U.S. Fish and Wildlife Service

U.S. Geological Survey

**Sediment Quality within the Impounded Reaches of Cape Fear River Locks and Dams**

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 U.S. Fish and Wildlife Service U.S. Geological Survey

 Ecological Services Columbia Environmental Research Center

 Raleigh, North Carolina Columbia, Missouri

**Preface**

The U.S. Fish and Wildlife Service assisted the U.S. Army Corps of Engineers (Wilmington District) in an assessment of the chemical contaminants in, and toxicity of, sediments at Cape Fear River locks and dams. The work was coordinated by Tom Augspurger (Ecologist / Environmental Contaminant Specialist) and Sara Ward (Ecologist / Environmental Contaminant Specialist) in the U.S. Fish and Wildlife Service’s Raleigh Field Office and was funded through a transfer agreement between the U.S. Fish and Wildlife Service and the U.S. Army Corps of Engineers. Toxicity tests were perfomed by the U.S. Geological Survey’s Columbia Environmental Research Center (CERC) under the direction of Chris Ingersoll through an intra-agency agreement with the U.S. Fish and Wildlife Service. Other CERC scientists primarily responsible for the toxicity testing component of the project include Nile Kemble and James Kunz. Analyical chemistry was performed by Environmental Conservation Laboraties, Inc. and Severn Trent Laboratories, Inc.

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Additional questions, comments, and suggestions related to this draft are encouraged. Inquires can be directed to the U.S. Fish and Wildlife Service at the following address:

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**Sediment Quality within the Impounded Reaches of Cape Fear River Locks and Dams**

**Executive Summary**

This report documents an evaluation of chemical contaminants in, and toxicity of, sediments collected from impoundments created by locks and dams on the Cape Fear River in Bladen and Cumberland Counties, North Carolina. Twelve whole-sediment samples from within the impounded reaches of the three locks and dams were collected in November 2006. All samples were analyzed for elemental contaminants and polycyclic aromatic hydrocarbons (PAHs), and a subset of nine of the samples were used in toxicity tests. Whole sediments had no significant effect on survival or growth in 28-d toxicity tests with *Hyallela azteca* (freshwater amphipod) or in 10-d tests with *Chironomus dilutus* (freshwater midge). In 2-d sediment elutriate (water-extractable fraction) tests with *Ceriodaphnia dubia* (freshwater cladoceran), statistically-significant reductions in survival occurred in four of the nine exposures. Pairwise correlation analyses indicated several whole-sediment and elutriate water chemistry variables were significantly and negatively correlated with *C. dubia* survival. Of the correlated variables, elutriate manganese had among the strongest correlation (r2 = 0.79, *p* = 0.0006) and the most plausible biological association with reduced *C. dubia* survival because some elutriate manganese concentrations exceeded published lethal concentrations for *C. dubia* in water exposures. All elemental contaminants for which published freshwater whole-sediment *threshold effects concentrations* (*TECs*, concentrations of contaminants in whole sediment below which adverse effects to sensitive aquatic organisms are not expected to occur) are available were less than their corresponding TECs and are therefore considered toxicologically insignificant. Sediment PAHs with published freshwater whole-sediment TECs were in excess of TECs at two of the 12 sampling stations (river mile 112.9 near Fayetteville and river mile 88.5 about 0.2 miles upstream of Tar Heel Landing Road). Five individual PAHs exceeded TECs at these locations, but no samples exceeded the *probable effects concentrations* (*PECs*, concentrations of contaminants in whole sediment above which adverse effects to sediment-dwelling organisms may be expected). Based on review of existing data (Tier 1) and results of sediment chemistry and toxicity tests (Tier 2 and 3), contamination in surface sediments behind Cape Fear River locks and dams is unlikely to be a concern in-place. Mobilization of sediments may be a short-term water column concern based on the elutriate toxicity test results. While sediment re-suspension and contaminant release in the elutriate tests may be near a worst case simulation of actual conditions following sediment disturbing activities, additional synthesis is needed to characterize the nature and magnitude of this issue. In particular, data regarding the modeled or measured sediment re-suspension caused by specific sediment disturbing activities (e.g., dredging, dam alterations, etc.) will help put the elutriate test results in context for normal sediment management practices. Sediment disturbing activities proposed for the Cape Fear River would benefit from development of a sediment management plan to address these issues.

**CONTENTS**

PREFACE . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . ii

EXECUTIVE SUMMARY . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . iii

LIST OF TABLES . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . v

LIST OF FIGURES . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . vi

INTRODUCTION . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 1

METHODS . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 2

RESULTS / DISCUSSION . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 18

REFERENCES . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 34

Appendix

**TABLES**  Page

1. Cape Fear River sediment sampling location data and the date and time of collection 3

2. Response of *Hyalella azteca* in 28-d whole-sediment exposures, *Chironomus dilutus*

in 10-d whole-sediment exposures, and *Ceriodaphnia dubia* in 2-d elutriate exposures

prepared from sediment samples collected from the Cape Fear River 19

3. Water quality characteristics in 2-d elutriate exposures with *Ceriodaphnia dubia*

prepared from Cape Fear River sediment samples and a control sediment 20

4. Elemental contaminant concentrations of 2-d elutriate exposures with *Ceriodaphnia*

*dubia* prepared from Cape Fear River sediment samples and a control sediment 21

5. Water quality characteristics of pore water isolated from Cape Fear River whole-

sediment samples 22

6. Elemental contaminant concentrations of pore water isolated from Cape Fear

River whole-sediment samples 23

7. Mean water quality characteristics of overlying water in whole-sediment toxicity

tests with *Hyalella azteca* and Cape Fear River sediments 24

8. Mean water quality characteristics of overlying water in whole-sediment toxicity

tests with *Chironomus dilutus* and Cape Fear River sediments 25

9. Total organic carbon, grain size and moisture measured in whole-sediment samples

collected from the Cape Fear River 26

10. Elemental contaminants in whole-sediment samples collected from the Cape Fear

River. For each element, results are compared to threshold-effects concentration

guidelines of MacDonald et al. (2000) 27

11. Polycyclic aromatic hydrocarbons measured in whole-sediment samples collected

from the Cape Fear River. For each compound, results are compared to threshold-effects concentrations of MacDonald et al. (2000) 28

12. Probable effect concentration quotients (PEC-Qs) and polycyclic aromatic

hydrocarbon equilibrium partitioning sediment benchmark toxic units (ΣESBTUs)

for whole-sediment samples collected from the Cape Fear River 29

13. Elutriate and pore water exceedences of North Carolina water quality standards

or action levels for elemental contaminants (NCDENR 2004) 30

14. Pearson’s correlations for 2-d *C. dubia* survival in sediment elutriates versus whole-

sediment and elutriate physical and chemical constituents 31

**FIGURES** Page

1. Vicinity map for Cape Fear River lock and dam sediment sampling sites 4

2. Cape Fear River sediment sampling site downstream of Fayetteville (furthest 5

upstream sample in this assessment)

3. Cape Fear River sediment sampling sites between confluence with Rock Fish 6

Creek and Lock and Dam #3

4. Cape Fear River sediment sampling sites upstream and downstream of Lock 7

and Dam #3

5. Cape Fear River sediment sampling site at Tar Heel Landing 8

6. Cape Fear River sediment sampling site upstream of Elizabethtown 9

7. Cape Fear River sediment sampling sites downstream of Elizabethtown and

upstream of Lock and Dam #2 10

8. Cape Fear River sediment sampling sites upstream of Lock and Dam #1, near 11

Elwell Ferry

9. Cape Fear River sediment sampling sites upstream of Lock and Dam #1, near 12

Riegelwood

10. Relationship between elutriate manganese concentrations and *Ceriodaphnia* 30

*dubia* survival in 2-d elutriate exposures prepared from sediment samples collected

from Cape Fear River locks and dams

**Sediment Quality within the Impounded Reaches of Cape Fear River Locks and Dams**

**Introduction**

This report documents an evaluation of chemical contaminants in, and toxicity of, sediments collected from impoundments created by locks and dams on the Cape Fear River in Bladen and Cumberland Counties, North Carolina. Lock and Dam #1 is located about 39 miles (63 km) upstream of Wilmington, Lock and Dam #2 is located at river mile 71 near Elizabethtown, and Lock and Dam #3 (the William O. Huske Lock and Dam) is located at river mile 95 just downstream of the Cumberland County / Bladen County border. The locks and dams were built between 1915 (Lock and Dam #1) and 1935 (Lock and Dam #3). The U.S. Army Corps of Engineers (Corps) owns and operates the three locks and dams, and these structures are no longer used for commercial navigation which was their original purpose. The Corps is interested in "decommissioning" the dams and is currently studying their options as part of a General Reevaluation Report (GRR) for the Wilmington Harbor Deepening Project. The U.S. Fish and Wildlife Service (Service) is assisting the Corps and others interested in the Cape Fear River’s natural resources by providing data to support the GRR.

Between May and August 2006, the Service conducted a review of existing information on pollutant sources within a one-mile boundary of the impounded reach of each of the three locks and dams. The work, which entailed database searches, file reviews, interviews, synthesis of environmental monitoring data, and a field reconnaissance, was similar to an environmental audit. In Augst 2006, the Service provided the Corps with a report, ***Tier 1 Preliminary Evaluation of Pollutant Sources to the Impounded Reaches of Cape Fear River Locks and Dams 1, 2, and 3***, which concluded there were no known significant sediment pollutant problems in the assessment area. However, existing data were limited, and there were indications of pollutant sources of concern in the upper portion of the assessment area including controlled or uncontrolled releases from facilities such as Borden Chemical, Monsanto, and EI DuPont Nemours. The 2006 report narrowed the scope of potential contaminant concerns to a targeted list of chemicals and several sites along the about 75 miles of impounded river reach. The Corps concluded that chemical analyses and toxicity testing of sediment samples from depositional areas would be a prudent next step. The Service coordinated an effort to assess area sediments; while low contaminant burdens were expected, the objectives of the effort were to:

1) generate current data on the chemical and physical quality of the sediments behind the locks and dams for comparison to sediment toxicological screening values; and,

2) assess the toxicity of whole sediments and sediment elutriates prepared from these whole-sediment samples to sensitive aquatic organisms.

The following summary presents the sediment sampling and testing methods, results, and an interpretation of the findings.

**Methods**

*Sample sites*

Factors considered in determining the number and location of samples included the location of identified potential sources of concern and areas of sediment accumulation. Physical factors considered included the area and depth of potentially affected sediments behind the dams, distribution of sediments, and the length and breadth of the impounded reaches. By comparing the results of surveys of the river performed in 1903 and contemporary (2005 and 2006) hydrographic surveys, the Corps mapped changes in elevation of the river bottom within about 1,500-feet upstream of each lock and dam. At Lock and Dam #1, there is only an average of about two feet of sediment accumulation with a maximum of about eight feet of accumulation in a small area near at the base of the dam (and opposite the lock). Similar efforts at Lock and Dam #2 indicated even less accumulation with the greatest depths again near the dam and opposite the lock while there was little overall accumulation at Lock and Dam #3. These depositional areas were among the targeted sites for sample collection.

During reconnaissance of the river, Service staff examined the nature of sediments at inside channel bends of the river. These are typically more quiescent areas where fine-grained sediments (which have the greatest potential to accumulate contaminants) would settle. We found silty, highly organic samples indicative of depositional environments within 20-feet of the bank at river bends, such as Kellys Cove (about 5 miles upstream of Lock and Dam #1). These depositional areas were also targeted sites for sample collection. Sediments farther from the bank were typically coarse sand which is characteristic of the straighter portions of the river channel as well; this sandy material has little potential for contaminant accumulation and sediments from these areas were not retained for analyses. Twelve samples were collected by Service Ecologists / Environmental Contaminants Specialists between November 1 and 3, 2006. Table 1 lists the collection locations, collection date, time, and specific coordinates. Figures 1 through 9 depict the collection locations.

*Sediment sample collection and storage*

Samples were collected, transported, stored, and shipped for analyses under chain of custody.

Because the Corps determined minimal depth of sediment accumulation, surface sediment grab sampling was conducted for this characterization. A stainless-steel petit Ponar dredge was used to collect the top 5 to 10 cm of sediment; multiple grabs were collected and composited to form one sample at each site. The composite of the grab samples was homogenized by stirring with a stainless-steel spoon in a stainless-steel bucket. Debris (e.g., sticks, leaves, rocks bigger than about 0.5 cm3) were physically removed during homogenization. Collection equipment was thoroughly cleaned (ambient water rinse, detergent and water scrub, distilled / demineralized water rinse, 10% nitric acid rinse, another distilled / demineralized water rinse, acetone rinse, and a final rinse with distilled / demineralized water) before sampling at each site.

Aliquants of the homogenate were split in into chemically cleaned glass jars (provided by the analytical laboratory) with Teflon®-lined lids for chemical analyses with about 4 L of the same sediment homogenate from each site placed in a high-density polyethylene jar (EP Scientific

Table 1. Cape Fear River sediment sampling location data and the date and time of collection.

|  |  |  |
| --- | --- | --- |
| **Sample ID** | **River Mile** | **Description and GPS Coordinates** |
| CF 1  | 76.1  | About 1.2 miles upstream of Elizabethtown Dock and upstream of most local sources, about 15 to 20 feet from west bank (11-01-06; 10:15 am)N 34.65373˚ W 78.64207˚ |
| CF 2  | 72.8 | About 0.3 miles downstream of Hwy 701 bridge at Elizabethtown andabout 15 to 20 feet from north bank (11-01-06; 11:25 am)N 34.62982˚ W 78.59916˚ |
| CF 3 | 71.8 | About 0.3 miles upstream of Lock and Dam #2 and about 15 feet from south bank (11-01-06; 11:54 am) N 34.62848˚ W 78.58287˚ |
| CF 4 | 93.4  | Back eddy behind rock downstream of DuPont, along south side of channel and about 0.1 mile downstream of gaging station (11-01-06; 3:47 pm) N 34.81243˚ W 78.81832˚ |
| CF 5 | 88.5 | About 0.2 miles upstream of Tar Heel Landing Road crossing, downstream of Smithfield Foods and about 15 feet from northwest shore(11-01-06; 4:39 pm) N 34.74638˚ W 78.78783˚ |
| CF 6 | 44.7 | About 1.0 mile downstream of confluence with Carvers Creek upstream of Locks Point, about 15 to 20 feet from west bank (11-02-06; 11:09 am)  N 34.44568˚ W 78.34975˚ |
| CF 7 | 46.3 | About 1.5 miles downstream of Elwell’s Ferry at inside bend of Kellys Cove, about 15 to 20 feet from west bank (11-02-06; 11:48 am) N 34.46391˚ W 78.35083˚ |
| CF 8 | 39.0 | About 0.25 miles upstream of Lock and Dam #1 at depositional area along southwest bank noted on Corps bathymetry map (11-02-06; 2:00 pm) N 34.40745˚ W 78.29622˚ |
| CF 9 | 105.9 | About 0.2 miles downstream of unnamed tributary adjacent to Monsanto and Rohm and Haas (11-03-06; 9:47 am)N 34.97574˚ W 78.77853˚ |
| CF 10 | 108.4 | About 0.3 miles downstream of confluence with Rock Fish Creek, downstream of Rock Fish Creek Wastewater Treatment Plant, along north side of channel (11-03-06; 10:24 am)N 34.96910˚ W 78.81459˚ |
| CF 11  | 112.9 | About 2.5 miles upstream of I-95 crossing, downstream of Borden Chemical, and about 15 to 20 feet from east bank (11-03-06; 1:50 pm) N 35.01809˚ W 78.85653˚ |
| CF 12  | 95.2 | About 0.2 miles upstream of Lock and Dam #3 and about 10 to 15 feet from west bank (11-03-06; 2:47 pm) N 34.83860˚ W 78.82367˚ |



Figure 1. Vicinity map for Cape Fear River lock and dam sediment sampling sites. Green boxes delineate areas enlarged on subsequent maps.

Figure 2. Cape Fear River sediment sampling site downstream of Fayetteville

(furthest upstream sample in this assessment).

Figure 3. Cape Fear River sediment sampling sites between confluence with Rock

Fish Creek and Lock and Dam #3.

Figure 4. Cape Fear River sediment sampling sites upstream and downstream of

Lock and Dam #3.

Figure 5. Cape Fear River sediment sampling site at Tar Heel Landing.

Figure 6. Cape Fear River sediment sampling site upstream of Elizabethtown.

Figure 7. Cape Fear River sediment sampling sites downstream of Elizabethtown

and upstream of Lock and Dam #2.

Figure 8. Cape Fear River sediment sampling sites upstream of Lock and Dam #1,

near Elwells Ferry

.

Products, Miami, OK) for toxicity testing. Samples were stored in a cooler on ice (about 4oC) in the field. Upon reaching the Service lab in Raleigh each evening, samples were stored refrigerated (about 4oC). The analytical chemistry fractions were held at this temperature for seven days when a refrigerator thermostat failed causing samples to be frozen (at least -10oC) overnight. The samples were subsequently transferred to another refrigerator. The temperature change would not affect analyses of elemental contaminants because their concentrations cannot be altered by freezing. Chemists we conferred with advised that significant degradation of the polycyclic aromatic hydrocarbons (PAHs) was also unlikely, so the analytical chemistry fractions were retained for analyses. The sample fractions for toxicity testing had been shipped prior to the temperature change.

*Sediment toxicity tests*

Nine samples were sent to the U.S. Geological Survey Columbia Environmental Research Center (CERC), Columbia, MO by overnight delivery on November 7, 2006. Toxicity tests included 28-d whole-sediment exposures of *Hyallela azteca* (freshwater amphipod) evaluating effects on growth and survival, 10-d whole-sediment exposures of *Chironomus dilutus* (freshwater midge) evaluating effects on growth and survival (USEPA 2000a, ASTM 2007), and 2-d sediment elutriate (water-extractable fraction of the sediment) exposures of *Ceriodaphnia dubia* (freshwater cladoceran) evaluating effects on survival (USEPA 1993a). This battery of toxicity tests was selected to help evaluate the toxicity of sediments, both in-place and upon re-suspension. Toxicity tests were performed on nine of the twelve samples based on prioritizing areas of interest from the results of the Tier 1 assessment (USFWS 2006).

Test organism culture - Amphipods were mass cultured at 23oC with a luminance of about 800 lux using 80-L glass aquaria containing 50 L of well water (hardness 283 mg/L as CaCO3, alkalinity 255 mg/L as CaCO3, pH 7.8; Ingersoll et al. 2002). Amphipods used to start the tests were obtained by collecting amphipods that passed through a #35 U.S. Standard size (500-µm opening) and were collected on a #40 (425-um opening) sieve placed under water. Amphipods were held in 3 L of water with gentle aeration and with a small amount of Tetramin® and a maple leaf for 24 hours before the start of the test. Use of this sieving method resulted in mean amphipod lengths at the start of the exposure of 1.78 mm (0.09 standard error of the mean (SE)).

Midge were mass cultured under static conditions in 5.7-L polyethylene cylindrical chambers containing about 3 L of water and 25 ml of silica sand as a substrate at a temperature of 25oC and a light intensity of about 800 lux. Second instar midge (10-d old) to start the tests were obtained by isolating <24 h old midge larvae ten days before starting the toxicity test.

Cladocerans where cultured under static conditions in 30-ml disposable clear plastic cups each containing 15 ml of well water and one adult at a temperature of 25oC and a light intensity of about 200 lux (USEPA 1993a). Cladocerans in each cup were fed 0.1 ml/d Yeast-Cerophyll-trout chow (YCT; 1.7 to 1.9 g/L) and 0.1 ml/d unicellular green algae (3 x 107 cells of *Selenastrum capricornutum*). Neonates (<24 hours) to start the tests were obtained from adults that produce eight or more young in their third brood or subsequent broods.

Whole-sediment toxicity tests - Whole-sediment toxicity tests with *H. azteca* were conducted for 28 d and with *C. dilutus* were conducted for 10 d in accordance with methods described in ASTM (2007) and USEPA (2000a) starting within 3 weeks of sediment collection. Endpoints measured in the amphipod exposures included survival and length on Day 28. Endpoints measured in the midge exposures included survival and growth (ash-free dry weight) on Day 10. Test sediments were homogenized in a stainless-steel bowl using a plastic spoon and added to exposure beakers 1 d before test organisms were added (Day -1). Sub-samples of sediment were then collected for physical characterizations and for isolating pore water. Amphipods and midge were exposed to 100 ml of sediment with 175 ml of overlying water in 300-ml beakers with four replicates/treatment. The photoperiod was 16 h light: 8 h dark at a light intensity of about 200 lux at the surface of the exposure beakers and the temperature was 23oC. Each beaker received 2-volume additions/d of overlying water starting on Day -1 (Ingersoll et al. 2002). Overlying water was prepared by diluting well water with deionized water to a hardness of 100 mg/L as CaCO3, alkalinity of 85 mg/L as CaCO3, and pH about 7.8. The water delivery system cycled every 4 h with each cycle delivering 125 ml of water to each beaker. Tests were started on Day 0 by placing 10 amphipods or 10 midge into each beaker using an eyedropper. Amphipods in each beaker were fed 1.0 ml of YCT (1.7 to 1.9 g/L) in a water suspension daily (USEPA 2000a, ASTM 2007). Midge in each beaker were fed 1.5 ml of Zeigler prime tropical flake fish food (red) (Zeigler Bros, Inc., Gardens, PA) (6.0 mg of dry solids) in a water suspension daily (USEPA 2000a, ASTM 2007). Beakers were observed daily for the presence of animals, signs of animal activity (i.e., burrowing), and to monitor test conditions (mainly water clarity).

Midge were isolated from each beaker on Day 10 of the exposure and amphipods were isolated from each beaker on Day 28 of the exposure by pouring off most of the overlying water, gently swirling the remaining overlying water and upper layer of sediment, and washing the sediment through a No. 50 (300-µm opening) U.S. Standard stainless-steel sieve. The materials that were retained on the sieve were washed into a glass pan and the surviving midge and amphipods were removed. This process was repeated with the remainder of the sediment in a beaker if all 10 of the amphipods or midge were not recovered from the upper layer of the sediment. Amphipods from each sediment were counted and preserved in 8% sugar formalin for subsequent length measurements (Ingersoll et al. 2002). Length of amphipods was measured along the dorsal surface from the base of the first antenna to the tip of the third uropod along the curve of the dorsal surface. Amphipod length measurements were made using an EPIX imaging system (PIXCI® SV4 imaging board and XCAP software; EPIX Inc., Buffalo Grove, IL) connected to a computer and a microscope (Ingersoll et al. 2002). Midge from each sediment were counted and dried at 100oC in a drying oven for subsequent ash-free dry weight measurements (ASTM 2007). Ash-free dry weight was obtained by recording the weights after drying, transferring to an ashing oven and ashing at 500oC. Ash-free dry weights were determined by subtracting the ashed weight from the dry weight.

About 50 ml of pore water was isolated from sediment samples by centrifugation at 5200 rpm (7000 x G) for 15 min at 4oC (Kemble et al. 1994). Immediately after pore water was isolated, dissolved oxygen, pH, alkalinity, temperature, conductivity, total ammonia, and hardness were measured using methods outlined in Kemble et al. (1994). Mean characteristics of pore-water water quality (ranges in parentheses) were: pH 6.95 (4.5 to 7.4), alkalinity 491 (248 to 824 mg/L as CaCO3), hardness 298 (144 to 498 mg/L as CaCO3), dissolved oxygen 1.8 (1.0 to 5.7 mg/L), conductivity 1016 (524 to 1597 µmho/cm), total ammonia 13.5 (2.1 to 33.7 mg N/L), and unionized ammonia 0.057 (0.001 to 0.078 mg N/L; Table 5).

In the amphipod test, conductivity, pH, alkalinity, hardness, dissolved oxygen, and total ammonia were measured in overlying test water on Day 0 (the day amphipods were added to the exposure beakers) and Day 28 of the exposure. Conductivity and dissolved oxygen, in overlying water were also measured weekly. Temperature in the water baths containing the exposure beakers was recorded daily. Overlying water pH, alkalinity, total hardness, conductivity and total ammonia measurements were similar among all treatments. Mean characteristics of overlying water quality (ranges in parentheses) are as follows: pH 8.0 (7.9 to 8.0), alkalinity 88 (81 to 94 mg/L as CaCO3), hardness 93 (90 to 96 mg/L as CaCO3), dissolved oxygen 7.1 (6.7 to 7.5 mg/L), conductivity 257 (252 to 263 µmho/cm), total ammonia 0.39 (0.19 to 0.70 mg N/L), and unionized ammonia 0.010 (0.004 to 0.010 mg N/L; Table 7). Dissolved oxygen in overlying water was at or above the acceptable level of 2.5 mg/L in all treatments throughout the study (USEPA 2000a, ASTM 2007).

In the midge test, conductivity, pH, alkalinity, hardness, dissolved oxygen, and total ammonia were measured in overlying test water on Day 0 (the day midge were added to the exposure beakers) and Day 10 of the exposure. Conductivity and dissolved oxygen in overlying water were also measured weekly. Temperature in the water baths holding the exposure beakers was monitored daily. Overlying water pH, alkalinity, total hardness, conductivity and total ammonia measurements were similar among all treatments. Mean characteristics of overlying water quality (ranges in parentheses) are as follows: pH 8.0 (7.6 to 8.1), alkalinity 87 (83 to 96 mg/L as CaCO3), hardness 92 (85 to 100 mg/L as CaCO3), dissolved oxygen 7.4 (7.1 to 7.6 mg/L), conductivity 255 (249 to 263 µmho/cm), total ammonia 0.38 (0.20 to 0.64 mg N/L), and unionized ammonia 0.010 (0.011 to 0.017 mg N/L; Table 8). Dissolved oxygen in overlying water was above acceptable levels of 2.5 mg/L in all treatments throughout the study (USEPA 2000a, ASTM 2007).

Elutriate toxicity tests - Toxicity tests with *C. dubia* were conducted for 2 d in accordance with methods outlined in USEPA (1993a) starting within 5 weeks of sediment collection.Elutriate samples were prepared by mixing one part sediment with four parts water (prepared by diluting well water, the characteristics of which are described at page 13 above under Test organism culture, with deionized water to a hardness of 100 mg/L as CaCO3, alkalinity of 85 mg/L as CaCO3, and pH about 7.8). Elutriate samples were prepared following procedures outlined in USEPA (1993a). About 50 ml of sediment was placed into two 250-ml high-density polypropylene centrifuge tubes and topped off with 200 ml of water. The tubes were sealed and tumbled on a rolling mill for 30 min. The tubes were then be centrifuged for 15 min at 5200 rpm (7000 x G). The water was then decanted through a US Standard #50 stainless steel sieve (300-µm opening) into a 500-ml beaker. A sub-sample of the elutriate samples was collected for water quality characterization, with the remaining sample used in toxicity tests.

The cladoceran test was started on Day 0 with <24-h old *C. dubia*. A total of 10 cladocerans were exposed to each elutriate sample and were exposed individually in 30-ml disposable clear plastic cups containing 15 ml of 100% elutriate test solution. Trays holding the plastic cups were covered with plastic sheets to prevent evaporation. Cladocerans were maintained in a water bath at 25±1º C on a 16 h light: 8 h darkness photoperiod at a light intensity of about 200 lux. Cladocerans were not fed during the exposures. On Day 2, survival of the cladocerans was determined by pouring all the test water into a petri dish and using a light table to facilitate counting the neonates. Four of the elutriate samples that were found to be toxic to *C. dubia* (CF1, CF6, CF10, and CF11) were re-tested in a dilution series prepared 21 d after the start of the initial toxicity tests with 100% elutriate sample. The four samples were prepared by the same procedures outlined above and a 50 % dilution series (elutriate sample was prepared with the 100 mg/L hardness water, testing 100, 50, 25, 12.5, 6.25% elutriate) and a dilution water control.

Conductivity, pH, alkalinity, hardness, dissolved oxygen, and total ammonia were measured in elutriate test water on Day 0 (the day cladocerans were added to the exposure beakers). Temperature in the water baths containing the exposure beakers was recorded daily. Elutriate water pH, alkalinity, total hardness, conductivity and total ammonia measurements were similar among all treatments. Mean characteristics of elutriate water quality (ranges in parentheses) are as follows: pH 7.4 (6.2 to 7.8), alkalinity 100 (32 to 150 mg/L as CaCO3), hardness 100 (64 to 154 mg/L as CaCO3), dissolved oxygen 5 (4 to 7 mg/L), conductivity 359 (259 to 491 µmho/cm), total ammonia 3.63 (0.8 to 6.7 mg N/L), and unionized ammonia 0.049 (0.001 to 0.088 mg N/L). Dissolved oxygen in overlying water was above 2.5 mg/L in all treatments throughout the study.

*Sediment chemical and physical analyses*

Elemental contaminants and PAHs were targeted for analyses in all samples; in addition to being detected in the assessment area above threshold effect levels by others (USFWS 2006), these classes of compounds include many common pollutants. Additionally, these compounds have consensus-based freshwater effects sediment quality guidelines (MacDonald et al. 2000, USEPA 2000b) with which to evaluate the results.

Sediment samples were delivered to Environmental Conservation Laboratories, Inc. (ENCO) in Cary, NC on November 9, 2006. ENCO has the North Carolina Laboratory Certification for the requested analyses. Sediment samples were analyzed for Al, As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Se, Ag, and Zn by USEPA method 6010B (inductively coupled plasma-atomic emission spectrometry, or ICP-AES). Analyses of mercury in sediment samples were by USEPA method 7471A (cold-vapor atomic absorption spectrophotometry). ENCO analyzed sediments for PAHs, including 1-methylnaphthalene, 2-methylnaphthalene, acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorine, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene and pyrene, by USEPA method 8270C (gas chromatography / mass spectrometry, or GC/MS).

Pore-water and elutriate samples prepared by CERC (described above) were also analyzed for elemental contaminants by ENCO. Elements in the analyses included Al, As, Cd, Cr, Cu, Fe, Pb, Mn, Ni, Se and Zn by USEPA method 200.8 (Trace Elements by ICP/Mass Spectrometry). This method was also used to measure Mg and Ca which provided the measures of hardness for these samples (by the calculation method in Standard Methods SM 2340B).

Physical characterization of whole sediments included percentage water, particle size and total organic carbon (TOC). Particle size analysis was conducted at CERC by sieve series following methods described in Foth et al. (1982), Gee and Bauder (1986), and Kemble et al. (1994). The TOC analyses were by the Walkley Black method (Schumacher 2002) and were conducted by Severn Trent Laboratories of Tampa, FL (under subcontract to ENCO).

All analyses were accompanied by batch-specific quality control / quality assurance samples (blanks, spikes, and duplicates). Review of quality assurance data (provided in their entirety in the appendices) indicate acceptable precision and accuracy for all analyses.

Threshold effects concentrations (TECs) and probable effects concentrations (PECs) were used to assess the signficance of the sediment chemistry results. The TECs are concentrations of contaminants in whole sediment below which adverse effects to sensitive aquatic organisms are not expected to occur, and the PECs are effect-based sediment quality guidelines established as concentrations of contaminants in whole sediment above which adverse effects are expected to frequently occur in field-collected sediments (MacDonald et al. 2000). Mean quotients based on PECs were calculated to provide an overall measure of chemical contamination and to support an evaluation of the combined effects of multiple contaminants in sediments (MacDonald et al. 2000; USEPA 2000b). A PEC quotient (PEC-Q) was calculated for each chemical in each sediment sample by dividing the dry-weight concentration of a chemical by the PEC for that chemical. To equally weight the contribution of metals and PAHs in the evaluation of sediment chemistry and toxicity (assuming these diverse groups of chemicals exert some form of collective toxic action), an average PEC-Q for metals was calculated based on concentrations of each metal. A mean overall quotient was then calculated for each sample by summing the average quotient for metals and the quotient for total PAHs, and dividing this sum by two (n = 2 quotients/sample). Use of this approach for calculating the quotients was selected to avoid over-weighting the influence of an individual chemical (e.g., a single metal) on the combined mean quotient (USEPA 2000b).

Relationships between toxicity and concentrations of PAHs in sediment samples were calculated using equilibrium partitioning sediment guideline toxic units (ESGTU; USEPA 2003). The ESGTUs are a way of summarizing many PAHs compounds into one representative number. An ESGTU is basically the organic carbon normalized concentration of a chemical divided by a toxicity threshold (this is a toxic unit for the specific chemical). Then the ESGTU for specific chemicals are summed for each sample yielding ΣESGTUs.

*Statistical analyses*

Statistical analyses for the amphipod exposures were conducted using one-way analysis of variance (ANOVA) at *p* = 0.05 for all endpoints except length which was analyzed using a one-way nested ANOVA at *p* = 0.05 (amphipods nested within a beaker; Snedecor and Cochran 1982). Before statistical analyses were performed, all data were tested for normality. Variance among treatment means for both endpoints was heterogeneous, therefore, a rank analysis of variance was performed and mean differences were determined using a t-test on ranked means (at *p* = 0.05). A sample was designated as toxic when mean survival or growth was significantly reduced relative to the control sediment. Statistical analyses on toxicity tests were performed with Statistical Analysis System programs (SAS 2001). Pairwise correlations among the *C. dubia* survival results and all sediment and elutriate chemical analyses were calculated with JMP 5.1.2 software (SAS Institute, Inc., Cary, NC).

Contaminant concentrations were analyzed for goodness of fit to a normal distribution by the Shapiro-Wilk test (JMP 5.1.2, SAS Institute, Inc.). All elemental contaminants concentrations were normally-distributed. Most of the sediment PAH concentrations were not normally-distributed and these were log-transformed prior to additional analyses. No statistical analyses were conducted for compounds detected in less than 40% of the samples, these included (in sediment samples) silver, 1-methylnaphthalene, 2-methylnaphthalene, acenaphthene, acenapthylene, anthracene, dibenzo(a,h)anthracene, fluorene and naphthalene. For analytes present in at least 40% of samples at concentrations greater than detection limits, a value of the detection limit was substituted for non-detects in statistical comparisons. For analytes reported as estimated values (near the detection limits), the estimated concentration was used.

**Results and Discussion**

*Toxicity tests*

Control survival of *H. azteca* was 98% and met the test acceptability requirment of 80% survival recommended in USEPA (2000a) and ASTM (2007). Control survival of *C. dilutus* was 65% and did not meet the test acceptability requirment of 70% survival recommended in USEPA (2000a) and ASTM (2007). USEPA (2000a) and ASTM (2007) state that a study in which

organisms in the negative control do not meet performance criteria must be considered questionable because it suggests that adverse factors affected the response of test organisms. Some studies have reported cases where performance in the negative control is poor, but

performance criteria are met in reference sediment included in the study design. In these cases, it might be resonable to infer that other samples that show good performance are probably

not toxic; however, any samples showing poor performance should not be judged to have shown toxicity, since it is unknown whether the adverse factors that caused poor control

performance might have also caused poor performance in the test treatments (USEPA 2000a, ASTM 2007). In the current study, survival of midge in all of the test sediments was above 75% and was frequently above 90% suggesting that the test sediments were not toxic to midge. No reason for the poor performance of midge in the control sediment could be identified other than the control sample had been held in the laboratory for over three years for use in routine testing.

Whole sediments had no signficant effect on survial or growth in 28-d toxicity tests with *H. azteca* or in 10-d tests with *C. dilutus* (Table 2). Results indicate the contaminants associated with the whole-sediment samples were not chronically toxic to amphipods or midge.

In 2-d sediment elutriate (water-extractable fraction) tests with *C. dubia*, statistically-significant reductions in survival occurred in four (CF 11, 10, 1 and 6) of the nine exposures (Table 2). Sample site CF 11 is downstream of Borden Chemical and the 25 million gallon per day [MGD] discharge of Fayetteville-Cross Creek wastewater treatment plant. Site CF 10 is downstream of the Fayetteville-Rockfish Creek wastewater treatment plant discharge. However, site CF 1 (upstream of Elizabethtown) and CF 6 (near Locks Point) have no known nearby pollutant

Table 2. Response of *Hyalella azteca* in 28-d whole-sediment exposures, *Chironomus dilutus* in

10-d whole-sediment exposures, and *Ceriodaphnia dubia* in 2-d elutriate exposures prepared from sediment samples collected from the Cape Fear River and to a control sediment (WB). Means (standard error of the means in parentheses) with an asterisk (CF 11, 10, 1 and 6) within a column are significantly different than the control (*p* <0.05).

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | AmphipodSurvival (%) | AmphipodGrowth (mm) | MidgeSurvival (%) | MidgeGrowth (mg) | CladoceranSurvival (%) |
| 112.9 | CF 11 | 98 (2.50) | 4.25 (0.09) | 93 (4.79) | 0.99 (0.02) |  20 \* |
| 108.4 | CF 10 | 100 (0.00) | 4.37 (0.07) | 75 (12.9)  | 0.74 (0.08) |  0 \* |
| 105.9 | CF 9 | 93 (4.15) | 4.04 (0.06) | 85 (6.45) | 1.03 (0.04) | 70 |
| 93.4 | CF 4 | 90 (5.77) | 4.12 (0.09) | 88 (6.29) | 1.08 (0.13) | 80 |
| 88.5 | CF 5 | 98 (2.50) | 4.10 (0.05) | 88 (2.50) | 1.03 (0.05) | 80 |
| 76.1 | CF 1 | 98 (2.50) | 4.34 (0.05) | 98 (2.50) | 0.86 (0.04) |  0 \* |
| 71.8 | CF 3 | 100 (0.00) | 4.03 (0.07) | 95 (2.89) | 0.93 (0.06) | 80 |
| 44.7 | CF 6 | 98 (2.89) | 3.96 (0.08) | 93 (4.79) | 0.93 (0.05) |  40 \* |
| 39.0 | CF 8 | 95 (2.89) | 4.32 (0.08) | 95 (2.89) | 1.04 (0.05) | 80 |
|  | WB | 98 (2.50) | 4.32 (0.06) | 65 (8.66) | 0.51 (0.06) | 80 |

sources. As discussed later in this report, elutriate sample toxicity appears to be related to sediment manganese concentrations rather than any particular point source. Tables 3 through 8 summarize the chemistry of elutriates and overlying water in the whole-sediment toxicity tests.

*Whole-sediment analytical chemistry*

The reports from ENCO and Severn Trent are reprinted in Appendix A and summarized here. Total organic carbon in the samples averaged 3.1% and the average percent fines (sum of silt and clay fraction) was 55% indicating depositional areas were located for the assessment (Table 9).

There are no North Carolina or federal sediment quality criteria, but Tables 10 and 11 compare the elemental contaminants and PAHs in whole sediment to the TECs and PECs of McDonald et al. (2000). Florida and Wisconsin recommend TECs and PECs for use as guidance in their

programs, including evaluation of dredged material and risk assessment of contaminated sites (MacDonald et al. 2003). While no regulatory implications are inferred in our use of the TECs and PECs, sediment quality guidelines like these have been found to offer good utility in site assessment by various government and non-government organizations (Wenning et al. 2005).

Table 3. Water quality characteristics in 2-d elutriate exposures with *Ceriodaphnia dubia* prepared from Cape Fear River sediment samples and a control sediment (WB).

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID |  | Dissolved Oxygen (mg/L) | Conductivity (umho @25oC) | pH | Total Ammonia (mg/L) | Hardness (mg/L as CaCO3) | Alkalinity (mg/L as CaCO3) | Sulfide (mg/L) | Dissolved Organic Carbon (mg/L) |
|  |  | Lab | CERC | CERC | CERC | CERC | ENCO | CERC | MERC | CERC |
| 112.9 | CF 11 |  | 4 | 478 | 7.8 | 6.7 | 154 | 150 | 0.013 | 2.77 |
| 108.4 | CF 10 |  | 5 | 491 | 7.8 | 3.6 | 149 | 130 | 0.021 | 4.51 |
| 105.9 | CF 9 |  | 5 | 299 | 7.7 | 2.0 |  85.8 |  92 |  0.0195 | 2.93 |
| 93.4 | CF 4 |  | 6 | 302 | 7.7 | 2.7 |  63.8 |  84 | 0.049 | 3.15 |
| 88.5 | CF 5 |  | 5 | 262 | 7.6 | 2.8 |  83.8 |  93 | 0.019 | 2.75 |
| 76.1 | CF 1 |  | 5 | 404 | 7.0 | 6.4 | 135 | 120 | 0.016 | 7.25 |
| 71.8 | CF 3 |  | 6 | 297 | 7.1 | 3.5 |  81.7 |  88 |  0.0175 | 4.25 |
| 44.7 | CF 6 |  | 5 | 411 | 7.6 | 6.2 | 101 | 120 |  0.0165 | 4.71 |
| 39.0 | CF 8 |  | 5 | 259 | 7.7 | 1.6 |  70.6 |  86 | 0.045 | 4.45 |
|  | WB |  | 7 | 387 | 6.2 | 0.8 |  78.0 |  32 | 0.035 | 4.85 |

Table 4. Elemental contaminant concentrations (µg/L) of 2-d elutriate exposures with *Ceriodaphnia dubia* prepared from Cape Fear River sediment samples and a control sediment (WB).

|  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Al | As | Cd | Cr | Cu | Fe | Pb | Mn | Ni | Se | Zn |
| 112.9 | CF 11 | 6680 B | 6.07 | 0.26 J | 27.9 | 15.4 | 32200 | 113 | 15700 | 19.5 | 0.74 J |  79.9 |
| 108.4 | CF 10 | 2680 B | 7.42 | 0.12 U |  6.85 |  9.32 | 26800 | 366 | 18600 | 10.8 | 0.95 J |  63.8 |
| 105.9 | CF 9 | 9590 B | 4.89 | 0.33 J | 19.7 | 16.9 | 24400 |  59.3 | 7710 | 13.2 | 0.51 J | 133 |
| 93.4 | CF 4 | 7900 B | 3.91 | 0.27 J | 18.0 | 15.4 | 21400 |  47.7 | 5700 | 10.8 | 0.49 J |  79.2 |
| 88.5 | CF 5 | 12000 B | 5.17 | 0.46 J | 23.1 | 18.0 | 29700 |  62.2 | 6780 | 12.9 | 0.42 J  | 110 |
| 76.1 | CF 1 | 5380 B | 5.01 | 0.24 J | 24.1 | 11.7 | 24400 |  44.8 | 11500 | 11.4 | 1.10  |  77.0 |
| 71.8 | CF 3 | 12300 B | 4.86 | 0.41 J | 23.2 | 18.8 | 27300 | 110 | 6900 | 13.6 | 0.73 J | 108 |
| 44.7 | CF 6 | 3970 B | 6.46 | 0.13 J |  9.51 |  9.01 | 25100 |  49.4 | 9250 |  8.20 | 0.78 J |  76.1 |
| 39.0 | CF 8 | 15600 B | 5.44 | 0.39 J | 30.3 | 24.2 | 33200 | 231 | 5010 | 19.8 | 0.52 J | 140 |
|  | WB | 513 B | 1.27 |  0.12 U |  5.92 |  9.96 | 17100 |  31.2 | 2690 |  7.84 | 0.32 U |  20.2 |

B = aluminum was detected in the blank at 47.7 µg/L. Because sample results were typically two orders of magnitude higher than this concentration, the presence of aluminum in the blank should not affect interpretation of results

J = less than reporting limit (estimated value)

U = not detected

Table 5. Water quality characteristics of pore water isolated from Cape Fear River whole-sediment samples and a

 control sediment (WB). NA = not analyzed

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID |  | Dissolved Oxygen (mg/L) | Conductivity (umho@25oC) | pH | Total Ammonia (mg/L) | Alkalinity (mg/L as CaCO3) | Hardness (mg/L as CaCO3) |
|  |  | Lab | CERC | CERC | CERC | CERC | CERC | ENCO |
| 112.9 | CF 11 |  | 1.1 | 1471 | 7.2 | 21.4 | 760 | 498 |
| 108.4 | CF 10 |  | 1.0 | 1597 | 7.3 | 11.5 | 824 | 507 |
| 105.9 | CF 9 |  | 1.4 |  824 | 7.4 | 10.2 | 380 | 246 |
| 93.4 | CF 4 |  | 1.3 |  894 | 7.1 | 10.9 | 268 | 144 |
| 88.5 | CF 5 |  | 1.2 |  750 | 7.2 | 7.2 | 340 | 231 |
| 76.1 | CF 1 |  | 2.7 | 1243 | 7.2 | 33.7 | 582 | 377 |
| 71.8 | CF 3 |  | 1.3 |  840 | 7.1 | 13.4 | 400 | 218 |
| 44.7 | CF 6 |  | 1.0 | 1235 | 7.3 | 21.5 | 616 | 382 |
| 39.0 | CF 8 |  | 1.3 |  524 | 7.2 | 3.6 | 248 | 161 |
|  | WB |  | 5.7 |  779 | 4.5 | 2.1 | NA | 220 |

Table 6. Elemental contaminant concentrations (µg/L) of pore water isolated from Cape Fear River whole-sediment samples and a control sediment (WB).

|  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Al | As | Cd | Cr | Cu | Fe | Pb | Mn | Ni | Se | Zn |
| 112.9 | CF 11 |  0.6 U | 14.5 | 0.30 J |  1.40 | 14.8 | 102000 | 13.7 | 48800 |  5.77 | 2.01 |  76.4 |
| 108.4 | CF 10 |  823 B | 22.5 | 0.34 J |  0.74 J | 19.8 | 102000 | 19.7 | 64600 |  9.06 | 2.95 |  52.6 |
| 105.9 | CF 9 | 2060 B | 11.6 | 0.28 J |  5.75 | 18.5 |  61900 | 19.3 | 23400 |  6.21 | 1.46 |  68.8 |
| 93.4 | CF 4 | 7090 B |  7.67 | 0.83 J | 16.6 | 48.5 |  69000 | 54.2 | 19200 |  7.98 | 1.51 | 122 |
| 88.5 | CF 5 | 3130 B | 10.8 | 0.42 J |  8.50 | 22.3 |  68000 | 23.2 | 20600 |  6.77 | 1.26 | 103 |
| 76.1 | CF 1 | 1380 B | 14.7 | 0.37 J |  2.22 | 19.5 |  96100 | 19.6 | 43300 |  6.22 | 2.17 |  59.5 |
| 71.8 | CF 3 | 4540 B | 10.7 | 0.66 J | 11.6 | 36.5 |  81500 | 36.2 | 24900 |  8.17 | 1.83 | 121 |
| 44.7 | CF 6 | 1650 B | 18.6 | 0.34 J |  2.37 | 18.7 | 111000 | 18.2 | 37200 |  6.99 | 2.41 |  67.0 |
| 39.0 | CF 8 | 8680 B |  9.62 | 0.63 J | 22.7 | 32.7 | 52900 | 32.6 | 10300 |  9.01 | 1.37 | 127 |
|  | WB |  0.6 U |  2.56 | 0.39 J |  2.14 | 34.6 | 67900 |  6.49 |  9830 | 17.5 | 0.78 J |  80.4 |

B = aluminum was detected in the blank at 47.7 µg/L. Because sample results were typically two orders of magnitude higher than this concentration, the presence of aluminum in the blank should not affect interpretation of results

J = less than reporting limit (estimated value)

U = not detected

Table 7. Mean water quality characteristics of overlying water in whole-sediment toxicity tests with *Hyalella azteca*

 and Cape Fear River sediments and a control sediment (WB). NA = not applicable.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Dissolved oxygen(mg/L) | Conductivity(µmho@25oC) | Hardness(mg/L as CaCO3) | Alkalinity(mg/L as CaCO3) | pH | Totalammonia(mg/L) |
| 112.9 | CF 11 | 7.1 | 261 | 96 | 91 | 8.0 | 0.22 |
| 108.4 | CF 10 | 6.7 | 263 | 94 | 94 | 8.0 | 0.70 |
| 105.9 | CF 9 | 7.4 | 255 | 92 | 86 | 8.0 | 0.24 |
| 93.4 | CF 4 | 7.1 | 254 | 92 | 87 | 8.0 | 0.34 |
| 88.5 | CF 5 | 7.2 | 253 | 93 | 87 | 8.0 | 0.38 |
| 76.1 | CF 1 | 7.0 | 259 | 95 | 90 | 7.9 | 0.66 |
| 71.8 | CF 3 | 7.2 | 254 | 90 | 88 | 8.0 | 0.50 |
| 44.7 | CF 6 | 7.0 | 257 | 92 | 90 | 8.0 | 0.39 |
| 39.0 | CF 8 | 7.2 | 254 | 95 | 86 | 7.9 | 0.23 |
|  | WB | 7.5 | 252 | 90 | 81 | 7.9 | 0.19 |

Table 8. Mean water quality characteristics of overlying water in whole-sediment toxicity tests with *Chironomus dilutus*

 and Cape Fear River sediments and a control sediment (WB).

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Dissolved oxygen(mg/L) | Conductivity(µmho@25oC) | Hardness(mg/L as CaCO3) | Alkalinity(mg/L as CaCO3) | pH | Totalammonia(mg/L) |
| 112.9 | CF 11 | 7.3 | 263 | 85 | 83 | 8.0 | 0.20 |
|  108.4 | CF 10 | 7.5 | 260 | 95 | 96 | 8.0 | 0.64 |
|  105.9 | CF 9 | 7.6 | 252 | 90 | 86 | 8.1 | 0.27 |
|  93.4 | CF 4 | 7.5 | 250 | 92 | 85 | 8.0 | 0.29 |
|  88.5 | CF 5 | 7.6 | 250 | 92 | 86 | 7.9 | 0.40 |
|  76.1 | CF 1 | 7.5 | 255 | 100 | 89 | 7.9 | 0.55 |
|  71.8 | CF 3 | 7.5 | 252 | 88 | 86 | 8.0 | 0.55 |
|  44.7 | CF 6 | 7.1 | 258 | 93 | 88 | 8.0 | 0.43 |
|  39.0 | CF 8 | 7.1 | 249 | 96 | 83 | 7.9 | 0.25 |
|  | WB | 7.3 | 263 | 85 | 83 | 8.0 | 0.20 |

Table 9. Total organic carbon, grain size and moisture measured in whole-sediment samples collected

from the Cape Fear River.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Total Organic Carbon (%) | Sand (%) | Clay (%) | Silt (%) | Moisture (%) |
|   |  | STL1 | CERC2 | CERC | CERC | ENCO3 |
| 112.9 | CF 11 | 4.5 | 48 | 19 | 33 | 48.4 |
| 108.4 | CF 10 | 4.3 | 32 | 25 | 44 | 59.6 |
| 105.9 | CF 9 | 2.7 | 62 | 18 | 19 | 46.3 |
| 95.2 | CF 12 | 4.1 | 78 | 18 | 5 | 51.9 |
| 93.4 | CF 4 | 2.0 | 37 | 20 | 44 | 45.4 |
| 88.5 | CF 5 | 1.9 | 53 | 27 | 21 | 40.3 |
| 76.1 | CF 1 | 2.9 | 31 | 24 | 44 | 53.3 |
| 72.8 | CF 2 | 2.9 | 45 | 22 | 33 | 59.4 |
| 71.8 | CF 3 | 3.5 | 36 | 24 | 40 | 49.7 |
| 46.3 | CF 7 | 3.7 | 41 | 24 | 35 | 65.0 |
| 44.7 | CF 6 | 2.6 | 28 | 30 | 41 | 59.4 |
| 39.0 | CF 8 | 2.2 | 46 | 20 | 35 | 55.0 |

1STL = Severn Trent Laboratories, Inc., Tampa, FL

2CERC = USGS Columbia Environmental Research Center, Columbia, MO

3ENCO = Environmental Conservation Laboratories, Inc., Cary, NC and Jacksonville, FL

Table 10. Elemental contaminants in whole-sediment samples collected from the Cape Fear River. All data are mg/kg dry weight (parts per million). For each element, results are compared to threshold-effects concentration (TEC) guidelines of MacDonald et al. (2000) -- values below which adverse effects to sensitive aquatic organisms are not expected to occur, and probable effects concentrations (PECs) -- values above which adverse effects to sediment dwelling organisms may be expected. No samples exceeded TECs. Silver was below detection in all samples.

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID |  Al |  As | Ba | Cd |  Cr | Cu |  Fe | Pb | Mn |  Hg | Ni | Se |  Zn |
| 112.9 | CF 11 |  6750 | 3.0 |  95.7 | 0.22 | 15.6 | 15.1 |  14800 D | 17.8 |  930 | 0.051 | 5.6 | 1.8 |  76.1 |
| 108.4 | CF 10 | 10500 | 4.3 | 139 | 0.28 | 22.4 | 21.5 |  20600 D | 22.9 | 1610 | 0.101 | 8.2 | 2.5 | 101 |
| 105.9 | CF 9 |  4940 | 1.8 |  64.8 | 0.15 | 11.9 |  9.81 | 10300  | 13.6 |  506 | 0.057 |  3.8 J | 1.3 |  57.5 |
| 95.2 | CF 12 |  8310 | 2.9 | 108 | 0.30 | 19.0 | 17.7 |  16800 D | 19.4 |  693 | 0.073 | 6.4 | 2.1 |  96.6 |
| 93.4 | CF 4 |  9790 D | 3.6 |  97.5 | 0.15 | 18.7 | 15.9 |  20100 D | 16.1  |  777 | 0.064 | 6.2 | 2.5 |  70.7 |
| 88.5 | CF 5 |  5600 | 2.3 |  74.4 | 0.22 | 13.3 | 11.7 |  12400 D | 13.7 |  544 | 0.040 | 4.4 | 1.8 |  62.9 |
| 76.1 | CF 1 |  9470 | 3.6 | 116 | 0.22 | 19.6 | 17.3 |  19600 D | 18.7 | 1040 | 0.087 | 7.1 | 2.7 |  93.8 |
| 72.8 | CF 2 | 10100 | 3.5 | 125 | 0.25 | 20.2 | 17.4 |  19800 D | 19.0 | 1050 | 0.097 | 7.7 | 2.5 | 101 |
| 71.8 | CF 3 |  9320 | 3.2 | 113 | 0.24 | 18.8 | 18.0 |  18700 D | 17.4 |  815 | 0.097 | 6.8 | 2.4 |  93.2 |
| 46.3 | CF 7 | 13000  | 4.3 | 148 | 0.22 | 24.1 | 19.2 |  24400 D | 21.9 | 1260 | 0.084 | 9.1 | 3.0 | 105 |
| 44.7 | CF 6 |  13500 D | 4.3 | 148 | 0.27 | 24.2 | 19.8 |  24700 D | 21.8 | 1160 | 0.092 | 9.0 | 3.1 | 109 |
| 39.0 | CF 8 | 8730 | 2.7 | 100 | 0.24 | 16.7 | 13.6 |  16600 D | 15.2 |  595 | 0.072 | 6.4 | 1.9 |  78.7 |
| TEC  |  |  |  9.79 |  | 0.99 |  43.4 | 31.6 |  | 35.8 |  |  0.18  | 22.7 |  | 121 |
| PEC  |  |  | 33 |  | 4.98 | 111 | 149 |  | 128 |  |  1.06 | 48.6 |  | 459 |

D = data reported from a dilution

J = less than reporting limit (estimated value)

Table 11. Polycyclic aromatic hydrocarbons measured in whole-sediment samples collected from the Cape Fear River. All data are µg/kg dry weight (parts per billion). For each compound, results are compared to threshold-effects concentration (TEC) guidelines of MacDonald et al. (2000) -- values below which adverse effects to sensitive aquatic organisms are not expected to occur, and probable effects concentrations (PECs) -- values above which adverse effects to sediment dwelling organisms may be expected. Only CF 5 and CF 11 exceeded TECs (exceedences designated with and asterisk), and no samples exceeded PECs. 1-methylnaphthalene, 2-methylnaphthalene, acenaphthene, acenapthylene, anthracene, fluorene and naphthalene were detected in one or no samples. J = less than reporting limit (estimated value), U = not detected.

|  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Benzo(a)anthracene  | Benzo(a)pyrene  | Benzo(b)fluoranthene  | Benzo(g,h,i)perylene  | Benzo(k)fluoranthene  | Chrysene  | Dibenzo(a,h)anthracene | Fluoranthene  | Indeno(1,2,3-cd)pyrene  | Phenanthrene  | Pyrene  |
| 112.9 | CF 11 |  194\* |  232\* | 316 | 200 | 110 |  226\* | 168\* |  355 | 232 |  136 |  336\* |
| 108.4 | CF 10 |  57.8J |  57.8J |  90.8 | 124 |  17.1U |  66.0J |  27.7U |  140 | 165 |  74.3J |  107 |
| 105.9 | CF 9 |  55.8J |  55.8J |  86.8 |  99.2 |  37.2J |  55.8J |  20.8U |  99.2 | 136 |  12.8U |  80.6 |
| 95.2 | CF 12 |  55.4J |  55.4J |  90.0 | 111 |  14.3U |  62.3J |  23.3U |  118 | 152 |  41.6J |  90.0 |
| 93.4 | CF 4 |  42.7J |  42.7J |  67.2 |  91.6 |  30.5J |  54.9J |  20.5U |  79.4 | 128 |  30.5J |  67.2 |
| 88.5 | CF 5 |  89.3 |  100 | 162 | 123 |  55.8 |  134 | 134\* |  346 | 156 |  78.1 |  262\* |
| 76.1 | CF 1 |  14.8U |  14.8U |  11.4U |  21.8U |  14.8U |  15.6U |  24.0U |  42.8J |  24.8U |  14.8U |  35.7J |
| 72.8 | CF 2 |  65.7J |  49.3J |  73.9J | 123 |  41.0J |  49.3J |  27.6U |  73.9J | 164 |  17.0U |  65.7J |
| 71.8 | CF 3 |  46.4J |  39.8J |  59.6J |  99.4 |  13.7U |  39.8J |  22.3U |  66.3 | 133 |  13.7U |  59.6J  |
| 46.3 | CF 7 |  19.7U |  19.7U |  15.2U |  29.2U |  19.7U |  20.9U |  32.0U |  47.7J |  33.2U |  19.7U |  25.5U |
| 44.7 | CF 6 |  17.0U |  17.0U |  13.1U |  25.1U |  17.0U |  18.0U |  27.6U |  21.9U |  28.6U |  17.0U |  21.9U |
| 39.0 | CF 8 |  15.3U |  15.3U |  11.8U |  22.7U |  15.3U |  16.2U |  24.9U |  19.8U |  25.8U |  15.3U |  19.8U |
|  | TEC |  108 |  150 |  |  |  |  166 |  33 |  423 |  |  204 |  195 |
|  | PEC | 1050 | 1450 |  |  |  | 1290 |  | 2230 |  | 1170 | 1520 |

No samples exceeded the TECs (concentrations below which adverse effects to sensitive aquatic organisms are not expected to occur) for elemental contaminants; these are therefore considered toxicologically insignificant. Sediment PAHs with freshwater sediment TECs were in excess of TECs at two of the 12 sampling stations: CF 11 (river mile 112.9 near Fayetteville) and CF 5 (river mile 88.5 about 0.2 miles upstream of Tar Heel Landing Road). Five individual PAHs exceeded TECs at these locations, but no samples exceeded the PECs. MacDonald et al. (2000) and USEPA (2000b) concluded that infrequent exceedences of TECs is not associated with sediment toxicity; however, exceedences of PECs (by frequency or by magnitude) is frequently associated with sediment toxicity. Probable effect concentration quotients (PEC-Qs) and PAH equilibrium partitioning sediment guideline toxic units (ΣESGTUs) for sediment samples from the Cape Fear River were all much less than 1 (unitless) indicating no anticipated toxicity from these constituents (Table 12). Collectively, the whole-sediment chemistry results indicate minimal contamination which is consistent with the lack of toxicity in the whole-sediment samples in the amphipod and midge tests (Table 2).

Table 12. Probable effect concentration quotients (PEC-Qs) and polycyclic aromatic hydrocarbon equilibrium partitioning sediment guideline toxic units (ΣESBTUs) for whole-sediment samples collected from the Cape Fear River. Values are unitless with levels of concern typically not indicated until exceeding 1 on these scales.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| River Mile | Sample ID | Mean PEC-Q | Mean Metal PEC-Q  | Total PAH PEC-Q  | ΣESGTU for PAHs |
| 112.9 | CF 11 | 0.089 | 0.105 | 0.072 | 0.160 |
| 108.4 | CF 10  | 0.084 | 0.147 | 0.020 | 0.066 |
| 105.9 | CF 9 | 0.045 | 0.074 | 0.014 | 0.078 |
| 95.2 | CF 12 | 0.069 | 0.120 | 0.017 | 0.058 |
| 93.4 | CF 4 | 0.063 | 0.111 | 0.013 | 0.095 |
| 88.5 | CF 5 | 0.067 | 0.085 | 0.049 | 0.252 |
| 76.1 | CF 1 | 0.065 | 0.123 | 0.007 | 0.043 |
| 72.8 | CF 2 | 0.069 | 0.127 | 0.010 | 0.072 |
| 71.8 | CF 3 | 0.064 | 0.119 | 0.010 | 0.048 |
| 46.3 | CF 7 | 0.077 | 0.146 | 0.007 | 0.038 |
| 44.7 | CF 6 | 0.077 | 0.148 | 0.006 | 0.043 |
| 39.0 | CF 8 | 0.054 | 0.104 | 0.005 | 0.046 |

Although the elutriates and pore-water samples are not surface waters, we compared elemental contaminant concentrations in these media to State water quality standards and action levels (Table 13). This is not a regulatory application of the standards; it is rather a comparison of test results to the standards as toxicological benchmarks, or estimates of safe water column concentrations. Copper, lead, and zinc exceed State standards, an indication that aggressive re-suspension of sediments like those tested could temporarily impair surface water quality.

Table 13. Elutriate and pore water exceedences of North Carolina water quality standards or action levels for elemental contaminants (NCDENR 2004). Analytical data for elutriates and pore water are in Tables 4 and 6.

|  |  |  |  |
| --- | --- | --- | --- |
| Contaminant | Standard or Action Level | Elutriate samples in excess of threshold (%) | Pore water in excess of threshold (%) |
| Arsenic | 50 μg/L | 0 (max = 7.42 μg/L) | 0 (max = 22.5 μg/L) |
| Cadmium | 2 μg/L | 0 (max = 0.46 μg/L) | 0 (max = 0.83 μg/L) |
| Chromium | 50 μg/L | 0 (max = 30.3 μg/L)  | 0 (max = 22.7 μg/L) |
| Copper | 7 μg/L | 100 (max = 24.2 μg/L) | 100 (max = 48.5 μg/L) |
| Lead | 25 μg/L | 100 (max = 366 μg/L)  | 33 (max = 54.2 μg/L) |
| Nickel | 88 μg/L | 0 (max = 19.8 μg/L)  | 0 (max = 9.06 μg/L) |
| Selenium | 5 μg/L | 0 (max = 1.10 μg/L)  | 0 (max = 2.95μg/L) |
| Zinc | 50 μg/L | 100 (max = 140 μg/L)  | 100 (max = 127μg/L) |

In 2-d sediment elutriate tests with *C. dubia*, statistically-significant reductions in survival occurred in four of the nine exposures. Pairwise correlation analyses indicated several sediment and elutriate water chemistry variables were significantly and negatively correlated with *C. dubia* survival in the elutriates (Table 14). Of the correlated variables, hardness, alkalinity, and conductivity are unlikely to impair cladocerans at the concentrations we encountered based on their tolerance of much higher salt concentrations (Mount et al. 1997). The correlations with selenium and arsenic also lack strong toxicological support because effects concentrations for these parameters were generally above concentrations found in the elutriates (USEPA 1996, 2004). Elutriate manganese had among the strongest correlation (r2 = 0.79, *p* = 0.0006) (Figure 10) and the most plausible biological association with reduced *C. dubia* survival.



Figure 10. Relationship between elutriate manganese concentrations and *Ceriodaphnia dubia* survival in 2-d elutriate exposures prepared from sediment samples collected from Cape Fear River locks and dams (and to a control sediment, WB).

Table 14. Pearson’s correlations for 2-d *C. dubia* survival in sediment elutriates versus whole-sediment and elutriate physical and chemical constituents (presented in Tables 3, 4, 9-11). All significant correlations are presented; only select variables that were not significant are included.

|  |  |  |  |
| --- | --- | --- | --- |
| Variable | Correlation to *C. dubia* survival | Threshold | Samples in excess of threshold (%) |
| n | R | r2 | *p* |  |  |
| Elutriate Hardness | 10 | -0.9411 | 0.8857 | <0.0001 |  |  |
| Elutriate Conductivity | 10 | -0.8452 | 0.7144 |  0.0021 |  |  |
| Elutriate Mn | 10 | -0.8873 | 0.7873 |  0.0006 | 7.2 mg/L1 | 56 |
| Elutriate Se | 10 | -0.8785 | 0.7718 |  0.0008 | 220 µg/L2 |  0 |
| Elutriate As | 10 | -0.5880  | 0.3457 |  0.0738 | 760 µg/L3 |  0 |
| Elutriate DO | 10 |  0.5491 | 0.3015 |  0.1002 |  |  |
| Elutriate pH | 10 | -0.1855 | 0.0344 |  0.6079 |  |  |
| Elutriate Ammonia | 10 | -0.7444  | 0.5541 |  0.0135 | 26 mg-N/L4 |  0 |
| Elutriate Alkalinity | 10 | -0.7739 | 0.5989 |  0.0086 |  |  |
| Elutriate Sulfide | 10 |  0.5431 | 0.2950 |  0.1047 |  |  |
| Elutriate DOC | 10 | -0.4689 | 0.2199 |  0.1717 |  |  |
| Sediment TOC | 9 | -0.6633 | 0.4400 |  0.0515 |  |  |
| Sediment Mn | 9 | -0.8044 | 0.6471 |  0.0089 |  |  |
| Sediment Pb | 9 | -0.7415 | 0.5498 |  0.0222 |  |  |

1 Lasier et al. (2000) report a *C. dubia* LC50 (median lethal concentration) of 14.5 mg/L for manganese at hardness of 93 mg/L as CaCO3. This value was multiplied by 0.5 to approximate a lethal effects threshold for the Cape Fear River sediment elutriates (which ranged in hardness from 70.6 to 154 mg/L as CaCO3). Evaluation of acute toxicity data over a large number of tests and species has generally shown that multiplying an LC50 or EC50 by 0.5 provides an estimate of a concentration near or below the lethality threshold. The technical rationale is found in the *Federal Register* notice soliciting comment on USEPA’s methodology for deriving water quality criteria (43 FR 21506, May 18, 1978) (Stephan et al. 1985).

2 USEPA (2004) report a *C. dubia* LC50 of 842 µg/L for selenate and 440 µg/L for selenite. The lower of these values was multiplied by 0.5 to approximate a lethal effects threshold for the Cape Fear River sediment elutriates.

3 USEPA (1996) report a *C. reticulata* LC50 of 1,511 µg/L for arsenic. This was multiplied by 0.5 to approximate a lethal effects threshold for the Cape Fear River sediment elutriates.

4 USEPA (1999) report a *C. acanthina*  LC50 of 25.78 mg total ammonia nitrogen /L at pH=8. This was multiplied by 0.5 to approximate a lethal effects threshold of 12.9 mg-N/L at pH=8. This was adjusted for the average pH (7.6) of our *C. dubia* elutriate tests with USEPA’s (1999) equation 11 resulting in a threshold of 26 mg-N/L for the Cape Fear River sediment elutriates.

Stubblefield and Hockett (2000) report a geometric mean LC50 (median lethal concentration) from eight manganese toxicity tests with *C. dubia* of 15.4 mg/L (data normalized to a hardness of 50 mg/L as CaCO3). Similarly, Lasier et al. (2000) report a *C. dubia* LC50 of 14.5 mg/L for manganese at a hardness of 93 mg/L as CaCO3. The Cape Fear River sediment elutriates ranged in hardness from 70.6 to 154 mg/L as CaCO3. To approximate a threshold at which lethal effects may begin, we multiplied the LC50 reported by Lasier et al. (2000) by 0.5. This yields an estimated lethal effects threshold of 7.2 mg/L for manganese. Evaluation of acute toxicity data over a large number of tests and species has generally shown that dividing an LC50 or EC50 by 2 (i.e., multiplying by 0.5) provides an estimate of a concentration near or below the lethality threshold. The technical rationale for dividing by 2 is found in the *Federal Register* notice soliciting comment on USEPA’s methodology for deriving water quality criteria (43 FR 21506, May 18, 1978) (Stephan et al. 1985). Because the estimate was derived for a large battery of species and chemicals, it may not be accurate for particular individual species and chemical combinations, but it is useful for risk screening purposes.

Elutriate manganese concentrations from samples sites CF 1 (11.5 mg/L), CF 6 (9.25 mg/L), CF 9 (7.71 mg/L), CF 10 (18.6 mg/L) and CF 11 (15.7 mg/L) exceeded the 7.2 mg/L estimated lethal effects threshold concentration for *C. dubia* and manganese (Table 4). The four samples with significant *C. dubia* toxicity were CF 1, CF 6, CF 10 and CF 11 (Table 2). Manganese is a naturally occurring and very common element in soils and surface waters; among the heavy metals, only iron is more abundant in the earth’s crust (ATSDR 2000). Impoundments have the potential to increase metal concentrations due to soil disturbance and increased surface area exposed to water. Elevated manganese was the number one problem associated with water quality downstream of Tennessee dams in a recent evaluation (Arnwine et al 2006).

Anthropogenic enrichment of manganese can occur through burning fossil fuels, steel production, battery manufacturing, animal feed supplements, fertilizers, wastewater treatments plants (using potassium permanganate), manganese-based fungicides, and antiknock fuel additives (ATSDR 2000). This project did not include any manganese source assessments for the Cape Fear River system.

Ammonia is often implicated in sediment toxicity evaluations. While ammonia is a natural constituent of freshwater sediments, excess ammonia from pollution can lead to toxic concentrations, and ammonia can also contribute to the toxicity of other sediment contaminants. Ammonia concentrations at the start of our elutriate test were significantly and inversely correlated with *C. dubia* survival in the elutriate test (Table 14). Unlike manganese, however, the elutriate ammonia concentrations did not exceed those expected to be toxic to *C. dubia*. USEPA (1999) report a *C. acanthina* LC50 of 25.78 mg total ammonia nitrogen /L at pH=8. Multiplying that LC50 by 0.5 yields an approximate lethal effects threshold of 12.9 mg-N/L at pH=8. Because ammonia toxicity decreases as pH decreases, we adjusted this lethal effects threshold for the average pH (7.6) of our *C. dubia* elutriate tests using USEPA’s (1999) equation 11. This yields an estimated total ammonia toxicity threshold of 26 mg-N/L for the Cape Fear River sediment elutriates. The measured elutriate ammonia concentrations (Table 3) were 4- to 14-times less than this estimated ammonia effects threshold.

If it becomes important to more definitively assess the cause of toxicity in the sediment elutriate samples, there are well established toxicity identification evaluation (TIE) approaches that can be applied (USEPA 1993 b, c, Besser at al. 1998, Boucher and Watzin 1999). Non-toxic elutriate samples could be spiked with increasing concentrations of manganese or ammonia to determine toxic concentrations of these constituents to *C. dubia* in a manner inclusive of site-specific elutriate chemistry. Procedures to reduce concentrations of elutriate components (e.g. sequester metals like manganese) also exist to investigate how their presence or absence explains toxicity.

Elutriate tests aid in the evaluation of the effects of suspended sediments (e.g., dredged material evaluations) within the water column. Mobilization of sediments we tested may be a short-term water column concern based on the elutriate toxicity test results. While sediment re-suspension and contaminant release in the elutriate tests may be near a worst case simulation of actual conditions following sediment disturbing activities, additional synthesis is needed to characterize the nature and magnitude of this issue. In particular, data on the modeled or measured sediment re-suspension caused by specific sediment disturbing activities will help put the elutriate test results in context for normal sediment management practices. The joint U.S. EPA and U.S. Army Corps of Engineers Inland Testing Manual (USEPA/USACE 1998) contains sediment fate models which may be helpful for this purpose. Other issues to be further evaluated regarding the significance of the elutriate test results include more background on manganese concentrations and sources in the Cape Fear River and the relative sensitivity of *C. dubia* to other freshwater organisms, particularly those resident to the Cape Fear River.

In summary, 12 sediment samples from within the impounded reaches of three locks and dams on the Cape Fear River were collected in November 2006. Elemental contaminant and PAH concentrations in whole sediments were below those of toxicological significance. Whole-sediment toxicity tests with midges and amphipods showed no adverse effects of the sediments on test organism survival or growth. In 2-d sediment elutriate tests with *Ceriodaphnia dubia*, statistically-significant reductions in survival occurred in samples from four sites. Manganese had among the strongest correlation and the most plausible biological association with reduced *C. dubia* survival; elutriate manganese concentrations exceeded published lethal concentrations for *C. dubia* in all four of the toxic samples. Elutriates and pore water samples are not surface waters, but copper, lead, and zinc in these sediment fractions or preparations frequently exceed State water quality standards, an indication that aggressive re-suspension of sediments like those tested could temporarily impair surface water quality. Based on review of existing data (Tier 1) and results of sediment chemistry and toxicity tests (Tier 2 and 3), contamination in surface sediments behind Cape Fear River locks and dams is unlikely to be a concern in-place. Mobilization of sediments may be a short-term water column concern based on the elutriate toxicity test results. While sediment re-suspension and contaminant release in the elutriate tests may be near a worst case simulation of actual conditions following sediment disturbing activities, additional synthesis is needed to characterize the nature and magnitude of this issue. In particular, data regarding the modeled or measured sediment re-suspension caused by specific sediment disturbing activities (e.g., dredging, dam alterations, etc.) will help put the elutriate test results in context for normal sediment management practices. Sediment disturbing activities proposed for the Cape Fear River would benefit from development of a sediment management plan to address these issues. The work presented here addresses the toxicological properties of the sediments evaluated; it does not address sediments other than surface deposits. This report does not inventory the accumulated sediments or address the potential physical impacts of sediment mobilization; these issues could also be addressed in a sediment a management plan.

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 Appendix A. Analytical Chemistry Reports and Chain of Custody Forms