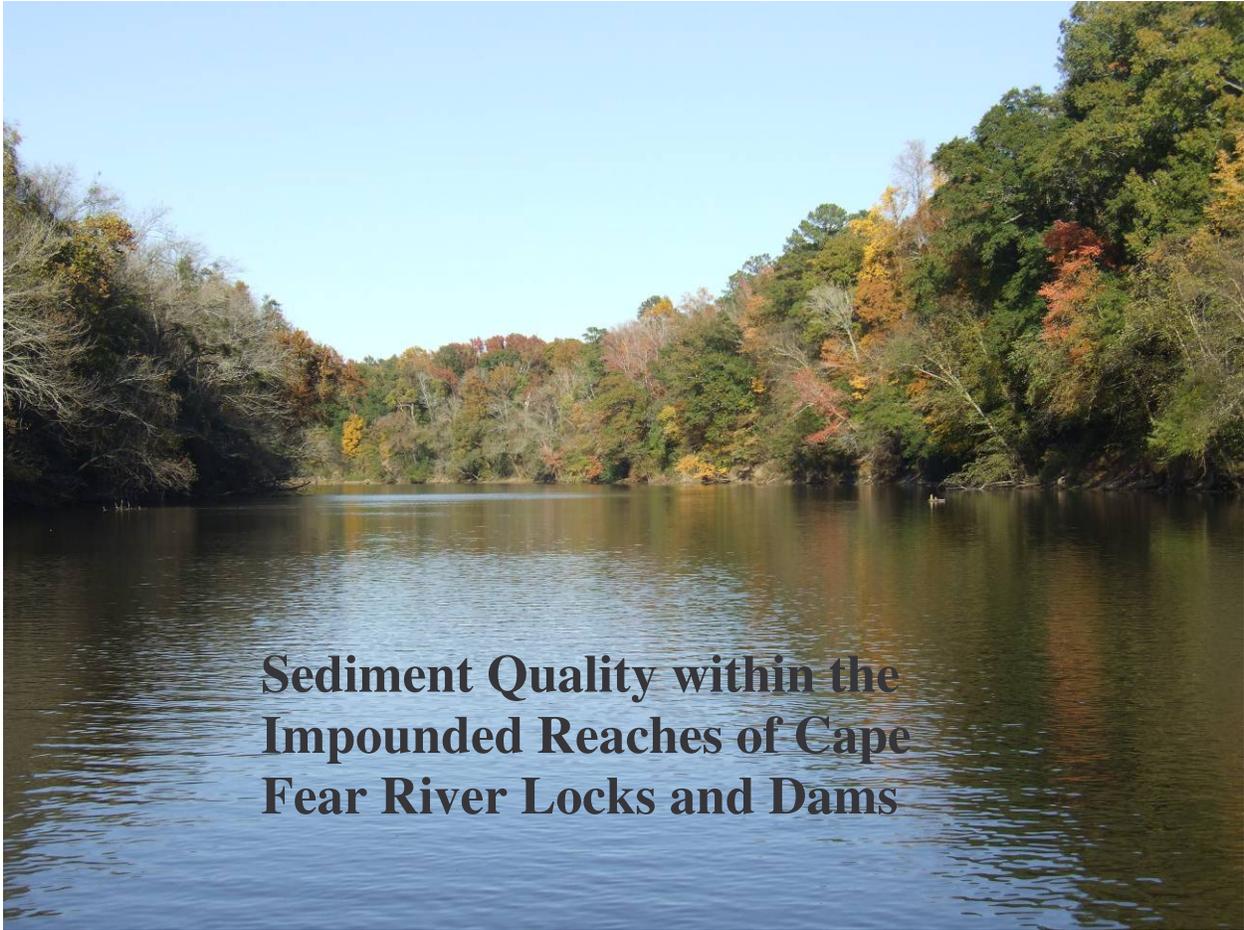


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U.S. Fish and Wildlife Service
U.S. Geological Survey



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

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Draft - May 2007



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U.S. Fish and Wildlife Service
Ecological Services
Raleigh, North Carolina

U.S. Geological Survey
Columbia Environmental Research Center
Columbia, Missouri

13 **Preface**

14
15 The U.S. Fish and Wildlife Service assisted the U.S. Army Corps of Engineers (Wilmington
16 District) in an assessment of the chemical contaminants in, and toxicity of, sediments at Cape
17 Fear River locks and dams. The work was coordinated by Tom Augspurger (Ecologist /
18 Environmental Contaminant Specialist) and Sara Ward (Ecologist / Environmental Contaminant
19 Specialist) in the U.S. Fish and Wildlife Service's Raleigh Field Office and was funded through
20 a transfer agreement between the U.S. Fish and Wildlife Service and the U.S. Army Corps of
21 Engineers. Toxicity tests were performed by the U.S. Geological Survey's Columbia
22 Environmental Research Center (CERC) under the direction of Chris Ingersoll through an intra-
23 agency agreement with the U.S. Fish and Wildlife Service. Other CERC scientists primarily
24 responsible for the toxicity testing component of the project include Nile Kemble and James
25 Kunz. Analytical chemistry was performed by Environmental Conservation Laboratories, Inc. and
26 Severn Trent Laboratories, Inc.

27
28 **The information in this draft is distributed solely for the purpose of pre-dissemination peer**
29 **review under applicable information quality guidelines. It has not been formally**
30 **disseminated by U.S. Fish and Wildlife Service. It does not represent and should not be**
31 **construed to represent any agency determination or policy.** A final report will be prepared
32 that addresses comments received on this draft.

33
34 Additional questions, comments, and suggestions related to this draft are encouraged. Inquires
35 can be directed to the U.S. Fish and Wildlife Service at the following address:

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42
43
44 Suggested citation: Augspurger, T.P., C.G. Ingersoll, N.E. Kemble, J.L. Kunz and S.E. Ward.
45 2007. Sediment Quality within the Impounded Reaches of Cape Fear River Locks and Dams -
46 May 2007 Draft. U.S. Fish and Wildlife Service and U.S. Geological Survey. USFWS, Raleigh,
47 NC.

48 Sediment Quality within the Impounded Reaches of Cape Fear River Locks 49 and Dams

50

51 Executive Summary

52

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

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

197

198 **Introduction**

199

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

213

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

229

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

232

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

235

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

238

239 **Methods**

240

241 *Sample sites*

242

243 Factors considered in determining the number and location of samples included the location of
244 identified potential sources of concern and areas of sediment accumulation. Physical factors
245 considered included the area and depth of potentially affected sediments behind the dams,
246 distribution of sediments, and the length and breadth of the impounded reaches. By comparing
247 the results of surveys of the river performed in 1903 and contemporary (2005 and 2006)
248 hydrographic surveys, the Corps mapped changes in elevation of the river bottom within about
249 1,500-feet upstream of each lock and dam. At Lock and Dam #1, there is only an average of
250 about two feet of sediment accumulation with a maximum of about eight feet of accumulation in
251 a small area near at the base of the dam (and opposite the lock). Similar efforts at Lock and Dam
252 #2 indicated even less accumulation with the greatest depths again near the dam and opposite the
253 lock while there was little overall accumulation at Lock and Dam #3. These depositional areas
254 were among the targeted sites for sample collection.

255

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

268

269 *Sediment sample collection and storage*

270

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

281

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

285 Table 1. Cape Fear River sediment sampling location data and the date and time of collection.
 286

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 and about 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.

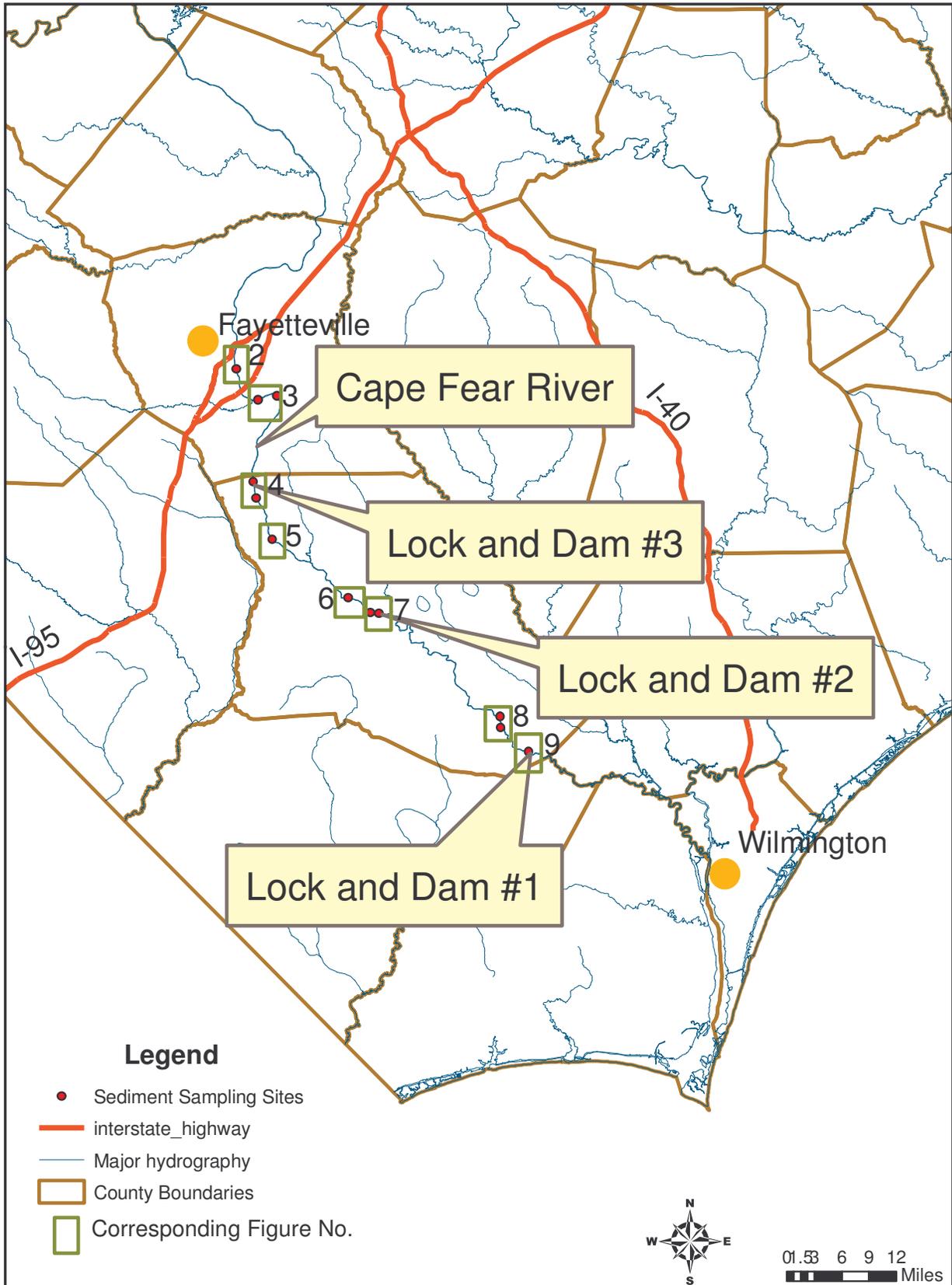
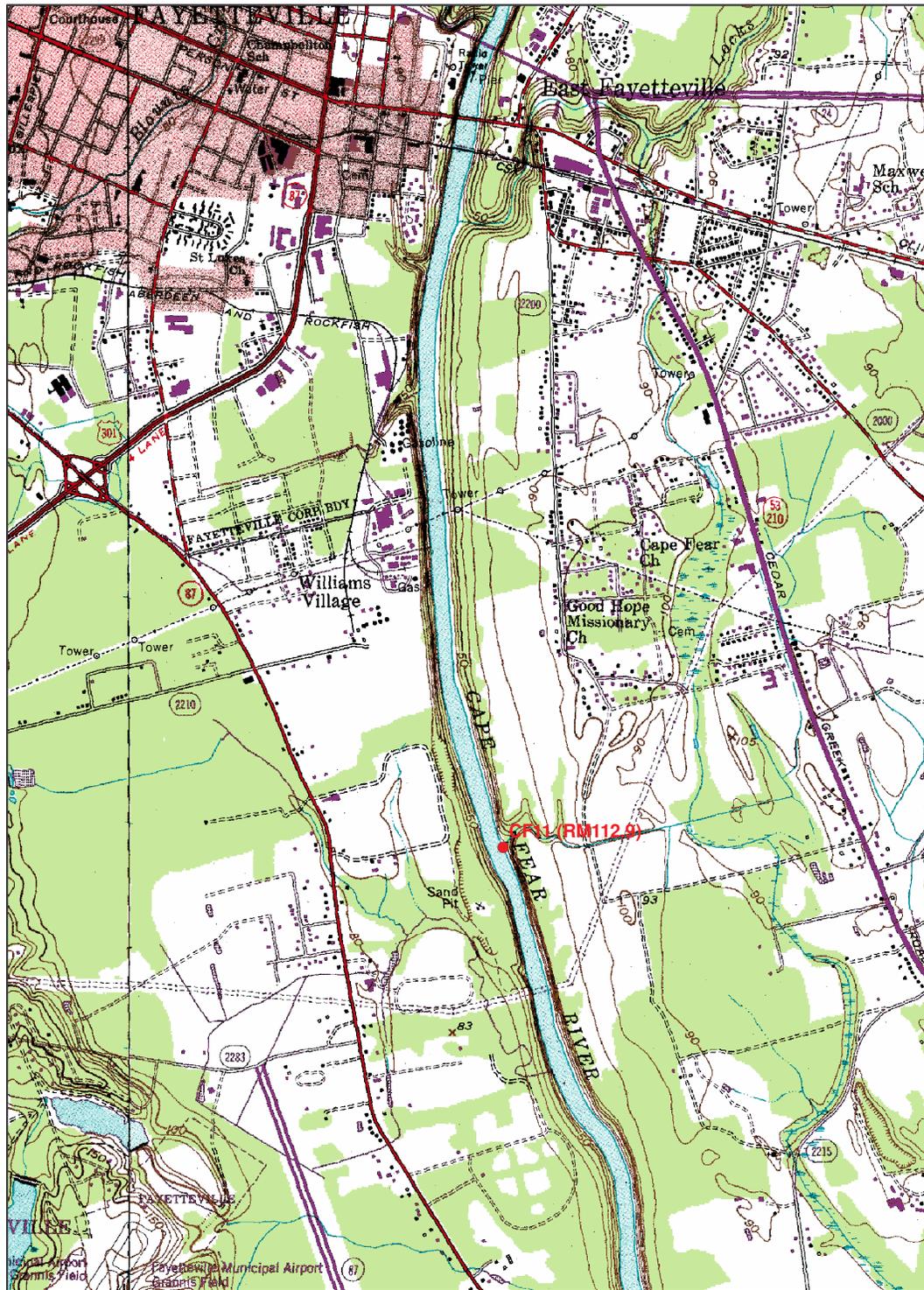
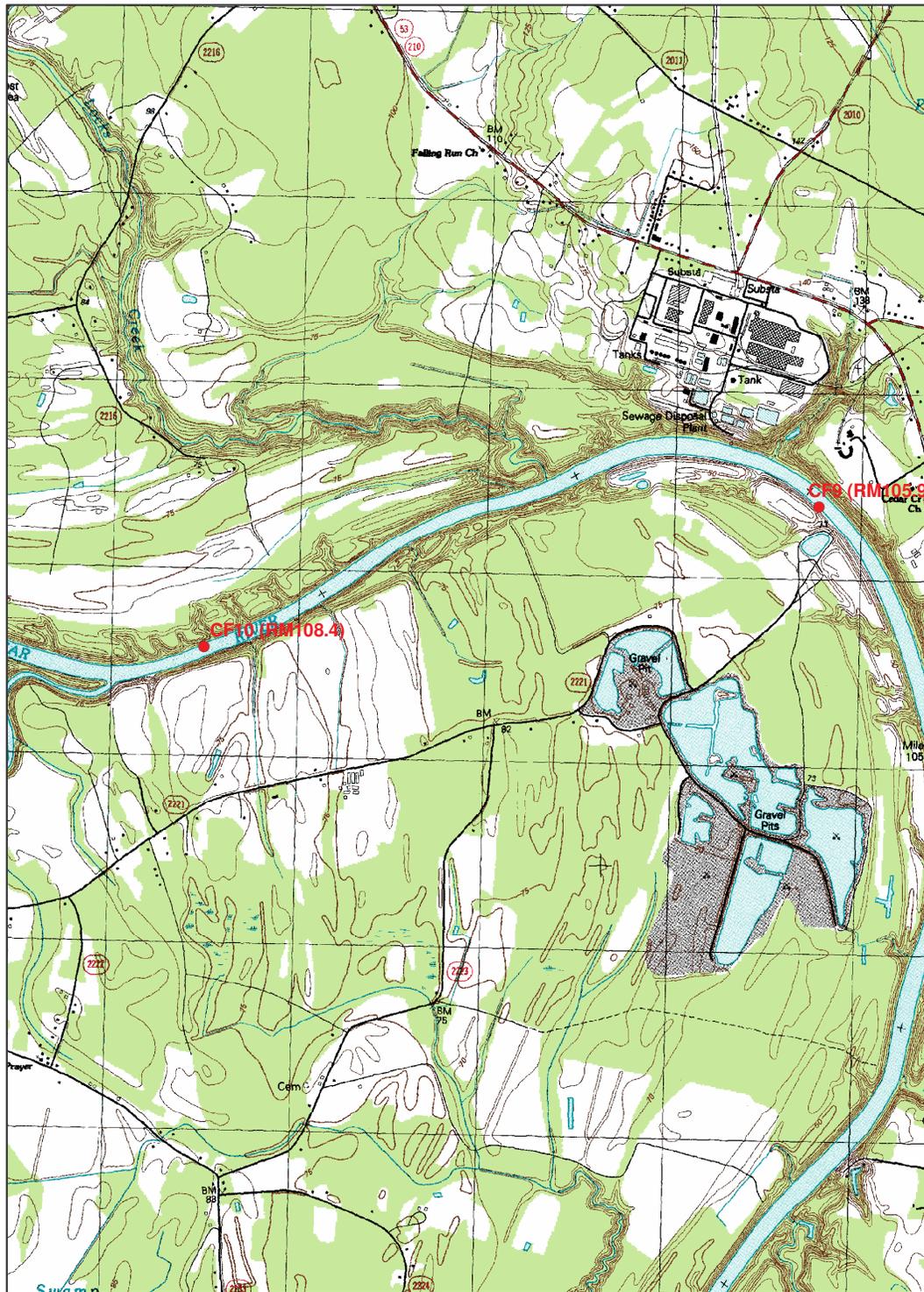


Figure 2. Cape Fear River sediment sampling site downstream of Fayetteville (furthest upstream sample in this assessment).



Tom Augspurger, USFWS 02-20-07

Figure 3. Cape Fear River sediment sampling sites between confluence with Rock Fish Creek and Lock and Dam #3.



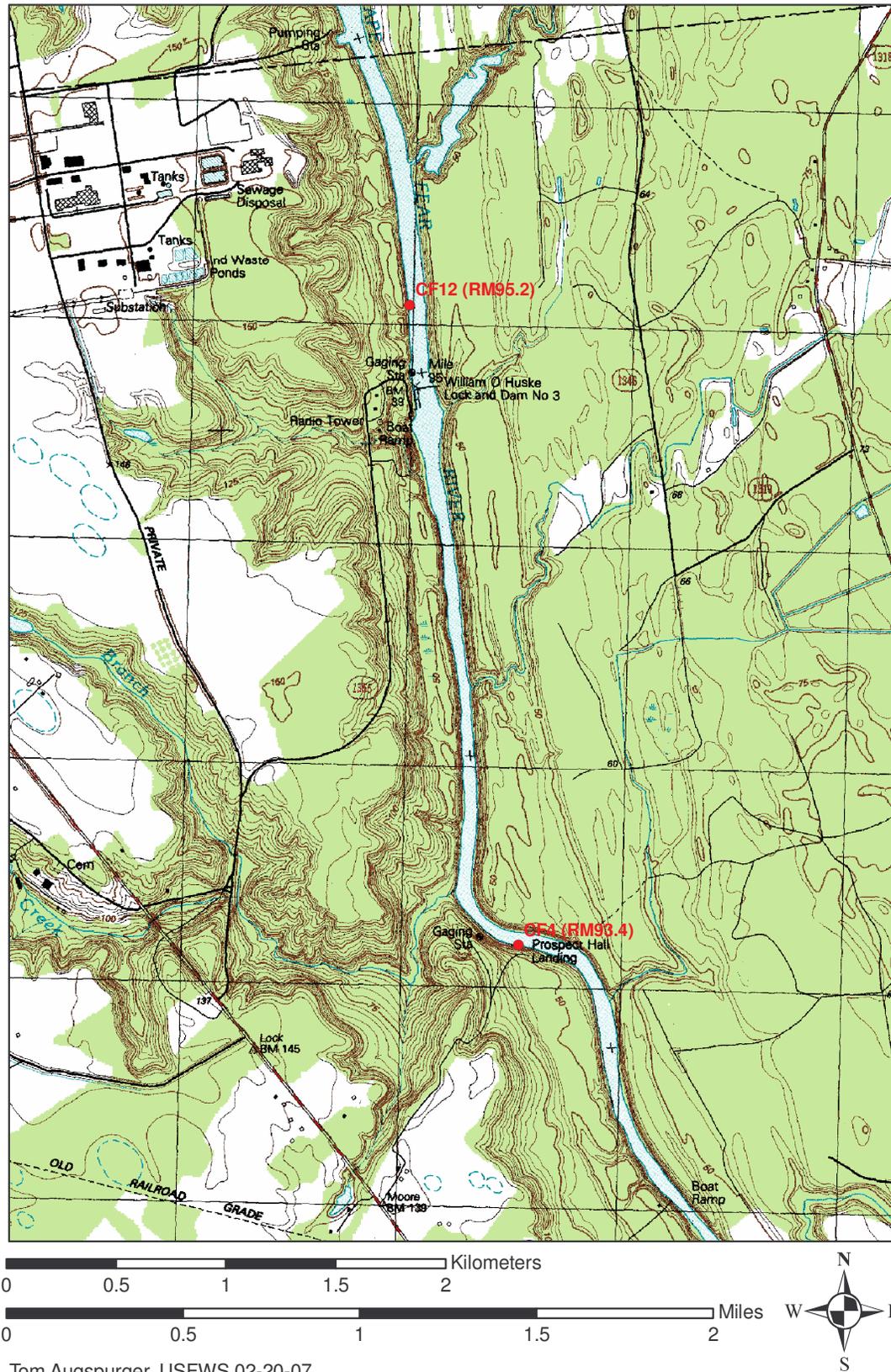
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0 0.5 1 1.5 2 Miles



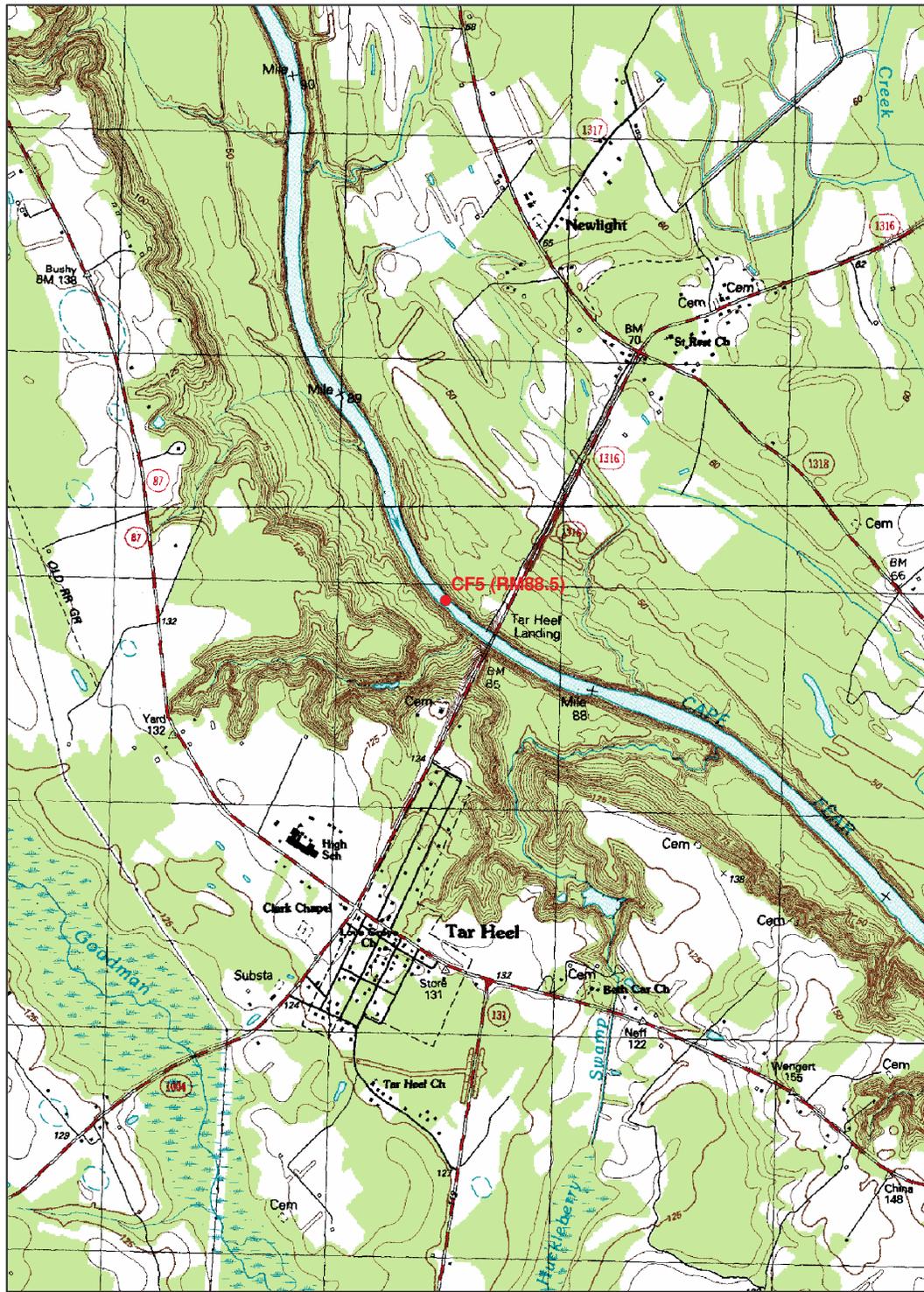
Tom Augspurger, USFWS 02-20-07

Figure 4. Cape Fear River sediment sampling sites upstream and downstream of Lock and Dam #3.



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



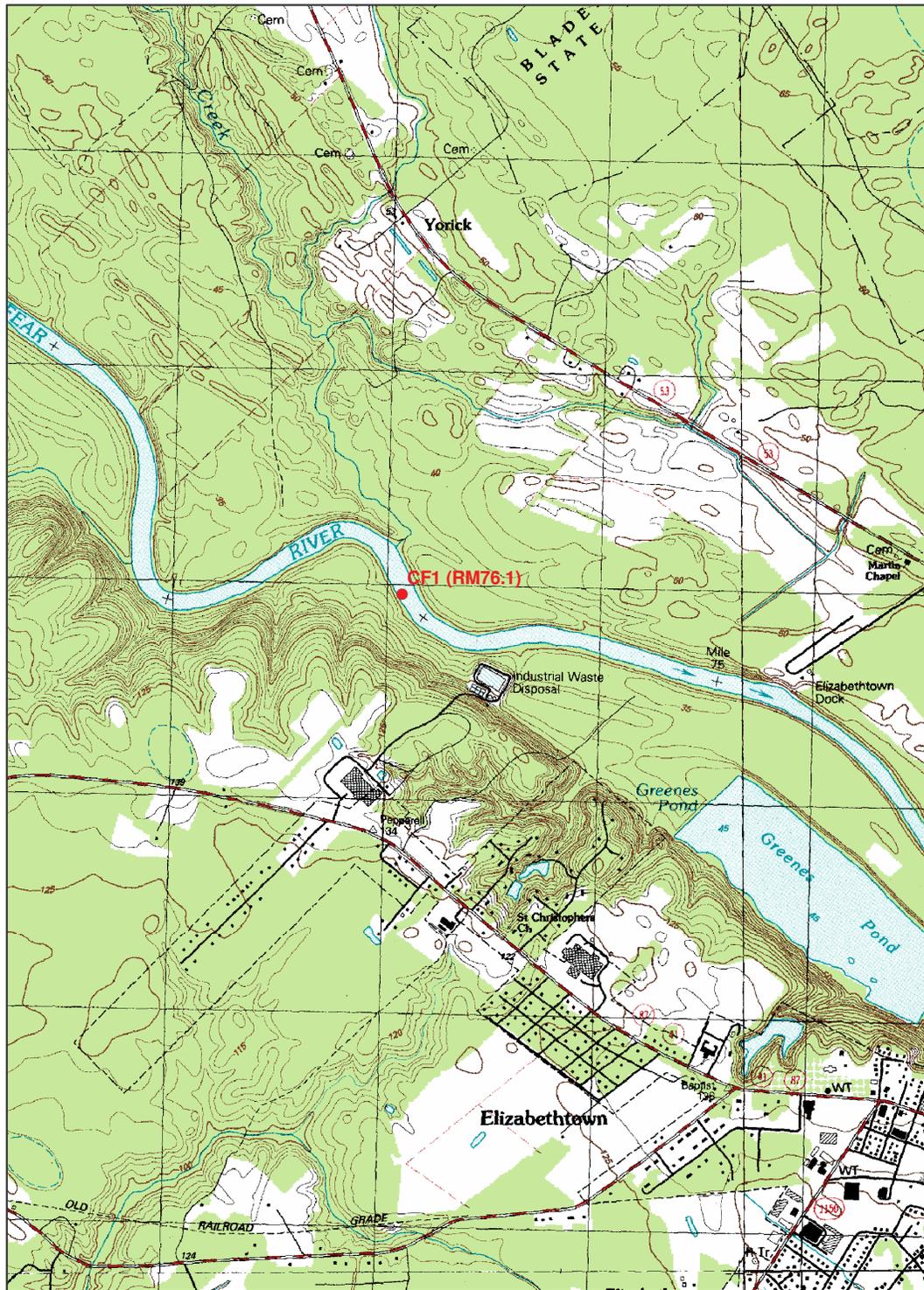
0 0.5 1 1.5 2 Kilometers

0 0.5 1 1.5 2 Miles



Tom Augspurger, USFWS 02-20-07

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



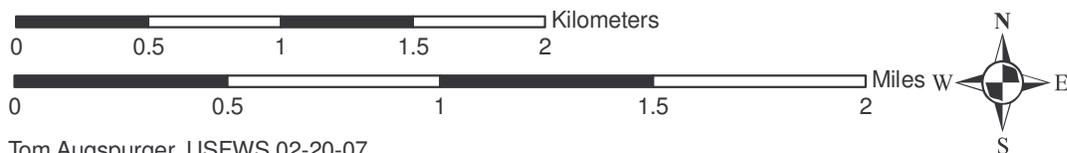
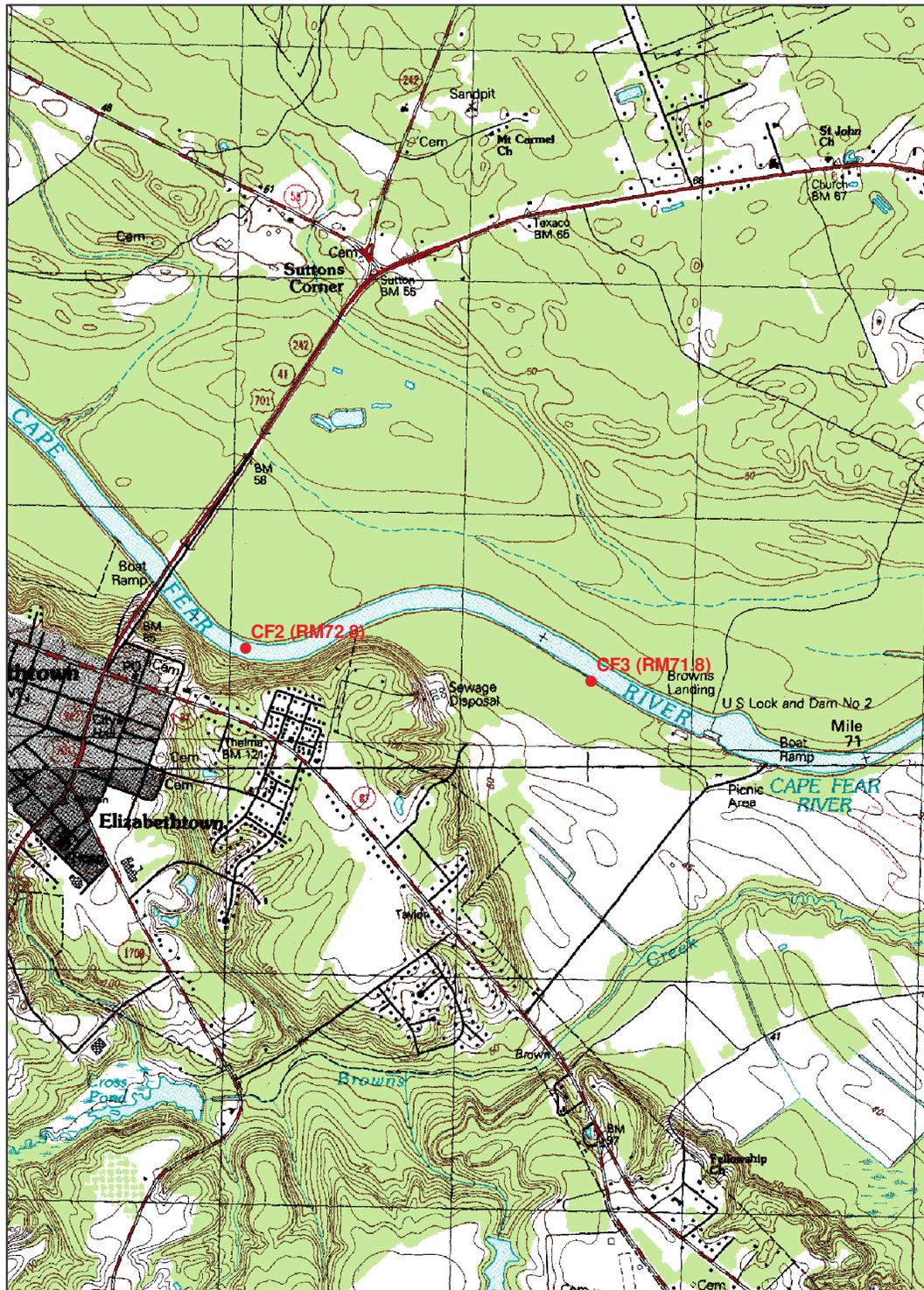
0 0.5 1 1.5 2 Kilometers

0 0.5 1 1.5 2 Miles

Tom Augspurger, USFWS 02-20-07

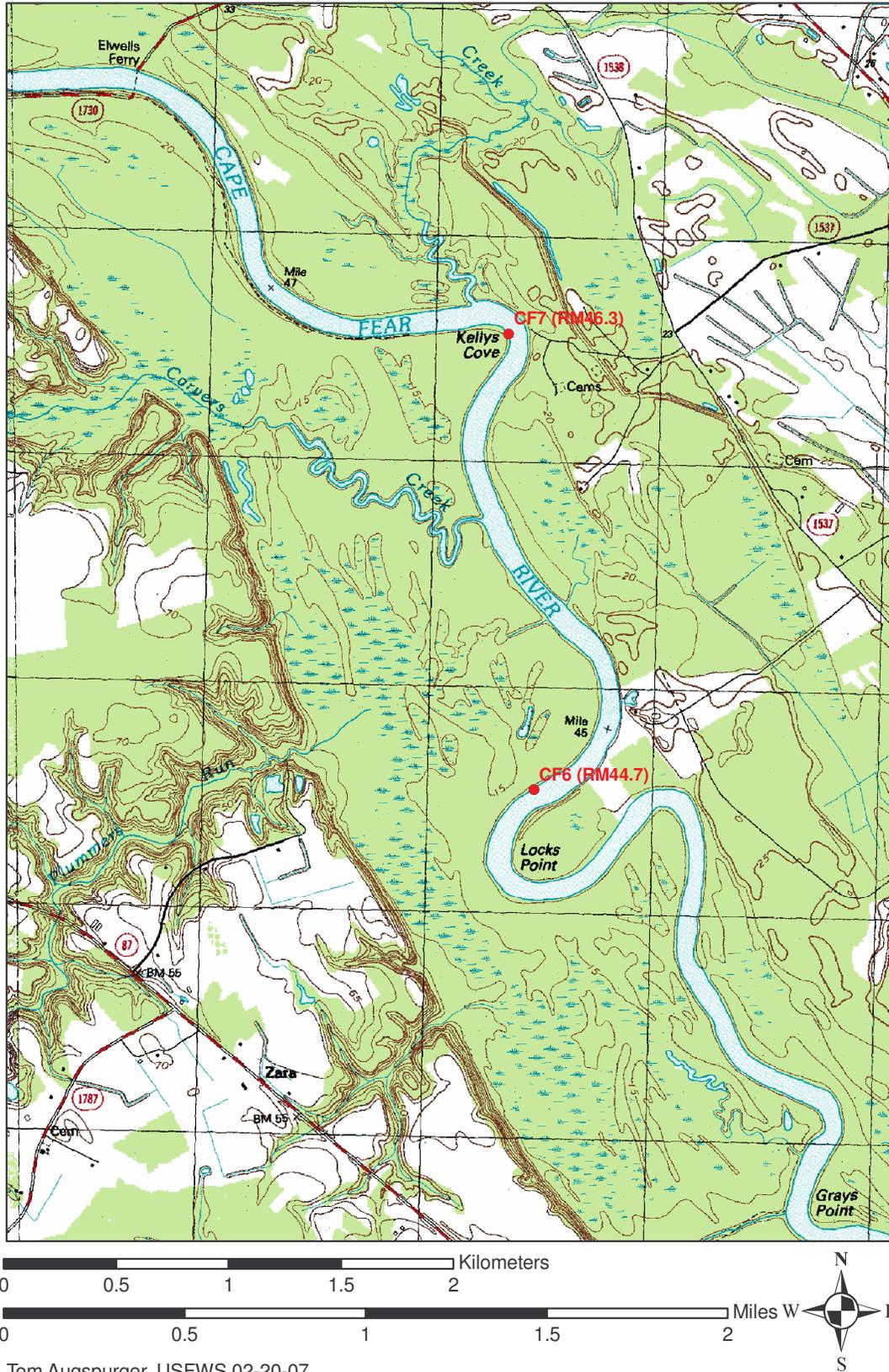


Figure 7. Cape Fear River sediment sampling sites downstream of Elizabethtown and upstream of Lock and Dam #2.



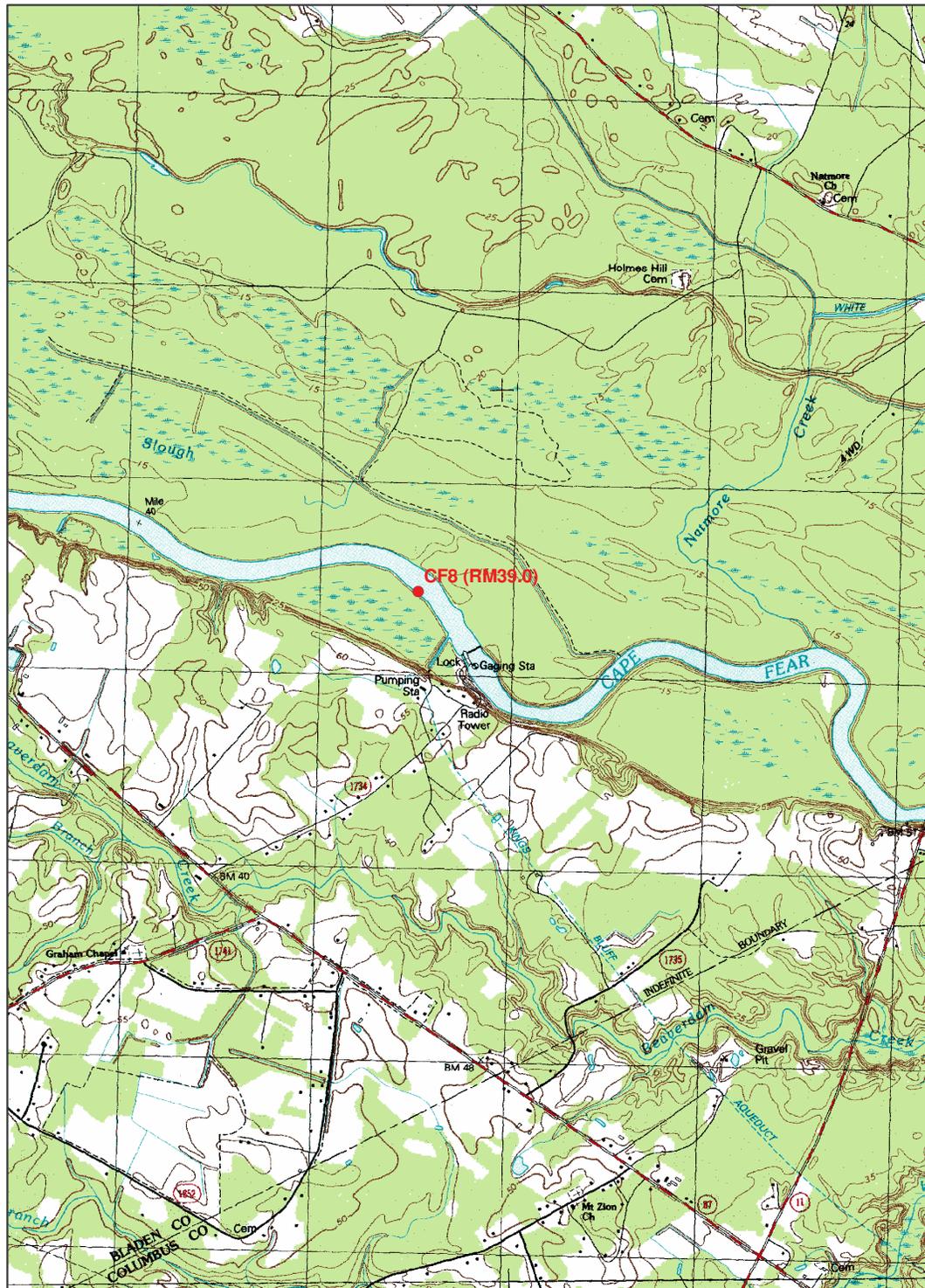
Tom Augspurger, USFWS 02-20-07

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



Tom Augspurger, USFWS 02-20-07

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



0 0.5 1 1.5 2 Kilometers

0 0.5 1 1.5 2 Miles



Tom Augspurger, USFWS 02-20-07

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

306

307 *Sediment toxicity tests*

308

309 Nine samples were sent to the U.S. Geological Survey Columbia Environmental Research Center
310 (CERC), Columbia, MO by overnight delivery on November 7, 2006. Toxicity tests included
311 28-d whole-sediment exposures of *Hyallorella azteca* (freshwater amphipod) evaluating effects on
312 growth and survival, 10-d whole-sediment exposures of *Chironomus dilutus* (freshwater midge)
313 evaluating effects on growth and survival (USEPA 2000a, ASTM 2007), and 2-d sediment
314 elutriate (water-extractable fraction of the sediment) exposures of *Ceriodaphnia dubia*
315 (freshwater cladoceran) evaluating effects on survival (USEPA 1993a). This battery of toxicity
316 tests was selected to help evaluate the toxicity of sediments, both in-place and upon re-
317 suspension. Toxicity tests were performed on nine of the twelve samples based on prioritizing
318 areas of interest from the results of the Tier 1 assessment (USFWS 2006).

319

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

328

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

333

334 Cladocerans were cultured under static conditions in 30-ml disposable clear plastic cups each
335 containing 15 ml of well water and one adult at a temperature of 25°C and a light intensity of
336 about 200 lux (USEPA 1993a). Cladocerans in each cup were fed 0.1 ml/d Yeast-Cerophyll-trout
337 chow (YCT; 1.7 to 1.9 g/L) and 0.1 ml/d unicellular green algae (3×10^7 cells of *Selenastrum*
338 *capricornutum*). Neonates (<24 hours) to start the tests were obtained from adults that produce
339 eight or more young in their third brood or subsequent broods.

340

341 Whole-sediment toxicity tests - Whole-sediment toxicity tests with *H. azteca* were conducted for
342 28 d and with *C. dilutus* were conducted for 10 d in accordance with methods described in
343 ASTM (2007) and USEPA (2000a) starting within 3 weeks of sediment collection. Endpoints
344 measured in the amphipod exposures included survival and length on Day 28. Endpoints
345 measured in the midge exposures included survival and growth (ash-free dry weight) on Day 10.
346 Test sediments were homogenized in a stainless-steel bowl using a plastic spoon and added to
347 exposure beakers 1 d before test organisms were added (Day -1). Sub-samples of sediment were
348 then collected for physical characterizations and for isolating pore water. Amphipods and midge
349 were exposed to 100 ml of sediment with 175 ml of overlying water in 300-ml beakers with four
350 replicates/treatment. The photoperiod was 16 h light: 8 h dark at a light intensity of about 200
351 lux at the surface of the exposure beakers and the temperature was 23°C. Each beaker received
352 2-volume additions/d of overlying water starting on Day -1 (Ingersoll et al. 2002). Overlying
353 water was prepared by diluting well water with deionized water to a hardness of 100 mg/L as
354 CaCO₃, alkalinity of 85 mg/L as CaCO₃, and pH about 7.8. The water delivery system cycled
355 every 4 h with each cycle delivering 125 ml of water to each beaker. Tests were started on Day 0
356 by placing 10 amphipods or 10 midge into each beaker using an eyedropper. Amphipods in each
357 beaker were fed 1.0 ml of YCT (1.7 to 1.9 g/L) in a water suspension daily (USEPA 2000a,
358 ASTM 2007). Midge in each beaker were fed 1.5 ml of Zeigler prime tropical flake fish food
359 (red) (Zeigler Bros, Inc., Gardens, PA) (6.0 mg of dry solids) in a water suspension daily
360 (USEPA 2000a, ASTM 2007). Beakers were observed daily for the presence of animals, signs
361 of animal activity (i.e., burrowing), and to monitor test conditions (mainly water clarity).

362
363 Midge were isolated from each beaker on Day 10 of the exposure and amphipods were isolated
364 from each beaker on Day 28 of the exposure by pouring off most of the overlying water, gently
365 swirling the remaining overlying water and upper layer of sediment, and washing the sediment
366 through a No. 50 (300-µm opening) U.S. Standard stainless-steel sieve. The materials that were
367 retained on the sieve were washed into a glass pan and the surviving midge and amphipods were
368 removed. This process was repeated with the remainder of the sediment in a beaker if all 10 of
369 the amphipods or midge were not recovered from the upper layer of the sediment. Amphipods
370 from each sediment were counted and preserved in 8% sugar formalin for subsequent length
371 measurements (Ingersoll et al. 2002). Length of amphipods was measured along the dorsal
372 surface from the base of the first antenna to the tip of the third uropod along the curve of the
373 dorsal surface. Amphipod length measurements were made using an EPIX imaging system
374 (PIXCI® SV4 imaging board and XCAP software; EPIX Inc., Buffalo Grove, IL) connected to a
375 computer and a microscope (Ingersoll et al. 2002). Midge from each sediment were counted and
376 dried at 100°C in a drying oven for subsequent ash-free dry weight measurements (ASTM 2007).
377 Ash-free dry weight was obtained by recording the weights after drying, transferring to an ashing
378 oven and ashing at 500°C. Ash-free dry weights were determined by subtracting the ashed
379 weight from the dry weight.

380
381 About 50 ml of pore water was isolated from sediment samples by centrifugation at 5200 rpm
382 (7000 x G) for 15 min at 4°C (Kemble et al. 1994). Immediately after pore water was isolated,
383 dissolved oxygen, pH, alkalinity, temperature, conductivity, total ammonia, and hardness were
384 measured using methods outlined in Kemble et al. (1994). Mean characteristics of pore-water
385 water quality (ranges in parentheses) were: pH 6.95 (4.5 to 7.4), alkalinity 491 (248 to 824 mg/L
386 as CaCO₃), hardness 298 (144 to 498 mg/L as CaCO₃), dissolved oxygen 1.8 (1.0 to 5.7 mg/L),

387 conductivity 1016 (524 to 1597 $\mu\text{mho/cm}$), total ammonia 13.5 (2.1 to 33.7 mg N/L), and
388 unionized ammonia 0.057 (0.001 to 0.078 mg N/L; Table 5).

389
390 In the amphipod test, conductivity, pH, alkalinity, hardness, dissolved oxygen, and total
391 ammonia were measured in overlying test water on Day 0 (the day amphipods were added to the
392 exposure beakers) and Day 28 of the exposure. Conductivity and dissolved oxygen, in overlying
393 water were also measured weekly. Temperature in the water baths containing the exposure
394 beakers was recorded daily. Overlying water pH, alkalinity, total hardness, conductivity and
395 total ammonia measurements were similar among all treatments. Mean characteristics of
396 overlying water quality (ranges in parentheses) are as follows: pH 8.0 (7.9 to 8.0), alkalinity 88
397 (81 to 94 mg/L as CaCO_3), hardness 93 (90 to 96 mg/L as CaCO_3), dissolved oxygen 7.1 (6.7 to
398 7.5 mg/L), conductivity 257 (252 to 263 $\mu\text{mho/cm}$), total ammonia 0.39 (0.19 to 0.70 mg N/L),
399 and unionized ammonia 0.010 (0.004 to 0.010 mg N/L; Table 7). Dissolved oxygen in overlying
400 water was at or above the acceptable level of 2.5 mg/L in all treatments throughout the study
401 (USEPA 2000a, ASTM 2007).

402
403 In the midge test, conductivity, pH, alkalinity, hardness, dissolved oxygen, and total ammonia
404 were measured in overlying test water on Day 0 (the day midge were added to the exposure
405 beakers) and Day 10 of the exposure. Conductivity and dissolved oxygen in overlying water
406 were also measured weekly. Temperature in the water baths holding the exposure beakers was
407 monitored daily. Overlying water pH, alkalinity, total hardness, conductivity and total ammonia
408 measurements were similar among all treatments. Mean characteristics of overlying water
409 quality (ranges in parentheses) are as follows: pH 8.0 (7.6 to 8.1), alkalinity 87 (83 to 96 mg/L as
410 CaCO_3), hardness 92 (85 to 100 mg/L as CaCO_3), dissolved oxygen 7.4 (7.1 to 7.6 mg/L),
411 conductivity 255 (249 to 263 $\mu\text{mho/cm}$), total ammonia 0.38 (0.20 to 0.64 mg N/L), and
412 unionized ammonia 0.010 (0.011 to 0.017 mg N/L; Table 8). Dissolved oxygen in overlying
413 water was above acceptable levels of 2.5 mg/L in all treatments throughout the study (USEPA
414 2000a, ASTM 2007).

415
416 Elutriate toxicity tests - Toxicity tests with *C. dubia* were conducted for 2 d in accordance with
417 methods outlined in USEPA (1993a) starting within 5 weeks of sediment collection. Elutriate
418 samples were prepared by mixing one part sediment with four parts water (prepared by diluting
419 well water, the characteristics of which are described at page 13 above under Test organism
420 culture, with deionized water to a hardness of 100 mg/L as CaCO_3 , alkalinity of 85 mg/L as
421 CaCO_3 , and pH about 7.8). Elutriate samples were prepared following procedures outlined in
422 USEPA (1993a). About 50 ml of sediment was placed into two 250-ml high-density
423 polypropylene centrifuge tubes and topped off with 200 ml of water. The tubes were sealed and
424 tumbled on a rolling mill for 30 min. The tubes were then be centrifuged for 15 min at 5200 rpm
425 (7000 x G). The water was then decanted through a US Standard #50 stainless steel sieve (300-
426 μm opening) into a 500-ml beaker. A sub-sample of the elutriate samples was collected for
427 water quality characterization, with the remaining sample used in toxicity tests.

428
429 The cladoceran test was started on Day 0 with <24-h old *C. dubia*. A total of 10 cladocerans
430 were exposed to each elutriate sample and were exposed individually in 30-ml disposable clear
431 plastic cups containing 15 ml of 100% elutriate test solution. Trays holding the plastic cups were
432 covered with plastic sheets to prevent evaporation. Cladocerans were maintained in a water bath

433 at 25±1° C on a 16 h light: 8 h darkness photoperiod at a light intensity of about 200 lux.
434 Cladocerans were not fed during the exposures. On Day 2, survival of the cladocerans was
435 determined by pouring all the test water into a petri dish and using a light table to facilitate
436 counting the neonates. Four of the elutriate samples that were found to be toxic to *C. dubia* (CF1,
437 CF6, CF10, and CF11) were re-tested in a dilution series prepared 21 d after the start of the
438 initial toxicity tests with 100% elutriate sample. The four samples were prepared by the same
439 procedures outlined above and a 50 % dilution series (elutriate sample was prepared with the 100
440 mg/L hardness water, testing 100, 50, 25, 12.5, 6.25% elutriate) and a dilution water control.

441
442 Conductivity, pH, alkalinity, hardness, dissolved oxygen, and total ammonia were measured in
443 elutriate test water on Day 0 (the day cladocerans were added to the exposure beakers).
444 Temperature in the water baths containing the exposure beakers was recorded daily. Elutriate
445 water pH, alkalinity, total hardness, conductivity and total ammonia measurements were similar
446 among all treatments. Mean characteristics of elutriate water quality (ranges in parentheses) are
447 as follows: pH 7.4 (6.2 to 7.8), alkalinity 100 (32 to 150 mg/L as CaCO₃), hardness 100 (64 to
448 154 mg/L as CaCO₃), dissolved oxygen 5 (4 to 7 mg/L), conductivity 359 (259 to 491
449 µmho/cm), total ammonia 3.63 (0.8 to 6.7 mg N/L), and unionized ammonia 0.049 (0.001 to
450 0.088 mg N/L). Dissolved oxygen in overlying water was above 2.5 mg/L in all treatments
451 throughout the study.

452
453 *Sediment chemical and physical analyses*

454
455 Elemental contaminants and PAHs were targeted for analyses in all samples; in addition to being
456 detected in the assessment area above threshold effect levels by others (USFWS 2006), these
457 classes of compounds include many common pollutants. Additionally, these compounds have
458 consensus-based freshwater effects sediment quality guidelines (MacDonald et al. 2000, USEPA
459 2000b) with which to evaluate the results.

460
461 Sediment samples were delivered to Environmental Conservation Laboratories, Inc. (ENCO) in
462 Cary, NC on November 9, 2006. ENCO has the North Carolina Laboratory Certification for the
463 requested analyses. Sediment samples were analyzed for Al, As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Ni,
464 Se, Ag, and Zn by USEPA method 6010B (inductively coupled plasma-atomic emission
465 spectrometry, or ICP-AES). Analyses of mercury in sediment samples were by USEPA method
466 7471A (cold-vapor atomic absorption spectrophotometry). ENCO analyzed sediments for PAHs,
467 including 1-methylnaphthalene, 2-methylnaphthalene, acenaphthene, acenaphthylene,
468 anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene,
469 benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorine, indeno(1,2,3-
470 cd)pyrene, naphthalene, phenanthrene and pyrene, by USEPA method 8270C (gas
471 chromatography / mass spectrometry, or GC/MS).

472
473 Pore-water and elutriate samples prepared by CERC (described above) were also analyzed for
474 elemental contaminants by ENCO. Elements in the analyses included Al, As, Cd, Cr, Cu, Fe, Pb,
475 Mn, Ni, Se and Zn by USEPA method 200.8 (Trace Elements by ICP/Mass Spectrometry). This
476 method was also used to measure Mg and Ca which provided the measures of hardness for these
477 samples (by the calculation method in Standard Methods SM 2340B).

478 Physical characterization of whole sediments included percentage water, particle size and total
479 organic carbon (TOC). Particle size analysis was conducted at CERC by sieve series following
480 methods described in Foth et al. (1982), Gee and Bauder (1986), and Kemble et al. (1994). The
481 TOC analyses were by the Walkley Black method (Schumacher 2002) and were conducted by
482 Severn Trent Laboratories of Tampa, FL (under subcontract to ENCO).
483

484 All analyses were accompanied by batch-specific quality control / quality assurance samples
485 (blanks, spikes, and duplicates). Review of quality assurance data (provided in their entirety in
486 the appendices) indicate acceptable precision and accuracy for all analyses.
487

488 Threshold effects concentrations (TECs) and probable effects concentrations (PECs) were used
489 to assess the significance of the sediment chemistry results. The TECs are concentrations of
490 contaminants in whole sediment below which adverse effects to sensitive aquatic organisms are
491 not expected to occur, and the PECs are effect-based sediment quality guidelines established as
492 concentrations of contaminants in whole sediment above which adverse effects are expected to
493 frequently occur in field-collected sediments (MacDonald et al. 2000). Mean quotients based on
494 PECs were calculated to provide an overall measure of chemical contamination and to support an
495 evaluation of the combined effects of multiple contaminants in sediments (MacDonald et al.
496 2000; USEPA 2000b). A PEC quotient (PEC-Q) was calculated for each chemical in each
497 sediment sample by dividing the dry-weight concentration of a chemical by the PEC for that
498 chemical. To equally weight the contribution of metals and PAHs in the evaluation of sediment
499 chemistry and toxicity (assuming these diverse groups of chemicals exert some form of
500 collective toxic action), an average PEC-Q for metals was calculated based on concentrations of
501 each metal. A mean overall quotient was then calculated for each sample by summing the
502 average quotient for metals and the quotient for total PAHs, and dividing this sum by two ($n = 2$
503 quotients/sample). Use of this approach for calculating the quotients was selected to avoid over-
504 weighting the influence of an individual chemical (e.g., a single metal) on the combined mean
505 quotient (USEPA 2000b).
506

507 Relationships between toxicity and concentrations of PAHs in sediment samples were calculated
508 using equilibrium partitioning sediment guideline toxic units (ESGTU; USEPA 2003). The
509 ESGTUs are a way of summarizing many PAHs compounds into one representative number. An
510 ESGTU is basically the organic carbon normalized concentration of a chemical divided by a
511 toxicity threshold (this is a toxic unit for the specific chemical). Then the ESGTU for specific
512 chemicals are summed for each sample yielding Σ ESGTUs.
513

514 *Statistical analyses*

515

516 Statistical analyses for the amphipod exposures were conducted using one-way analysis of
517 variance (ANOVA) at $p = 0.05$ for all endpoints except length which was analyzed using a one-
518 way nested ANOVA at $p = 0.05$ (amphipods nested within a beaker; Snedecor and Cochran
519 1982). Before statistical analyses were performed, all data were tested for normality. Variance
520 among treatment means for both endpoints was heterogeneous, therefore, a rank analysis of
521 variance was performed and mean differences were determined using a t-test on ranked means
522 (at $p = 0.05$). A sample was designated as toxic when mean survival or growth was significantly
523 reduced relative to the control sediment. Statistical analyses on toxicity tests were performed

524 with Statistical Analysis System programs (SAS 2001). Pairwise correlations among the *C.*
 525 *dubia* survival results and all sediment and elutriate chemical analyses were calculated with JMP
 526 5.1.2 software (SAS Institute, Inc., Cary, NC).

527
 528 Contaminant concentrations were analyzed for goodness of fit to a normal distribution by the
 529 Shapiro-Wilk test (JMP 5.1.2, SAS Institute, Inc.). All elemental contaminants concentrations
 530 were normally-distributed. Most of the sediment PAH concentrations were not normally-
 531 distributed and these were log-transformed prior to additional analyses. No statistical analyses
 532 were conducted for compounds detected in less than 40% of the samples, these included (in
 533 sediment samples) silver, 1-methylnaphthalene, 2-methylnaphthalene, acenaphthene,
 534 acenaphthylene, anthracene, dibenzo(a,h)anthracene, fluorene and naphthalene. For analytes
 535 present in at least 40% of samples at concentrations greater than detection limits, a value of the
 536 detection limit was substituted for non-detects in statistical comparisons. For analytes reported
 537 as estimated values (near the detection limits), the estimated concentration was used.

538

539 **Results and Discussion**

540

541 *Toxicity tests*

542

543 Control survival of *H. azteca* was 98% and met the test acceptability requirement of 80% survival
 544 recommended in USEPA (2000a) and ASTM (2007). Control survival of *C. dilutus* was 65%
 545 and did not meet the test acceptability requirement of 70% survival recommended in USEPA
 546 (2000a) and ASTM (2007). USEPA (2000a) and ASTM (2007) state that a study in which
 547 organisms in the negative control do not meet performance criteria must be considered
 548 questionable because it suggests that adverse factors affected the response of test organisms.
 549 Some studies have reported cases where performance in the negative control is poor, but
 550 performance criteria are met in reference sediment included in the study design. In these cases, it
 551 might be reasonable to infer that other samples that show good performance are probably
 552 not toxic; however, any samples showing poor performance should not be judged to have shown
 553 toxicity, since it is unknown whether the adverse factors that caused poor control
 554 performance might have also caused poor performance in the test treatments (USEPA 2000a,
 555 ASTM 2007). In the current study, survival of midge in all of the test sediments was above 75%
 556 and was frequently above 90% suggesting that the test sediments were not toxic to midge. No
 557 reason for the poor performance of midge in the control sediment could be identified other than
 558 the control sample had been held in the laboratory for over three years for use in routine testing.

559

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

563

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

570 Table 2. Response of *Hyalella azteca* in 28-d whole-sediment exposures, *Chironomus dilutus* in
 571 10-d whole-sediment exposures, and *Ceriodaphnia dubia* in 2-d elutriate exposures prepared
 572 from sediment samples collected from the Cape Fear River and to a control sediment (WB).
 573 Means (standard error of the means in parentheses) with an asterisk (CF 11, 10, 1 and 6) within a
 574 column are significantly different than the control ($p < 0.05$).
 575

River Mile	Sample ID	Amphipod Survival (%)	Amphipod Growth (mm)	Midge Survival (%)	Midge Growth (mg)	Cladoceran Survival (%)
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

576
 577
 578 sources. As discussed later in this report, elutriate sample toxicity appears to be related to
 579 sediment manganese concentrations rather than any particular point source. Tables 3 through 8
 580 summarize the chemistry of elutriates and overlying water in the whole-sediment toxicity tests.
 581

582 *Whole-sediment analytical chemistry*

583
 584 The reports from ENCO and Severn Trent are reprinted in Appendix A and summarized here.
 585 Total organic carbon in the samples averaged 3.1% and the average percent fines (sum of silt and
 586 clay fraction) was 55% indicating depositional areas were located for the assessment (Table 9).
 587

588 There are no North Carolina or federal sediment quality criteria, but Tables 10 and 11 compare
 589 the elemental contaminants and PAHs in whole sediment to the TECs and PECs of McDonald et
 590 al. (2000). Florida and Wisconsin recommend TECs and PECs for use as guidance in their
 591 programs, including evaluation of dredged material and risk assessment of contaminated sites
 592 (MacDonald et al. 2003). While no regulatory implications are inferred in our use of the TECs
 593 and PECs, sediment quality guidelines like these have been found to offer good utility in site
 594 assessment by various government and non-government organizations (Wenning et al. 2005).
 595
 596

597 Table 3. Water quality characteristics in 2-d elutriate exposures with *Ceriodaphnia dubia* prepared from Cape Fear River sediment
 598 samples and a control sediment (WB).
 599

River Mile	Sample ID	Dissolved Oxygen (mg/L)		Conductivity (umho @25°C)		pH	Total Ammonia (mg/L)	Hardness (mg/L as CaCO ₃)	Alkalinity (mg/L as CaCO ₃)	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	

600

601 Table 4. Elemental contaminant concentrations ($\mu\text{g/L}$) of 2-d elutriate exposures with *Ceriodaphnia dubia* prepared from Cape Fear
 602 River sediment samples and a control sediment (WB).
 603

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

604
 605 B = aluminum was detected in the blank at 47.7 $\mu\text{g/L}$. Because sample results were typically two orders of magnitude higher than
 606 this concentration, the presence of aluminum in the blank should not affect interpretation of results
 607 J = less than reporting limit (estimated value)
 608 U = not detected

609 Table 5. Water quality characteristics of pore water isolated from Cape Fear River whole-sediment samples and a
 610 control sediment (WB). NA = not analyzed
 611

River Mile	Sample ID	Dissolved Oxygen (mg/L)		Conductivity (umho@25°C)	pH	Total Ammonia (mg/L)	Alkalinity (mg/L as CaCO ₃)	Hardness (mg/L as CaCO ₃)
		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

612

613 Table 6. Elemental contaminant concentrations (µg/L) of pore water isolated from Cape Fear River whole-sediment samples and a
 614 control sediment (WB).
 615

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

616
 617 B = aluminum was detected in the blank at 47.7 µg/L. Because sample results were typically two orders of magnitude higher than
 618 this concentration, the presence of aluminum in the blank should not affect interpretation of results
 619 J = less than reporting limit (estimated value)
 620 U = not detected

621 Table 7. Mean water quality characteristics of overlying water in whole-sediment toxicity tests with *Hyalella azteca*
 622 and Cape Fear River sediments and a control sediment (WB). NA = not applicable.
 623

River Mile	Sample ID	Dissolved oxygen (mg/L)	Conductivity (µmho@25°C)	Hardness (mg/L as CaCO ₃)	Alkalinity (mg/L as CaCO ₃)	pH	Total ammonia (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

624

625 Table 8. Mean water quality characteristics of overlying water in whole-sediment toxicity tests with *Chironomus dilutus*
 626 and Cape Fear River sediments and a control sediment (WB).
 627

River Mile	Sample ID	Dissolved oxygen (mg/L)	Conductivity ($\mu\text{mho}@25^\circ\text{C}$)	Hardness (mg/L as CaCO_3)	Alkalinity (mg/L as CaCO_3)	pH	Total ammonia (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

628

629 Table 9. Total organic carbon, grain size and moisture measured in whole-sediment samples collected
 630 from the Cape Fear River.
 631

River Mile	Sample ID	Total Organic Carbon (%)	Sand (%)	Clay (%)	Silt (%)	Moisture (%)
		STL ¹	CERC ²	CERC	CERC	ENCO ³
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

632 ¹STL = Severn Trent Laboratories, Inc., Tampa, FL
 633 ²CERC = USGS Columbia Environmental Research Center, Columbia, MO
 634 ³ENCO = Environmental Conservation Laboratories, Inc., Cary, NC and Jacksonville, FL

635 Table 10. Elemental contaminants in whole-sediment samples collected from the Cape Fear River. All data are mg/kg dry weight
 636 (parts per million). For each element, results are compared to threshold-effects concentration (TEC) guidelines of MacDonald et al.
 637 (2000) -- values below which adverse effects to sensitive aquatic organisms are not expected to occur, and probable effects
 638 concentrations (PECs) -- values above which adverse effects to sediment dwelling organisms may be expected. No samples exceeded
 639 TECs. Silver was below detection in all samples.
 640

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

641
 642 D = data reported from a dilution
 643 J = less than reporting limit (estimated value)

644 Table 11. Polycyclic aromatic hydrocarbons measured in whole-sediment samples collected from the Cape Fear River. All data are
 645 µg/kg dry weight (parts per billion). For each compound, results are compared to threshold-effects concentration (TEC) guidelines of
 646 MacDonald et al. (2000) -- values below which adverse effects to sensitive aquatic organisms are not expected to occur, and probable
 647 effects concentrations (PECs) -- values above which adverse effects to sediment dwelling organisms may be expected. Only CF 5 and
 648 CF 11 exceeded TECs (exceedences designated with and asterisk), and no samples exceeded PECs. 1-methylnaphthalene, 2-
 649 methylnaphthalene, acenaphthene, acenaphthylene, anthracene, fluorene and naphthalene were detected in one or no samples. J = less
 650 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

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

665
 666
 667 Table 12. Probable effect concentration quotients (PEC-Qs) and polycyclic aromatic
 668 hydrocarbon equilibrium partitioning sediment guideline toxic units (Σ ESBTUs) for whole-
 669 sediment samples collected from the Cape Fear River. Values are unitless with levels of concern
 670 typically not indicated until exceeding 1 on these scales.
 671

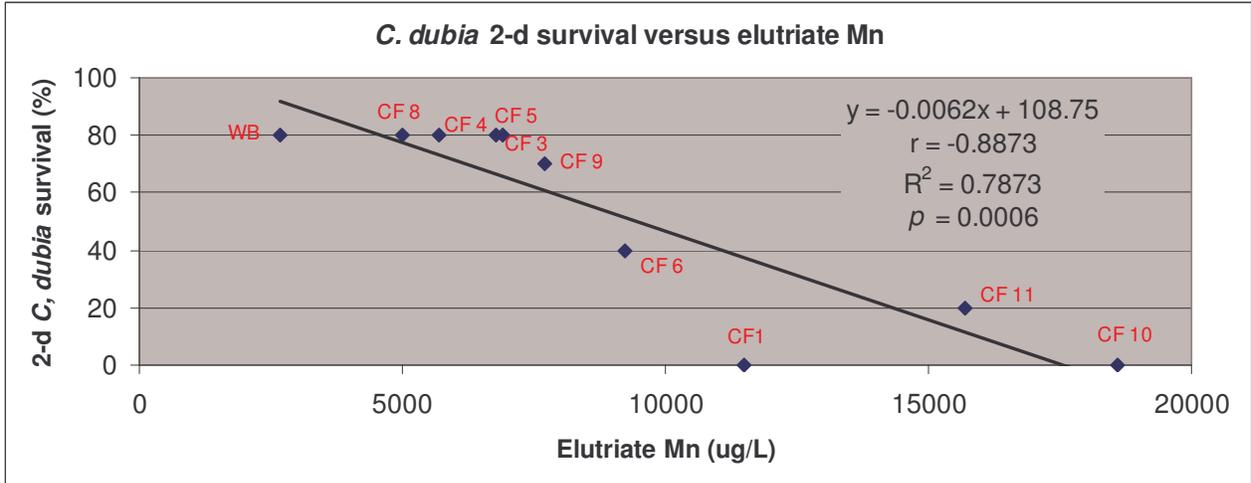
River Mile	Sample ID	Mean PEC-Q	Mean Metal PEC-Q	Total PAH PEC-Q	Σ ESGTU for PAHs
112.9	CF 11	0.089	0.105	0.072	0.160
108.4	CF 10	0.084	0.147	0.020	0.066
105.9	CF 9	0.045	0.074	0.014	0.078
95.2	CF 12	0.069	0.120	0.017	0.058
93.4	CF 4	0.063	0.111	0.013	0.095
88.5	CF 5	0.067	0.085	0.049	0.252
76.1	CF 1	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

672
 673
 674 Although the elutriates and pore-water samples are not surface waters, we compared elemental
 675 contaminant concentrations in these media to State water quality standards and action levels
 676 (Table 13). This is not a regulatory application of the standards; it is rather a comparison of test
 677 results to the standards as toxicological benchmarks, or estimates of safe water column
 678 concentrations. Copper, lead, and zinc exceed State standards, an indication that aggressive re-
 679 suspension of sediments like those tested could temporarily impair surface water quality.

680 Table 13. Elutriate and pore water exceedences of North Carolina water quality standards or
 681 action levels for elemental contaminants (NCDENR 2004). Analytical data for elutriates and
 682 pore water are in Tables 4 and 6.
 683

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)

684
 685
 686 In 2-d sediment elutriate tests with *C. dubia*, statistically-significant reductions in survival
 687 occurred in four of the nine exposures. Pairwise correlation analyses indicated several sediment
 688 and elutriate water chemistry variables were significantly and negatively correlated with *C.*
 689 *dubia* survival in the elutriates (Table 14). Of the correlated variables, hardness, alkalinity, and
 690 conductivity are unlikely to impair cladocerans at the concentrations we encountered based on
 691 their tolerance of much higher salt concentrations (Mount et al. 1997). The correlations with
 692 selenium and arsenic also lack strong toxicological support because effects concentrations for
 693 these parameters were generally above concentrations found in the elutriates (USEPA 1996,
 694 2004). Elutriate manganese had among the strongest correlation ($r^2 = 0.79, p = 0.0006$) (Figure
 695 10) and the most plausible biological association with reduced *C. dubia* survival.
 696



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

699 Table 14. Pearson’s correlations for 2-d *C. dubia* survival in sediment elutriates versus whole-
 700 sediment and elutriate physical and chemical constituents (presented in Tables 3, 4, 9-11). All
 701 significant correlations are presented; only select variables that were not significant are included.
 702

Variable	Correlation to <i>C. dubia</i> survival				Threshold	Samples in excess of threshold (%)
	n	R	r ²	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/L ¹	56
Elutriate Se	10	-0.8785	0.7718	0.0008	220 µg/L ²	0
Elutriate As	10	-0.5880	0.3457	0.0738	760 µg/L ³	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/L ⁴	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		

703 ¹ Lasier et al. (2000) report a *C. dubia* LC50 (median lethal concentration) of 14.5 mg/L for manganese at hardness
 704 of 93 mg/L as CaCO₃. This value was multiplied by 0.5 to approximate a lethal effects threshold for the Cape Fear
 705 River sediment elutriates (which ranged in hardness from 70.6 to 154 mg/L as CaCO₃). Evaluation of acute toxicity
 706 data over a large number of tests and species has generally shown that multiplying an LC50 or EC50 by 0.5 provides
 707 an estimate of a concentration near or below the lethality threshold. The technical rationale is found in the *Federal*
 708 *Register* notice soliciting comment on USEPA’s methodology for deriving water quality criteria (43 FR 21506, May
 709 18, 1978) (Stephan et al. 1985).
 710

711 ² USEPA (2004) report a *C. dubia* LC50 of 842 µg/L for selenate and 440 µg/L for selenite. The lower of these
 712 values was multiplied by 0.5 to approximate a lethal effects threshold for the Cape Fear River sediment elutriates.
 713

714 ³ USEPA (1996) report a *C. reticulata* LC50 of 1,511 µg/L for arsenic. This was multiplied by 0.5 to approximate a
 715 lethal effects threshold for the Cape Fear River sediment elutriates.
 716

717 ⁴ USEPA (1999) report a *C. acanthina* LC50 of 25.78 mg total ammonia nitrogen /L at pH=8. This was multiplied
 718 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)
 719 of our *C. dubia* elutriate tests with USEPA’s (1999) equation 11 resulting in a threshold of 26 mg-N/L for the Cape
 720 Fear River sediment elutriates.
 721
 722
 723

724 Stubblefield and Hockett (2000) report a geometric mean LC50 (median lethal concentration)
725 from eight manganese toxicity tests with *C. dubia* of 15.4 mg/L (data normalized to a hardness of
726 50 mg/L as CaCO₃). Similarly, Lasier et al. (2000) report a *C. dubia* LC50 of 14.5 mg/L for
727 manganese at a hardness of 93 mg/L as CaCO₃. The Cape Fear River sediment elutriates ranged
728 in hardness from 70.6 to 154 mg/L as CaCO₃. To approximate a threshold at which lethal effects
729 may begin, we multiplied the LC50 reported by Lasier et al. (2000) by 0.5. This yields an
730 estimated lethal effects threshold of 7.2 mg/L for manganese. Evaluation of acute toxicity data
731 over a large number of tests and species has generally shown that dividing an LC50 or EC50 by
732 2 (i.e., multiplying by 0.5) provides an estimate of a concentration near or below the lethality
733 threshold. The technical rationale for dividing by 2 is found in the *Federal Register* notice
734 soliciting comment on USEPA's methodology for deriving water quality criteria (43 FR 21506,
735 May 18, 1978) (Stephan et al. 1985). Because the estimate was derived for a large battery of
736 species and chemicals, it may not be accurate for particular individual species and chemical
737 combinations, but it is useful for risk screening purposes.

738
739 Elutriate manganese concentrations from samples sites CF 1 (11.5 mg/L), CF 6 (9.25 mg/L), CF
740 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
741 lethal effects threshold concentration for *C. dubia* and manganese (Table 4). The four samples
742 with significant *C. dubia* toxicity were CF 1, CF 6, CF 10 and CF 11 (Table 2). Manganese is a
743 naturally occurring and very common element in soils and surface waters; among the heavy
744 metals, only iron is more abundant in the earth's crust (ATSDR 2000). Impoundments have the
745 potential to increase metal concentrations due to soil disturbance and increased surface area
746 exposed to water. Elevated manganese was the number one problem associated with water
747 quality downstream of Tennessee dams in a recent evaluation (Arnwine et al 2006).
748 Anthropogenic enrichment of manganese can occur through burning fossil fuels, steel
749 production, battery manufacturing, animal feed supplements, fertilizers, wastewater treatments
750 plants (using potassium permanganate), manganese-based fungicides, and antiknock fuel
751 additives (ATSDR 2000). This project did not include any manganese source assessments for
752 the Cape Fear River system.

753
754 Ammonia is often implicated in sediment toxicity evaluations. While ammonia is a natural
755 constituent of freshwater sediments, excess ammonia from pollution can lead to toxic
756 concentrations, and ammonia can also contribute to the toxicity of other sediment contaminants.
757 Ammonia concentrations at the start of our elutriate test were significantly and inversely
758 correlated with *C. dubia* survival in the elutriate test (Table 14). Unlike manganese, however,
759 the elutriate ammonia concentrations did not exceed those expected to be toxic to *C. dubia*.
760 USEPA (1999) report a *C. acanthina* LC50 of 25.78 mg total ammonia nitrogen /L at pH=8.
761 Multiplying that LC50 by 0.5 yields an approximate lethal effects threshold of 12.9 mg-N/L at
762 pH=8. Because ammonia toxicity decreases as pH decreases, we adjusted this lethal effects
763 threshold for the average pH (7.6) of our *C. dubia* elutriate tests using USEPA's (1999) equation
764 11. This yields an estimated total ammonia toxicity threshold of 26 mg-N/L for the Cape Fear
765 River sediment elutriates. The measured elutriate ammonia concentrations (Table 3) were 4- to
766 14-times less than this estimated ammonia effects threshold.

767
768 If it becomes important to more definitively assess the cause of toxicity in the sediment elutriate
769 samples, there are well established toxicity identification evaluation (TIE) approaches that can be

770 applied (USEPA 1993 b, c, Besser et al. 1998, Boucher and Watzin 1999). Non-toxic elutriate
771 samples could be spiked with increasing concentrations of manganese or ammonia to determine
772 toxic concentrations of these constituents to *C. dubia* in a manner inclusive of site-specific
773 elutriate chemistry. Procedures to reduce concentrations of elutriate components (e.g. sequester
774 metals like manganese) also exist to investigate how their presence or absence explains toxicity.
775

776 Elutriate tests aid in the evaluation of the effects of suspended sediments (e.g., dredged material
777 evaluations) within the water column. Mobilization of sediments we tested may be a short-term
778 water column concern based on the elutriate toxicity test results. While sediment re-suspension
779 and contaminant release in the elutriate tests may be near a worst case simulation of actual
780 conditions following sediment disturbing activities, additional synthesis is needed to characterize
781 the nature and magnitude of this issue. In particular, data on the modeled or measured sediment
782 re-suspension caused by specific sediment disturbing activities will help put the elutriate test
783 results in context for normal sediment management practices. The joint U.S. EPA and U.S.
784 Army Corps of Engineers Inland Testing Manual (USEPA/USACE 1998) contains sediment fate
785 models which may be helpful for this purpose. Other issues to be further evaluated regarding the
786 significance of the elutriate test results include more background on manganese concentrations
787 and sources in the Cape Fear River and the relative sensitivity of *C. dubia* to other freshwater
788 organisms, particularly those resident to the Cape Fear River.
789

790 In summary, 12 sediment samples from within the impounded reaches of three locks and dams
791 on the Cape Fear River were collected in November 2006. Elemental contaminant and PAH
792 concentrations in whole sediments were below those of toxicological significance. Whole-
793 sediment toxicity tests with midges and amphipods showed no adverse effects of the sediments
794 on test organism survival or growth. In 2-d sediment elutriate tests with *Ceriodaphnia dubia*,
795 statistically-significant reductions in survival occurred in samples from four sites. Manganese
796 had among the strongest correlation and the most plausible biological association with reduced
797 *C. dubia* survival; elutriate manganese concentrations exceeded published lethal concentrations
798 for *C. dubia* in all four of the toxic samples. Elutriates and pore water samples are not surface
799 waters, but copper, lead, and zinc in these sediment fractions or preparations frequently exceed
800 State water quality standards, an indication that aggressive re-suspension of sediments like those
801 tested could temporarily impair surface water quality. Based on review of existing data (Tier 1)
802 and results of sediment chemistry and toxicity tests (Tier 2 and 3), contamination in surface
803 sediments behind Cape Fear River locks and dams is unlikely to be a concern in-place.

804 Mobilization of sediments may be a short-term water column concern based on the elutriate
805 toxicity test results. While sediment re-suspension and contaminant release in the elutriate tests
806 may be near a worst case simulation of actual conditions following sediment disturbing
807 activities, additional synthesis is needed to characterize the nature and magnitude of this issue.

808 In particular, data regarding the modeled or measured sediment re-suspension caused by specific
809 sediment disturbing activities (e.g., dredging, dam alterations, etc.) will help put the elutriate test
810 results in context for normal sediment management practices. Sediment disturbing activities
811 proposed for the Cape Fear River would benefit from development of a sediment management
812 plan to address these issues. The work presented here addresses the toxicological properties of
813 the sediments evaluated; it does not address sediments other than surface deposits. This report
814 does not inventory the accumulated sediments or address the potential physical impacts of
815 sediment mobilization; these issues could also be addressed in a sediment management plan.

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Appendix A. Analytical Chemistry Reports and Chain of Custody Forms