS	NS
BLANK(FIAS)										0.000202 J				0.0352 U		
LCS or										0.00547				4.92		
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.00527	N/A	N/A	N/A	4.92	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	107%	N/A	N/A	N/A	98%	N/A	N/A
MATRIX SPIKE RESULTS																
		Amount Spiked	NS	100	NS	NS	NS	NS	100	NS	100	NS	NS	NS	10	100
1979-1		NAV-OF9-SDB2-FF		1840					2390		92.6				1.00	433
		MS		1920					2360		192				9.29	534
		Amount Recovered	N/A	80.0	N/A	N/A	N/A	N/A	-30	N/A	99.4	N/A	N/A	N/A	8.29	101
		Percent Recovery	N/A	80%	N/A	N/A	N/A	N/A	0% w	N/A	99%	N/A	N/A	N/A	83%	101%
		Amount Spiked	NS	100	NS	NS	NS	NS	100	NS	100	NS	NS	NS	10	100
1979-1		NAV-OF9-SDB2-FF		1840					2390		92.6				1.00	433
		MSD		2080					2610		189				9.70	540
		Amount Recovered	N/A	240	N/A	N/A	N/A	N/A	220	N/A	96.4	N/A	N/A	N/A	8.70	107
		Percent Recovery	N/A	240% w	N/A	N/A	N/A	N/A	220% w	N/A	96%	N/A	N/A	N/A	87%	107%
		Amount Spiked	NS	100	5.0	NS	NS	NS	100	NS	100	NS	NS	5.0	10	100
1979-16		NAV-OF9-SDB2-FF		16.6 J	0.695				18.5		28.7			0.132 J	0.165 J	218
		MS		119	5.38				104		138			4.80	10.6	335
		Amount Recovered	N/A	102	4.69	N/A	N/A	N/A	85.5	N/A	109	N/A	N/A	4.67	10.4	117
		Percent Recovery	N/A	102%	94%	N/A	N/A	N/A	86%	N/A	109%	N/A	N/A	93%	104%	117%
		Amount Spiked	NS	100	NS	NS	NS	NS	100	NS	100	NS	NS	NS	10	100
1979-16		NAV-OF9-SDB2-FF		16.6 J					18.5		28.7				0.165 J	218
		MSD		125					102		133				10.5	328
		Amount Recovered	N/A	108	N/A	N/A	N/A	N/A	83.5	N/A	104	N/A	N/A	N/A	10.3	110
		Percent Recovery	N/A	108%	N/A	N/A	N/A	N/A	84%	N/A	104%	N/A	N/A	N/A	103%	110%
		Amount Spiked	10	NS	NS	10	10	10	NS	NS	NS	10	10	NS	NS	NS
1979-18		NAV-OF14-SDB2-FF	0.0267 J			0.983	0.804 J	22.1				5.78	0.916			
		MS	9.90			9.46	9.46	30.3				14.6	9.47			
		Amount Recovered	9.87	N/A	N/A	8.48	8.66	8.20	N/A	N/A	N/A	8.82	8.55	N/A	N/A	N/A
		Percent Recovery	99%	N/A	N/A	85%	87%	82%	N/A	N/A	N/A	88%	86%	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.0158	NS	NS	NS	NS	NS	NS
1979-12		NAV-BAY14-SDB2-D								0.00229 J						
		MS								0.0188						
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.0165	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	104%	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	0.0155	NS	NS	NS	NS	NS	NS
1979-12		NAV-BAY14-SDB2-D								0.00229 J						
		MSD								0.01820						
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.0159	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	103%	N/A	N/A	N/A	N/A	N/A	N/A

METALS QA/QC (CONT.)

MSL		Instrument	GFAA	ICP-MS	FIAS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF	ICP-MS	ICP-MS	ICP-MS	FIAS	ICP-MS
Code	Rep	Sponsor I.D.	Ag	Al	As	Cd	Cr	Cu	Fe	Hg	Mn	Ni	Pb	Se	Sn
REPLICATE RESULTS															
1979-1		NAV-OF9-SDB2-FF	0.168 J	1840	1.58	0.987	6.56	54.2	2390	0.0173	92.6	12.5	22.7	0.187 J	1.00
1979-1	2	NAV-OF9-SDB2-FF	NA	1790	NA	NA	NA	NA	2180	0.0174	87.8	NA	NA	NA	1.02
		% difference	NA	3%	NA	NA	NA	NA	9%	1%	5%	NA	NA	NA	2%
1979-12		NAV-BAY14-SDB2-D	0.0324 J	107	1.17	0.109	1.75	5.01	152	0.00230 J	12.5	1.93	0.623	0.0539 J	0.253 J
1979-12	2	NAVBAY14-SDB2-D	0.0388 J	NA	1.20	0.113	1.74	4.99	NA	NA	NA	1.94	0.602	0.0352 U	NA
		% difference	18%	NA	3%	4%	1%	0%	NA	NA	NA	1%	3%	NA	NA
1979-16		OF9-SDB2-FF	0.0203 J	16.6 J	0.695	0.388	1.22	25.8	18.5	0.00367 J	28.7	6.95	0.369	0.132 J	0.165 J
1979-16	2	OF9-SDB2-FF	NA	16.4 J	NA	NA	NA	NA	25.5	NA	29.9	NA	NA	NA	0.149 J
		% difference	NA	1%	NA	NA	NA	NA	32%	NA	4%	NA	NA	NA	10%

(1)= Fe/Pd MDL Study, Ag from Graphite Furnace report, and Hg from 2002 MDL Study; NC = Analyte not certified; NS= Analyte not spike; # = Data quality outside the accuracy criteria of ±20% or precision/MS recovery criteria of ±25%; U= Analyte not detected above the laboratory achieved MDL, which is reported; J = Analyte detected above the MDL, but below the reporting limit.

3.3 Phase II/III TIE Evaluation

3.3.1 Species Sensitivity to Toxicants Identified

The absolute and relative sensitivity of the three species tested to various constituents of concern based on classes of compounds identified during the Phase I TIE characterization phase provides further evidence as to the causes of toxicity in the stormwater samples. Mussel larvae were clearly the most sensitive species tested, with adverse effects observed at concentrations as low as 12 percent sample. Based on the survival endpoint, mysid shrimp and topsmelt were similar in sensitivity, but both were less sensitive than mussel larvae.

Cationic trace metals and surfactants were identified as the classes of compounds responsible for observed toxicity during Phase I TIE testing. A summary of analytical results for total and dissolved trace metals in stormwater outfall samples is provided in Table 4. A review of metal concentrations in the samples and available toxicity data identified only copper and zinc as the most likely causes of toxicity attributable to divalent metals in any of the stormwater samples tested. Copper and zinc are the only two metals that exceeded EPA water quality criteria for the protection of aquatic marine life in any of the samples tested (EPA 2002b). For comparison, concentrations of copper and zinc (both pre- and post-C_{18} extraction) and toxicity values for each of the three species tested in laboratory dilution water at Nautilus are shown in Figure 12. For both metals, mussels were clearly more sensitive; mysids and topsmelt were similar.

Surfactant concentrations in screening samples over time and post aeration are shown in Figure 13.

A summary of available EC_{50}/LC_{50} point estimate values for the primary toxicants of concern identified is provided in Table 5 for all species tested. Due to the limited zinc and surfactant data available for mussels and topsmelt, a summary of EC_{50}/LC_{50} point estimate values for a few closely related species is also provided in Table 6.

The following results for trace metals focus on the dissolved fraction, as it is well-documented that this fraction, rather than total, is much more closely associated with biological effects (Bergman and Dorward-King 1997)

Copper

Mussel larvae are clearly the most sensitive of the three species to copper; our long-term mean EC_{50} for this metal (n=20) is 9.5 µg/L, which can be compared with long-term average LC_{50} values of 163 µg/L and 233 µg/L for 96-hour topsmelt and mysid shrimp exposures, respectively. Published mean 48- to 96-hour EC_{50} literature values for *M. galloprovincialis* are similar to values obtained at Nautilus, ranging from 5.8 µg/L (Martin et al. 1981) to 7.9 (EPA 1998). Published acute 96-hour LC_{50} values for mysids are slightly less than those derived at Nautilus at 153 to 181 µg/L copper (Lussier et al. 1985 and Cripes 1994), while published values for topsmelt are slightly greater at 288 to 365 µg/L (Anderson et al. 1991 and McNulty et al. 1994). Thus, given the range of dissolved copper concentrations in the samples (83 to 212 µg/L), mussels would have exhibited the greatest response to copper, with much lower responses exhibited by mysids or topsmelt. Topsmelt, however, exhibited no toxicity in Sample NAB OF 9, which had the highest copper concentration among the stormwater samples tested, but mysids showed improvement in survival when this sample was treated with EDTA.

Based on the amount of data generated at Nautilus for copper, and because TIE procedures performed during this study using the same dilution water with methods that are consistent with our standard reference toxicant procedures, the following toxic unit (TU) values were calculated using sensitivity data derived at Nautilus.

In order to apply these general sensitivity guidelines more directly to the samples tested, predicted TUs based on metal concentrations in the samples were calculated (TU = metal concentration in the sample/ EC or LC_{50} values derived at Nautilus)

Predicted TU values based on copper concentrations range from 6.4 to 16.3 among the three toxic samples for the bivalve embryo development test (Table 7). Predicted TU values based on copper for mysids and topsmelt range from 0.3 to 0.7 and 0.5 to 1.3, respectively among the three toxic samples (Tables 8 and 9). Based on these TU values, and the observation that topsmelt exhibited no toxicity in the sample with the highest copper concentration (NAB OF 9), copper does not appear to be primarily responsible for observed toxicity to either mysids or topsmelt in any of the samples tested. It is possible, however, that copper may still contribute to zinc toxicity through additivity.

Zinc

Similar to copper, mussels were again the most sensitive species to zinc, with an EC_{50} of 159 µg/L determined previously at Nautilus. This is similar to values published in the literature for this species and endpoint; 175 µg/L by Martin et al. (1981) and 178 µg/L by Phillips (2000). Mysid shrimp and topsmelt are both much less sensitive; we determined that the 96-hour LC_{50} values for *Americamysis bahia* and *Atherinops affinis* were 647 and 880 µg/L, respectively. A previous mysid test at Nautilus resulted in an acute LC_{50} value of 448 µg/L. Published mysid 96-hour LC_{50} estimates for zinc range from approximately 303 to 547 µg/L, with most of the values approaching 500 µg/L (Lussier et al. 1985 and Cripe 1994). Zinc toxicity data was not found in the literature for topsmelt. For similar reasons mentioned in the prior section for copper (dilution water and test method consistency), the following TU values were performed using values derived at Nautilus for all three test species.

The concentration of zinc in NASNI OF 23a, at 297 µg/L, is enough to potentially cause toxicity to bivalve larvae (TU = 1.7), but not great enough to cause toxicity to either mysids or topsmelt (Table 7). Concentrations of zinc, at 985 and 742 µg/L in and NAB OF 18 and NAB OF 9, respectively, are greater than those expected to cause toxicity to all three test species with zinc TU values of 4.2 and 5.6 for bivalves, 1.2 and 1.5 for mysids, and 0.8 to 1.1 for topsmelt (Tables 7 through 9). Zinc TUs for mussels, although elevated, were two to four times less than those for copper, thus indicating that zinc appears to contribute a lesser proportion of toxicity to bivalves than copper. The additivity of these two metals, however, suggests that both could be contributing to observed toxicity to bivalve larvae in all three toxic samples. On the contrary, TU data indicate that zinc, rather than copper, is more likely to be responsible for toxicity to mysids in Sample NAB OF 9. Despite zinc TU values of 1.1 to 1.5 in NAB OF 18 for topsmelt and mysids, EDTA failed to reduce toxicity, indicating that cationic trace metals were not responsible for toxicity to these two species in this sample.

Trace Metal Reduction by C_{18} Extraction

The conclusion that divalent cationic metals are contributing to toxicity is based on the effectiveness of EDTA in removing toxicity. While reduction of toxicity following extraction with SPE C_{18} columns is generally attributed to the presence of non-polar organic toxicants, metals concentrations can also be reduced by C_{18} extraction (USEPA 1991). Therefore, the ability of SPE columns to remove some metals requires that the presence of an organic constituent be further confirmed by: 1) a comparative lack of effect of EDTA; and/ or 2) toxicity in the solvent

elution of the SPE column.

To evaluate the potential reduction in trace metal toxicity in this study due to the C_{18} extraction procedure, the concentration of trace metals was measured both prior to and after C_{18} treatment in Samples NAB OF 18 and NASNI OF23a. These data are presented in Figure 12 for copper and zinc. Copper was reduced 34 percent in NAB OF 18 and 38 percent in NASNI OF 23a by the SPE C_{18} procedure. Zinc was reduced 17 percent in NAB OF 18, but not reduced in sample NASNI OF 23a by the SPE C_{18} procedure. These results indicate that while the C_{18} extraction may have reduced toxicity due to cationic metals, however, the degree of reduction likely had little bearing on the overall Phase I TIE results and interpretation as confirmation of additional toxicity due to organics was performed by 1) testing methanol elutions from the columns, and 2) performing combined EDTA + C_{18} treatments.

Surfactants

Concentrations of surfactants, measured as MBAS, were 1.0, 1.9 and 1.1 mg/L in NAB OF 9, NAB OF 18, and NASNI OF 23a, respectively. Non-toxic NASNI OF 26 had an MBAS concentration of 0.47 mg/L.

Published surfactant toxicity values for all three tests species is very limited. Surfactants, measured as MBAS, include anionic forms; nonionic surfactants, such as ethoxylates and nonyl phenol, are not captured by this method. Due to the limited data currently available, and the wide range of chemicals with surfactant properties, a summary of available toxicity data for both anionic and nonionic surfactants is presented in Table 7 for the three species tested. A search for closely related fish and bivalve species was also performed, with published toxicity values summarized in Table 6.

Published anionic surfactant values for *Mytilus galloprovincialis* range from 0.3 to 50 mg/L (Grammo 1972, Grammo et al. 1989, and Swedmark et al. 1971), and a single LAS surfactant EC_{50} value of 0.46 mg/L was published by Cardwell et al. (1979) for embryo development of the Pacific oyster. Published anionic surfactant toxicity data, however, was not found for *Americamysis bahia*, *Atherinops affinis*, or closely related species.

In summary, the range of published toxicity values for surfactants (anionic and nonionic) varies widely depending on both the specific type of surfactant tested and species. Sufficient side-by-side testing has not been performed to determine whether there are general sensitivity trends for the three marine species tested in this study. Some anionic surfactant toxicity values for the

bivalves *M. galloprovincialis* and *C. gigas*, are below MBAS concentrations measured in all stormwater outfall samples, thus providing evidence that surfactants may be of concern based on concentration alone. Prior experience at Nautilus has frequently identified toxicity due to anionic surfactants at MBAS concentrations above approximately 1.0 mg/L for a variety of marine and freshwater species.

Table 4. Trace Metal Analysis Results for San Diego Bay Stormwater Samples.

Trace Metal	Reporting Limit (µg/L)	Measurement	Concentration (µg/L)			
			NAB OF 9	NAB OF 18	NASNI OF 23a	NASNI OF 26
Aluminum	50	Dissolved	ND	ND	ND	ND
		Total	405	659	224	241
Antimony	15	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Arsenic	15	Dissolved	23.0[a]	ND	ND	ND
		Total	ND	ND	ND	ND
Barium	10	Dissolved	143	94.4	29.6	27.0
		Total	169	110	39.7	29.0
Beryllium	1	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Cadmium	5	Dissolved	ND	8.00	ND	ND
		Total	ND	9.3	ND	ND
Chromium	5	Dissolved	7.00[a]	ND	ND	5.0
		Total	ND	7.00	ND	6.0
Cobalt	5	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Copper	5	Dissolved	**212**	**144**	**83.3**	**9.0**
		Total	**278**	**178**	**93.6**	**24.0**
Iron	100	Dissolved	ND	ND	ND	ND
		Total	1190	1050	346	337
Lead	10	Dissolved	10.0	ND	ND	ND
		Total	11.0	13.5	ND	13.0
Manganese	5	Dissolved	250	179	ND	21.0
		Total	311	209	42.9	35.0

[a] Dissolved metal was reported at a higher concentration than total metal. However, concentrations were near the reporting limit where true differences in concentration are difficult to detect.

Bold values in red exceed published US EPA national recommended water quality criteria for acute exposures to aquatic marine life (criteria maximum concentration), (EPA 2002b)

ND - Not Detected

Table 4 (cont'd). Trace Metal Analysis Results for San Diego Bay Stormwater Samples.

Trace Metal	Reporting Limit (µg/L)	Measurement	Concentration (µg/L)			
			NAB OF 9	NAB OF 18	NASNI OF 23a	NASNI OF 26
Molybdenum	5	Dissolved	ND	6.70	ND	ND
		Total	ND	6.90	ND	ND
Nickel	5	Dissolved	14.0[a]	13.9	8.1	ND
		Total	12.0	16.2	9.00	ND
Phosphorus	100	Dissolved	ND	319	2190	ND
		Total	166	455	2280	130
Silver	5	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Silicon	50	Dissolved	1600	2140	2310	3650
		Total	2380	3490	2790	4120
Strontium	30	Dissolved	ND	554	86.6	2960
		Total	892	570	93.4	3050
Thallium	15	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Tin	50	Dissolved	ND	ND	ND	ND
		Total	ND	ND	ND	ND
Titanium	15	Dissolved	ND	ND	ND	ND
		Total	69.0	36.5	16.7	ND
Vanadium	5	Dissolved	ND	ND	ND	21.0
		Total	ND	ND	5.40	23.0
Zinc	10	Dissolved	**742**	**985**	**297**	**80.0**
		Total	**1540**	**1220**	**398**	**121**

[a] Dissolved metal was reported at a higher concentration than total metal. However, concentrations were near the reporting limit where true differences in concentration are difficult to detect.

Bold values in red exceed published US EPA national recommended water quality criteria for acute exposures to aquatic marine life (criteria maximum concentration), (EPA 2002b)

ND - Not Detected

Table 5. Toxicity Values for Selected Metals and Surfactants of Potential Concern for *Mytilus galloprovincialis* Embryo Development and Acute Survival of *Americamysis bahia* and *Atherinops affinis*

Species	Chemical of Concern	Test Duration	Endpoint	NOEC (µg/L)	LOEC (µg/L)	Mean LC_{50} (µg/L)	Reference
A. bahia	Cu	96 hr	Survival	nr	nr	233	Nautilus (2005)
	Cu	96 hr	Survival	77	140	181	Lussier et al (1985)
	Cu	96 hr	Survival	nr	nr	153	Cripe (1994)
	Zn	96 hr	Survival	nt	nt	499	Lussier et al (1985)
	Zn	96 hr	Survival	nr	nr	303	Cripe (1994)
	Zn	96 hr	Survival	nr	nr	547	Lussier and Gentile (1985)
	Zn	96 hr	Survival	nr	nr	448	Nautilus (2005)
	Surfactants[a]	96 hr	Survival	nr	nr	<1000 to >4 x 10^6	Hall et al (1989)
	4-Nonyl Phenol	96 hr	Survival	nr	nr	>50 - <150	Lussier et al (2000)
A. affinis	Cu	96 hr	Survival	nr	nr	288	Anderson et al (1991)
	Cu	96 hr	Survival	nr	nr	365	McNulty et al (1994)
	Cu	96 hr	Survival	160	nr	nr	Isensee et al (1973)
	Cu	96 hr	Survival	nr	nr	163	Nautilus (2005)
	Zn	96 hr	Survival	nr	nr	880	Nautilus (2005)
M. galloprovincialis	Cu	48 hr	Develop.	nr	nr	5.8	Martin et al (1981)
	Cu	48 hr	Develop.	nr	nr	9.5	Nautilus (2005)
	Cu	96 hr	Develop.	nr	nr	7.9	EPA (1998)
	Zn	96hr	Develop.	nr	nr	178	Phillips (2000)
	Zn	48hr	Develop.	nr	nr	175	Martin et al (1981)
	Zn	48hr	Develop.	nr	nr	159	Nautilus (2005)
	Surfactants	96hr	Mortality	nr	nr	50,000	Swedmark et al (1971)
	Nonyl Phenol	96hr	Mortality	nr	nr	3000	Granmo et al (1989)
	LAS	96hr	Develop.	nr	nr	300	Granmo (1972)
	LAS	96hr	Mortality	nr	nr	1.66 (mg/kg)	Bressan et al (1989)

[a] - includes Alkyl Phenol Ethoxylate, Nonyl Phenol Ethoxylate, Octyl Phenol Ethoxylate, Decyl Alcohol Ethoxylate, Tridecyl Alcohol Ethoxylae, and Tripropylene

nr – not reported

Table 6. Selected Toxicity Data for Selected Metals and Surfactants of Potential Concern for Closely Related Species (The inland silverside minnow *Menidia beryllina*, the Pacific oyster *Crassostrea gigas*, and the Sheepshead minnow *Cyprinidon variegatus*)

Species	Chemical of Concern	Test Duration	Endpoint	NOEC (µg/L)	LOEC (µg/L)	Mean LC_{50} (µg/L)	Reference
M. beryllina	Zn	96hr	Survival	nr	nr	1000 - 10,000	Lewis (1993)
	Nonyl Phenol	96hr	Survival	nr	nr	70	Lussier et al (2000)
C. gigas	Zn	48hr	Development	100	nr	200	Chapman et al (1993)
	Zn	48hr	Development	nr	nr	206	Dinnel et al (1983)
	LAS	48hr	Development	nr	nr	460	Cardwell et al (1979)
C. variegatus	Nonyl Phenol	96hr	Survival	nr	nr	460	Sappington et al (2001)
	Nonyl Phenol	96hr	Survival	nr	nr	142	Lussier et al (2000)

nr – not reported

Table 7. Comparisons of Predicted Copper and Zinc TUs for Mussel Embryos (Dissolved Concentrations).

Site ID	Dissolved Cu (µg/L)	Dissolved Zn (µg/L)	Screening Test EC_{50} (% Sample)	Screening Test TU[a]	Predicted Cu TU[b]	Predicted Zn TU[b]	Predicted Cu + Zn TU
NAB OF 9	212	742	12.5	**8.00**	**16.3**	**4.24**	**20.5**
NAB OF 18	144	985	13.7	**7.30**	**11.1**	**5.63**	**16.7**
NASNI OF 23a	83.3	297	22.1	**4.52**	**6.41**	**1.70**	**8.10**
NASNI OF 26	9.00	80.0	>69.0	**<1.00**	**0.69**	**0.46**	**1.15**

[a] TU is equal to 100 divided by the screening test EC_{50}.

[b] TU is equal to the concentration of the trace metal in the stormwater sample divided by the concurrent reference toxicant test EC_{50} (13 µg/L Cu, and 175 µg/L Zn).

Table 8. Comparisons of Predicted Copper and Zinc TUs for Mysid Shrimp (Dissolved Concentrations).

Site ID	Total Copper (µg/L)	Total Zinc (µg/L)	Screening Test EC_{50} (% Sample)	Screening Test TU[a]	Predicted Copper TU[b]	Predicted Zinc TU[b]	Predicted Copper + Zinc TU
NAB OF9	212	742	>100	**<1.00**	**0.731**	**1.40**	**2.13**
NAB OF18	144	985	42.4	**2.36**	**0.497**	**1.86**	**2.36**
NASNI OF23a	83.3	297	>100	**<1.00**	**0.287**	**0.560**	**0.848**
NASNI OF26	9.00	80.0	>100	**<1.00**	**0.031**	**0.151**	**0.182**

[a] TU is equal to 100 divided by the screening test EC_{50}.
[b] TU is equal to the concentration of the trace metal in the stormwater sample divided by the reference toxicant test EC_{50} (290 µg/L Cu, and 530 µg/L Zn).

Table 9. Comparisons of Predicted Copper and Zinc TUs for Pacific Topsmelt (Dissolved Concentrations).

Site ID	Total Copper (µg/L)	Total Zinc (µg/L)	Screening Test EC_{50} (% Sample)	Screening Test TU[a]	Predicted Copper TU[b]	Predicted Zinc TU[b]	Predicted Copper + Zinc TU
NAB OF9	212	742	>100	**<1.00**	**1.30**	**0.843**	**2.14**
NAB OF18	144	985	38.2	**2.62**	**0.883**	**1.12**	**2.00**
NASNI OF23a	83.3	297	>100	**<1.00**	**0.511**	**0.338**	**0.849**
NASNI OF26	9.00	80	>100	**<1.00**	**0.055**	**0.091**	**0.146**

[a] TU is equal to 100 divided by the screening test EC_{50}.
[b] TU is equal to the concentration of the trace metal in the stormwater sample divided by the reference toxicant test EC_{50} (163 µg/L Cu, and 880 µg/L Zn).

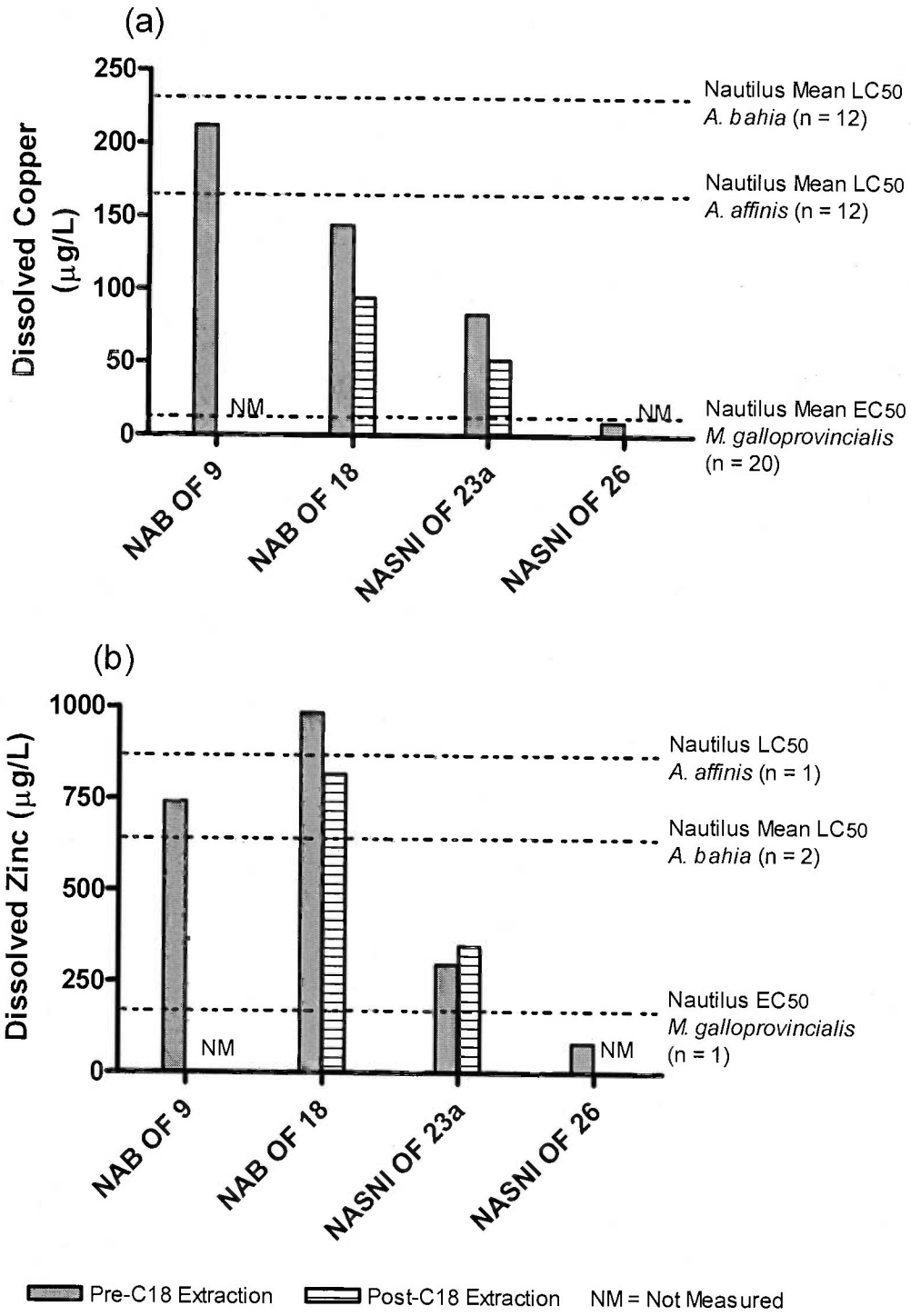

Figure 12. Dissolved copper (a), and dissolved zinc (b) measurements for San Diego Bay stormwater samples before and after C_{18} column extraction. Mean EC_{50} values (mussel embryos) and acute LC_{50} values for mysids and topsmelt are displayed on each figure.

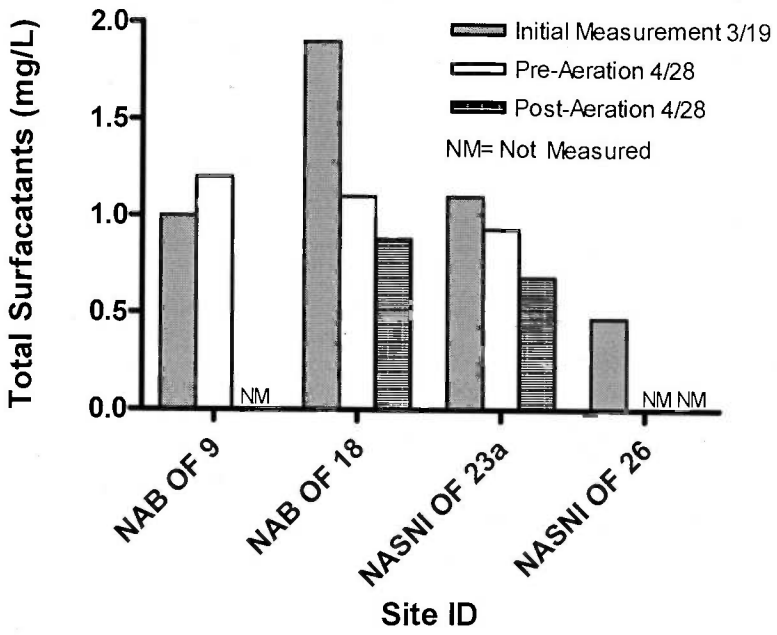

Figure 13. Anionic surfactant (as MBAS) analytical results for San Diego Bay stormwater samples.

3.3.2 Copper and Zinc Mixture Studies

The results of the Phase I TIE manipulations strongly suggest that divalent cationic metals were the primary cause of toxicity to mussel embryos and mysids in NAB OF 9 and a significant contributor to mussel toxicity in NAB OF 18 and NASNI OF 23a. A comparison of concentrations with available toxicity data further supports these conclusions in that sufficient metal is present to account for the presence of toxicity.

To help evaluate the extent to which each metal contributed to toxicity to both bivalve embryos and mysids, and to understand how they interacted when present in solution together, a series of tests were performed to identify the level of toxicity associated with each metal and their degree of interaction. Zinc and copper were tested alone, and as a mixture at two different ratios to evaluate whether the ratios affected the interactive characteristics of the metals. Mysids were tested May 19, 2005 at ratios of 2.2:1 and 5.4:1. The 5.4 to 1 ratio was the mean ratio of the four stormwater outfall samples analyzed, with ratios ranging from 3.8 to 8.9. The 2.2 value is equal to the LC_{50} ratio between zinc and copper for mysids. Mediterranean mussels were tested in a prior study (May 2004) at ratios of 4.5:1 and 13.6:1, corresponding to ratios obtained for stormwater samples collected February 19, 2004 (4.5:1) and the LC_{50} ratio (13.6:1)

between zinc and copper for this species. The metal mixture studies with the mussel are included here, but were previously reported and submitted to SPAWAR as a part of TIE stormwater evaluations conducted in May 2004.

Mediterranean Mussel

The EC_{50} estimates determined in May 2004 for copper and zinc individually during this test series was 9.6 and 160 µg/L, respectively. These values are likely conservative as they were obtained in laboratory seawater. Irrespective of the ratios tested, toxicity appeared to be additive; in mixtures of the two metals in laboratory seawater toxic units of 1.2 to 1.3 were calculated. Figure 14 shows the response curves for zinc and copper individually, as well as for the two mixtures. Clearly, similar dose-responses were exhibited in all four of the tests, suggesting similar modes of action and additive toxicity.

Applying these laboratory-derived EC_{50} estimates to metals concentrations measured in the actual samples collected March 18, 2005 suggested that, in all cases, the predicted toxicity over-estimated the actual toxicity observed in the original screening tests (Table 7). In other words, there was frequently less toxicity present in the original samples than would have been predicted on the basis the concentrations of total metals present and their additivity. Thus, these data suggest that some portion of the metals present in the samples was not bioavailable. Reduced bioavailability of trace metals due to binding by various ligands (e.g., dissolved organic carbon) is well-documented in the literature (Bergman and Dorward-King 1997). On average, the actual bivalve TUs in the stormwater samples were 46 percent of those that would have been predicted on the basis of the joint toxicity of copper and zinc in laboratory seawater.

In order to address the relative importance of each of the metals to overall toxicity, predicted TUs for copper and zinc alone and in combination were plotted against the actual TUs determined in the screening tests on the original samples (Figure 15). The relationships for copper alone and in combination with zinc were statistically significant ($p \leq 0.05$). A positive relationship was also observed for zinc; however, it was not statistically significant. The relationship between actual toxicity and the toxicity predicted by the combination of metals, however, was the strongest, with an r^2 value of 0.98. This finding suggests that both metals contributed to toxicity in all three toxic samples.

Mysid Shrimp

Mysid acute LC_{50} estimates determined concurrently during this study for copper and zinc alone

were 291 and 647 µg/L, respectively. As with bivalves, these values are likely conservative as they were obtained in laboratory seawater. Regardless of the ratios tested, toxicity appeared to be somewhat less than additive, in mixtures of the two metals in laboratory seawater, toxic units for the two mixtures were 1.45 and 1.62 compared with a predicted TU of 1. Figure 16 shows the response curves for zinc and copper individually, as well as for the two mixtures. Clearly, similar dose-responses were exhibited in all four of the tests, suggesting similar modes of action and additive toxicity. Interestingly, however, the two mixture studies appeared to actually be less than additive in toxicity. Not only were the actual TUs required to elicit a response greater than predicted, the slope of the response curves appeared to diverge from those associated with the individual metals.

Applying these laboratory-derived EC_{50} estimates to metals concentrations measured in the actual samples collected March 18, 2005 found that the predicted toxicity for the sum of copper and zinc concentrations over-estimated the actual toxicity observed in the original screening test for Sample NAB OF 9 (Table 8). These data suggests that at least some portion of the metals present in Sample OF 9 was not bioavailable. Predicted toxicity based on copper and zinc concentrations in NAB OF 18 was identical to the actual toxicity observed; despite this observation, toxicity in sample NAB OF 18 was clearly attributable to an organic compound, and not to trace metals. Again, this result indicates that a substantial fraction of copper and zinc is not bioavailable. The sum of predicted copper and zinc toxic units for NASNI OF 23a and NASNI OF 26 was less than 1, which corresponds to the actual toxic units of less than 1 that were found for both of these samples.

Unfortunately, due to a lack of mysid data with sufficient toxic responses in this study, predicted versus actual TU plots for copper and zinc alone and in combination were not meaningful for this species and are, therefore, not included.

Pacific Topsmelt

A copper and zinc mixture study was not performed for topsmelt; however an evaluation of predicted versus actual toxicity units is provided below.

Topsmelt acute LC_{50} estimates determined for copper and zinc based on internal data collected at Nautilus are 163 (n=12) and 880 µg/L (n=1), respectively. As with bivalves and mysids, these values are likely conservative as they were obtained in laboratory seawater.

Applying laboratory-derived EC_{50} estimates to copper and zinc concentrations measured in the

actual samples collected March 18, 2005 found that the predicted toxicity over-estimated the actual toxicity observed in the original screening test for Sample NAB OF 9 (Table 9). Sample NAB OF 9 was not toxic to topsmelt, therefore, this data further suggests that a substantial fraction of the metals present in Sample OF 9 were not bioavailable. In contrast to the data for mussels and mysids, the predicted summed copper and zinc toxic units for topsmelt exposed to Sample NAB OF 18 were less than the actual toxicity observed. This observation supports the finding that an organic constituent, and not a trace metal, was responsible for toxicity in this sample. The sum of predicted copper and zinc toxic units for NASNI OF 23a and NASNI OF 26 was less than 1, which corresponds to actual toxic units of less than 1 for both of these samples.

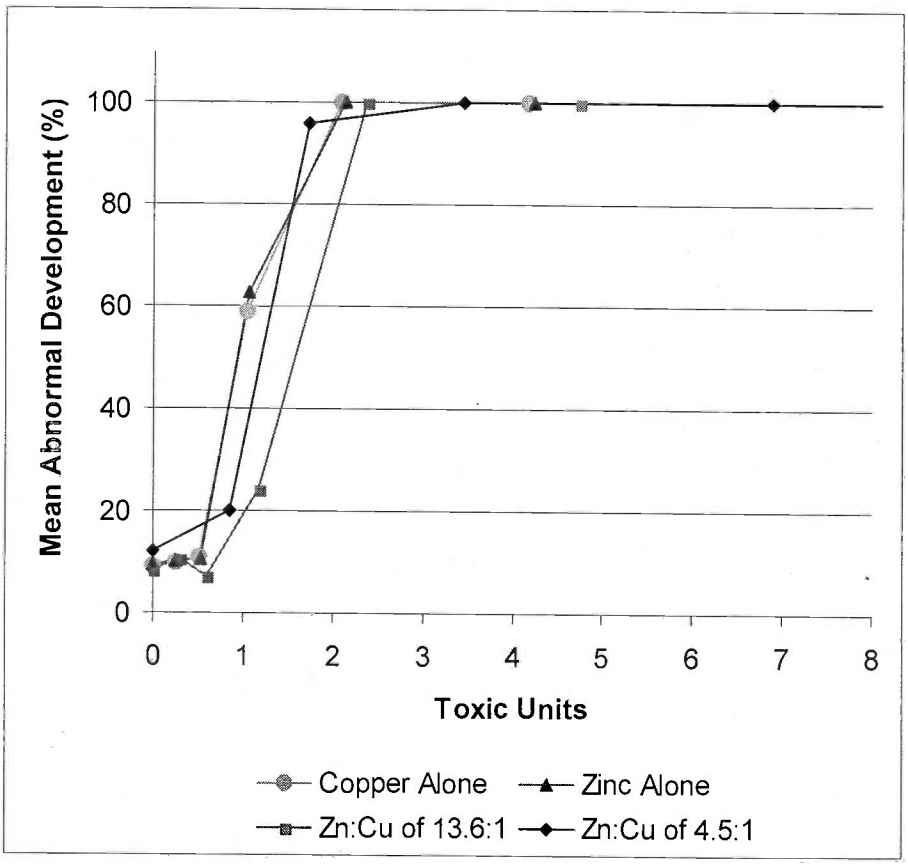

Figure 14. Response of mussel embryos to copper and zinc alone and in combination. Metals are expressed as TUs. February 2004 study.

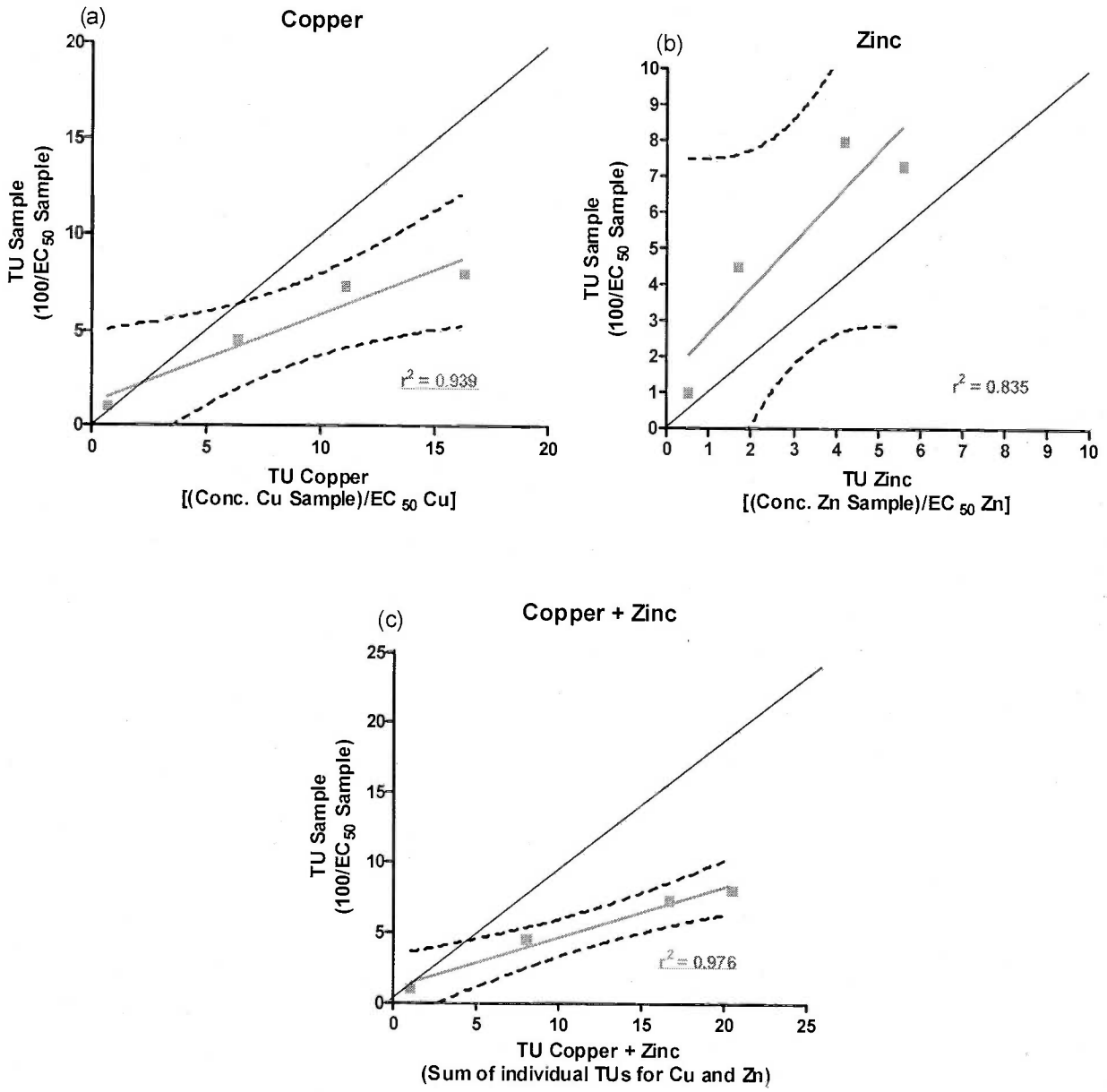

Figure 15. Comparisons of predicted and actual copper and zinc TU values. February 2004 study. Underlined r^2 values are statistically significant ($p \leq 0.05$).

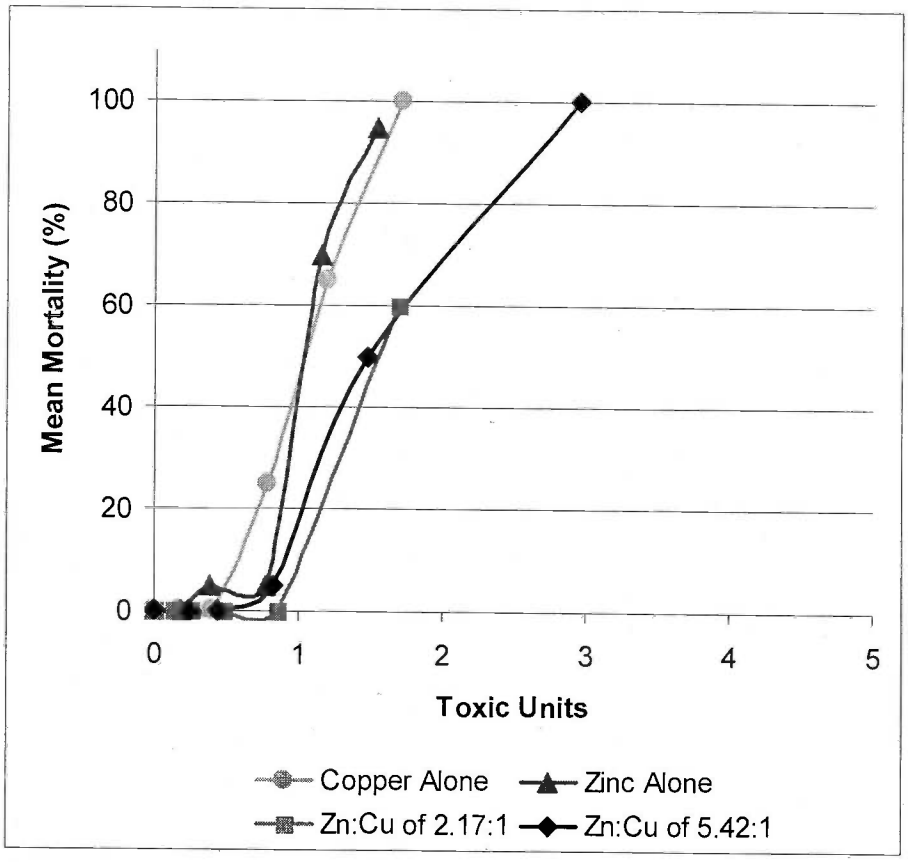

Figure 16. Response of mysid shrimp to copper and zinc alone and in combination. Metals are expressed as TUs.

3.3.3 Toxicity Relationships to Identified Toxicants of Concern

Relationships between toxicity and concentrations of chemicals of concern across samples provide additional lines of supporting evidence, depending the amount of data available. Relationships alone, however, must be evaluated with caution as chemical constituents are often correlated and interactions between chemical and physical parameters may also effect such relationships.

Copper and Zinc

Linear regression relationships between toxicity and concentrations of dissolved copper and zinc to the three species tested are provided in Figures 17 though 19. Despite the limited number of data points available for analysis (n=4), strong relationships were observed between

mussel embryo development and concentrations of both copper and zinc with r^2 values of 0.98 and 0.90, respectively. The relationship between zinc and mysid survival was also reasonably strong with an r^2 value of 0.76. Relationships between mysid survival and copper (r^2 = 0.49), and both copper and zinc for topsmelt (r^2 = 0.23 and 0.44, respectively) were relatively weak.

These relationships support conclusions that: 1) copper and zinc contributed toxicity to mussel embryos in all three toxic samples; 2) toxicity to mysids was attributed to zinc in one sample (NAB OF 9); and 3) toxicity to topsmelt was not attributed to copper or zinc in any of the stormwater samples tested.

Surfactants

Multiple lines of evidence from Phase I TIE tests indicated that anionic surfactants are a contributing toxic class of compounds in Samples NAB OF 18 and NASNI OF 23a for both mussels and mysids. Linear regression relationships between concentrations of MBAS and initial screening test responses for all test organisms are provided in Figures 20 through 22. In summary, despite the limited number of data points available for analysis, strong relationships were obtained for mysids and topsmelt with r^2 values of 0.91 and 0.92, respectively. The relationships between bivalve embryo development and MBAS was less compelling, with an r^2 value of 0.57.

These relationships are consistent with the results of the TIE manipulations that suggest that: 1) surfactants contributed some toxicity to mussel embryos in two samples (NAB OF 18 and NASNI OF 23a); and 2) surfactants appear to be primarily responsible for observed toxicity to mysids in these two samples. Evidence suggests that surfactants may also be primarily responsible for toxicity to topsmelt in these two samples, however, additional Phase II/III TIE procedures are required to confirm this hypothesis for the species.

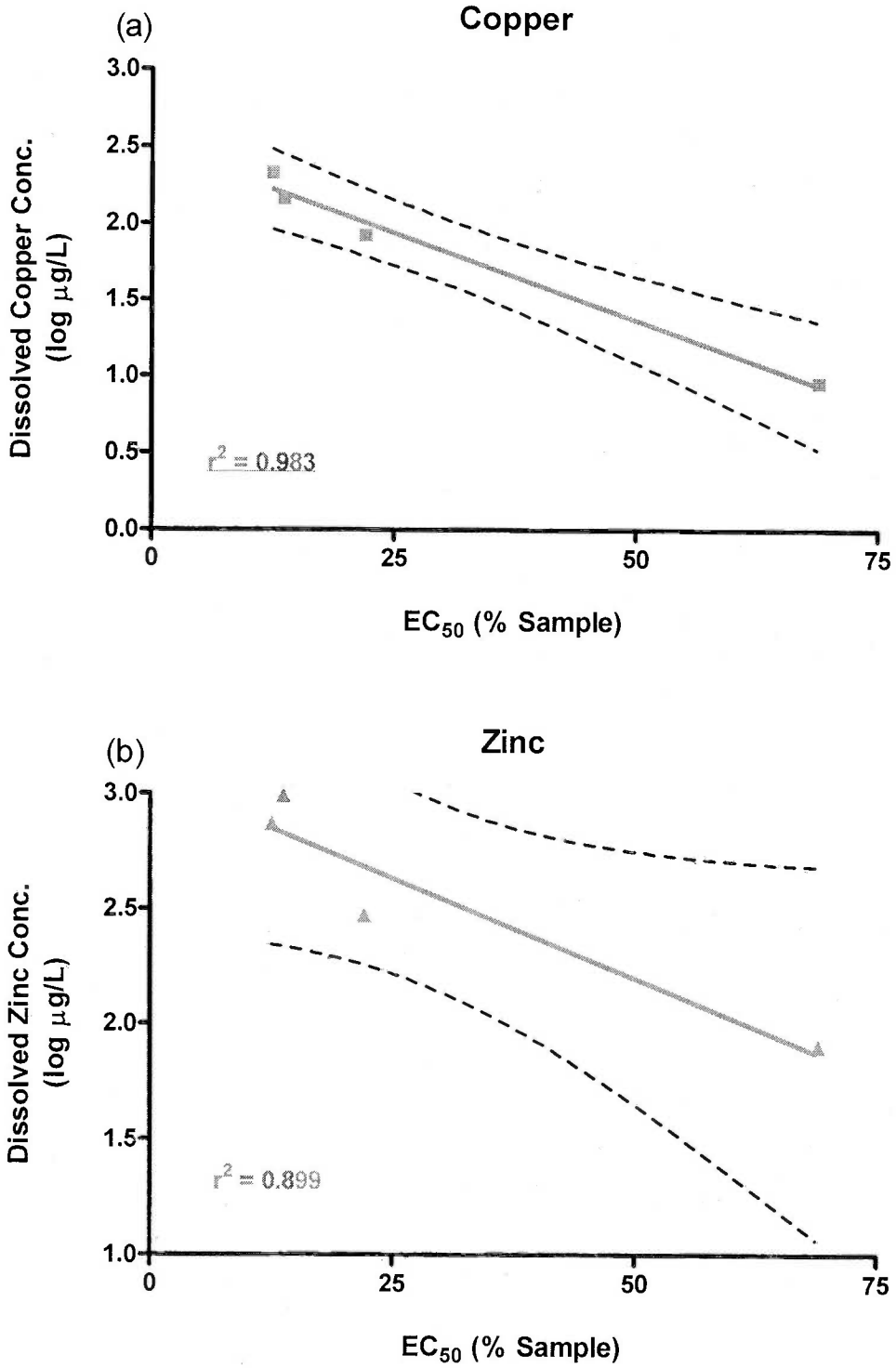

Figure 17. Relationship between mussel embryo development and (a) dissolved copper and (b) dissolved zinc. Underlined r^2 values are statistically significant ($p \leq 0.05$)

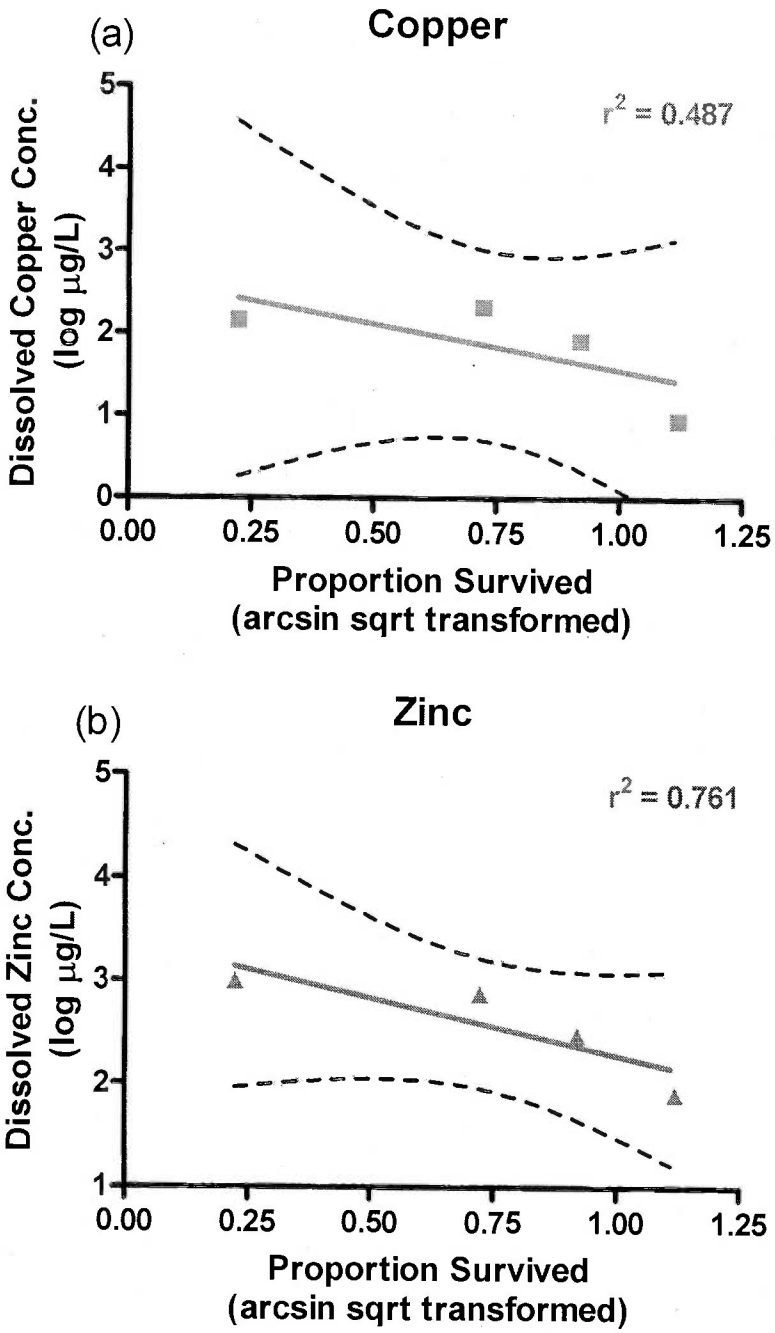

Figure 18. Relationship between acute mysid survival in undiluted sample and (a) dissolved copper and (b) dissolved zinc.

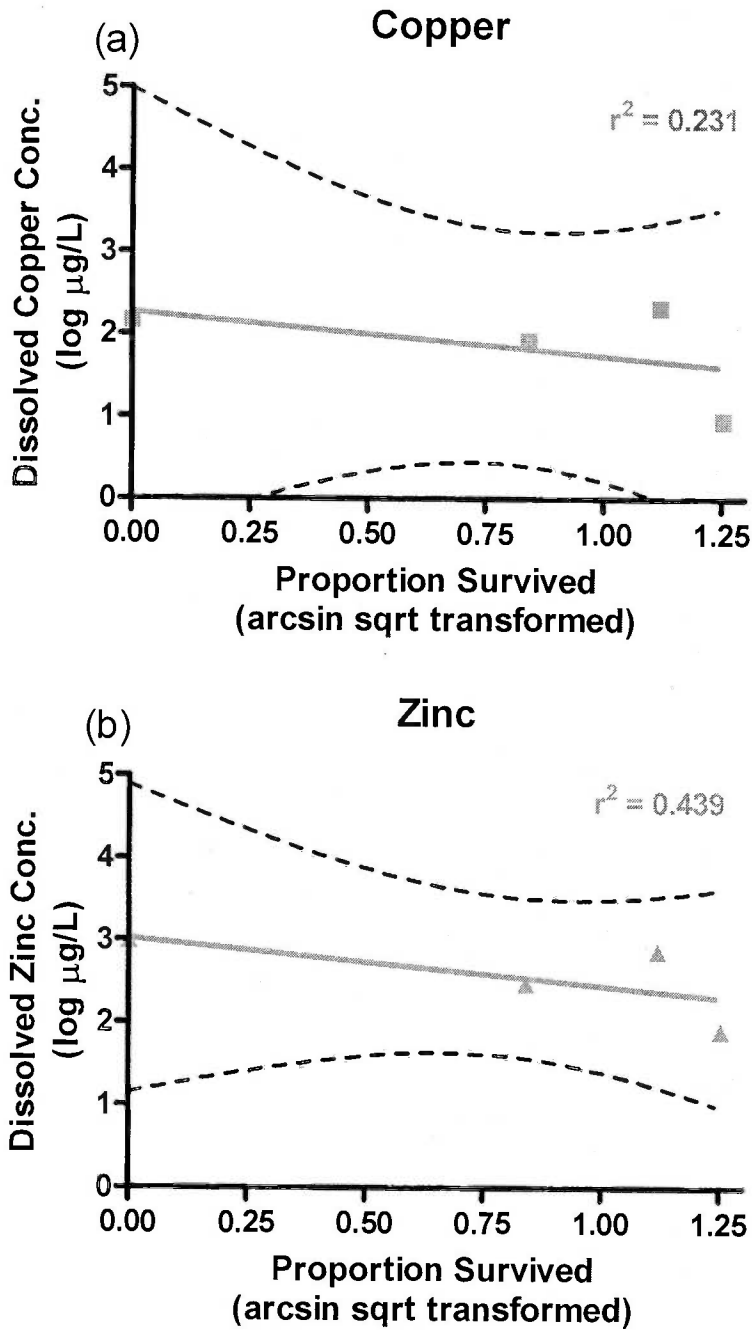

Figure 19. Relationship between acute topsmelt survival in undiluted sample and (a) dissolved copper and (b) dissolved zinc.

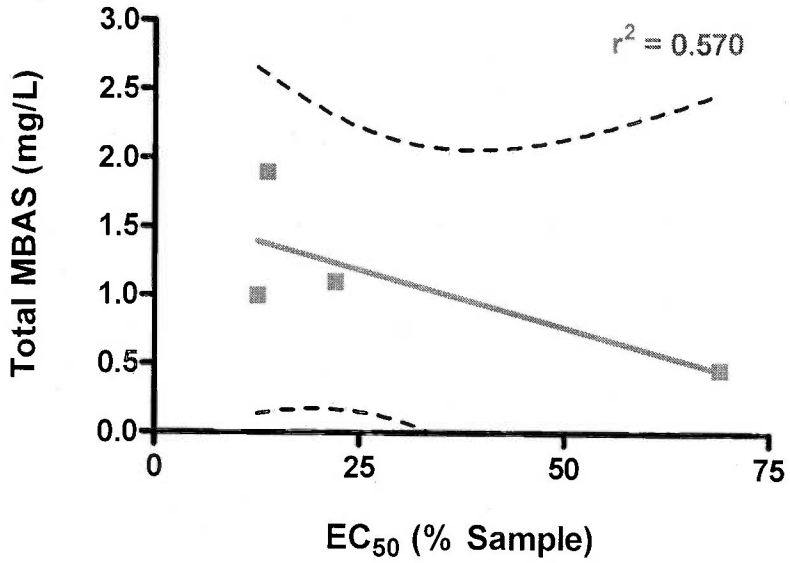

Figure 20. Relationship between mussel embryo development and MBAS concentrations. The EC_{50} was plotted on the X axis for this species due to zero percent normal in the highest concentrations tested in all three toxic samples.

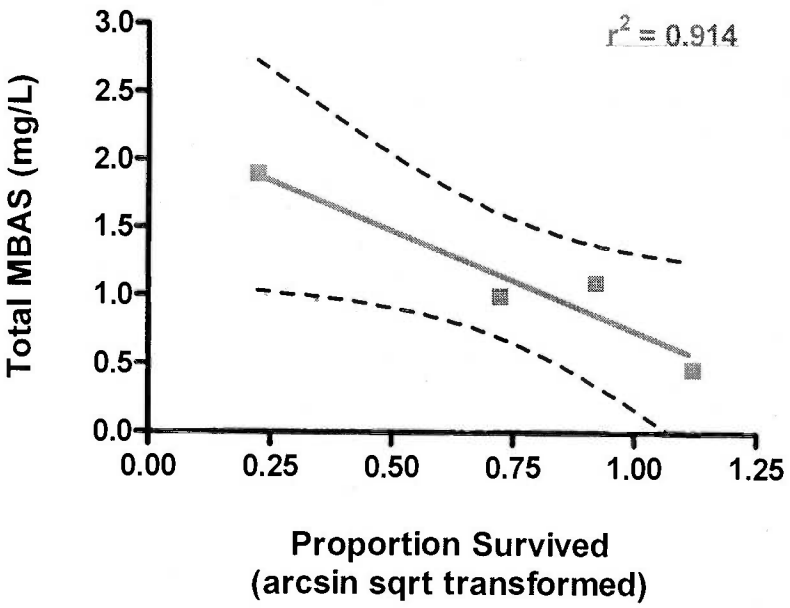

Figure 21. Relationship between acute mysid survival and MBAS concentrations in undiluted sample.

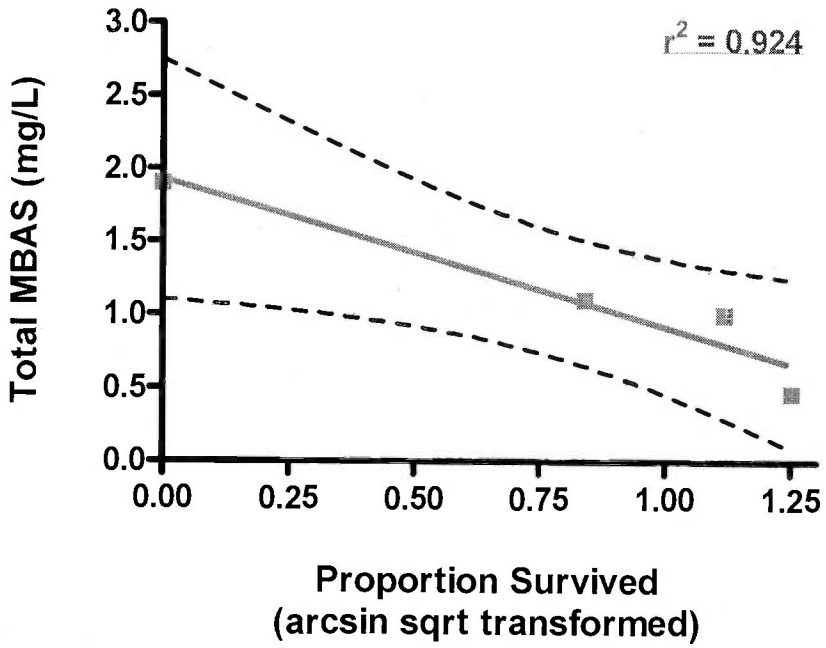

Figure 22. Relationship between acute topsmelt survival and MBAS concentrations in undiluted sample.

4.0 CONCLUSIONS

Results for each of the toxic samples are summarized below in the context of the findings of the TIE investigation. A final summary of results is provided in Table 10.

Table 10. Summary of Identified Toxicants of Concern

Sample ID	Species/ Endpoint	Primary Toxicant(s)
NAB OF 9	Bivalve embryo development	Copper and zinc
	Mysid acute survival	Zinc and copper
	Topsmelt acute survival	Not toxic
NAB OF 18	Bivalve embryo development	Copper and zinc (50%), Anionic surfactants (50%)
	Mysid acute survival	Surfactants[a]
	Topsmelt acute survival	Surfactants[a]
NASNI OF 23a	Bivalve embryo development	Copper and zinc (50%), Anionic surfactants (50%)
	Mysid acute survival	Surfactants[b]
	Topsmelt acute survival	Surfactants[b]

[a] Weight of evidence suggests surfactants despite the lack of confirmatory TIE data available for interpretation due to loss of toxicity in the sample. The type of surfactant (e.g. anionic vs nonionic) was not confirmed.

4.1 NAB OF 9

4.1.1 Mediterranean Mussel

TIE results clearly identified both copper and zinc as potential causes of toxicity in Sample NAB OF 9 based on 1) the success and specificity of the EDTA treatment, 2) toxic unit calculations for these two metals; and 3) the strong relationship between actual and predicted TU values across all outfall samples for these two metals. Copper, with a predicted TU value of 16.3, potentially contributes a greater proportion of toxicity than zinc, with a much lower predicted TU value of 4.2. The actual proportion of toxicity contributed by each metal, however, is not possible to derive at this point due to unknown differences in bioavailability at the time of sample collection.

4.1.2 Mysid

Zinc and copper were identified as the primary toxicants of concern in Sample NAB OF 9 based on 1) the success and specificity of the EDTA treatment; 2) toxic unit calculations for these two metals; and 3) documented additivity of these two metals. The TU value for zinc (1.2) is greater than that derived during this study for copper (0.7). Based on the range of mysid sensitivity data collected over time at Nautilus, a copper TU value as high as 1.3 may be derived based on its concentration in NAB OF 9. Without data to document their relative bioavailability in the sample, it is not possible to know whether toxicity was due to zinc alone, copper alone, or to a combination of copper and zinc.

4.2 NAB OF 18

4.2.1 Mussel

Toxicity to mussels in Sample NAB OF 18 was attributed to a combination of copper, zinc, and anionic surfactants. Addition of EDTA removed approximately 50 percent of the observed toxicity in the Phase I TIE. Results of this treatment and an evaluation of toxic units indicate that copper and zinc are the only cationic trace metals of concern. Copper, with a predicted TU value of 11.1, potentially contributes a greater proportion of toxicity than zinc, with a predicted TU value of 5.6. Toxicity not removed by EDTA (the remaining 50 percent) may be attributable to anionic surfactants based on the following observations, in concert: 1) reduction in toxicity following extraction of the sample through a C_{18} column; 2) a similar reduction in toxicity following aeration; 3) recovery of toxicity in both C_{18} methanol extracts and foam collected

during aeration tests; 4) complete removal of toxicity in the C_{18} methanol extract following anion exchange; 5) a concentration of surfactants, as MBAS, greater than documented levels of potential concern for some surfactants; and 6) a reduction in surfactant concentrations following aeration. Combined treatments (C_{18} + EDTA and aeration + EDTA) completely removed toxicity in the sample, thus providing additional evidence that a combination of trace metals and anionic surfactants may explain all of the toxicity observed in the sample for this species.

4.2.2 Mysid

Toxicity to mysids in Sample NAB OF 18 was attributed to surfactants based on the following combination of observations: 1) removal of toxicity following both extraction of the sample through a C_{18} column and aeration; 2) recovery of toxicity in foam collected during aeration tests; 3) a concentration of surfactants greater than that found to cause toxicity to mysids in prior studies at Nautilus; 4) a reduction in surfactant concentrations following aeration, 5) a strong correlation between surfactant concentrations (i.e. MBAS) and survival of mysids across all samples tested; and 6) anionic surfactants were identified as a cause of toxicity to mussels in this sample. Unlike mussels, trace metals were not identified as a toxicant of concern to mysids in this sample due to the lack of toxicity reduction following addition of EDTA. The loss of toxicity between the screening test and round two TIE Baseline test limited the ability to make interpretations based on most of the TIE treatments performed during this round. Rapid loss of toxicity, however, is another characteristic routinely observed for surfactants as they break down over time and adhere to the sides of sample containers (EPA 1991). In support of this observation, a decrease in surfactant concentrations over time was measured in this study for this sample.

4.2.3 Topsmelt

Toxicity to topsmelt in Sample NAB OF 18 was attributed to surfactants based on the following combined observations: 1) complete removal of toxicity following both extraction of the sample through a C_{18} column and aeration; 2) a concentration of surfactants greater than that found to cause toxicity to other marine species; 3) a reduction in surfactant concentrations following aeration; 4) a strong correlation between surfactant concentrations and survival of topsmelt across all samples tested; and 5) anionic surfactants were identified as a cause of toxicity to mussels in this sample. Trace metals were not identified as a toxicant of concern to topsmelt in this sample due to the lack of toxicity reduction following addition of EDTA.

4.3 NASNI OF 23a

4.3.1 Mussel

Toxicity to mussels in Sample NASNI OF 23a, like that for NAB OF 18, was attributed to a combination of copper, zinc, and surfactants. Addition of EDTA removed approximately ½ of observed toxicity in the screening test. Results of this treatment and an evaluation of toxic units indicate that copper and zinc are the only cationic trace metals of concern. Copper, with a predicted TU value of 6.4, potentially contributes a much greater proportion of toxicity than zinc, with a predicted TU value of 1.7. Toxicity not removed by EDTA may be attributable to surfactants based on these observations in concert: 1) complete removal of toxicity following extraction of the sample through a C_{18} column; 2) a reduction in toxicity following aeration; 3) recovery of toxicity in foam collected during aeration tests; 4) a concentration of surfactants greater than documented levels of potential concern depending on the specific type of surfactant; and 5) a reduction in surfactant (MBAS) concentrations following aeration. The combined aeration and EDTA treatment completely removed toxicity in the sample, thus providing additional evidence that a combination of trace metals and surfactants may explain all of the toxicity observed in the sample for this species.

4.3.2 Mysid

Toxicity to mysids in Sample NASNI OF 23a, like that in NAB OF 18, appears to also be attributed to surfactants based on the following combined observations: 1) a strong correlation between surfactant (MBAS) concentrations and survival of mysids in all samples tested; 2) a concentration of surfactants greater than documented levels of potential concern; and 3) surfactants were identified as a cause of toxicity to mussels in this sample. The loss of toxicity between the screening and TIE Baseline tests limited the ability to make any interpretations based on TIE treatments. Rapid loss of toxicity, as above, is a characteristic routinely observed for surfactants (EPA 1991). In support of this observation, a decrease in surfactant concentrations over time was measured in this study for this sample.

4.3.3 Topsmelt

A loss of toxicity between the screening and TIE Baseline tests limited our ability to make additional interpretations based on TIE treatments. Toxicity to topsmelt in Sample NASNI OF 23a, like that in NAB OF 18, may be attributable to surfactants based on the following combined observations: 1) a strong correlation between surfactant concentrations and survival of topsmelt

in all samples; 2) a concentration of surfactants higher than reported levels of concern for other marine species; and. 3) surfactants were identified as a cause of toxicity to mussels in this sample. Rapid loss of toxicity is a routinely observed characteristic for surfactants as they break down over time and adhere to the sides of sample containers (EPA 1991). In support of this observation, a decrease in surfactant concentrations over time was measured in this study for this sample.

5.0 QA/QC

5.1 Screening Bioassays

5.1.1 Mediterranean Mussel

Mean normal development of mussel larvae in all laboratory seawater and hypersaline brine controls tested during the screening phase of the study ranged between 89 and 96 percent. MSDs ranged between 3.0 and 4.7 percent, indicating test sensitivity was within a suitable range.

5.1.2 Mysid Shrimp

At 96-hours, control performance met the 90 percent acute criterion in all cases, with mean survival ranging from 95 to 100 percent across laboratory seawater and artificial salt controls. MSDs calculated in comparison with the artificial salt controls ranged from 12.0 to 28.9 percent across samples.

5.1.3 Pacific Topsmelt

Both laboratory seawater and artificial salt controls met survival acceptability criteria. At 96-hours, mean control survival was 100 percent across all controls (> 90 percent acute criterion). MSDs ranged from 10 to 12.5 percent across samples.

5.2 TIEs

5.2.1 Mediterranean Mussel

Baseline controls exhibited a mean of 90 to 98 percent normal larvae across all rounds of testing, indicating that the organisms used were healthy and test conditions were adequate. Method controls among the various treatments utilized exhibited 84 to 99 percent normal larvae,

indicating that the test organisms were not adversely affected by the test methods.

5.2.2 Mysid Shrimp

Survival in the baseline laboratory seawater and artificial salt controls ranged from 90 to 100 percent, indicating that the organisms were healthy and test conditions were adequate. Method controls of the treatments ranged from 90 to 100 percent, suggesting that the treatments themselves had no adverse affect on the test animals.

5.2.3 Pacific Topsmelt

Topsmelt survival in the baseline and method controls ranged from 95 to 100 percent, demonstrating that the organisms were healthy and were not affected by testing conditions.

5.3 Reference Toxicant Tests

All reference toxicant test results were within +/- 2 standard deviations of the long-term laboratory control chart averages, suggesting that the sensitivity of the test organisms and the laboratory techniques were consistent throughout the study.

6.0 LITERATURE CITED

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Appendix G

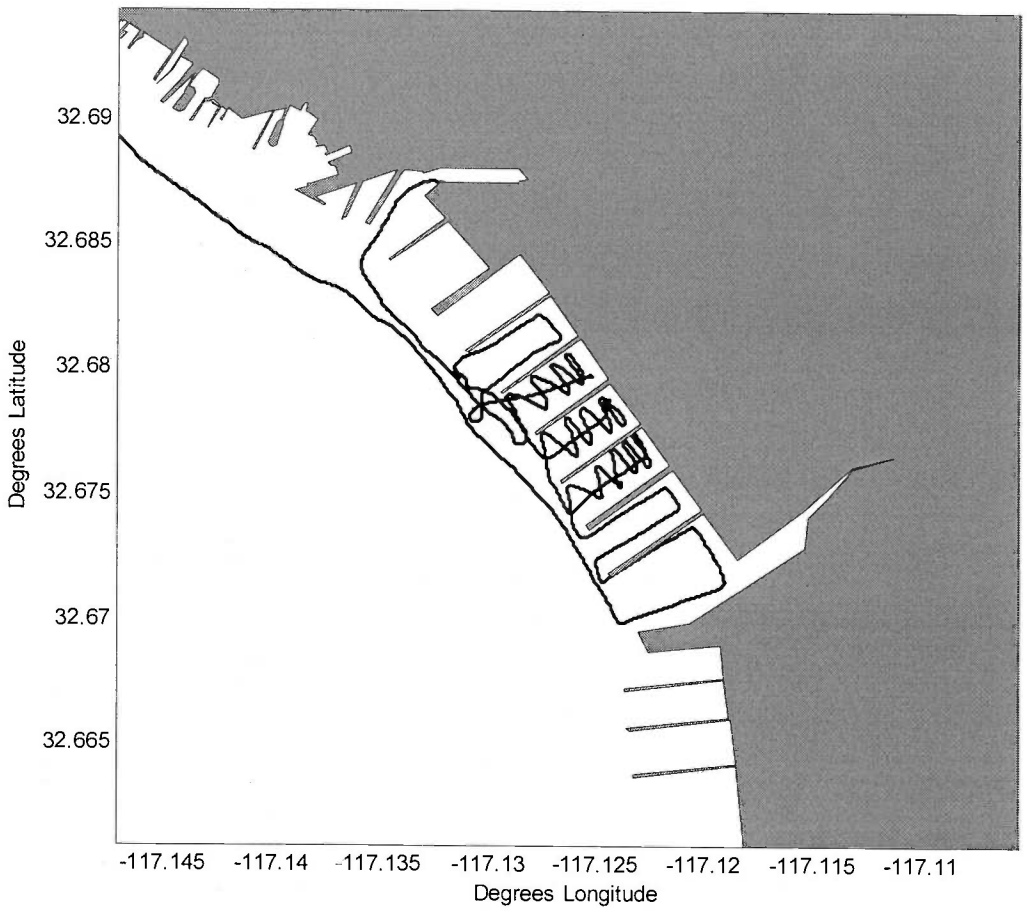
Plume Mapping Data Plots

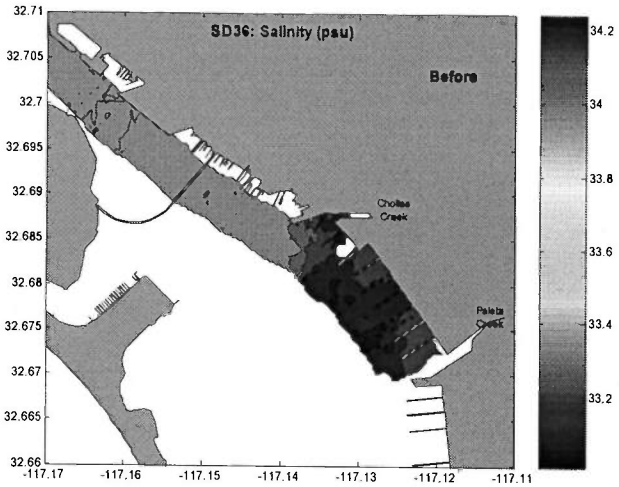
Appendix G1

NAV

SDB1- 11/7/2002
SDB2- 2/24/2003

SDB1- 11/7/2002

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Before
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Paleta Creek
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32.705
32.7
32.695
32.69
32.685
32.68
32.675
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32.665
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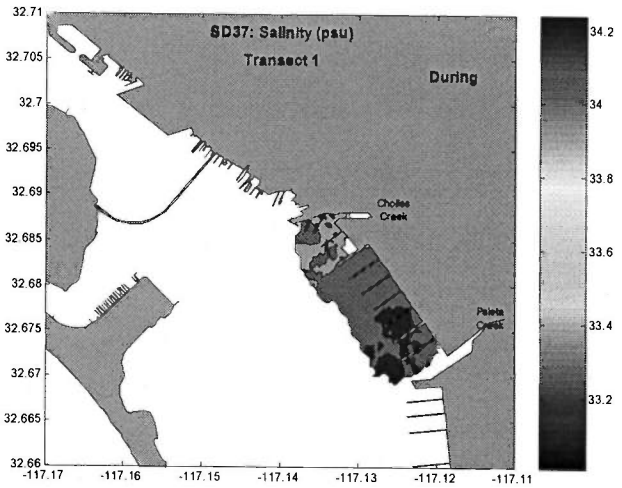
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Paleta Creek
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32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
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-117.17
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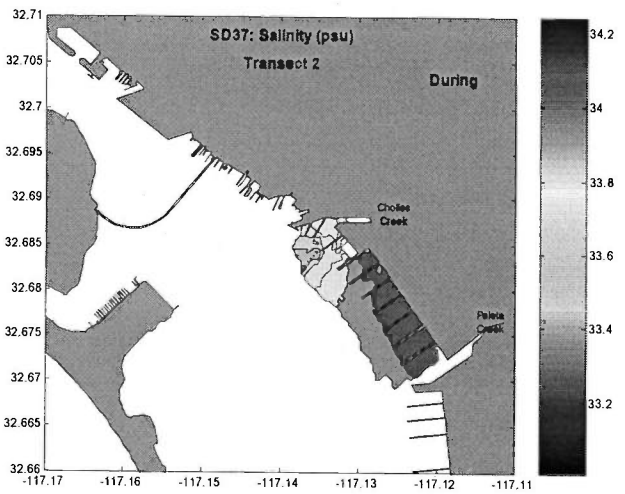
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Transect 2
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Paleta Creek
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32.675
32.67
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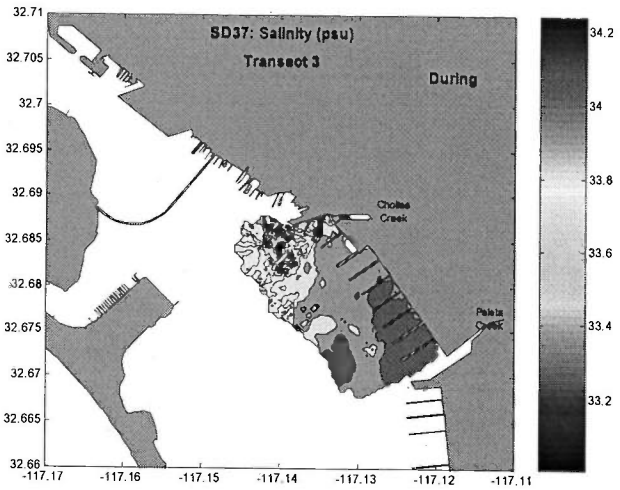
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Paleta Creek
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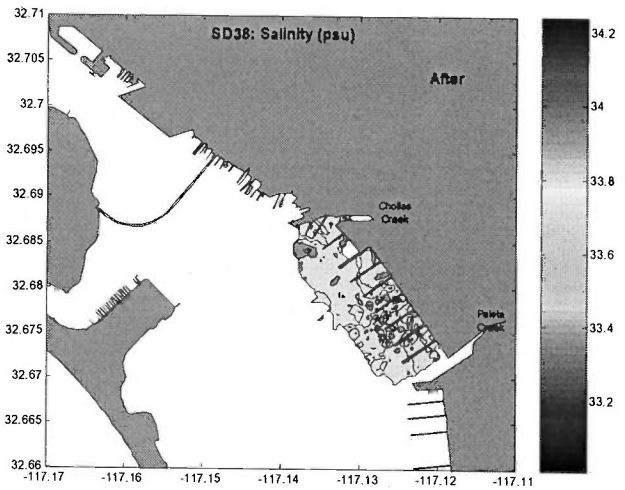
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Paleta Creek
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32.69
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32.68
32.675
32.67
32.665
32.66
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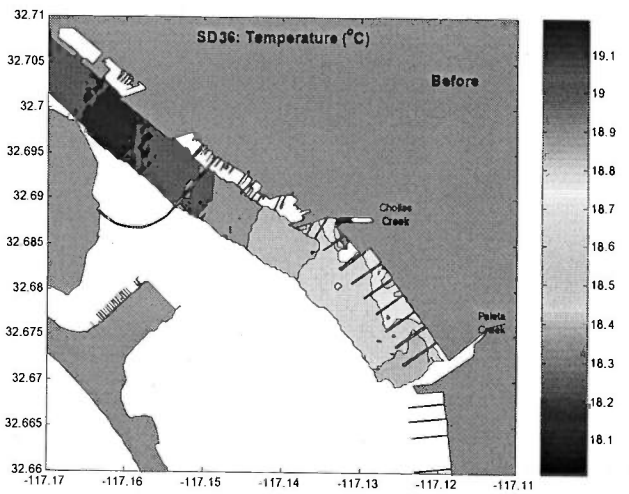
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Paleta Creek
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32.69
32.685
32.68
32.675
32.67
32.665
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-117.16
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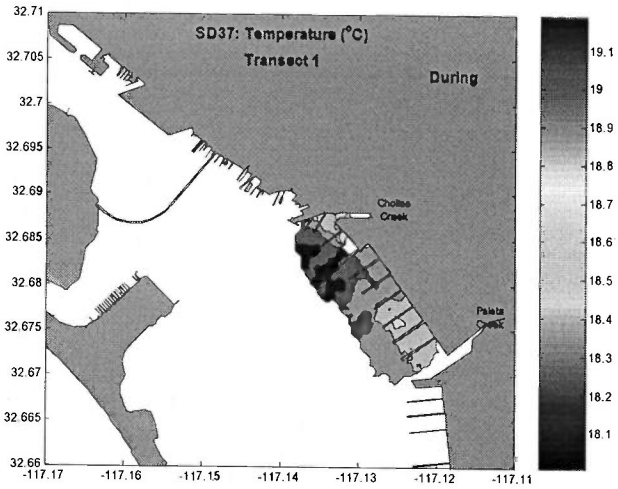
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Transect 1
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Paleta Creek
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32.7
32.695
32.69
32.685
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32.675
32.67
32.665
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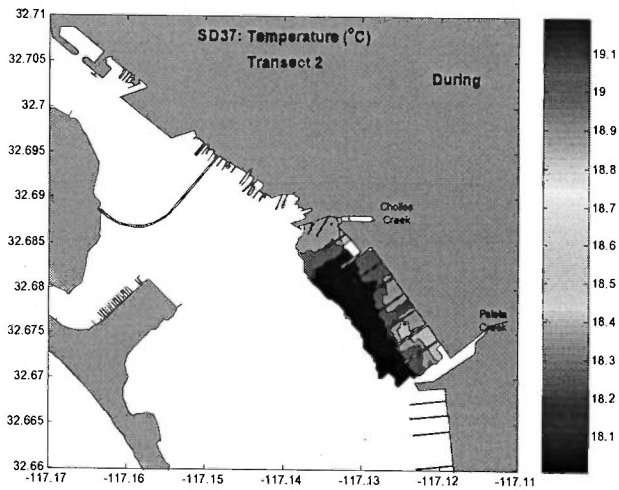
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Transect 2
During
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Paleta Creek
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32.7
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32.68
32.675
32.67
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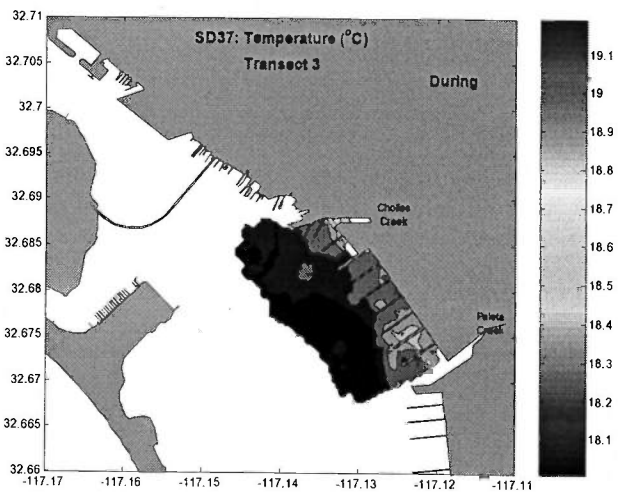
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Transect 3
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Paleta Creek
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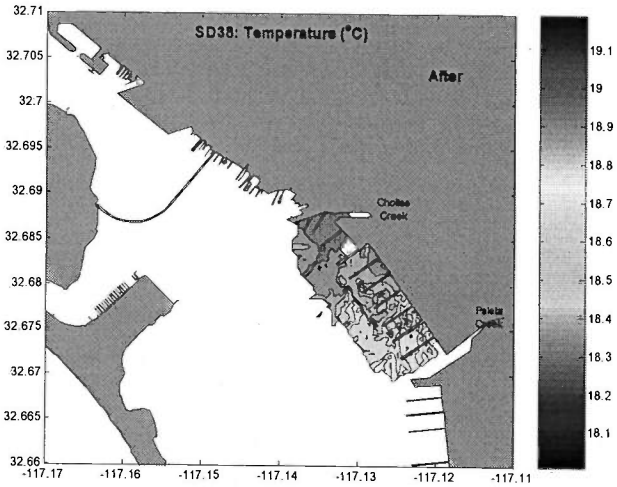
SD38: Temperature (°C)
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Pescadero Creek
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32.675
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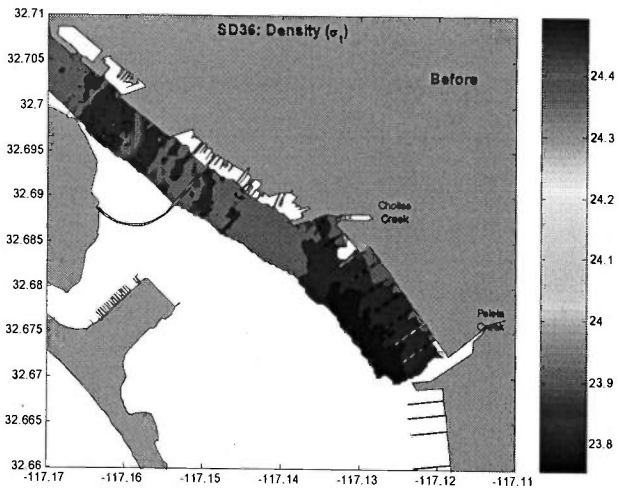
SD36: Density (σ_t)
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Cholse Creek
Pescadero Creek
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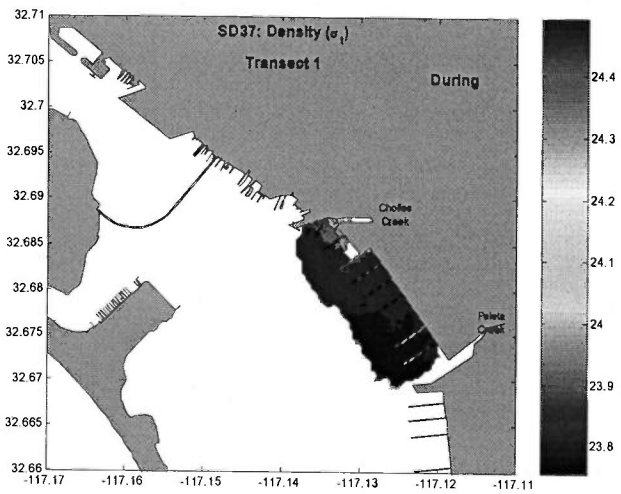
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Transect 1
During
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Pescadero Creek
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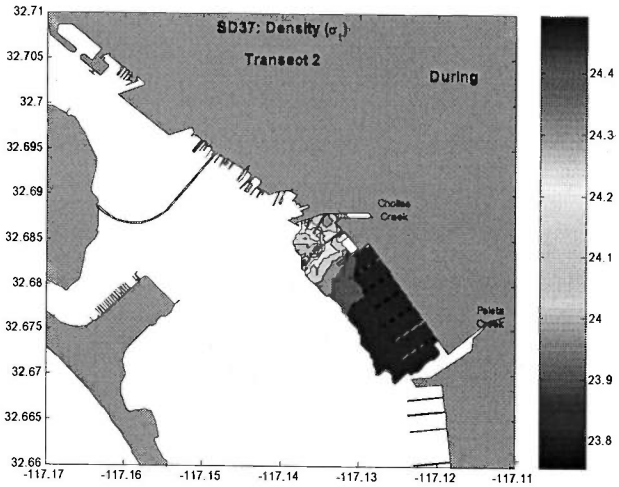
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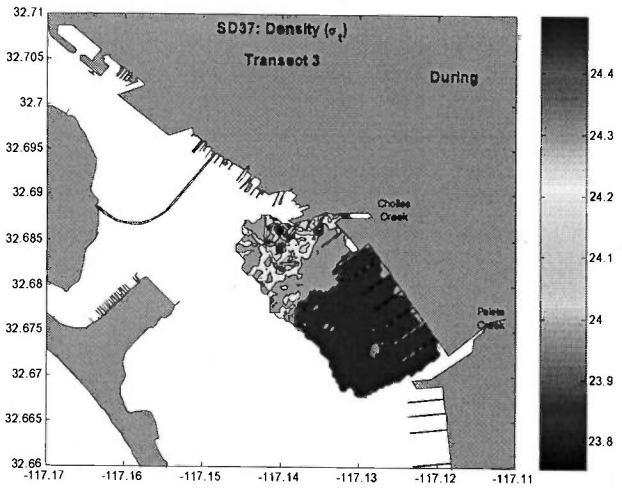
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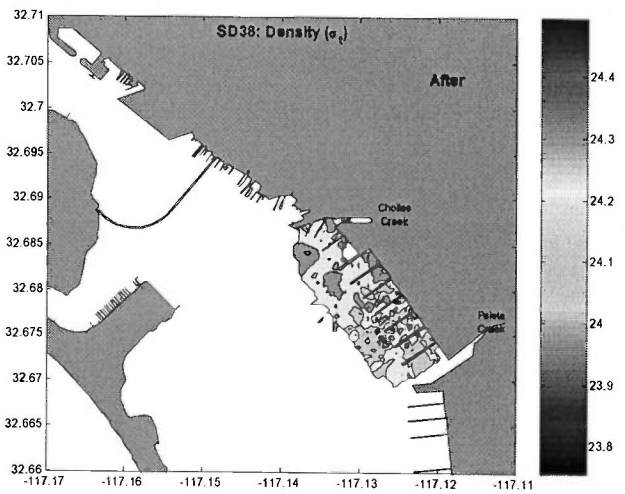
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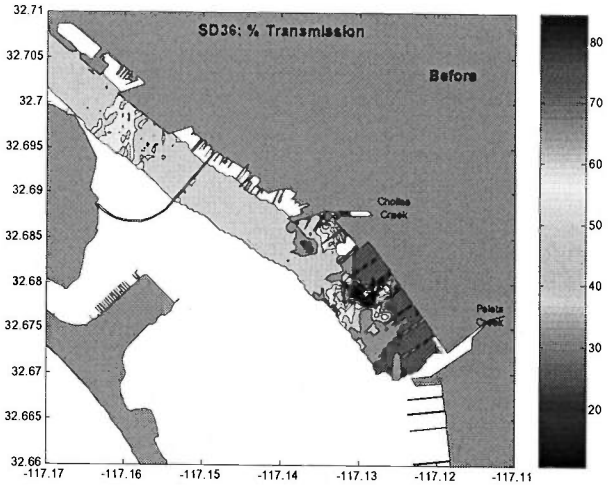
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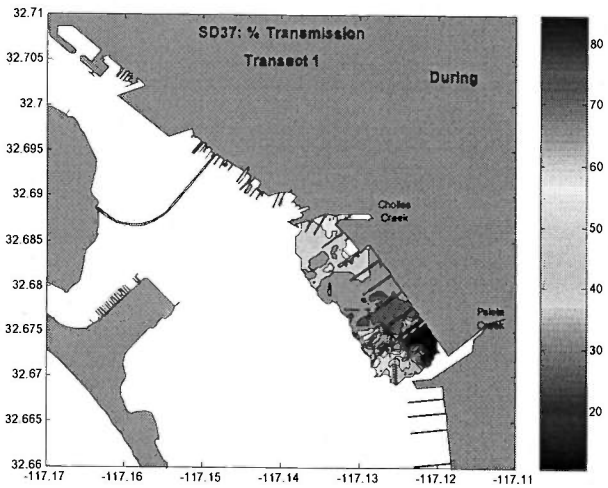
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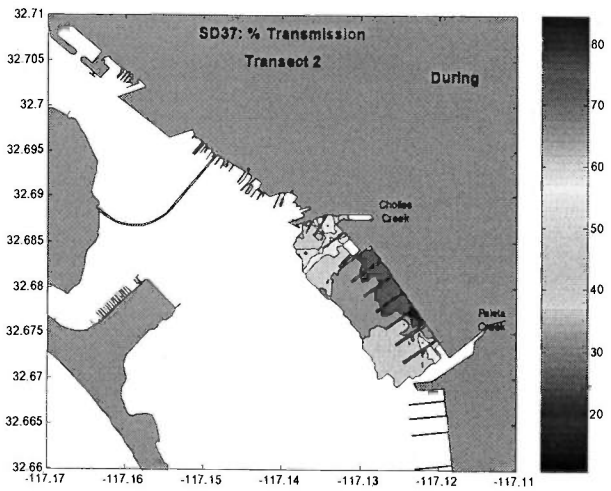
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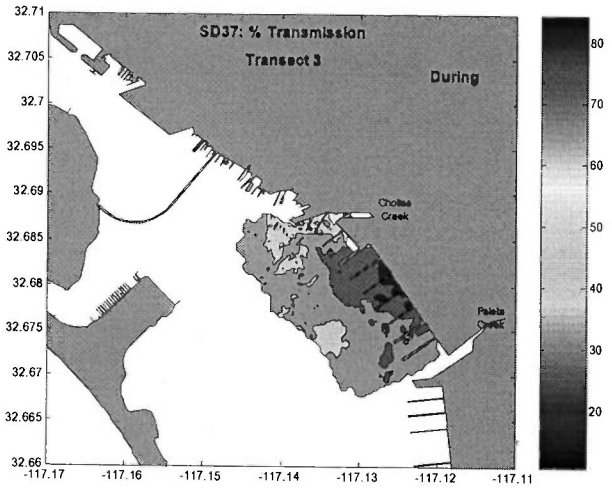
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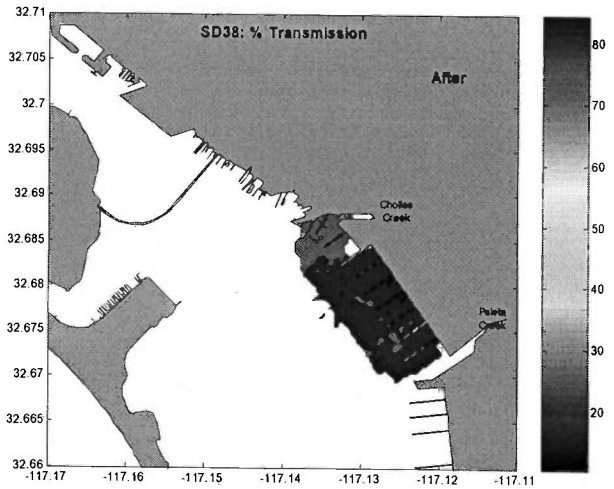
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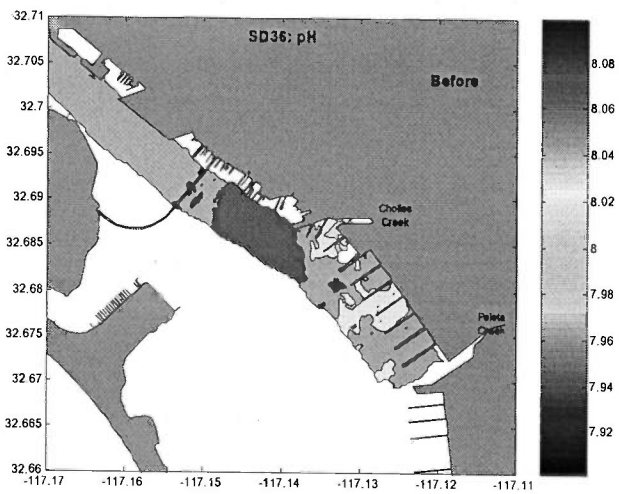
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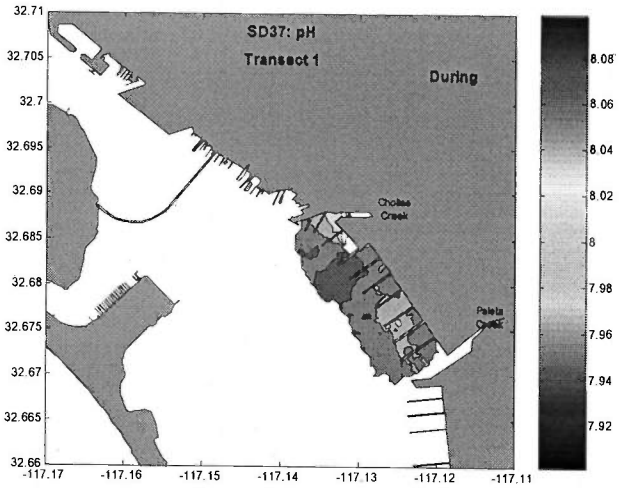
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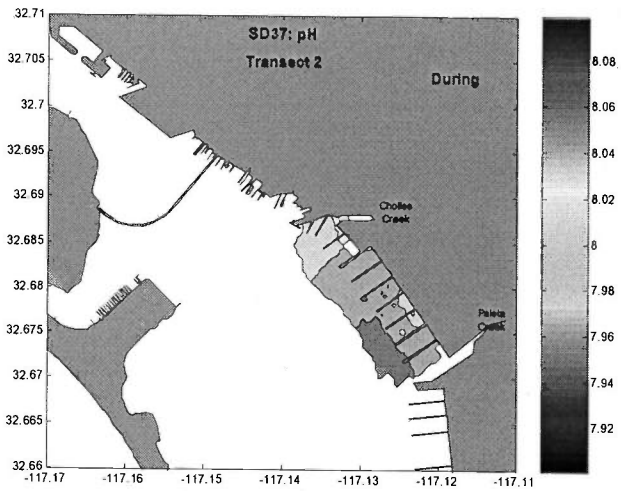
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Transect 2
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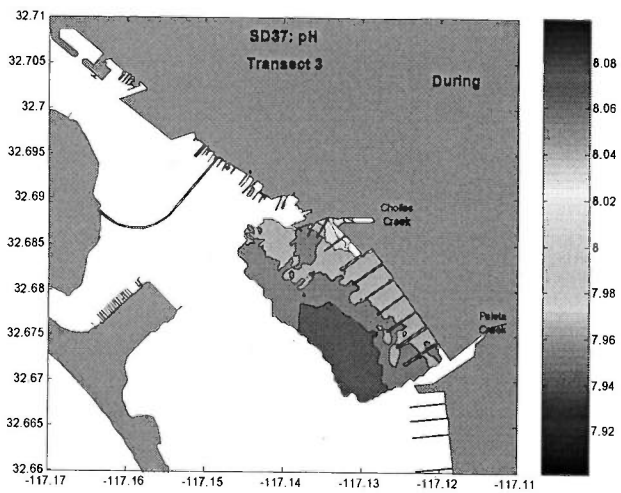
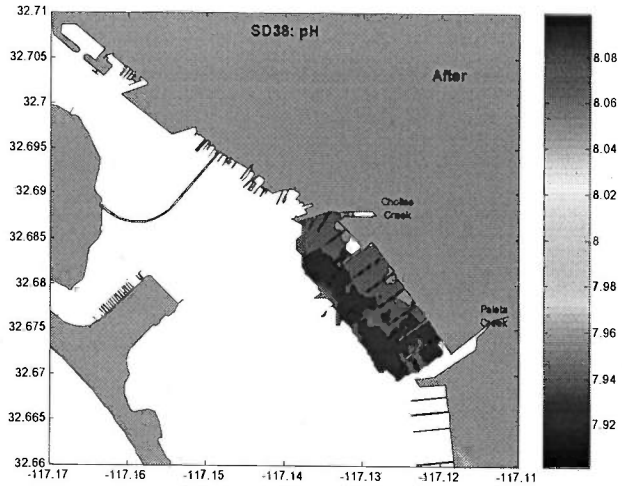
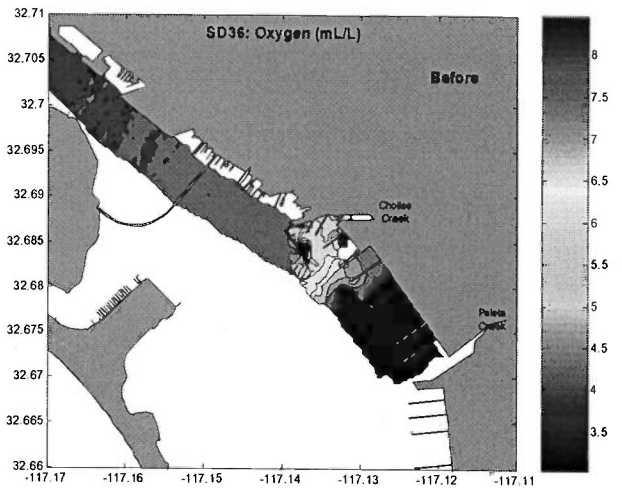
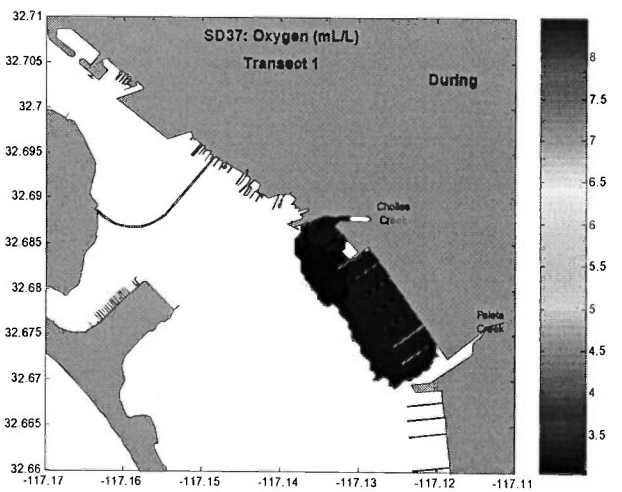
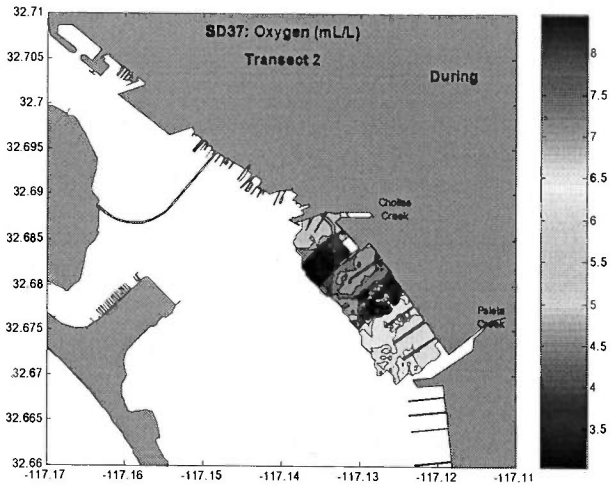
8.02
8
7.98
7.96
7.94
7.92

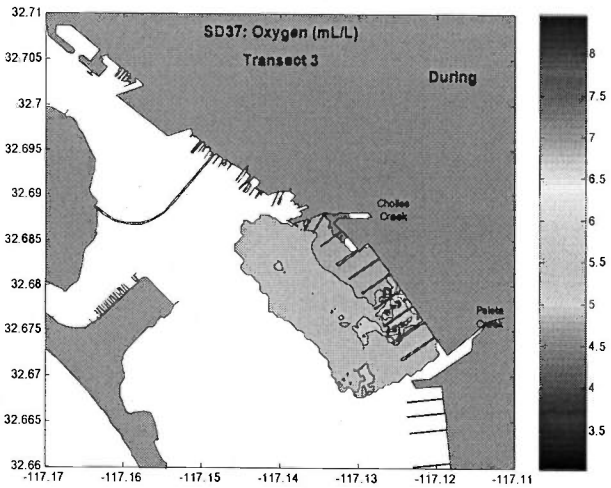

SD37: pH
Transect 3
During
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8.08
8.06
8.04
8.02
8
7.98
7.96
7.94
7.92

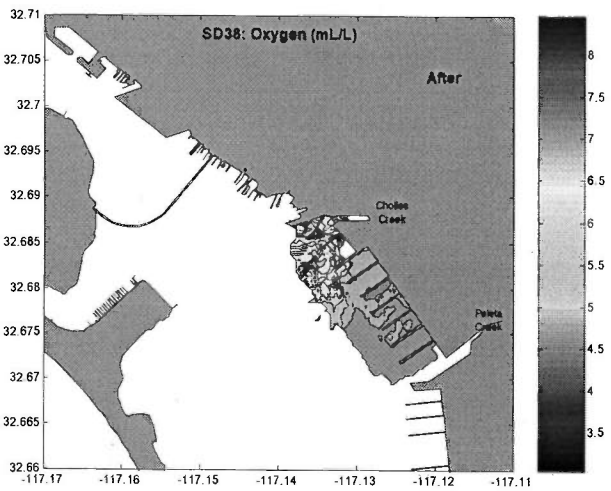

SD38: pH
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8.08
8.06
8.04
8.02
8
7.98
7.96
7.94
7.92

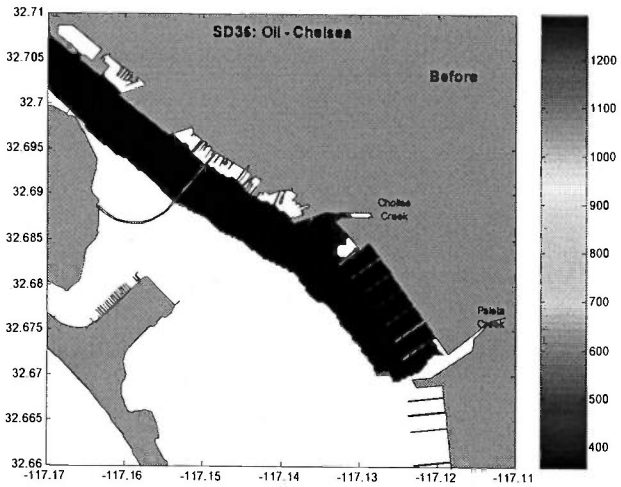

SD36: Oxygen (mL/L)
Before
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5

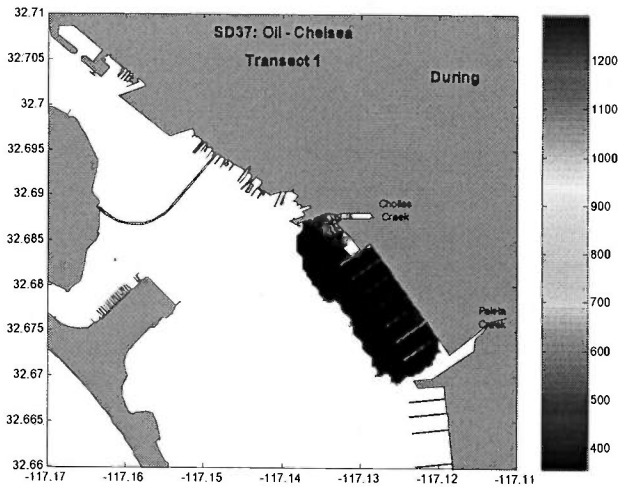

SD37: Oxygen (mL/L)
Transect 1
During
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5

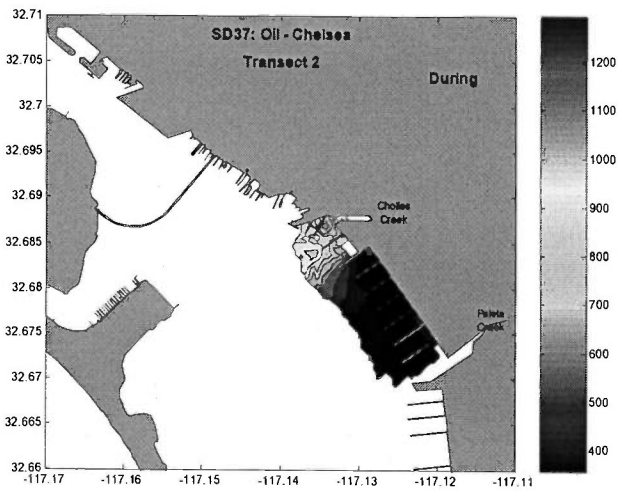

SD37: Oxygen (mL/L)
Transect 2
During
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5

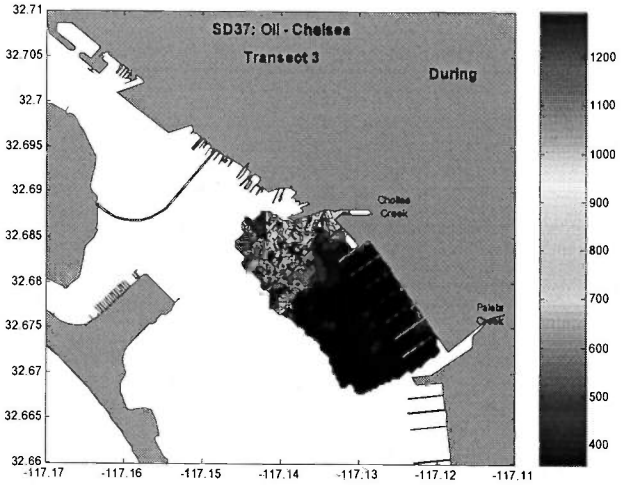

SD37: Oxygen (mL/L)
Transect 3
During
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5

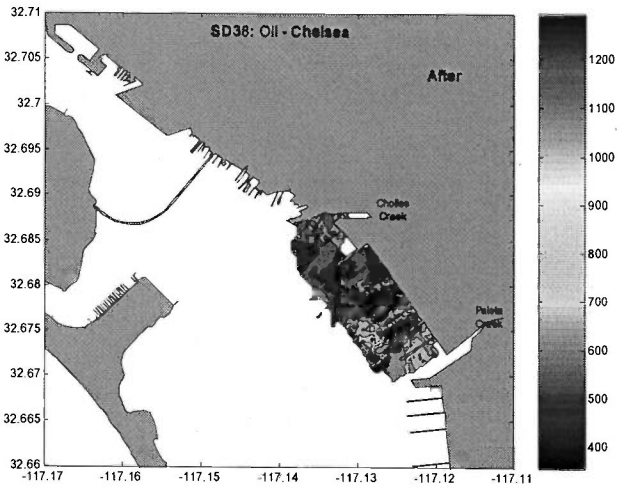

SD38: Oxygen (mL/L)
After
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8
7.5
7
6.5
6
5.5
5
4.5
4
3.5

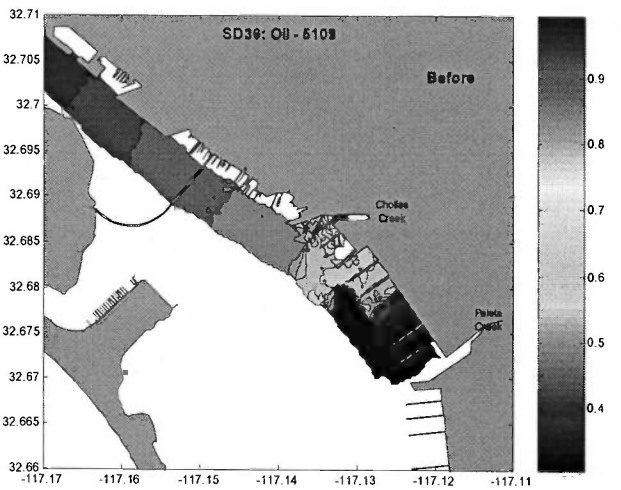

SD36: Oil - Chelsea
Before
Chelsea Creek
Pelekis Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1100
1000
900
800
700
600
500
400

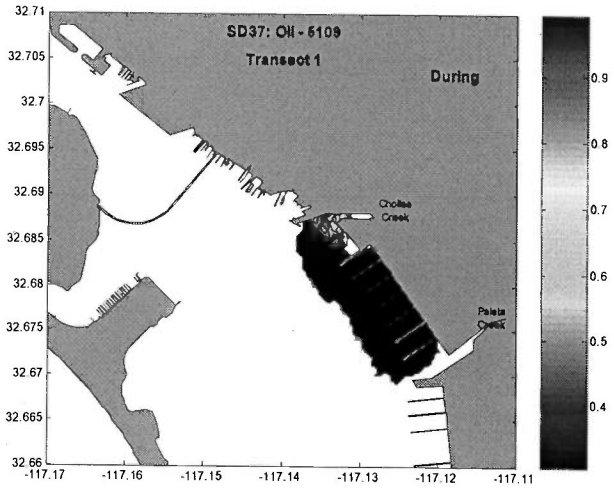

SD37: Oil - Chelsea
Transect 1
During
Chelsea Creek
Pelekis Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1100
1000
900
800
700
600
500
400

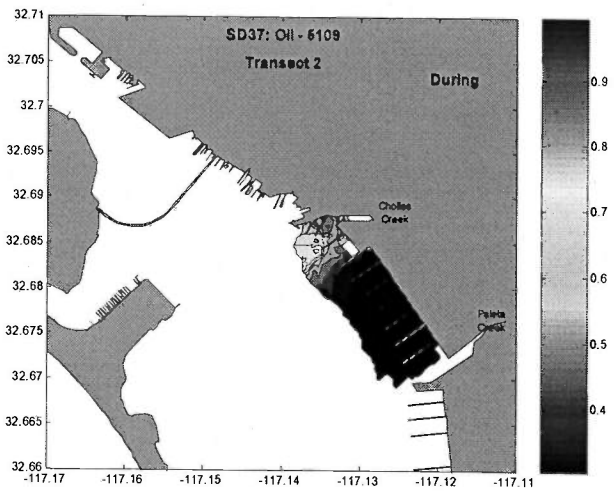

SD37: Oil - Chelsea
Transect 2
During
Chelsea Creek
Pelekis Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1100
1000
900
800
700
600
500
400

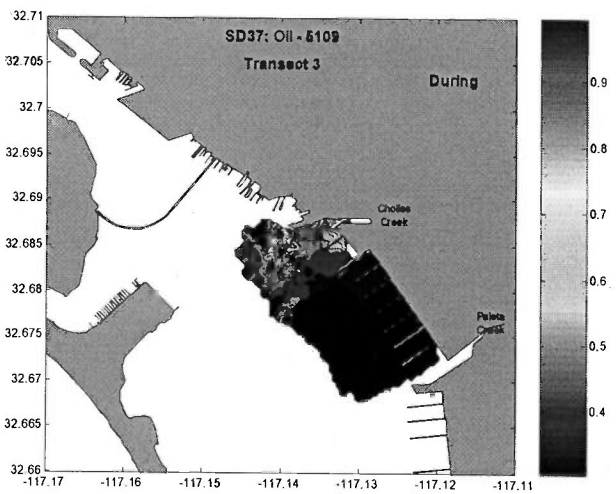

SD37: Oil - Chelsea
Transect 3
During
Chelsea Creek
Peleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1100
1000
900
800
700
600
500
400


SD38: Oil - Chelsea
After
Chelsea Creek
Peleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1100
1000
900
800
700
600
500
400


SD39: Oil - 5109
Before
Chelsea Creek
Peleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.9
0.8
0.7
0.6
0.5
0.4


SD37: Oil - 5109
Transect 1
During
Cholla Creek
Pelela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.9
0.8
0.7
0.6
0.5
0.4


SD37: Oil - 5109
Transect 2
During
Cholla Creek
Pelela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.9
0.8
0.7
0.6
0.5
0.4


SD37: Oil - 5109
Transect 3
During
Cholla Creek
Pelela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
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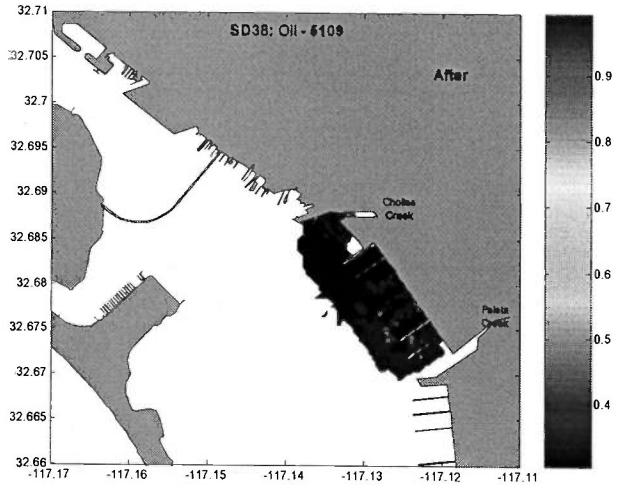
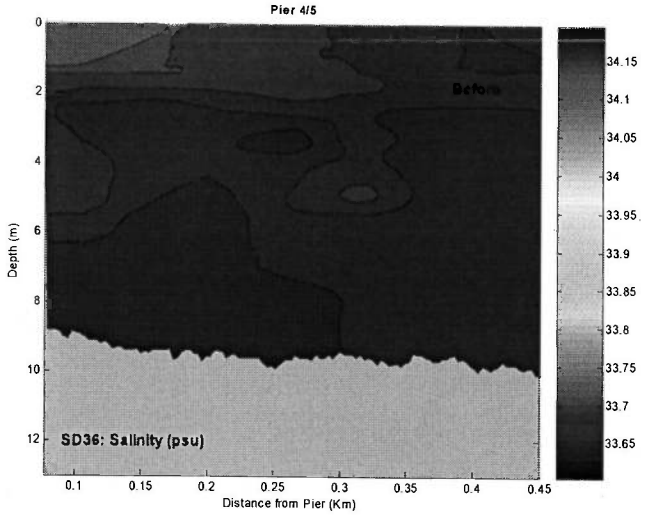
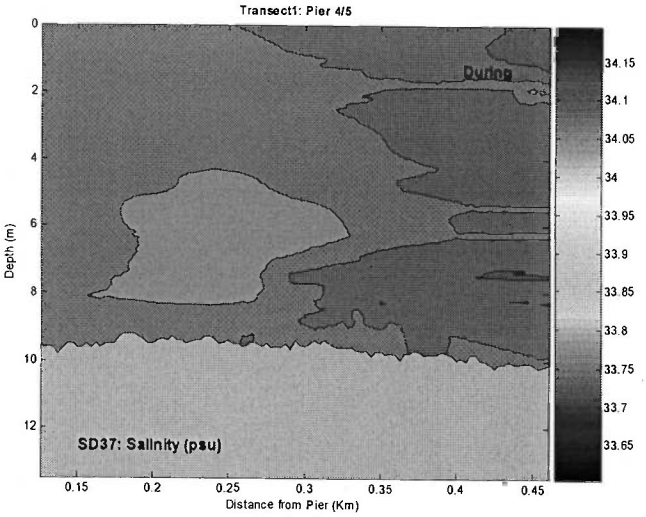
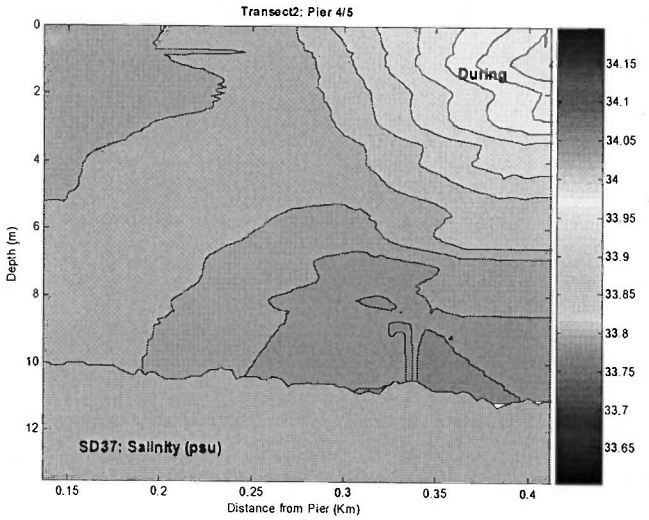
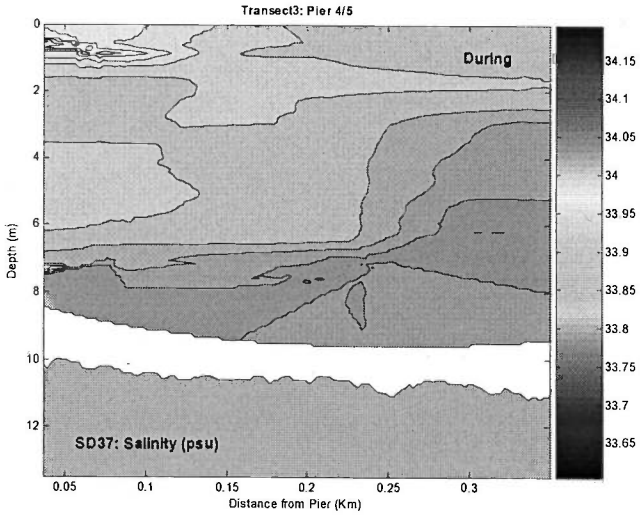
-117.13
-117.12
-117.11
0.9
0.8
0.7
0.6
0.5
0.4

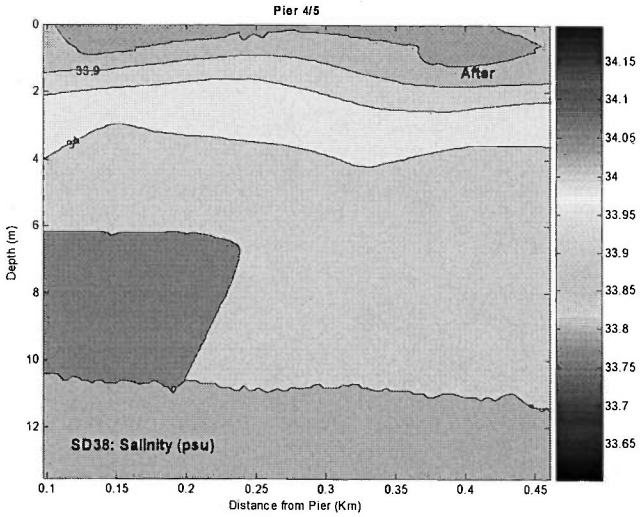

SD38: Oil - 5109
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.9
0.8
0.7
0.6
0.5
0.4

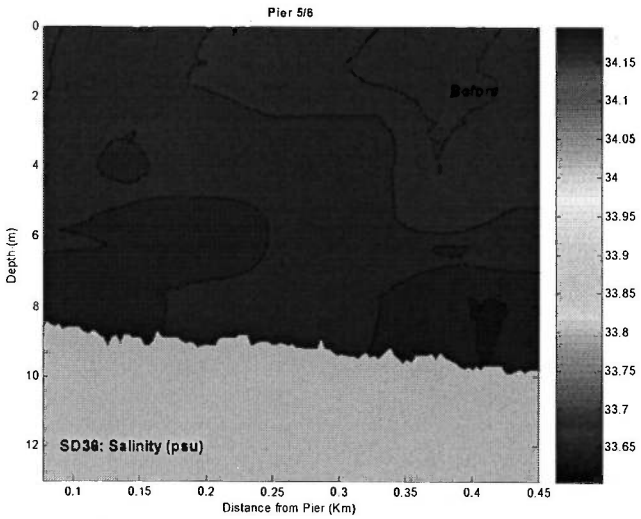

Pier 4/5
Before
SD36: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

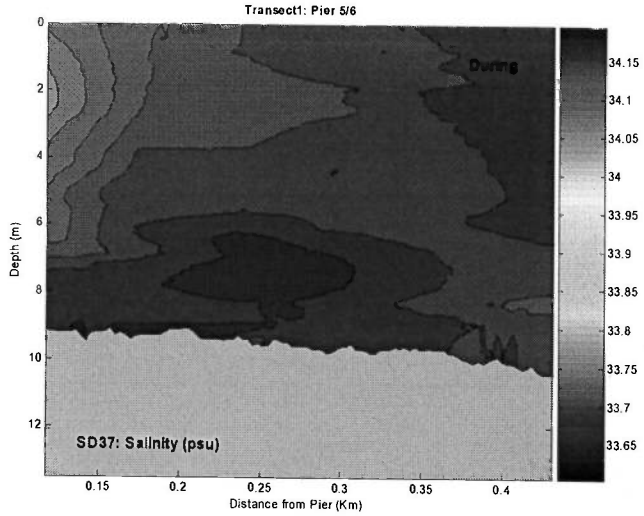

Transect1: Pier 4/5
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.15
0.2
0.25
0.3
0.35
0.4
0.45
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

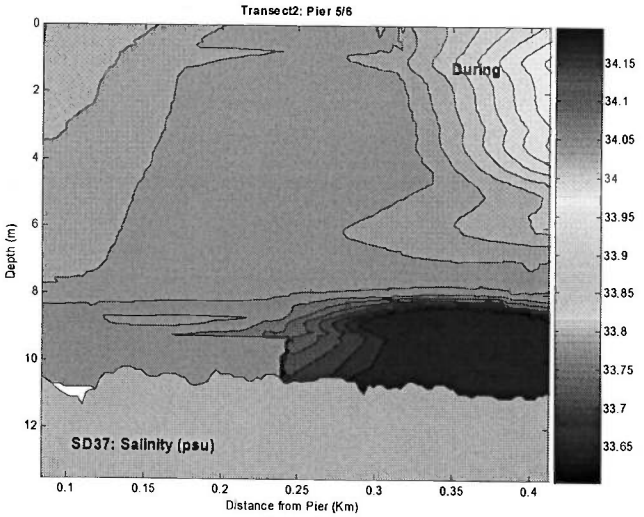

Transect2: Pier 4/5
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.15
0.2
0.25
0.3
0.35
0.4
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

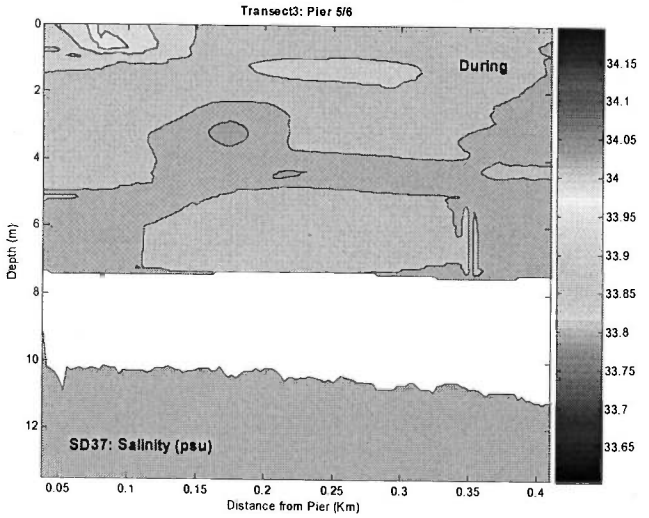

Transect3: Pier 4/5
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.05
0.1
0.15
0.2
0.25
0.3
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

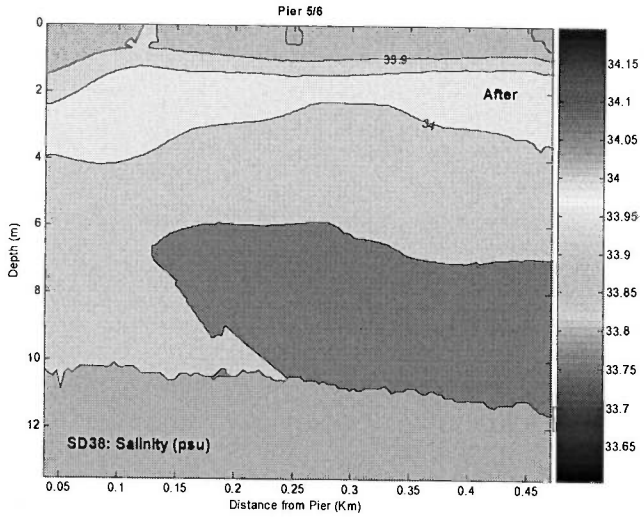

Pier 4/5
33.9
After
SD38: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

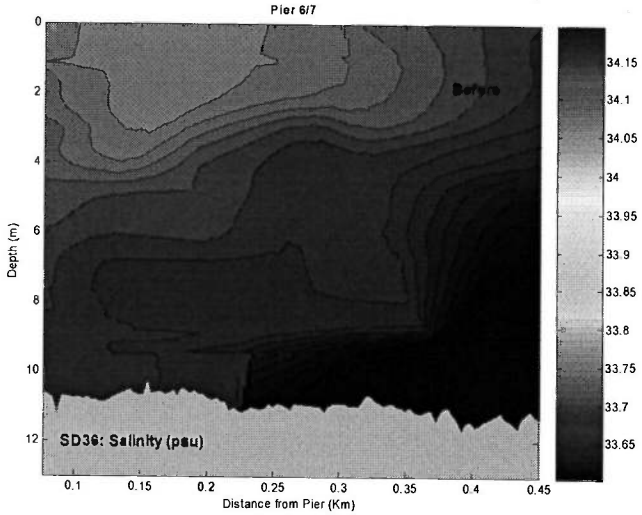

Pier 5/6
Before
SD39: Salinity (psu)
Depth (m)
Distance from Pier (Km)
0
2
4
6
8
10
12
0.1
0.15
0.2
0.25
0.3
0.35
0.4
0.45
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

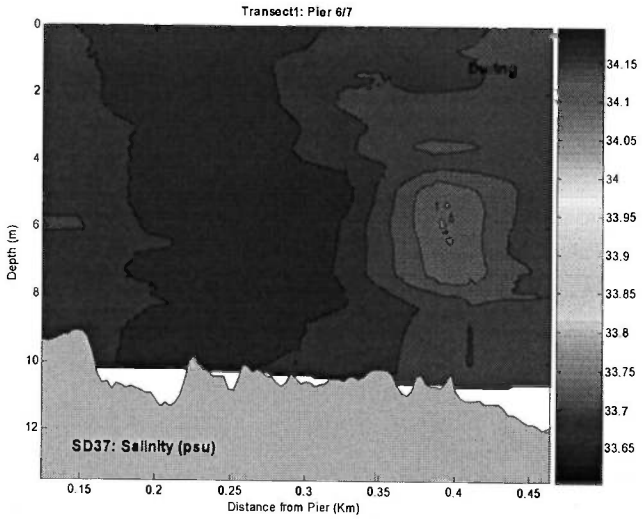

Transect1: Pier 5/6
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

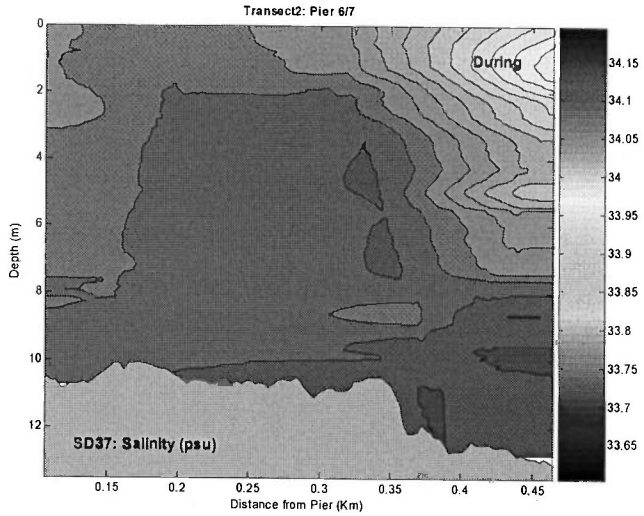

Transect2: Pier 5/6
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

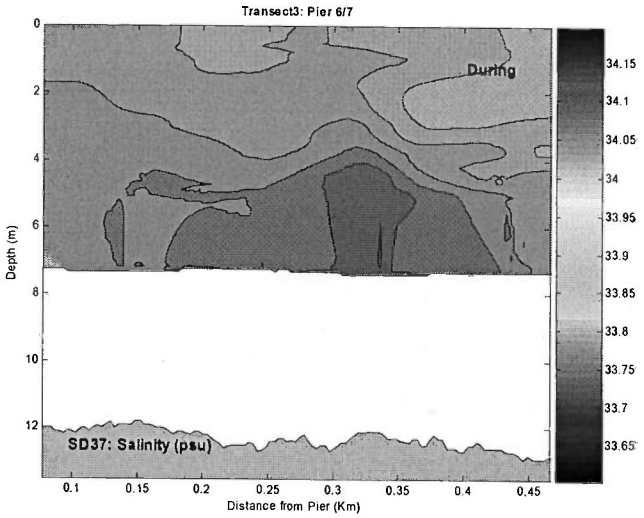

Transect3: Pier 5/6
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

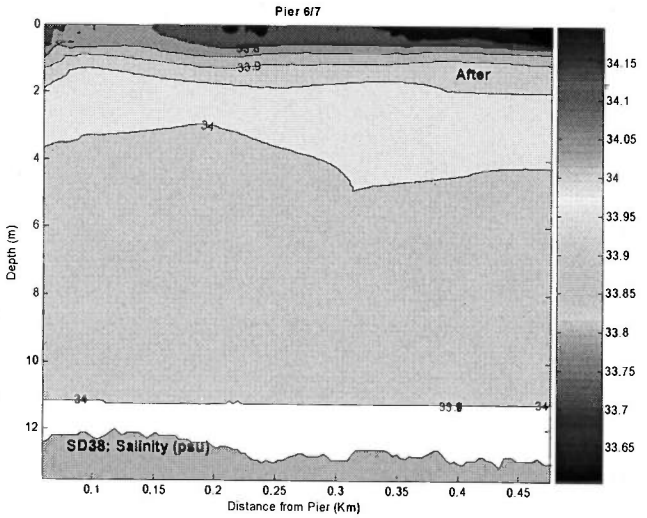

Pier 5/6
After
SD38: Salinity (psu)
Depth (m)
Distance from Pier (Km)


Pier 6/7
SD36: Salinity (psu)
Depth (m)
Distance from Pier (Km)

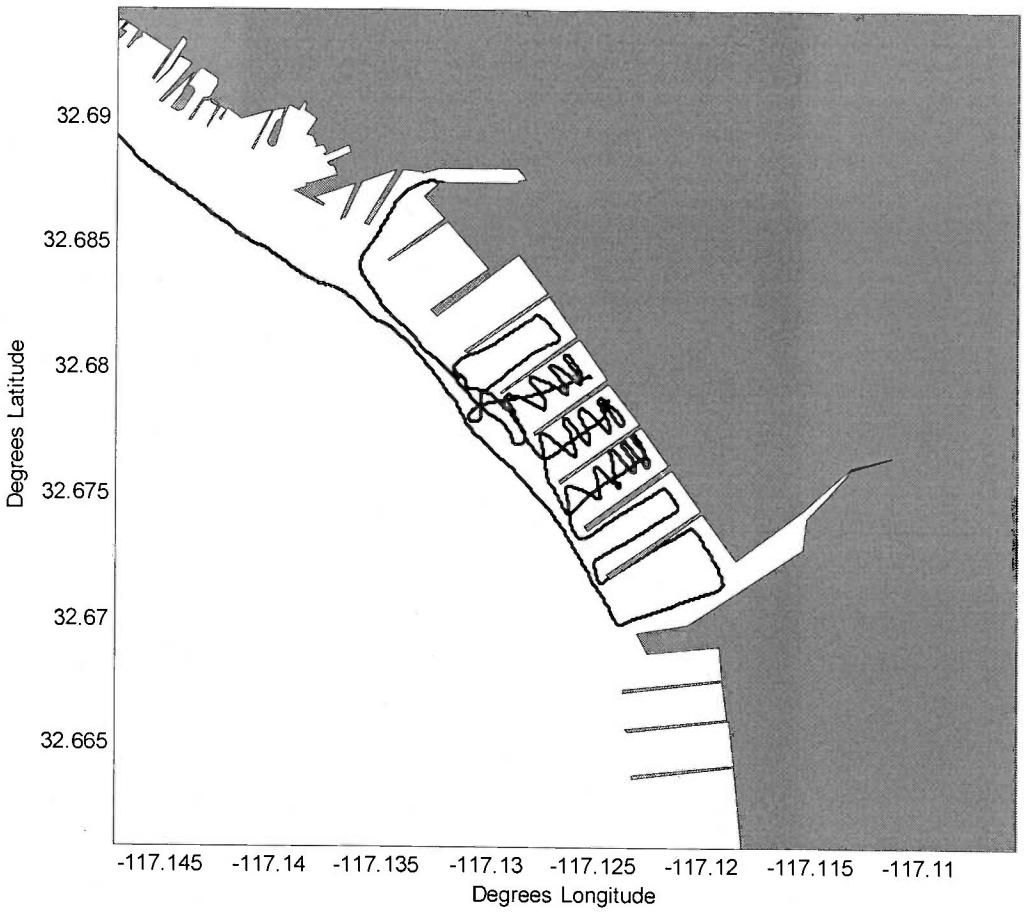

Transect1: Pier 6/7
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)

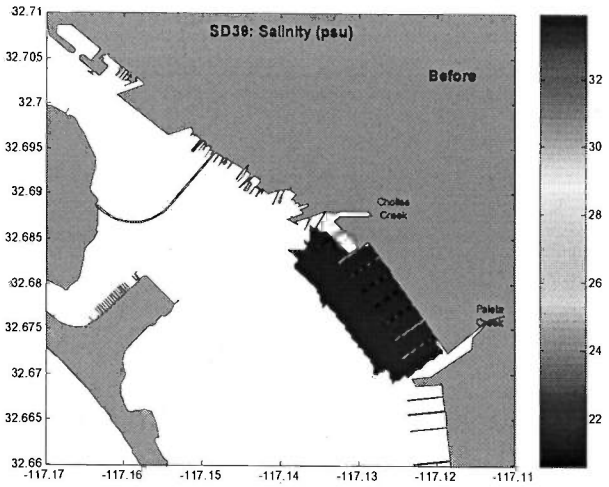

Transect2: Pier 6/7
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

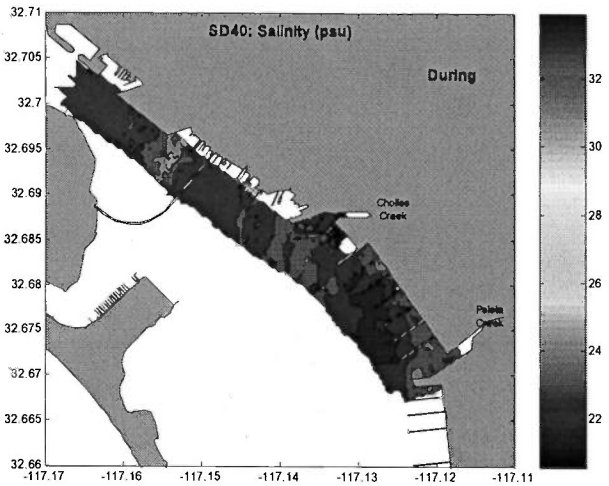

Transect3: Pier 6/7
During
SD37: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

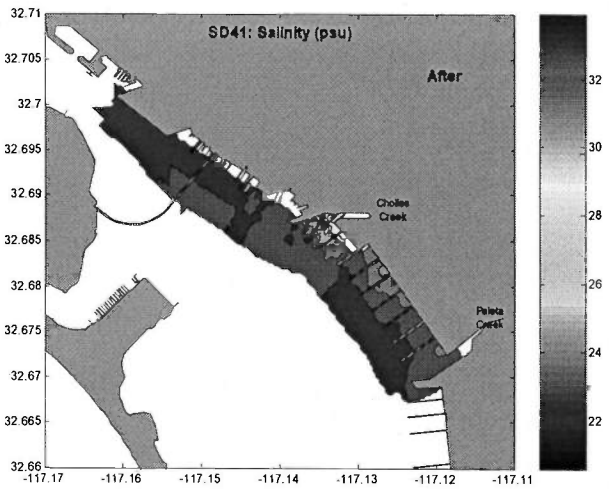

Pier 6/7
After
SD38: Salinity (psu)
Depth (m)
Distance from Pier (Km)
34.15
34.1
34.05
34
33.95
33.9
33.85
33.8
33.75
33.7
33.65

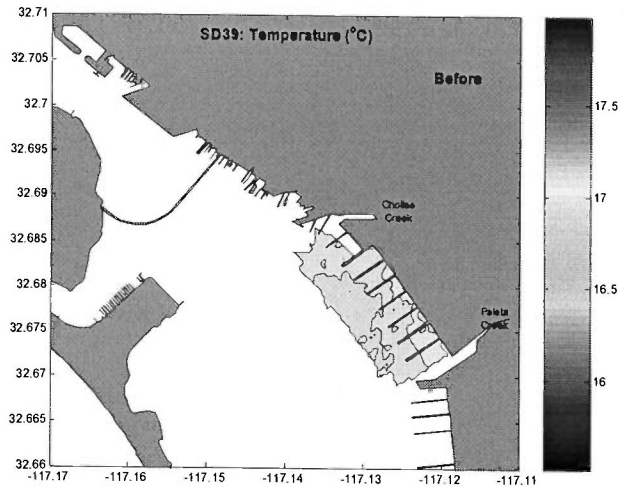
SDB2- 2/24/2004

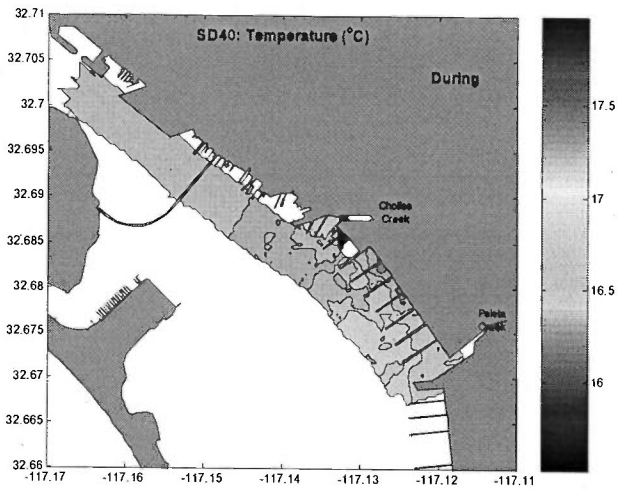


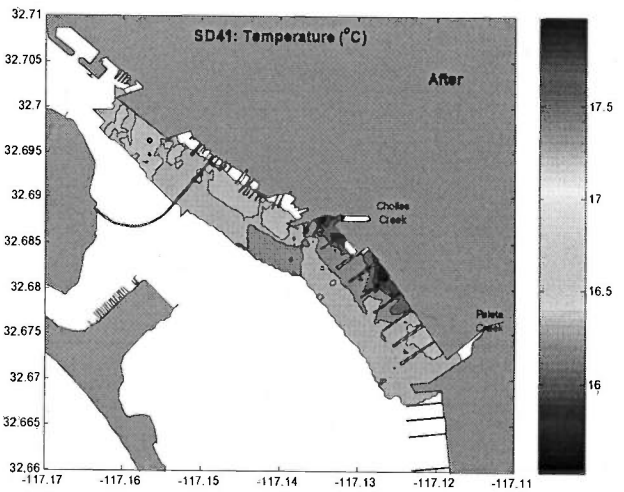

SD39: Salinity (psu)
Before
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
32
30
28
26
24
22

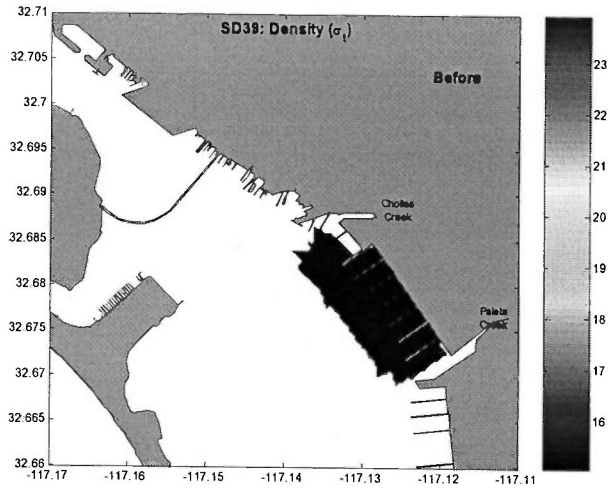

SD40: Salinity (psu)
During
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
32
30
28
26
24
22

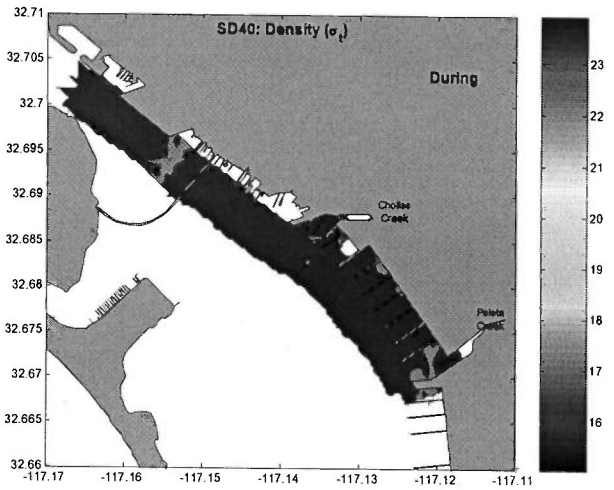

SD41: Salinity (psu)
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
32
30
28
26
24
22

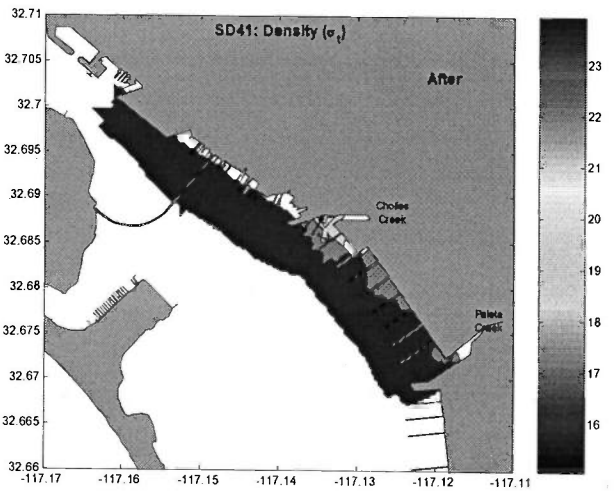

SD39: Temperature (°C)
Before
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
17.5
17
16.5
16

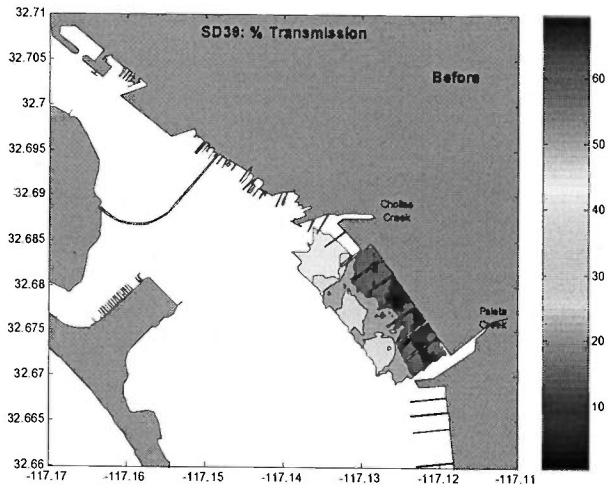

SD40: Temperature (°C)
During
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
17.5
17
16.5
16

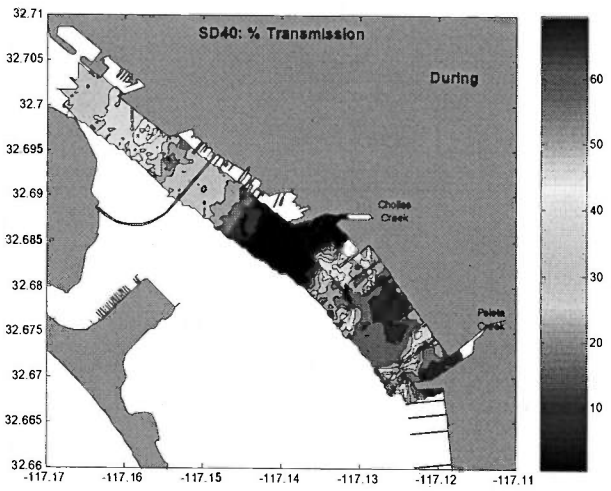

SD41: Temperature (°C)
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
17.5
17
16.5
16

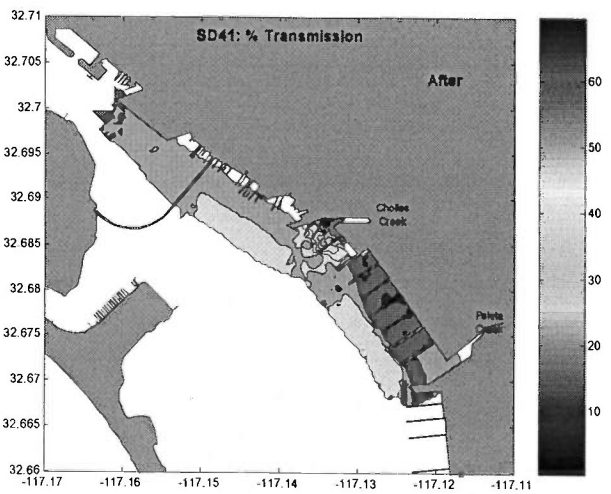

SD39: Density (σ_t)
Before
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
23
22
21
20
19
18
17
16

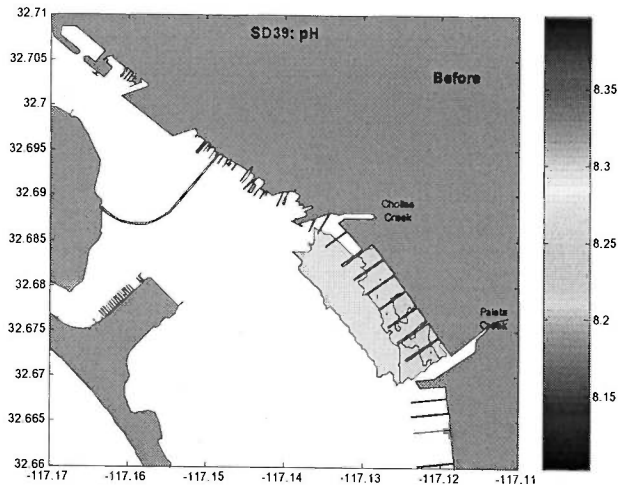

SD40: Density (σ_t)
During
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
23
22
21
20
19
18
17
16

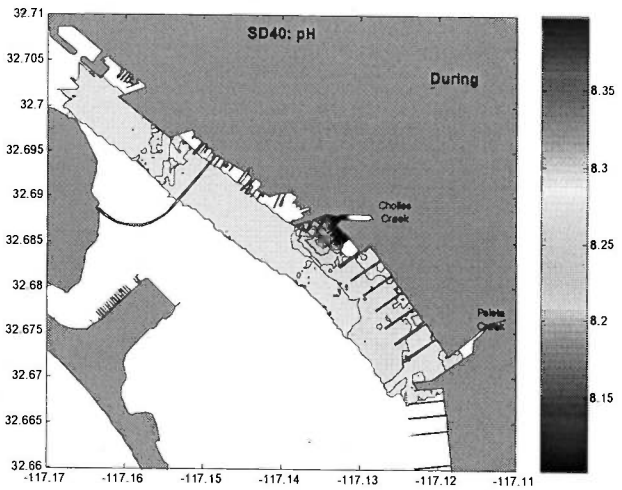

SD41: Density (σ_t)
After
Cholas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
23
22
21
20
19
18
17
16

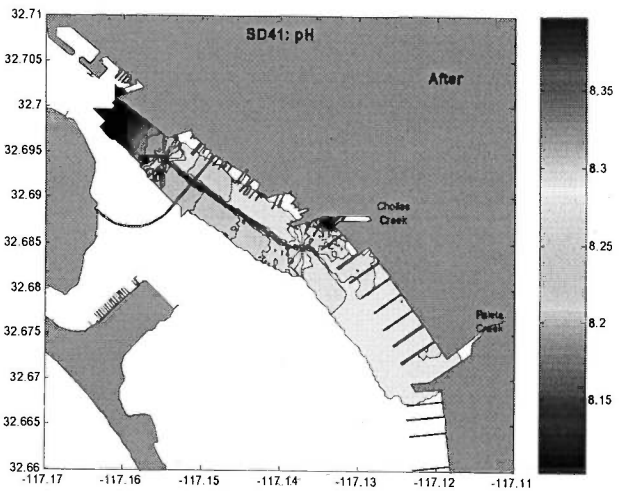

SD39: % Transmission
Before
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
60
50
40
30
20
10

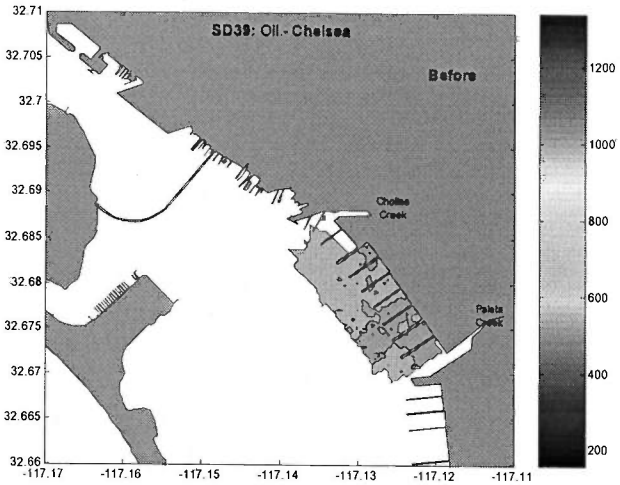

SD40: % Transmission
During
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
60
50
40
30
20
10

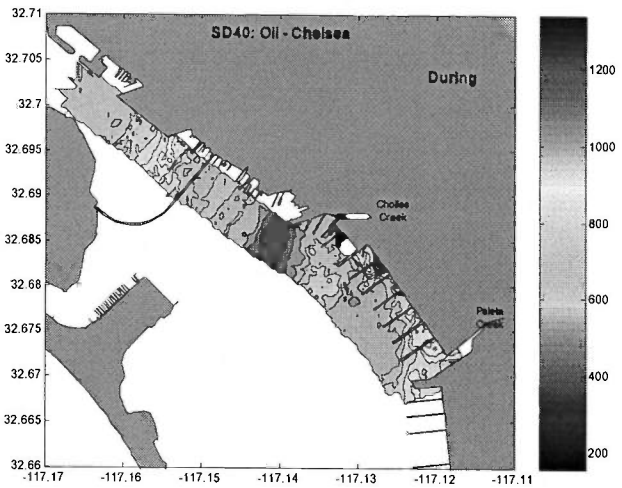

SD41: % Transmission
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
60
50
40
30
20
10

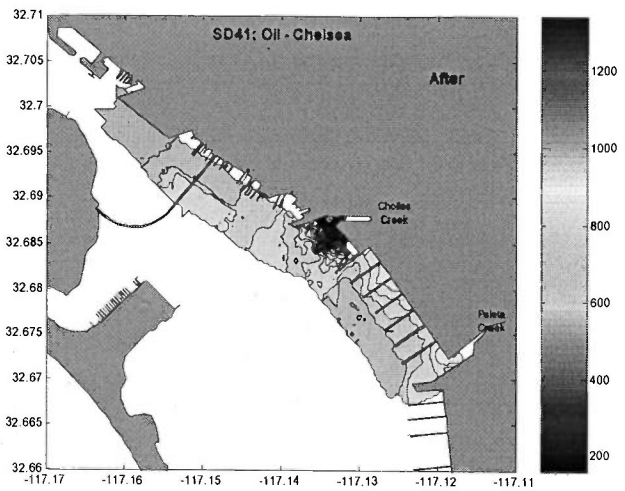

SD39: pH
Before
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8.35
8.3
8.25
8.2
8.15

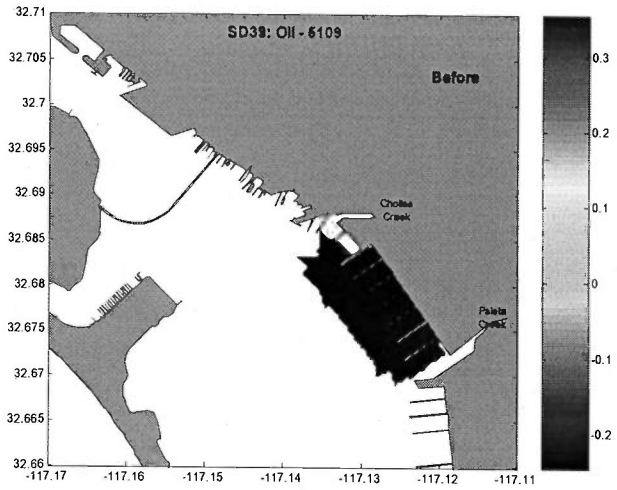

SD40: pH
During
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8.35
8.3
8.25
8.2
8.15

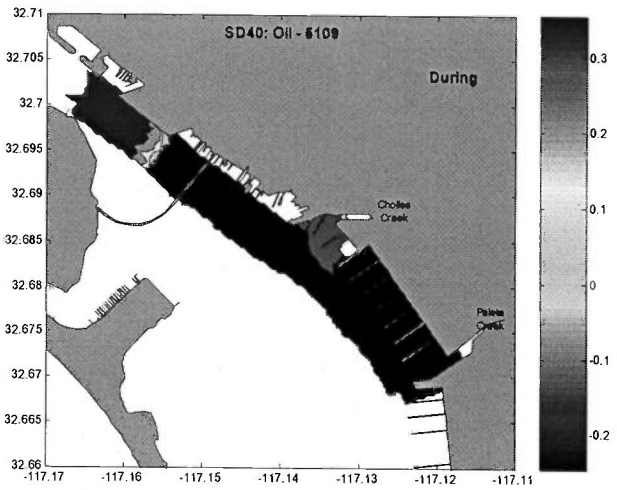

SD41: pH
After
Chollas Creek
Paleta Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
8.35
8.3
8.25
8.2
8.15

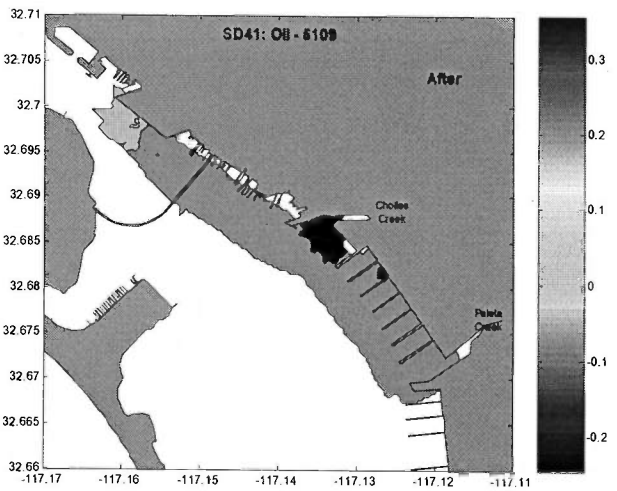

SD39: Oil - Chelsea
Before
Chelsea Creek
Pajaro Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1000
800
600
400
200

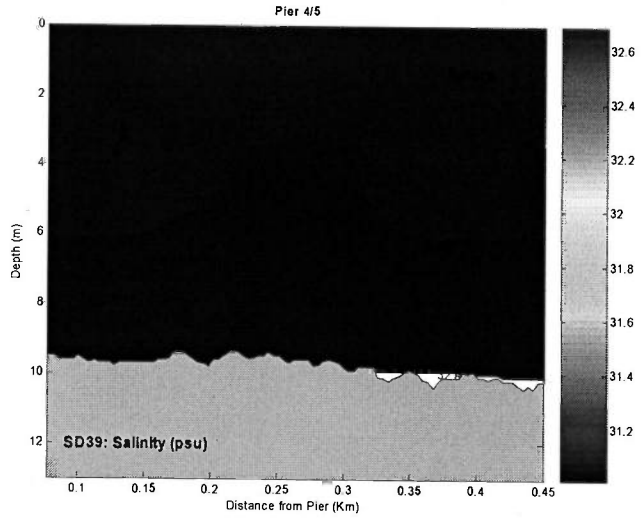

SD40: Oil - Chelsea
During
Chelsea Creek
Pajaro Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1000
800
600
400
200

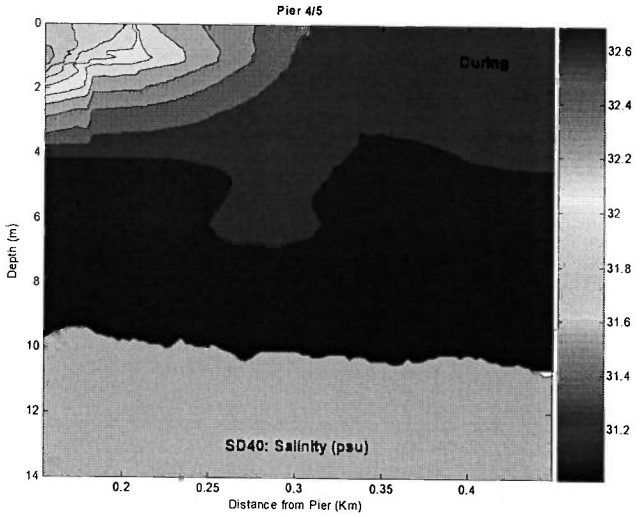

SD41: Oil - Chelsea
After
Chelsea Creek
Pajaro Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
1200
1000
800
600
400
200

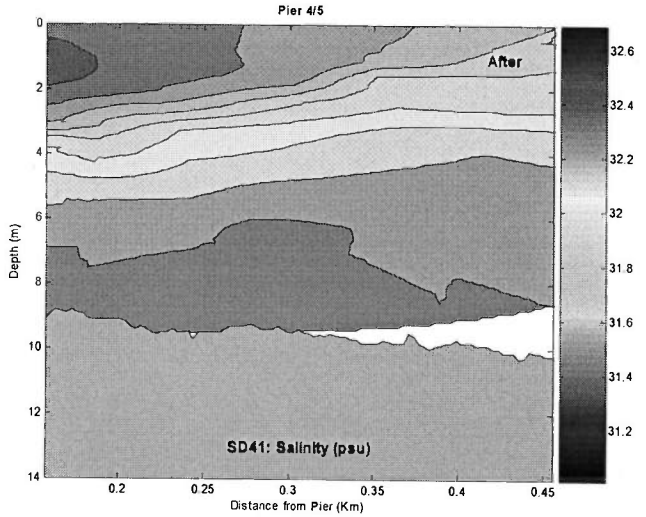

SD39: Oil - 5109
Before
Cholla Creek
Palela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.3
0.2
0.1
0
-0.1
-0.2

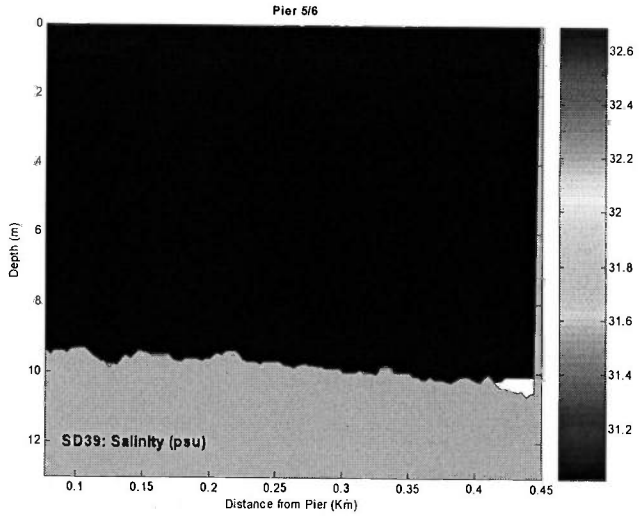

SD40: Oil - 5109
During
Cholla Creek
Palela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.3
0.2
0.1
0
-0.1
-0.2

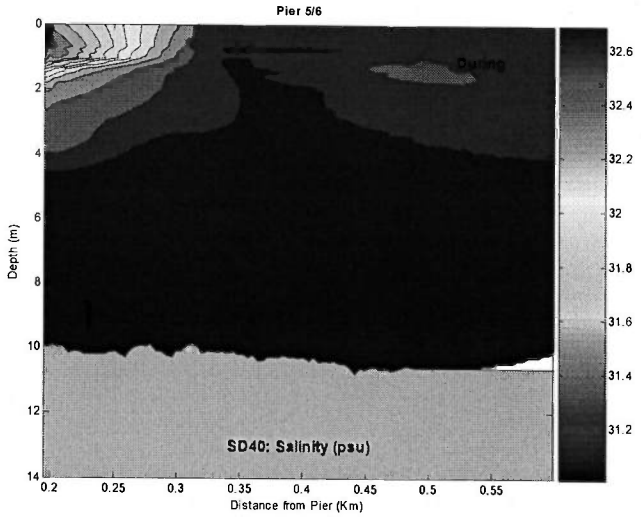

SD41: Oil - 5109
After
Cholla Creek
Palela Creek
32.71
32.705
32.7
32.695
32.69
32.685
32.68
32.675
32.67
32.665
32.66
-117.17
-117.16
-117.15
-117.14
-117.13
-117.12
-117.11
0.3
0.2
0.1
0
-0.1
-0.2

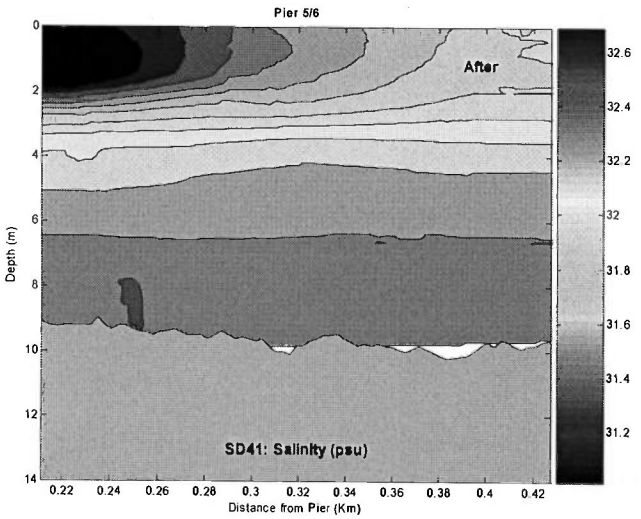

Pier 4/5
Depth (m)
SD39: Salinity (psu)
Distance from Pier (Km)
32.6
32.4
32.2
32
31.8
31.6
31.4
31.2

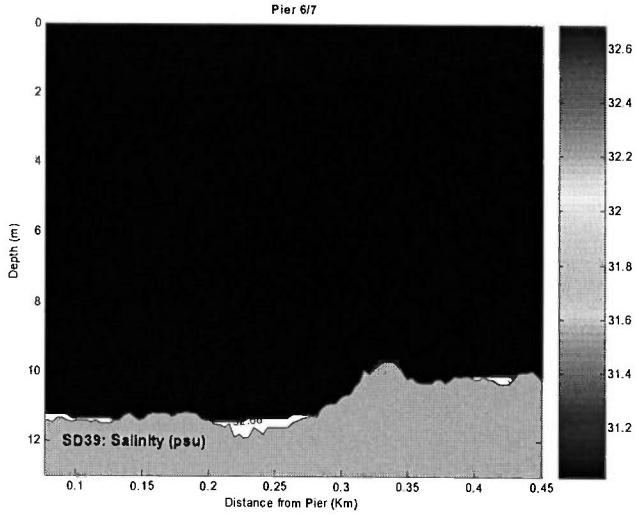

Pier 4/5
During
Depth (m)
SD40: Salinity (psu)
Distance from Pier (Km)
32.6
32.4
32.2
32
31.8
31.6
31.4
31.2

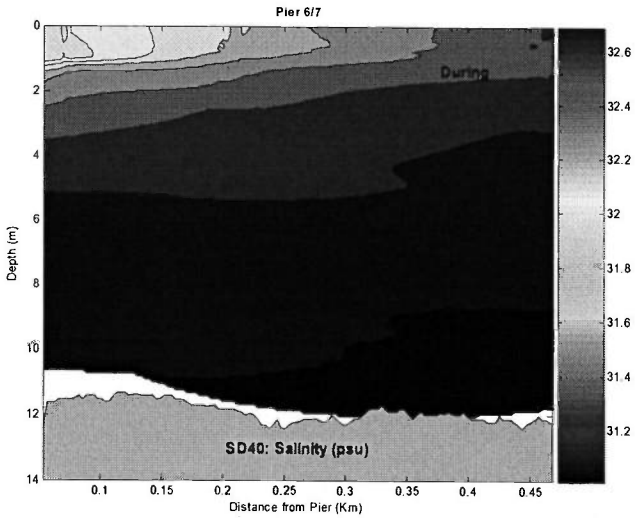

Pier 4/5
After
Depth (m)
SD41: Salinity (psu)
Distance from Pier (Km)
32.6
32.4
32.2
32
31.8
31.6
31.4
31.2

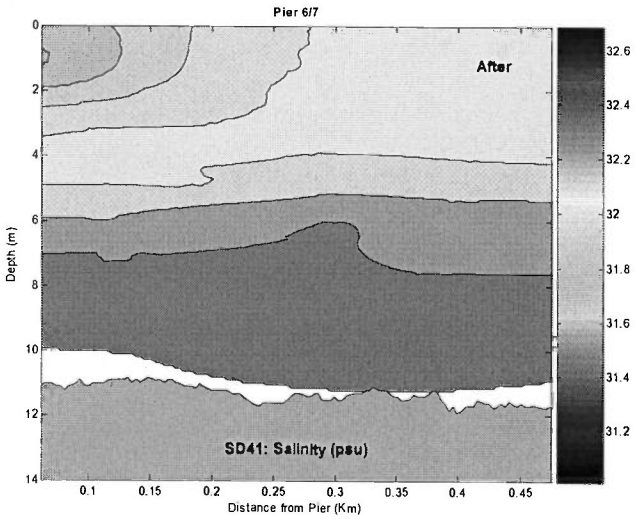

Pier 5/6
Depth (m)
Distance from Pier (Km)
SD39: Salinity (psu)


Pier 5/6
Depth (m)
Distance from Pier (Km)
SD40: Salinity (psu)


Pier 5/6
After
Depth (m)
Distance from Pier (Km)
SD41: Salinity (psu)


Pier 6/7
SD39: Salinity (psu)
Depth (m)
Distance from Pier (Km)


Pier 6/7
During
SD40: Salinity (psu)
Depth (m)
Distance from Pier (Km)

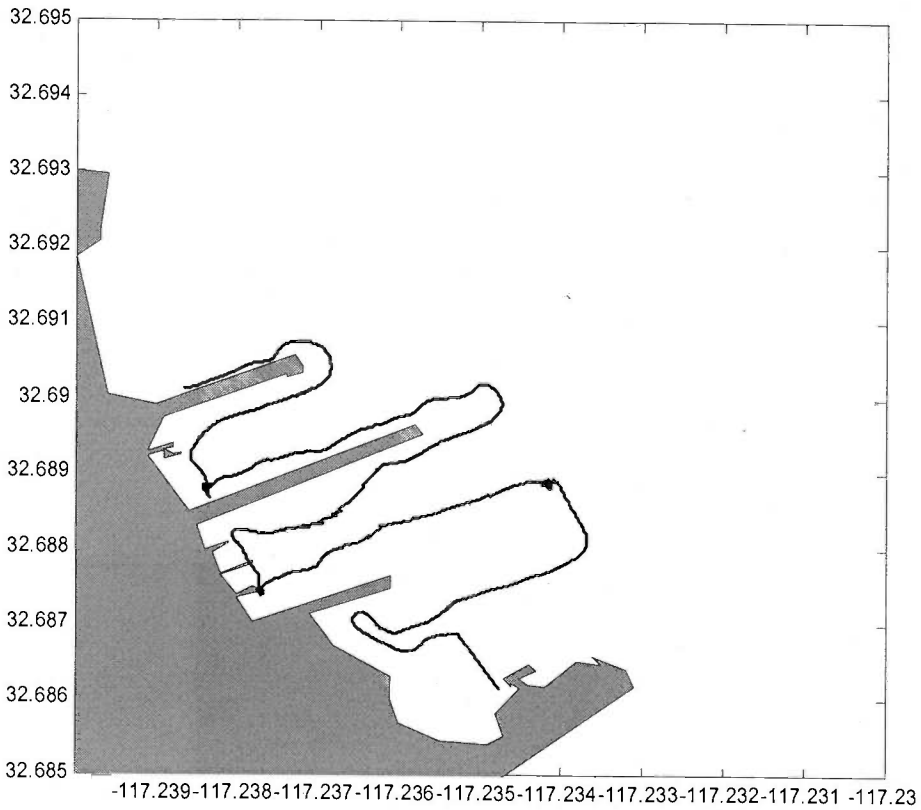

Pier 6/7
After
SD41: Salinity (psu)
Depth (m)
Distance from Pier (Km)

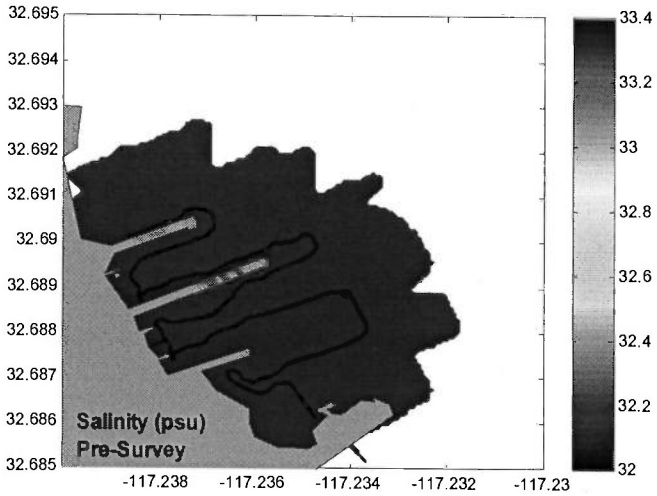
Appendix G2

SUB

SDB2- 2/24/2003

SDB2- 2/24/2004

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Salinity (psu)
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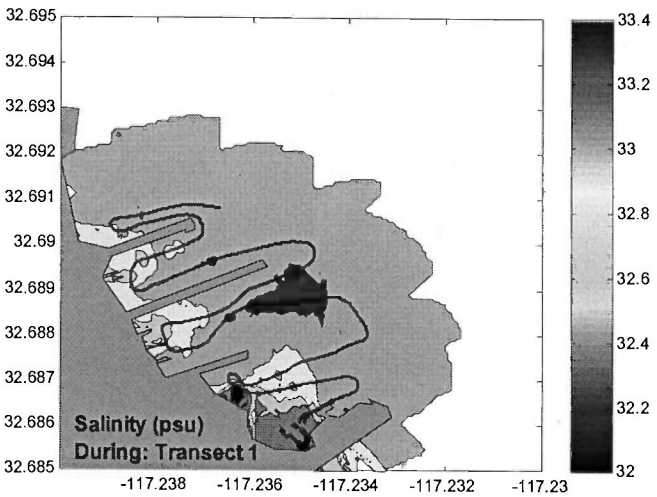
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Salinity (psu)
During: Transect 1
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-117.236
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-117.232
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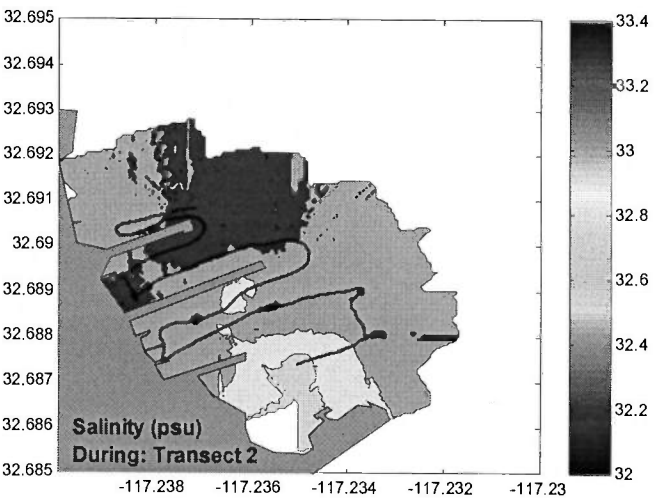
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Salinity (psu)
During: Transect 2
-117.238
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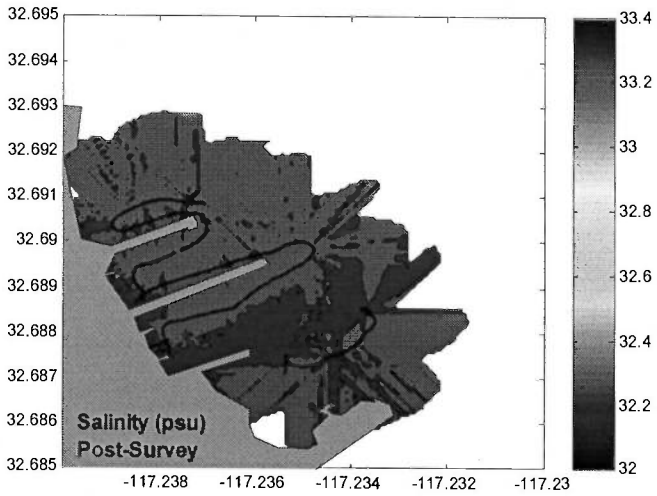
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Salinity (psu)
Post-Survey
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-117.23
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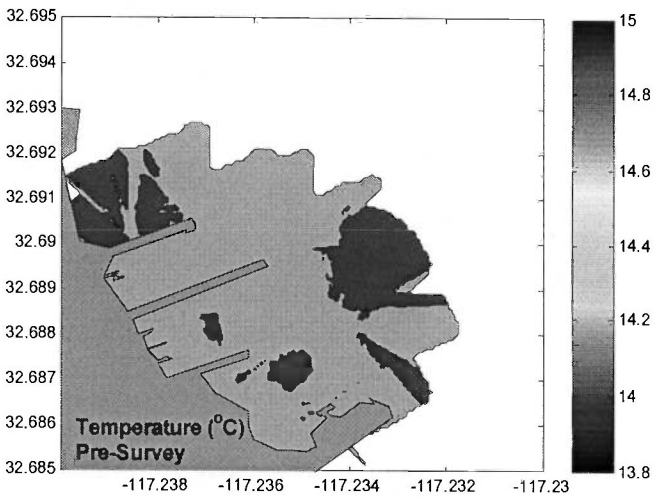
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Temperature (°C)
Pre-Survey
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-117.236
-117.234
-117.232
-117.23
15
14.8
14.6
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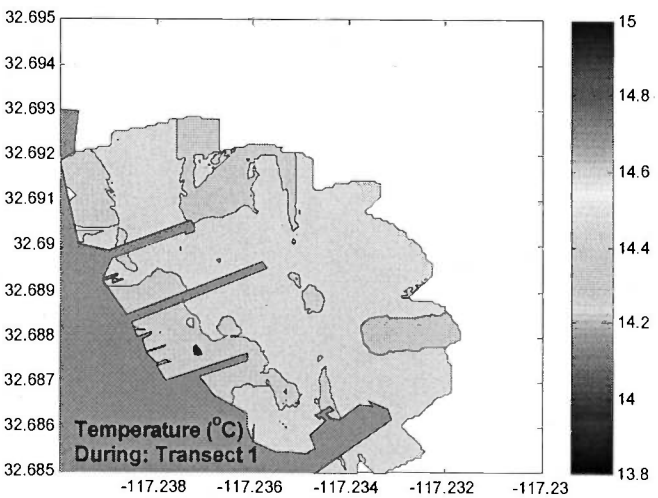
13.8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Temperature (°C)
During: Transect 1
-117.238
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-117.234
-117.232
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14.8
14.6
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