

**AN ENVIRONMENTAL QUALITY ASSESSMENT
OF
TENNESSEE NATIONAL WILDLIFE REFUGE**



**U.S. Fish and Wildlife Service
Ecological Services
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Cookeville, Tennessee 38501**

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**U.S. FISH and WILDLIFE SERVICE / SOUTHEAST REGION / ATLANTA,
GEORGIA**

U.S. Fish and Wildlife Service
Southeast Region

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OF
TENNESSEE NATIONAL WILDLIFE REFUGE

by

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EXECUTIVE SUMMARY

Samples were collected at the Duck River Unit, Busseltown Unit, and Big Sandy Unit of Tennessee National Wildlife Refuge (NWR)(Figure 1) from 1996 to 1998. Fish and sediment samples were obtained at each of these units. Semi-permeable membrane devices (SPMDs) were deployed at the Duck River Unit. Wood duck (*Aix sponsa*) eggs were also collected at the Duck River Unit.

Comparisons of the data collected for this investigation are made to previous data collected at Tennessee NWR in the Tennessee River watershed, Cross Creeks NWR in the Cumberland River watershed, and 5 refuges in the Mississippi River watershed of West Tennessee. Our data is also compared to data generated as part of the Biomonitoring of Environmental Status and Trends (BEST) program conducted by the United States Geological Survey (USGS) and United States Fish and Wildlife Service (FWS). These comparisons are intended to document temporal and spatial patterns of various contaminants in select species and sediments in the major watersheds within the State of Tennessee.

This study, Project ID No. 4N45, was initiated in September 1996 at the Duck River Unit of the refuge. Composite young-of-year bluegill (*Lepomis macrochirus*) and gizzard shad (*Dorosoma cepedianum*); common carp (*Cyprinus carpio*) muscle, liver and gonad; white crappie (*Pomoxis annularis*) muscle; and sediment samples were collected and submitted (ECDMS Catalog No. 4050023) for analyses. Organochlorine analyses for the fish and sediment samples and a metals scan for the sediment samples were performed by the Hand Chemical Laboratory at Mississippi State University and Midwest Research Institute, in Kansas City, Missouri, respectively. Largemouth bass (*Micropterus salmoides*) were also collected and a fish health assessment performed. Histological examination of the common carp and largemouth bass livers revealed discoloration, macrophage aggregations, and external and internal lesions. Nine of the ten liver samples submitted had PCB-total concentrations ranging from 0.12 to 1.80 ppm (wet weight (ww)). The common carp muscle samples also had PCB-total concentrations of 0.077 and 0.066 ppm ww.

In 1997, our efforts included the collection of wood duck eggs from the Duck River Unit. Four SPMD's were also deployed (Figure 2) and sediment samples collected at these locations. Bigmouth buffalo (*Ictiobus cyprinellus*), bowfin (*Amia calva*), largemouth bass, white crappie, bluegill, and gizzard shad were collected at the Busseltown Unit (Figure 3). These samples and two archived 1996 whole-body carp samples from the Duck River Unit (ECDMS Catalog No. 4050025) were submitted for organic and inorganic analyses (the archived carp samples were analyzed for inorganic parameters only). Organochlorine pesticide and AHH-active polychlorinated biphenyl (PCB) congener analyses for the fish, wood duck eggs, SPMDs, and sediment samples were performed by the Hand Chemical Laboratory at Mississippi State

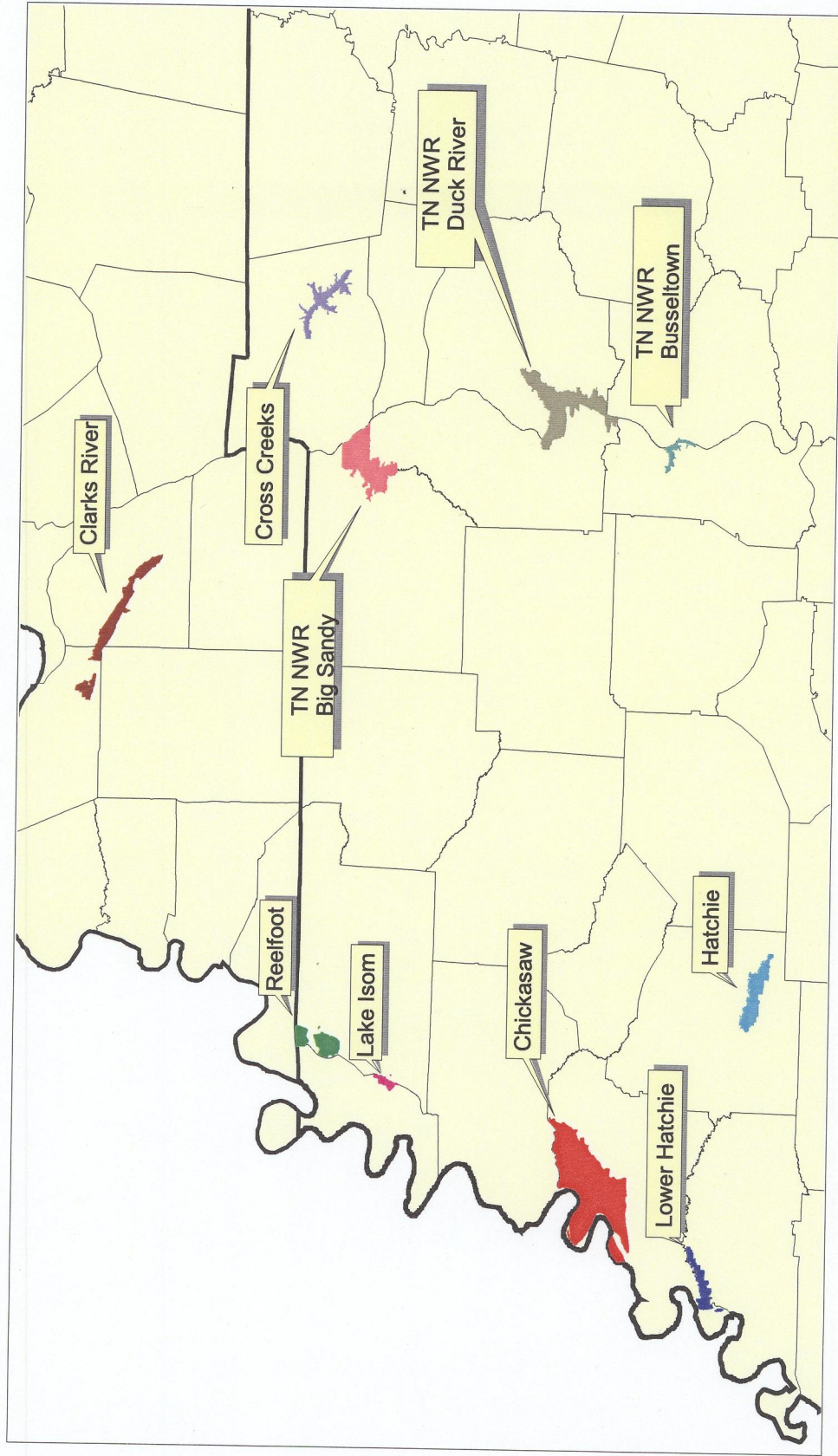
University. This laboratory also performed the organochlorine, organophosphate, and triazine pesticide analyses of the SPMD dialysates. The metals scan for fish and sediment samples was performed at Research Triangle Institute, Research Triangle Park, North Carolina.

Our efforts in 1998 included the collection of largemouth bass, gizzard shad, emerald shiners (*Notropis atherinoides*), bluegill, longear sunfish (*Lepomis megalotis*), and sediment samples at the Big Sandy Unit (Figure 4). Additional sediment samples were also collected at the Busseltown Unit. Significant electroshocking effort did not produce the desired number of fish samples in the West Sandy embayment at the Big Sandy Unit. The Tennessee Department of Environment and Conservation (TDEC) has periodically sampled sediment on the upstream side of the dike on the embayment, and elevated levels of metals and organic contaminants were found. These contaminants are likely associated with individual National Pollutant Discharge Elimination System (NPDES) discharges from municipal and industrial wastewater treatment facilities. Additional sampling was conducted in June 1999 along the eastern shore of the Big Sandy River above the confluence of West Sandy Creek. Spotted sucker (*Minytrema melanops*), freshwater drum (*Aplodinotus grunniens*), and largemouth bass were collected. The spotted sucker had discolored and malformed gill covers. These samples (ECDMS Catalog No. 4050027) were submitted for chlorinated hydrocarbon insecticide, AHH-active PCB congener, and inorganic analyses.

Nine of the ten liver samples submitted had PCB-total concentrations ranging from 0.12 to 1.80 ppm ww. The common carp muscle samples also had PCB-total concentrations of 0.077 and 0.066 ppm ww. Total PCBs in wood duck eggs, fish, sediment, and SPMD dialysates were all below the reported detection limits, however, specific individual AHH-active congeners were detected in all sample matrices. DDD (p-p') was detected in bowfin liver and gonads (0.022 and 0.024 ppm ww, respectively). DDE (p-p') was detected in wood duck eggs (0.017-0.023 ppm ww), bowfin liver and gonads (0.190 and 0.220 ppm ww, respectively), and in whole-body bigmouth buffalo, bowfin, bluegill, and gizzard shad (0.010-0.070 ppm ww). Trans-nonachlor was detected (0.0140 ppm ww) in bowfin liver and gonads. These organochlorines were not detected in the largemouth bass and white crappie filets.

Aluminum (1.39-2.67 ppm ww), barium (1.76-3.40 ppm ww), mercury (0.0366-0.0701 ppm ww), and selenium (0.354-0.674 ppm ww) were detected in wood duck eggs. Arsenic (0.249 ppm ww) and chromium (1.30 ppm ww) were also detected in one composite wood duck egg sample. Mercury ranged from 0.045 to 0.417 ppm ww in whole-body fish samples. Selenium was detected at 11.6 ppm ww in the bowfin liver sample. Lead values of 0.339, 0.365, and 0.321 ppm ww were found in bluegill, carp, and gizzard shad, respectively. Elevated levels (2.59 - 12.40 ppm dw) of arsenic and slightly elevated levels of cadmium (0.444 - 0.940 ppm dw) were detected in the sediment samples. Elevated barium levels were detected in whole-body fish samples (0.419 - 5.11 ppm ww) and sediment (78.9 - 121.0 ppm dw).

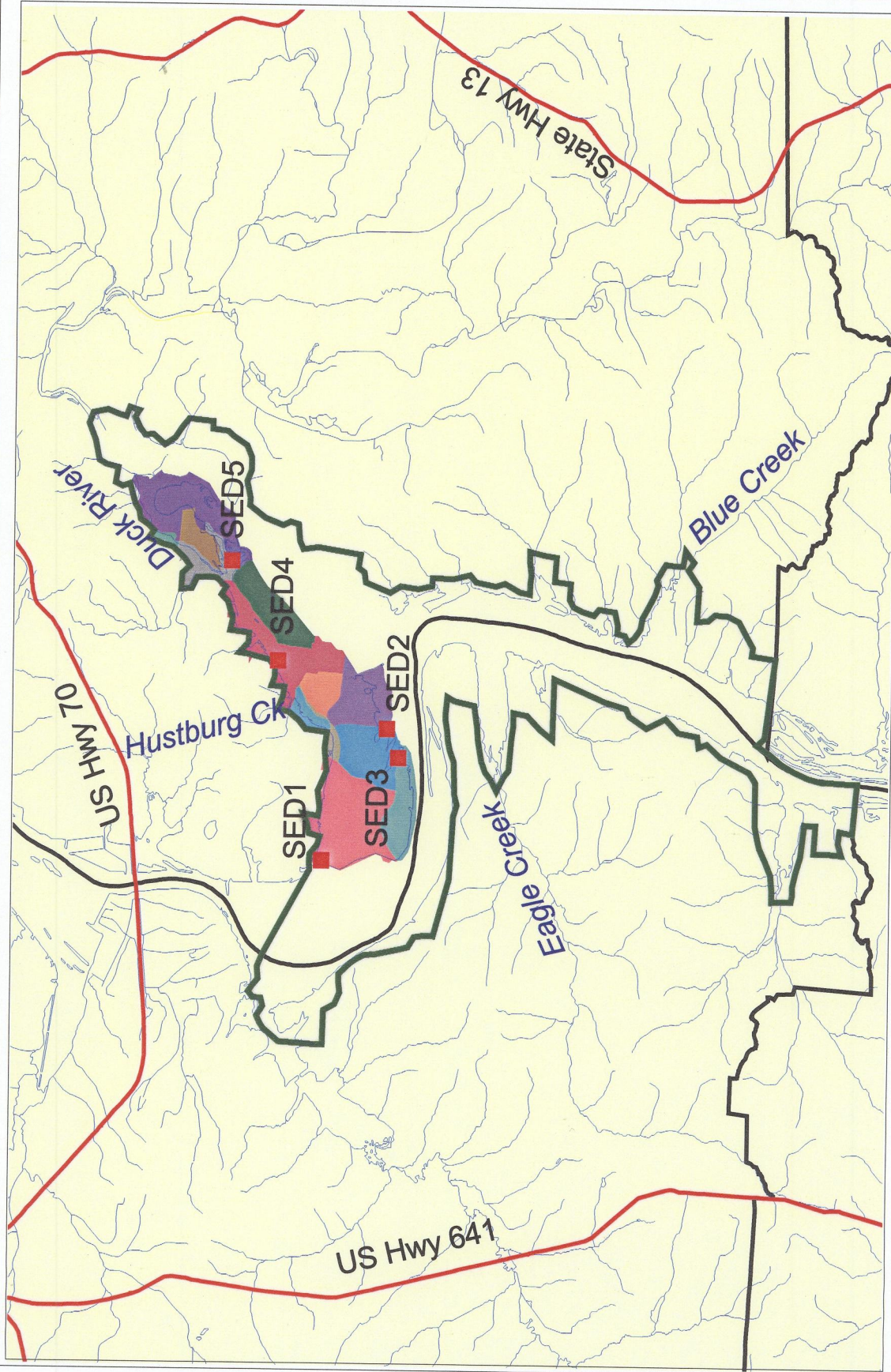
Figure 1. National Wildlife Refuges in Tennessee and Kentucky



Map produced on June 25, 2002 by the Area II
GIS Center in the Cookeville, TN Field Office



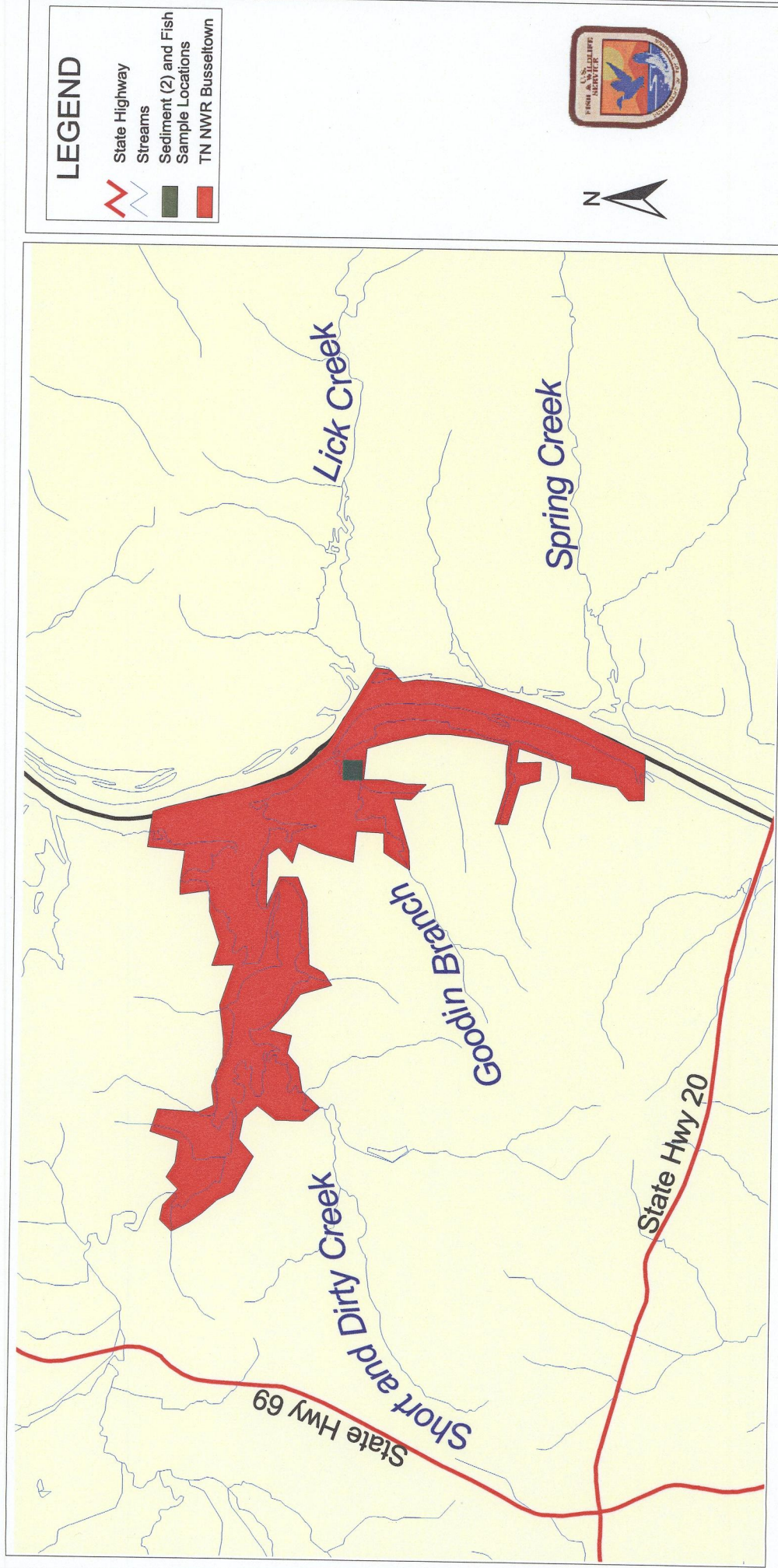
Figure 2. Sample Locations in Duck River Unit Tennessee National Wildlife Refuge



Map produced on June 25, 2002 by the Area II
GIS Center in the Cookeville, TN Field Office

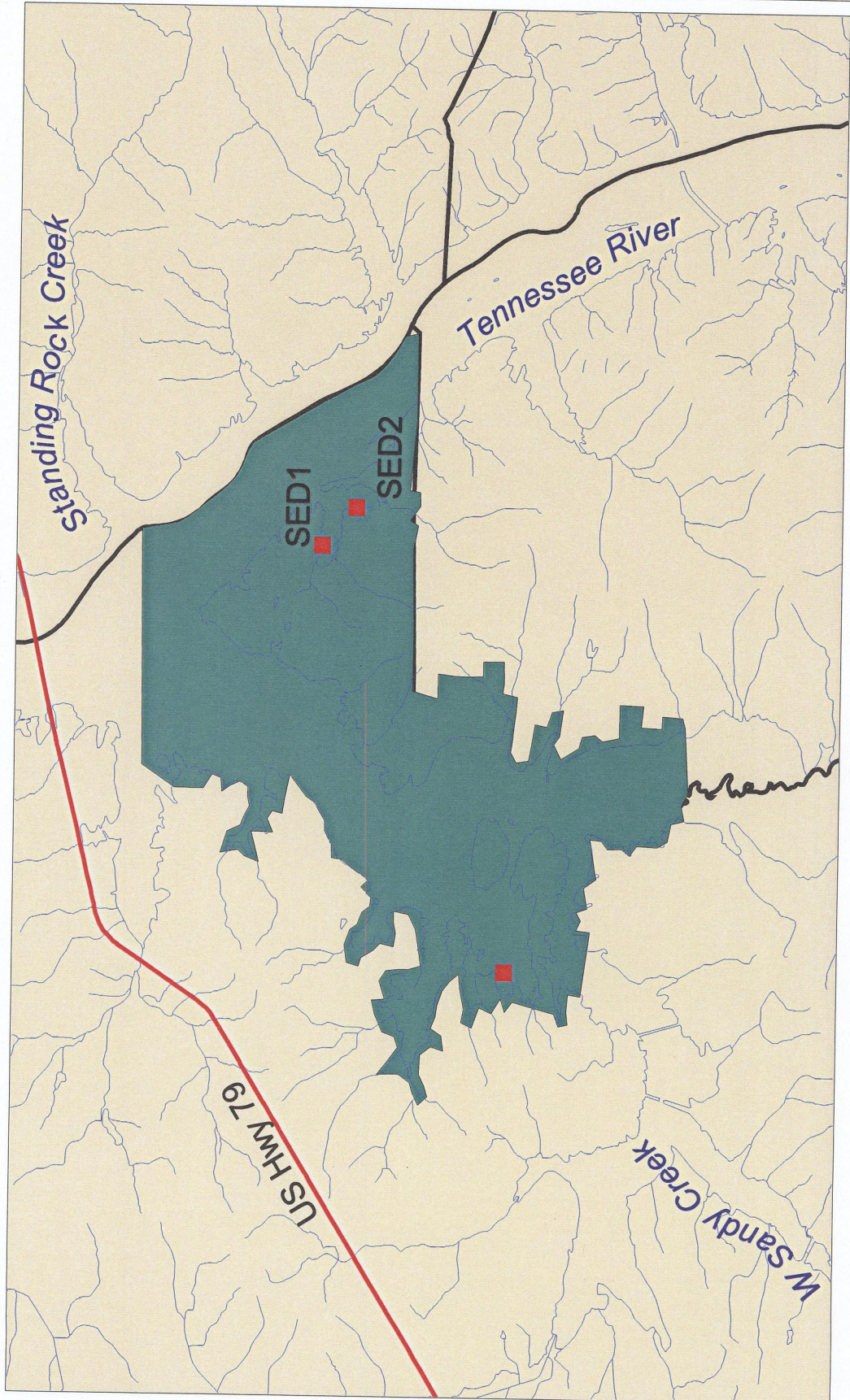
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Figure 3. Sampling Locations in Busseltown Unit Tennessee National Wildlife Refuge



Map produced on June 25, 2002 by the Area II GIS Center in the Cookeville, TN Field Office

Figure 4. Sample Locations in Big Sandy Unit Tennessee National Wildlife Refuge



Map produced on June 25, 2002 by the Area II
GIS Center in the Cookeville, TN Field Office

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INTRODUCTION

Tennessee NWR was established in 1945 in conjunction with the creation of Kentucky Lake. Executive Order 9670, Tennessee NWR's enabling legislation, calls for the refuge to "further the purposes of the Migratory Bird Conservation Act (45 Stat. 1222)" and to use the land "as a refuge and wildlife management area for migratory birds and other wildlife."

The 51,358-acre Tennessee NWR, located along approximately 65 river miles of Kentucky Lake in Benton, Decatur, Henry, and Humphreys counties, is divided into three units: the Big Sandy, Duck River, and Busseltown units (Figure 1). All three units are located within the Western Highland Rim of the Interior Plateau ecoregion (Omernik 1987). There is a total of 3,000 acres of cropland on all three units, with approximately 700 acres on the Big Sandy Unit, 1600 on the Duck River Unit, and 700 acres on the Busseltown Unit. This land is farmed each year through cooperative farming agreements to provide supplemental food and cover for thousands of waterfowl. In addition, approximately 1,500 acres in the Duck River Bottoms are managed for moist-soil vegetation production. These areas are compartmentalized by a series of levees and water control structures that allow water levels to be controlled for optimum waterfowl food production. The refuge contains approximately 20,000 acres of forest, with the majority being comprised of upland stands. Small isolated blocks of bottomland hardwoods occur on the Duck River and Busseltown units. The predominant forest type is oak-hickory. Acreage not falling into the forested, agricultural, or moist-soil categories primarily consists of open water habitats.

There are presently six Federally-listed animal species that occur on the refuge. They are the bald eagle (*Haliaeetus leucocephalus*), least tern (*Sterna antillarum*), wood stork (*Mycteria americana*), and three freshwater mussels: Cumberland pigtoe (*Pleurobema gibberum*), pink mucket (*Lampsilis abrupta*) and ring pink (*Obovaria retusa*). There are no known Federally-listed flora on the refuge.

The refuge serves as an important wintering ground for thousands of migratory waterfowl and shorebirds in the Mississippi Flyway. Tennessee NWR winters about 200,000 ducks and approximately 20,000 Canada geese. The Southern James Bay population of Canada geese make up about 40-60% of the geese using the refuge. There is much concern for this population, as it has been in steady decline in recent years. The refuge is also the most significant area for wintering American black ducks in Tennessee, holding 50-75% of the population migrating to this state (Tennessee NWR and Tennessee Wildlife Resource Agency, unpub. data). Other waterfowl species present in significant numbers during fall and winter include the mallard (*Anas platyrhynchos*), gadwall (*Anas strepera*), American wigeon (*Anas americana*), blue-winged teal (*Anas discors*), green-winged teal (*Anas crecca*), northern pintail (*Anas acuta*), ring-necked duck (*Aythya collaris*), and canvasback (*Aythya valisineria*). The only waterfowl species to nest in any significant numbers on the refuge is the wood duck (*Aix sponsa*).

In addition to waterfowl management, the refuge is involved in developing innovative management techniques for a wide variety of migratory songbirds that breed in upland forested habitats. Many of these bird species have experienced severe population declines over the past few decades, alarming both wildlife professionals and the general public. The refuge is located within the Tennessee River Corridor, which currently is one of the largest forested areas in the Southeast, making the refuge's forest management research program critical in the effort to reverse the declining population trends of several bird species.

WETLAND RESOURCES

Wetlands provide essential watershed functions related to floodwater storage, groundwater flow moderation, sediment removal, nutrient cycling, and water purification. They provide diverse habitats for wildlife foraging and reproduction, and essential refugia for a wide variety of mammals, reptiles, amphibians, and fish. Three broad categories of wetlands exist within the boundaries of Tennessee NWR: lacustrine, palustrine, and riverine.

Lacustrine wetland systems include wetlands and deepwater habitats that are 1) situated in a topographical depression or dammed river channel; 2) lacking trees, shrubs, persistent emergents, emergent mosses or lichens with greater than 30% areal coverage; and 3) a total area exceeding 20 acres. *Palustrine* systems include all wetlands dominated by trees, shrubs, persistent emergents, and emergent mosses or lichens. They also include: 1) wetlands lacking this type of vegetation, but less than 20 acres in size with active wave-formed or bedrock shorelines and a water depth less than 2 meters, and 2) small, shallow, permanent or intermittent water bodies (ponds). *Riverine* systems include all wetlands and deepwater habitats contained within a stream channel excluding those areas dominated by trees, shrubs, persistent emergents, emergent mosses, or lichens. These systems are further defined by water permanence, gradient, water velocity, substrate, extent of floodplain development, and vegetation type.

Our GIS analyses indicated that the following general wetland types and acreages are present on Tennessee NWR:

Duck River Unit

Lacustrine

Impounded	7,262 acres
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Palustrine

Unconsolidated Bottom	11 acres
Emergent	1,394 acres
Scrub-Shrub	1,217 acres
Forested	3,747 acres

Riverine	100 acres
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Busseltown Unit

Lacustrine

Impounded	1,062 acres
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Palustrine

Unconsolidated Bottom	12 acres
Emergent	47 acres
Scrub-Shrub	173 acres
Forested	550 acres

Big Sandy Unit

Lacustrine

Impounded	13,406 acres
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Palustrine

Unconsolidated Bottom	9 acres
Emergent	73 acres
Scrub-Shrub	60 acres
Forested	305 acres

Field verification has not been performed in all of the mapped wetland areas. Field verification of NWI mapped wetland areas should be conducted as part of future biological and water quality monitoring efforts on Tennessee NWR.

METHODS AND MATERIALS

Sample Collection. Fish specimens were collected by Service personnel using standard boat-electrofishing techniques. Samples were kept in the boat's live-well until collecting efforts were completed. Fish were sorted by species and age, and those retained for analysis were weighed and measured (total length). Liver and gonad samples were also taken from select species. Specimens for the composite samples were then wrapped in clean aluminum foil, labeled and sealed in plastic bags. The samples were held on wet ice during transport, frozen and shipped to the analytical laboratory for analysis.

Wood duck eggs were collected from nest boxes on the Duck River Unit. Five composite (2-3 eggs) samples were prepared, held on wet ice during transport, refrigerated and shipped to the analytical laboratory for analysis.

Sediment samples were collected by Service personnel using a petite ponar dredge. Samples were collected in areas where fine-grained, silty, depositional substrate was overlain by relatively shallow water. The samples were held on wet ice during transport, refrigerated, and shipped to the analytical laboratory for analysis.

SPMDs were deployed at four locations on the Duck River Unit (Figure 2). A SPMD blank was utilized and exposed to the ambient atmosphere during deployment and retrieval. The membranes were refrigerated and held until shipment to CIA/EST Laboratories in St. Joseph, Missouri. The membranes were extracted, and the dialysates returned. The dialysates were then shipped on dry ice to the analytical laboratory for analysis.

Sample Analysis. Fish samples were prepared for chlorinated hydrocarbon pesticides, total PCBs, and AHH-active PCB congener analyses (Table 1) by taking a five-gram (g) aliquot from a well-mixed composite sample and combining with up to 150 g of anhydrous sodium sulfate. These samples were then extracted with hexane for seven hours in a Soxhlet extractor and concentrated using a rotary evaporator. The extract was then air-dried to a constant weight (usually 4 days) for lipid determinations. After weighing, the lipid sample was dissolved in petroleum ether (12 ml) saturated with acetonitrile and extracted four times each with 30 ml of acetonitrile saturated with petroleum ether. Residues were partitioned into petroleum ether, washed, concentrated to 5 ml, and transferred to a glass elution column containing 20 g of Florisil.

The Florisil column was eluted with a 200 ml mixture of 6% diethyl ether and 94% petroleum ether (Fraction I) followed by 200 ml of 15% diethyl ether and 85% petroleum ether (Fraction II). Fraction II was then concentrated for quantification of chemical residues using capillary or megabore columns and electron capture gas chromatography. Fraction I was concentrated to 5 ml and transferred to a silicic acid chromatographic column for additional cleanup and separation

of PCBs. Three elutriate fractions were obtained and concentrated to 10 ml for quantification using capillary or megabore columns and electron capture gas chromatography. Elution profiles for the separation columns are shown in Appendix A.

Sediment samples were prepared for analysis of insecticides and AHH-active PCB congeners by taking a 20 g aliquot and mixing with sulfuric acid, acetone, and a 1:1 mixture of petroleum ether and ethyl ether. Detailed procedures are included in Appendix C.

Pesticide, total PCB, and AHH-active PCB congener assays on fish, wood duck eggs, SPMD dialysates, and sediment samples were performed by the Hand Chemical Laboratory at Mississippi State University. Organophosphate and triazine pesticide analyses (Table 2) of the SPMD dialysates were also performed by the Hand Chemical Laboratory.

Fish samples were prepared for metal analyses (Table 3) by freeze-drying aliquots of well homogenized composite samples. After moisture content was determined, a 0.25 to 0.5 g aliquot of freeze-dried tissue was digested with nitric acid. Specific procedures are included in Appendix B. Fish, wood duck eggs, and sediment were analyzed for metals by Midwest Research Institute in Kansas City, Missouri, and Research Triangle Institute in Research Triangle Park, North Carolina. Arsenic and Se were analyzed using atomic absorption (AA) spectrophotometry. Mercury was analyzed using a standard cold vapor AA technique. All other metals were analyzed using inductively coupled plasma (ICP) emission spectrometry.

Results for tissue samples are given in wet weight values to provide comparisons with other studies. Results for sediment samples are given in dry weight values. Conversion between wet weight and dry weight values was accomplished using the following formulas:

$$\text{Dry weight concentration} = \text{wet weight concentration} / (1 - (\% \text{Moisture} \div 100))$$

$$\text{Wet weight concentration} = \text{dry weight concentration} * (1 - (\% \text{Moisture} \div 100))$$

Both wet and dry weight values are provided to ease comparisons with data reported from previous or future studies by other investigators.

Table 1. Chlorinated Hydrocarbon Insecticides and AHH-active Polychlorinated Biphenyl (PCB) Congeners Analyzed in Samples from Tennessee National Wildlife Refuge.

Insecticides

HCB (hexachlorobenzene) aka perchlorobenzene
BHC (benzene hexachloride) aka hexachlorocyclohexane
alpha, beta, gamma, delta isomers

Oxychlorthane

Chlordane (alpha, gamma)

Nonachlor (trans, cis)

Heptachlor epoxide

DDT (*o,p'* and *p,p'*)

DDE (*o,p'* and *p,p'*)

DDD (*o,p'* and *p,p'*)

Endrin

Dieldrin

Mirex

Toxaphene

Total PCBs

AHH-active PCB Congeners 77, 81, 105, 114, 118, 126, 128, 138, 156, 157, 158, 166, 167, 169, 170, 189

Table 2. Organophosphate Pesticides and Triazine Herbicides Analyzed in SPMD Samples from Tennessee National Wildlife Refuge.

chlorpyrifos	atrazine
diazinon	simazine
malathion	propazine
methyl parathion	
parathion	
profenaphos	

Table 3. Metals Analyzed in Samples from Tennessee National Wildlife Refuge.

Aluminum	(Al)	Chromium	(Cr)	Molybdenum	(Mo)
Arsenic	(As)	Copper	(Cu)	Nickel	(Ni)
Barium	(Ba)	Iron	(Fe)	Selenium	(Se)
Beryllium	(Be)	Lead	(Pb)	Strontium	(Sr)
Boron	(B)	Magnesium	(Mg)	Vanadium	(Vn)
Cadmium	(Cd)	Manganese	(Mn)	Zinc	(Zn)
Cobalt	(Co)	Mercury	(Hg)		

Quality Assurance/Quality Control (QA/QC)

Laboratory quality control was verified by the Patuxent Analytical Control Facility at the Patuxent National Wildlife Research Center in Laurel, Maryland. The precision and accuracy of the analytical results were confirmed with matrix and reagent blanks, duplicate analysis of randomly selected samples, recoveries of spiked analytes, and analysis of samples from the National Institute of Standards and Technology, the U.S. Environmental Protection Agency (USEPA), and the use of FWS reference materials. With only a few exceptions, all analytical results were considered to be acceptable.

The laboratories used in these investigations completed a considerable amount of QA/QC analyses. This included analyzing several samples in duplicate. The analytical results for these laboratory duplicates and other detailed QA/QC information is not included in this report, but is available upon request.

RESULTS

Duck River Unit

This study, Project ID No. 4N45, was initiated in September 1996 at the Duck River Unit of Tennessee National Wildlife Refuge (Figure 2). Composite young-of-year bluegill (*Lepomis macrochirus*) and gizzard shad (*Dorosoma cepedianum*); common carp (*Cyprinus carpio*) filet, liver and gonad; and white crappie (*Pomoxis annularis*) filet were collected from the Duck River Unit and analyzed for organochlorines (Table 4). Largemouth bass (*Micropterus salmoides*) were also collected and a fish health assessment performed. Histological examination of the common carp and largemouth bass revealed discoloration of, and macrophage aggregations in, the liver. Internal and external lesions were also present. No gross abnormalities were observed in the other species collected.

Two DDT-related compounds (*p,p'*-DDD and DDE) were detected (Table 5). DDD was detected in six of the common carp liver samples, ranging from 0.013 to 0.029 ppm. DDE was detected in all common carp filet, liver, and gonad samples (0.063 - 0.270 ppm), as well as whole-body bluegill and gizzard shad (0.037 and 0.018 ppm, respectively). All ten common carp liver samples submitted had total PCB concentrations ranging from 0.12 to 1.80 ppm wet weight (Table 5). The two common carp filet samples had total PCB concentrations of 0.077 and 0.066 ppm ww, respectively.

Wood duck eggs were collected in 1997 and analyzed for organochlorines and metals. Eggs contained low levels (<0.0058 ppm ww) of PCB congeners 81, 105, 114, 118, 128, 138, 156, 167, 170, and 189. DDE (*p-p'*) was detected in three wood duck egg samples (0.017-0.023 ppm ww). DDD (*p,p'*-) was not detected in any of the composite egg samples (Table 12). Eleven of nineteen metals analyzed were present (Table 13). Aluminum (1.39-2.67 ppm ww), barium (1.76-3.40 ppm ww), mercury (0.0366-0.0701 ppm ww), and selenium (0.354-0.674 ppm ww) were detected in the samples. Arsenic (0.249 ppm ww) and chromium (1.30 ppm ww) were also detected in one composite wood duck egg sample. Cadmium was not detected.

Sediment samples were collected from Pool 1, Pool 2, Pool 10, Pool 6, and Pool 9 (Gaynor Slough). Semi-permeable membrane devices were also deployed at select sites at the Duck River Unit (Figure 2). Nineteen metals were detected in the sediment samples collected (Table 6). Mercury (Hg) was present at two sites, Pool2 (0.076 ppm) and Blue Creek (Table 11). Elevated levels of barium (121-209 ppm dry weight (dw)) were detected at all of the impoundments and Blue Creek. Average strontium concentrations were approximately four-fold higher in Duck River Unit sediment samples than those collected at the Busseltown and Big Sandy Units. No chlorinated hydrocarbon insecticides or PCBs were detected in the sediment samples. PCB congeners 105, 118, 135, and 8 were detected at low levels (<0.0034 mg/l) in the SPMD dialysates.

Busseltown Unit

Whole-body bigmouth buffalo (*Ictiobus cyprinellus*), bowfin (*Amia calva*), bluegill, and gizzard shad, largemouth bass and white crappie fillets, as well as bowfin liver and gonads were collected from the Busseltown Unit Impoundment (Figure 3) for organochlorine and metal analysis (Table 8). In addition, whole-body common carp was collected from the Duck River/Gaynor Slough for metal analysis (Tables 7 and 8). Bowfin liver and gonad samples contained low levels (<0.0180 ppm ww) of PCB congeners 81, 105, 118, 128, 157, 158, 167, 170, and 189. Young-of-year gizzard shad contained low levels (<0.0029 ppm ww) of PCB congeners 81, 105, 114, 118, 138, 156, 157, and 167. Young-of-year bluegill contained low levels (<0.0040 ppm ww) of congeners PCB 81, 105, 118, 138, 156, 167, and 170. DDE (p,p'-) was detected in bowfin liver and gonads (0.190 and 0.220 ppm ww, respectively), and in whole-body bigmouth buffalo, bowfin, bluegill, and gizzard shad (0.010-0.580 ppm ww) (Table 7). DDD (p,p'-) was detected in bowfin liver and gonads (0.022 and 0.024 ppm ww, respectively) and in a whole-body bigmouth buffalo (0.010 ppm ww). Trans-nonachlor was detected (0.014 ppm ww) in bowfin liver and gonads. These organochlorines were not detected in the largemouth bass and white crappie filets.

Mercury ranged from 0.045 to 0.417 ppm ww in whole-body fish samples. Selenium was detected at 11.6 ppm ww in the bowfin liver sample. Lead values of 0.339, 0.365, and 0.321 ppm ww were found in bluegill, carp, and gizzard shad, respectively. Elevated barium levels were detected in whole-body fish samples (0.419 - 5.11 ppm ww). Cadmium was found in bowfin liver gonads and liver (0.046 and 0.260 ppm, respectively), as well as whole-body samples of bigmouth buffalo, common carp, bluegill, and gizzard shad (Table 9).

Sediment was collected from impoundments and Goodin Branch on the Busseltown Unit (Tables 10 and 17). Elevated levels (2.59 - 12.40 ppm dw) of arsenic, barium (78.9 - 121.0 ppm dw) and slightly elevated levels of cadmium (0.444 - 0.940 ppm dw) were detected in sediment samples (Tables 11 and 18). Magnesium concentrations in sediment samples collected from impoundments at the Busseltown Unit and Goodin Branch were 4-fold higher than samples from the Big Sandy Unit and from creeks on the Duck River Unit. Cadmium was detected in sediments from Busseltown with an average value of 0.478 ppm. Aluminum concentrations were approximately three-fold higher in Busseltown sediments (15335 ppm average) compared to Big Sandy sediments (5352 ppm average).

Big Sandy Unit

Whole-body bluegill, gizzard shad, emerald shiner (*Notropis atherinoides*), longear sunfish (*Lepomis megalotis*), largemouth bass, smallmouth bass (*Micropterus dolomieu*), freshwater drum (*Aplodinotus grunniens*), and spotted sucker (*Minytrema melanops*) were collected at the Big Sandy Unit (Figure 4) and analyzed for organochlorines and metals (Tables 14 and 15). Low levels of p,p'-DDE and DDD were found in all samples except gizzard shad and drum

(Table 14). DDD was detected only in the emerald shiner (0.016 ppm ww). Concentrations of *p,p'*-DDE ranged from 0.010 to 0.035 ppm. Fifteen of nineteen metals analyzed were detected in fish samples (Table 16). Mercury concentrations ranged from 0.010 ppm in gizzard shad to 0.384 ppm in smallmouth bass. Cadmium was found in emerald shiner, gizzard shad, smallmouth bass and largemouth bass and one spotted sucker sample in concentrations ranging from 0.055 ppm (emerald shiner) to 0.124 ppm (smallmouth bass).

Sediment was collected from the Big Sandy Unit and analyzed for organochlorines and metals. None of the organochlorines analyzed were detected in sediment samples. Eighteen of nineteen metals analyzed were found in some or all of the samples. Percent moisture, total organic carbon, sand, silt, and clay of each sample are listed in Table 17. Mercury concentrations ranged from 0.046 ppm dw to 0.092 ppm dw. Cadmium was not found in sediments from Big Sandy, although samples from four fish species did contain detectable levels of Cd.

Table 4. Information for 1996 Fish Samples Collected at Duck River Unit, Tennessee NWR.

Sample Number	Species	Composite Sample	Weight (lbs)	Length (cm)
CC1	Common carp	NA	2 lbs. 13oz.	45.5
CC2	Common carp	NA	2 lbs. 7 oz.	43.5
CC3	Common carp	NA	3 lbs. 12 oz.	50.0
CC4	Common carp	NA	2 lbs. 6 oz.	41.0
CC5	Common carp	NA	3 lbs. 8 oz.	53.3
CC6	Common carp	NA	2 lbs. 15 oz.	48.1
CC7	Common carp	NA	2 lbs. 13 oz.	43.0
CC8	Common carp	NA	2 lbs. 14 oz.	45.5
CC9	Common carp	NA	3 lbs. 6 oz.	47.0
CC10	Common carp	NA	2 lbs. 12 oz.	45.3
LMB1	Largemouth bass	NA	4 lbs. 0 oz.	48.9
LMB2	Largemouth bass	NA	8 oz.	26.6
WB1	White bass	NA	4 lbs. 5 oz.	49.0
WC1	White crappie	NA	1 lb. 0 oz	29.0
WC2	White crappie	NA	1 lb. 2 oz.	29.5
WC3	White crappie	NA	1 lb. 4 oz.	31.8
WC4	White crappie	NA	1 lb.	28.9
WC5	White crappie	NA	1 lb. 1 oz.	29.6
WC6	White crappie	NA	1 lbs. 2 oz.	30.2
WC7	White crappie	NA	1 lb. 1 oz.	29.0
WC8	White crappie	NA	15 oz.	27.8
WC9	White crappie	NA	12 oz.	26.6
WC10	White crappie	NA	6 oz.	21.9
BG	Bluegill	5		NA
GS	Gizzard shad	4		NA

Table 5. Organic Analyses of Fish Samples Collected in 1996 at Duck River Unit. Chlorinated Hydrocarbon Insecticides and Total PCBs Detected (ppm, ww).

Sample Number	Species	Sample Matrix	Weight	% Moisture	% Lipids	p,p'-DDE	p,p'-DDD	Total PCBs
96CC1	Common carp	muscle	1150	77.2	2.06	.064	<.010	.077
96CC2	Common carp	muscle	470	77.2	2.27	.071	<.010	.066
96CCR1	Common carp	gonad	157	68.2	3.22	.085	<.010	<.050
96CCR2	Common carp	gonad	194	69.2	2.70	.063	<.010	<.050
96CCR3	Common carp	gonad	172	86.4	2.74	.087	<.010	<.050
96CCL1	Common carp	liver	3.87	74.9	3.91	.270	.023	.750
96CCL2	Common carp	liver	1.76	80.1	2.97	.100	<.010	.430
96CCL3	Common carp	liver	3.94	75.0	3.93	.140	.013	.430
96CCL4	Common carp	liver	1.29	---	3.98	.066	<.010	.120
96CCL5	Common carp	liver	2.54	80.2	3.05	.094	<.010	.350
96CCL6	Common carp	liver	2.93	77.9	4.07	.210	.021	.250
96CCL7	Common carp	liver	2.26	80.8	2.44	.073	<.010	.260
96CCL8	Common carp	liver	2.77	78.5	4.03	.180	0.017	.280
96CCL9	Common carp	liver	1.64	72.1	4.99	.220	0.024	.410
96CCL10	Common carp	liver	3.06	78.4	4.02	.240	0.029	1.80
96BG	Bluegill	whole-body	381	77.2	2.56	.037	<.010	<.050
96GS	Gizzard shad	whole-body	172	82.4	1.57	.018	<.010	<.050
96WCF1	White crappie	muscle	422	78.4	0.74	<.010	<.010	<.050
96WCF2	White crappie	muscle	352	78.2	0.66	<.010	<.010	<.050

Table 6. Metal Concentrations (ppm, dw) in 1996 Sediment Samples from Duck River Unit.

Sample Number	96SED1 (Pool 1)	96SED2 (Pool 2)	96SED3 (Pool 10)	96SED4 (Pool 6)	96SED5 (Pool 9)
Al	27,293	16,818	20,339	24,656	20,453
As	3.82	3.33	2.95	3.54	3.88
B	3.41	3.66	4.47	4.03	4.22
Ba	209	121	155	184	157
Be	1.42	0.92	1.18	1.07	0.95
Cd	.343	.322	.383	.435	.039
Cr	24.0	16.8	19.9	20.5	17.6
Cu	16.1	11.9	13.4	13.6	11.4
Fe	29,621	19,312	22,039	24,608	22,905
Hg	<.074	.076	<.067	<.056	<.057
Mg	2170	1643	1885	1853	1591
Mn	2590	1787	1364	2164	2195
Mo	1.77	1.72	2.21	<.597	<.601
Ni	25.4	17.0	20.6	21.4	18.9
Pb	22.0	15.9	18.0	19.5	19.3
Se	.545	.341	.171	.225	<.158
Sr	46.9	29.1	32.7	40.0	37.5
V	37.9	23.8	28.1	33.8	28.3
Zn	85.3	59.6	70.9	72.8	63.0
Weight (g)	81.4	329	324	322	324
% Moisture	45.7	37.3	37.8	32.9	35.9

Table 7. Organic Analyses of 1997 Fish Samples Collected at Busseltown Unit. Chlorinated Hydrocarbon Insecticides Detected (ppm, ww).

Sample Number	Species	Sample Matrix	Weight (g)	% Moisture	% Lipids	p,p'-DDE	p,p'-DDD	trans-nonachlor
97BFING	Bowfin	gonads	21.7	72.0	11.7	.220	.024	.014
97BFINL	Bowfin	liver	35.6	65.5	12.8	.190	.022	.014
97LMB1	Largemouth bass	muscle	784	79.0	.569	<.048	<.048	<.048
97WC1	White crappie	muscle	467	79.5	.493	<.049	<.049	<.049
97BBUFF	Bigmouth buffalo	whole-body	4150	72.5	12.3	.580	.01	<0.01
97BFIN	Bowfin	whole-body	2780	75.5	3.94	.070	<.010	<0.01
97BG	Bluegill	whole-body	1160	74.5	2.76	.028	<.010	<0.01
97GS	Gizzard shad	whole-body	1010	78.5	5.9	.010	<.010	<0.01

Table 8. Metal Analyses of 1997 Fish Samples Collected at Busseltown Unit and Duck River Unit.

Sample Number	Species	Sample Matrix	Weight (g)	Moisture (%)
97BFING	Bowfin	gonads	17.2	72.4
97BFINL	Bowfin	liver	28.9	63.9
97LMB1	Largemouth bass	muscle	45.1	75.9
97WC1	White crappie	muscle	44.4	79.5
97BBUFF	Bigmouth buffalo	whole-body	417	70.2
97BFIN	Bowfin	whole-body	303	73.7
97BG	Bluegill	whole-body	304	73.6
97DRCAR1*	Common carp	whole-body	3570	68.9
97DRCAR2*	Common carp	whole-body	3030	68.1
97GS	Gizzard shad	whole-body	258	78.2

* Samples collected from Duck River Unit.

Table 9. Metal Concentrations in 1997 Fish Samples Collected at Busselton Unit (ppm, ww).

Sample Number	97 BFING	97 BFINL	97 LMBI	97 WC1	97 BBUFF	97 BFIN	97 BG	*97 DRCAR1	*97 DRCAR2	97 GS
Al	2.30	19.3	5.78	3.26	108	8.46	52.6	65.3	42.5	297
As	<.139	<.183	<.122	<.103	.161	<.131	<.132	.190	<.161	.259
Ba	<.139	<.183	.215	.200	3.75	.419	2.55	5.11	2.96	4.60
Cd	.046	.260	<.025	<.021	.037	<.026	.032	.151	.106	.024
Cr	<.139	<.183	1.39	.629	2.02	.181	1.85	2.96	5.47	1.65
Cu	.660	43.0	.181	.132	.854	.289	.398	1.07	1.15	.721
Fe	81.9	993	15.0	9.83	197	18.4	70.0	82.7	108	439
Hg	.045	.347	.326	.157	.190	.417	.101	.137	.101	.045
Mg	172	132	382	288	415	431	623	735	505	315
Mn	1.07	1.13	.989	1.10	27.9	3.26	76.6	40.5	19.8	110
Ni	<.139	<.183	.397	.322	.912	.489	.988	.288	.246	.983
Pb	<.278	<.366	<.245	<.206	<.299	<.261	.339	.365	<.323	.321
Se	.674	11.6	.275	.310	.247	.245	.364	.438	.370	<.107
Sr	<.056	<.073	2.82	.998	4.8	2.06	10.4	13.7	7.33	1.96
V	<.139	.321	<.122	<.103	.198	<.131	.182	.319	<.161	.484
Zn	27.1	36.6	12.1	8.84	15.9	11.1	38.4	86.3	90.1	14.8

* Samples Collected from Duck River Unit

Table 10. Metals Analyses of 1997 Sediment Samples Collected from Tennessee NWR.

Sample Number	Location	Weight (g)	% Moisture	% TOC	% Sand	% Silt	% Clay
97SED1	Eagle Creek	206	39.8	2.28	47.2	49.8	.230
97SED2	Duck River	318	29.9	2.16	74.2	25.9	.180
97SED3	Goodin Branch	203	39.6	4.16	52.0	40.0	.190
97SED4	Hustburg Creek	346	26.3	1.06	63.3	29.1	.200
97SED5	Blue Creek	155	43.2	1.70	68.0	31.9	.320

Table 11. Metal Concentrations (ppm, dw) in 1997 Sediment Samples from Tennessee NWR.

Sample Number	97SED1 Eagle Creek	97SED2 Duck River	97SED3 Goodin Branch	97SED4 Hustburg Creek	97SED5 Blue Creek
Al	6076	8745	5943	4513	14130
As	4.68	3.34	12.4	2.59	3.76
B	5.40	7.90	10.9	3.00	9.71
Ba	91.6	92.1	90.1	78.9	121
Be	.736	.847	.838	.985	1.03
Cd	.444	.610	.704	.451	.940
Cr	9.57	12.9	31.9	13.4	17.8
Cu	6.83	7.46	14.6	5.99	14.9
Fe	12200	15310	22560	8243	24540
Hg	<.098	<.102	<.101	<.101	.113
Mg	443	653	305	256	2036
Mn	678	867	603	153	903
Mo	<4.88	<5.09	19.4	<5.05	<5.03
Ni	13.2	14.7	12.8	13.8	17.5
Pb	10.2	11.1	16.4	9.01	14.1
Se	.670	.860	<.507	<.505	.720
Sr	8.00	41.1	9.76	4.60	9.26
V	19.6	15.4	44.1	20.8	23.4
Zn	31.2	46.6	63.0	30.9	75.2

Table 12. Organic Analyses of 1997 Wood Duck Egg Samples Collected at Duck River Unit. Chlorinated Hydrocarbon Insecticides Detected (ppm, ww).

Sample Number	Weight (g)	Moisture (%)	Lipids (%)	p,p'-DDE	p,p'-DDD
97WD1	87.5	70.0	14.4	<.010	<.010
97WD2	73.7	69.5	14.6	.017	<.010
97WD3	137	65.5	14.9	<.010	<.010
97WD4	348	69.5	15.1	.020	<.010
97WD5	154	68.5	17.1	.023	<.010

Table 13. Metal Concentrations (ppm, ww) in 1997 Wood Duck Eggs from Duck River Unit.

Sample Number	97WD1	97WD2	97WD3	97WD4	97WD5
Al	2.41	2.67	2.25	1.79	1.83
Ba	1.27	1.72	3.40	2.11	1.76
Cr	1.30	<.175	<.157	<.154	<.160
Cu	.696	1.02	1.19	1.01	1.06
Fe	36.4	38.4	29.7	28.6	34.5
Hg	.037	.045	.037	.046	.07
Mg	114	111	104	113	114
Mn	1.15	1.19	2.46	2.05	1.92
Se	.365	.370	.356	.354	.401
Sr	.624	.777	1.17	.742	.701
Zn	15.4	17.5	14.6	14.5	14.1
Weight (g)	53.6	39.6	44.5	40.3	39.0
Moisture (%)	69.3	65.5	69.3	69.2	68.4

Table 14. Organic Analyses of 1998 Fish Samples Collected at the Big Sandy Unit. Chlorinated Hydrocarbon Insecticides Detected (ppm, ww).

Sample Number	Weight (g)	Moisture (%)	Lipids (%)	p,p'-DDE	p,p'-DDD
98BG	39.6	89.1	4.13	.016	<.010
98ES	50.6	70.9	5.82	.110	.016
98GS	66.1	74.1	4.76	<.010	<.010
98LES1	102	75.7	3.26	.035	<.010
98LMB1	89.0	73.9	.942	.019	<.010
98SMB	259	79.8	1.03	.012	<.010
99Drum	184	73.7	7.12	<.010	<.010
99LMB	133	76.0	1.00	.019	<.010
99SS1	541	70.2	10.2	.010	<.010
99SS2	420	43.3	7.08	<.010	<.010

Table 15. Metal Analyses of 1998 Fish Samples Collected at the Big Sandy Unit.

Sample Number	Species	Sample Matrix	Weight (g)	Moisture (%)
98BG	Bluegill	Whole-body	17.5	73.1
98ES	Emerald shiner	Whole-body	20.2	73.1
98GS	Gizzard shad	Whole-body	28.0	74.5
98LES1	Longear sunfish	Whole-body	49.0	72.0
98LMB1	Largemouth Bass	Whole-body	38.0	73.1
98SMB	Smallmouth Bass	Whole-body	60.0	78.5
99Drum	Freshwater Drum	Whole-body	10.8	73.7
99LMB	Largemouth Bass	Whole-body	10.2	77.0
99SS1	Spotted shiner	Whole-body	30.7	69.5
99SS2	Spotted Shiner	Whole-body	18.0	72.7

Table 16. Metal Concentrations in 1998 Fish Samples Collected at the Big Sandy Unit (ppm, ww).

Sample Number	98BG	98ES	98GS	98LES1	98LMB1	98SMB	99Drum	99LMB	99SS1	99SS2
Al	26.4	10.6	439	9.05	9.15	8.57	30.5	10.8	12.9	13.7
Ba	1.82	3.18	5.58	1.31	.540	.610	5.15	.625	2.91	2.69
Cd	<.049	.055	.066	<.054	<.051	.124	<.051	.073	<.058	.061
Cr	3.09	.741	3.42	1.34	2.75	2.02	2.10	1.58	.626	.645
Cu	.406	.461	1.11	.320	.580	.739	.529	.439	.740	.791
Fe	50.4	20.4	478	16.5	20.7	23.0	49.7	19.8	24.5	21.7
Hg	.040	.074	.010	.072	.169	.384	.061	.125	.044	.034
Mg	12.2	7.35	59.3	8.87	4.92	.677	46.8	2.98	44.1	47.4
Mn	<.245	<.260	<.242	<.271	<.255	.235	<.255	<.215	<.288	<.264
Ni	.961	<.260	1.05	.333	.998	1.07	.542	.773	<.288	<.264
Pb	<.245	<.260	.416	.387	.402	.293	.324	.225	<.288	<.264
Se	.390	.460	.377	.386	.484	.338	.702	.368	.296	.489
Sr	12.9	12.5	7.29	13.3	15.3	4.30	14.8	13.5	22.7	19.4
V	<.245	<.260	.774	<.271	<.255	<.201	<.255	<.215	<.288	<.264
Zn	32.5	56.5	17.5	32.7	26.9	17.8	14.3	18.0	17.1	13.5

Table 17. Metal Analyses of 1998 Sediment Samples in the Big Sandy and Busseltown Units.

Sample Number	Location	Weight (g)	% Moisture	% TOC	% Sand	% Silt	% Clay
98SED1	Busseltown	326	42.8	1.86	66.3	31.2	.240
98SED2	Busseltown	300	46.6	2.16	70.3	21.7	.210
98SED3	Big Sandy	332	33.4	.640	19.7	80.0	.360
98SED4	Big Sandy	357	35.7	.660	42.2	53.9	.290

Table 18. Metal Concentrations (ppm, dw) in 1998 Sediment Samples from the Busseltown and Big Sandy Units.

Sample Number	Busseltown n 98SED1	Busseltown 98SED2	Big Sandy 98SED3	Big Sandy 98SED4
Al	14260	16410	5813	4890
As	2.85	3.67	1.23	3.07
B	12.2	15.0	4.68	5.42
Ba	89.0	132	46.4	45.6
Be	.857	1.17	.396	.411
Cd	.501	.473	<.204	<.202
Cr	19.8	21.4	8.71	9.51
Cu	15.3	16.1	6.27	6.16
Fe	20810	26610	6414	8729
Hg	.059	.092	.047	.046
Mg	2563	2234	526	516
Mn	399	1036	151	388
Mo	<5.08	<5.01	<5.09	<5.04
Ni	17.5	20.0	7.86	7.46
Pb	8.47	10.6	7.03	5.40
Se	<.508	.660	<.509	<.504
Sr	9.20	11.2	6.19	5.86
V	28.1	27.1	13.3	13.6
Zn	64.6	79.9	25.3	23.6

Summary of Results

Table 19. Organochlorine Constituents Found in Composite Whole-body Fish Samples from NWRs in Tennessee.

	alpha		gamma		p,p'		p,p'		DDT	Dieldrin	HCB	trans		cis		PCBs	Toxaphene
	Chlordane	Chlordane	DDD	DDD	DDE	DDE	Nonachlor	Nonachlor									
Tennessee NWR																	
<i>Duck River Unit (1996)</i>																	
Bluegill					x												
Gizzard shad					x												
Bussetown Unit (1997)																	
Bigmouth Buffalo					x												
Bowfin					x												
Bluegill					x												
Gizzard shad					x												
Big Sandy Unit (1998)																	
Bluegill					x												
Emerald shiner					x												
Longear sunfish					x												
Largemouth Bass (2)					x												
Smallmouth Bass					x												
Spotted Sucker					x												
Reelfoot NWR (1989)																	
Carp (2)*					x					x							x

Table 19 (continued).

Reelfoot NWR (1989)	alpha	gamma	p,p'	p,p'	DDT	Dieldrin	HCB	trans	cis	PCBs	Toxaphene
	Chlordane	Chlordane	DDD	DDE	DDT	Dieldrin	HCB	Nonachlor	Nonachlor	PCBs	Toxaphene
Channel catfish (2)			x	x		x					
White crappie (1)			x	x		x					
Gar** (2)	x		x	x		x		x			x
Yellow bass (2)			x	x		x					
Lake Isom NWR (1988)											
Spotted gar (2)			x	x							
Smallmouth buffalo (2)			x	x							
Chickasaw NWR (1988)											
<i>Wardlows Pocket</i>											
Gar*** (1)	x	x	x	x		x		x	x	x	
Bigmouth buffalo (1)			x	x							
<i>Chisolm Lake</i>											
Spotted gar (1)			x	x						x	
Smallmouth buffalo (1)			x	x						x	
Lower Hatchie NWR											
<i>Champion Lake (1988)</i>											
Spotted gar (1)			x	x				x		x	
Bowfin (2)			x	x							
Sunk Lake (1990)											
Gar** (2)	x		x	x		x		x			

Table 19 (continued).

	alpha Chlordane	gamma Chlordane	p,p' DDD	p,p' DDE	DDT	HCB	trans Nonachlor	cis Nonachlor	PCBs	Toxaphene
Sunk Lake (1990)										
Gizzard shad (2)				x						
Quillback (1)				x						
Hatchie NWR (1988)										
<i>Powell Lake</i>										
Spotted gar (1)			x	x	x					
Spotted gar (1)			x	x						
Bowfin (1)			x	x	x					
Cross Creeks NWR (1988)										
Channel catfish (3)										
<i>Pool 2</i>										
<i>Lower Pool 4</i>				x					x	
<i>Upper Pool 4</i>				x					x	
Blue catfish (2)				x					x	
Elk Reservoir										
<i>Pool 8</i>										
				x					x	
				x					x	

* Number of composite samples. ** Species not determined. *** Composite sample of spotted and shortnose gar.

Table 20. Organochlorine Concentrations (ppm, ww) in Composite Whole-body Fish Samples from NWRs in Tennessee.

	alpha		p,p'		DDT	Dieldrin	HCB	trans	cis	PCBs	Toxaphene
	Chlordane	gamma	DDD	DDE							
Tennessee NWR											
<i>Duck River Unit (1996)</i>											
Bluegill			<0.01	0.04							
Gizzard shad			<0.01	0.02							
<i>Busselton Unit (1997)</i>											
Bigmouth Buffalo			0.01	0.58							
Bowfin			<.01	0.07							
Bluegill			<.01	0.03							
Gizzard shad			<.01	0.01							
<i>Big Sandy Unit (1998)</i>											
Bluegill			<.01	0.02							
Emerald shiner			0.11	0.11							
Longear sunfish			<.01	0.04							
Largemouth Bass (2)			<.01	0.02							
Smallmouth Bass			<.01	0.01							
Spotted Sucker			<.01	0.01							
Reelfoot NWR (1989)											
Carp	<0.01	<0.01	<0.01	0.1	<0.01	0.03	<0.01	<0.01	<0.01	<0.05	0.21
Carp	<0.01	<0.01	<0.01	0.06	<0.01	0.03	<0.01	<0.01	<0.01	<0.05	0.05
Channel catfish	<0.01	<0.01	<0.01	0.02	<0.01	0.03	<0.01	<0.01	<0.01	<0.05	<0.05
Channel catfish	<0.01	<0.01	<0.01	0.03	<0.01	0.03	<0.01	<0.01	<0.01	<0.05	<0.05

Table 20 (continued).

Reelfoot NWR (1989)	alpha	gamma	p,p' p,p'		Dieldrin	HCB	trans Nonachlor	cis Nonachlor	PCBs	Toxaphene
	Chlordane	Chlordane	DDD	DDE						
White crappie	<0.01	<0.01	<0.01	0.02	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Gar*	0.02	<0.01	<0.01	0.26	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Gar*	<0.01	<0.01	0.06	1.3	<0.01	<0.01	0.03	<0.01	<0.05	0.3
Yellow bass	<0.01	<0.01	<0.01	0.03	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Yellow bass	<0.01	<0.01	<0.01	0.06	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Lake Isom NWR (1988)										
Spotted gar	<0.01	<0.01	0.02	0.12	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Spotted gar	<0.01	<0.01	0.02	0.23	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Smallmouth buffalo	<0.01	<0.01	0.02	0.08	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Smallmouth buffalo	<0.01	<0.01	0.02	0.08	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05
Chickasaw NWR (1988)										
<i>Wardlows Pocket</i>										
Gar**	0.03	0.01	0.03	0.18	<0.01	<0.01	0.01	0.02	0.71	<0.05
Bigmouth buffalo	<0.01	<0.01	<0.01	0.06	<0.01	<0.01	<0.01	<0.01	0.42	<0.05
<i>Chisolm Lake</i>										
Spotted gar	<0.01	<0.01	<0.01	0.06	<0.01	<0.01	<0.01	<0.01	0.24	<0.05
Smallmouth buffalo	<0.01	<0.01	<0.01	0.11	<0.01	<0.01	<0.01	<0.01	0.45	<0.05
Lower Hatchie NWR										
<i>Champion Lake (1988)</i>										
Spotted gar	<0.01	<0.01	0.04	0.19	<0.01	<0.01	<0.01	<0.01	0.51	<0.05
Bowfin	<0.01	<0.01	<0.01	0.05	<0.01	<0.01	<0.01	<0.01	<0.05	<0.05

Table 20 (continued).

Lower Hatchie NWR	alpha		p,p'		Dieldrin	HC	trans		PCB	Toxaphene
	Chlordane	gamma	DDD	DDE			Nonachlor	cis		
Sunk Lake (1990)										
Gar*	0.01	<0.01	0.03	0.14	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05
Gar	0.02	<0.01	0.04	0.18	<0.01	0.01	<0.01	<0.01	<0.01	<0.05
Gizzard shad	<0.01	<0.01	<0.01	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05
Gizzard Shad	<0.01	<0.01	<0.01	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05
Quillback	<0.01	<0.01	<0.01	0.04	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05
Hatchie NWR (1988)										
<i>Powell Lake</i>										
Spotted gar	<0.01	<0.01	0.03	0.2	0.04	<0.01	<0.01	<0.01	<0.01	<0.05
Spotted sucker	<0.01	<0.01	0.05	0.15	0.01	0.01	<0.01	<0.01	<0.01	<0.05
Bowfin	<0.01	<0.01	0.01	0.05	0.01	0.01	<0.01	<0.01	<0.01	<0.05
<i>Kelso Lake</i>										
Spotted gar	<0.01	<0.01	0.02	0.14	<0.05	<0.01	<0.01	<0.01	<0.01	<0.05
Bowfin	<0.01	<0.01	0.01	0.07	0.01	<0.01	<0.01	<0.01	<0.01	<0.05
Cross Creeks NWR (1988)										
<i>Channel catfish</i>										
<i>Pool 2</i>	<0.01	<0.01	<0.01	0.12	<0.01	<0.01	<0.01	<0.01	<0.01	0.39
<i>Lower Pool 4</i>	<0.01	<0.01	<0.01	0.15	<0.01	<0.01	<0.01	<0.01	<0.01	0.82
<i>Upper Pool 4</i>	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05
<i>Blue catfish</i>										
<i>Elk Reservoir</i>	<0.01	<0.01	<0.01	0.1	<0.01	<0.01	<0.01	<0.01	<0.01	0.17
<i>Pool 8</i>	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.05

* Species not determined.

** Composite sample of spotted and shortnose gar specimens.

Table 21. Metals Detected in Composite Whole-Body Fish Samples from NWRs in Tennessee.

	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn
Tennessee NWR																		
<i>Busselton Unit (1997)</i>																		
Bigmouth buffalo	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Bowfin	X		X		X	X	X	X		X	X	X	X	X	X			X
Bluegill	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Common carp1	X	X	X		X	X	X	X	X	X	X	X	X	X	X			X
Common carp2	X		X		X	X	X	X		X	X	X	X	X	X			X
Gizzard shad	X	X	X		X	X	X	X	X	X	X	X	X		X			X
<i>Big Sandy Unit (1998)</i>																		
Bluegill	X		X		X	X	X	X		X	X	X	X	X	X			X
Emerald Shiner	X		X		X	X	X	X		X	X	X	X	X	X			X
Gizzard shad	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Longear sunfish	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Largemouth bass1	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Largemouth bass99	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Smallmouth bass	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Drum	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Spotted sucker1	X		X		X	X	X	X		X	X	X	X	X	X			X
Spotted sucker2	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Reelfoot NWR (1989)																		
Carp (2)*	X		X		X	X	X	X	X	X	X	X	X	X	X			X
Channel catfish (2)	X		X		X	X	X	X	X	X	X	X	X	X	X			X

Table 21 (continued).

	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn	
Reelfoot NWR (1989)																			
White crappie (1)			X				X	X		X	X	X		X	X				X
Gar** (2)			X				X	X		X	X	X		X	X	X			X
Yellow bass (2)			X				X	X		X	X	X		X	X				X
Lake Isom NWR (1988)																			
Spotted gar (2)	X			X	X	X	X	X		X	X	X	X	X					X
Smallmouth buffalo (2)	X	X		X	X	X	X	X		X	X	X	X	X					X
Chickasaw NWR (1988)																			
<i>Wardlows Pocket</i>																			
Gar*** (1)	X					X	X	X		X	X	X	X	X					X
Bigmouth buffalo (1)	X	X			X	X	X	X		X	X	X	X	X					X
<i>Chisolm Lake</i>																			
Spotted gar (1)	X	X		X	X	X	X	X	X	X	X	X	X	X					X
Smallmouth buffalo (1)	X	X			X	X	X	X		X	X	X	X	X					X
Lower Hatchie NWR																			
<i>Champion Lake (1988)</i>																			
Spotted gar (1)	X		X	X	X	X	X	X		X	X	X	X	X					X
Bowfin (1)	X	X		X	X	X	X	X		X	X	X	X	X					X
Sunk Lake (1990)																			
Gar** (2)	X	X			X	X	X	X		X	X	X	X	X	X				X
Gizzard shad (2)	X	X			X	X	X	X		X	X	X	X	X	X				X
Quillback (1)	X	X			X	X	X	X		X	X	X	X	X	X				X

Table 21 (continued).

	Al	As	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn	
Hatchie NWR (1988)																			
<i>Powell Lake</i>																			
Spotted gar (1)	x					x	x	x		x	x	x	x	x					x
Spotted sucker (1)	x				x	x	x	x	x	x	x	x	x	x					x
Bowfin (1)		x			x	x	x	x		x	x	x	x	x					x
<i>Kelso Lake</i>																			
Spotted gar (1)	x				x	x	x	x	x	x	x	x	x	x					x
Bowfin (1)	x	x			x	x	x	x		x	x	x	x	x					x
Cross Creeks NWR (1988)																			
Channel catfish (3)																			
<i>Pool 2</i>																			
							x												
<i>Lower Pool 4</i>																			
<i>Upper Pool 4</i>																			
Blue catfish (2)																			
<i>Elk Reservoir</i>																			
<i>Pool 8</i>																			

* Number of composite samples. ** Species not determined. *** Composite sample of spotted and shortnose gar specimens.

Table 22. Metal Concentrations (ppm, ww) in Composite Whole-Body Fish Samples from NWRs in Tennessee.

	Al	As	B	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn
Tennessee NWR																			
<i>Busselton Unit (1997)</i>																			
Bigmouth buffalo	0.11	0.16		3.75		0.04	2.02	0.85	197	<0.30	415	27.9	0.19	0.91	0.25	4.80		0.20	15.9
Bowfin	8.46	<0.13		0.42		<0.03	0.18	0.29	18.4	<0.26	431	3.26	0.42	0.49	0.25	2.06		<0.13	11.1
Bluegill	52.6	<0.13		2.55		0.03	1.85	0.40	70.0	0.34	623	76.6	0.10	0.99	0.36	10.4		0.18	38.4
Common carp1	65.3	0.19		5.11		0.15	2.96	1.07	82.7	0.37	735	40.5	0.14	0.29	0.44	13.7		0.32	86.3
Common carp2	42.5	<0.16		2.96		0.11	5.47	1.15	108	<0.32	505	19.8	0.10	0.25	0.37	7.33		<0.16	90.1
Gizzard shad	297	0.26		4.60		0.02	1.65	0.72	439	0.32	315	110	0.05	0.98	<0.11	1.96		0.48	14.8
Big Sandy Unit (1998)																			
Bluegill	26.4	<0.91		1.82		<0.05	3.09	0.41	50.4	<0.25	12.2	<0.25	0.04	0.96	0.39	12.9		<0.25	32.5
Emerald Shiner	10.6	<0.97		3.18		0.06	0.74	0.46	20.4	<0.26	7.35	<0.26	0.07	<0.26	0.46	12.5		<0.26	56.5
Gizzard shad	439	<0.95		5.58		0.07	3.42	1.11	478	0.42	59.3	<0.24	0.01	1.05	0.38	7.29		0.77	17.5
Longear sunfish	9.05	<0.97		1.31		<0.05	1.34	0.32	16.5	0.39	8.87	<0.27	0.07	0.33	0.39	13.3		<0.27	32.7
Largemouth bass1	9.15	<0.95		0.54		<0.05	2.75	0.58	20.7	0.40	4.92	<0.26	0.17	1.00	0.48	15.3		<0.26	26.9
Largemouth bass99	10.8	<0.94		0.63		0.07	1.58	0.44	19.8	0.23	2.98	<0.22	0.13	0.77	0.37	13.5		<0.22	18.0
Smallmouth bass	8.57	<0.93		0.61		0.12	2.02	0.74	23.0	0.29	0.68	0.24	0.38	1.07	0.34	4.30		<0.20	17.8
Drum	30.5	<0.97		5.15		<0.05	2.10	0.53	49.7	0.32	46.8	<0.26	0.06	0.54	0.70	14.8		<0.26	14.3
Spotted sucker1	12.9	<0.94		2.91		<0.06	0.63	0.74	24.5	<0.29	44.1	<0.29	0.04	<0.29	0.30	22.7		<0.29	17.1
Spotted sucker2	13.9	<0.97		2.69		0.06	0.65	0.79	21.7	<0.26	47.4	<0.26	0.03	<0.26	0.49	19.4		<0.26	13.5
Reelfoot NWR (1989)																			
Carp	16.0	<0.09		1.80				0.96	81.0		276	13.9	0.08		0.44	5.2	<14		69
Carp		<0.11		3.12				<0.74	50.0		337	8.4	0.05		0.34	12.8	<13		86
Channel catfish	9.3	<0.08		2.74				0.8	51		289	42	0.02		0.31	5.6	<14		15.8
Channel catfish	6.6	<0.08		<0.57				0.72	46		271	7	0.01		0.219	3.7	<14		17.1

Table 22 (continued).

Reelfoot NWR (1989)	Al	As	B	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn
White crappie	<5	<0.07		1.48				0.49	16.5		362	4.5	0.03		0.203	15.5	<15		17.7
Gar*	<8	<0.12		1.77				<0.8	47		2750	14.9	0.1		0.231	26.4	19.2		26.2
Gar*	<7	<0.11		2.11				0.89	32.5		2741	4.7	0.17		0.269	25.7	13.6		20.6
Yellow bass	<5	<0.07		2.27				2.26	24.4		417	13.4	0.01		0.366	13.5	<15		25.4
Yellow bass	<5	<0.08		2.38				3.35	38.8		406	16.3	0.01		0.393	12.5	<15		25.8
Lake Isom NWR (1988)																			
Spotted gar	6					0.02	2.6	0.6	42			40	0.04	1.3	0.23				18.1
Spotted gar	24.6				0.01		6.9	0.4	76			37	0.50	3.2	0.2				21.4
Smallmouth buffalo	13.9					0.01	0.17	1.39	91			16.1	0.11	0.1	0.23				19.3
Smallmouth buffalo	58	0.1			0.01		0.1	0.91	112			29.9	0.08	0.1	0.22				18.6
Chickasaw NWR (1988)																			
<i>Wardlows Pocket</i>																			
Gar**	8						7	0.6	62			14	0.10	3.2	0.4				22.4
Bigmouth buffalo	28.9	0.2				0.02	0.23	0.98	46			8.8	0.03	0.1	0.4				18.6
<i>Chisolm Lake</i>																			
Spotted gar	7	1.9			0.01	0.02	3	0.7	42	118		30	0.20	1.5	0.29				23.8
Smallmouth buffalo	64	0.1				0.02	0.4	0.69	88			31	0.09	0.3	0.4				15.9
Lower Hatchie NWR																			
<i>Champion Lake (1988)</i>																			
Spotted gar	10						1.9	0.5	44			57	0.22	1.3	0.29				19
Bowfin	9	0.1					0.09	0.68	60			8.5	0.7	0.21	0.4				12.4

Table 22 (continued).

	Al	As	B	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Hg	Ni	Se	Sr	Sn	V	Zn
Lower Hatchie NWR																			
<i>Sunk Lake (1990)</i>																			
Gar*	12	0.1		4			5	0.9	64		2573	26.4	0.25	2.5	0.29	27.3			21.5
Gar*	9		3.3			2.8	0.7	54			1992	20.5	0.27	1	0.29	22.2			19.3
Gizzard shad	62	0.1		3.3				0.7	147		304	29	0.05		0.4	5.1		0.2	14.1
Gizzard shad	76	0.2		2.9				0.7	120		304	34	0.07		0.3	5.9		0.2	14.6
Quillback	46	0.2		3			1	0.6	88		345	9.3	0.03		0.4	9.9			12.7
Hatchie NWR (1988)																			
<i>Powell Lake</i>																			
Spotted gar	10					0.02	5.3	0.8	57	0.3		156	0.5	2.5	0.29				20.2
Spotted sucker	7					0.01		0.87	53			22.5	0.22	0.1	0.4				17.3
Bowfin	15.2	0.19				0.01	0.18	1.14	70			13.3	0.4	0.14	0.3				11.3
Kelso Lake																			
Spotted gar	13					0.02	3	0.5	49	0.3		117	0.4	1.7	0.27				18.4
Bowfin	36	0.17				0.02	0.3	0.82	80			10.4	0.4	0.3	0.3				12.5
Cross Creeks NWR (1988)																			
Channel catfish																			
<i>Pool 2</i>	333		0.6	2.9		0	0.45	0.38	247	0.49	318	35	0.08	0.67	0.34	2.9		0.62	16.2
<i>Lower Pool 2</i>	98		0.33	1.08			0.24	0.2	128	0.41	307	15.5	0.07	0.26	0.35	1.46		0.25	13.8
<i>Upper Pool 4</i>	125	0.06	0.6	1.03		0.2	0.3	0.37	125	0.56	288	17	0.06	0.3	0.4	0.91		0.29	17
Blue catfish																			
<i>Elk Reservoir</i>	17.7		0.27	0.2		0	0.1	0.22	37	0.43	256	1.92	0.06		0.29	0.27		0.06	7.8
<i>Pool 8</i>	37		0.1	0.5			0.45	0.34	49		318	4.5	0.03		0.2	4.6		0.13	14.8

* Species not determined. ** Composite sample of spotted and shortnose gar specimens.

Table 23. Metals Detected in Sediment Samples From Tennessee NWRs.

	Sb	Al	As	B	Ba	Be	Cd	Co	Cr	Cu	Fe	Pb	Mg	Mo	Mn	Hg	Ni	Se	Ag	Sr	Tl	V	Zn
Tennessee NWR																							
<i>Duck River Unit (1996)</i>																							
Pool 1	NA	x	x	x	x	x	x	NA	x	x	x	x	x	x	x	ND	x	x	NA	x	NA	x	x
Pool2	NA	x	x	x	x	x	x	NA	x	x	x	x	x	x	x	x	x	x	NA	x	NA	x	x
Pool10	NA	x	x	x	x	x	x	NA	x	x	x	x	x	x	x	ND	x	x	NA	x	NA	x	x
Pool6	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	ND	x	x	NA	x	NA	x	x
Pool9	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	ND	x	ND	NA	x	NA	x	x
1997																							
Sed1-Eagle Creek	NA	x	x	x	x	x	x	NA	x	x	x	x	x	ND	x	ND	x	x	NA	x	NA	x	x
Sed2-Duck River	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	ND	x	x	NA	x	NA	x	x
Sed3-Goodin Branch	NA	x	x	x	x	x	x	NA	x	x	x	x	x	x	x	ND	x	ND	NA	x	NA	x	x
Sed4-Hustburg Creek	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	ND	x	ND	NA	x	NA	x	x
Sed5-Blue Creek	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	ND	x	ND	NA	x	NA	x	x
1998																							
Sed1 Busseltown Unit	NA	x	x	x	x	x	x	NA	x	x	x	x	x	ND	x	x	x	ND	NA	x	NA	x	x
Sed2 Busseltown Unit	NA	x	x	x	x	x	x	NA	x	x	x	x	ND	x	x	x	x	x	NA	x	NA	x	x
Sed3 Big Sandy Unit	NA	x	x	x	x	x	ND	NA	x	x	x	x	ND	x	x	x	x	ND	NA	x	NA	x	x
Sed4 Big Sandy Unit	NA	x	x	x	x	x	ND	NA	x	x	x	x	ND	x	x	x	x	ND	NA	x	NA	x	x

Table 23 (continued).

	Sb	Al	As	B	Ba	Be	Cd	Co	Cr	Cu	Fe	Pb	Mg	Mo	Mn	Hg	Ni	Se	Ag	Sr	Tl	V	Zn
Reelfoot NWR (1988)*																							
Donaldson Ditch	NA	ND	x	ND	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	NA	x	NA	x	x
Samburg	NA	ND	x	ND	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	NA	x	NA	x	x
Horse Island Ditch	NA	ND	x	ND	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	NA	x	NA	x	x
Buck Basin	NA	ND	x	ND	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	NA	x	NA	x	x
Upper Blue Basin	NA	ND	x	ND	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	NA	x	NA	x	x
Lower Hatchie NWR																							
<i>Sunk Lake (1990)</i>																							
NA	x	x	x	x	x	x	x	NA	x	x	x	x	x	NA	x	x	x	x	x	x	ND	x	x
Cross Creeks NWR																							
<i>Pool 2</i>																							
ND	x	x	x	x	x	x	x	x	x	x	x	x	x	NA	x	ND	x	x	ND	x	NA	x	x
<i>Lower Pool 4</i>																							
ND	x	x	x	x	x	x	x	x	x	x	x	x	x	NA	x	x	x	x	ND	x	NA	x	x
<i>Upper Pool 4</i>																							
ND	x	x	x	x	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	ND	x	NA	x	x
<i>Elk Reservoir</i>																							
ND	x	x	x	x	x	x	ND	x	x	x	x	x	x	NA	x	x	x	x	ND	x	NA	x	x
<i>Pool 8</i>																							
ND	x	x	x	x	x	x	ND	x	x	x	x	x	x	NA	x	x	x	ND	ND	x	NA	x	x

* based on results reported by Broshears (1991) for upper layer core samples.

NA-Not analyzed. ND-Not detected.

Table 24. Metal Concentrations (ppm, dw) in Sediment Samples From Tennessee NWRs.

	Al	As	B	Ba	Be	Cd	Cr	Cu	Fe	Pb	Mg	Mn	Mo	Hg	Ni	Se	Sr	V	Zn
Tennessee NWR																			
<i>Duck River Unit (1996)</i>																			
Pool 1	27293	3.82	3.41	209	1.42	0.34	24.0	16.1	29621	22.0	2170	2590	1.77	ND	25.4	0.55	46.9	37.9	85.3
Pool2	16818	3.33	3.66	121	0.92	0.32	16.8	11.9	19312	15.9	1643	1787	1.72	0.08	17.0	0.34	29.1	23.8	59.6
Pool10	20339	2.95	4.47	155	1.18	0.38	19.9	13.4	22039	18.0	1885	1364	2.21	ND	20.6	0.17	32.7	28.1	70.9
Pool6	24656	3.54	4.03	184	1.07	0.44	20.5	13.6	24608	19.5	1853	2164	ND	ND	21.4	0.23	40.0	33.8	72.8
Pool9	20453	3.88	4.22	157	0.95	0.04	17.6	11.4	22905	19.3	1595	2195	ND	ND	18.9	ND	37.5	28.3	63.0
Average **	21912	3.50	3.96	165	1.1	0.30	20	13	23697	18.9	1829	2020	1.90	0.08	20.7	0.32	37.2	30.4	70.3
1997																			
Sed1-Eagle Creek	6076	4.68	5.40	91.6	0.74	0.44	9.57	6.83	12200	10.2	443	678	ND	ND	13.2	0.67	8.00	19.6	31.2
Sed2-Duck River	8745	3.34	7.90	92.1	0.85	0.61	12.9	7.46	15310	11.1	653	867	ND	ND	14.7	0.86	41.1	15.4	46.6
Sed3-Goodin Branch	5943	12.4	10.9	90.1	0.84	0.70	31.9	14.6	22560	16.4	305	603	19.4	ND	12.8	ND	9.76	44.1	63.0
Sed4-Hustburg Creek	4513	2.59	3.00	78.9	0.99	0.45	13.4	5.99	8243	9.01	256	153	ND	ND	13.8	ND	4.60	20.8	30.9
Sed5-Blue Creek	14130	3.76	9.71	121	1.03	0.94	17.8	14.9	24540	14.1	2036	903	ND	0.11	17.5	0.72	9.26	23.4	75.2
1998																			
Sed1 Busseltown Unit	14260	2.85	12.2	89.0	0.86	0.50	19.8	15.3	20810	8.47	2563	399	ND	0.06	17.5	ND	9.20	28.1	64.6
Sed2 Busseltown Unit	16410	3.67	15.0	132	1.17	0.47	21.4	16.1	26610	10.6	2234	1036	ND	0.09	20.0	0.66	11.2	27.1	79.9
Sed3 Big Sandy Unit	5813	1.23	4.68	46.4	0.40	ND	8.71	6.27	6414	7.03	526	151	ND	0.05	7.86	ND	6.19	13.3	25.3
Sed4 Big Sandy Unit	4890	3.07	5.42	45.6	0.41	ND	9.51	6.16	8729	5.4	516	388	ND	0.05	7.46	ND	5.86	13.6	23.6

Table 24. (continued)

	Sb	Al	As	B	Ba	Be	Cd	Co	Cr	Cu	Fe	Pb	Mg	Mn	Mo	Hg	Ni	Se	Ag	Sr	Tl	V	Zn
Reelfoot NWR (1988)*																							
Donaldson Ditch	NA	69000	5.80	NA	740	2.00	ND	14.0	64.0	23.0	NA	14.0	8300	600	ND	0.0	30.	0.7	NA	160	NA	110	83
Samburg	NA	64000	8.40	NA	660	2.00	ND	14.0	62.0	25.0	NA	19.0	9300	1400	ND	0.1	34.	0.4	NA	120	NA	95.	97.
Horse Island Ditch	NA	78000	11.0	NA	620	2.00	ND	18.0	80.0	32.0	NA	29.0	9200	1200	ND	0.1	41.	1.0	NA	99.	NA	130	120
Buck Basin	NA	90000	18.0	NA	660	2.00	ND	19.0	91.0	44.0	NA	28.0	10000	1600	ND	0.1	53.	1.0	NA	80.	NA	150	160
Upper Blue Basin	NA	70000	12.0	NA	580	2.00	ND	14.0	73.0	27.0	NA	21.0	8600	1200	ND	0.1	35.	1.2	NA	110	NA	120	110
Average**		74200	11.0		652	2.00		15.8	74.0	30.2		22.2	9080	1200		0.1	38.	0.8		114		121	114
Lower Hatchie NWR																							
Sunk Lake (1990)																							
Sediment 1	NA	58200	14.9	13.0	348	2.20	0.90	NA	44.0	35.8	56600	56.0	6800	1450	ND	0.0	47.	1.1	3.0	51.	ND	81.	160
Sediment 2	NA	56700	13.0	15.0	354	2.10	0.90	NA	32.0	34.3	55400	53.0	6600	1570	ND	0.1	42.	0.8	3.0	52.	ND	73.	153
Sediment 3	NA	46100	18.0	10.0	335	2.00	2.00	NA	46.0	36.0	47700	61.0	6340	1780	ND	0.1	50.	1.3	10.	52.	ND	78.	182
Sediment 4	NA	26300	8.40	6.00	247	1.30	0.60	NA	21.0	18.0	24800	28.0	4830	1040	ND	0.0	26.	0.4	6.0	39.	ND	47.	81.
Average**		46825	13.5	11	321	1.90	1.10		35.8	31.0	46125	49.5	6143	1460		0.0	41.	0.9	5.5	48.		70.	144

Table 24 (continued).

	Sb	Al	As	B	Ba	Be	Cd	Co	Cr	Cu	Fe	Pb	Mg	Mn	Mo	Hg	Ni	Se	Ag	Sr	Tl	V	Zn
Cross Creeks NWR																							
Pool 2	ND	7300	2.69	41.8	281	1.01	0.86	13.2	37.3	5.81	19800	26.2	836	870	ND	ND	22.6	0.47	ND	21.4	NA	62.0	69.0
Lower Pool 4	ND	7570	3.86	40.8	283	1.67	0.85	15.8	42.0	6.32	22600	36.1	735	1440	ND	0.07	27.5	0.49	ND	23.7	NA	68.0	77.2
Upper Pool 4	ND	8460	3.05	32	236	1.16	ND	11.4	32.6	5.91	17400	24.3	864	758	ND	0.05	20.9	0.36	ND	21.3	NA	50.0	65.1
Elk Reservoir	ND	6040	0.98	22.7	151	0.72	ND	8.40	21.6	3.04	22500	12.9	443	1100	ND	0.01	14.8	ND	ND	10.9	NA	36.0	43.2
Pool 8	ND	4250	3.97	27.6	154	0.93	ND	14.3	28.8	2.91	34200	31.4	30	2440	ND	0.05	18.4	0.47	ND	11.1	NA	48.0	50.6
Average**		6724	2.91	33	221	1.10	0.86	12.6	32.5	4.80	23300	26.2	582	1322		0.05	20.8	0.45		17.7		52.8	61.0

* based on results reported by Broshears (1991) for upper layer core samples.

NA-Not analyzed. ND-Not detected.

Table 25. Comparison of Maximum Sediment Metal Concentrations (ppm, dw) at Tennessee NWR and Average Sediment Concentrations at West Tennessee NWRS with Soil Values (geometric means) Reported by Shacklette and Boerngen(1984).

Metals	Eastern United States		Tennessee NWR			West Tennessee NWRs		
	33000	4.80	Duck River	Busseltown	Big Sandy	Reelfoot	Lower Hatchie	Cross Creeks
			27293	16410	5813	74200	46825	6724
Al	33000		27293	16410	5813	74200	46825	6724
As	4.80		3.88	12.40	3.07	11.0	13.6	2.91
B	31.0		4.47	15.00	5.42	NA	11.0	33.0
Ba	290		209	132.0	46.4	652	321	221
Be	0.55		1.42	1.17	0.41	2.00	1.90	1.10
Cr	33.0		24.0	31.9	9.5	74.0	35.8	32.5
Cu	13.0		16.1	16.10	6.3	30.2	31.0	4.80
Fe	14000		29621	26610	8729	NA	46125	23300
Hg	0.08		0.11	0.09	0.05	0.12	0.10	0.04
Mg	2100		2170	2563	526	9080	6143	582
Mn	300		2590	1036	388	1200	1460	1322
Mo	0.32		2.21	19.4	ND	ND	ND	ND
Ni	11.0		25.4	20.0	7.9	38.6	41.3	20.8
Pb	14.0		22.0	16.4	7.03	22.2	49.5	26.2
Se	0.30		0.72	0.66	ND	0.86	0.91	0.36
Sr	53.0		46.9	11.2	6.2	114.0	48.9	17.7
V	43.0		37.9	44.1	13.6	121.0	70.0	52.8
Zn	40.0		85.3	79.9	25.3	114.0	144	61.0

DISCUSSION

Schultz *et al.* (1986) collected largemouth bass and freshwater drum samples at Cross Creeks NWR in 1985. A total of six composite samples (three per species) was collected. Three chemicals (p,p'-DDT, dieldrin and oxychlorane) were each detected at low concentrations in one largemouth bass sample. Toxaphene was found in four samples (2 per species) at average concentrations of 0.23 ppm (drum) and 0.21 ppm (largemouth bass). Schultz *et al.* (1986) found trans-nonachlor in all samples at concentrations ranging from 0.01 to 0.08 ppm (wet weight).

In this study, no dieldrin, toxaphene, or chlordane isomer residues were detected in any sample collected on Tennessee NWR. Trans-nonachlor was only detected in the gonad and livers of bowfin collected at the Busseltown Unit.

Schultz *et al.* (1985) collected black crappie, bluegill, gizzard shad, and sediment samples at Reelfoot NWR in 1983. Low levels of α -BHC, p,p'-DDE, and p,p'-DDD were detected in their whole-body fish samples. While only one bluegill sample contained DDD (0.10 ppm, wet weight), nine of their 12 composite fish samples (75%) contained DDE at concentrations ranging from 0.007 ppm to 0.018 ppm (wet weight), with the highest concentration found in bluegill. Winger *et al.* (1988) reported results for 588 composite whole-body fish samples, representing 36 species, collected from 20 NWRs in the Southeast during 1980-1986. Samples from Cross Creeks, Tennessee, and Wheeler NWRs were collected as part of their survey. Total DDT-related residues ranged from about 150 ppm (mean, wet-weight) at Wheeler NWR to 0.01 ppm at Reelfoot NWR. Of the three NWRs sampled in Tennessee, mean DDT-related residues were highest (0.31 ppm) at Tennessee NWR. Previous results (Robison *et al.* 2000) for DDT-related compounds from NWRs in West Tennessee were generally lower.

No DDT isomers were detected in any of the filet samples collected for this study, however, low levels of p,p'-DDE were detected in all liver and gonad samples, and in all but two whole-body fish samples. One bigmouth buffalo at the Busseltown Unit had a p,p'-DDE level of 0.58 ppm (ww). This level exceeded USEPA (1980), Environment Canada (EC) (1999), and New York State (Newell *et al.*, 1987) guidelines for the protection of piscivorous species. Residues of p,p'-DDE were not detected in gizzard shad and drum collected at the Big Sandy Unit. Low levels of p,p'-DDD were prevalent in common carp and bowfin livers, as well as whole-body bigmouth buffalo and bluegill collected at the Busseltown Unit. No DDT-related residues were detected in any filet sample collected at the three units in this study.

These results suggest a continuation of the declining trends reported in our West Tennessee NWR investigation and the 1995 Biomonitoring of Environmental Status and Trends (BEST) program effort (USGS 2002). This is likely indicative of the continued weathering of these compounds in sediments at the refuge. Low levels of p,p'-DDE (0.017-0.023 μ g/g, ww) were detected in the

composite wood duck eggs collected at the Duck River Unit, but these levels were well below residues reported to be deleterious to reproductive success (Blus 1996).

Schultz *et al.* (1986) found PCBs in all samples at concentrations ranging from 0.08 to 0.50 ppm. Freshwater drum and largemouth bass had average PCB concentrations of 0.12 and 0.27 ppm, respectively. On average, PCBs results (Robison *et al.* 2000) from NWRs in West Tennessee were lower than those reported by Winger *et al.* (1988) for refuges in Tennessee. Since congener-specific PCB levels in some samples were evaluated in this study, comparisons of total congener-specific PCBs present in those samples were made to total PCB levels in samples from previous studies. No PCBs were detected in the wood duck eggs collected from the Duck River Unit.

None of the organochlorine results observed in our West Tennessee NWRs investigation or this study exceeded the maximum values reported by Schmitt *et al.* (1985, 1990) and the 1995 BEST program (USGS 2002), and most concentrations were below the geometric means calculated for previous National Contaminant Biomonitoring Program (NCBP) data collection efforts. With the exception of very low congener-specific levels in common carp filets collected at the Duck River Unit, no PCBs were detected in whole-body samples or filets from other species collected at the Duck River, Busseltown, and Big Sandy Units. The low levels detected in the carp samples would not be expected to cause toxicity to the species (Eisler 1986) or pose serious toxicity concerns in piscivorous predators (Niimi 1996).

Four of the 15 sediment samples (27%) collected by Schultz *et al.* (1985) contained DDE at concentrations ranging from 0.9 ppm to 11.0 ppm, wet weight. No organochlorines were detected in any of the sediment samples collected at the Duck River Unit, Busseltown Unit, or Big Sandy Unit. The very low detected levels of congener-specific PCBs in our SPMD dialysates suggest potential resuspension, due to hydraulic manipulations on the Tennessee River at Kentucky Dam, of sediment-borne contamination into the water column. These levels were well below recommended and existing water quality criteria for PCBs (NAS 1973).

Schultz *et al.* (1985) found four metals (Cu, Fe, Hg and Zn) in all 12 fish samples collected in 1983. They reported two additional metals (Pb and V) in gizzard shad. Most metal concentrations were higher in the West Tennessee NWRs study (Robison *et al.* 2000), however, no direct comparison between the same species is possible. Except for the 1989 gar samples, which were higher, Hg concentrations were similar in the 1983 and the 1989 fish samples. Average Hg values reported by Winger *et al.* (1988) varied from 1.41 ppm (wet weight) at Overflow NWR to 0.08 ppm at Reelfoot NWR. Their mean Hg residues were similar at Cross Creeks and Tennessee NWRs (0.12 and 0.16 ppm, respectively). Results for six metals (As, Cd, Cu, Pb, Se, Zn) in the West Tennessee NWRs study (Robison *et al.* 2000) typically did not exceed maximum values for NCBP samples reported by May and McKinney (1981), Lowe *et al.* 1985, Schmitt and Brumbaugh (1990), or from the 1995 BEST program (USGS 2002). Six of 34 Hg samples were between maximum values for 1984 NCBP samples and the 1980-81 NCBP

samples. Gar or bowfin comprised these samples from Lake Isom, Chickasaw, and Hatchie NWRs. In the present study, one freshwater drum collected at the Big Sandy Unit had a selenium concentration of 0.70 ppm (ww). This level exceeds whole-fish thresholds for Se toxicity and the protection of piscivorous wildlife (Lemly 1996).

Average Hg values at Tennessee NWR did not exceed mean values reported by Winger *et al.* (1988) for NWRs in Tennessee and the 1995 BEST samples ($<0.15 \mu\text{g/g}$) collected in the Tennessee River at Savannah, Tennessee, and the Cumberland River in Clarksville, Tennessee (USGS 2002). Although most of the Hg results observed for our samples were considered low, three of 20 samples (15%) fell between maximum values for 1984 NCBP samples (0.37 ppm) and the 1980-81 NCBP samples (0.77 ppm) reported by Schmitt and Brumbaugh (1990). Bowfin and largemouth bass comprised two of these samples from the Busseltown Unit. One smallmouth bass collected on the Big Sandy Unit had a mercury concentration of 0.384 ppm.

None of the other metal concentrations in fish samples from the present study exceeded the respective mean values reported from the 1995 BEST effort (USGS 2002) or published action levels for fish tissue. Although there is not enough data to perform a statistically-significant trend analyses of the Hg concentrations within similar species for the samples we collected during this investigation, our empirical observations indicate that mercury concentrations may be increasing in higher-trophic level organisms on the refuge. Geometric mean Hg concentrations at the NCBP stations sampled in 1995 were also generally higher than when those sites were last sampled in the mid-1980s. The maximum Hg concentration found ($0.45 \mu\text{g/g}$) in the 1995 BEST effort was in a largemouth bass collected from the Mississippi River at Memphis, Tennessee.

At the three refuges sampled in the West Tennessee NWRs study (Robison *et al.* 2000), average sediment concentrations for five metals (Be, Mg, Mn, V and Zn) were 2-3 times greater than the geometric means reported by Shacklette and Boerngen (1984) for soils in the eastern United States. An additional four metals (As, Ba, Pb and Se) were in this category at two of the three refuges samples. Chromium and Sr substantially exceeded the soil values of Shacklette and Boerngen (1984) only at Reelfoot NWR, while Fe did so only at Lower Hatchie NWR. Based on the Illinois stream sediment classification reported by Kelly and Hite (1984), Cr and Mn were considered elevated ($\geq 23 \text{ ppm}$ and $\geq 0.18\%$, respectively) at all three refuges sampled in the West Tennessee NWRs study (Robison *et al.* 2000). Arsenic ($\geq 11 \text{ ppm}$) and Zn ($\geq 100 \text{ ppm}$) were elevated at both Reelfoot and Lower Hatchie NWRs, while Cd and Pb (≥ 1.0 and $\geq 38 \text{ ppm}$, respectively) were elevated only at Lower Hatchie.

In this investigation, average strontium concentrations were approximately four-fold higher in Duck River Unit sediment samples than those collected at the Busseltown and Big Sandy Units. Elevated ($\geq 0.18\%$) levels of manganese were also found in three sediment samples from this unit. Chromium in two sediment samples from de-watering areas (pools) on the Duck River Unit and Goodin Branch (Busseltown Unit) were considered elevated ($\geq 23.0 \text{ ppm}$). Copper was elevated in sediment samples from Goodin Branch (14.6 ppm) and Blue Creek (14.9 ppm). Molybdenum

was only detected in samples from three de-watering areas on the Duck River Unit (1.90 ppm) and in Goodin Branch (19.4 ppm) on the Busseltown Unit.

Canadian sediment quality guidelines published by Persaud *et al.* (1989) and Jaagumaji (1992) indicated that average Mn concentrations at three refuges in West Tennessee (Robison *et al.* 2000) exceeded the limit of tolerance or severe effects level (0.111%). Also, Fe was greater than the limit of tolerance (4%) at Lower Hatchie NWR. Lowest effect levels (LELs) published by Persaud *et al.* (1989) and Jaagumaji (1992) were exceeded for Cr (26 ppm) and Ni (16 ppm) at all three NWRs. Arsenic and Cu LELs were exceeded at Reelfoot and Lower Hatchie NWRs, while Pb and Zn did so only at Lower Hatchie. Average Hg concentrations did not exceed the Canadian tolerance values, although values at Reelfoot and Lower Hatchie NWRs approached the LEL (0.12 ppm) reported by Persaud *et al.* (1989).

In this study, manganese levels at the Duck River Unit exceeded the limit of tolerance or severe effects level of 0.111 % (Persaud *et al.* (1989) and Jaagumaji (1992)). The arsenic concentration in one sediment sample (12.4 ppm) collected in Goodin Branch within the Busseltown Unit exceeded their published LELs. Mercury concentrations also approached the Canadian tolerance value in Goodin Branch (0.11 ppm) while average nickel concentrations exceeded this level only within the de-watering areas on the Duck River Unit (20.7 ppm).

CONCLUSIONS AND RECOMMENDATIONS

Based on the whole-body fish sample results, none would be expected to exceed any applicable action levels established by the Food and Drug Administration (FDA 1990). The filet values for largemouth bass and white crappie collected at the Busseltown Unit were below the FDA action level of 1.0 ppm for Hg, however, these levels may exceed current risk-based screening levels for consumption by special populations utilized in several states in the Southeast Region. Unfortunately, filet samples were not analyzed for metals at the Duck River Unit and suitable specimens were not collected at the Big Sandy Unit. Populations of concern would include women of child-bearing age and children under the age of 12 years old. Precautionary fish consumption advisories, particularly those which involve mercury, place recommended limits on the number of meals of fish that these special populations should consume on a weekly, monthly, and/or yearly basis. The State of Tennessee does not currently utilize a risk-based screening approach that has resulted in the issuance of site-specific precautionary advisories or a state-wide advisory for Hg. The FWS has not formally adopted a similar approach on National Wildlife Refuges in the United States. Subsequent investigation and evaluation of contaminants in fish filet samples may be warranted on all units of Tennessee National Wildlife Refuge.

Concentrations of p,p'-DDE and Hg were sufficient at several locations to warrant concern for fish-eating birds, particularly when cumulative exposures and effects are considered. Concentrations of As and Se were also elevated in a small percentage of the samples collected. This concern is somewhat ameliorated because many of the fish collected that contained elevated concentrations of these contaminants were too large for consumption by most piscivorous birds, except raptors. Depending upon the foraging and feeding habits of specific raptor species, elevated levels of these and other contaminants in tissue and internal organs of the fish collected may pose additional un-quantified risks.

Overall there does not appear to be any immediate need for mitigation or clean-up of environmental contamination at the three units of Tennessee National Wildlife Refuge sampled in this study. The elevated concentrations of various inorganic contaminants in sediment within the Busseltown Unit (Goodin Branch) may warrant further investigation. Future biota sampling activities should also include amphibians and reptiles to provide a more complete evaluation of contaminant exposure pathways.

Refuge managers can best enhance the overall environmental quality on Tennessee NWR by:

- 1) improving cooperative farming practices on the refuge to reduce soil erosion and the associated transport of environmental contaminants to aquatic systems;
- 2) continue the implementation of the integrated pest management program on the refuge that couples the proper use of appropriate pesticides with other techniques;

- 3) installing and protecting vegetative buffer strips along stream channels, ditches, swales, and other water-conveyance conduits on the refuge; and
- 4) working actively with private landowners, other Federal and State agencies, and non-governmental organizations in the refuge watershed to improve land use practices.

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APPENDICES

Appendix A

Elution Profiled For Florisil, Silica Gel and Silicic Acid Column Separations

Florisil Column (used for tissue samples)

Fraction I (6% ethyl ether containing 2% ethanol, and 94% petroleum ether): HCB, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordan, heptachlor epoxide, gamma-chlordane, trans-nonachlor, toxaphene, PCBs, o,p'-DDE, alpha-chlordane, p,p'-DDE, p,p'-DDT, cis-nonachlor, o,p'-DDT, p,p'-DDD, mirex, dicofol, endosulfan I (split with F-II).

Fraction II (15% ethyl ether containing 2% ethanol, and 85% petroleum ether): dieldrin, endrin, dacthal, endosulfan I (split with F-I), endosulfan II (split with F-III), endosulfan sulfate (split with F-III).

Fraction III (50% ethyl ether containing 2% ethanol, and 50% petroleum ether): endosulfan II (split with F-II), endosulfan sulfate (split with F-II), malathion.

Florisil Mini-Column (used for soils)

Fraction I (12 ml of hexane, followed by 12 ml of 1% methanol in hexane): HCB, gamma-BHC (25%), alpha-BHC (splits with F-II), trans-nonachlor, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD (splits with F-II), o,p'-DDT, p,p'-DDT, cis-nonachlor, cis-chlordane, trans-chlordane, PCBs, mirex, photomirex and derivatives.

Fraction II (24 ml of 1% methanol in hexane): g-BHC (75%), b-BHC, a-BHC (splits with F-I), delta-BHC, oxychlordan, heptachlor epoxide, toxaphene, dicofol, dacthal, endosulfan I, endosulfan II, endosulfan sulfate, octachlorostyrene, kepone (with additional 12 ml 1% methanol in hexane).

Silicic Acid

SA Fraction I (20 ml petroleum ether): HCB, mirex

SA Fraction II (100 ml petroleum ether): PCBs, p,p'-DDE (splits with SA-III)

SA Fraction III (20 ml of 1% acetonitrile, 80% methylene chloride, 19% hexane): alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, oxychlordan, heptachlor epoxide, gamma-chlordane, trans-chlordane, toxaphene, o,p'-DDE, alpha-chlordane, p,p'-DDE (splits with SA-II), cis-nonachlor, o,p'-DDT, p,p'-DDD, p,p'-DDT, dicofol.

Appendix B

Tissue Sample Preparation For Metal Analyses

Tissue Preparation

Samples are homogenized using a Kitchen Aid food processor. Portions are then freeze dried for determination of moisture content and subsequent acid digestions.

Digestion for Inductively Coupled Plasma Emission (ICP) measurement. About 0.25 to 0.5 grams of freeze-dried tissue are placed in a 120 ml teflon microwave vessel and five ml of Baker Instra-Analyzed HNO₃ are added to the vessel. The vessel is then capped according to the manufacturer's instructions and heated in a CEM microwave oven for 3 minutes at 120 watts, 3 minutes at 300 watts, and 15 minutes at 450 watts. The resulting residue is diluted to 50 ml with 5% HCl.

Digestion for Graphite Furnace Atomic Absorption (GFAA) measurement. Using a CEM microwave oven, 0.25 to 0.5 grams of freeze-dried tissue are heated in a capped 120 ml teflon vessel in the presence of 5 ml of Baker Instra-Analyzed HNO₃ for 3 minutes at 120 watts, 3 minutes at 300 watts, and 15 minutes at 450 watts. The residue was is then diluted to 50 ml with laboratory pure water.

Digestion for mercury measurement by Cold Vapor Atomic Absorption (CVAA). About 0.25 to 0.5 grams of sample are refluxed for 2 hours in 10 ml HNO₃ (Baker Instr-Analyzed) and diluted to 50 ml with 1% HCl.

Metal Analyses

ICP measurements were made using a Leeman Labs Plasma Spec I sequential spectrometer. GFAA measurements were made using a Perkin-Elmer Zeeman 3030 AA spectrophotometer with an HGA-600 graphite furnace and an AS-60 autosampler. Mercury measurements were conducted using SNCL₄ (as the reducing agent) and an Instrumentation Laboratories Model 251 AA spectrophotometer.

Appendix C

Sediment Sample Preparation for Chlorinated Hydrocarbon Pesticides, Polychlorinated Biphenyls, and Chlorophenoxy Acid Herbicide Analyses

Sample Preparation

Twenty grams of soil are weighed into a pesticide residue quality (PRQ)* centrifuge bottle and 10 ml of PRQ H₂O are added to the dry samples. The pH is adjusted to ≤ 2 using PRQ 12N sulfuric acid (usually about 1 ml). Fifty ml of acetone are added and the sample is shaken well six times over a 90 minute period (about every 15 minutes). Fifty ml of a 1:1 petroleum ether/ethyl ether (PE:EtoEt) mixture are added and the shaking is repeated. The sample is then centrifuged and the liquid decanted into a 500 ml separatory funnel containing 200 ml of PRQ water. The soil is re-extracted by shaking one minute with 50 ml 1:1 PE:EtoEt (10 ml H₂O may need to be added and the pH re-adjusted to ≤ 2), centrifuging again, and decanting the liquid into a separatory funnel.

Using PRQ 6N KOH (5 ml), the contents of separatory funnel are adjusted to pH ≥ 12 , shaken vigorously for two minutes, and then allowed to stand for 30 minutes with intermittent shaking. The H₂O layer is separated and re-extracted with 100 ml 1:1 PE:EtoEt. The two petroleum ether extracts are then combined, capped, and reserved for analysis (this contains the chlorohydrocarbon pesticides, aliphatic and polynuclear aromatic hydrocarbons).

The aqueous layer is adjusted to pH ≤ 2 using three ml of PRQ 12N sulfuric acid and extracted with 100 ml 1:1 PE:EtoEt. The H₂O layer is separated and re-extracted with 100 ml 1:1 PE:EtoEt. The two petroleum ether extracts are then combined, capped, and reserved for analysis (this contains the chlorophenoxy acid herbicides).

Both the acid and the basic extracts are concentrated with Kuderna-Danish evaporators and their volumes reduced to adequate size for column clean-up.

Column Cleanup

Neutral Fraction (N/P and chlorohydrocarbon pesticides, aliphatic and polynuclear aromatic hydrocarbons). The sample extract is adjusted to exact volume and an appropriate aliquot removed for column clean-up techniques specific to analyte. For pesticides, a mini-florisil column is used, and for hydrocarbons a 1% deactivated silica gel column is used (Appendix A and B).

Acid Fraction (Chlorophenoxy acid herbicides)

Derivitization. The sample volume is reduced to approximately 0.5 ml and ethylated using diazoethane (15 minutes). The sample is exchanged to hexane (N-EVAP) and the volume reduced to 0.2 ml.

Column Clean-Up. Two grams of 1% deactivated silica gel are placed in a 7 mm i.d. chromatography column (#22 Kontes). This is topped with one cm of Na₂SO₄ and the column pre-wetted with 10 ml of hexane. The sample is then divided into three fractions as follows:

Fraction A: The sample is added and the container rinsed with two 0.5 ml washes of 20 % benzene in hexane. The column is then eluted with nine ml of the same solution (this fraction contains PCP).

Fraction B: Ten ml of 40% benzene in hexane are added. This is followed by ten ml of 60% benzene in hexane (this fraction contains Dalapon, PNP, Silvex, Dinoseb and a portion of Dicamba).

Fraction C: Ten ml of 80% benzene in hexane are added and followed by ten ml of 100% benzene (this fraction contains Dichlorprop, 2,4-D, 2,4,5-T, 2,4-DB, Bentazon, Blazer, and the remaining Dicamba).

Reference for column clean-up for chlorophenoxy acid herbicides:

Shafik, T. A., H.C. Sullivan and H.R. Enos. 1973. Multi-residue procedure for halo and nitrophenols: measurement of exposure to biodegradable pesticides yielding these compounds as metabolites. *J. Agr. Food Chemistry* 21:295-298.

* PRQ glassware and other equipment is obtained by rinsing 3 times with acetone followed by 3 rinses with petroleum ether.