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# BACKGROUND CONTAMINANTS EVALUATION OF FORT NIOBRARA AND VALENTINE NATIONAL WILDLIFE REFUGE

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## ABSTRACT

The objectives of this study were to determine background concentrations of metals and organic compounds in biotic and abiotic components of the Fort Niobrara/Valentine National Wildlife Refuge (NWR) Complex and document these results to utilize as baseline information for future contaminants investigations. To establish this baseline, sediment, water, and biotic samples (fish, double crested cormorant eggs, and vegetation) were collected from Fort Niobrara and Valentine National Wildlife Refuge in 1995 and tested for inorganic and organic contaminants. Inorganics were determined by inductively coupled plasma atomic emission spectrophotometer (ICP) scans. Arsenic and selenium were analyzed by atomic absorption spectrophotometry (AAS). Mercury levels were determined by cold vapor atomic absorption. Organochlorine scans were performed by capillary gas chromatography with an electron capture detector to determine organic contaminant levels.

Elevated inorganic contaminant concentrations were detected in water, sediment, plant, and fish samples collected from each refuge. Only aluminum appeared elevated in water samples collected from Valentine NWR. Aluminum and arsenic appeared elevated at Fort Niobrara NWR. At Valentine NWR, elevated concentrations of metals in sediment were limited to one of the three sediment samples from Pelican Lake (Valentine NWR), which contained elevated levels in 11 of 19 metals analyzed. Aquatic plants collected from Valentine NWR showed very limited contamination; only boron was elevated in the common star duckweed collected from Marsh Lake. Concentrations of boron and selenium were elevated in aquatic macrophytes collected from Fort Niobrara NWR. Elevated concentrations of copper, molybdenum, and zinc were detected in fish collected from Valentine NWR. Concentrations of aluminum, copper, selenium, and zinc appeared elevated in fish collected from Fort Niobrara NWR. None of the concentrations detected in double-crested cormorant eggs appeared elevated.

Concentrations of organics did not appear to be elevated in any of the media sampled from both refuges. The lack of intensive agriculture and absence of industrial development have likely allowed these refuges to remain in fairly pristine condition.

## INTRODUCTION

The Fort Niobrara/Valentine National Wildlife Refuge (NWR) Complex is located in Cherry County, Nebraska, in the heart of the Sandhills (Figure 1). The Sandhills contain the largest remaining tract of mid- and tall-grass prairie in North America. Land use surrounding the refuge complex is dominated by rangeland which likely results in minimal inputs of point or non-point source pollution. Industrial development in the area is minimal and the NWR Complex is believed to be in fairly pristine condition. Nonetheless, a few concerns do exist for both refuges. The past use (i.e., 1950's) of persistent organochlorine pesticides on Valentine NWR and the detection of elevated levels of selenium in the Niobrara River upstream of Fort Niobrara NWR (Esmoil et al. In Press) are potential contaminant concerns. The objectives of this study were to determine background concentrations of metals and organic compounds in biotic and abiotic components of the Refuge complex and document these results to utilize as baseline information for future contaminants investigations.

## STUDY AREAS

### Valentine National Wildlife Refuge

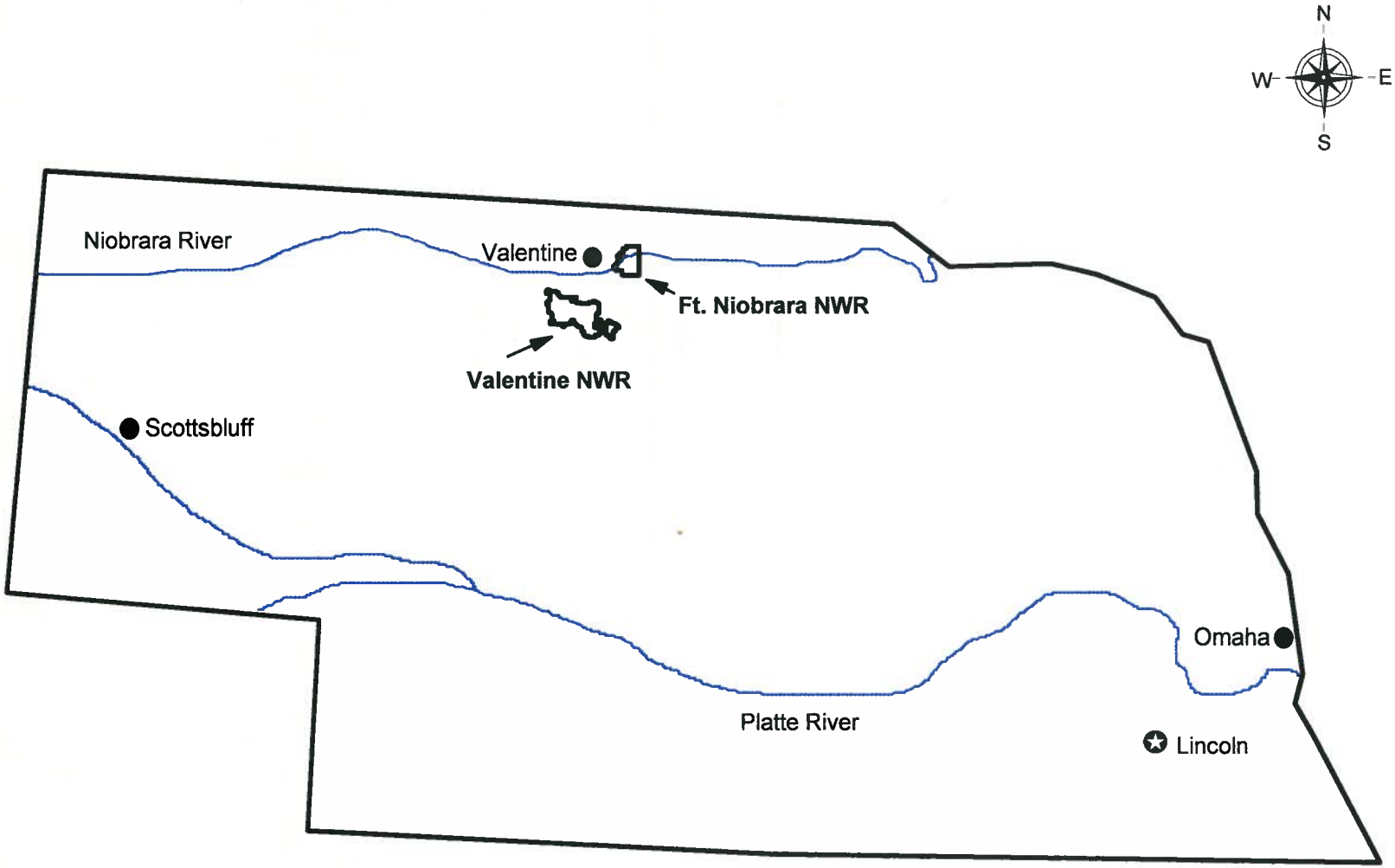
Established in 1935, Valentine NWR contains 38 natural lakes and marshes which occupy one sixth of the 71,516 acre refuge. Ninety percent of the lakes in Cherry County are classified as slightly alkaline, while the remaining 10% range from medium to strongly alkaline (average pH 8.8) (McCarraher 1977). The remaining lands consist of meadows, and grassy dunes. Land use adjacent to the refuge is primarily rangeland used for cattle grazing. A few center pivot irrigation systems are present primarily for alfalfa irrigation. Agricultural pesticides and fertilizers are likely applied on cropland adjacent to the refuge.

The primary objective of the refuge is waterfowl management. During migration up to 150,000 waterfowl utilize the refuge. Federally listed threatened species observed on the refuge include the piping plover (Charadrius melodus), bald eagle (Haliaeetus leucocephalus) and western prairie fringed orchid (Platanthera praeclara). Endangered species occurring on the refuge include the peregrine falcon (Falco peregrinus), least tern (Sterna antillarum), whooping crane (Grus americana), American burying beetle (Nicrophorus americanus), and blowout penstemon (Penstemon haydenii). A total of 270 bird species have been observed with 107 species nesting on the refuge. Additional wildlife using the refuge include six species of amphibians, 16 species of reptiles, and 24 species of mammals (Bogan 1995).

Some pesticides have been used on the refuge. During the 1950's, toxaphene was applied to Hackberry and Dewey Lakes to control common carp (Cyrinus carpio) populations. Refuge personnel have also applied very limited amounts of Tordon (picloram) and Banvel (dicamba) for spot control of leafy spurge. Warbex (famphur) was used in the past on cattle which graze on the refuge. Perhaps the only other known contaminant on the refuge would be presence of lead shot from a private hunting club that was previously located on Marsh Lake. The refuge now requires the use of non-toxic shot for all hunting.

Atmospheric deposition of contaminants is not a major concern as a grain elevator is the only registered air emissions producer within 50 km of the refuge. Further, it does not appear

Figure 1. Location of Valentine and Ft. Niobrara NWR, Nebraska.



that groundwater transport of contaminants is likely. Only surface water runoff from adjacent lands enters the refuge. Further, the watershed of most of the lakes are small because of the topography of the region. One livestock confined winter feeding operation is located on the western edge of the refuge, and is within the watershed of West Long Lake.

**Fort Niobrara National Wildlife Refuge**

Fort Niobrara NWR is located 4 miles east of Valentine, Nebraska, and encompasses 19,122 acres. The refuge is situated on the abandoned military post of Fort Niobrara Military Reservation. In 1912 it became a preserve and breeding ground for native birds, and is now additionally managed to maintain bison (Bison bison), elk (Cervus elaphus), and Texas longhorns (Bos indicus) (to preserve the gene pool). The refuge contains sandhill prairie, mixed grass prairie, rocky mountain forest, eastern deciduous forest, and northern boreal forest. The portion of the refuge north of the Niobrara River contains irregular plateaus cut by dry ravines and small streams. The southern portion of the refuge is comprised mostly of rolling Sandhills.

Federally listed species observed on the refuge include the bald eagle, peregrine falcon, whooping crane. While habitat exists for the following federally listed species, documented occurrences are lacking: American burying beetle, blowout penstemon, eskimo curlew (Numenius borealis), and the black-footed ferret (Mustela nigripes). Over 250 bird species have been documented on the refuge, as well as 7 species of amphibians, 17 species of reptiles, and 14 species of mammals (Bogan 1995).

Atmospheric deposition of contaminants is not a major concern as a grain elevator is the only registered air emissions producer within 50 km of the refuge. Further, groundwater transport of contaminants is unlikely. The sewage outfall for the city of Valentine, Nebraska, is located on Minnechaduza Creek which flows into the refuge. Elevated selenium levels have been detected on the Niobrara upstream of the refuge (Esmoil et al. In Press). Heavy use of the Niobrara River in the summer for canoeing and tubing has caused concern for potential human-related water quality impacts (M. Lindvall, USFWS, pers. comm).

## MATERIALS AND METHODS

This survey included eight sampling locations on the two refuges. Five lakes were sampled at Valentine NWR and three rivers/creeks were sampled at Fort Niobrara NWR. Water, sediment, aquatic plants, and fish or double-crested cormorant (Phalacrocorax auritus) eggs were collected from each aquatic system in July of 1995 (sampling locations are denoted in Figures 2 and 3). Grab samples (approximately 1 liter) of surface water were stored in pre-cleaned plastic jars. These unfiltered samples were preserved with nitric acid (pH <2), stored in ice, and later shipped to the analytical laboratory. A stainless steel spoon was used to collect the top 2-3 cm of sediment. Approximately 800 g of sediment was placed in pre-cleaned glass sample jars. Plants were collected as close as possible to the location of water and sediment collections. The entire plant was collected for analysis. Tables 1 and 2 show the species collected from each refuge.

Samples were sent to Geochemical and Environmental Research Group (GERG) and analyzed for organochlorine compounds (fish/double-crested cormorant eggs and sediment) and

Figure 2. Valentine National Wildlife Refuge sampling locations

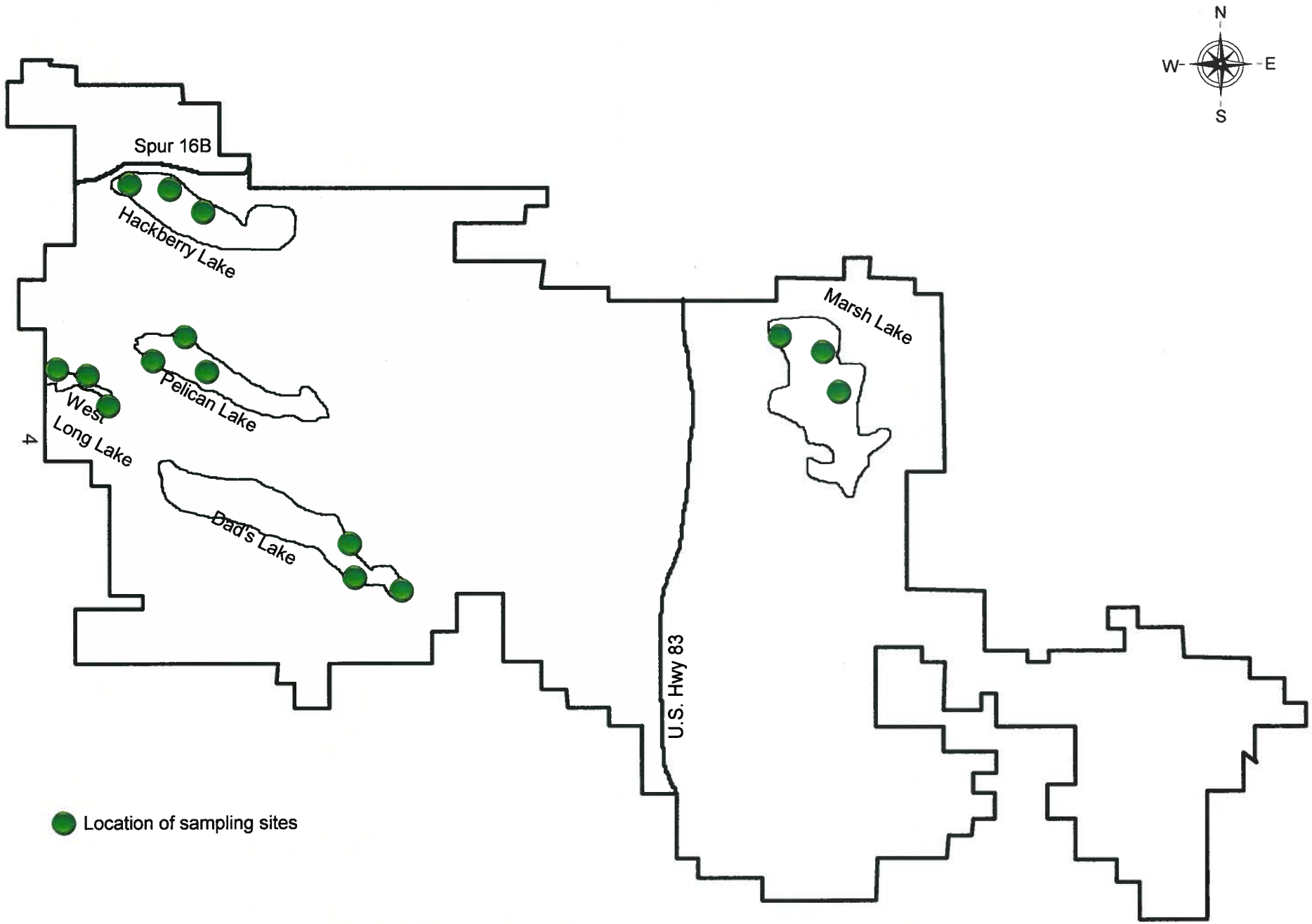
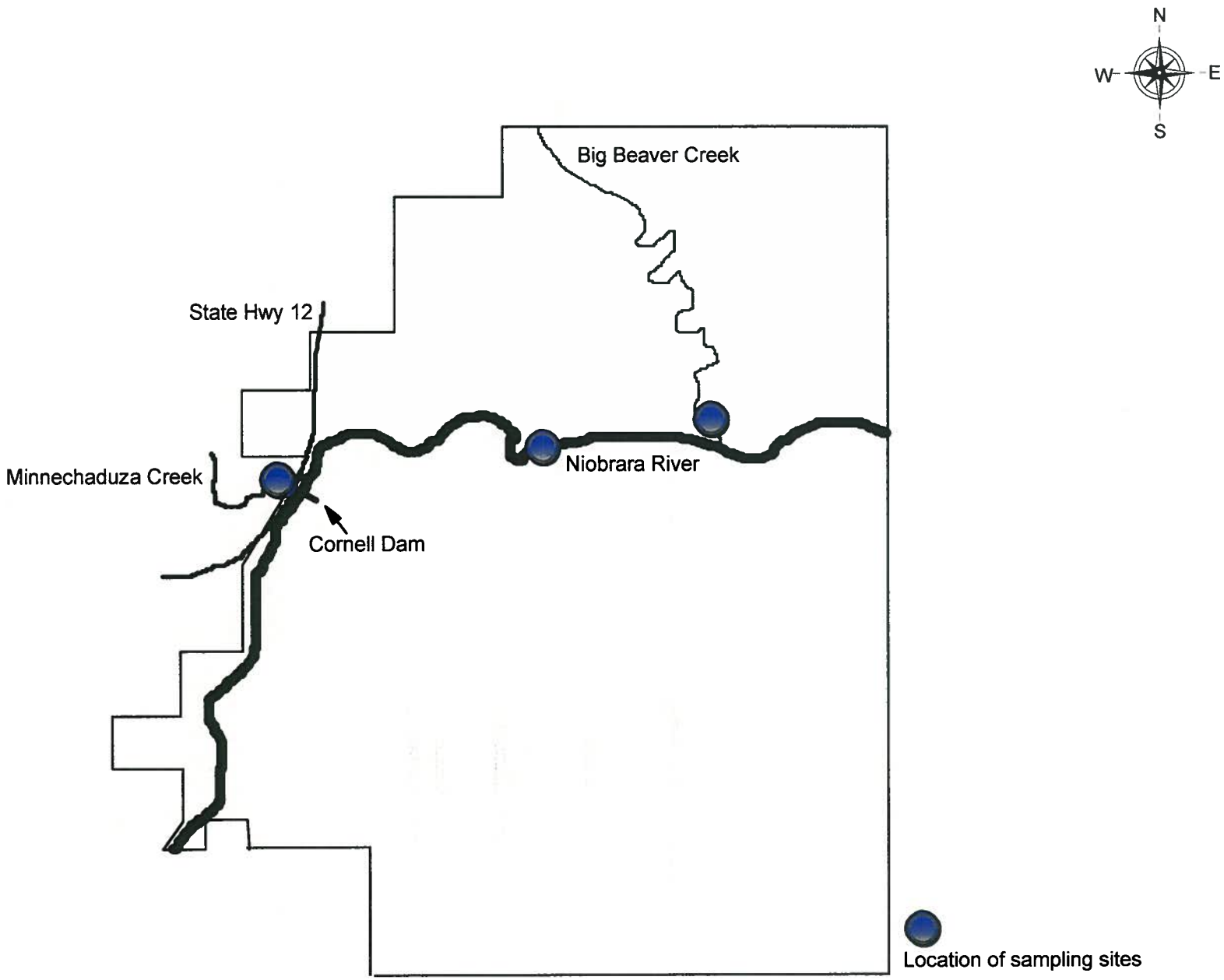


Figure 3. Fort Niobrara National Wildlife Refuge sampling locations.



inorganics (water, sediment, plants, and fish/double-crested cormorant eggs). Inorganics were determined by inductively coupled plasma atomic emission spectrophotometer (ICP) scans. Arsenic and selenium were analyzed by atomic absorption spectrophotometry (AAS). Mercury levels were determined by cold vapor atomic absorption. Analysis of organochlorine contaminants included organochlorine compounds and PCB congeners. Organochlorine scans were performed by capillary gas chromatography with an electron capture detector to determine organochlorine contaminant levels.

Concentrations are reported in µg/g or ppm dry weight, unless noted otherwise (or if it refers to a concentration in water).

## RESULTS and DISCUSSION

### Valentine National Wildlife Refuge

#### Aluminum

Aluminum is abundant in the earth's crust and production and consumption of this metal is high. The largest contributor of anthropogenic aluminum to surface waters is the discharge of alum sludge from municipal water treatment plants. However, the resulting increase of aluminum in water from these additions appears to be minimal (Moore 1991). Toxicity of aluminum is dependent on pH, and the high pH found in most Sandhills lakes reduces the bioavailability, and thus the likelihood of aluminum toxicity. The pH dependent toxicity of aluminum in plants, although well documented, remains unclear. One possible theory is that higher pH may reduce the plant's sensitivity to aluminum (Parker et al. 1989). Speciation of aluminum in aquatic environments is further dictated by total organic carbon and other limnological parameters. Total aluminum, therefore, is not a useful measurement to determine possible toxic effects without additional environmental parameters.

Aluminum is the third most common metal in the earth's crust averaging 72,000 ppm in the conterminous U.S. (Shacklette and Boerngen 1984), and sediment concentrations from the refuge lakes were much lower ranging from 997 ppm to 5,485 ppm. Water samples collected from refuge lakes varied in levels of aluminum detected. A water sample from West Long Lake contained 1.435 ppm of aluminum, exceeding the U.S. Environmental Protection Agency's chronic toxicity value for the protection of aquatic life, of 0.087 ppm and acute toxicity value of 0.75 ppm (USEPA 1988). Marsh Lake had the highest aluminum concentration at 4.37 ppm. This level is similar to the 4.4 ppm aluminum sulfate $LC_{50}$ for the fathead minnow, and exceeds the 4.25 ppm aluminum nitrate $LC_{50}$ for the fathead minnow (Mayer and Ellersiek 1986).

Aluminum detected in aquatic plants varied greatly between lakes, between species within lakes, as well as within species. Concentrations in aquatic macrophytes ranged from 18.27 to 2303 ppm. Comparatively, concentrations of aluminum in aquatic mosses collected in a Welsh metal mine drainage contained 54,000 ppm aluminum, and in less contaminated reaches the levels of aluminum in the mosses dropped to 2,000-7,000 ppm (Moore 1991). High concentrations of aluminum in plants can result in decreased root growth, increased mucilage production (Crowder 1991), as well as reduction in plant biomass (Parker et al. 1989). Although toxic thresholds for aquatic plants are not well documented, a toxicity threshold has been

developed for rice at 300 ppm (Crowder 1991).

Fish exposed to aluminum may exhibit respiratory stress, histopathological changes in the liver, kidney, skin, and muscle, and reproductive abnormalities (Brumbaugh and Kane 1985). Further, fish exposed to aluminum were smaller than fish exposed to control conditions in a study by Cleveland et al. (1989), which also documented reduced feeding and abnormal swim bladder development. Concentrations of aluminum in fish collected from the refuge lakes ranged from 9.18 to 40.47 ppm (2.08 to 10.24 ppm w.w.). This is lower than the mean detection of 13.8 ppm w.w. (whole body concentrations) detected in smallmouth bass (Micropterus dolomieui) from a reservoir with lower aluminum concentrations in water (Brumbaugh and Kane 1985). The range in aluminum concentrations detected from fish collected from refuge lakes could be from whole body analyses. Brumbaugh and Kane (1985) found gut contents could greatly skew results of aluminum concentrations, and recommended removal for more accurate determination of aluminum levels. Double-crested cormorant eggs contained aluminum concentrations below the level of detection (5 ppm).

**Arsenic**

The major anthropogenic sources of arsenic include industrial smelters, coal-fired power plants, and production and use of arsenical pesticides. These anthropogenic inputs are significant and exceed natural additions (i.e., rock weathering) of arsenic in the environment by a factor of three (Eisler 1994). Arsenic can be mutagenic, teratogenic, and carcinogenic, and can cause bronchitis, pneumonia, and gangrene (Eisler 1988a).

Arsenic water concentrations in the five refuge lakes sampled ranged from 0.005 to 0.017 ppm. Dad's Lake (0.013 and 0.014 ppm) and Marsh Lake (0.014 and 0.017 ppm) contained slightly elevated levels of arsenic compared to the background concentration in water of 0.010 ppm (Eisler 1988a). Adverse acute effects of arsenic have not been recorded in fresh water below 0.048 ppm. However, chronic effects of 1 ppb $As^{+5}$ resulted in plant community structure changes (Eisler 1994). Concentrations of arsenic in refuge lakes were lower than the chronic and acute national criteria for the protection of aquatic life for $As^{+3}$ of 0.19 ppm and 0.360 ppm, respectively (USEPA 1985c). Sediment concentrations detected from refuge lakes were lower than the average for U.S. soils of 5.2 ppm (Shacklette and Boerngen 1984) and ranged from below detection to 2.4 ppm.

Background levels of arsenic in terrestrial and freshwater flora and fauna are typically less than 1 ppm w.w. (Eisler 1994). Of the aquatic macrophytes sampled, only arrowhead (Sagittaria spp.) from Marsh Lake exceeded this with a concentration of 1.2 ppm w.w. However, this level has not been documented to be detrimental when ingested by birds (Eisler 1994). Other aquatic vegetation sampled contained concentrations ranging from 0.09 to 0.36 ppm w.w. Arsenic was below the limit of detection in fish and double-crested cormorant eggs. Arsenic levels detected in refuge components sampled do not appear to be a concern to fish and wildlife.

**Barium**

Barium is a relatively abundant element found most frequently in the environment in the form of barite (barium sulfate) or witherite (barium carbonate) (Moore 1991). Barium is used as a drilling fluid in oil and gas wells which accounts for 90% of barium usage (Moore 1991).

Barium is also used in the production of various barium chemicals (Moore 1991). Anthropogenic inputs of barium result from mining, refining, and processing of barium ore, the burning of fossil fuels (International Programme on Chemical Safety (IPCS) 1990), as well as drilling fluid spills (Moore 1991).

Water quality standards for aquatic life have not been established for barium, but all concentrations were much lower than the drinking water criteria (1 ppm) for barium set by the Nebraska Department of Environmental Quality (NDEQ 1995). Concentrations ranged from 0.15 to 0.355 ppm in refuge lakes. Concentrations of barium in freshwater typically range from 0.007 to 15 ppm (IPCS 1990). Sandhills lakes are typically hard water lakes and the high concentrations of carbonate and sulfate (McCarraher 1977) likely bind with barium to form an insoluble salt that precipitates, resulting in low concentrations of dissolved barium (Moore 1991). Sediment samples collected from refuge lakes ranged from 14.66 to 292.96 ppm and were below the mean barium concentration of 440 ppm in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Barium concentrations in sediment are usually below 100 ppm, and higher levels are usually associated with geologic deposits (IPCS 1990).

Concentrations of barium in macrophytes collected from refuge lakes ranged from 73 to 382 ppm. Barium is not known to accumulate in plants in sufficient quantities to cause toxicity to wildlife (IPCS 1990). Concentrations detected in fish collected from the refuge ranged from 9.99 to 34.56 ppm. Barium concentrations in double-crested cormorant eggs ranged from 1.89 to 5.97 ppm. The lack of data on the toxicity of barium does not allow for a clear interpretation of detected concentrations, at this time, it does not appear that barium concentrations detected on the refuge are a concern.

**Beryllium**

The major anthropogenic source of beryllium in the environment is the combustion of fossil fuels. Beryllium inputs to the aquatic environment occurs via atmospheric deposition, weathering of rocks and soils, as well as municipal/industrial point source inputs (USEPA 1980a). The toxicity of beryllium increases in soft waters and the solubility of beryllium salts changes dramatically with changes in pH (Wilber 1980).

Beryllium was below the limit of detection (0.0005 ppm) in water samples collected from the refuge. Concentrations in surface waters are usually less than 0.001 ppm (USEPA 1980a). Further, guidelines for the protection of aquatic life have been established for water at 0.005 and 0.130 ppm of beryllium for chronic and acute exposures, respectively (NDEQ 1996). Concentrations of beryllium in sediment from this study ranged from below the limit of detection to 26.34 ppm. Sediment samples collected from the five refuge lakes revealed higher beryllium concentrations than the average concentration (0.63 ppm) determined in soils of the conterminous U.S. (Shacklette and Boerngen 1984). A sample collected from Pelican Lake contained the highest beryllium concentration.

Beryllium in aquatic plants collected from refuge lakes were for the most part below the limit of detection. Arrowhead collected from Hackberry Lake and Marsh Lake contained the only detectable levels of beryllium at 0.13 and 0.19 ppm, respectively. Beryllium was below the limit of detection in fish and double-crested cormorant eggs. Therefore, bioavailability from sediment does not seem apparent.

**Boron**

Boron is an essential trace element for higher plants but is not required by fungi or animals (Eisler 1990a). Sources of boron additions to the environment include laundry products, agricultural chemicals including fertilizers, coal combustion, and mining and processing (Eisler 1990a).

Boron levels in surface waters of the U.S. are typically less than 0.5 ppm (Eisler 1990a). All lakes sampled ranged from below detection (0.1 ppm) to 0.12 ppm. These concentrations exceed 1 ppb where teratogenic effects have been documented in rainbow trout (Weis and Weis 1991). The concentrations detected were lower than levels documented to be detrimental to aquatic macrophytes which start as low as 1 ppm. Some plants, however, exhibit no detrimental effects at concentrations as high as 50 ppm (Powell et al. 1997). The average boron concentration in soils in the conterminous U.S. is 26 ppm (Shacklette and Boerngen 1984). All sediment samples collected were below this average, except for one Pelican Lake sample containing 1117 ppm of boron.

Aquatic macrophytes typically contain less than 20 ppm boron and range from 1.2 to 100 ppm (Eisler 1990a). Most plants collected were in this range. However, the common star duckweed (Lemna trisulca) sample from Marsh Lake contained 1297 ppm (83.53 ppm w.w.) boron. Comparatively, the highest boron concentration in filamentous algae collected for an irrigation drainwater study California was 280 ppm (Saiki et al. 1993). Aquatic macrophytes sampled from a boron-contaminated wetland contained up to 142 ppm (Powell et al. 1997). Further, the level of boron detected in duckweed collected from Marsh Lake was higher than levels ingested by mallards which resulted in lower weight gain in ducklings (Smith and Anders 1989). Diets containing over 30 ppm w.w. appear to be detrimental to waterfowl (Eisler 1990a). A composite sample of common carp collected from Hackberry Lake contained the only detectable level of boron of 16.2 ppm. This was the only detectable level of boron in fish, and similar to values from common carp in an unimpacted area (Eisler 1990a). Double-crested cormorant eggs contained boron concentrations ranging from below the limit of detection to 3.03 ppm (0.49 ppm w.w.). In comparison, the 1.0 ppm w.w. $LD_{50}$ concentration determined by injection of boron to the yolk sack of chicken embryos was higher (Eisler 1990a). Although boron concentrations in aquatic plants collected from Marsh Lake appeared elevated, the effects on double-crested cormorant eggs collected from the same lake were not apparent.

**Cadmium**

Cadmium is used in electroplating or in alloys as protection against corrosion. It is also used in batteries, ceramics, some biocides (Moore 1991), and manufacturing of plastic stabilizers (Eisler 1985a). Anthropogenic sources of cadmium include refining and smelting, manufacturing processes, and domestic wastewater (Moore 1991). Cadmium is not a biologically essential element and is toxic to all forms of life. It is a known teratogen, carcinogen, and possible mutagen (Eisler 1985a).

All water samples collected from refuge lakes were below the limit of detection (0.0005 ppm). This was well below background cadmium concentrations from surface waters which range from less than 0.001 to 0.07 ppb (Wren et al. 1995). Cadmium was also below the limit of

detection in sediment, double-crested cormorant eggs, and fish. The average bioconcentration factor for cadmium in aquatic macrophytes is less than 50 (Wren et al. 1995). However, some aquatic species have shown bioconcentration factors as high as 10,000 (Crowder 1991). Concentrations of cadmium in aquatic vegetation sampled from refuge lakes ranged from below detection to 0.1 ppm. Cadmium residues in macrophytes are typically below 5 ppm (Moore 1991). Cadmium uptake in aquatic plants is enhanced at neutral or basic pH, and inhibited by the presence of manganese and iron in the water (through competition for binding sites)(Moore 1991). The level detected in aquatic vegetation from lakes on the refuge is much lower than levels of cadmium having detrimental effects on birds (Eisler 1985a). Cadmium concentrations detected in samples collected from the refuge are below levels of concern.

**Chromium**

Elevated levels of chromium are often found in surface waters near electroplating and metal finishing industries (Eisler 1986a). Other sources of chromium include chromium alloy and metal producing industries, coal combustion, municipal incinerators, cement production, and cooling towers (Eisler 1986a). Chromium is an essential trace element (Moore 1991) but in elevated quantities can be a mutagen, teratogen, and carcinogen (Eisler 1986a). The toxicity of chromium is dependent on hardness, pH, and temperature (Eisler 1986a). Chromium is typically found in the environment in two oxidation states (trivalent and hexavalent oxidation). Both oxidative states exhibit different toxicity and do not appear to be additive (USEPA 1985a). Only information on total chromium was reported by the analytical laboratory.

Chromium was below the limit of detection in all water samples collected from the refuge. Sediment samples ranged from below the limit of detection to 219.03 ppm. The average concentration of chromium in soils of the conterminous U.S. is 37 ppm (Shacklette and Boerngen 1984). Background concentrations of chromium in the Great Lakes ranged from 9 to 86 ppm (Moore 1991). All levels detected in sediment were well below this average except for one Pelican Lake sample containing 219.03 ppm. The level of chromium detected at Pelican Lake sediment exceeds the effects range median (ER-M) of 145 ppm delineated by Long and Morgan (1990). The median effects range delineates the concentration above which effects were frequently or always observed in most species (Long and Morgan 1990).

Concentrations of chromium in aquatic plants ranged from below the limit of detection to 79.39 ppm. The highest concentration detected was still below the concentration that caused detrimental effects in black ducks (Anas rubripes) when ingested (Eisler 1986a). Chromium concentrations in both fish and double-crested cormorant eggs sampled were below the 4 ppm characterizing possible chromium contamination (Eisler 1986a). Although one sediment sample from Pelican Lake appeared elevated, all other abiotic and biotic samples collected from the refuge are below levels of concern.

**Copper**

Copper is a required nutrient for plants and animals but is toxic at levels only slightly higher than those required nutritionally (USEPA 1985b). Anthropogenic inputs of copper include municipal and industrial effluents (especially smelting), refining, metal plating industries (USEPA 1985b), copper containing algicides, paints, wood preservatives, and metal corrosion

(Novotny and Olem 1994). Global copper releases approach nearly 1.8 million metric tons per year of which the majority results from anthropogenic releases (Eisler 1997).

Surface water copper samples from the refuge lakes were below the limit of detection (5 ppb). Concentrations of copper in unpolluted surface waters range from 1 to 10 ppb (USEPA 1985b). However, concentrations of 4 ppb and above can severely alter migratory and other behaviors in fish (Sorenson 1991). Except for one sediment sample from Pelican Lake, which contained 113 ppm of copper, all sediment concentrations from sampled lakes were below the mean (37 ppm) copper concentrations for soils of the conterminous U.S (Shacklette and Boerngen 1984). In the United Kingdom, sediments from uncontaminated estuaries contained 10 ppm copper in sediments, while sediments from contaminated estuaries contained over 2000 ppm (Eisler 1997). Copper detected in Pelican Lake sediment lies between the effects range low ER-L concentration of 70 ppm and the ER-M of 390 ppm (Long and Morgan 1990). The ER-L is the concentration representing the lower 10 percentile of the range of concentrations reviewed (Long and Morgan 1990). The ER-L was the concentration where effects may begin or be predicted among sensitive species (Long and Morgan 1990).

Concentrations of copper in aquatic plants ranged from 0.81 to 13.16 ppm (most concentrations were below 5 ppm). Concentrations of copper in aquatic macrophytes inhabiting copper contaminated streams are typically much higher than those found in the present study, with levels exceeding 600 ppm (Stokes 1979).

Copper concentrations in fish ranged from 1.97 to 4.67 ppm (0.44 to 1.06 ppm w.w.). In comparison, fish from Dad's Lake contained copper concentrations exceeding the 85th percentile of 1 ppm w.w. detected in the National Contaminants Biomonitoring Program (NCBP) (Schmitt and Brumbaugh 1990). Similarly, fish from Pelican Lake exceeding the mean of 0.65 ppm w.w. in the NCBP (Schmitt and Brumbaugh 1990). Copper exposure in fish can result in reduced egg production, abnormalities in fry, and reduced survival of young, and in adults exposure causes alterations in the gill, kidney, and respiration (Sorenson 1991). Concentrations of copper in double-crested cormorant eggs ranged from 5.36 to 6.85 ppm. The paucity of data on the toxicity of copper does not allow clear interpretation of detected concentrations in refuge components sampled.

**Iron**

Iron is used for the production of steel and is the fourth most abundant element in the earth's crust (Moore 1991); it is a required nutrient for almost all organisms (National Research Council (NRC) 1979). Natural erosion is responsible for the majority of iron delivered to the aquatic environment although anthropogenic activities such as mining and municipal effluents also deliver iron to aquatic systems (Moore 1991).

Concentrations of iron in surface waters are highly variable and differences reflect the geomorphology of the watershed and lake sediment composition. Iron concentrations in refuge lakes ranged from below detection (0.1 ppm) to 2.09 ppm. One sample (Marsh Lake) exceeded the chronic criteria for the protection of aquatic life of 1 ppm established by the Nebraska Department of Environmental Quality (NDEQ 1996). All sediment concentrations were below the average (26,000 ppm) for the conterminous U.S. (Shacklette and Boerngen 1984).

Iron concentrations in aquatic macrophytes collected from refuge lakes ranged from 123

to 4906 ppm. While iron concentrations are typically elevated in aquatic plants, knowledge of the concentrations causing toxic effects of iron are lacking (Moore 1991). Concentrations detected in fish and double-crested cormorant eggs are likely not present at concentrations high enough to result in any toxicological effects (Moore 1991). Iron is not known to be toxic at levels present in the environment (NRC 1979) and therefore likely not a concern to refuge fish and wildlife.

**Lead**

Lead is the fifth most utilized metal and is found in batteries, solder and ammunition, and previously in gasoline, paint, and pesticides (USEPA 1992). After centuries of anthropogenic inputs resulting from mining and smelting, lead detection in the environment is becoming ubiquitous (Pain 1996). Lead is not an essential nutrient and is highly toxic (Pain 1995).

Lead was below the limit of detection in water, sediment, fish, and double-crested cormorant eggs collected from the refuge. The only samples with detectable lead levels were samples of aquatic macrophytes with concentrations ranging from below the limit of detection (0.5 ppm) to 2.8 ppm. Plants inhabiting uncontaminated environments generally contain less than 1 ppm w.w., and all w.w. samples of vegetation were below this level (Pain 1995). Further, it does not appear that the concentrations detected in aquatic macrophytes would cause problems for waterfowl using the plants as a food source (Eisler 1988b). Lead accumulation in aquatic plants occurs via uptake from contaminated sediment and to a lesser degree from the water column (Demayo et al. 1982). Concentrations of lead detected in refuge components sampled from the refuge are below levels of concern.

**Magnesium**

All chlorophyllous plants require magnesium and it is generally not a limiting factor in most aquatic systems (Wetzel 1983). Magnesium concentrations in lentic systems remain fairly constant throughout the year. For example, concentrations in Lawrence Lake (a hard-water lake in Michigan) ranged from 24 to 28 ppm over a year (Wetzel 1983). Concentrations in the five lakes sampled on the refuge ranged from 6.99 to 17.69 ppm. Magnesium concentrations in sediment samples were well below average (9,000 ppm) concentrations for soils in the conterminous U.S. (Shacklette and Boerngen 1984). Magnesium concentrations in aquatic macrophytes ranged from 738 to 6,015 ppm. Concentrations in fish ranged from 784 to 1398 ppm, and from 441 to 613 ppm in double-crested cormorant eggs. Studies on magnesium are lacking, and at this time, concentrations detected in refuge components do not appear to be a concern.

**Manganese**

Manganese is detected widely in surface water and sediment, and concentrations can vary greatly (Stubblefield et al. 1997). Because manganese is used mainly in metal alloys (Moore 1991), surface water concentrations adjacent to mining and smelting operations are often elevated as a result of point and nonpoint discharges (Stubblefield et al. 1997).

Manganese concentrations in refuge lakes ranged from 0.0399 to 0.24 ppm. All but two of the samples exceeded the maximum contaminant level (MCL) for drinking water of 0.05 ppm

(Novotny and Olem 1994). However, this criteria is based more on its objectionable taste and staining capacity rather than toxicity (Moore 1991). Stubblefield et al. (1997) recommended determination of an IC25 (25% inhibition concentration based on survival and body weight endpoints) based on water hardness (using $e^{0.2064(\ln \text{hardness}) + 7.092}$). Manganese toxicity is inversely related to water hardness. Prior measurements of water hardness in Dad's Lake and Pelican Lake revealed respective mean levels of 77 and 109 mg/L as $CaCO_3$ (McCarraher 1977), resulting in IC25 levels of 5.5 and 5.9 ppm manganese. Levels detected in refuge lakes were much lower than this range, so it appears that detrimental effects from detected manganese levels in water are unlikely. Sediment manganese concentrations sampled from the lakes were for the most part lower than average (330 ppm) for soils in the conterminous U.S. (Shacklette and Boerngen 1984). However, one sediment sample taken from Pelican Lake contained 1,102 ppm of manganese. No biological effects guidelines have been created for manganese in sediment.

Concentration of manganese in aquatic vegetation ranged from 131 to 1,981 ppm. Manganese is an essential micronutrient in plants and assists in nitrate assimilation in photosynthesis (Wetzel 1983). Manganese tends to saturate metal binding sites in aquatic plants which protects the plants against the effects of more toxic heavy metals (Moore 1991). Concentrations of manganese in fish ranged from 5.58 to 10.95 ppm in refuge lakes. Manganese concentrations in double-crested cormorant eggs collected from Marsh Lake ranged from below the limit of detection to 1.94 ppm. Manganese is normally considered the least toxic of the trace elements for poultry and mammals (Pais and Jones 1997) and therefore, not a concern to refuge fish and wildlife.

**Mercury**

Mercury has no known biological function and has the potential to bioconcentrate and biomagnify (Eisler 1987). Although natural sources emit mercury to the environment (e.g., volcanic activity) (Thompson 1996), anthropogenic sources deliver nearly 9000 metric tons per year to freshwater systems (Moore 1991). The major anthropogenic sources of mercury include coal burning power plants and the manufacturing of chemicals and metals (Moore 1991). Other anthropogenic activities resulting in increases in mercury include battery and florescent light disposal as well as the mining and processing of gold, lead, and copper (Eisler 1987). Mercury poisoning most often results from methyl mercury (the most stable and toxic to wildlife). Symptoms include loss of coordination, numbness in the extremities, and hampered awareness and mental activity (Thompson 1996).

Mercury was below the limit of detection in water and vegetation collected from refuge lakes. However, the limit of detection (0.5 ppb) in water exceeds the chronic criteria (0.012 ppb) for the protection of aquatic life (USEPA 1985d). Sediment samples collected from refuge lakes contained lower levels of mercury in comparison to the average (0.58 ppm) concentration of mercury in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Levels of mercury were below the limit of detection (0.05 ppm) in fish except for a composite carp sample collected from Hackberry Lake containing 0.05 ppm (0.016 ppm w.w). Concentrations causing toxic effects are much higher than those found in common carp samples (Wiener and Spry 1995). Mercury levels in double-crested cormorant eggs ranged from 0.13 to 6 ppm (0.004 to 0.925 ppm w.w.), lower than the 2 ppm w.w. when detrimental effects of mercury often occur (Thompson

1996). Concentrations of mercury detected in refuge components sampled are below levels of concern.

**Molybdenum**

Molybdenum is mainly used in the production of steel alloys as well as in the production of pigments, spark plugs, x-ray tubes and catalysts (Eisler 1989). Anthropogenic sources of molybdenum include coal combustion, molybdenum mining and milling, and oil refining (Eisler 1989). Molybdenum is an essential nutrient for most life forms although it can be toxic; and toxicity of molybdenum is dependent on copper and inorganic sulfate intake (Eisler 1989).

Molybdenum concentrations in surface waters are normally less than 20 ppb, and detrimental effects are usually not detected until concentrations exceed 50 ppm (Eisler 1989). Water samples taken from refuge lakes were below the limit of detection (50 ppb). Concentrations of molybdenum in sediment samples were mostly below the limit of detection. Detected concentrations were from Marsh Lake and Pelican Lake containing 6.65 and 444.46 ppm, respectively. Both these concentrations exceed the average concentration (0.59 ppm) for soils in the conterminous U.S. (Shacklette and Boerngen 1984). However, the concentration detected in Marsh Lake is similar to the average molybdenum concentration detected in western states of 6 ppm (Kubota 1977). The sediment sample from Pelican Lake is similar to that detected in sediments near a molybdenum mine averaging 530 ppm (Eisler 1989). Soil concentrations exceeding 5 ppm are usually indicative of a geologic anomaly or industrial contamination (Eisler 1989).

Vegetation samples from the lakes ranged from mostly below the limit of detection (2 ppm) to 5.73 ppm. Molybdenum is biologically more available to plants in alkaline soils, and the high alkalinity of the refuge lakes may facilitate increased uptake (Eisler 1989). It is unlikely that the concentrations detected in aquatic plants are detrimental based on documentation of freshwater algae containing 20,000 ppm of molybdenum without apparent detrimental effects (Eisler 1989). Concentrations detected in plants were also below dietary levels causing detrimental effects in birds (Eisler 1989). Whole body fish concentrations were for the most part below the limit of detection. The common carp composite sample from Hackberry Lake contained 3.21 ppm of molybdenum. This is higher than the 0.6 ppm average for fish (Saiki and May 1988), and higher than fish inhabiting the lower San Joaquin River and its tributaries which receive elevated concentrations of molybdenum from irrigation drainwater (Saiki et al. 1993). In the study by Saiki et al. (1993), molybdenum showed no signs of biomagnification and was not elevated in any food chain organism tested. Molybdenum concentrations in double-crested cormorant eggs ranged from below detection (2 ppm) to 3.6 ppm. Molybdenum concentrations in refuge components sampled do not appear to be present at levels of concern to fish and wildlife.

**Nickel**

The major anthropogenic sources of nickel in the environment are from the combustion of fossil fuels, electroplating, and smelting industries (USEPA 1986), as well as production of alloys, batteries, and electronics (Birge and Black 1980). The toxicity of nickel is dependent on alkalinity, hardness, salinity, pH, and temperature (USEPA 1986).

Nickel was below the limit of detection in water samples collected from the refuge (0.005 ppm). The detection limit itself was also below the level recommended for the protection of aquatic life for the Great Lakes by the International Joint Commission (Birge and Black 1980). Nickel concentrations in sediment samples were below the limit of detection (5 ppm) except for one Pelican Lake sediment sample which contained 119.33 ppm. Comparatively, the average for soils of the conterminous U.S. is 13 ppm (Shacklette and Boerngen 1984). Nickel concentrations detected in sediment from a confined disposal facility for dredged material contained levels ranging from 12 to 150 ppm (Beyer et al. 1990). The ER-M for nickel in sediment is 50 ppm, at this concentration, effects are frequently or always observed or predicted (Long and Morgan 1984).

Nickel concentrations in aquatic macrophytes ranged from below the limit of detection (0.5 ppm) to 36.71 ppm (below the limit of detection to 3.07 ppm w.w.). The highest concentration was from an arrowhead plant collected in Hackberry Lake. Aquatic macrophytes inhabiting uncontaminated areas contained nickel concentrations less than 6 ppm w.w. (Jenkins 1980). Concentrations of nickel in macrophytes from refuge lakes were lower, and thus nickel levels do not appear problematic for aquatic plants from the refuge lakes sampled. Similarly, concentrations of nickel in fish collected from refuge lakes were much lower than the nickel concentrations in whole fish from unpolluted locations (less than 2.0 ppm w.w.) (Jenkins 1980). Nickel concentrations detected in fish from refuge lakes ranged from 0.13 to 0.38 ppm w.w.. Concentrations of nickel in double-crested cormorant eggs ranged from below the limit of detection (0.5 ppm) to 1.31 ppm. These are similar to levels found in American coot (Fulica americana), mallard (Anas platyrhynchos), and blue-winged teal (Anas discors) eggs from the Belle Fourche and James River in South Dakota (Sowards et al. 1991). Although one sediment sample from Pelican Lake was elevated, bioavailabilty from the sediment is not apparent. Nickel concentrations detected in refuge components sampled do not appear detrimental to fish and wildlife.

**Selenium**

Selenium is an essential micronutrient. However, levels exceeding the capacity of metabolic regulation leads to toxicity. Two anthropogenic activities are the major contributors to elevated levels of selenium: 1) production and use of fossil fuels; and 2) irrigation of seleniferous soils in semiarid regions of the country (Lemly 1996). High selenium concentrations (exceeding 100 ppm) in the diets of adult mallards can result in fatality. Lower levels of selenium can result in reduced hatching success. In fish, toxic levels of selenium results in loss of equilibrium, loss of coordination, liver degeneration, and an increase in white blood cell count (Eisler 1985b).

Selenium concentrations in water, sediment, and aquatic vegetation collected from refuge lakes were below the limit of detection. Concentrations of selenium detected in fish sampled from the refuge ranged from below detection (0.5 ppm) to 1.3 ppm (below the limit of detection to 0.295 ppm w.w.). This is lower than the 4 ppm toxic effects threshold reported by Lemly (1996). Further, concentrations in fish were below the national average determined by the NCBP of 0.42 ppm w.w. (Schmitt and Brumbaugh 1990). Similarly, selenium concentrations detected in double-crested cormorant eggs collected from Marsh Lake ranged from 0.216 to 0.309 ppm

w.w. This is much lower than the 3 ppm w.w. threshold for reproductive impairment (Heinz 1996), and lower than levels reported by Heinz et al. (1989) of possible reproductive impairment when selenium concentrations exceed 1 ppm w.w.. Selenium concentrations detected in refuge components sampled are below levels of concern.

**Strontium**

Strontium is an alkaline earth element and like calcium, accumulates in bone tissue (Pais and Jones 1997). Strontium concentrations detected in refuge lakes ranged from 0.213 to 0.375 ppm and sediment concentrations ranged from 6.2 to 545 ppm. The average concentration of strontium in soils for the conterminous U.S. is 120 ppm (Shacklette and Boerngen 1984). All concentrations of strontium are well below this average except for one Pelican Lake sample containing 545 ppm. Aquatic macrophytes collected from refuge lakes contained strontium ranging from 25 to 214 ppm. Fish contained from 40 to 112 ppm, and double-crested cormorant eggs contained from 5 to 11 ppm. Much of the research available on strontium focuses on the radioactive form, $^{90}Sr$. Non-radioactive strontium is not know to be toxic (Pais and Jones 1997).

**Vanadium**

Vanadium is used in metallurgy, dyes, inks, paints, as well as being used as a catalyst in the production of polymeric plastics (Moore 1991). The major anthropogenic source of vanadium in the environment results from the combustion of oil and coal (Moore 1991).

Vanadium levels in water samples from refuge lakes ranged from below the limit of detection (0.001 ppm) to 0.006 ppm. Levels detected in surface waters generally are below 0.22 ppm (International Programme on Chemical Safety 1988). Sediment concentrations ranged from 2.02 to 218.99 ppm. The average concentration for soils of the conterminous U.S. is 58 ppm (Shacklette and Boerngen 1984). Sediment vanadium levels typically range from 20 to 150 ppm (Moore 1991). All concentrations were well below this level except for one Pelican Lake sample containing 218.99 ppm.

Concentrations of vanadium detected in aquatic macrophytes collected from refuge lakes ranged from below the limit of detection (0.5 ppm) to 8.23 ppm. Vanadium appears to be relatively nontoxic to plants and concentrations in freshwater plants normally range from 0.10 to 5.7 ppm (Moore 1991). Vanadium also appears to be relatively nontoxic to fish with $LC_{50}$s ranging from 2.9 to 5.6 ppm (in hard water) (Moore 1991). Concentrations of vanadium detected in fish collected from refuge lakes ranged from 0.76 to 1.65 ppm. Concentrations of vanadium in double-crested cormorant eggs ranged from below the limit of detection (0.5 ppm) to 1.1 ppm. The paucity of data available on vanadium toxicity does not allow for clear interpretation of the detected concentrations from refuge components sampled. At this time, it does not appear that vanadium concentrations detected on the refuge are hazardous to fish and wildlife.

**Zinc**

Zinc is one of the most widely used metals worldwide and its principal uses include galvanizing steel, an additive for paint, and an ingredient in rubber (USEPA 1987b). The major anthropogenic sources occur from smelting operations, combustion of fossil fuels, as well as from corroded galvanized electrical transmission towers (Eisler 1993). Once zinc enters the

aquatic environment, it is usually partitioned into the sediments and release is then dependent on high dissolved oxygen and low salinity and pH (Eisler 1993). In water, speciation is dependent on oxygen levels, pH, and salinity. Fish exposure to toxic zinc concentrations however, can increase in alkaline waters. The change in pH around the gill due to the release of $CO_2$ may cause the release of toxic soluble zinc from the zinc precipitates present in some alkaline environments (Sorenson 1991). While zinc is a required nutrient and is essential for normal growth, reproduction, and the ability to heal, it can be teratogenic, mutagenic, and carcinogenic (Eisler 1993).

Concentrations of zinc rarely exceed 0.04 ppm in water (Eisler 1993), and all water samples collected from the refuge were lower than this benchmark (concentrations ranged from below the limit of detection (0.01 ppm) to 0.0135 ppm). The proposed zinc criteria for the protection of aquatic life in surface waters is 0.047 ppm (Eisler 1993). Zinc concentrations in sediment samples ranged from below the limit of detection (5 ppm) to 565 ppm. All but one of the samples were well below the average concentration (180 ppm) for soils in the conterminous U.S. (Shacklette and Boerngen 1984). The level detected in Pelican Lake sediment (565 ppm) exceeds the ER-M of 270 ppm (Long and Morgan 1990). The chemistry of the lakes in the Sandhills, however, likely reduces the availability of zinc in the sediment (i.e., high pH, low dissolved oxygen, and high salinity) (McCarraher 1977).

Levels of zinc detected in aquatic macrophytes ranged from 9.65 to 60.72 ppm. Marginal sublethal effects of dietary zinc concentrations for birds occur starting at 178 ppm (Eisler 1993). This concentration is much higher than levels detected in aquatic macrophytes from refuge lakes. Concentrations of zinc in fish ranged from 63.22 to 235.16 ppm (14.21 to 74.96 ppm w.w.). Zinc concentrations in three of the four fish samples exceeded the national average of 21.7 ppm w.w. Further, two concentrations exceeded the 85th percentile (34.2 ppm w.w.) of fish collected as part of the NCBP in 1984 (Schmitt and Brumbaugh 1990). The highest concentrations of zinc detected were in common carp collected from Pelican Lake and Hackberry Lake. Common carp appear to accumulate zinc to a greater degree than other species (Schmitt and Brumbaugh 1990). Increased zinc concentrations in fish tissue can lead to growth retardation, inhibition of spawning, as well as mortality (Sorenson 1991). Zinc concentrations can vary dramatically depending on diet, age, and reproductive state (Eisler 1993). Further, metabolic rate, previous zinc exposure, and feeding patterns can change zinc uptake or toxicity (Sorenson 1991). Zinc concentrations in double-crested cormorant eggs ranged from 48.42 to 66.78 (8.04 to 11.31 ppm w.w.). Zinc concentrations in marine bird eggs typically occur at 12 ppm w.w. (Eisler 1993). Zinc concentrations detected in refuge components sampled do not appear to be above levels of concern.

## Fort Niobrara NWR

### Aluminum

Water samples from the Niobrara River and its tributaries collected from the refuge ranged from 3,458 to 14,744 ppm which are much higher than the background levels of 64 ppm (Salomons and Förstner 1984). Further, these concentrations revealed levels exceeding the chronic and acute toxicity value established by the EPA for the protection of aquatic life of 748

ppm and 1,496 ppm, respectively (USEPA 1988). Both the chronic and acute toxicity values are for pH ranges of 6.5-9, and aluminum becomes more toxic in acidic environments (USEPA 1988). Aluminum concentrations in sediment samples collected from the three sampling locations ranged from 9,098 ppm to 17,782 ppm. This range is lower than the average aluminum concentration of 72,000 ppm in soils of the conterminous U.S. (Shacklette and Boerngen 1984).

Aluminum concentrations in aquatic plants ranged from 1,557 ppm to 6,384 ppm. Comparatively, concentrations of aluminum in aquatic mosses collected in a Welsh metal mine drainage contained 54,000 ppm aluminum and in less contaminated reaches the levels of aluminum in the mosses dropped to 2,000-7,000 ppm (Moore 1991). Levels detected from plants collected from the refuge are similar to the less contaminated reaches of the Welsh mine drainage study (Moore 1991). High concentrations of aluminum in plants can result in decreased root growth, increased mucilage production (Crowder 1991), as well as reduction in plant biomass (Parker et al. 1989). Toxic thresholds for aquatic plants are not well documented, although a toxicity threshold has been developed for rice at 300 ppm (Crowder 1991). Concentrations of aluminum in fish from the refuge ranged from 90.94 ppm to 142.05 ppm (22.70 to 33.41 ppm w.w.). This is higher than concentrations detected by Brumbaugh and Kane (1985) whose mean detection was 13.8 ppm w.w.(whole body concentrations) detected in smallmouth bass (Micropterus dolomieui) from a reservoir with lower aluminum concentrations in water. Toxicological effects of aluminum on fish and wildlife remain largely unknown. At this time, it does not appear that concentrations of aluminum detected in refuge components sampled are a concern.

**Arsenic**

Concentrations of arsenic in surface waters collected at Fort Niobrara NWR were all lower than the background levels for surface water of 10 ppb (Eisler 1988a). However, unpolluted river waters in the U.S contain less than 5 ppb, which all samples from this study slightly exceed (Eisler 1988a). Concentrations of arsenic were similar from all sampling locations and ranged from 6 to 8 ppb. These concentrations exceed the 1.4 ppb chronic criteria established for aquatic life in the Nebraska surface water quality standards (NDEQ 1996). Standards established by the EPA for the protection of aquatic life do not include a total arsenic chronic toxicity criteria, but list chronic and acute toxicity criteria for inorganic arsenic (USEPA 1985c). The EPA chronic criteria is 190 ppb $As^{3+}$ and 48 ppb $As^{5+}$), whereas the acute criteria is 360 ppb $As^{3+}$ and 850 ppb $As^{5+}$ (USEPA 1985c). Sediment and aquatic vegetation concentrations were below background levels (ranging from 0.8 to 1.8 ppm and 0.9 to 2.2 ppm, respectively), and arsenic levels in fish were below detection limits. Further, arsenic has a low bioconcentration factor (Weis and Weis 1991). Arsenic concentrations detected in refuge components sampled do not appear to be above levels of concern.

**Barium**

All concentrations in refuge streams ranged from 0.11 to 0.21 ppm which is much lower than the drinking water criteria (1 ppm) for barium established by NDEQ (1996). Water quality standards for aquatic life have not been established for barium. Concentrations of barium in freshwater typically range from 0.007 to 15 ppm (IPCS 1990). Concentrations of barium in

sediment ranged from 83 to 198 ppm, and all sediment samples collected from the refuge were below the mean barium concentration of 440 ppm in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Barium concentrations in sediment are typically less than 100 ppm. Higher concentrations of barium are usually associated with geologic deposits (IPCS 1990).

Barium is not known to accumulate in plants in sufficient quantities to cause toxicity to wildlife (IPCS 1990). Barium concentrations in macrophytes collected from the refuge ranged from 50 to 277 ppm. Concentrations of barium in fish collected from the refuge ranged from 12 to 30 ppm. Studies on the toxicity of barium to fish and wildlife are lacking. At this time, it does not appear that barium concentrations detected in refuge components sampled are at levels of concern.

**Beryllium**

Beryllium was below the limit of detection (0.5 ppm) in all water and fish samples collected from the refuge. The mean beryllium concentration in soils of the conterminous U.S. is 0.63 ppm (Shacklette and Boerngen 1984), and sediments in Illinois ranged from 1.4 to 7.4 ppm (IPCS 1990). Sediment samples collected from the refuge ranged from 0.61 to 1.22 ppm. Beryllium concentrations in aquatic macrophytes collected from the refuge range from 0.11 to 0.42 ppm. Beryllium concentrations detected in refuge components sampled appear to be below levels of concern.

**Boron**

Boron was not detected in water samples from the three lotic systems sampled. Sediment samples ranged from below detection (10 ppm) to 12 ppm, well below the average boron concentration (26 ppm) in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Three of the vegetation samples collected from the refuge contained higher than average concentrations of boron in comparison to a study wherein 22 aquatic macrophytes were sampled. Of the 22 macrophytes, concentrations of boron usually were less than 20 ppm and ranged from 1.2 to 100 ppm (mean 11.3) (Eisler 1990a). Pondweed (Potamogeton spp.) collected from Minnechaduza Creek and the Niobrara River contained 28.02 ppm and 48.26 ppm, respectively. An elevated level of boron (187 ppm) was detected in sago pondweed (Potamogeton pectinatus) collected from Minnechaduza Creek. This concentration exceeded levels fed to mallards (30 ppm w.w.) which produced adverse affects in the growth rates of their ducklings (Smith and Anders 1989). Boron was below the limit of detection in all but one of the fish samples. The white sucker (Catostomus commersoni) composite sample collected from Minnechaduza Creek had detectable levels of boron (11.2 ppm) similar to values in fish from a reference area as reported by Eisler (1990a). Aside from the high concentrations detected in aquatic vegetation, boron concentrations do not appear to be above levels of concern.

**Cadmium**

Cadmium concentrations in all water and sediment samples collected from Fort Niobrara NWR were below the limit of detection. Concentrations of cadmium in aquatic vegetation sampled from refuge streams ranged from below the limit of detection (0.1 ppm) to 0.3 ppm. Background cadmium concentrations in plants are typically less than 1 ppm (Eisler 1985a).

Cadmium concentrations in fish collected on the refuge were below the limit of detection in all but a flathead chub composite sample collected from the Niobrara River which contained 0.2 ppm (0.05 ppm w.w.). This is much lower than the 2.0 ppm w.w. suggested by Eisler (1985a) as a probable level of cadmium contamination. Cadmium concentrations detected in components sampled from the refuge are below levels of concern.

**Chromium**

Chromium was not detected in water collected from three lotic systems and all sediment samples were well below the average concentration of chromium in soils of the conterminous U.S (Shacklette and Boerngen 1984). Similarly concentrations of chromium in aquatic plants and fish sampled were below concentrations that reveal contamination. Chromium concentrations detected in components sampled from the refuge are below levels of concern.

**Copper**

Copper was not detected in water samples from the refuge fluvic systems. Sediment concentrations ranged from 3.7 to 7.1 ppm, well below the mean copper concentration (37 ppm) in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Concentrations of copper in aquatic plants ranged from 7.3 to 30.51 ppm, which is slightly higher than concentrations detected in Alamosa and Monte Vista NWR in Colorado (Archuleta and DeWeese 1992). Concentrations of copper in aquatic macrophytes from uncontaminated areas generally range from 10 to 100 ppm (Stokes 1979). Fish samples collected from the refuge contained copper ranging from 4.6 to 7.63 ppm. While copper is an essential nutrient for fish, it can be toxic at higher levels (Weis and Weis 1991). Copper concentrates in the liver of fish resulting in copper toxicosis (Leland and Kuwabara 1985). Copper concentrations detected in fish from refuge lotic systems revealed concentrations exceeding the NCBP's 85th percentile in all fish (Schmitt and Brumbaugh 1990). Tissue concentrations, especially threshold values for fish are not available. At this time it does not appear that concentrations of copper detected in refuge components sampled are above levels of concern.

**Iron**

Concentrations of iron in surface waters are highly variable and differences reflect the geomorphology of the watershed and lake sediment composition. Iron concentrations in refuge fluvial environments ranged from 0.21 to 0.95 ppm. All samples were below the chronic criteria for the protection of aquatic life of 1 ppm established by NDEQ (1996). All sediment samples contained concentrations that were below average (26,000 ppm) for soils in the conterminous U.S. (Shacklette and Boerngen 1984). Concentrations in aquatic macrophytes collected from the refuge ranged from 1,074 to 4,436 ppm. While iron concentrations are typically high in aquatic plants, toxic effects are rarely observed (Moore 1991). Similarly, concentrations detected in fish would not likely result in any toxicological effects (Moore 1991). Iron is not known to be toxic at levels present in the environment (NRC 1979).

**Lead**

Lead was below the limit of detection in water and fish collected from the refuge. Sediment concentrations ranged from below detection (5 ppm) to 7.5 ppm. This range is below the 16 ppm average for the conterminous U.S. (Shacklette and Boerngen 1984). Aquatic vegetation sampled on the refuge contained lead levels ranging from 1.7 to 3.2 ppm (0.20 to 0.32 ppm w.w.) Plants inhabiting uncontaminated environments generally contain less than 1 ppm w.w. (Pain 1995). Concentrations of lead in vegetation sampled for this study fell below this level. Further, it does not appear that the concentrations detected in aquatic macrophytes would cause problems for waterfowl using the plants as a food source (Eisler 1988b). Lead concentrations detected in refuge components sampled are below levels of concern.

**Magnesium**

Concentrations of magnesium in water from the rivers sampled on the refuge were below the levels detected in refuge lakes on Valentine NWR. Magnesium concentrations in sediment samples were well below the average (9,000 ppm) concentration for soils in the conterminous U.S. (Shacklette and Boerngen 1984). Magnesium concentrations detected in aquatic macrophytes ranged from 1,925 to 3,733 ppm and all chlorophyllous plants require magnesium (Wetzel 1983). Concentrations in fish collected from the refuge contained magnesium concentrations ranging from 1,360 to 1,436 ppm. Studies on magnesium are lacking, and at this time, concentrations detected in refuge components do not appear to be a concern.

**Manganese**

Manganese concentrations in refuge streams ranged from 0.0241 to 0.0635 ppm. All but two of the samples exceeded the MCL of 0.05 ppm for drinking water (Novotny and Olem 1994). However, this criteria is based more on the objectionable taste and staining capacity of this metal (Moore 1991). Because manganese toxicity is inversely related to water hardness, Stubblefield et al. (1997) recommended determination of an IC25 (25% inhibition concentration) based on water hardness (using $e^{0.2064(\ln \text{hardness}) + 7.092}$). For the Niobrara River, the IC25 would be 3.07 ppm manganese based on the hardness of the Niobrara River at Sparks, Nebraska, of 95 mg/l (Bentall 1990). Sediment concentrations sampled from the tributaries were lower than the Niobrara River, and were also similar to the average for soils (330 ppm) in the conterminous U.S. (Shacklette and Boerngen 1984). No biological effects guidelines have been created for manganese in sediment.

Concentrations of manganese in aquatic vegetation ranged from 248 to 1037 ppm. Manganese is an essential micronutrient in plants and assists in nitrate assimilation in photosynthesis (Wetzel 1983). Manganese also tends to saturate metal binding sites in aquatic plants protecting the plants against the effects of more toxic heavy metals (Moore 1991). Concentrations of manganese in fish ranged from 9.66 to 35.76 ppm in refuge streams. Manganese is normally considered the least toxic of the trace elements for poultry and mammals (Pais and Jones 1997) and therefore, not likely a concern to refuge fish and wildlife.

**Mercury**

Mercury was below the limit of detection in water collected from the refuge. Sediment mercury concentrations ranged from below the limit of detection to 0.02 ppm, lower than the average (0.58 ppm) concentration in soils of the conterminous U.S. (Shacklette and Boerngen 1984). Mercury was only detected in one vegetation sample, and at 0.05 ppm was well below concentrations depicting mercury contamination as well as below levels of concern for dietary intake by waterfowl (Eisler 1987). Levels of mercury in fish ranged from 0.22 to 0.26 ppm (0.56 to 0.64 ppm w.w.), which is lower than the national average calculated from the NCBP (Schmitt and Brumbaugh 1990). Concentrations of mercury detected in components of the refuge sampled are below levels of concern.

**Molybdenum**

Molybdenum was below the limit of detection (50 ppb) in water, sediment, and fish samples collected from the refuge. Vegetation samples were below the limit of detection (2 ppm) except for one pondweed sample collected from the Niobrara River containing 2.16 ppm. Concentrations detected in plants are well below dietary levels causing detrimental effects in birds (Eisler 1989). Molybdenum concentrations detected in components sampled from the refuge are below levels of concern.

**Nickel**

Nickel was below the limit of detection in water and fish samples collected from the refuge. Concentrations of nickel in sediment samples ranged from below the limit of detection (0.5 ppm) to 6.41, which is lower than the average for soils (13 ppm) of the conterminous U.S. (Shacklette and Boerngen 1984). Nickel concentrations in aquatic macrophytes ranged from 1.68 to 7.39 ppm (0.18 to 0.92 ppm w.w.). This range is similar to aquatic macrophytes inhabiting uncontaminated areas, which contain nickel concentrations less than 6 ppm w.w. (Jenkins 1980). Nickel concentrations detected in components sampled from the refuge are below levels of concern.

**Selenium**

Selenium was not detected in water or sediment samples from Fort Niobrara NWR. However, the limit of detection by the contract laboratory for selenium in water is also the current National Water Quality Criteria chronic level of 5 ppb (USEPA 1987a). Aquatic vegetation contained selenium concentrations from below detection (0.5 ppm) to 1 ppm which reveals a slight elevation in comparison to background concentrations of selenium reported for aquatic macrophytes of 0.1 ppm to 0.4 ppm in control areas (Eisler 1985b). Heinz et al. (1989) found dietary concentrations of selenium causing reproductive impairment in mallards were between 4 and 8 ppm. Further, Lemly (1997) recommended food chain items containing 3 ppm selenium or more be considered potentially lethal to fish and aquatic birds when used as a food source. Aquatic vegetation from refuge lotic systems do not appear to be a potential contaminant source to waterfowl using the macrophytes as a food source.

Selenium levels from a composite sample of longnose dace (Rhinichthys cataractae) from Big Beaver Creek contained 6.1 ppm, which exceeds the toxic effect threshold of 4 ppm reported

by Lemly (1996). High levels of selenium can also cause problems in reproduction. Bluegills (Lepomis macrochirus) containing over 6 ppm w.w. produced larvae with edema that did not survive to the swim-up stage (Gillespie and Baumann 1986). All other fish sampled were below this toxic effects threshold. However, the flathead chub (Hybopsis gracilis) sample collected from the Niobrara River had a higher concentration of selenium (0.81 ppm w.w.) than the NCBP national average (0.42 ppm w.w.) and the 85th percentile (0.73 ppm w.w.) (Schmitt and Brumbaugh 1990). Trophic transfer of selenium through benthic organisms exposed to selenium in sediment is believed to be the major route of selenium accumulation in fish. However, selenium was below the limit of detection in sediment at these river sites. The limit of detection for selenium in sediment is 1 ppm and it is possible that low total organic carbon may be the reason for the non-detection of selenium. Van Derveer and Canton (1997) proposed that the level of selenium in sediment is related to total organic carbon and dissolved selenium. The sand substrate found in most Sandhills lotic environments may not provide binding sites for selenium that a more organic based substrate would, yielding selenium levels below the level of detection.

Heinz et al. (1989) found dietary concentrations of selenium causing reproductive impairment in mallards between 8 and 16 ppm. Concentrations detected in plants and fish on the refuge were lower, indicating dietary intake of fish or aquatic macrophytes from the refuge would likely not impair reproduction for waterfowl.

Selenium levels in fish collected from Big Beaver Creek exceed the toxic effect threshold of 4 ppm reported by Lemly (1996), all other components sampled were below levels of concern.

**Strontium**

Strontium concentrations detected in refuge river water ranged from 0.196 to 0.383 ppm. Concentrations of strontium in sediment ranged from 30.7 to 83 ppm. The average concentration of strontium in soils for the conterminous U.S. is 120 ppm (Shacklette and Boerngen 1984). Strontium concentrations in aquatic vegetation ranged from 49 to 217 ppm and in fish from 36 to 82 ppm. Non-radioactive strontium is not know to be toxic (Pais and Jones 1997). Levels detected in refuge components sampled are therefore likely below levels of concern.

**Vanadium**

Vanadium levels in refuge rivers ranged from 0.008 to 0.0139 ppm, well within the levels normally detected in surface waters (below 0.22 ppm) (International Programme on Chemical Safety 1988). Sediment concentrations ranged from 14.43 to 26.56 ppm, and the average concentration for soils of the conterminous U.S. is 58 ppm (Shacklette and Boerngen 1984). Sediment vanadium levels typically range from 20 to 150 ppm (Moore 1991). Concentrations of vanadium detected in aquatic macrophytes collected from refuge streams ranged from 6.93 to 17.51 ppm. Vanadium appears to be relatively nontoxic to plants and concentrations in freshwater plants typically range from 0.10 to 5.7 ppm (Moore 1991). Vanadium also appears to be relatively nontoxic to fish with $LC_{50}$ values ranging from 2.9 to 5.6 ppm (in hard water) (Moore 1991). Concentrations of vanadium detected in fish collected from refuge lakes ranged from 0.83 to 1.68 ppm. The paucity of data available on vanadium toxicity does not allow for clear interpretation of the detected concentrations from refuge components sampled. At this time, it does not appear that vanadium concentrations detected on the refuge are

hazardous to fish and wildlife.

**Zinc**

Concentrations of zinc rarely exceed 0.04 ppm in unpolluted surface water (Eisler 1993). All water samples collected from the refuge were lower, ranging from below the limit of detection (0.01 ppm) to 0.0137 ppm. The proposed zinc criteria for the protection of aquatic life in surface waters is 0.047 ppm (Eisler 1993). Zinc concentrations in sediment collected from the refuge ranged from 16.06 to 31.86 ppm, and were lower than the average concentration for soils (180 ppm) in the conterminous U.S. of 180 ppm (Shacklette and Boerngen 1984).

Aquatic macrophytes contained zinc concentrations ranging from 27.66 to 38.73 ppm. Marginal sublethal effects of dietary zinc concentrations for birds occur starting at 178 ppm (Eisler 1993). Concentrations of zinc in aquatic macrophytes were much lower than this benchmark, but the benchmark was exceeded by a composite sample of longnose dace from Big Beaver Creek. Concentrations of zinc in fish ranged from 77.61 to 198.68 ppm (18.25 to 49.25 ppm w.w.). Two of the concentrations exceeded the national average of 21.7 ppm w.w., and one concentration exceeded the 85th percentile (34.2 ppm w.w.) of fish collected as part of the NCBP in 1984 (Schmitt and Brumbaugh 1984). Increased zinc concentrations in fish tissue can lead to growth retardation, inhibition of spawning, as well as mortality (Sorenson 1991). Zinc concentrations can vary dramatically depending on diet, age, and reproductive state (Eisler 1993). Further, metabolic rate, previous zinc exposure, and feeding patterns can change zinc uptake or toxicity (Sorenson 1991). Aside from the elevated concentration of zinc detected in fish collected from Big Beaver Creek, zinc concentrations detected in refuge components sampled are below levels of concern.

**Organochlorines and PCB Congeners**

**Valentine National Wildlife Refuge**

**PCBs**

Polychlorinated biphenyl (PCB) describes the chemical structure whose base can be used to form 209 compounds or congeners. Only about 150 of these congeners have been used and are now found in the environment (Rice and O'keefe 1995). Further, if the congeners potential toxicity, environmental occurrence, and detection in animal tissue are considered, the number of PCB congeners that could be considered a toxic threat only number about 36 (McFarland and Clarke 1989). PCBs were widely used from their introduction in 1920 until the 1970's when health problem caused by PCBs became evident. In July of 1979, the EPA implemented a ban on PCBs prohibiting their manufacture, processing, distribution, and commerce (Eisler 1986b). PCBs have low water solubilities and therefore high log $K_{ow}$ values. Highly chlorinated congeners of PCBs have the highest log $K_{ow}$ values. Upon entry into aquatic systems the partitioning of PCBs occurs which normally involves adsorption to sediment or other organic matter (Rice and O'keefe 1995). Benthic organisms feeding on PCB contaminated sediment, accumulated lower levels of PCB from sediment with higher concentrations of organic carbon in comparison to PCB contaminated sediment with lower levels of organic carbon (Means and

McElroy 1997).

**Total PCBs**

Total PCBs detected in sediment from refuge lakes ranged from below the limit of detection (0.001) to 1.10 ppm. This is much lower than concentrations found in the Lower Hudson River which ranged from 1 to 15 ppm (Eisler 1986b). Total PCBs detected in fish collected from refuge lakes ranged from 0.016 to 0.22 ppm (0.003 to 0.077 ppm w.w.), below the 0.4 ppm w.w. that has been demonstrated as harmful to teleosts (Eisler 1986b). Mean concentrations of total PCBs detected in the NCBP have decreased since the programs inception in 1976; the mean concentration of total PCBs in 1984 were 0.39, down from 0.89 ppm w.w. in 1976 (Schmitt et al. 1990). Further, the level contained in whole bodies of fishes is considerably lower than the level of PCBs (3 ppm w.w.) in the diets of birds associated with PCB poisoning (Eisler 1986b). Total PCB concentrations detected in double-crested cormorant eggs collected on the refuge ranged from 1.24 to 10.41 ppm (0.21 to 1.86 ppm w.w.). This is lower than mean concentrations of total PCBs detected in double-crested cormorant eggs collected from colonies on Lake Michigan (8 ppm w.w.), and similar to a control site, Lake Winnipegosis (1 ppm w.w.) (Larson et al. 1996). Although concentrations detected on the refuge were much lower than the total PCB concentration of 16 ppm w.w. in eggs associated with PCB poisoning (Tillit et al. 1992). Total PCB concentrations of 0.8 ppm w.w. have resulted in an 8% egg mortality in double-crested cormorant eggs collected from Lake Winnipegosis (Tillit et al. 1992). It appears that the total PCB concentrations detected in refuge components sampled are below levels of concern. However, the differential toxicity in PCB congeners can make interpretation of total PCB concentration meaningless.

**Congener specific PCBs**

Congener specific PCB analyses were completed on samples collected from the refuge. Because PCB congeners have vastly different toxicities, congener specific analysis provides a better indication of PCB contamination (McFarland and Clarke 1989). Further, the reporting of specific Araclors can be hard to interpret as some Araclor mixtures can contain 140 different congeners (Niimi 1996). Individual PCB congeners that have the greatest potential to bioaccumulate are those containing five to seven chlorine atoms per molecule (the penta-, hexa-, and heptachlorobiphenyls). Polychlorinated biphenyls that are more highly chlorinated are not generally bioavailable as they are more tightly bound to soils and sediments as well as being less frequently detected in the environment. Further, lesser chlorinated PCBs are unlikely to bioaccumulate as they are metabolized and eliminated more readily (McFarland and Clarke 1989).

McFarland and Clarke (1989) delineated 36 PCB congeners that were of greatest concern as environmental contaminants based on their potential toxicity, frequency of occurrence in the environment, and abundance in tissue. Toxicity of PCB congeners can also be related to how closely they approach the molecular configuration of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (Rice and O'Keefe 1995). Quantifying the toxicity of PCB congeners is now possible through the use of toxic equivalency factors (TEFs) based on their relative potency to 2,3,7,8-TCDD. However, the coelution that occurred in the congener specific PCB analyses

completed on refuge samples does not allow the use of TEFs. Of the twelve PCB congeners identified as exhibiting dioxin like toxicity, only six were included in the congener specific PCB analysis. Further, of the six PCB congeners that were included, only three were not coeluted; PCB 105, 167, and 189. All three of these congeners were below the limit of detection in sediment and fish samples collected from the refuge. Congeners 105 and 167 were detected in double-crested cormorant eggs and concentrations ranged from 0.06 to 0.22 ppm and 0.013 to 0.39 ppm respectively. These congeners are not highly toxic to birds and are likely below levels of concern.

**Chlorinated Hydrocarbon Insecticides**

The chlorinated hydrocarbons includes three distinct groups of compounds; DDT and its analogs, the cylclodienes, and benzene hexachloride isomers. They are characterized by their apolarity, lipophilicity, and their stability in the environment (Matsumura 1985).

**BHC (Benzene Hexachloride)**

The insecticidal properties of benzene hexachloride's four (of the eight possible) isomers were first discovered in 1942. The gamma isomer was named lindane after the discoverer, Van der Linden. The gamma isomer is 50 - 10,000 times more toxic than the other isomers, and the toxicity of BHC is proportional to the amount of the gamma isomer (Matsumura 1985). The insecticide lindane contains at least 99% of the gamma isomer (Blus et al. 1984). The four isomers of BHC affect the central nervous system differently. The alpha and gamma isomers are stimulants with the most prevailing symptom being convulsions, whereas the beta and delta isomers are depressants (Matsumura 1985). Technical BHC consists of 65-70% of the alpha isomer (a pair of optical isomers), 5-6% of the beta isomer, 13% of the gamma isomer, and 6% of the delta isomer (Matsumura 1985). Because several of the BHC's isomers have a musty odor resulting in a bad flavor in fruits and vegetables after application, this lead to voluntarily cancellation by the manufacturer in 1978. Lindane however, is odorless and tasteless (Blus 1995) and it is still widely used on seeds, hardwood lumber, and livestock (Wiemeyer 1996).

Gamma BHC (lindane) was detected in the sediments of three of the lakes sampled with concentrations ranging from 0.0001 to 0.0016 ppm. Comparatively, concentrations of gamma BHC in sediments sampled in the South Platte River Basin study were below the limit of detection (0.001 ppm) (Tate and Heiny 1996). Gamma BHC was detected in fish collected from Hackberry Lake and Pelican Lake where fish contained 0.0014 and 0.0011 ppm (0.0005 and 0.0003 ppm w.w.), respectively. These levels are lower than those found during the NCBP, where levels detected were 0.01 ppm w.w., which was also their limit of detection (Schmitt et al. 1990). The National Academy of Sciences, National Academy of Engineering (NAS, NAE 1973) guideline for the whole body concentration of fish for the protection of fish eating wildlife is 0.1 ppm w.w. which none of the detections on the refuge exceeded. Gamma BHC was below the limit of detection in all double-crested cormorant eggs collected. Because lindane is rapidly metabolized to chlorophenols and chlorobenzenes (which are water soluble and readily excreted), birds ingesting lindane treated seeds do not retain the pesticide long (Blus et al. 1984). Lindane appears to be less harmful to wild birds than the previously used seed treatments of heptachlor,

aldrin, or dieldrin (Blus et al. 1984). Concentrations of lindane detected in refuge components sampled are below levels of concern.

The delta isomer comprises only 6% of technical BHC (Matsumura 1985) and was almost below the limit of detection at all sites. Delta BHC was not detected in double-crested cormorant eggs or fish collected from refuge lakes and only one sediment sample (Pelican Lake at 0.0006 ppm) contained detectable levels of delta BHC. The delta isomer concentrations detected on the refuge are below levels of concern.

Technical BHC contains 5-6% of the beta isomer (Matsumura 1985). The beta isomer was not detected in fish collected from refuge lakes and was detected in only one sediment sample from West Long Lake (0.0012 ppm). Concentrations of beta BHC detected in double-crested cormorant eggs ranged from 0.01 to 0.013 ppm (0.001 to 0.002 ppm w.w.). This range is below levels detected in eggs (up to 10 ppm) exhibiting normal reproduction (Wiemeyer 1996). The beta isomer appears to be present at concentrations lower than levels of concern.

The alpha isomer of BHC makes up the greatest proportion of technical BHC, typically 65-70% (Matsumura 1985). Alpha BHC was not detected in sediment samples collected from the refuge and only one double-crested cormorant egg had a concentration above detection (0.013 ppm). Two of the composite fish samples collected from Hackberry and Pelican Lake also had detectable levels of the alpha isomer, but were below levels of concern.

**HCB (Hexachlorobenzene)**

Hexachlorobenzene is a fungicide used to control fungal diseases in seed grains (Wiemeyer 1996) and its main use was for the treatment of wheat seeds intended for planting. Seeds treated with HCB were excluded for use as animal feed or human consumption (USEPA 1980c). Production of HCB was discontinued in 1976 but is still largely produced as a byproduct in the manufacture of rubber for tires (Wiemeyer 1996). It is also present as a byproduct in perchloroethylene, carbon tetrachloride, trichloroethylene, and other chlorinated hydrocarbons (USEPA 1980c). Hexachlorobenzene is a priority pollutant, has the potential to bioaccumulate in aquatic systems, and is persistent (USEPA 1980c).

HCB was not detected in sediment samples collected from refuge lakes and concentrations detected in fish were below the national average of <0.01 ppm w.w. as part of the NCBP (Schmitt et al. 1990). All double-crested cormorant eggs sampled contained detectable levels of HCB ranging from 0.01 to 0.076 ppm (0.002 to 0.012 ppm w.w.). The levels detected in eggs from the refuge were much lower than concentrations found in eggs of Canada geese which appeared normal when residues of HCB reached 2.97 ppm w.w. (Blus et al. 1984). Concentrations of HCB detected in refuge components sampled appear to be below levels of concern.

**DDT and its analogs and metabolites**

The insecticidal activity of DDT was first discovered in 1939 and its usage peaked in 1959 when 78 million pounds were applied. By 1972 almost all uses for DDT were banned.

However, prior to the ban DDT usage had decreased dramatically because of the large scale resistance conferred in many insect pests (Blus 1995). Signs of DDT poisoning in the American cockroach (Periplaneta americana) include uncoordinated movement followed by tremors resulting from repetitive discharges from a single stimulus in the nervous system (Matsumura 1985).

The para-para isomers of DDT, its analogs and metabolites occur more commonly and appear to be responsible for the majority of toxic effects (Blus 1995). The paucity of information on the ortho-para isomers has made interpretations of some of the levels detected difficult to interpret.

**Total DDT**

Several guidelines have been produced on total DDT concentrations. A sediment quality guideline for protection of aquatic communities with levels based on a lowest effects level for the majority of benthic macroinvertebrates and total DDT, should not exceed 0.007 ppm (Persaud et al. 1993). All sediment concentrations on the refuge were below this standard ranging from no detections at Hackberry Lake, to 0.0004 ppm and 0.004 ppm at West Long Lake, 0.0018 and 0.0002 ppm at Marsh Lake, and 0.002 at Pelican Lake. Concentrations of total DDT in fish should not exceed 1 ppm w.w. to protect fish eating wildlife (NAS, NAE 1973). Concentrations in fish collected from the refuge were much lower. Whole fish concentrations ranged from 0.004 and 0.002 ppb w.w. at Dads Lake, 0.02 ppb w.w. at Hackberry Lake, and 0.0034 ppb w.w. at Pelican Lake. Concentrations of total DDT detected in refuge components sampled are below levels of concern.

**p,p'-DDT**

The active ingredient in DDT (p,p'-DDT) is one of the most apolar compounds known to exist and its toxicological effects increase with decreasing temperature. Commercial DDT contains o,p-DDT and p,p'-DDT (Matsumura 1985).

Fish collected from refuge lakes contained levels of DDT below the limit of detection and only one sediment sample (Pelican Lake contained 0.0012 ppm) contained detectable level of DDT. Similarly, only one sediment sample of the 6 collected from the South Platte River basin study contained a detectable level (0.0023 ppm) of DDT (Tate and Heiny 1996). DDT was detected at 0.15 ppm (0.023 ppm w.w.) in only one of the eggs sampled. Comparatively, the average concentration of DDT detected in double-crested cormorant eggs in Lake Huron was 0.22 ppm w.w. (Weseloh et al. 1983). The low detection frequency and low levels of DDT detected on the refuge indicates that DDT continues to weather, and recent or continuing inputs to the refuge are unlikely.

**o,p-DDT**

The o,p-DDT isomer of DDT has a lower toxicity than p,p-DDT and it is the major contaminant of commercial DDT consisting of 10 to 25% (Matsumura 1985). The o,p-DDT is an estrogenic isomer, and gull eggs injected with as little as 2 ppm resulted in feminization of the embryos (Fry and Toone 1981). However, the resulting concentrations in the eggs were not given. The ortho-para isomer of DDT was below the limit of detection in all fish sampled and

was detected in only one sediment sample collected from Marsh Lake (0.0018 ppm). Concentrations found in double-crested cormorant eggs collected on the refuge ranged from below the limit of detection (0.005) to 0.054 ppm. The paucity of data on the toxicological effects of o,p-DDT does not allow for interpretation of the concentrations detected on the refuge. However, the low concentrations detected are likely below levels of concern.

**p,p'-DDE**

The metabolite of DDT, p,p'-DDE, is formed in the presence of alcoholic alkali which declorinates DDT using iron, aluminum, or chromium salts as a catalyst. DDE is also formed by DDT metabolism in insects. Metabolism of DDT is accomplished by DDT-dehydrochlorinase, an enzyme capable of dehydrochlorination. Because DDE is non-toxic to insects (it does not react with the central nervous system), elevated levels of this enzyme are correlated with resistance. Only p,p'-DDT is metabolized by this enzyme, o,p-DDT is not (Matsumura 1985).

Sediment samples collected from the refuge contained DDE samples ranging from below the limit of detection (0.0001 ppm) to 0.0019 ppm. Concentrations detected in sediment in the South Platte River basin were higher than those detected on the refuge ranging from below the limit of detection (0.001 ppm) to 0.067 ppm (Tate and Heiny 1996).

DDE concentrations detected in fish collected from refuge lakes ranged from 0.019 to 0.0075 ppm (0.0063 to 0.0019 ppm w.w.). DDE was detected in 98.2% of fish sampled in the 1984 NCBP, down from 100% in 1976-77, 1978-79, 1980-81. Similarly the mean concentrations of DDE have decreased since the NCBP's inception, decreasing from 0.26 ppm w.w. to 0.19 ppm w.w. in 1984 (Schmitt et al. 1990). Levels detected in fish from the refuge were much lower than the NCBP's national average. Metabolism of DDT in fish is not extensive but studies have indicated microbial activity in the gut capable of dehydrochlorination and the liver of rainbow trout is capable of breaking down DDT to DDE (Matsumura 1985).

Levels of DDE contained in avian eggs are inversely related to eggshell thickness. Concentrations of DDE in double-crested cormorant eggs from 32, 24, and 24 ppm w.w. resulted in egg shell thinning of 11%, 30%, and 15%, respectively (Blus 1996). Highly contaminated eggs of double-crested cormorant eggs collected from Lake Huron contained an average DDE level of 14.5 ppm w.w. (Weseloh et al. 1983). DDE concentrations detected in double-crested cormorant eggs collected on the refuge ranged from 1.05 to 9.77 ppm (0.180 to 1.5 ppm w.w.), which is much lower than concentrations listed for causing eggshell thinning in contaminated eggs collected from Lake Huron. A no-effects level for DDE regarding eggshell thickness has been described for the brown pelican (0.1 ppm) and for the peregrine falcon (2 ppm) (Blus 1996). Therefore, with respect to these levels, it is possible that double-crested cormorant eggs collected on the refuge had some degree of eggshell thinning. However, eggshell thickness was not measured in this study. In addition to the impacts of DDE on eggshell thickness, DDE has a greater impact on eggshell strength and avian productivity (Blus 1996). While DDT and DDD metabolism occurs in birds, DDE remains stable. Further, DDT and DDD metabolism results in some DDE formations (Matsumura 1985). Of the DDT analogs, it appears DDE poses the most significant threat to avian species. Concentrations of p,p'-DDE detected in refuge components sampled are likely below levels of concern.

**o,p-DDE**

The o,p-DDE analog was not detected in sediment samples collected from refuge lakes. This follows the lack of detection in the South Platte River basin study (Tate and Heiny 1996). Concentrations of o,p-DDE in fish ranged from below the limit of detection to 0.0196 ppm (below the limit of detection to 0.006 ppm w.w.), which is similar to the levels detected in fish collected as part of the South Platte River basin study of 0.0006 ppm w.w. (Tate and Heiny 1996). Concentrations of o,p-DDE in double-crested cormorant eggs ranged from below the limit of detection to 0.01 ppm (below the limit of detection to 0.0009 ppm w.w.). The paucity of data on the toxicological effects of o,p-DDE does not allow for interpretation of the concentrations detected on the refuge. However, the low concentrations detected are likely below levels of concern.

**p,p'-DDD**

The insecticide Rhothane or p,p'-DDD is both an analog of DDT as well as a metabolite. DDD does not compare in toxicity to DDT but it is more effective in controlling certain pests. Black fly larvae, leafrollers, and hornworms are controlled better with DDD and it is not as toxic to mammals in comparison to DDT (Matsumura 1985). Nonetheless, DDD is present in almost all animal fat tissue and the major route of DDT to DDD metabolism is through reductive dechlorination (Matsumura 1985).

Concentrations of p,p'-DDD were only detected in the sediment samples from West Long Lake with concentrations of 0.00022 and 0.0025 ppm. Comparatively, DDD concentrations detected in sediment in the South Platte River basin study were similar containing 0.0025 and 0.0022 ppm (Tate and Heiny 1996). Only a common carp (Cyprinus carpio) composite sample from Hackberry Lake contained detectable levels of DDD at 0.012 ppm (0.0047 ppm w.w.). For the NCBP, the average DDD concentration in 1984 was 2.55 ppm w.w. (Schmitt et al. 1990). DDD was detected in two of the double-crested cormorant eggs collected on the refuge with concentrations of 0.012 and 0.014 ppm (0.0017 and 0.0023 ppm w.w.). The average concentration detected in eggs collected from colonies of double-crested cormorants on Lake Huron was 0.17 ppm w.w. (Weseloh et al. 1983). Concentrations of p,p'-DDD in refuge components sampled appear to be below levels of concern.

**o,p-DDD**

The ortho-para isomer of DDD was not detected in sediment samples collected from refuge lakes. This compares to the lack of detection in the South Platte River basin study (Tate and Heiny 1996). Concentrations of o,p-DDD were below the limit of detection in fish except for a common carp composite sample from Hackberry Lake containing 0.002 ppm (0.0002 ppm w.w.). Comparatively, concentrations detected in the South Platte River basin study ranged from below the limit of detection to 0.039 ppm w.w. (Tate and Heiny 1996). Concentrations in double-crested cormorant eggs ranged from below the limit of detection to 0.07 (below the limit of detection to 0.013 ppm w.w.). The paucity of data on the toxicological effects of o,p-DDD does not allow for interpretation of the concentrations detected on the refuge. However, the low concentrations detected are likely below levels of concern.

**Cyclodiene Insecticides**

The cyclodiene insecticides include chlordane, heptachlor, aldrin, dieldrin, isodrin, endrin, and endosulfan. Unlike DDT, the cyclodienes have a positive temperature correlation (i.e., toxicity increases as temperature increases). After exposure to cyclodienes, a characteristic lag period up to several hours will pass before symptoms become apparent. Cyclodiene poisoning manifests itself similar to DDT in that repetitive discharges occur from a single stimulus (Matsumura 1985).

**Chlordane**

The cyclodiene insecticide, chlordane, has been used since the late 1940's and technical chlordane consists of about 45 components (Eisler 1990b). The primary components of chlordane consist of 24% trans-chlordane (gamma chlordane) and 19% cis-chlordane (alpha chlordane), while the remainder of technical chlordane is composed of heptachlor, cis-nonachlor, and trans-nonachlor (Eisler 1990b). Chlordane metabolites include oxychlordane and heptachlor epoxide (Wiemeyer 1996).

Chlordane was introduced in 1947 and was widely used until 1978 when EPA restricted its use. After 1978, limited use on certain crops and pests was allowed until 1983 (Eisler 1990b). During the 40 years that chlordane and heptachlor were registered, over 250 million pounds were used (Cassidy et al. 1994). Chlordane is still used in foreign countries and its global distribution is primarily a result of atmospheric transport (Cassidy et al. 1994). Some chlordane isomers persist in the soil for up to 15 years (Eisler 1990b). Chlordane can be absorbed through the skin, (Matsumura 1985) inhaled, or ingested and it is a mutagen and a carcinogen (USEPA 1980b).

Guidelines for chlordane have been suggested for the protection of benthic macroinvertebrates and fish-eating wildlife. Chlordane components should not exceed 0.007 ppm in sediment (Persaud et al. 1993). All sediment samples collected from the refuge contained lower levels after summation of chlordane components and metabolites. Similarly, the summation of chlordane components and metabolites did not exceed the guideline for the protection of fish-eating wildlife of 100 ppm w.w. concentration in fish (National Academy of Sciences, National Academy of Engineering (NAS, NAE) 1973). Chlordane residues in refuge components sampled are below levels of concern.

**Alpha Chlordane**

Alpha or cis-chlordane is the active ingredient in technical chlordane (Schmitt et al. 1990) and it usually makes up 19% of technical chlordane (Eisler 1990b). The toxicity of the alpha-chlordane isomer is eight times greater than the gamma isomer in bluegills, and the alpha isomer persists longer in fish tissue than the gamma isomer (Johnson and Finley 1980). Alpha-chlordane was below the limit of detection in double-crested cormorant eggs collected on the refuge. Only one sediment sample collected from West Long Lake on the refuge contained alpha-chlordane above the limit of detection (0.005 ppm). In comparison, the South Platte River basin study contained concentrations above the limit of detection and ranged from 0.001 to 0.005 (Tate and Heiny 1996). Concentrations of alpha-chlordane were detected in fish collected from all refuge lakes and ranged from 0.001 to 0.004 ppm (0.0002 to 0.0013 ppm w.w.). These

concentrations were lower than the average for the NCBP of 0.03 ppm w.w. (Schmitt et al. 1990). Alpha chlordane concentrations detected in refuge components sampled appear to be below levels of concern.

**Gamma Chlordane**

Gamma-chlordane or trans-chlordane is not as toxic as alpha-chlordane and technical chlordane is composed of 24% gamma-chlordane (Eilser 1990b). Gamma-chlordane was not detected in double-crested cormorant eggs, or sediment samples collected from the refuge. It was detected in fish from the three lakes sampled with concentrations ranging from 0.0005 to 0.0028 ppm (0.0002 to 0.0007 ppm w.w.). These detected concentrations were well below the national average determined by the NCBP of 0.02 ppm w.w. (Schmitt et al. 1990). Concentrations of gamma chlordane detected in refuge components sampled are likely below levels of concern.

**Cis-Nonachlor**

Cis-nonachlor is a constituent of chlordane and technical chlordane contains approximately 7% of cis and trans-nonachlor (Eisler 1990b). Cis-nonachlor was not detected in sediment samples collected from refuge lakes. Cis-nonachlor was detected in two common carp composite samples collected from two of the three refuge lakes. Concentrations from Hackberry Lake and Pelican Lake were 0.001 and 0.002 ppm (0.0003 and 0.0006 ppm w.w.), respectively. These were much lower than the national average computed by the NCBP for 1984 of 0.02 ppm w.w. (Schmitt et al. 1990). The samples collected from the refuge also contained lower amounts of cis-nonachlor in comparison to common carp sampled from the South Platte River basin of 0.006, 0.008, and 0.009 ppm w.w. (Tate and Heiny 1996). Cis-nonachlor was detected in only one of the eggs collected from the refuge and contained 0.008 ppm (0.0013 ppm w.w.). The concentration detected from the double-crested cormorant egg collected from the refuge was lower than the average concentration of cis-nonachlor detected in great blue heron eggs (0.032 ppm w.w.) collected from the Mississippi River (Custer et al. 1997). The half-life of cis-nonachlor in the tissue of northern gannets (Sula bassanus) was 19.4 years, eight years longer than cis chlordane, and 16 years shorter than oxychlordane (Eisler 1990b). Concentrations of cis-chlordane detected in refuge components sampled are likely below levels of concern.

**Trans-Nonachlor**

Trans-nonachlor is the most persistent component of technical chlordane (Schmitt et al. 1990), and trans and cis-nonachlor comprise about 7% of technical chlordane (Eisler 1990b). Trans-nonachlor is known to accumulate in human adipose tissue at a higher rate than rats because of lower metabolic capacity in humans (Matsumura 1985). The half-life of trans-nonachlor in the brain of birds was 19 days, much less than the chlordane metabolites oxychlordane and heptachlor epoxide (Eisler 1990b).

Trans-nonachlor was below the limit of detection in sediments collected from refuge lakes. It was however, detected in all fish samples collected. Concentrations of trans-nonachlor detected in fish ranged from 0.002 to 0.004 ppm (0.0004 to 0.001 ppm w.w.), lower than the national average of 0.03 ppm w.w. detected by the NCBP (Schmitt et al. 1990). Trans-nonachlor

was also detected in 3 of the 8 double-crested cormorant egg samples with levels ranging from 0.007 to 0.011 ppm (0.0012 to 0.0016 ppm w.w.). Levels of trans-nonachlor detected in great blue heron eggs from British Columbia ranged from 0.014 to 0.062 ppm w.w. and did not appear to be causing detrimental effects (Elliott et al. 1989). Similarly, great blue heron eggs collected from the Mississippi River contained an average of 0.08 ppm w.w. which did not appear to engender any detrimental effects (Custer et al. 1997). In comparison to levels detected in these studies, trans-nonachlor levels in eggs collected from double-crested cormorant colonies on Marsh Lake were lower and therefore not likely to produce detrimental effects.

**Heptachlor and Heptachlor epoxide**

Heptachlor is four to five times more toxic than technical chlordane (Matsumura 1985). Heptachlor does comprise approximately 10% of technical chlordane (Eisler 1990b). Heptachlor was used widely until 1983 when most of its uses were withdrawn (Blus 1995). One of the main uses of heptachlor was for eradication of fire ants (Solenopsis invicta) in the southwestern U.S. This treatment however, resulted in excessive mortality in non-target invertebrates and passerine birds. It has been also used for the treatment of wheat seeds (Blus 1995). Mammals, plants, insects, and soil microorganisms metabolize heptachlor to heptachlor epoxide which is also toxic (Matsumura 1985). The presence of heptachlor epoxide is more often related to the application of heptachlor than the application of technical chlordane (Eisler 1990b). Heptachlor epoxide is more potent than its parent compound (Cassidy et al. 1994). Heptachlor is rarely found in the environment because of its proclivity for epoxidation (Blus et al. 1983). The lack of detection of heptachlor in sediment, double-crested cormorant eggs, and fish sampled from the refuge, and the detection of heptachlor epoxide in biotic and abiotic samples saliently depicts this process.

Heptachlor epoxide was detected in one Pelican Lake sediment sample at a concentration of 0.001. All other samples were below the limit of detection. Heptachlor epoxide was below the limit of detection in all samples collected as part of the South Platte River basin study (the limit of detection was 0.001 ppm) (Tate and Heiny 1996). Common carp composite samples collected from Hackberry and Pelican Lake on the refuge contained 0.005 and 0.001 ppm (0.0018 and 0.00024 ppm w.w.), respectively. These concentrations were much lower than the average concentration (0.01 ppm w.w.) of heptachlor epoxide (including traces of heptachlor) in the NCBP in 1984 (Schmitt et al. 1990). Further, the residues in fish sampled from the refuge were well below the level determined for protection of fish eating wildlife (0.1 ppm w.w.) (NAS, NAE 1973).

Heptachlor epoxide was detected in all double-crested cormorant eggs collected on the refuge and concentrations ranged from 0.009 to 0.055 ppm (0.001 to 0.009 ppm w.w.). Concentrations were higher in great blue heron eggs collected from the upper Mississippi River where the average detection was 0.05 ppm w.w. with no apparent detrimental effects (Custer et al. 1997). Concentrations above 1.5 ppm w.w. adversely affected productivity in American kestrel (Falco sparverius) eggs (Henny et al. 1983). The hatching success of Canada geese (Branta canadensis) was 95% when eggs contained $\leq 1$ ppm w.w., but showed 20% hatching success when the concentration of heptachlor epoxide exceeded 10 ppm w.w. (Blus et al. 1979). It appears that the levels of heptachlor epoxide detected in double-crested cormorant eggs collected from Marsh Lake are below levels of concern. Heptachlor and heptachlor epoxide

concentrations detected in refuge components sampled appear to be below levels of concern.

**Oxychlordane**

Both alpha and gamma chlordane are metabolized to form oxychlordane (Blus et al. 1983). Oxychlordane can also result from the breakdown of heptachlor and trans-nonachlor (Eisler 1990b). Oxychlordane is a more potent epoxide than its parent compound (Cassidy et al. 1994). Oxychlordane was below the limit of detection in all sediment samples. Oxychlordane was detected in only one fish sample collected from the refuge at 0.004 ppm (0.001 ppm w.w.). This was an order of magnitude lower than the national average found by the NCBP in 1984 of 0.01 ppm w.w. (Schmitt et al. 1990). Oxychlordane was detected in all double-crested cormorant eggs collected from the refuge with concentrations ranging from 0.01 to 0.09 ppm (0.002 to 0.014 ppm w.w.). This is lower than concentrations detected in great blue heron eggs collected from the upper Mississippi River (Custer et al. 1997) and British Columbia (Elliot et al. 1989) where no effects were observed. Concentrations of oxychlordane detected in refuge components sampled are likely below levels of concern.

**Mirex**

Mirex is known for its stability and persistence in the environment (Wiemeyer 1996). It was widely used to control fire ants as well as a fire retardant in electronic components, fabrics, and plastics (Eisler 1985c). Although mirex was banned in 1978 it is possible for mirex and its metabolites to persist in the environment for another 600 years (Eisler 1985c). The use of mirex has been associated with birth defects, high death rates, tumors, metabolism disruption, and it has the potential to biomagnify and bioconcentrate (Eisler 1985c).

Mirex was not detected in sediment or fish samples collected from the refuge. Double-crested cormorant eggs collected had detectable levels of mirex in all but one of the eggs with concentrations ranging from 0.009 ppm to 0.1 ppm (0.0016 to 0.017 ppm w.w.). Concentrations of mirex in eggs from birds receiving mirex in their diet appeared normal when concentrations did not exceed 150 ppm w.w. (Wiemeyer 1996). Concentrations detected in the double-crested cormorant eggs collected from the refuge were much lower and are therefore not likely to effect development. Mirex concentrations detected in refuge components sampled are below levels of concern.

**Aldrin**

Aldrin was used primarily as an insecticide and was normally applied using soil injection or aerial methods. In 1974, the U.S. EPA restricted the use of aldrin and dieldrin to termite (Reticulitermes spp.) control by direct soil injection and non-food seed and plant treatments (USEPA 1980d). Soil erosion and sediment transport were the major routes of aldrin addition to aquatic environments (USEPA 1980d). Aldrin is converted readily in plant and animal tissues to its epoxide dieldrin which is more stable and apolar (Matsumura 1985).

Aldrin was only detected in one fish sample from the refuge, and was below the limit of detection in sediment and double-crested cormorant eggs. A common carp composite sample collected from Hackberry Lake contained 0.002 ppm aldrin, lower than the guideline for dieldrin (which has similar toxicity) (USEPA 1980d) for the protection of fish eating wildlife of 0.1 ppm

w.w. (NAS,NAE 1973). Aldrin concentrations detected on the refuge are below levels of concern.

**Dieldrin**

Dieldrin, the epoxide of aldrin, is one of the most persistent chemicals known and was often applied to areas where this persistence was advantageous (Matsumura 1985). Dieldrin was detected in more samples than aldrin.

Concentrations of dieldrin in sediment collected from refuge lakes ranged from mostly below the limit of detection to 0.0002 ppm. These concentrations are lower than the Canadian guideline of 0.002 ppm which can be tolerated by the majority of benthic macroinvertebrates (Persaud et al. 1993). Concentrations in fish collected from refuge lakes ranged from below the limit of detection to 0.003 ppm (below the limit of detection to 0.001 ppm w.w.), which is lower than the 1984 NCBP national average of 0.04 ppm w.w. (Schmitt et al. 1990). These levels are also lower than the fish consumption advisory levels for the protection of fish eating wildlife (0.1 ppm w.w.) (NAS/NAE 1973). Concentrations of dieldrin in double-crested cormorant eggs ranged from 0.007 to 0.091 ppm (0.0013 to 0.014 ppm w.w.). Concentrations on dieldrin leading to reproductive impairment have not been determined but are thought to exceed 1 ppm w.w. for the brown pelican (Peakall 1996). This concentration is greater than those detected in eggs collected from the refuge. Dieldrin concentrations detected in refuge components sampled are below levels of concern.

**Endrin**

Endrin is the endo-endo isomer of dieldrin and is more toxic than either aldrin or dieldrin. However, it is easily degraded by light and heat (Matsumura 1985). Endrin can metabolize to 12-ketoendrin which is more toxic than the parent compound (Blus 1995).

Endrin was below the limit of detection in sediment collected from the refuge lakes except for one Pelican Lake sample containing 0.002 ppm. Similarly, endrin was below the limit of detection (0.002 ppm) in sediment samples collected in a South Platte River Basin study (Tate and Heiny 1996). Concentrations of endrin in fish collected were all below detection except for one composite sample of common carp from Hackberry Lake which contained 0.002 ppm (0.00086 ppm w.w.). Concentrations of endrin from the NCBP ranged from below the level of detection (0.01 ppm w.w.) to 0.22 ppm w.w. in 1984, with detectable concentrations documented at 29% of the stations sampled (Schmitt et al. 1990). Concentrations of endrin in double-crested cormorant eggs were below the limit of detection except for one egg containing 0.026 ppm (0.037 ppm w.w.). The concentration of endrin causing detrimental effects for brown pelicans has been estimated to be 0.5 ppm w.w. (Peakall 1996), which is higher than the level detected from the refuge. Concentrations of endrin detected on the refuge are below levels of concern.

**Fort Niobrara National Wildlife Refuge**

**Total PCBs**

Total PCBs in sediment collected from three lotic systems on the refuge ranged from below the limit of detection (0.0007 ppm) to 0.037 ppm. This is much lower than concentrations

found in the Lower Hudson River which ranged from 1 to 15 ppm (Eisler 1986b). Concentrations in fish collected from the refuge ranged from 0.0544 to 0.294 ppm (0.013 to 0.068 ppm w.w.) well below the 0.4 ppm w.w. that has been demonstrated as harmful to teleosts (Eisler 1986b). Concentrations of total PCBs detected on the refuge appear to be below levels of concern.

**Congener specific PCBs**

McFarland and Clarke (1989) delineated 36 PCB congeners that were of greatest concern as environmental contaminants based on their potential toxicity, frequency of occurrence in the environment and abundance in tissue. Toxicity of PCB congeners can also be related to how closely they approach the molecular configuration of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (Rice and O'Keefe 1995). Quantifying the toxicity of PCB congeners is now possible through the use of toxic equivalency factors (TEFs) based on their relative potency to 2,3,7,8-TCDD. However, the coelution that occurred in the congener specific PCB analyses completed on refuge samples does not allow the use of TEFs. Of the twelve PCB congeners identified as exhibiting dioxin like toxicity, only six were included in the congener specific PCB analysis. Further, of the six PCB congeners that were included, only three were not coeluted; PCB 105, 167, and 189. Only PCB 105 was above the limit of detection in one fish sample from Minnechaduza Creek at 0.0039 ppm. Because of the low concentration detected and the low toxicity of this congener in comparison to other congeners, PCB levels on the refuge appear to be below levels of concern.

**BHC (Benzene Hexachloride)**

The gamma isomer is 50-10,000 times more toxic than the other isomers, and the toxicity of BHC is proportional to the amount of the gamma isomer (Matsumura 1985). The insecticide lindane contains at least 99% of the gamma isomer (Blus et al. 1984). The four isomers of BHC affect the central nervous system differently. The alpha and gamma isomers are stimulants with the most prevailing symptom being convulsions, while the beta and delta isomers are depressants (Matsumura 1985). Technical BHC consists of 65-70% of the alpha isomer (a pair of optical isomers), 5-6% of the beta isomer, 13% of the gamma isomer, and 6% of the delta isomer (Matsumura 1985). Several of the BHC's isomers have a musty odor that imparts a bad flavor in fruits and vegetable after application so the manufacturer voluntarily canceled its use in 1978. Lindane is odorless and tasteless and is still in use (Blus 1996). It is widely used on seed, hardwood lumber, and livestock (Wiemeyer 1996). The beta and delta isomers were not detected in Fort Niobrara NWR.

Gamma BHC was only detected in the sediments of Minnechaduza Creek which contained 0.0001 ppm. Only one fish sample from Minnechaduza Creek contained a detectable level of gamma BHC which was 0.0014 ppm (0.0003 ppm w.w.). This is lower than levels detected in the NCBP study of 0.01 ppm w.w. (Schmitt et al. 1990). The NAS, NAE (1973) guideline for whole fish tissue for the protection of fish eating wildlife is 0.1 ppm w.w., which is much higher than concentrations detected in fish collected from the refuge. Concentrations of gamma BHC detected on the refuge appear to be below levels of concern.

The alpha isomer of BHC typically comprises 65-70% of technical BHC (Matsumura 1985). Alpha BHC was not detected in sediment collected from the refuge and only a composite sample of longnose dace from Big Beaver Creek had a detectable level of 0.002 ppm (0.00025 ppm w.w.). This is lower than many of the levels detected as part of the NCBP in 1984 (Schmitt et al. 1990). The alpha isomer of BHC appears to be below levels of concern in samples collected from the refuge.

**HCB (Hexachlorobenzene)**

HCB was not detected in sediment samples collected from the refuge's rivers and creeks. The white sucker composite sample collected from Minnechaduza Creek contained 0.0015 ppm (0.0005 ppm w.w.). This level was below those reported for the NCBP (Schmitt et al. 1990). Hexachlorbenzene concentrations detected in refuge components sampled appear to be below levels of concern.

**Total DDT**

Guidelines for the protection of aquatic life and fish eating wildlife have been established for total DDT concentrations. The sediment quality guideline (0.007 ppm total DDT) for protection of aquatic communities is based on a lowest effects level for the majority of benthic macroinvertebrates (Persaud et al. 1993). All sediment concentrations on the refuge were well below this guideline. Total DDT concentrations in sediments from refuge rivers ranged from below the limit of detection in Big Beaver Creek, 0.001 ppm in Minnechaduza Creek, and 0.0005 ppm in the Niobrara River.

Concentrations of total DDT in fish should not exceed 1 ppm w.w. to protect fish eating wildlife (NAS, NAE 1973). All concentrations in fish collected from the refuge were much lower than this benchmark. Whole fish concentrations were 0.02, 0.01, and 0.01 ppm w.w. at Minnechaduza Creek, Big Beaver Creek and the Niobrara River, respectively. Concentrations of DDT detected on the refuge are below levels of concern.

**p,p'-DDT**

Two of the three sediment samples collected from the refuge contained low but detectable levels of p,p'-DDT (0.00008 and 0.00016 ppm). Comparatively, only one of 6 sediment samples collected as part of the South Platte River basin study contained a detectable level of p,p'-DDT (0.0023 ppm) (Tate and Heiny 1996). A white sucker composite sample collected from Minnechaduza Creek, had detectable levels of p,p'-DDT (0.0081 ppm, 0.0019 ppm w.w.). This is lower than the average concentration detected as part of the NCBP study of 0.03 ppm w.w. (Schmitt et al. 1990). The low detection frequency and low levels of DDT detected on the refuge indicate that DDT continues to weather, and recent or continuing inputs to the refuge's aquatic systems are unlikely.

**o,p'-DDT**

The ortho para isomer of DDT has a lower toxicity than p,p'-DDT and it is the major contaminant of commercial DDT consisting of 10 to 25% (Matsumura 1985). Sediment samples

collected from the refuge contained o,p'-DDT levels below the limit of detection. Similarly, o,p'-DDT was not detected in the South Platte River basin study (Tate and Heiny 1996). Fish collected from Minnechaduza Creek and Big Beaver Creek on the refuge contained 0.0077 and 0.002 ppm (0.0018 and 0.0005 ppm w.w.), respectively. Levels of o,p'-DDT detected in fish collected as part of the South Platte River basin study were higher ranging from 0.005 to 0.027 ppm w.w. (Tate and Heiny 1996). The o,p'-DDT concentrations detected in the refuge components sampled appear to be below levels of concern.

**p,p'-DDE**

Sediment samples collected from Minnechaduza Creek and the Niobrara River contained p,p'-DDE levels of 0.005 and 0.0002 ppm, respectively. Concentrations detected in sediment in the South Platte River basin were higher than those detected on the refuge ranging from below the limit of detection (0.001 ppm) to 0.067 ppm (Tate and Heiny 1996).

All fish collected on the refuge contained p,p'-DDE ranging from 0.03 to 0.06 ppm (0.008 to 0.014 w.w.). The p,p'-DDE isomer of DDT was detected in 98.2% in the 1984 NCBP study, which was down from 100% in 1976-77, 1978-79, 1980-81. Similarly the mean concentration of p,p'-DDE has decreased since the NCBP's inception from 0.26 ppm in w.w. 1976-77 to 0.19 ppm w.w. in 1984 (Schmitt et al. 1990). Levels detected in fish from the refuge were much lower than the national average. Metabolism of DDT in fish is not extensive but studies have indicated microbial activity in the gut is capable of dehydrochlorination, and the liver of rainbow trout is capable of breaking down DDT to p,p'-DDE (Matsumura 1985). Concentrations of p,p'-DDE detected in refuge components sampled appear to be below levels of concern.

**o,p-DDE**

The ortho, para isomer of DDE was not detected in sediment samples collected from the refuge. Fish concentrations ranged from below the limit of detection to 0.002 ppm (below the limit of detection to 0.0005 ppm w.w.). Tate and Heiny (1996) reported one sample from the South Platte River basin study contained detectable concentrations of o,p-DDE (0.006 ppm). This is higher than concentrations detected in fish samples collected from the refuge. The NCBP did not report o,p-DDE concentrations, and studies reporting this isomer appear lacking. Concentrations of o,p'-DDE detected in refuge components sampled appear to be below levels of concern.

**p,p'-DDD**

Concentrations of p,p'-DDD were detected in the sediment of Minnechaduza Creek and the Niobrara River at 0.0002 and 0.0001 ppm, respectively. Comparatively, p,p'-DDD concentrations detected in sediment in the South Platte River basin study were higher with two sites showing detectable levels of 0.0025 and 0.0022 ppm (Tate and Heiny 1996). All fish samples had detectable levels of p,p'-DDD, ranging from 0.02 to 0.007 ppm (0.005 to 0.001 ppm w.w.). The NCBP national average in 1984 was 2.55 ppm w.w. (Schmitt et al. 1990). The low concentrations of p,p'-DDD detected in refuge components sampled appear to be below levels of concern.

**o,p-DDD**

The ortho, para isomer of DDD levels in sediment samples collected from the refuge ranged from 0.0001 ppm in Minnechaduza Creek to 0.00008 ppm in the Niobrara River. This isomer was not detected in the six sediment samples from the South Platte River basin study (Tate and Heiny 1996). It should be noted however, that the limit of detection was 0.001 ppm (Tate and Heiny 1996). Of all fish collected, a white sucker composite sample from Minnechaduza Creek contained the only detectable level of o,p-DDD at 0.0028 ppm (0.0007 ppm w.w.). Comparatively, the highest concentration detected in the South Platte River basin study was 0.039 ppm w.w. (Tate and Heiny 1996). The low concentrations of o,p'-DDD detected in this study are likely below levels of concern.

**Chlordane**

Guidelines for chlordane have been suggested for the protection of benthic macroinvertebrates and fish-eating wildlife. Chlordane components should not exceed 0.007 ppm in sediment (Persaud et al. 1993). All sediment samples collected contained lower levels after summation of chlordane components and metabolites. Similarly, the summation of chlordane components and metabolites in fish did not exceed the guideline for the protection of fish eating wildlife of 100 ppm w.w. (NAS, NAE 1973). Chlordane levels detected on the refuge are below levels of concern.

**Alpha Chlordane**

Alpha or cis-chlordane is the active ingredient in technical chlordane (Schmitt et al. 1990). Two sediment samples collected from the refuge contained alpha-chlordane above the limit of detection at levels of 0.0003 and 0.0008 ppm. Concentrations above the limit of detection for the South Platte River basin study ranged from 0.001 to 0.005 (Tate and Heiny 1996).

Alpha-chlordane was detected in fish collected from all refuge sites and ranged from 0.001 to 0.012 ppm (0.0002 to 0.0027 ppm w.w.). These concentrations were lower than the 0.03 ppm w.w. average from the NCBP in 1984 (Schmitt et al. 1990). The toxicity of the alpha-chlordane isomer is eight times greater than the gamma isomer in bluegills (Johnson and Finley 1980). Further, the alpha isomer persists longer in fish tissue than the gamma isomer (Johnson and Finley 1980). The low concentrations of alpha chlordane detected on the refuge appear to be below concern.

**Gamma Chlordane**

Gamma-chlordane or trans-chlordane was detected in two sediment samples collected from the refuge containing (0.0001 and 0.0002 ppm). This is lower than the concentrations detected in the South Platte River basin study (Tate and Heiny 1996). Detected concentrations in fish from the refuge ranged from 0.002 to 0.007 ppm (0.0004 to 0.0017 ppm w.w.), well below the national average determined by the NCBP of 0.02 ppm w.w. in 1984 (Schmitt et al. 1990). The low concentrations of gamma chlordane detected in refuge components sampled appear to be below levels of concern.

**Cis-Nonachlor**

Cis-nonachlor was not detected in sediment samples collected from the refuge. Cis-nonachlor was detected in fish collected from all three rivers sampled on the refuge and ranged from 0.0001 to 0.007 ppm (0.0002 and 0.0016 ppm w.w.). This is much lower than the 0.02 ppm w.w. NCBP national average for 1984 (Schmitt et al. 1990). The samples collected from the refuge also contained lower amounts of cis-nonachlor in comparison to concentrations in common carp (0.006, 0.008, and 0.009 ppm w.w.) sampled from the South Platte River basin (Tate and Heiny 1996). The low concentrations detected in refuge components sampled appear to be below levels of concern.

**Trans-Nonachlor**

Trans-nonachlor was below the limit of detection in two of the three sediment samples collected from refuge. Minnechaduza Creek contained 0.0001 ppm, which is lower than the detected levels of samples collected as part of the South Platte River basin study (Tate and Heiny 1996). Concentrations of trans-nonachlor detected in fish were below the 0.03 ppm w.w. NCBP national average in 1984 (Schmitt et al.1990). The low concentrations of trans-nonachlor detected in refuge components sampled are likely below levels of concern.

**Heptachlor and Heptachlor epoxide**

Heptachlor is rarely found in the environment because of its proclivity for epoxidation (Blus et al. 1983). Heptachlor epoxide was not detected in sediment samples collected from the refuge. A white sucker composite sample from Minnechaduza Creek was the only fish sample containing heptachlor epoxide at 0.001 ppm (0.0003 ppm w.w.). This is much lower than the average concentration (0.01 ppm w.w.) of heptachlor epoxide (including traces of heptachlor) in the NCBP of 1984 (Schmitt et al. 1990). Further, the residues in fish sampled from the refuge were well below the concentration suggested for protection of fish eating wildlife (0.1 ppm w.w.) (NAS, NAE 1973). Concentrations detected on the refuge appear to be below levels of concern.

**Oxychlordane**

Both alpha and gamma chlordane are metabolized to form oxychlordane (Blus et al. 1983). Oxychlordane can also result from the breakdown of heptachlor and trans-nonachlor (Eisler 1990b). Oxychlordane is a more potent epoxide than the parent compound (Cassidy et al. 1994). Oxychlordane was below the limit of detection in all sediment samples, which is similar to the lack of detection in the South Platte River basin study (Tate and Heiny 1996). Oxychlordane was detected in two of the fish collected from the refuge containing 0.003 and 0.001 ppm (0.0003 to 0.0007 ppm w.w.). This is lower than the 0.01 ppm w.w. NCBP national average of 1984 (Schmitt et al. 1990). The low concentrations detected in refuge components sampled from the refuge appear to be below levels of concern.

**Mirex**

Mirex was not detected in sediment samples collected from the refuge. A composite sample of white suckers collected from Minnechaduza Creek was the only fish collected containing concentrations of mirex above the limit of detection with a level of 0.0016 ppm

(0.0004 ppm w.w.). Comparatively, the concentration detected in this study was lower than the NCBP's study average of <0.01 ppm w.w. and a maximum w.w. residue concentration of 0.44 ppm (Schmitt et al. 1990). The concentrations of mirex detected in refuge components sampled appear to be below levels of concern.

**Dieldrin**

Concentrations of dieldrin in sediment collected from the refuge were below the limit of detection except for one sample from the Niobrara River which contained 0.0001 ppm. This is lower than the Canadian guideline of 0.002 ppm which can be tolerated by the majority of benthic macroinvertebrates (Persaud et al. 1993). Concentrations in fish collected from the refuge ranged from below the limit of detection to 0.0029 ppm (below the limit of detection to 0.0007 ppm w.w.). This is also lower than the national average of 0.04 ppm w.w. determined by the NCBP in 1984 (Schmitt et al. 1990), as well as the fish consumption advisory levels for the protection of fish eating wildlife (0.1 ppm w.w.) (NAS/NAE 1973). Dieldrin concentrations detected on the refuge are below levels of concern.

**Endrin**

Endrin was detected (0.0002 ppm) in only one sediment sample which was collected from Minnechaduza Creek. Endrin was below the limit of detection in sediment samples collected in a South Platte River Basin study with a detection limit of 0.002 ppm (Tate and Heiny 1996). Concentrations of endrin in fish collected from the refuge were all below the limit of detection similar to the lack to detection in the NCBP 1984 (Schmitt et al. 1990). The low concentrations of endrin detected on the refuge appear to be below levels of concern.

## SUMMARY

**Inorganics**

Elevated inorganic contaminant concentrations detected in water samples collected from the lakes of Valentine NWR and the rivers and creeks of Fort Niobrara NWR were limited. Only aluminum appeared elevated in water samples collected from Valentine NWR and aluminum and arsenic at Fort Niobrara NWR. The toxicity of these elevated concentrations to biota however could be questionable, as pH, water hardness, temperature, and oxygen concentration can all affect the bioavailability of these metals.

Concentrations of inorganics contained in the sediments from Valentine NWR were in some cases elevated. However, the actual toxicity of the detected metals is dependent on many parameters not tested. Measurements of water hardness, pH, Eh, and percent organic carbon in the sediment would provide information on the bioavailability of sediment associated metals (McIntosh 1991). Further, in anoxic sediments, determination of acid-volatile sulfides (AVS) and simultaneously extracted metals (SEM) for calculation of the SEM/AVS ratio would help determine bioavailability of some metals (i.e., Cu, Cd, Ni, Pb, and Zn). An SEM/AVS ratio <1 are generally non-toxic, whereas an SEM/AVS ration >1 are typically toxic (Di Toro et al. 1990, Hare et al. 1994, and Berry et al. 1996).

Elevated concentrations of metals in sediment from Valentine NWR were limited to one of the three sediment samples from Pelican Lake. This sediment sample contained elevated levels in 11 of 19 metals evaluated for this study. However, these metals were not elevated in other media. These elevated readings may warrant further sampling of Pelican Lake to determine if the levels detected in the sample depict a highly contaminated portion of the lake or laboratory error. None of the sediment samples collected from Fort Niobrara NWR appeared to contain elevated concentrations of inorganic contaminants.

Aquatic plants collected as part of this study did show elevated concentrations of inorganics at both refuges. Elevated detections were not frequent and the paucity of studies on concentrations causing detrimental effects to the plants or their consumers made interpretation difficult. Aquatic macrophyte uptake of inorganic contaminants, or in many cases required nutrients or micronutrients, depends on several environmental factors such as pH and Eh. Further, submerged aquatic vegetation accumulates greater amounts of metals than emergent vegetation, and concentrations of trace metals can be 100,000 times greater in aquatic vegetation than in the water (Albers and Camardese 1993). Also exacerbating interpretations of detected concentrations is the potential that metal accumulation in macrophytes may not be representative of the metal availability of the system due to competitive exclusion at the plants binding sites (Wile and Hitchin 1983).

Aquatic plants collected from Valentine NWR showed very limited contamination. Boron was elevated in the one common star duckweed composite sample collected from Marsh Lake. Concentrations of boron and selenium were elevated in aquatic macrophytes collected from Fort Niobrara NWR.

Elevated concentrations of copper, molybdenum, and zinc were detected in fish collected from Valentine NWR. Concentrations of aluminum, copper, selenium, and zinc appeared elevated at Fort Niobrara NWR. None of the concentrations detected in double-crested cormorant eggs appeared elevated.

Future investigations including the collection of benthic macroinvertebrates may be helpful in tracing the trophic transfer of metals in these systems, revealing a potential cause of the elevated metal concentrations detected in some of the fish samples collected as part of this study.

Although elevated levels of inorganic contaminants were detected in this study, the elevated levels were only seen in one or two of the components of the aquatic ecosystem sampled (i.e., an elevated level detected in fish would not be corroborated by elevated levels in water, sediment, or aquatic plants) so it does not appear that the levels of metal detected are cause for concern in this refuge complex.

**Organochlorines and PCB Congeners**

Concentrations of organochlorines did not appear to be elevated in any of the media sampled from both refuges. The lack of intensive agriculture and absence of industrial development have likely allowed these refuges to remain in fairly pristine condition.

Table 1. Valentine NWR sampling locations, dates and species collected.

| Sample | Location | Common Name | Scientific name | Latitude | Longitude | Date |
|---|---|---|---|---|---|---|
| 95DCC1 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC2 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC3 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC4 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC5 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC6 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC7 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| 95DCC8 | Marsh Lake | double-crested cormorant | Phalacrocorax auritus | 42N35 | 100W31 | 7/21/95 |
| MLP1 | Marsh Lake | arrowhead | Sagittaria | 42N35 | 100W30 | 7/21/95 |
| MLP2 | Marsh Lake | bulrush | Scirpus | 42N35 | 100W30 | 7/21/95 |
| MLP3 | Marsh Lake | common star duckweed | Lemna trisulca | 42N35 | 100W31 | 7/21/95 |
| MLS1 | Marsh Lake | sediment | | 42N35 | 100W30 | 7/21/95 |
| MLS2 | Marsh Lake | sediment | | 42N35 | 100W31 | 7/21/95 |
| MLS3 | Marsh Lake | sediment | | 42N35 | 100W32 | 7/21/95 |
| MLW1 | Marsh Lake | water | | 42N35 | 100W30 | 7/21/95 |
| MLW2 | Marsh Lake | water | | 42N35 | 100W31 | 7/21/95 |
| DALF1 | Dad's Lake | brown bullhead | Ictaluridae nebulosus | 42N30 | 100W40 | 7/21/95 |
| DALF2 | Dad's Lake | brown bullhead | Ictaluridae nebulosus | 42N30 | 100W40 | 7/19/95 |
| DALF3 | Dad's Lake | green sunfish | Lepomis cyanellus | 42N30 | 100W40 | 7/19/95 |
| DALF4 | Dad's Lake | green sunfish | Lepomis cyanellus | 42N30 | 100W40 | 7/19/95 |
| DALP1 | Dad's Lake | arrowhead | Sagittaria | 42N30 | 100W40 | 7/19/95 |
| DALP2 | Dad's Lake | bulrush | Scirpus | 42N30 | 100W41 | 7/19/95 |
| DALP3 | Dad's Lake | bladderwort | Utricularia | 42N30 | 100W42 | 7/19/95 |
| DALS1 | Dad's Lake | sediment | | 42N30 | 100W40 | 7/19/95 |
| DALS2 | Dad's Lake | sediment | | 42N30 | 100W41 | 7/19/95 |
| DALS3 | Dad's Lake | sediment | | 42N30 | 100W42 | 7/19/95 |
| DALW1 | Dad's Lake | water | | 42N30 | 100W40 | 7/19/95 |
| DALW2 | Dad's Lake | water | | 42N30 | 100W41 | 7/19/95 |
| HLF1-6 | Hackberry Lake | common carp | Cyprinus carpio | 42N40 | 100W43 | 7/20/95 |
| HALP1 | Hackberry Lake | arrowhead | Sagittaria | 42N40 | 100W43 | 7/20/95 |
| HALP2 | Hackberry Lake | bulrush | Scirpus | 42N40 | 100W44 | 7/20/95 |
| HALP3 | Hackberry Lake | smartweed | Polygonum | 42N40 | 100W45 | 7/20/95 |
| HALS1 | Hackberry Lake | sediment | | 42N40 | 100W43 | 7/20/95 |
| HALS2 | Hackberry Lake | sediment | | 42N40 | 100W44 | 7/20/95 |
| HALS3 | Hackberry Lake | sediment | | 42N40 | 100W45 | 7/20/95 |
| HALW1 | Hackberry Lake | water | | 42N40 | 100W43 | 7/20/95 |
| HALW2 | Hackberry Lake | water | | 42N40 | 100W44 | 7/20/95 |
| LLP1 | Long Lake | bulrush | Scirpus | 42N36 | 100W46 | 7/21/95 |
| LLP2 | Long Lake | pondweed | Potamogeton | 42N36 | 100W46 | 7/21/95 |
| LLP3 | Long Lake | arrowhead | Sagittaria | 42N36 | 100W46 | 7/21/95 |
| LLS1 | Long Lake | sediment | | 42N36 | 100W46 | 7/21/95 |
| LLS2 | Long Lake | sediment | | 42N36 | 100W47 | 7/21/95 |
| LLS3 | Long Lake | sediment | | 42N36 | 100W48 | 7/21/95 |
| LLW1 | Long Lake | water | | 42N36 | 100W46 | 7/21/95 |
| LLW2 | Long Lake | water | | 42N36 | 100W47 | 7/21/95 |
| PL1-3 | Pelican Lake | common carp | Cyprinus carpio | 42N36 | 100W43 | 7/21/95 |
| PLP1 | Pelican Lake | arrowhead | Sagittaria | 42N36 | 100W43 | 7/21/95 |
| PLP2 | Pelican Lake | arrowhead | Sagittaria | 42N36 | 100W43 | 7/21/95 |
| PLP3 | Pelican Lake | bulrush | Scirpus | 42N36 | 100W44 | 7/21/95 |
| PLS1 | Pelican Lake | sediment | | 42N36 | 100W43 | 7/21/95 |
| PLS2 | Pelican Lake | sediment | | 42N36 | 100W44 | 7/21/95 |
| PLS3 | Pelican Lake | sediment | | 42N36 | 100W45 | 7/21/95 |
| PLW1 | Pelican Lake | water | | 42N36 | 100W43 | 7/21/95 |
| PLW2 | Pelican Lake | water | | 42N36 | 100W44 | 7/21/95 |

Table 2. Fort Niobrara NWR sampling locations, dates and species collected.

| Sample | Location | Common Name | Scientific Name | Latitude | Longitude | Date |
|---|---|---|---|---|---|---|
| NRAFA | Minnechaduza Creek | white sucker | Catostomus commersoni | 42N50 | 100W30 | 7/17/95 |
| NRAP1 | Minnechaduza Creek | pondweed | Potamogeton | 42N50 | 100W30 | 7/17/95 |
| NRAP2 | Minnechaduza Creek | sago pondweed | Potamogeton pectinatus | 42N50 | 100W30 | 7/17/95 |
| NRAS1 | Minnechaduza Creek | sediment | | 42N50 | 100W30 | 7/17/95 |
| NRAS2 | Minnechaduza Creek | sediment | | 42N50 | 100W30 | 7/17/95 |
| NRAW1 | Minnechaduza Creek | water | | 42N50 | 100W30 | 7/17/95 |
| NRAW2 | Minnechaduza Creek | water | | 42N50 | 100W30 | 7/17/95 |
| NRBFA | Big Beaver Creek | longnose dace | Rhinichthys cataractae | 42N48 | 100W26 | 7/18/95 |
| NRBP1 | Big Beaver Creek | arrowhead | Sagittaria | 42N48 | 100W26 | 7/18/95 |
| NRBP2 | Big Beaver Creek | waterweed | Elodea | 42N48 | 100W26 | 7/18/95 |
| NRBS1 | Big Beaver Creek | sediment | | 42N48 | 100W26 | 7/18/95 |
| NRBS2 | Big Beaver Creek | sediment | | 42N48 | 100W26 | 7/18/95 |
| NRBW1 | Big Beaver Creek | water | | 42N48 | 100W26 | 7/18/95 |
| NRBW2 | Big Beaver Creek | water | | 42N48 | 100W26 | 7/18/95 |
| NRCFA | Niobrara River | flathead chub | Hybopsis gracilis | 42N48 | 100W28 | 7/18/95 |
| NRCP1 | Niobrara River | pondweed | Potamogeton | 42N48 | 100W28 | 7/18/95 |
| NRCP2 | Niobrara River | arrowhead | Sagittaria | 42N48 | 100W28 | 7/18/95 |
| NRCS1 | Niobrara River | sediment | | 42N48 | 100W28 | 7/18/95 |
| NRCS2 | Niobrara River | sediment | | 42N48 | 100W28 | 7/18/95 |
| NRCW1 | Niobrara River | water | | 42N48 | 100W28 | 7/18/95 |
| NRCW2 | Niobrara River | water | | 42N48 | 100W28 | 7/18/95 |

Table 3. Concentrations of inorganics detected in water collected from Valentine NWR, in ppm (mg/L)

| Location | Sample | Aluminum | Arsenic | Barium | Boron | Iron | Magnesium | Manganese | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Dad's Lake | DALW1 | < .05 | 0.014 | 0.2302 | 0.1232 | < .1 | 15.3126 | 0.0456 | 0.213 | 0.0035 | 0.0105 |
| Dad's Lake | DALW2 | 0.0787 | 0.013 | 0.2333 | 0.1175 | < .1 | 15.3459 | 0.051 | 0.2129 | 0.0034 | 0.0122 |
| Hackberry Lake | HLW1 | < .05 | 0.009 | 0.1677 | < .1 | 0.1157 | 11.9275 | 0.121 | 0.2821 | < .001 | 0.0103 |
| Hackberry Lake | HLW2 | < .05 | 0.007 | 0.165 | 0.1051 | 0.1036 | 11.7893 | 0.1048 | 0.2793 | < .001 | < .01 |
| Long Lake | LLW1 | 0.1435 | 0.006 | 0.1582 | < .1 | 0.3816 | 7.0281 | 0.0554 | 0.2806 | 0.0011 | < .01 |
| Long Lake | LLW2 | < .05 | 0.006 | 0.1502 | < .1 | 0.208 | 6.9903 | 0.0399 | 0.2759 | < .001 | < .01 |
| Marsh Lake | MLW1 | 0.7306 | 0.014 | 0.2462 | 0.118 | 0.9672 | 16.6381 | 0.1155 | 0.3427 | 0.0024 | 0.0108 |
| Marsh Lake | MLW2 | 4.3716 | 0.017 | 0.3548 | 0.1091 | 2.0932 | 17.6898 | 0.1065 | 0.375 | 0.0061 | 0.0135 |
| Pelican Lake | PLW1 | < .05 | 0.005 | 0.2248 | < .1 | 0.4978 | 8.3985 | 0.1602 | 0.2687 | 0.0012 | < .01 |
| Pelican Lake | PLW2 | 0.0701 | 0.005 | 0.2282 | < .1 | 0.4926 | 8.6169 | 0.2401 | 0.2722 | 0.0011 | < .01 |

Table 4. Concentrations of inorganics detected in sediment collected from Valentine NWR, in ppm (µg/g) dry weight.

| Location | Sample | Aluminum | Arsenic | Barium | Beryllium | Boron | Chromium | Copper | Iron |
|---|---|---|---|---|---|---|---|---|---|
| Dad's Lake | DALS1 | 1187.88 | 0.8 | 18.8 | 0.24 | 10.75 | 0.52 | < 1 | 1030.47 |
| Dad's Lake | DALS2 | 2197.84 | 1.8 | 39.8 | 0.26 | < 10 | < .5 | 1.76 | 1622.94 |
| Dad's Lake | DALS3 | 1438.18 | 0.8 | 18.62 | < .2 | < 10 | < .5 | 1.28 | 1253.43 |
| Hackberry Lake | HLS1 | 1310.85 | < .5 | 14.66 | < .2 | < 10 | < .5 | < 1 | 1023.74 |
| Hackberry Lake | HLS2 | 1386.08 | < .5 | 16.01 | < .2 | < 10 | < .5 | < 1 | 1217.35 |
| Hackberry Lake | HLS3 | 997.31 | < .5 | 16.78 | 1.72 | < 10 | 2.13 | 3.78 | 676.44 |
| Long Lake | LLS1 | 1167.29 | 0.7 | 24.17 | < .2 | < 10 | < .5 | < 1 | 1231.52 |
| Long Lake | LLS2 | 2511.11 | 2.4 | 100.02 | 0.22 | 13.34 | 4.19 | 2.57 | 2530.92 |
| Long Lake | LLS3 | 1406.85 | 0.7 | 27.07 | < .2 | < 10 | < .5 | < 1 | 1441.66 |
| Marsh Lake | MLS1 | 3917.48 | 1 | 111.98 | 0.29 | < 10 | .83 | 1.78 | 2552.75 |
| Marsh Lake | MLS2 | 5485.41 | 1.2 | 78.59 | 0.34 | < 10 | 1.59 | 2.32 | 3838.83 |
| Marsh Lake | MLS3 | 3472.31 | 1 | 58.21 | 0.25 | < 10 | 1.26 | 1.9 | 2838.51 |
| Pelican Lake | PLS1 | 1616.87 | < .5 | 19.8 | 0.37 | 24.99 | 2.12 | 1.87 | 1312.81 |
| Pelican Lake | PLS2 | 2923.3 | .7 | 56.25 | 0.31 | < 10 | 1.44 | 2.15 | 2604.02 |
| Pelican Lake | PLS3 | 3656.83 | 1.5 | 292.26 | 26.34 | 1117.33 | 219.03 | 113.81 | 2402.42 |

Table 4. Concluded

| Location | Sample | Magnesium | Manganese | Mercury | Molybdenum | Nickel | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|
| Dad's Lake | DALS1 | 356.52 | 18.55 | 0.01 | < 5 | < 5 | 6.73 | 2.9 | 6.41 |
| Dad's Lake | DALS2 | 644.21 | 28.5 | 0.01 | < 5 | < 5 | 11.8 | 6.54 | 9.42 |
| Dad's Lake | DALS3 | 364.76 | 15.87 | < .01 | < 5 | < 5 | 5.25 | 4.04 | 5.49 |
| Hackberry Lake | HLS1 | 265.87 | 15.01 | 0.02 | < 5 | < 5 | 6.2 | 2.02 | < 5 |
| Hackberry Lake | HLS2 | 234.75 | 19.2 | 0.01 | < 5 | < 5 | 6.76 | 3.09 | < 5 |
| Hackberry Lake | HLS3 | 281.73 | 24.89 | < .01 | < 5 | < 5 | 5.95 | 4.64 | 8.36 |
| Long Lake | LLS1 | 244.55 | 20.29 | 0.01 | < 5 | < 5 | 8.18 | 3.04 | < 5 |
| Long Lake | LLS2 | 766.86 | 94.27 | 0.03 | < 5 | < 5 | 43.15 | 17.09 | 11.96 |
| Long Lake | LLS3 | 306.87 | 23.24 | 0.02 | < 5 | < 5 | 9.77 | 2.06 | 5.66 |
| Marsh Lake | MLS1 | 737.59 | 48.05 | 0.02 | < 5 | < 5 | 31.26 | 4.84 | 9.43 |
| Marsh Lake | MLS2 | 750.73 | 25.01 | 0.02 | < 5 | < 5 | 21.82 | 6.92 | 10.87 |
| Marsh Lake | MLS3 | 548.9 | 25.99 | 0.03 | < 5 | < 5 | 17.3 | 5.49 | 9.54 |
| Pelican Lake | PLS1 | 395.16 | 24.74 | 0.01 | 6.65 | < 5 | 12.59 | 5.34 | 11.2 |
| Pelican Lake | PLS2 | 546.69 | 40.85 | 0.01 | < 5 | < 5 | 19.51 | 6.69 | 13.67 |
| Pelican Lake | PLS3 | 11442.13 | 1102.6 | 0.03 | 444.46 | 119.33 | 545.02 | 218.99 | 565.16 |

Table 5. Concentrations of inorganics detected in aquatic vegetation collected from Valentine NWR, in ppm (µg/g) dry weight.

| Location | Sample | Genus | Aluminum | Arsenic | Barium | Beryllium | Boron | Cadmium | Chromium | Copper | Iron |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Dad's Lake | DALP1 | Sagittaria | 851.24 | 3.2 | 128.28 | < .1 | 19.41 | < .1 | 7.59 | 2.1 | 620.6 |
| Dad's Lake | DALP2 | Scirpus | 88.57 | 1.8 | 73.25 | < .1 | 9.65 | < .1 | 2.34 | 4.52 | 123.42 |
| Dad's Lake | DALP3 | Utricularia | 658.98 | 2.2 | 453.35 | < .1 | 21.64 | 0.1 | .66 | 8.42 | 452.74 |
| Hackberry Lake | HLP1 | Sagittaria | 1611.78 | 3.5 | 140.39 | 0.13 | 23.98 | 0.1 | 79.39 | 8.28 | 3927.25 |
| Hackberry Lake | HLP2 | Scirpus | 18.27 | 1.1 | 116.93 | < .1 | 7.97 | < .1 | 3.42 | 1.6 | 240.29 |
| Hackberry Lake | HLP3 | Polygonum | 23.52 | 1.2 | 139.4 | < .1 | 21.95 | < .1 | 0.5 | 4.3 | 735.68 |
| Long Lake | LLP1 | Sagittaria | 20.27 | 1.7 | 149.18 | < .1 | 6.08 | < .1 | 2.2 | 0.81 | 149.56 |
| Long Lake | LLP2 | Scirpus | 149.7 | 0.9 | 381.73 | < .1 | 16.31 | < .1 | 1.69 | 4.55 | 563.15 |
| Long Lake | LLP3 | Lemna | 405.73 | 1.2 | 150.34 | < .1 | 16.48 | < .1 | 1.23 | 3.98 | 1159.66 |
| Marsh Lake | MLP1 | Sagittaria | 2302.87 | 14.4 | 216.14 | 0.19 | 21.08 | < .1 | 2.5 | 13.16 | 4906.75 |
| Marsh Lake | MLP2 | Scirpus | 294.6 | 1.5 | 103.11 | < .1 | 16.64 | < .1 | 4.27 | 1.81 | 725.43 |
| Marsh Lake | MLP3 | Lemna | 254.06 | 3.3 | 306.93 | < .1 | 1297.06 | 0.1 | 1.78 | 2.08 | 1026.15 |
| Pelican Lake | PLP1 | Sagittaria | 654.33 | 3.3 | 133.98 | < .1 | 17.39 | < .1 | 1.37 | 2.34 | 3406.55 |
| Pelican Lake | PLP2 | Sagittaria | 489.37 | 2.8 | 105.2 | < .1 | 16.03 | < .1 | 2.17 | 2.35 | 2083.87 |
| Pelican Lake | PLP3 | Scirpus | 51.33 | 0.5 | 159.59 | < .1 | 15.09 | < .1 | < .5 | 0.88 | 134.49 |

Table 5. Concluded

| Location | Sample | Genus | Lead | Magnesium | Manganese | Molybdenum | Nickel | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|---|
| Dad's Lake | DALP1 | Sagittaria | 1.2 | 3323.91 | 164 | < 2 | 5.34 | 67.1 | 8.23 | 40.86 |
| Dad's Lake | DALP2 | Scirpus | < .5 | 1594.13 | 185.32 | < 2 | 2.16 | 26.73 | 1.24 | 23.09 |
| Dad's Lake | DALP3 | Utricularia | 1.8 | 4711.68 | 1958.76 | < 2 | 1.14 | 134.71 | 5.75 | 25.29 |
| Hackberry Lake | HLP1 | Sagittaria | 2 | 2888.42 | 231.11 | 4.13 | 36.71 | 80.21 | 4.63 | 46.5 |
| Hackberry Lake | HLP2 | Scirpus | < .5 | 738.34 | 213.34 | < 2 | 3.16 | 25.47 | 1.56 | 11.62 |
| Hackberry Lake | HLP3 | Polygonum | < .5 | 3070.63 | 210.5 | < 2 | < .5 | 99.6 | 1.97 | 23.02 |
| Long Lake | LLP1 | Sagittaria | < .5 | 1126.5 | 200.38 | < 2 | 2.08 | 35.83 | < .5 | 13.46 |
| Long Lake | LLP2 | Scirpus | < .5 | 5916.98 | 858.05 | < 2 | 0.56 | 214.03 | 1.3 | 15.72 |
| Long Lake | LLP3 | Lemna | 0.7 | 2223.1 | 467.05 | < 2 | 2.5 | 78.86 | 1.86 | 16.91 |
| Marsh Lake | MLP1 | Sagittaria | 2.8 | 3173.2 | 372.49 | < 2 | 3.05 | 88.61 | 5.82 | 60.72 |
| Marsh Lake | MLP2 | Scirpus | < .5 | 1575.5 | 131.13 | 3.23 | 2.26 | 37.61 | 1.03 | 17.92 |
| Marsh Lake | MLP3 | Lemna | 0.9 | 6015.14 | 1981.08 | 5.73 | < .5 | 217.44 | 3.23 | 9.65 |
| Pelican Lake | PLP1 | Sagittaria | 1 | 2225.8 | 382.93 | < 2 | 2.25 | 70.03 | 3.32 | 42.52 |
| Pelican Lake | PLP2 | Sagittaria | 1.1 | 2484.05 | 177.3 | < 2 | 4.4 | 67.64 | 2.47 | 32.85 |
| Pelican Lake | PLP3 | Scirpus | < .5 | 928.67 | 204.74 | < 2 | 0.91 | 27.93 | 1.23 | 13.98 |

Table 6. Concentrations of inorganics detected in fish (whole body) and eggs collected from Valentine NWR, in ppm (µg/g) dry weight.

| Location | Sample | Matrix | Aluminum | Barium | Boron | Chromium | Copper | Iron | Magnesium | Manganese |
|---|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < 5 | 1.96 | < 2 | < .5 | 6.39 | 129.2 | 612.18 | 1.14 |
| Marsh Lake | 95DCC2 | Avian Egg | < 5 | 3.29 | < 2 | 1.18 | 5.92 | 152.71 | 535.17 | 1.33 |
| Marsh Lake | 95DCC3 | Avian Egg | < 5 | 1.89 | < 2 | < .5 | 5.94 | 179.27 | 587 | 1.63 |
| Marsh Lake | 95DCC4 | Avian Egg | < 5 | 4.16 | < 2 | < .5 | 6.06 | 121.5 | 507.73 | < 1 |
| Marsh Lake | 95DCC5 | Avian Egg | < 5 | 5.97 | 3.03 | 1.87 | 5.65 | 118.69 | 495.2 | 1.12 |
| Marsh Lake | 95DCC6 | Avian Egg | < 5 | 3.76 | < 2 | 1.62 | 5.94 | 144.39 | 441.87 | 1.91 |
| Marsh Lake | 95DCC7 | Avian Egg | < 5 | 1.91 | < 2 | < .5 | 5.36 | 131.02 | 518.89 | 1.9 |
| Marsh Lake | 95DCC8 | Avian Egg | < 5 | 4.38 | < 2 | < .5 | 6.85 | 174.22 | 613.62 | 1.94 |
| Dad's Lake | DALF1 | Whole Body | 16.17 | 34.56 | < 2 | < .5 | 1.97 | 120.23 | 1205.78 | 6.59 |
| Dad's Lake | DALF3 | Whole Body | 9.18 | 20.19 | < 2 | < .5 | 4.67 | 57.85 | 1398.9 | 10.81 |
| Hackberry Lake | HLF1-6 | Whole Body | 9.57 | 9.99 | 16.2 | 2.85 | 3.23 | 96.57 | 784.96 | 5.58 |
| Pelican Lake | PLF1-3 | Whole Body | 40.47 | 22.93 | < 2 | < .5 | 3.5 | 106.21 | 1270.31 | 10.95 |

Table 6. Concluded

| Location | Sample | Matrix | Mercury | Molybdenum | Nickel | Selenium | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | 0.42 | < 2 | < .5 | 1.5 | 5.35 | < .5 | 48.42 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.73 | < 2 | 0.55 | 1.4 | 8.25 | < .5 | 47.98 |
| Marsh Lake | 95DCC3 | Avian Egg | 6 | < 2 | 0.61 | 1.4 | 11.61 | 0.53 | 59.14 |
| Marsh Lake | 95DCC4 | Avian Egg | 0.13 | < 2 | 0.77 | 1.6 | 7.71 | 0.77 | 51.15 |
| Marsh Lake | 95DCC5 | Avian Egg | 0.55 | 3.6 | < .5 | 1.4 | 10.78 | < .5 | 50.38 |
| Marsh Lake | 95DCC6 | Avian Egg | 1.05 | 3.27 | 0.62 | 1.8 | 9.29 | 0.74 | 66.03 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.44 | 2.21 | 0.58 | 1.8 | 5.2 | 0.94 | 57.59 |
| Marsh Lake | 95DCC8 | Avian Egg | 0.49 | 2.27 | 1.31 | 1.7 | 8.3 | 1.1 | 66.78 |
| Dad's Lake | DALF1 | Whole Body | < .05 | < 2 | 0.91 | 0.7 | 114.21 | 1.65 | 63.22 |
| Dad's Lake | DALF3 | Whole Body | < .05 | < 2 | 0.66 | 1.3 | 152.34 | 0.76 | 104.72 |
| Hackberry Lake | HLF1-6 | Whole Body | 0.05 | 3.21 | 1.18 | < .5 | 40.91 | 0.87 | 235.16 |
| Pelican Lake | PLF1-3 | Whole Body | < .05 | < 2 | < .5 | 0.7 | 112.22 | 1.2 | 214.29 |

Table 7. Concentrations of inorganics detected in water collected from Fort Niobrara NWR, in ppm (mg/L).

| Location | Sample | Aluminum | Arsenic | Barium | Iron | Magnesium | Manganese | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAW1 | 0.3586 | 0.008 | 0.1589 | 0.2562 | 6.2937 | 0.0635 | 0.2596 | 0.0105 | 0.0137 |
| Minnechaduza Creek | NRAW2 | 0.3548 | 0.007 | 0.1599 | 0.262 | 6.2643 | 0.063 | 0.2608 | 0.0103 | < .01 |
| Big Beaver Creek | NRBW1 | 1.1584 | 0.006 | 0.2177 | 0.6428 | 7.887 | 0.0483 | 0.3827 | 0.009 | 0.0107 |
| Big Beaver Creek | NRBW2 | 0.3549 | 0.006 | 0.1973 | 0.2095 | 7.5848 | 0.0241 | 0.3739 | 0.008 | < .01 |
| Niobrara River | NRCW1 | 1.3857 | 0.007 | 0.1057 | 0.8926 | 5.7408 | 0.0589 | 0.1981 | 0.0139 | < .01 |
| Niobrara River | NRCW2 | 1.4744 | 0.006 | 0.1096 | 0.9545 | 5.8313 | 0.0614 | 0.1964 | 0.0137 | 0.0101 |

Table 8. Concentrations of inorganics detected in sediment collected from Fort Niobrara NWR,in ppm (µg/g) dry weight.

| Location | Sample | Aluminum | Arsenic | Barium | Beryllium | Boron | Chromium | Copper | Iron |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAS1 | 9249.11 | 1 | 102.88 | 0.81 | 10.39 | 6.97 | 5.61 | 7477.66 |
| Minnechaduza Creek | NRAS2 | 8532.42 | 0.9 | 83.18 | 0.86 | 10.35 | 4.99 | 4.51 | 6837.55 |
| Big Beaver Creek | NRBS1 | 17782.72 | 1.8 | 198.48 | 1.22 | 12 | 9.23 | 7.09 | 11263.49 |
| Big Beaver Creek | NRBS2 | 7881.31 | 0.8 | 137.29 | 0.61 | < 10 | 4.1 | 3.67 | 6302.04 |
| Niobrara River | NRCS1 | 10871.3 | 1.1 | 98.98 | 0.81 | < 10 | 6.95 | 4.27 | 8724.63 |
| Niobrara River | NRCS2 | 9098.2 | 0.9 | 90.79 | 0.7 | < 10 | 6.48 | 3.9 | 7833.07 |

Table 8. Concluded

| Location | Sample | Lead | Magnesium | Manganese | Mercury | Nickel | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAS1 | 6.3 | 2312.22 | 315.27 | 0.02 | 5.1 | 54.16 | 21.02 | 27.76 |
| Minnechaduza Creek | NRAS2 | 6.8 | 1923.33 | 287.04 | 0.02 | 5.08 | 30.7 | 19.95 | 24.83 |
| Big Beaver Creek | NRBS1 | 7.5 | 3796.69 | 380.22 | 0.02 | 6.41 | 83.17 | 25.38 | 31.86 |
| Big Beaver Creek | NRBS2 | <.5 | 1846.39 | 177.07 | 0.01 | < 5 | 32.58 | 14.43 | 16.06 |
| Niobrara River | NRCS1 | 5.3 | 2829.02 | 165.87 | 0.02 | < 5 | 35.92 | 26.56 | 24.5 |
| Niobrara River | NRCS2 | 5.1 | 2545.85 | 130.82 | < .01 | < 5 | 33.35 | 23.27 | 22.49 |

Table 9. Concentrations of inorganics detected in aquatic vegetation collected from Fort Niobrara NWR, in ppm (µg/g) dry weight.

| Location | Sample | Genus | Aluminum | Arsenic | Barium | Beryllium | Boron | Cadmium | Chromium |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAP1 | Potamogeton | 3964.84 | 2.2 | 107.9 | 0.27 | 28.02 | 0.3 | 4.48 |
| Minnechaduza Creek | NRAP2 | Potamogeton | 1556.76 | 0.9 | 125.45 | 0.11 | 186.79 | 0.1 | 1.49 |
| Big Beaver Creek | NRBP1 | Sagittaria | 5915.35 | 1.3 | 89.53 | 0.33 | 13.3 | < .1 | 4.84 |
| Big Beaver Creek | NRBP2 | Elodea | 5671.85 | 2.2 | 277.62 | 0.34 | 14.23 | 0.1 | 6.12 |
| Niobrara River | NRCP1 | Potamogeton | 6384.2 | 1.2 | 111.74 | 0.42 | 48.26 | 0.2 | 13.12 |
| Niobrara River | NRCP2 | Sagittaria | 2653.45 | 0.9 | 50.65 | 0.15 | 13.47 | < .1 | 12.36 |

Table 9. Continued

| Location | Sample | Genus | Copper | Iron | Lead | Magnesium | Manganese | Mercury |
|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAP1 | pondweed | 5.99 | 2862.16 | 3.2 | 1925.07 | 1037.63 | < .05 |
| Minnechaduza Creek | NRAP2 | sago pondweed | 4.88 | 1074.84 | 1.7 | 3376.71 | 725.68 | < .05 |
| Big Beaver Creek | NRBP1 | arrowhead | 30.51 | 3886.79 | 2 | 2602.4 | 290.08 | < .05 |
| Big Beaver Creek | NRBP2 | waterweed | 5.06 | 3196.99 | 1.7 | 3566.11 | 669.78 | 0.05 |
| Niobrara River | NRCP1 | pondweed | 7.3 | 4436.7 | 1.7 | 3733.05 | 346.1 | < .05 |
| Niobrara River | NRCP2 | arrowhead | 15.87 | 2270.89 | 1.8 | 2709.91 | 248.38 | < .05 |

Table 9. Concluded

| Location | Sample | Genus | Molybdenum | Nickel | Selenium | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAP1 | pondweed | < 2 | 1.8 | 0.8 | 62.8 | 13.18 | 36.91 |
| Minnechaduza Creek | NRAP2 | sago pondweed | < 2 | 1.68 | 1 | 78.58 | 8.73 | 28.08 |
| Big Beaver Creek | NRBP1 | arrowhead | < 2 | 3.66 | < .5 | 55.06 | 11.74 | 38.73 |
| Big Beaver Creek | NRBP2 | waterweed | < 2 | 6.84 | 0.8 | 217.65 | 14.49 | 29.61 |
| Niobrara River | NRCP1 | pondweed | 2.16 | 7.39 | 1 | 73.3 | 17.51 | 30.04 |
| Niobrara River | NRCP2 | arrowhead | < 2 | 6.54 | < .5 | 49.39 | 6.93 | 27.66 |

Table 10. Concentrations of inorganics detected in fish (whole body) collected from Fort Niobrara NWR, in ppm (µg/g) dry weight.

| Location | Sample | Common name | Aluminum | Barium | Boron | Cadmium | Chromium | Copper | Iron |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | white sucker | 142.05 | 12.53 | 11.29 | < .1 | 0.69 | 4.6 | 173.69 |
| Big Beaver Creek | NRBFA | longnose dace | 91.56 | 30.49 | < 2 | < .1 | 0.93 | 7.63 | 125.24 |
| Niobrara River | NRCFA | flathead chub | 90.94 | 21.03 | < 2 | 0.2 | 1.01 | 5.14 | 112.33 |

Table 10. Concluded

| Location | Sample | Common name | Magnesium | Manganese | Mercury | Selenium | Strontium | Vanadium | Zinc |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | white sucker | 1436.14 | 35.76 | 0.25 | 1.4 | 36.29 | 1.68 | 77.61 |
| Big Beaver Creek | NRBFA | longnose dace | 1529.67 | 11.75 | 0.26 | 6.1 | 82.05 | 0.83 | 198.68 |
| Niobrara River | NRCFA | flathead chub | 1360.89 | 9.66 | 0.22 | 3.2 | 54.99 | 1.59 | 113.29 |

Table 11. Concentrations of chlorinated hydrocarbons detected in sediment from Valentine NWR, in ppm (µg/g) dry weight.

| Location | Sample | alpha chlordane | beta BHC | delta BHC | dieldrin | endrin | gamma BHC | heptachlor epoxid | o,p'-DDT |
|---|---|---|---|---|---|---|---|---|---|
| Hackberry Lake | HLS1 | < .00008176 | < .00008176 | < .00008176 | < .00008176 | < .00008176 | 0.00012225 | < .00008176 | < .00008176 |
| Hackberry Lake | HLS2 | < .00009227 | < .00009227 | < .00009227 | < .00009227 | < .00009227 | < .00009227 | < .00009227 | < .00009227 |
| Hackberry Lake | HLS3 | < .00010485 | < .00010485 | < .00010485 | < .00010485 | < .00010485 | < .00010485 | < .00010485 | < .00010485 |
| Long Lake | LLS1 | < .00009877 | < .00009877 | < .00009877 | < .00009877 | < .00009877 | < .00009877 | < .00009877 | < .00009877 |
| Long Lake | LLS2 | 0.00520733 | 0.00115382 | < .00088591 | < .00088591 | < .00088591 | 0.00158538 | < .00088591 | < .00088591 |
| Marsh Lake | MLS1 | < .00019943 | < .00019943 | < .00019943 | < .00019943 | < .00019943 | 0.00026468 | < .00019943 | 0.00178063 |
| Marsh Lake | MLS2 | < .0000813 | < .0000813 | < .0000813 | 0.00010763 | < .0000813 | < .0000813 | < .0000813 | < .0000813 |
| Pelican Lake | PLS1 | < .000091 | < .000091 | < .000091 | 0.00020253 | < .000091 | < .000091 | < .000091 | < .000091 |
| Pelican Lake | PLS2 | < .00037644 | < .00037644 | 0.0005788 | < .00037644 | 0.00213231 | < .00037644 | 0.00096715 | < .00037644 |

Table 11. Continued

| Location | Sample | p,p'-DDD | p,p'-DDE | p,p'-DDT | PCB# 101 | PCB# 110/77 | PCB# 118/108/149 | PCB# 138 | PCB# 15 |
|---|---|---|---|---|---|---|---|---|---|
| Hackberry Lake | HLS1 | < .00008176 | < .00008176 | < .00008176 | < .00016352 | < .00016352 | < .00016352 | < .00016352 | < .00016352 |
| Hackberry Lake | HLS2 | < .00009227 | < .00009227 | < .00009227 | < .00018454 | < .00018454 | < .00018454 | < .00018454 | < .00018454 |
| Hackberry Lake | HLS3 | < .00010485 | < .00010485 | < .00010485 | < .00020969 | < .00020969 | < .00020969 | < .00020969 | < .00020969 |
| Long Lake | LLS1 | 0.00022000 | 0.00013540 | < .00009877 | < .00019753 | < .00019753 | < .00019753 | < .00019753 | < .00019753 |
| Long Lake | LLS2 | 0.00249667 | 0.00192600 | < .00088591 | < .00177182 | < .00177182 | < .00177182 | < .00177182 | < .00177182 |
| Marsh Lake | MLS1 | < .00019943 | < .00019943 | < .00019943 | < .00039887 | < .00039887 | 0.00072111 | < .00039887 | 0.00055594 |
| Marsh Lake | MLS2 | < .0000813 | 0.00021759 | < .0000813 | < .00016261 | < .00016261 | < .00016261 | < .00016261 | < .00016261 |
| Pelican Lake | PLS1 | < .000091 | < .000091 | < .000091 | < .000182 | < .000182 | < .000182 | < .000182 | < .000182 |
| Pelican Lake | PLS2 | < .00037644 | 0.00081485 | 0.00121846 | 0.00116515 | 0.00124131 | 0.00079200 | 0.00146977 | < .00075288 |

Table 11. Concluded

| Location | Sample | PCB# 153 | PCB# 16/32 | PCB# 170 | PCB# 183 | PCB# 41/64 | PCB# 52 | PCB# 60/56 | PCB-TOTAL |
|---|---|---|---|---|---|---|---|---|---|
| Hackberry Lake | HLS1 | < .00016352 | < .00016352 | 0.00024986 | < .00016352 | 0.13737186 | < .00016352 | 0.00024651 | 0.13802753 |
| Hackberry Lake | HLS2 | < .00018454 | < .00018454 | < .00018454 | < .00018454 | < .00018454 | < .00018454 | 0.00032293 | < .00092272 |
| Hackberry Lake | HLS3 | < .00020969 | < .00020969 | < .00020969 | < .00020969 | 0.00032228 | < .00020969 | 0.00036862 | < .00104847 |
| Long Lake | LLS1 | < .00019753 | < .00019753 | < .00019753 | < .00019753 | 0.00069400 | < .00019753 | < .00019753 | 0.00132200 |
| Long Lake | LLS2 | < .00177182 | < .00177182 | < .00177182 | 0.00183683 | 1.09307568 | < .00177182 | < .00177182 | 1.10254517 |
| Marsh Lake | MLS1 | < .00039887 | < .00039887 | < .00039887 | < .00039887 | 0.05306030 | < .00039887 | < .00039887 | 0.05567887 |
| Marsh Lake | MLS2 | < .00016261 | 0.00055146 | < .00016261 | < .00016261 | 0.00487841 | < .00016261 | < .00016261 | 0.00555776 |
| Pelican Lake | PLS1 | < .000182 | < .000182 | < .000182 | < .000182 | 0.00052000 | < .000182 | 0.00033572 | 0.00140856 |
| Pelican Lake | PLS2 | 0.00121846 | 0.00245215 | < .00075288 | < .00075288 | 0.92779014 | 0.00090623 | 0.00105854 | 0.94558730 |

Table 12. Concentrations of chlorinated hydrocarbons detected in fish (whole body) and eggs from Valentine NWR, in ppm (µg/g) dry weight.

| Location | Sample | Matrix | Aldrin | alpha BHC | alpha chlordane | beta BHC | cis-nonachlor | dieldrin | endrin |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .00522538 | < .00522538 | < .00522538 | < .00522538 | < .00522538 | 0.00740603 | < .00522538 |
| Marsh Lake | 95DCC2 | Avian Egg | < .00547952 | < .00547952 | < .00547952 | 0.01241136 | 0.00841106 | 0.09089938 | < .00547952 |
| Marsh Lake | 95DCC3 | Avian Egg | < .00635229 | < .00635229 | < .00635229 | 0.01231699 | < .00635229 | 0.01646043 | 0.02588055 |
| Marsh Lake | 95DCC4 | Avian Egg | < .00526376 | < .00526376 | < .00526376 | < .00526376 | < .00526376 | 0.02240948 | < .00526376 |
| Marsh Lake | 95DCC5 | Avian Egg | < .00597624 | < .00597624 | < .00597624 | < .00597624 | < .00597624 | 0.02091140 | < .00597624 |
| Marsh Lake | 95DCC6 | Avian Egg | < .00581705 | < .00581705 | < .00581705 | 0.01250730 | < .00581705 | 0.02900763 | < .00581705 |
| Marsh Lake | 95DCC7 | Avian Egg | < .00595238 | 0.01296305 | < .00595238 | < .00595238 | < .00595238 | 0.02781617 | < .00595238 |
| Marsh Lake | 95DCC8 | Avian Egg | < .0070015 | < .0070015 | <.0070015 | 0.01007384 | < .0070015 | 0.01502372 | < .0070015 |
| Dad's Lake | DALF2 | Whole Body | < .00085944 | < .00085944 | 0.00102574 | < .00085944 | < .00085944 | < .00085944 | < .00085944 |
| Dad's Lake | DALF4 | Whole Body | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 |
| Hackberry Lake | HLF1-6 | Whole Body | 0.0020288 | 0.00234526 | 0.00372416 | < .00052884 | 0.00096071 | 0.00335118 | 0.00243850 |
| Pelican Lake | PLF1-3 | Whole Body | < .00073709 | 0.00197269 | 0.00199595 | < .00073709 | 0.00231375 | 0.00106580 | < .00073709 |

Table 12. Continued

| Location | Sample | Matrix | gamma BHC | gamma chlordane | HCB | heptachlor epoxide | mirex | o,p'-DDD | o,p'-DDE |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .00522538 | < .00522538 | 0.01048508 | 0.00940625 | < .00522538 | < .00522538 | 0.00850819 |
| Marsh Lake | 95DCC2 | Avian Egg | < .00547952 | < .00547952 | 0.01926530 | 0.05191272 | 0.04666151 | 0.06930817 | 0.01051545 |
| Marsh Lake | 95DCC3 | Avian Egg | < .00635229 | < .00635229 | 0.03615769 | 0.02321034 | 0.01835862 | < .00635229 | < .00635229 |
| Marsh Lake | 95DCC4 | Avian Egg | < .00526376 | < .00526376 | 0.01524740 | 0.04758308 | 0.00867283 | 0.07134664 | < .00526376 |
| Marsh Lake | 95DCC5 | Avian Egg | < .00597624 | < .00597624 | 0.02678430 | 0.02602692 | 0.10840166 | < .00597624 | 0.01098843 |
| Marsh Lake | 95DCC6 | Avian Egg | < .00581705 | < .00581705 | 0.07601220 | 0.05523587 | 0.04911741 | 0.01759449 | < .00581705 |
| Marsh Lake | 95DCC7 | Avian Egg | < .00595238 | < .00595238 | 0.05812406 | 0.03598416 | 0.03907755 | 0.01566748 | < .00595238 |
| Marsh Lake | 95DCC8 | Avian Egg | < .0070015 | < .0070015 | 0.04047701 | 0.02119215 | 0.04378449 | < .0070015 | 0.00884015 |
| Dad's Lake | DALF2 | Whole Body | < .00085944 | 0.00248636 | < .00085944 | < .00085944 | < .00085944 | < .00085944 | < .00085944 |
| Dad's Lake | DALF4 | Whole Body | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 | < .00086887 |
| Hackberry Lake | HLF1-6 | Whole Body | 0.00139 | 0.00054252 | 0.00145237 | 0.00504089 | < .00052884 | 0.00215312 | 0.01958715 |
| Pelican Lake | PLF1-3 | Whole Body | 0.00109 | 0.00277495 | 0.00079838 | 0.00093402 | < .00073709 | < .00073709 | 0.00565841 |

Table 12. Continued

| Location | Sample | Matrix | o,p'-DDT | oxychlordane | p,p'-DDD | p,p'-DDE | p,p'-DDT | PCB# 101 | PCB# 105 |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .00522538 | 0.01081747 | < .00522538 | 1.05074708 | < .00522538 | 0.01719716 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.04839454 | 0.09078862 | < .00547952 | 9.76959822 | 0.1528324 | 0.02551984 | 0.1014865 |
| Marsh Lake | 95DCC3 | Avian Egg | 0.02389737 | 0.03864376 | 0.01187077 | 6.95672622 | < .00635229 | < .01270457 | < .01270457 |
| Marsh Lake | 95DCC4 | Avian Egg | 0.05373799 | 0.04116518 | < .00526376 | 2.15975918 | < .00526376 | 0.01598039 | < .01052752 |
| Marsh Lake | 95DCC5 | Avian Egg | 0.02746466 | 0.04090495 | < .00597624 | 3.92098341 | < .00597624 | < .01195247 | < .01195247 |
| Marsh Lake | 95DCC6 | Avian Egg | 0.02941008 | 0.04943183 | < .00581705 | 3.9442334 | < .00581705 | < .01163411 | < .01163411 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.02337968 | 0.05022954 | 0.01417245 | 3.07047451 | < .00595238 | < .01190476 | 0.06443846 |
| Marsh Lake | 95DCC8 | Avian Egg | 0.02350815 | 0.09133059 | < .0070015 | 6.95427945 | < .0070015 | 0.02544572 | 0.22867086 |
| Dad's Lake | DALF2 | Whole Body | < .00085944 | < .00085944 | < .00085944 | 0.0190211400 | < .00085944 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00086887 | < .00086887 | < .00086887 | 0.0108178900 | < .00086887 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00052884 | < .00052884 | 0.01207667 | 0.0179285100 | < .00052884 | < .00105769 | < .00105769 |
| Pelican Lake | PLF1-3 | Whole Body | < .00073709 | 0.00427869 | < .00073709 | 0.0074954500 | < .00073709 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 110/77 | PCB# 118/108/149 | PCB# 128 | PCB# 137 | PCB# 138 | PCB# 146 | PCB# 149 |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .01045076 | 0.07701109 | 0.01999046 | < .01045076 | 0.16209883 | < .01045076 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | < .01095904 | 0.23179602 | 0.04066757 | 0.01392288 | 0.44763643 | < .01095904 | < .01095904 |
| Marsh Lake | 95DCC3 | Avian Egg | < .01270457 | 0.43170375 | 0.10347971 | 0.01320942 | 0.74375067 | 0.14067149 | < .01270457 |
| Marsh Lake | 95DCC4 | Avian Egg | < .01052752 | 0.33896228 | 0.06553864 | < .01052752 | 1.03212865 | 0.50464696 | < .01052752 |
| Marsh Lake | 95DCC5 | Avian Egg | < .01195247 | 0.22580202 | 0.03089855 | < .01195247 | 0.55424191 | 0.14026298 | < .01195247 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | 0.42881999 | 0.07064834 | < .01163411 | 0.59117597 | 0.14590595 | < .01163411 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.01599566 | 0.26110246 | 0.05552294 | < .01190476 | 0.4223475 | 0.09766349 | < .01190476 |
| Marsh Lake | 95DCC8 | Avian Egg | < .01400301 | 0.53501094 | 0.07738162 | < .01400301 | 0.88373533 | 0.17917962 | < .01400301 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | < .00105769 | < .00105769 | < .00105769 | < .00105769 | 0.00214464 | 0.00143824 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 153 | PCB# 156/171/202 | PCB# 158 | PCB# 16/32 | PCB# 167 | PCB# 170 | PCB# 172 |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | 0.34194299 | < .01045076 | < .01045076 | < .01045076 | < .01045076 | 0.05400573 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.74852562 | 0.03373545 | < .01095904 | < .01095904 | 0.01681560 | 0.17784406 | 0.01809257 |
| Marsh Lake | 95DCC3 | Avian Egg | 1.19068952 | 0.03019398 | 0.04188060 | < .01270457 | 0.03920330 | 0.23494356 | 0.02502353 |
| Marsh Lake | 95DCC4 | Avian Egg | 3.38402744 | 0.09608380 | 0.04470705 | < .01052752 | 0.02278997 | 0.59411143 | 0.07406040 |
| Marsh Lake | 95DCC5 | Avian Egg | 1.39467087 | 0.05449287 | 0.02673938 | < .01195247 | 0.01347238 | 0.39641156 | 0.05204743 |
| Marsh Lake | 95DCC6 | Avian Egg | 1.43176855 | 0.03649692 | 0.01880183 | < .01163411 | 0.03018982 | 0.28134199 | 0.02387015 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.70913904 | 0.05149364 | 0.04203116 | < .01190476 | 0.03094602 | 0.19890656 | 0.03370515 |
| Marsh Lake | 95DCC8 | Avian Egg | 1.41064784 | 0.05489524 | 0.06512802 | < .01400301 | 0.02069262 | 0.33322384 | 0.03437669 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | 0.00187659 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | 0.00232408 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | < .00105769 | < .00105769 | 0.00138172 | < .00105769 | 0.00127153 | < .00105769 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | 0.00270906 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 177 | PCB# 178 | PCB# 180 | PCB# 183 | PCB# 185 | PCB# 187/182/15 | PCB# 188 |
|---|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .01045076 | < .01045076 | 0.14751420 | 0.01478874 | < .01045076 | 0.05302020 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.01798181 | 0.02175409 | 0.50483945 | 0.08032529 | 0.02962439 | 0.16960239 | < .01095904 |
| Marsh Lake | 95DCC3 | Avian Egg | 0.01623378 | 0.02083051 | 0.57355098 | 0.07434815 | < .01270457 | 0.17868488 | < .01270457 |
| Marsh Lake | 95DCC4 | Avian Egg | < .01052752 | 0.10155048 | 1.09596629 | 0.26241194 | < .01052752 | 0.87672268 | < .01052752 |
| Marsh Lake | 95DCC5 | Avian Egg | < .01195247 | 0.04782407 | 0.86058940 | 0.14774050 | < .01195247 | 0.31573772 | < .01195247 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | 0.03405709 | 0.62642153 | 0.08042026 | <.01163411 | 0.22786682 | < .01163411 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.03006480 | 0.03224050 | 0.42536188 | 0.07966229 | < .01190476 | 0.19403250 | < .01190476 |
| Marsh Lake | 95DCC8 | Avian Egg | < .01400301 | 0.03289324 | 0.86266428 | 0.12706207 | < .01400301 | 0.30091338 | < .01400301 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | < .00105769 | < .00105769 | < .00105769 | < .00105769 | < .00105769 | 0.00986422 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 191 | PCB# 194 | PCB# 195 | PCB# 196 | PCB# 200 | PCB# 201 |
|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .01045076 | 0.02292955 | 0.01082914 | 0.02766474 | < .01045076 | 0.02698246 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.03198939 | 0.08442332 | 0.05360014 | 0.10776710 | < .01095904 | 0.10756513 |
| Marsh Lake | 95DCC3 | Avian Egg | 0.02420193 | 0.08627559 | 0.04237639 | 0.11392684 | 0.01780616 | 0.12931776 |
| Marsh Lake | 95DCC4 | Avian Egg | 0.10885804 | 0.29948131 | 0.12492796 | 0.35446707 | 0.02684102 | 0.31281509 |
| Marsh Lake | 95DCC5 | Avian Egg | 0.05021816 | 0.20820256 | 0.08381247 | 0.24290084 | 0.02195761 | 0.24737452 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | 0.12316140 | 0.07528277 | 0.14896203 | 0.01401019 | 0.15455227 |
| Marsh Lake | 95DCC7 | Avian Egg | < .01190476 | 0.09105738 | 0.04172121 | 0.09828338 | 0.01593489 | 0.09787620 |
| Marsh Lake | 95DCC8 | Avian Egg | 0.03257536 | 0.13685587 | 0.05861900 | 0.17264033 | 0.01895184 | 0.12201380 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | < .00105769 | < .00105769 | < .00105769 | < .00105769 | < .00105769 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 206 | PCB# 209 | PCB# 24 | PCB# 25 | PCB# 28 | PCB# 29 |
|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | 0.01333669 | < .01045076 | < .01045076 | < .01045076 | < .01045076 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.03754681 | 0 .02143484 | < .01095904 | < .01095904 | 0.01149924 | < .01095904 |
| Marsh Lake | 95DCC3 | Avian Egg | 0.04786556 | < .01270457 | < .01270457 | < .01270457 | 0.01789115 | < .01270457 |
| Marsh Lake | 95DCC4 | Avian Egg | 0.08875945 | 0.01231542 | < .01052752 | < .01052752 | < .01052752 | < .01052752 |
| Marsh Lake | 95DCC5 | Avian Egg | 0.06703457 | < .01195247 | < .01195247 | < .01195247 | < .01195247 | < .01195247 |
| Marsh Lake | 95DCC6 | Avian Egg | 0.09049401 | 0.05326136 | < .01163411 | 0.02329792 | < .01163411 | < .01163411 |
| Marsh Lake | 95DCC7 | Avian Egg | 0.03775268 | < .01190476 | < .01190476 | < .01190476 | 0.04853395 | < .01190476 |
| Marsh Lake | 95DCC8 | Avian Egg | 0.07011574 | 0.02059423 | < .01400301 | < .01400301 | < .01400301 | < .01400301 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | < .00105769 | 0.02809224 | < .00105769 | 0.00317034 | 0.00578403 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | 0.00239126 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 40 | PCB# 41/64 | PCB# 44 | PCB# 47/48 | PCB# 60/56 | PCB# 66 |
|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .01045076 | 0.04469279 | < .01045076 | < .01045076 | 0.03035891 | 0.02062610 |
| Marsh Lake | 95DCC2 | Avian Egg | 0.0110106 | < .01095904 | < .01095904 | < .01095904 | 0.03355954 | 0.03320772 |
| Marsh Lake | 95DCC3 | Avian Egg | < .01270457 | 0.01975393 | < .01270457 | < .01270457 | 0.06832777 | 0.04541491 |
| Marsh Lake | 95DCC4 | Avian Egg | < .01052752 | 0.02752925 | < .01052752 | 0.02548134 | 0.04268712 | 0.05222724 |
| Marsh Lake | 95DCC5 | Avian Egg | < .01195247 | < .01195247 | < .01195247 | < .01195247 | 0.03292037 | 0.02754168 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | < .01163411 | < .01163411 | < .01163411 | 0.03080607 | 0.02154979 |
| Marsh Lake | 95DCC7 | Avian Egg | < .01190476 | < .01190476 | < .01190476 | 0.01408129 | 0.01505975 | 0.06436553 |
| Marsh Lake | 95DCC8 | Avian Egg | < .01400301 | < .01400301 | < .01400301 | < .01400301 | 0.04049214 | 0.07266637 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | 0.00838083 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | 0.00616508 | 0.00319619 |
| Hackberry Lake | HLF1-6 | Whole Body | < .00105769 | 0.00328054 | 0.00576143 | < .00105769 | 0.03575531 | < .00105769 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 |

Table 12. Continued

| Location | Sample | Matrix | PCB# 7 | PCB# 74 | PCB# 82 | PCB# 84 | PCB# 87 | PCB# 88 |
|---|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | < .01045076 | < .01045076 | < .01045076 | < .01045076 | < .01045076 | < .01045076 |
| Marsh Lake | 95DCC2 | Avian Egg | < .01095904 | 0.01957151 | < .01095904 | 0.04384696 | < .01095904 | < .01095904 |
| Marsh Lake | 95DCC3 | Avian Egg | < .01270457 | < .01270457 | 0.03528651 | < .01270457 | < .01270457 | < .01270457 |
| Marsh Lake | 95DCC4 | Avian Egg | < .01052752 | 0.03080814 | < .01052752 | < .01052752 | < .01052752 | < .01052752 |
| Marsh Lake | 95DCC5 | Avian Egg | < .01195247 | 0.01480101 | < .01195247 | < .01195247 | < .01195247 | < .01195247 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | 0.03196939 | < .01163411 | < .01163411 | 0.01223062 | < .01163411 |
| Marsh Lake | 95DCC7 | Avian Egg | < .01190476 | 0.07266115 | < .01190476 | < .01190476 | < .01190476 | < .01190476 |
| Marsh Lake | 95DCC8 | Avian Egg | < .01400301 | 0.08773550 | 0.02649776 | < .01400301 | < .01400301 | < .01400301 |
| Dad's Lake | DALF2 | Whole Body | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 | < .00171888 |
| Dad's Lake | DALF4 | Whole Body | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 | < .00173774 |
| Hackberry Lake | HLF1-6 | Whole Body | 0.0014693 | < .00105769 | 0.00537432 | < .00105769 | 0.0018649 | < .00105769 |
| Pelican Lake | PLF1-3 | Whole Body | < .00147418 | < .00147418 | < .00147418 | < .00147418 | < .00147418 | 0.00264705 |

Table 12. Concluded

| Location | Sample | Matrix | PCB# 92 | PCB# 97 | PCB# 99 | PCB-TOTAL | trans-nonachlor |
|---|---|---|---|---|---|---|---|
| Marsh Lake | 95DCC1 | Avian Egg | 0.02173992 | < .01045076 | 0.09623762 | 1.24206184 | < .00522538 |
| Marsh Lake | 95DCC2 | Avian Egg | < .01095904 | 0.01568848 | 0.26453465 | 3.60520404 | < .00547952 |
| Marsh Lake | 95DCC3 | Avian Egg | < .01270457 | 0.03090226 | 0.28384321 | 5.05617015 | < .00635229 |
| Marsh Lake | 95DCC4 | Avian Egg | 0.01171112 | < .01052752 | 0.39635965 | 10.41017463 | 0.00679838 |
| Marsh Lake | 95DCC5 | Avian Egg | 0.03213731 | 0.01658534 | 0.16025653 | 5.71406840 | < .00597624 |
| Marsh Lake | 95DCC6 | Avian Egg | < .01163411 | < .01163411 | 0.33036508 | 5.59115396 | 0.01018065 |
| Marsh Lake | 95DCC7 | Avian Egg | < .01190476 | < .01190476 | 0.12442219 | 3.54241342 | < .00595238 |
| Marsh Lake | 95DCC8 | Avian Egg | 0.03253752 | < .01400301 | 0.29526719 | 6.42384753 | 0.01133023 |
| Dad's Lake | DALF2 | Whole Body | 0.00278888 | < .00171888 | < .00171888 | 0.01582101 | 0.00446694 |
| Dad's Lake | DALF4 | Whole Body | 0.00339567 | < .00173774 | < .00173774 | 0.02038791 | 0.00187875 |
| Hackberry Lake | HLF1-6 | Whole Body | 0.00860682 | 0.10856005 | < .00105769 | 0.21803561 | 0.00348116 |
| Pelican Lake | PLF1-3 | Whole Body | 0.00390275 | < .00147418 | < .00147418 | 0.04598425 | 0.00259279 |

Table 13. Concentrations of chlorinated hydrocarbons detected in sediment from Fort Niobrara NWR, in ppm (µg/g) dry weight.

| Location | Sample | alpha chlordane | dieldrin | endrin | gamma BHC | gamma chlordane | o,p'-DDD | p,p'-DDD |
|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAS2 | 0.00029347 | < .00008789 | 0.00018074 | 0.00009663 | 0.00021474 | 0.00011095 | 0.00017251 |
| Big Beaver Creek | NRBS2 | < .00007937 | < .00007937 | < .00007937 | < .00007937 | < .00007937 | < .00007937 | < .00007937 |
| Niobrara River | NRCS2 | 0.00008005 | 0.00009964 | < .00007419 | < .00007419 | 0.00010839 | 0.00008276 | 0.00009799 |

Table 13. Continued

| Location | Sample | p,p'-DDE | p,p'-DDT | PCB# 138 | PCB# 180 | PCB# 29 | PCB# 41/64 | PCB# 52 |
|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAS2 | 0.00049926 | 0.00016105 | 0.00018253 | 0.00031853 | 0.00109695 | 0.03301042 | 0.00056547 |
| Big Beaver Creek | NRBS2 | < .00007937 | < .00007937 | < .00015874 | < .00015874 | < .00015874 | < .00015874 | < .00015874 |
| Niobrara River | NRCS2 | 0.00021707 | 0.00007628 | < .00014837 | < .00014837 | < .00014837 | 0.00149993 | < .00014837 |

Table 13. Concluded

| Location | Sample | PCB# 60/56 | PCB-TOTAL | trans-nonachlor |
|---|---|---|---|---|
| Minnechaduza Creek | NRAS2 | < .00017578 | 0.03667348 | 0.00012956 |
| Big Beaver Creek | NRBS2 | 0.00019910 | < .00079369 | < .00007937 |
| Niobrara River | NRCS2 | 0.00032561 | 0.00249937 | < .00007419 |

Table 14. Concentrations of chlorinated hydrocarbons in fish (whole body) from Fort Niobrara NWR, in ppm (µg/g), dry weight.

| Location | Sample | alpha BHC | alpha chlordane | cis-nonachlor | dieldrin | gamma BHC | gamma chlordane | HCB | heptachlor epoxide |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | < .00086016 | 0.01179698 | 0.00684874 | 0.00290054 | 0.00141564 | 0.00743751 | 0.00089181 | 0.0012165 |
| Big Beaver Creek | NRBFA | 0.00203305 | 0.00097475 | 0.00093099 | < .00077782 | < .00077782 | 0.00179036 | < .00077782 | < .00077782 |
| Niobrara River | NRCFA | < .00095219 | 0.00097273 | 0.00137964 | 0.00104636 | < .00095219 | 0.00157341 | < .00095219 | < .00095219 |

Table 14. Continued

| Location | Sample | mirex | o,p'-DDD | o,p'-DDE | o,p'-DDT | oxychlordane | p,p'-DDD | p,p'-DDE | p,p'-DDT |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00163643 | 0.00286591 | 0.00220355 | 0.00773622 | 0.00317761 | 0.02017392 | 0.06232269 | 0.0080739 |
| Big Beaver Creek | NRBFA | < .00077782 | < .00077782 | 0.00100658 | 0.00201316 | < .00077782 | 0.00535516 | 0.03025704 | < .00077782 |
| Niobrara River | NRCFA | < .00095219 | < .00095219 | < .00095219 | < .00095219 | 0.00149978 | 0.00720437 | 0.03176278 | < .00095219 |

Table 14. Continued

| Location | Sample | PCB# 101 | PCB# 105 | PCB# 110/77 | PCB# 118/108/149 | PCB# 128 | PCB# 137 | PCB# 138 | PCB# 141 |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00631193 | 0.00395253 | 0.0067232 | 0.00993977 | 0.00351961 | 0.00451532 | 0.01177101 | 0.00289188 |
| Big Beaver Creek | NRBFA | < .00155565 | < .00155565 | 0.00163121 | 0.00301178 | < .00155565 | < .00155565 | 0.00372792 | < .00155565 |
| Niobrara River | NRCFA | < .00190437 | < .00190437 | 0.00218573 | < .00190437 | < .00190437 | < .00190437 | < .00190437 | < .00190437 |

Table 14. Continued

| Location | Sample | PCB# 146 | PCB# 149 | PCB# 153 | PCB# 156/171/202 | PCB# 16/32 | PCB# 170 | PCB# 180 | PCB# 183 |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00387893 | 0.00414734 | 0.01185759 | 0.00339407 | 0.02308745 | 0.00237671 | 0.05803249 | 0.00196977 |
| Big Beaver Creek | NRBFA | < .00155565 | < .00155565 | 0.00262984 | < .00155565 | 0.0017824 | < .00155565 | 0.00307146 | < .00155565 |
| Niobrara River | NRCFA | < .00190437 | < .00190437 | < .00190437 | < .00190437 | 0.00229811 | 0.00563483 | 0.0072470 | < .00190437 |

Table 14. Continued

| Location | Sample | PCB# 187/182/159 | PCB# 41/64 | PCB# 44 | PCB# 49 | PCB# 50 | PCB# 52 | PCB# 60/56 | PCB# 66 |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00188319 | 0.00593529 | 0.00310834 | 0.00172734 | 0.00564523 | 0.04315314 | 0.00545475 | 0.00770592 |
| Big Beaver Creek | NRBFA | < .00155565 | 0.00241897 | 0.00371201 | < .00155565 | < .00155565 | 0.00170283 | 0.00456342 | 0.00406610 |
| Niobrara River | NRCFA | < .00190437 | 0.00315845 | 0.00260427 | < .00190437 | < .00190437 | 0.00566583 | 0.0065688 | 0.00570071 |

Table 14. Continued

| Location | Sample | PCB# 70 | PCB# 74 | PCB# 8 | PCB# 87 | PCB# 88 | PCB# 92 | PCB# 97 | PCB# 99 |
|---|---|---|---|---|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00196111 | < .00172031 | 0.00182691 | 0.00404777 | 0.0040824 | 0.01594432 | 0.01121254 | 0.01106102 |
| Big Beaver Creek | NRBFA | < .00155565 | 0.00271736 | 0.00269747 | 0.00179036 | < .00155565 | 0.0055143 | < .00155565 | < .00155565 |
| Niobrara River | NRCFA | < .00190437 | < .00190437 | < .00190437 | 0.00279416 | < .00190437 | 0.00604175 | 0.00222448 | 0.0022051 |

Table 14. Concluded

| Location | Sample | PCB# UNK | PCB-TOTAL | trans-nonachlor |
|---|---|---|---|---|
| Minnechaduza Creek | NRAFA | 0.00182258 | 0.2942968 | 0.02352902 |
| Big Beaver Creek | NRBFA | < .00155565 | 0.05446664 | 0.00383534 |
| Niobrara River | NRCFA | < .00190437 | 0.05927424 | 0.0074989 |

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