92-06

United States
Environmental Protection
Agency

EPA/600/R-94/058
July 1994

Research and Development

BIOLOGICAL AND CHEMICAL ASSESSMENTS OF SEDIMENT COLLECTED FROM ELEVEN LOCATIONS IN PAMLICO SOUND, NORTH CAROLINA

Prepared for
U.S. Environmental Protection Agency
Region IV

Prepared by
Environmental Research Laboratory
Gulf Breeze, FL 32561

EPA/600/X-92/076
July 1994

BIOLOGICAL AND CHEMICAL ASSESSMENTS OF SEDIMENT COLLECTED FROM ELEVEN LOCATIONS IN PAMLICO SOUND, NORTH CAROLINA

by

James C. Moore, E.M. Lores, J. Forester and R. Stanley
U.S. Environmental Protection Agency
Environmental Research Laboratory
Gulf Breeze, FL 32561

Barbara Albrecht, and
Peggy S. Harris
TRI, Inc.
Sabine Island
Gulf Breeze, FL 32561

Submitted to:
F. Theodore Bisterfeld
Project Officer, Coastal Section
U.S. Environmental Protection Agency
Region IV
345 Courtland Street, N.E.
Atlanta, GA 30365

U.S. ENVIRONMENTAL PROTECTION AGENCY
ENVIRONMENTAL RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
GULF BREEZE, FLORIDA 32561-5299

DISCLAIMER

The research described in this article has not been subjected to Agency review and is intended for internal Agency distribution. It should be considered a draft document and should not be cited or quoted. Mention of trade names does not constitute endorsement or recommendation for use.

EXECUTIVE SUMMARY

Sediments from 11 locations within Pamlico Sound, North Carolina were used in a four-day acute toxicity test with *Mysidopsis bahia* and 10-day acute toxicity tests with *Ampelisca abdita*, and *Neanthes arenaceodentata*. Chemical analyses were performed on each sediment sample for selected pesticides, PCBs, total aliphatic and aromatic petroleum hydrocarbons, and heavy metals.

The sediments were not acutely toxic to *M. bahia* or to *N. arenaceodentata* in the 10-day acute tests. Four sediments caused a statistically significant reduction in mean percentage survival of *A. abdita* when data were analyzed with the t-test: Hancock Creek ($p = 0.0545$) and E. Prong Slocum Creek, Kennedy Creek, and Oriental Harbor ($p < 0.05$). Mortality ranged from 12 to 37%.

Mean percentage survival in Kennedy Creek sediment (94%) was higher than the mean percentage survival in other sediments that did not cause statistically significant reduction in survival. This statistical difference is attributed to the variances differing among the locations tested: 0.200 for Kennedy Creek, 1.80 for Middle Pamlico River, and 4.20 for Upper Slocum Creek.

MEAN PERCENTAGE SURVIVAL

Sediment Location	A. abdita	M. bahia	N. arenaceodentata
Reference	99	100	100
Upper Slocum Creek	91	100	100
Middle Pamlico River (30A-30B)	96	100	100
E. Prong Slocum Creek	[a]63	100	100
Chocowinity Bay	94	100	100
Green & Kershaw Creeks	97	100	100
Kennedy Creek	[a]94	100	100
Middle Pamlico River (TG 1-2A)	93	100	100
Hancock Creek	[b]89	100	100
Middle Pamlico River (21-24)	96	100	100
Oriental Harbor	[a]88	100	100
Broad Creek	97	100	100

[a]Statistically significant at $P<.05$.
[b]Statistically significant at $P=.0545$.

The reasons for the acute toxicity are not known. However sediment from E. Prong Slocum Creek SLO (18-19) contained much higher concentrations of total petroleum hydrocarbons than any of the other sediments (3680 $\mu g/g$ dry weight).

Based on these acute toxicity tests, we believe the survival of Ampelisca in sediments from E. Prong Slocum Creek, Kennedy Creek, Hancock Creek, and Oriental Harbor could be reduced if exposure is 10 days, and that benthic life may be adversely affected at these locations.

INTRODUCTION

Sediment collected from eleven locations within Pamlico Sound were analyzed for toxicity to three invertebrate species, chemical contamination, and physical characteristics, at the request of EPA Region IV and the Albemarle and Pamlico Sounds National Estuary Program. Coxicity was determined from acute toxicity tests conducted with the sediments and each of the following estuarine crustaceans, M. bahia and A. abdita, and the marine polychaete, N. arenaceodentata. Neanthes arenaceodentata, although not specified for testing in the original study plan, was included to obtain toxicity data on a benthic organism in addition to the tube-dwelling, A. abdita. Mysidopsis bahia is epibenthic. Each sediment was chemically analyzed for residues of selected organochlorine pesticides, PCB's aliphatic and aromatic hydrocarbons, and metals, and physically characterized by determining moisture, carbon, and sand and silt/clay content.

MATERIALS AND METHODS

Test Sediments

Eleven sediment samples were collected in tributaries to Pamlico Sound, by the North Carolina Division of Environmental Management field crew as coordinated through Ted Bisterfeld of Region IV. Each sample consisted of a one gallon Nalgene polyethylene jar filled to capacity with composite sediment from one of the 11 sites. Site names with core numbers given in parentheses are as follows: E. Prong Slocum Creek (SLO 18-19), Upper Slocum Creek (SLO 1-13), Hancock Creek (HCK 3-4-5), Oriental Harbor (CMP 1-2), Green and Kershaw Creeks (ORL 4-5), Kennedy Creek (NAT 10-12), Broad Creek (BRD 1-3), Chocowinity Bay (PAM 3), Middle Pamlico River (30A-30B), Middle Pamlico River (TG 1-2A), Middle Pamlico River (21-24), and Perdido Bay (a reference sediment from Florida).

Biological Test Methods

Within 24 hours of arrival, the chilled samples were homogenized using a Teflon paddle. After homogenization, a subsample of each sediment was removed for physical characterization and classification. Each sediment sample was analyzed for percentage moisture and carbon (measured by weight on ignition), sand and silt/clay content following the procedures of American Society for Testing Materials (ASTM 1990) and Folk (1957). The remaining homogenous mixture was then press-sieved through a 1.0 millimeter (mm) stainless steel screen to remove large particles and possible predators to the test organisms. An aliquot of the homogenized, press-sieved sample was removed for chemical analyses.

Triplicates of each sediment sample were used in three different acute toxicity tests: one of 96 hours duration with M. bahia and two of 10 days duration, one with A. abdita and one with N. arenaceodentata. The tests were conducted according to ASTM guidelines (ASTM 1988). Natural seawater from Santa Rosa sound, adjacent to the laboratory, was used in the tests. It was filtered to 20 μm; its salinity was adjusted to 20‰ for M. bahia, 30‰ for A. abdita, and 28‰ for N. arenaceodentata. All tests were conducted at a nominal temperature of 20°C.

The three species were selected for testing for the following reasons. Mysidopsis bahia was chosen because it is an ecologically important mysid crustacean, contributing to the estuarine food chain; it is toxicologically important because of its amenability to laboratory culture and toxicity test conditions, and its sensitivity to a variety of toxicants; and there is a rich comparative literature concerning its usage. Ampelisca abdita was chosen because it is an amphipod crustacean that dwells in tubes in the sediment and its feeds on particles in suspension and on the surface of the sediment. Neanthes arenaceodentata was chosen because it is a representative benthic infaunal species recommended in the EPA/COE dredge material testing guidelines (EPA/COE 1991).

Five-day old M. Bahia, cultured at ERL/GB, were acclimated to test conditions before use in the cute toxicity tests. Each test exposure replicate consisted of a 1.9 L glass Carolina bowl to which were added 200 mL of homogenous, press-sieved sediment, 800 mL of filtered seawater, and a mysid containment device. The containment device consisted of a glass petri plate (2.0 cm x 15 cm diameter) with a collar of nylon screen (Nitex with 316 μm openings); it was added to the test dish while there was high sediment turbidity to ensure passage of sediment through the screen collar and into the Petri plate where the mysids were contained. Ten M. bahia were added to each of three replicates approximately 1 h after the addition of the sediment. Daily, the mysids were counted and then fed, and the dissolved oxygen (DO), pH, and temperature were measured. Salinity was measured at 0, 48, and 96 h of the test.

Feral A. abdita were obtained from John Brezina Associates in Dillon Beach, California. Over a 5-h period, beginning with their arrival at the laboratory, the amphipods were acclimated from shipment conditions of 26‰ salinity and 15°C to test conditions of 30‰ salinity and 20°C. Each of five test replicates consisted of a 1-L mason jar containing 200 mL of test sediment and 700 mL of filtered seawater. Test sediments were allowed to settle 1 to 2 h before 20 acclimated A. abdita were added to each replicate. Test water was aerated and the amphipods were not fed during the test (ASTM 1990). This same test design is used in the Environmental Monitoring and assessment Program (EMAP). Daily, DO, pH, and temperature were measured. Salinity was measured on test days 0, 3, 7, and 10.

Juvenile N. arenaceodentata were acquired from cultures at the University of California. They were acclimated from shipment conditions to test conditions of 28‰ salinity and 20°C over a period of 7 days. Each of three test replicates consisted of a 1-L mason jar containing 200 mL of test sediment and 600 mL of filtered seawater. Ten acclimated N. arenaceodentata were added to each replicate. Test water was aerated during the test. Daily, the DO, pH, and salinity were measured in each replicate and, in replicate 1, the temperature was measured. Test water bath temperature was measured continuously. The polychaetes were fed powdered Tetramin® on test days 2, 4, 6, and 8.

Chemical Analyses

A. Chlorinated Hydrocarbon Pesticides and PCBs

Sediments were air-dried and blended as necessary, then weighed into 150-mm by 25-mm Teflon-lined screw top test tube. Twenty mℓ of 20% (v/v) acetone in petroleum ether were added and samples were tumbled on a Rotorack at 60-90 rpm for 30 minutes. Samples were then centrifuged (1600 x g) for 10 minutes. The solvent extracts were transferred to an oil sample bottle containing 50 mℓ of 2.0% (w/v) aqueous sodium sulfate. This extraction was repeated twice more. The solvent extracts were hand shaken for one minute. The solvent was transferred to a 25 mℓ-concentrator tube after the two phases separated. Five mℓ of petroleum ether were added to the sodium sulfate solution and a second extraction of the aqueous phase performed. A second 5 mℓ extraction was performed. Sample extracts from the three aqueous washes were combined and concentrated to 1 mℓ on a nitrogen evaporator in preparation for Florisil® cleanup. Samples analyzed with electron-capture detectors were shaken with 500 μℓ of elemental mercury to remove sulfur before gas chromatographic analysis. Cleanup columns were prepared by adding 3 g of PR-grade Florisil (stored at 130°C) and 2 g of anhydrous sodium sulfate (powder) to a 200-mm by 9-mm i.d. Chromaflex column (Kontes Glass Co., Vineland, NJ) and rinsing with 10 ml of hexane. Sediment extracts were transferred to the column with two additional 2-mℓ volumes of hexane. Pesticides and PCBs were eluted with 20 mℓ of 5% (v/v) diethyl ether in hexane. Dieldrin and Endosulfan were eluted with 20 ml of 10% (v/v) isopropanol in isooctane.

Quantification of pesticides was made with external standard methods. All standards were obtained from the EPA pesticide repository. Analyses were performed on a Hewlett-Packard Model 5890 gas chromatograph equipped with a ^{63}Ni electron-capture detector. Separations were performed by using 30 m (0.32 mm i.d.) RTX-5 and 30 m (0.32 mm i.d.) RTX-1 fused silica capillary columns. Other gas chromatographic parameters were: helium carrier gas flowing at 1.5 mℓ/min; column temperature was programmed at 50°C (held for 2 min),10°/min to 150°C, 2°/min to 260°C (held for 3 min). Inlet temperature 250°C; and detector temperature, 350°C; 10% methane in argon makeup gas was flowing at 60 mℓ/min.

B. Heavy Metals

One to two grams of wet sediment were weighed into a 40-mℓ reaction vessel. Five mℓ of concentrated nitric acid (Baker Chemical Instra-Analyzed) were added and the samples digested uncapped for 2 to 4 h at 70°C in a tube heater. Digestion was continued, with vessels capped, for 48 h at 70°C. After digestion, samples were transferred to graduated 15-mℓ centrifuge tubes and a 5-mℓ aliquot was diluted to 50 mℓ for aspiration into a Jarrell-Ash AtomComp 800 Series inductively-coupled argon-plasma emission spectrometer (ICP), an instrument that acquires data for 15 elements simultaneously. No detectable residues could be found in method blanks. Dilute hydrochloric acid was aspirated between samples to prevent carryover of residues from one sample to the next. Standards in 10% nitric acid were used to calibrate the instrument initially and adjustments were made when necessary. Concentrations are reported in two significant figures as our method allows, and were not corrected for percentage recovery. Concentrations of metals in wet sediment were corrected for moisture content using mean values from Table 1.

C. Petroleum Hydrocarbons

Ten grams of wet sediment were weighed into culture tubes and extracted as described by J.S. Warner (1976). Sample extracts were concentrated to approximately 0.50 mℓ for gas chromatographic analyses. Analyses were performed using a Hewlett Packard Perkin gas chromatograph (GC) equipped with flame ionization detectors (FID). Separations were performed by using a 30-m (0.32-mm i.d.) RTX-5 fused silica capillary column. Helium carrier gas was used at a flow of 1.5 mℓ/min. Other gas chromatographic parameters were: oven temperature programmed from 50°C (hold for 2 min) at a rate of 20°/min to 315°C (hold for 5 min); injector temperature was 250°C and detector temperature was 350°C.

<u>Quality Assurance of Chemical Analyses</u>

All standards used for quantitations of pesticides were obtained from EPA's Pesticide Repository in Las Vegas, Nevada. Standard solutions of metals were obtained from J.T. Baker Chemical Co., Phillipsburg, NJ, and were Instra-Analyzed quality. Five-alpha androstane was obtained from Sigma Chemical Company, St. Louis, MO, and was used as an internal standard to quantitate petroleum hydrocarbons. Reagent and glassware blanks were analyzed to verify that the analytical system was not contaminated with chemical residues that could interfere with quantifications.

Sediment Characterization

Percentage sand and silt/clay measurements were performed according to procedures described by Folk (1957). Percentage moisture was the difference in weight of the sediment wet and dry relative to the weight of the wet sediment. The sediment was dried approximately 48 h at 100°C. Percentage carbon was measured as the weight loss on ignition which was the difference in weight of the sediment dry and burned relative to the weight of the dry sediment. The sediment was burned by heating it to 537°C for 4 h (Buchanan and Kain 1971).

Statistical Analyses

Data were analyzed according to guidance described in "Evaluation of Dredged Material Proposed for Ocean Disposal," (U.S. EPA 1991). When variances were not homogeneous, data were transformed using arcsin $\sqrt{x}$. Statistical differences between mean number of survivors from five replicates of test sediment were determined by using t-test. Statistical analyses were performed by using Statistical Analysis System (SAS) procedures (SAS, 1990).

RESULTS AND DISCUSSION

Mean percentage moisture, carbon, sand and silt/clay were measured for each sediment (Table 1). Mean moisture content varied from 35.1% to 87.5%; mean percentage carbon content ranged from 1.9 to 21.2; mean percentage sand varied from 4.0 to 89.3; and mean percentage silt/clay varied from 10.7 to 96.0.

Data from sediment toxicity tests with M. bahia, A. abdita and N. arenaceodentata, are presented in Tables 2 and 3. Mortality observed with A. abdita was statistically significant ($P < .05$) when using t-test with arcsin $\sqrt{x}$ transformed data. Results of all reference toxicant tests with the anionic surfactant, sodium dodecyl sulfate (SDS), show that mysids and amphipods were healthy and responded in the expected range of sensitivity. Test concentrations of SDS were 0.0 μg/mℓ, 1.25 μg/mℓ, 2.5 μg/mℓ, and 5.0 μg/mℓ for both M. bahia and A. abdita. LC50 values for M. bahia were between 2 and 10 mg/ℓ; for A. abdita they were between 2 and 5 mg/ℓ.

Water quality was acceptable during the test (tables 4, 5, and 6).

Results of chemical analyses showed that no significant concentrations of pesticides or PCB congeners were detected in sediments (Table 7). Heavy metals were detected in every sediment (Table 8). Values were highest for copper, chromium, lead and nickel in Slocum Creek. Sediment from Upper Slocum Creek had the second highest values for chromium. Highest values for arsenic were determined in middle Pamlico River sediment. Kennedy Creek sediment had the second highest values for lead. Replicate chemical analyses performed on each sediment for metals are in close agreement except for zinc in E. Prong Slocum Creek sediment. This variability is attributed to sample replication not analytical error. One order of magnitude higher concentrations of aliphatic and aromatic petroleum hydrocarbons were determined in sediment from E. Prong Slocum Creek (Table 9; Core # SLO 18-19) than in other sediments. Several sediments contained aromatic hydrocarbons but no aliphatic hydrocarbons: Middle Pamlico River (30A-30B and 21-24), Hancock Creek, and Broad Creek. Sediment from Green & Kershaw Creeks, and Middle Pamlico River (TG 1-2A) did not contain detectable residues of petroleum hydrocarbons.

REFERENCES

ASTM. 1980. Conducting Acute Toxicity Tests with Fishes Macroinvertebrates, and Amphibians. E 729-80. Am. Soc. Test. Mater., Philadelphia, PA.

ASTM. 1988. Draft Guidelines, Amer. Soc. Test. Mater., Philadelphia, PA. Conducting Acute Toxicity Tests with Fishes, Macroinvertebrates, and Amphibians, E 729-88, 1988.Annual Book of ASTM Standards, Vol. 11.04, Amer. Soc. Test. Mater., Philadelphia, PA, pp. 304-323.

ASTM. 1990. Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods. E 1367-90. Am. Soc. Test. Mater., Philadelphia, PA.

ASTM. 1990a. Guide for Collection, Storage, Characterization, and Manipulation of Sediments for Toxicological Testing, E 1391.

ASTM. 1990b. Guide for conducting Solid Phase 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods,E 1167, Draft guideline, Amer. Soc. Test. Mater., Philadelphia, PA.

Buchanan, J.B. and J.M. Kain. 1971. Measurement of the physical and chemical environment. In: <u>Methods for the study of Marine Benthos, IBP Handbook No. 16</u>, N.A. Holme and A.D. McIntyre, editors, Blackwell Scientific Publications: Oxford.

Folk, R.L. 1957. <u>Petrology of Sedimentary Rock</u>. Hemphill Publishing Company, Austin, Texas.

Statistical Analysis System. 1990. SAS Institute Inc., SAS Procedures Guide, Version 6, Third Edition, Cary, NC: SAS Institute Inc., 705 pp.

U.S. Environmental Protection Agency and U.S. Army Corps of Engineers. 1991. Evaluation of Dredged Material Proposed for Ocean Disposal. Testing Manual. EPA-503/8-91/001.

Warner, J.S. 1976. Determination of Aliphatic and aromatic Hydrocarbons in Marine Organisms. Analytical Chemistry, 48, No. 3, 578-583.

ACKNOWLEDGEMENTS

We thank Dr. George Ryan for assistance with statistical analysis of data and Valerie A. Coseo for typing the report.

Table 1. Physical characteristics for 11 sediment samples from Albemarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL. All measurements were performed in triplicate.

	Mean percentage (standard deviation)			
Sediment	Moisture	[a]WLOI	Sand	Silt/Clay
SLO 18-19	87.5(0.33)	21.2(0.34)	26.3(1.10)	73.7(1.70)
SLO 1-2-3	83.5(0.43)	18.7(0.22)	22.2(0.46)	77.8(0.46)
HCK 3-4-5	74.9(2.07)	14.3(1.29)	32.0(9.8)	68.0(9.8)
CMP 1-2	69.9(0.82)	10.0(0.23)	32.4(1.72)	67.5(1.72)
ORL 4-5	69.6(0.33)	14.1(0.31)	7.6(1.09)	92.4(1.09)
NAT 10-11-12	81.6(0.06)	21.1(0.24)	6.6(0.75)	93.4(0.75)
BRD 1-2-3	77.9(0.33)	13.9(0.22)	9.0(0.80)	91.0(0.80)
PAM 3	78.7(0.44)	17.4(0.75)	17.7(0.35)	82.3(0.35)
PAM 30A-30B	79.4(0.46)	12.0(0.36)	4.0(0.67)	96.0(0.67)
TG 1-2A	35.1(0.92)	1.9(0.17)	89.3(0.18)	10.7(0.18)
PAM 21-24	71.4(1.34)	8.0(0.60)	55.2(8.83)	44.8(8.83)
Reference (C-17)	78.6(0.31)	17.7(1.73)	2.9(0.78)	97.1(0.78)

[a] WLOI = Weight loss on ignition at 537°C for four hours.

Table 2. Survival of three benthic species exposed in acute toxicity tests to 11 sediments from Albemarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL. Five replicates of 20 Ampelisca were used (total of 100) and three replicates of mysids (30 organisms total) and three replicates of Neanthes (30 organisms total) were used.

Sediment	10-day Acute Test - Ampelisca Number Survived	96-hour Acute Test - Mysids Number Survived	10-day Acute Test - Neanthes [a]Number Survived
SLO 18-19	63	29	31[a]
SLO 1-2-3	91	30	30
HCK 3-4-5	89	30	30
CMP 1-2	88	30	30
ORL 4-5	97	30	30
NAT 10-11-12	94	30	30
BRD 1-2-3	97	30	30
PAM 3	94	30	30
PAM 30A-30B	96	30	30
TG 1-2A	93	30	30
PAM 21-24	96	30	30
Reference (C-17)	99	30	30

[a] One additional animal was added in one replicate by mistake.

Table 3. Survival of Ampelisca exposed in 10-day acute toxicity tests to 11 sediments from Albemarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL. Five replicates with 20 animals per replicate were used in each test.

	Number of survivors per replicate							
Sediment	1	2	3	4	5	Mean	S.D.	S^2
[a]SLO 18-19	11	12	15	12	13	12.6	1.52	2.30
SLO 1-2-3 4.20	15	20	18	18	20	18.2	2.05	
[b]HCK 3-4-5	17	20	14	19	19	17.8	2.39	5.70
[a]CMP 1-2	19	18	16	16	19	17.6	1.52	2.30
ORL 4-5	20	20	18	19	20	19.4	0.89	0.80
[a]NAT 10-11-12	19	19	19	18	19	18.8	0.45	0.20
BRD 1-2-3	20	20	20	20	17	19.4	1.34	1.80
PAM 3	16	20	19	19	20	18.8	1.64	2.70
PAM 30A-30B	20	20	17	20	19	19.2	1.30	1.70
TG 1-2A	20	17	20	18	18	18.6	1.34	1.80
PAM 21-24	19	20	20	20	17	19.2	1.30	1.70
Reference (C-17)	20	19	20	20	20	19.8	0.45	0.200

[a] Statistically significant at $P < .05$ when using t-test with arcsin $\sqrt{x}$ transformed data.
[b] Statistically significant at $P = 0.545$.
S.D. = Standard deviation.
S^2 = Variance.

Table 4. Range of measured values for pH, dissolved oxygen (mg/ℓ), temperature (°C) and salinity (°/..) during the 96-hour acute toxicity test conducted with *Mysidopsis bahia* and 11 sediments from Albermarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
SLO 18-19				
Rep 1	7.5-8.1	6.4-8.8	18-24	15.9-16.4
2	7.8-8.0	5.5-8.6	18-20	17.6-18.2
3	7.9-8.2	6.4-9.0	18-21	17.5-17.9
SLO 1-2-3				
Rep 1	7.8-8.1	7.4-9.0	18-24	16.4-16.9
2	7.8-8.2	6.8-9.0	18-20	17.0-17.9
3	7.4-8.2	7.2-9.1	18-20	15.4-17.5
HCK 3-4-5				
Rep 1	7.2-8.1	7.2-8.8	18-26	15.5-16.8
2	7.8-8.0	7.5-9.0	18-22	16.7-17.9
3	7.9-8.9	6.4-8.8	18-20	17.3-17.9
CMP 1-2				
Rep 1	7.9-8.1	6.7-8.6	18-22	17.7-18.2
2	7.8-8.1	7.4-9.1	18-22	16.0-18.1
3	7.7-8.1	7.4-8.9	18-24	16.7-18.3
ORL 1-2				
Rep 1	7.2-8.1	7.4-8.9	18-26	15.3-16.9
2	7.3-8.1	7.2-9.0	18-23	15.9-17.5
3	7.4-8.1	6.0-8.7	18-22	17.8-18.1
NAT 10-11-12				
Rep 1	6.9-8.0	7.5-9.0	18-20	16.0-18.4
2	7.2-8.3	6.3-9.0	18-22	15.9-17.5
3	7.5-8.0	6.7-8.8	18-20	17.5-18.5
BRD 1-2-3				
Rep 1	7.7-8.0	7.2-8.9	18-20	17.6-18.8
2	7.4-8.1	6.3-9.0	18-24	15.2-17.2
3	7.7-7.9	7.2-8.9	18-22	17.2-17.7
PAM 3				
Rep 1	7.7-8.2	6.0-9.1	18-20	17.6-18.8
2	7.2-8.0	6.6-8.8	18-24	16.2-17.0
3	7.3-8.1	7.2-8.8	18-22	16.9-17.8

Table 4 continued.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
PAM 30A-30B				
Rep 1	7.9-8.1	7.2-9.0	18-24	15.8-17.5
2	7.8-8.1	7.3-8.7	18-22	17.6-19.0
3	7.9-8.1	6.9-9.0	18-20	17.8-19.3
TG 1-2A				
Rep 1	7.4-8.1	7.0-9.1	18-20	17.5-18.8
2	8.0-8.2	7.2-9.0	18-24	16.8-17.8
3	7.9-8.2	6.8-8.8	18-22	17.2-18.3
PAM 21-24				
Rep 1	7.8-8.1	6.5-8.9	18-24	15.8-17.5
2	7.8-8.1	7.2-8.9	18-22	17.2-18.2
3	7.9-8.2	7.5-9.1	18-22	17.8-18.5
Reference (C-17)				
Rep 1	7.9-8.1	6.8-9.0	18-22	17.4-18.7
2	7.9-8.2	6.7-8.9	18-24	16.7-18.0
3	7.9-8.1	7.5-8.7	18-20	17.5-18.5

Table 5. Range of measured values for pH, dissolved oxygen (mg/ℓ), temperature (°C) and salinity (°/‥) during the 10-day acute toxicity test conducted with <u>Ampelisca abdita</u> and 11 sediments from Albemarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
SLO 18-19				
Rep 1	7.7-8.4	5.8-8.7	27-28	17.4-19.0
2	7.8-8.4	5.6-8.7	28	16.6-18.4
3	7.9-8.5	5.3-8.6	26-28	16.9-19.5
4	8.0-8.4	6.9-9.0	26-28	16.8-18.5
5	7.9-8.5	5.5-8.8	26-28	17.2-18.6
SLO 1-2-3				
Rep 1	8.0-8.4	6.1-8.7	26-28	17.3-18.5
2	6.9-8.3	5.7-8.5	26-28	17.2-18.9
3	7.9-8.4	5.5-8.9	26-28	17.0-18.3
4	7.7-8.3	5.6-8.7	25-28	16.8-18.2
5	7.7-8.4	5.5-8.8	26-28	16.9-18.7
HCK 3-4-5				
Rep 1	7.9-8.3	6.7-8.9	28	16.8-18.4
2	8.0-8.2	6.5-8.7	28-30	17.3-18.5
3	7.9-8.3	6.4-8.9	28	17.3-18.6
4	8.0-8.2	6.4-8.8	27-29	17.1-18.4
5	7.9-8.3	6.7-8.8	28-29	16.9-18.3
CMP 1-2				
Rep 1	7.5-8.3	6.7-8.5	28-29	17.5-18.7
2	8.1-8.4	6.4-8.6	28-30	17.2-18.7
3	8.0-8.5	6.5-8.9	28-30	17.3-18.6
4	7.9-8.4	6.5-9.1	28-30	16.9-18.0
5	7.9-8.5	6.5-8.9	28-30	17.0-18.4
ORL 4-5				
Rep 1	7.9-8.2	6.7-9.0	28-30	16.6-18.6
2	7.9-8.3	6.9-9.0	28-29	16.7-18.3
3	8.0-8.3	6.6-8.8	28-30	17.0-18.4
4	8.0-8.2	6.5-8.7	28-29	17.0-18.6
5	8.0-8.1	6.6-8.7	28-29	17.3-18.9
NAT 10-11-12				
Rep 1	7.8-8.2	6.4-8.8	27-28	16.9-18.9
2	7.9-8.2	6.1-8.9	28-29	17.0-18.7
3	7.9-8.1	6.1-8.7	26-28	17.4-18.8
4	7.7-8.3	6.2-8.7	27-28	17.0-18.4
5	7.8-8.1	6.4-8.8	27-28	16.7-18.7

Table 5 Continued.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
BRD 1-2-3				
Rep 1	7.9-8.3	6.7-8.8	28-30	16.9-18.1
2	8.0-8.3	6.4-8.8	28-30	17.3-18.5
3	7.7-8.3	6.7-8.6	28-29	17.7-18.8
4	7.9-8.3	6.5-9.0	28-30	17.5-18.2
5	7.9-8.3	6.8-8.8	28	17.1-18.3
PAM 3				
Rep 1	7.9-8.3	6.9-8.7	28-30	16.8-18.6
2	8.0-8.2	6.6-8.9	28	16.7-18.4
3	7.8-8.3	6.6-8.7	28-30	16.7-18.0
4	8.0-8.3	6.6-8.8	27-28	16.7-18.4
5	8.0-8.3	6.7-8.9	28	16.7-18.4
PAM 30A-30B				
Rep 1	7.9-8.1	6.9-8.7	28-30	16.8-18.6
2	7.9-8.3	6.7-8.9	28-30	17.1-18.6
3	8.0-8.3	6.5-9.0	28-30	17.1-18.3
4	8.0-8.2	6.7-8.7	28-30	16.7-18.6
5	7.9-8.2	6.7-8.6	28-30	16.8-18.5
TG 1-2A				
Rep 1	7.1-8.2	6.6-8.7	28-30	17.2-18.6
2	7.8-8.2	6.8-8.9	28-30	16.9-18.6
3	8.0-8.3	6.5-8.7	28-30	17.1-18.6
4	8.0-8.3	6.7-8.8	28-31	17.2-17.9
5	7.9-8.3	6.8-8.7	26-30	16.9-18.6
PAM 21-24				
Rep 1	7.9-8.3	6.7-8.8	28-30	17.4-18.6
2	7.9-8.3	6.8-8.9	28-29	16.6-18.1
3	8.0-8.3	6.7-8.9	28-30	17.5-18.1
4	7.7-8.3	6.6-8.7	28-29	16.9-18.3
5	8.0-8.3	6.4-8.8	28-29	16.9-18.3
Reference (C-17)				
Rep 1	7.9-8.3	7.0-8.7	26-30	16.8-18.2
2	7.9-8.2	6.6-8.8	28-31	16.8-18.4
3	7.9-8.3	6.8-8.9	28-30	16.8-18.3
4	8.0-8.3	6.7-8.7	28-30	17.1-18.3
5	8.0-8.2	7.0-8.9	28-30	16.7-18.3

Table 6. Range of measured values of pH, dissolved oxygen (mg/ℓ), temperature (°C) and salinity (°/..) during the 10-day acute toxicity test conducted with *Neanthes arenaceodentata* and 11 sediments from Albemarle and Pamlico Sounds, North Carolina, and a reference sediment from Perdido Bay AL/FL.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
SLO 18-19				
Rep 1	8.1-8.2	7.0-7.3	24	
2	8.1-8.2	7.2-7.4	24	
3	8.1-8.3	7.0-7.4	24-25	
SLO 1-2-3				
Rep 1	7.9-8.1	7.3-7.6	22	
2	8.0-8.1	7.3-7.5	22-23	
3	8.0-8.1	7.3-7.5	24-28	20.0
HCK 3-4-5				
Rep 1	7.7-8.0	6.8-7.3	24-25	
2	7.7-8.1	7.2-7.4	25-26	
3	7.8-8.0	7.2-7.4	24-25	
CMP 1-2				
Rep 1	8.1-8.2	7.1-7.3	26	
2	8.1-8.2	7.1-7.3	26	
3	8.0-8.1	7.0-7.3	26	
ORL 4-5				
Rep 1	7.8-8.1	7.1-7.4	26-27	
2	7.8-8.1	7.2-7.3	24-26	
3	7.8-8.1	7.1-7.3	25-26	
NAT 10-11-12				
Rep 1	7.5-7.8	7.3-7.4	24-25	
2	7.5-7.8	7.2-7.4	23-24	
3	7.5-7.9	7.2-7.4	24-25	
BRD 1-2-3				
Rep 1	7.7-8.0	7.1-7.4	25	
2	7.7-8.0	7.1-7.4	24-25	
3	7.8-8.0	7.2-7.3	24-25	
PAM 3				
Rep 1	7.7-8.0	7.2-7.3	24-26	
2	7.7-8.0	7.1-7.3	24-26	
3	7.1-8.0	7.1-7.4	24-26	

Table 6 continued.

Sediment	pH	Dissolved Oxygen	Salinity	Temperature
PAM 30A-30B				
Rep 1	7.8-8.1	7.1-7.4	25-26	
2	7.8-8.1	6.7-7.4	25-26	
3	7.9-8.1	7.1-7.4	26	
TG 1-2A				
Rep 1	7.9-8.0	6.8-7.3	24-26	
2	7.8-8.0	6.5-7.3	26-28	
3	8.0-8.1	7.1-7.3	26	
PAM 21-24				
Rep 1	7.9-8.1	7.2-7.3	25-26	
2	7.8-8.1	7.1-7.3	25-26	
3	7.8-8.1	7.1-7.4	25-26	
Reference (C-17)				
Rep 1	7.9-8.1	7.1-7.3	26-28	
2	7.9-8.2	7.1-7.3	27-28	
3	8.0-8.2	7.1-7.3	27-28	

Table 7. Concentrations in nanograms/gram (ppb) of selected chlorinated pesticides and PCB congeners in samples of sediment from Albemarle and Pamlico Sounds, North Carolina.

		Concentrations in nanogram/gram wet sediment					
Kennedy		E. Prong	Upper	Hancock	Oriental	Green & Kershaw	
Compound	Core#	Slocum Creek SLO 18-19	Slocum Creek SLO 1-3	Creek HCK 3-4-5	Harbor CMP 1-2	Creeks ORL 4-5	Creek NAT 10-12
Aldrin		ND	ND	ND	ND	ND	ND
Isomers							
Alpha		ND	ND	ND	ND	ND	ND
Beta		ND	ND	ND	ND	ND	ND
Gamma (lindane)		ND	ND	ND	ND	ND	ND
Chlordane (alpha)		59	ND	ND	ND	ND	ND
DDE*		59	ND	ND	ND	ND	ND
DDD*		60	ND	ND	ND	ND	ND
DDT*		34	ND	15	ND	ND	ND
Dieldrin		15	ND	ND	ND	ND	ND
Endrin		ND	ND	ND	ND	ND	ND
Endosulfan I		22	ND	ND	ND	ND	ND
Endosulfan II		ND	ND	ND	ND	ND	ND
Endosulfan Sulfate		ND	ND	ND	ND	ND	ND
Heptachlor		ND	ND	ND	ND	ND	ND
Heptachlor epoxide		ND	ND	ND	ND	ND	ND
Hexachlorobenzene		ND	ND	ND	ND	ND	ND
Methoxychlor		ND	ND	ND	ND	ND	ND
Mirex		ND	ND	ND	ND	ND	ND
PCB Congeners		ND	ND	ND	ND	ND	ND
Toxaphene		ND	ND	ND	ND	ND	ND

ND = Not detected;
* Ortho-Para and Para-Para isomers are combined for DDD, DDE and DDT.

Table 7. (Continued)

Compound	Core#	Broad Creek BRD 1-3	Chocowinity Bay PAM 3	Middle Pamlico River 30A 30B	Middle Pamlico River Tg 1-2A	Middle Pamlico River 21-24	Method Detection Limit (μg/g)
		Concentrations in nanogram/gram wet sediment					
Aldrin		ND	ND	ND	ND	ND	0.0020
BHC Isomers							
Alpha		ND	ND	ND	ND	ND	0.0040
Beta		ND	ND	ND	ND	ND	0.0040
Gamma (lindane)		ND	ND	ND	ND	ND	0.0020
Chlordane (alpha)		ND	ND	ND	ND	ND	0.0040
DDE*		ND	ND	ND	ND	ND	0.0040
DDD*		ND	ND	ND	ND	ND	0.0080
DDT*		24	17	ND	ND	ND	0.010
Dieldrin		ND	ND	ND	ND	ND	0.0040
Endrin		ND	ND	ND	ND	ND	0.010
Endosulfan I		ND	ND	ND	ND	ND	0.010
Endosulfan II		ND	ND	ND	ND	ND	0.010
Endosulfan Sulfate		ND	ND	ND	ND	ND	0.050
Heptachlor		ND	ND	ND	ND	ND	0.0020
Heptachlor epoxide		ND	ND	ND	ND	ND	0.010
Hexachlorobenzene		ND	ND	ND	ND	ND	0.0020
Methoxychlor		ND	ND	ND	ND	ND	0.030
Mirex		ND	ND	ND	ND	ND	0.020
PCB Congeners		ND	ND	ND	ND	ND	0.010
Toxaphene		ND	ND	ND	ND	ND	0.20

ND = Not detected

Table 8. Concentrations in $\mu g/g$ dry weight of selected metals in sediment from the Albemarle and Pamlico Sounds, North Carolina. Analyses were performed by using inductively coupled argon plasma emission spectrometry.

Sediment Location (Core #)	Replicate	As[a]	Cd	Cr	Cu	Hg	Ni	Pb[a]	Se	Zn
E. Prong Slocum Creek (SLO 18-19)	1	41	10	100	260	ND	24	140	ND	320
	2	50	10	100	260	ND	22	140	ND	37
Upper Slocum Creek (SLO 1-3)	1	52	9.7	109	24	ND	21	50	ND	160
	2	33	9.7	103	30	ND	17	23	ND	160
Hancock Creek (HCK 3-4-5)	1	49	ND	21	10	ND	12	18	ND	4.4
	2	44	ND	20	7.6	ND	10	12	ND	ND
Oriental Harbor (CMP 1-2)	1	53	0.56	29	110	ND	13	43	ND	160
	2	50	ND	27	90	ND	12	33	ND	170
Green & Kershaw Creeks (ORL 4-5)	1	59	ND	30	13	ND	14	7.9	ND	4.9
	2	59	ND	28	14	ND	13	8.6	ND	6.7
Kennedy Creek (NAT 10-12)	1	51	2.1	36	45	ND	20	47	ND	350
	2	60	1.7	39	45	ND	21	51	ND	350
Broad Creek (BRD 1-3)	1	72	ND	27	81	ND	14	20	ND	50
	2	54	ND	23	72	ND	12	19	ND	59

Table 8. (Continued)

Sediment Location (Core #)	Replicate	As[a]	Cd	Cr	Cu	Hg	Ni	Pb[a]	Se	Zn
Chocowinity Bay (PAM 3)	1	89	ND	21	3.1	ND	14	25	ND	21
	2	47	ND	18	9.4	ND	12	ND	ND	14
Middle Pamlico River (30A-30B)	1	63	2.2	39	15	ND	20	18	ND	29
	2	73	2.3	39	13	ND	19	25	ND	18
Middle Pamlico River (TG 1-2A)	1	17	2.8	15	3.2	ND	6.0	ND	ND	22
	2	11	2.2	12	1.6	ND	4.5	ND	ND	17
Middle Pamlico River (21-24)	1	45	0.80	21	8.7	ND	11	16	ND	14
	2	45	1.2	23	11	ND	13	10	ND	2.8

ND = not detected

[a] Background subtraction techniques normally used could not be applied due to interference from unknown elements that cause intense background signal. Therefore these values are maximum possible because they include background.

Method Detection Limits[b]	1	2.5	0.20	0.75	0.10	0.75	0.75	2.5	0.75	0.25

[b] Based on final volume of 50 mℓ and a sample weight of 2 grams (maximum sample size).

Table 9. Concentrations in $\mu g/g$ total aliphatic and total aromatic petroleum hydrocarbons in sediments from Albermarle and Pamlico Sounds, North Carolina.

Location (Core #)	Concentrations in wet and dry sediment			
	Aliphatic		Aromatic	
	Wet	Dry	Wet	Dry
E. Prong Slocum Creek (SLO 18-19)	330	2640	130	1040
Upper Slocum Creek (SLO 1-3)	59	357	1.6	9.7
Hancock Creek (HCK 3-4-5)	ND	ND	2.8	11
Oriental Harbor (CMP 1-2)	70	230	6.0	20
Green & Kershaw Creeks (ORL 4-5)	ND	ND	ND	ND
Kennedy Creek (NAT 10-12)	18	97.8	6.1	33
Broad Creek (BRD 1-3)	ND	ND	1.5	6.8
Chocowinity Bay (PAM 3)	1.2	5.6	1.9	8.9
Middle Pamlico River (30A-30B)	ND	ND	2.5	12.1
Middle Pamlico River (TG 1-2A)	ND	ND	ND	ND
Middle Pamlico River (21-24)	ND	ND	1.3	4.6

ND = 1.0 $\mu g/g$ aromatic and aliphatic petroleum hydrocarbons