



U. S. Fish and Wildlife Service Region 2



CONTAMINANTS INVESTIGATION OF FAR NORTHWESTERN PORTION OF CADDO LAKE NATIONAL WILDLIFE REFUGE, TEXAS 2002

Project ID No. 94420-03-Y178

(Supplement for Project ID No. 94420-02-Y037)



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ABSTRACT

In December, 2002, a supplemental contaminants investigation was conducted at Caddo Lake National Wildlife Refuge by the United States Fish and Wildlife Service in support of the initial investigation conducted at the Refuge between April and May, 2002 (Project ID No. 94420-02-Y037). The purpose of this supplemental investigation was to determine contaminant (metals, semi-volatile organic compounds, organochlorine pesticides, total polychlorinated biphenyls, and perchlorate) levels in soils at six sites located in the far northwestern portion [approximately 50 acres (20 hectares)] of the Refuge.

Of the six sites sampled, one site contained levels of metals (beryllium and lead) that were high enough to warrant additional investigations. A separate site, contained levels of semi-volatile organic compounds (three of the eight polycyclic aromatic hydrocarbon compounds detected at this site exceeded ecological screening criteria), organochlorine pesticides (primarily total-dichloro-diphenyl-trichloroethane), total polychlorinated biphenyls, and perchlorate that warrant further investigation, while an additional site contained levels of perchlorate that also warrant further investigation. Additional investigations may also be necessary at still another separate site which contained levels of an organochlorine pesticide that exceeded the ecological benchmark value. Neither of the remaining two sites investigated contained contaminant levels where significant adverse impacts to ecological resources would be expected to occur.

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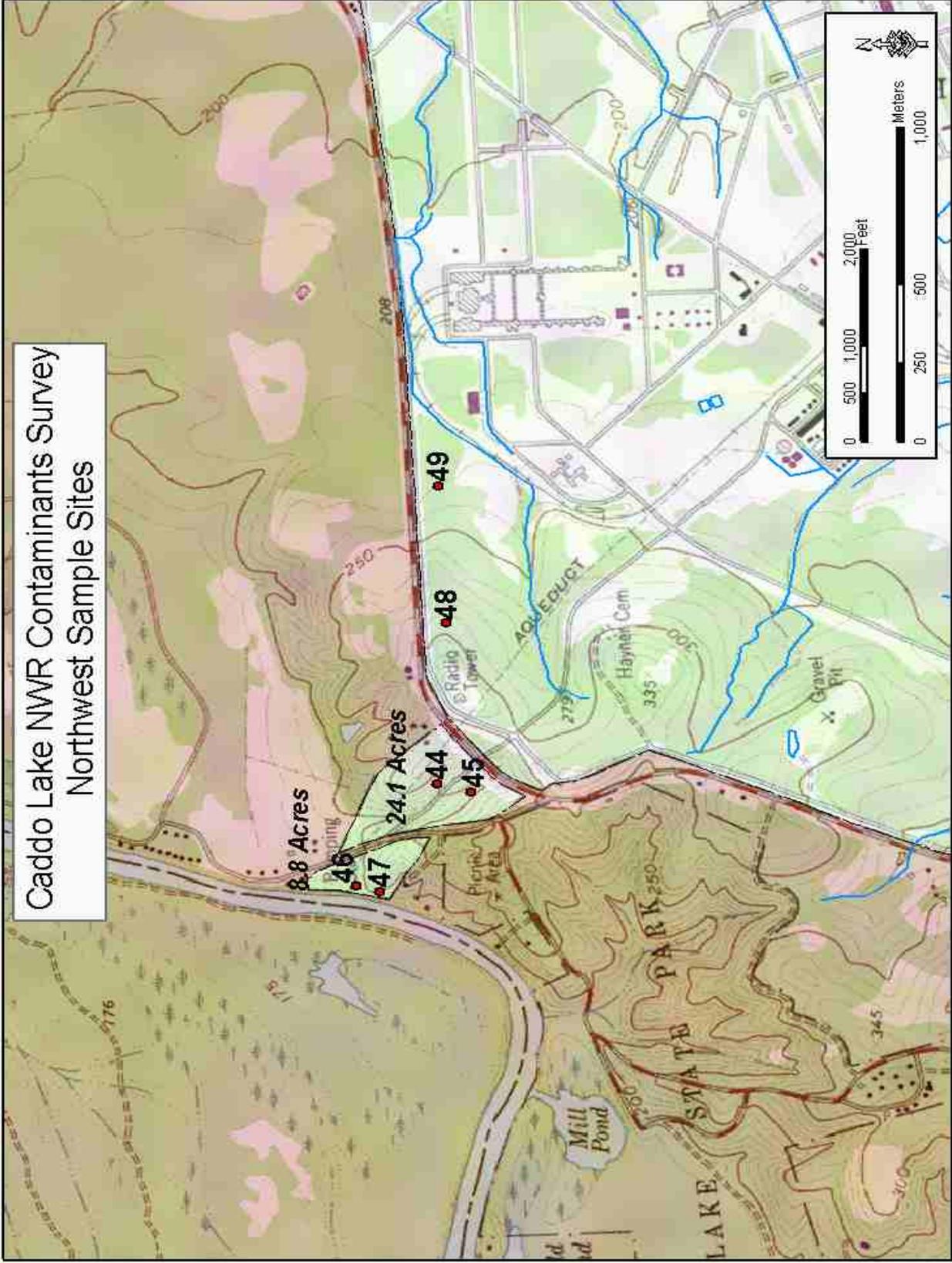
INTRODUCTION

In December, 2002, a contaminants investigation was conducted at Caddo Lake National Wildlife Refuge (CLNWR) by the United States Fish and Wildlife Service (USFWS). The purpose of this investigation was to determine contaminant (metals, semi-volatile organic compounds, organochlorine pesticides, total polychlorinated biphenyls, and perchlorate) levels in soils in the far northwestern portion [approximately 50 acres (20 hectares)] of the Refuge. Data resulting from this investigation would be used by the USFWS to determine the suitability of transfer of administrative control of this portion of the Refuge from the United States Army to the USFWS.

MATERIALS & METHODS

As stated in the completed report for Project ID No. 94420-02-Y037, CLNWR is an overlay refuge located on the site of a former military munitions production facility, Longhorn Army Ammunition Plant, in Harrison County, Texas, southwest of Caddo Lake (Giggleman and Lewis, 2002). The entire site encompasses 8,493 acres (3,437 hectares) of mixed upland pine and bottomland hardwood forests interlaced with remnant structures from the munitions plant. The area is drained by four principal lotic systems, Goose Prairie Bayou, Central Creek, Harrison Bayou, and Saunders Branch, all flowing into Caddo Lake (Giggleman and Lewis, 2002). Surficial soil samples were collected from 43 sites in the western portion of the Refuge [approximately 2,000 acres (810 hectares)] during April and May, 2002 (Project ID No. 94420-02-Y037), and analyzed for metals, semi-volatile organic compounds, organochlorine pesticides, total polychlorinated biphenyls (total-PCBs), dioxin/furans, and perchlorate to confirm that contaminant concentrations were at low enough levels (below ecological screening criteria) to allow for administrative transfer of this portion of the Refuge from the U.S. Army to the USFWS (Giggleman and Lewis, 2002). As a supplement to this sampling, surficial grab soil samples were collected at a depth of 0 to 6 inches [0 to 15 centimeters (cm)] from six sites within the far northwestern portion of CLNWR (Figure 1) by USFWS personnel in December, 2002. Each sample was collected in a pre-cleaned glass container using a disposable plastic scoop and placed on ice in a cooler. These samples were then transported to the USFWS Arlington, Texas Ecological Services Field Office (ESFO) and remained refrigerated at 4°C until submitted through the Patuxent Analytical Control Facility (PACF) to contract laboratories for chemical analyses. Samples from each site were analyzed for moisture, sand, silt, and clay content (as percentages); metals (aluminum, arsenic, barium, beryllium, boron, cadmium, chromium, copper, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, selenium, silver,

Caddo Lake NWR Contaminants Survey
Northwest Sample Sites



strontium, vanadium, and zinc) in milligrams/kilogram (mg/kg) dry weight; semi-volatile organic compounds [1,2,4-trichlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1-chloronaphthalene, 1-naphthylamine, 2,3,4,6-tetrachlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 2,4-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2,4-dinitrotoluene, 2,6-dichlorophenol, 2,6-dinitrotoluene, 2-chloronaphthalene, 2-chlorophenol, 2-methylphenol, 2-naphthylamine, 2-nitroaniline, 2-nitrophenol, 2-picoline, 2-methylnaphthalene, 3,3'-dichlorobenzidine, 3-methylcholanthrene, 3-nitroaniline, 4,6-dinitro-2-methylphenol, 4-aminobiphenyl, 4-bromophenyl-phenylether, 4-chloro-3-methylphenol, 4-chloroaniline, 4-chlorophenyl-phenylether, 4-methylphenol, 4-nitrophenol, 7,12-dimethylbenz(a)anthracene, acetophenone, aniline, benzidine, benzo(a)anthracene, benzoic acid, benzyl alcohol, bis(2-chloroethoxy)methane, bis(2-chloroethyl)ether, bis(2-ethylhexyl)phthalate, bis(2chloroisopropyl)ether, butylbenzylphthalate, carbazole, di-n-butylphthalate, di-n-octylphthalate, dibenz(a,h)anthracene, dibenz(a,j)acridine, dibenzofuran, diethyl phthalate, dimethylphthalate, diphenylamine, ethyl methanesulfonate, hexachlorobutadiene, hexachlorocyclopentadiene, hexachloroethane, isophorone, methyl methanesulfonate, n-nitroso-di-n-propylamine, n-nitrosopiperidine, nitrobenzene, pentachlorobenzene, pentachloronitrobenzene, pentachlorophenol, phenacetin, phenol, pronamide, a,a-dimethylphenylamine, acenaphthalene, acenaphthene, anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, n-nitrosodiphenylamine, naphthalene, p-dimethylaminoazobenzene, phenanthrene, and pyrene] in mg/kg dry weight; organochlorine pesticides [1,2,3,4-tetrachlorobenzene, 1,2,4,5-tetrachlorobenzene, aldrin, hexachlorobenzene (HCB), heptachlor, alpha hexachlorocyclohexane (α BHC), alpha (α) chlordane, beta hexachlorocyclohexane (β BHC), cis-nonachlor, delta hexachlorocyclohexane (δ BHC), dieldrin, endosulfan II, endrin, gamma hexachlorocyclohexane (γ BHC), gamma (γ) chlordane, heptachlor epoxide, mirex, o,p'-dichloro-diphenyl-dichloroethane (o,p'-DDD), o,p'-dichloro-diphenyl-dichloroethylene (o,p'-DDE), o,p'-dichloro-diphenyl-trichloroethane (o,p'-DDT), oxychlordane, p,p'-dichloro-diphenyl-dichloroethane (p,p'-DDD), p,p'-dichloro-diphenyl-dichloroethylene (p,p'-DDE), p,p'-dichloro-diphenyl-trichloroethane (p,p'-DDT), pentachloro-anisole, toxaphene, and trans-nonachlor] in mg/kg dry weight; and total polychlorobiphenyls (PCBs) in mg/kg dry weight (for analytical methods see Appendix A). Additional soil/sediment samples were also collected in the same manner from all six sites and submitted to the Institute of Environmental and Human Health at Texas Tech University to be analyzed for perchlorate content in micrograms/kilogram (μ g/kg) dry weight (for analytical methods see Appendix A). In addition to the sampling, all observed anomalies encountered in the field (i.e., drums, solid waste, foundations, etc.) were documented and their coordinates were entered into a geographical information systems (GIS).

RESULTS & DISCUSSION

Surface anomalies such as drums, discarded pipe, and concrete foundations encountered during the field sampling are presented in Appendix B. The majority of anomalies observed at the far northwestern portion of CLNWR (i.e., concrete slabs, discarded pipe, etc.) appeared to be associated

with former surface water supply operations previously conducted in the area. Other observed physical hazards (broken light posts) were also characterized and their locations documented for future corrective measures.

As would be expected, various metals were detected throughout the sites sampled; however, mercury, molybdenum, selenium, and silver were not detected above their respective analytical detection limits in any of the samples collected. Only one semi-volatile organic compound, aniline, was measured above the analytical detection limit in the sample collected from Site 47 and eight semi-volatile compounds [benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, phenanthrene, and pyrene] were detected at Site 46. None of the soil samples collected from the remaining four sites contained semi-volatile organic compounds above their respective analytical detection limits. Of the 27 organochlorine pesticides analyzed, 16 were detected above the analytical detection limits in the samples collected. In contrast, total-PCBs were measured above the analytical detection limits in soils collected from every site.

Analytical results were compared with available ecological benchmarks developed by Efroymsen *et al.* (1997), the U.S. Environmental Protection Agency (USEPA), the Texas Natural Resource Conservation Commission (TNRCC), and other sources, as well as with data from comparative studies and additional screening criteria such as background values to estimate the possible effects of contamination in soils collected from the northwestern portion of CLNWR. Ecological benchmarks are values derived from toxicity data resulting from multiple studies. These benchmarks are typically based on the degree of toxicity of a given contaminant to plants, earthworms, heterotrophic microbes, and other terrestrial invertebrates and are non-regulatory screening guidelines developed to assist in assessing the degree of contamination in a given area (Efroymsen *et al.*, 1997). For certain contaminants, no ecological benchmarks or screening criteria have been established. In this case, the corresponding analytical results were compared to soil-remedial target values which usually have been developed to address strictly human health concerns.

Moisture, sand, silt, and clay content as percentages for the soil/sediment samples collected from the six sites are presented in Table 1. The measured moisture content in the samples ranged from 17.2% at Site 47 to 33.3% at Site 45 (Table 1). The majority of the sites were dominated by sands. Soils

Table 1. Moisture, sand, silt, and clay content as percentages (%) measured in soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002.

Sample Site	% Moisture	% Sand	% Silt	% Clay
44	31.0	77.5	12.0	10.4
45	33.3	72.9	18.3	8.8
46	33.0	18.2	56.8	25.0
47	17.2	42.2	34.5	23.2
48	20.5	55.4	23.9	20.6
49	23.6	65.3	23.6	11.1

collected at Sites 46 and 47 were the only samples that were predominantly composed of silts and clays.

Metals

Results of the metals and perchlorate analyses for the six samples are presented in Table 2. All of these results are in mg/kg dry weight with the exception of the perchlorate data which is presented in $\mu\text{g}/\text{kg}$.

[Aluminum (Al)] Approximately 8.1% of the Earth's crust is composed of aluminum (Miller and Gardiner, 1998). Background surface soil concentrations in the western U.S. range up to 74,000 mg Al/kg (Shacklette and Boerngen, 1984). According to the TNRCC (2001), a soil-aluminum concentration of 30,000 mg Al/kg is considered background in the State of Texas. Efroymsen *et al.* (1997), proposed 600 mg Al/kg dry weight as a screening benchmark value for aluminum toxicity to soil microorganisms. Aluminum levels were detected above the analytical detection limits in all six of the soil samples collected (Table 2). These concentrations ranged from 950 mg Al/kg dry weight at Site 45 to 7,941 mg Al/kg dry weight at Site 49 (Table 2). All of these concentrations exceeded the ecological benchmark value proposed by Efroymsen *et al.* (1997), but none of the measured levels exceeded the soil background values suggested by Shacklette and Boerngen (1984) and the TNRCC (2001).

[Arsenic (As)] According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background elemental arsenic concentrations in surface soils in the western U.S. is 7 mg As/kg, while the TNRCC (2001), considers a soil-arsenic concentration of 5.9 mg As/kg as background in the State of Texas. Pennington (1991) reported soil-arsenic concentrations ranging up to 13.36 mg As/kg in the Texas Panhandle. Efroymsen *et al.* (1997), proposed an earthworm soils toxicity screening benchmark value of 60 mg As/kg dry weight, while the USEPA (2000a) considers a soil-arsenic concentration of 37 mg As/kg dry weight as a benchmark value for terrestrial plants. Arsenic concentrations were detected above the analytical detection limits in all six samples (Table 2). These concentrations ranged from 1.51 mg As/kg dry weight at Site 45 to 12.2 mg As/kg dry weight at Site 46 (Table 2). All of the measured arsenic levels were below screening criteria with the exception of the concentration detected at Site 46 which exceeded the soil background criterion proposed by Shacklette and Boerngen (1984) and the Texas soil background criterion (TNRCC, 2001). However, the concentration at Site 46 was below the ecological benchmarks suggested by Efroymsen *et al.* (1997) and the USEPA (2000a).

[Barium (Ba)] Barium compounds are used in a variety of industrial applications. In nature, barium chiefly occurs as the relatively insoluble salts, barite and witherite (USEPA, 1986). Shacklette and Boerngen (1984) reported an estimated arithmetic mean of 670 mg Ba/kg as background for soils in the western U.S. while a soils concentration of 300 mg Ba/kg dry weight is considered background in the State of Texas (TNRCC, 2001). According to Efroymsen *et al.* (1997), a proposed screening benchmark value for barium toxicity to soil microorganisms is 3000 mg Ba/kg dry weight, while the TNRCC (2001) considers a soil-barium concentration of 500 mg Ba/kg dry weight as a benchmark value for terrestrial plants. Barium levels were detected above the analytical

detection limits in all soil samples collected from the six sites (Table 2). These concentrations ranged from 13.3 mg Ba/kg dry weight at Site 45 to 82.2 mg Ba/kg dry weight at Site 46 (Table 2), all below screening criteria (Shacklette and Boerngen, 1984; Efroymson *et al.*, 1997; TNRCC, 2001).

Table 2. Results of metals and perchlorate analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; and bdl is below the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
Aluminum	3,522.00	950.00	6,086.00	4,743.00	5,462.00	7,941.00
dl	10.10	10.40	10.00	10.00	10.10	10.00
Arsenic	2.01	1.51	12.20	2.25	2.12	1.92
dl	0.50	0.52	0.50	0.50	0.50	0.50
Barium	71.90	13.30	82.20	42.20	62.60	66.70
dl	0.20	0.21	0.20	0.20	0.20	0.20
Beryllium	0.39	84.10	0.41	0.32	0.30	0.42
dl	0.02	0.02	0.02	0.02	0.02	0.02
Boron	3.41	2.11	5.01	3.48	2.56	3.22
dl	2.02	2.10	2.00	2.00	2.02	2.00
Cadmium	0.29	0.32	0.62	0.19	0.17	0.34
dl	0.10	0.10	0.10	0.10	0.10	0.10
Chromium	9.97	6.17	11.40	9.45	9.03	17.20
dl	0.51	0.52	0.50	0.50	0.50	0.50
Copper	6.55	5.40	16.30	3.59	5.99	5.58
dl	0.51	0.52	0.50	0.50	0.50	0.50
Iron	8,640.00	9,939.00	12,450.00	15,190.00	6,854.00	18,520.00
dl	10.10	10.40	10.00	10.00	10.10	10.00
Lead	12.10	67.20	33.90	13.30	25.30	19.20
dl	2.02	2.07	2.00	2.00	2.02	2.00
Magnesium	1,376.00	680.00	2,083.00	260.00	383.00	431.00
dl	10.10	10.40	10.00	10.00	10.10	10.00
Manganese	392.00	356.00	351.00	48.30	204.00	166.00
dl	1.01	1.04	1.00	1.00	1.01	1.00
Mercury	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.10	0.10	0.10	0.10	0.10	0.10
Molybdenum	bdl	bdl	bdl	bdl	bdl	bdl
dl	1.01	1.04	1.00	1.00	1.01	1.00
Nickel	24.60	17.30	28.80	6.93	7.37	12.40
dl	1.01	1.04	1.00	1.00	1.01	1.00
Selenium	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.51	0.52	0.50	0.50	0.50	0.50
Silver	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.10	0.10	0.10	0.10	0.10	0.10
Strontium	33.30	28.30	95.70	5.14	8.97	8.30
dl	0.51	0.52	0.50	0.50	0.50	0.50
Vanadium	13.20	10.20	12.40	21.30	13.60	24.70
dl	0.51	0.52	0.50	0.50	0.50	0.50
Zinc	32.40	20.50	65.50	13.80	14.70	21.6
dl	5.05	5.18	5.01	5.01	5.04	5.01
Perchlorate*	bdl	bdl	86.0	96.5	bdl	bdl
dl	1.7	1.8	1.5	1.8	1.8	2.1

*Perchlorate analytical results are presented as µg/kg.

[Beryllium (Be)] Although not truly a heavy metal, beryllium is a rare element that is considered potentially toxic (Irwin and Dodson, 1991; Manahan, 1991). The distribution of beryllium in the environment largely results from the combustion of coal and oil (Goyer, 1991; Manahan, 1991). Coal mined from the mid-west U.S. contains an average of about 2.5 mg Be/kg while crude oil can contain approximately 0.08 mg Be/kg (Goyer, 1991). Beryllium concentrations in soils in the U.S. can range up to 15 mg Be/kg (Shacklette and Boerngen, 1984), but according to Shacklette and Boerngen (1984), the estimated arithmetic mean for background beryllium concentrations in soils in the western U.S. is 0.97 mg Be/kg. In the State of Texas, a soil-beryllium concentration of 1.5 mg Be/kg dry weight is considered background (TNRCC, 2001). The TNRCC (2001) considers a soil-beryllium concentration of 10 mg Be/kg dry weight as a benchmark value for terrestrial plants. Beryllium concentrations were detected above the analytical detection limits in every soil sample collected (Table 2). The detected concentrations ranged from 0.30 mg Be/kg dry weight at Site 48 to 84.1 mg Be/kg dry weight at Site 45 (Table 2). All of these concentrations were below recommended screening criteria, except for the level measured at Site 45, which was high enough to warrant further investigation.

[Boron (B)] Boron compounds are used in the production of fertilizers and other agricultural chemicals such as herbicides and insecticides (Moore *et al.*, 1990; USDOJ, 1998). In the U.S., boron concentrations in soils typically range from 10-300 mg B/kg (USDOJ, 1998). According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background boron concentrations in western soils is 29 mg B/kg, while a soils concentration of 30 mg B/kg is considered background in the State of Texas (TNRCC, 2001). Efroymson *et al.* (1997), proposed a screening benchmark value of 20 mg B/kg dry weight for boron toxicity to soil microorganisms and microbial processes, while the TNRCC (2001) considers a soil-boron concentration of 0.5 mg B/kg dry weight as a benchmark value for terrestrial plants. Usually, arid, saline soils will contain higher boron concentrations in comparison to watered, loamy soils (USDOJ, 1998). Furthermore, soils formed from marine sediments typically contain higher concentrations of boron than those formed from igneous rocks (Moore *et al.*, 1990). Boron concentrations were detected above the analytical detection limits in every soil sample collected (Table 2). The detected concentrations ranged from 2.11 mg B/kg dry weight at Site 45 to 5.01 mg B/kg dry weight at Site 46 (Table 2). All six samples contained boron levels that exceeded the benchmark value for plants (TNRCC, 2001); however, none of the soils contained boron concentrations that exceeded any of the other recommended screening criteria (Shacklette and Boerngen, 1984; Efroymson *et al.*, 1997; USDOJ, 1998; TNRCC, 2001).

[Cadmium (Cd)] Ryan *et al.* (1980) reported that the normal range for elemental cadmium in surface soils in the U.S. is 0.06 to 0.5 mg Cd/kg. According to Efroymson *et al.* (1997), a proposed screening benchmark value for cadmium toxicity to soil microorganisms is 20 mg Cd/kg dry weight, while the TNRCC (2001) reports concentrations of 110 mg Cd/kg dry weight and 29 mg Cd/kg dry weight as ecological benchmarks for earthworms and terrestrial plants, respectively. Cadmium concentrations were detected above the analytical detection limits in every soil sample collected (Table 2). These concentrations ranged from 0.17 mg Cd/kg dry weight at Site 48 to 0.62 mg Cd/kg dry weight at Site 46 (Table 2). Only the sample from Site 46 contained a cadmium level that exceeded the expected soil background concentration (Ryan *et al.*, 1980); however, none of the

cadmium concentrations measured in any of the samples collected exceeded any of the ecological benchmark values suggested by Efroymsen *et al.* (1997) and the TNRCC (2001).

[Chromium (Cr)] Excessive chromium can be mutagenic, carcinogenic, and teratogenic to a wide variety of organisms (Eisler, 1986). Shacklette and Boerngen (1984) reported an estimated arithmetic mean of 56 mg Cr/kg as background for soils in the western U.S. According to the TNRCC (2001), a soil-chromium concentration of 30 mg Cr/kg dry weight can be considered background in the State of Texas. Efroymsen *et al.* (1997), proposed soil toxicity screening benchmark values ranging from 0.4 mg Cr/kg dry weight for earthworms to 10 mg Cr/kg dry weight for soil microorganisms. The USEPA (2000a) considers a soil-chromium concentration of 5 mg Cr/kg dry weight as a benchmark value for terrestrial plants. Chromium concentrations were detected above the analytical detection limits in all soil samples collected (Table 2). These concentrations ranged from 6.17 mg Cr/kg dry weight at Site 45 to 17.2 mg Cr/kg dry weight at Site 49 (Table 2). All of the samples contained chromium levels that exceeded soil benchmark values (Efroymsen *et al.*, 1997; USEPA, 2000); however, none of these samples contained chromium concentrations above the suggested soil background values (Shacklette and Boerngen, 1984; TNRCC, 2001).

[Copper (Cu)] Copper is primarily used in the manufacturing of electrical equipment, pipe, and machinery (1998a). It is also an essential micronutrient that interacts in animals with other essential trace elements such as iron, zinc, molybdenum, manganese, nickel, and selenium and also with nonessential elements including silver, cadmium, mercury, and lead (Goyer, 1991; Eisler, 1998). According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background copper concentrations in surface soils in the western U.S. is 27 mg Cu/kg, while a soil-copper concentration of 15 mg/kg dry weight is considered background in the State of Texas (TNRCC, 2001). Efroymsen *et al.* (1997) proposed a soils toxicity screening benchmark value of 100 mg Cu/kg dry weight. The TNRCC (2001) report 61 mg Cu/kg dry weight as the soils benchmark value for earthworms. Copper concentrations were detected above the analytical detection limits in each of the soil samples collected (Table 2). These concentrations ranged from 3.59 mg Cu/kg dry weight at Site 47 to 16.3 mg Cu/kg dry weight at Site 46 (Table 2). Only Site 46 contained a copper level that exceeded any of the cited screening criteria (TNRCC, 2001); however, the concentration detected at this site would not be expected to cause significant detrimental effects to ecological resources within this area.

[Iron (Fe)] Iron is a necessary nutrient that is a constituent of many enzymatic and other cellular processes (Horne and Goldman, 1994). It is absolutely essential both for the transport of oxygen to the tissues and for maintenance of oxidative systems within the tissue cells (Guyton, 1981). Iron composes approximately 5% of the Earth's crust (Miller and Gardiner, 1998). Background iron concentrations in surface soils in the western U.S. range up to 26,000 mg Fe/kg (Shacklette and Boerngen, 1984). In Texas, median background soil-iron concentrations are reported as 15,000 mg Fe/kg (TNRCC, 2001). Iron levels were detected above the analytical detection limits in every sample collected (Table 2). These concentrations ranged from 6,854 mg Fe/kg dry weight at Site 48 to 18,520 mg Fe/kg dry weight at Site 49 (Table 2). Only one of the six sites, Site 49, contained elevated iron in comparison to any of the criteria cited (TNRCC, 2001), however, the level measured at this site would not be expected to result in significant adverse effects to wildlife resources.

[Lead (Pb)] Listed by the USEPA as a priority pollutant, lead is used in pigment and chemical production, metallurgy and steel manufacturing, storage batteries, ceramics, petroleum products, cable sheathing, pipe and sheeting fabrication, and ammunition production (Eisler, 1988). Lead is neither essential nor beneficial to living organisms, and unlike mercury, lead does not exhibit bio-magnification through progressive trophic levels (Eisler, 1988; Pain 1995). Lead is naturally occurring in soils. According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background lead concentrations in surface soils in the western U.S. is 20 mg Pb/kg. The TNRCC (2001), considers a soil-lead concentration of 15 mg Pb/kg dry weight as background in the State of Texas. Soil benchmark values range from 50 mg Pb/kg dry weight for terrestrial plants to 500 mg Pb/kg dry weight for earthworms (TNRCC, 2001). Lead was detected above the analytical detection limits in every soil sample collected (Table 2). These concentrations ranged from 12.1 mg Pb/kg dry weight at Site 44 to 67.2 mg Pb/kg dry weight at Site 45 (Table 2). Detected soil lead levels at Sites 45, 46 (33.9 mg Pb/kg dry weight), and 48 (25.3 mg Pb/kg dry weight) exceeded all of the recommended background criteria (Shacklette and Boerngen, 1984; TNRCC, 2001). The concentration measured at Site 45 also exceeded the lower soil ecological benchmark value recommended by the TNRCC (2001). Based on these results, further investigation into lead contamination at Site 45 is warranted.

[Magnesium (Mg)] Magnesium is an essential nutrient that is required for energy transfer in all living cells because it catalyzes the change from adenosine triphosphate (ATP) to adenosine diphosphate (ADP) (Horne and Goldman, 1994). The Earth's crust is composed of approximately 2.1% magnesium (Miller and Gardiner, 1998). Shacklette and Boerngen (1984), estimated the arithmetic mean for background magnesium concentrations in surface soils in the western U.S. as 10,000 mg Mg/kg. Magnesium concentrations were detected above the analytical detection limits in all soil samples collected (Table 2). These concentrations ranged from 260 mg Mg/kg dry weight at Site 47 to 2,083 mg Mg/kg dry weight at Site 46 (Table 2), all below the background value reported by Shacklette and Boerngen (1984).

[Manganese (Mn)] Manganese is a widely distributed, abundant element that constitutes approximately 0.085% of the earth's crust (Irwin and Dodson, 1991). It is a necessary nutrient for plants and animals that is relatively nontoxic to aquatic biota (Wiener and Giesy, 1979; Cole 1983). According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background manganese concentrations in surface soils in the western U.S. is 480 mg Mn/kg. The TNRCC (2001), considers a soil-manganese concentration of 300 mg Mn/kg dry weight as background in the State of Texas. According to Efroymsen *et al.* (1997), a proposed screening benchmark value for manganese toxicity to soil microorganisms is 100 mg Mn/kg dry weight, while the TNRCC (2001) reports a soil-manganese concentration of 500 mg Mn/kg dry weight as a benchmark value for terrestrial plants. The ecological screening benchmark recommended by the USEPA for manganese in soils is 100 mg Mn/kg (RAIS, 2002). Manganese concentrations were detected above the analytical detection limits in every soil sample collected (Table 2). The detected concentrations ranged from 48.3 mg Mn/kg dry weight at Site 47 to 392 mg Mn/kg dry weight at Site 44 (Table 2). Only Sites 44, 45 (356 mg Mn/kg dry weight), and 46 (351 mg Mn/kg dry weight) contained manganese levels that exceeded any of the cited screening criteria (Efroymsen *et al.* 1997; TNRCC, 2001); however, the concentrations detected at these sites would not be expected to cause significant detrimental effects to ecological resources within these areas.

[Nickel (Ni)] Background surface soil-nickel concentrations range up to 19 mg Ni/kg in the western U.S. and up to 10 mg Ni/kg in the State of Texas (Shacklette and Boerngen, 1984; TNRCC, 2001). According to Efroymsen *et al.* (1997), a proposed screening benchmark value for nickel toxicity to soil microorganisms is 90 mg Ni/kg dry weight, while the TNRCC (2001) reports a soil-nickel concentration of 30 mg Ni/kg dry weight as a benchmark value for terrestrial plants. Nickel concentrations were detected above the analytical detection limits in all six of the samples collected (Table 2). The detected concentrations ranged from 6.93 mg Ni/kg dry weight at Site 47 to 28.8 mg Ni/kg dry weight at Site 46 (Table 2). The samples collected from Sites 44 (24.6 mg Ni/kg dry weight), 45 (17.3 mg Ni/kg dry weight), and 46 (28.8 mg Ni/kg dry weight) contained nickel levels that exceeded some of the cited benchmark values and screening criteria (Shacklette and Boerngen, 1984; TNRCC, 2001), but the concentrations detected at these sites were not at levels where significant adverse affects to wildlife resources would be expected to occur.

[Strontium (Sr)] Strontium compounds are used in the manufacturing of pyrotechnics including signal flares and tracer bullets, the production of glass and ceramics, and sugar refining (Merck, 1989). Strontium is a fairly common alkaline earth metal (Irwin and Dodson, 1991). According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background strontium concentrations in western soils in the U.S. is 270 mg Sr/kg while a soils concentration of 100 mg Sr/kg is considered background in the State of Texas (TNRCC, 2001). Strontium concentrations were detected above the analytical detection limits in every sample collected (Table 2). These concentrations ranged from 5.14 mg Sr/kg dry weight at Site 47 to 95.7 mg Sr/kg dry weight at Site 46 (Table 2), all below the suggested background values (Shacklette and Boerngen, 1984; TNRCC, 2001).

[Vanadium (V)] Approximately 0.01% of the Earth's crust is composed of vanadium (Merck, 1989). Vanadium compounds are used in the production of rust-resistant metals, the manufacturing of ammunition, in x-rays, as catalysts in the distillation of alcohols and the production of synthetic rubber, and to reduce mercuric and ferric salts to mercurous and ferrous salts in industrial processes (Sax and Lewis, 1987; Merck, 1989). Vanadium is also a component of fossil fuels (Merck, 1989). West Texas Intermediate Crude contains up to 3.2 mg V/L (ETC, 2000). Vanadium concentrations in soils in the U.S. can range up to 500 mg V/kg (Shacklette and Boerngen, 1984). According to Shacklette and Boerngen (1984), the estimated arithmetic mean for background vanadium concentrations in western soils in the U.S. is 88 mg V/kg, while 50 mg V/kg dry weight is considered background in soils from Texas (TNRCC, 2001). The ecological screening benchmark recommended by the USEPA for vanadium in soils is 2 mg V/kg (RAIS, 2002). According to Efroymsen *et al.* (1997), a proposed screening benchmark value for vanadium toxicity to soil microorganisms is 20 mg V/kg, while the TNRCC (2001) considers a soil-vanadium concentration of 2 mg V/kg dry weight as a benchmark value for terrestrial plants. Vanadium concentrations were detected above the analytical detection limits in all six samples collected (Table 2). The detected concentrations ranged from 10.2 mg V/kg dry weight at Site 45 to 24.7 mg V/kg dry weight at Site 49 (Table 2). All of the sites sampled contained vanadium concentrations exceeding the USEPA (RAIS, 2002) and TNRCC (2001) ecological benchmark values. However, only Sites 47 (21.3 mg V/kg dry weight) and 49 (24.7 mg V/kg dry weight) exceeded the screening criterion proposed by Efroymsen *et al.* (1997), while none of the sites sampled contained vanadium levels that exceeded suggested background values.

[Zinc (Zn)] Zinc is a naturally occurring metallic element found in soil but is also listed by the USEPA as a priority pollutant (Giggleman *et al.*, 1998). It is used in the production of non-corrosive alloys and brass and in galvanizing steel and iron products (Eisler, 1993). Shacklette and Boerngen (1984), estimated the arithmetic mean for background zinc concentrations in surface soils in the western U.S. at 65 mg Zn/kg. The TNRCC (2001), considers a soil-zinc concentration of 30 mg Zn/kg as background in the State of Texas. Efrogmson *et al.* (1997), proposed a soils toxicity screening benchmark value of 100 mg Zn/kg dry weight for soil microorganisms and invertebrates. The ecological screening benchmark recommended by the USEPA for zinc in soils is 50 mg Zn/kg (RAIS, 2002). Zinc concentrations were detected above the analytical detection limits in all samples collected (Table 2). These concentrations ranged from 13.8 mg Zn/kg dry weight at Site 47 to 65.5 mg Zn/kg dry weight at Site 46 (Table 2). Site 47 was the only site that contained a zinc level which exceeded the soil benchmark value recommended by the USEPA (RAIS, 2002); however, the detected concentration at this site was still below the benchmark values suggested by Efrogmson *et al.* (1997) for soil microorganisms and invertebrates.

Semi-volatile Organic Compounds

Results of the semi-volatile organic compound analyses for the six sites are presented in Table 3. As previously stated, one semi-volatile organic compound (aniline) was detected above the analytical detection limit at Site 47 and eight semi-volatile organic compounds [the polycyclic aromatic hydrocarbons benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, phenanthrene, and pyrene] were detected above their respective analytical detection limits at Site 46. No other semi-volatile organic compounds were measured above the analytical detection limits at the remaining four sites.

[Aniline] Also known as aminobenzene, aminophade, aminophen, anyvim, arylamine, benzamine, benzeneamine, blue oil, krystallin, kyanol, and phenylamine, aniline is an aromatic amine that is typically a colorless, oily liquid (USEPA, 1994a; PTCL, 2002). It is used in the production of explosives, pesticides, preservatives, rubber, and dyes and can also be released into the environment as a by-product through the incineration of plastics (USEPA, 1994a; PTCL, 2002). When released to terrestrial environments, depending on soil pH and ionic composition, aniline will moderately adsorb to organic material in soils, but will also slowly volatilize and bio-degrade (USEPA, 1994a; Kosson and Byrne, 1995; ATSDR, 2002). According to Kosson and Byrne (1995), once bound to soils, aniline is not readily bio-degradeable. This compound is considered a probable human carcinogen (USEPA, 1994a), consequently remedial target values for soils have been developed. In Texas, in order to protect groundwater, the remedial target value for aniline contaminated soils in residential areas is 1.5 mg/kg (TAC, 1993), while in the State of Louisiana, the cleanup standard for non-industrial soils is 2.5 mg/kg (AEHS, 2002). In a more conservative approach, the State of New York recommends a cleanup objective of 0.1 mg/kg for aniline contaminated soils (NYSDEC, 2002). The concentration detected at Site 47 (0.075 mg/kg dry weight) was less than the remedial values cited above, even the conservative concentration proposed by the State of New York (TAC, 1993; AEHS, 2002; NYSDEC, 2002).

Table 3. Results of semi-volatile analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; and bdl is below the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
1,2,4-trichlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
1,2-dichlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
1,3-dichlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
1,4-dichlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
1-chloronaphthalene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
1-naphthylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,3,4,6-tetrachlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4,5-trichlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4,6-trichlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4-dichlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4-dimethylphenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4-dinitrophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,4-dinitrotoluene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,6-dichlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2,6-dinitrotoluene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-chloronaphthalene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-chlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-methylphenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-naphthylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-nitroaniline	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-nitrophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-picoline	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
2-methylnaphthalene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
3,3'-dichlorobenzidine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
3-methylcholanthrene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
3-nitroaniline	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4,6-dinitro-2-methylphenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032

Table 3 (continued). Results of semi-volatile analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; bdl is below the analytical detection limit; and ★ is detected above the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
4-aminobiphenyl	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-bromophenyl-phenylether	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-chloro-3-methylphenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-chloroaniline	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-chlorophenyl-phenylether	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-methylphenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-nitroaniline	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
4-nitrophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
7,12-dimethylbenz(a)anthracene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
acetophenone	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
aniline★	bdl	bdl	bdl	0.075	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzidine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzo(a)anthracene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzoic acid	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzyl alcohol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
bis(2-chloroethoxy)methane	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
bis(2-chloroethyl)ether	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
bis(2-ethylhexyl)phthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
bis(2chloroisopropyl)ether	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
butylbenzylphthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
carbazole	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
di-n-butylphthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
di-n-octylphthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
dibenz(a,h)anthracene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
dibenz(a,j)acridine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
dibenzofuran	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
diethyl phthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
dimethylphthalate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032

Table 3 (continued). Results of semi-volatile analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; bdl is below the analytical detection limit; and ★ is detected above the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
diphenylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
ethyl methanesulfonate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
hexachlorobutadiene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
hexachlorocyclopentadiene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
hexachloroethane	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
isophorone	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
methyl methanesulfonate	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
n-nitroso-di-n-propylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
n-nitrosopiperidine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
nitrobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
pentachlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
pentachloronitrobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
pentachlorophenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
phenacetin	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
phenol	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
pronamide	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
a,a-dimethylphenylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
acenaphthalene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
acenaphthene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
anthracene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzo(a)pyrene★	bdl	bdl	0.046	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzo(b)fluoranthene★	bdl	bdl	0.212	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzo(g,h,i)perylene★	bdl	bdl	0.046	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
benzo(k)fluoranthene★	bdl	bdl	0.070	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
chrysene★	bdl	bdl	0.147	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
fluoranthene★	bdl	bdl	0.303	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
fluorene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
indeno(1,2,3-cd)pyrene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032

Table 3 (continued). Results of semi-volatile analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; bdl is below the analytical detection limit; and ★ is detected above the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
n-nitrosodiphenylamine	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
naphthalene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
p-dimethylaminoazobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
phenanthrene★	bdl	bdl	0.108	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032
pyrene★	bdl	bdl	0.326	bdl	bdl	bdl
dl	0.036	0.037	0.037	0.030	0.031	0.032

[Polycyclic aromatic hydrocarbons (PAHs)] Polycyclic aromatic hydrocarbons are semi-volatile organic compounds that consist of hydrocarbons arranged in the form of two or more benzene rings in linear, angular, or cluster arrangements which may or may not have substituted groups attached to one or more of the rings (Eisler, 1987). Lower molecular weight PAHs (molecular weight less than 202.26) contain two to three benzene rings that often exhibit acute toxicity but are generally non-carcinogenic, whereas high molecular weight PAHs (molecular weight greater than 202.26) contain four to seven benzene rings and are usually considered carcinogenic (Eisler, 1987). Sources of PAHs can include the incineration of municipal and industrial wastes, petroleum and/or crude oil releases, and emissions from the incomplete combustion of fossil fuels (Eisler, 1987). Eight PAH compounds were measured above the analytical detection limits in the sample collected from Site 46 (Table 3). No other site sampled contained PAH concentrations greater than the analytical detection limits (Table 3). Three of the PAH compounds detected at Site 46 exceeded ecological benchmark values which indicates that further investigations may be warranted in this area to determine effects to wildlife resources. The eight compounds detected were as follows:

[Benzo(a)pyrene] Considered a probable human carcinogen, benzo(a)pyrene is a heavy weight PAH (molecular weight equals 252.3) that contains five benzene rings (RAIS, 1994a). In soils, the ecological screening benchmark recommended by the USEPA for benzo(a)pyrene is 0.1 mg/kg (RAIS, 2002). The concentration detected at Site 46 (0.046 mg/kg dry weight) was less than the benchmark value cited for soils (RAIS, 2002).

[Benzo(b)fluoranthene] Benzo(b)fluoranthene is a heavy weight PAH (molecular weight equals 252) that contains four benzene rings and is also considered a probable human carcinogen (RAIS, 1994b). The Canadian Council of Ministers of the Environment (CCME) recommends a soils-benzo(b)fluoranthene screening criterion of 0.1 mg/kg for agricultural soils (EPT, 1999), whereas the cleanup standard for non-industrial soils in the State of Louisiana is 0.56 mg/kg (AEHS, 2002). The concentration detected at Site 46 (0.212 mg/kg dry weight) exceeded the CCME criterion, but was below the Louisiana standard (EPT, 1999; AEHS, 2002).

[Benzo(g,h,i)perylene] Benzo(g,h,i)perylene is a heavy weight PAH (molecular weight equals 276) that contains six benzene rings. Unlike other heavy chained PAHs, benzo(g,h,i)perylene has not been classified as a human carcinogen (RAIS, 1994c). In soils, the cleanup objective recommended by the State of New York is 50 mg/kg (NYSDEC, 2002). The concentration detected at Site 46 (0.046 mg/kg dry weight) was over 1000 times less than the target remedial value proposed by the State of New York (NYSDEC, 2002).

[Benzo(k)fluoranthene] Benzo(k)fluoranthene is a heavy weight PAH (molecular weight equals 252) that contains four benzene rings and is considered a probable human carcinogen (RAIS, 1994d). For non-industrial soils, the cleanup standard in the State of Louisiana is 5.5 mg/kg (AEHS, 2002), while the State of New York recommends a cleanup objective of 1.1 mg/kg (NYSDEC, 2002). The concentration detected at Site 46 (0.07 mg/kg dry weight) was well less than the soil cleanup levels cited above (AEHS, 2002; NYSDEC, 2002).

[Chrysene] Chrysene is a heavy weight PAH (molecular weight equals 228) that contains four benzene rings and is classified as a probable human carcinogen (RAIS, 1994e). The screening criterion in Ontario (Canada) for chrysene in coarse textured agricultural, residential, and parkland soils is 12 mg/kg (EPT, 1999), while the cleanup standard in Louisiana for non-industrial soils is 61 mg/kg (AEHS, 2002). In contrast to these values, the soils cleanup objective recommended by the State of New York is 0.4 mg/kg. The concentration detected at Site 46 (0.147 mg/kg dry weight) was less than all of the cited screening criteria (EPT, 1999; AEHS, 2002; NYSDEC, 2002).

[Fluoranthene] Fluoranthene is a light weight PAH (molecular weight equals 202) that contains three benzene rings. Like benzo(g,h,i)perylene, fluoranthene is not considered a human carcinogen (RAIS, 1993a). In soils, the ecological screening benchmark recommended by the USEPA for fluoranthene is 0.1 mg/kg (RAIS, 2002). The concentration detected at Site 46 (0.303 mg/kg dry weight) exceeded this benchmark value (RAIS, 2002).

[Phenanthrene] Not classified as a human carcinogen, phenanthrene is a light weight PAH (molecular weight equals 178) that contains three benzene rings (RAIS, 1993b). In soils, the ecological screening benchmark recommended by the USEPA for phenanthrene is 0.1 mg/kg (RAIS, 2002). The concentration detected at Site 46 (0.108 mg/kg dry weight) slightly exceeded the soil benchmark value (RAIS, 2002).

[Pyrene] Pyrene is a heavy weight PAH (molecular weight equals 202.26) that contains four benzene rings and like benzo(g,h,i)perylene, it is not considered a human carcinogen (RAIS, 1993c). In soils, the ecological screening benchmark recommended by the USEPA for pyrene is 0.1 mg/kg (RAIS, 2002). The concentration detected at Site 46 (0.326 mg/kg dry weight) exceeded this soil benchmark value (RAIS, 2002).

Organochlorine Pesticides

Results of the organochlorine analyses for the six samples, including total-PCBs are presented in Table 4. The compounds 1,2,3,4-tetrachlorobenzene, aldrin, dieldrin, alpha-, beta-, and delta-BHC, gamma- and oxy-chlordane, heptachlor, heptachlor epoxide, and toxaphene were not detected above the analytical detection limits in any of the soil samples collected.

[1,2,4,5-Tetrachlorobenzene] Listed by the USEPA as a persistent, bio-accumulative, and toxic chemical (PBT), 1,2,4,5-tetrachlorobenzene is a common component of many herbicides, insecticides, defoliants, and electrical insulation fluids (Sax and Lewis, 1987; NDDH, 2002). Buchman (1999) reports a remedial target value for agricultural soils of 0.1 mg/kg for residual chlorobenzenes as a group. In Texas, the remedial target value for residential soils is 1.1 mg/kg in order to protect groundwater (TAC, 1993), while in the State of Louisiana, the cleanup standard for non-industrial soils is also 1.1 mg/kg (AEHS, 2002). This compound was detected above the analytical detection limit in soils collected from all six sites (Table 4). These concentrations ranged from 0.000898 mg/kg dry weight at Site 45 to 0.00147 mg/kg dry weight at Site 46 (Table 4), all of which were below recommended screening criteria (TAC, 1993; Buchman, 1999; AEHS, 2002).

[Chlordane, isomers, and metabolites] Technical chlordane consists of the stereoisomers alpha (α) and gamma (γ) or *cis* and *trans*-chlordane, heptachlor, *cis*-nonachlor and *trans*-nonachlor, and the metabolites oxychlordane and heptachlor epoxide (ATSDR, 1994). First developed in 1946, chlordane was used as a general pesticide until 1983 (LMF, 2002). Between 1983 and 1988, use of chlordane in the United States was restricted by the USEPA to subterranean termite control (ATSDR, 1994). All commercial use of chlordane as a pesticide was banned by the USEPA in the United States in 1988 (ATSDR, 1994). In the environment, chlordane binds tightly with soil particles and can remain in the soil for more than 20 years (LMF, 2002). It can bio-accumulate in the tissues of fish, birds, and mammals and can adversely affect the nervous, digestive, and hepatic systems in both humans and animals (ATSDR, 1994; LMF, 2002). In Ontario (Canada), the screening criterion for chlordane in coarse textured agricultural, residential, and parkland soils is 0.29 mg/kg (EPT, 1999), while the cleanup standard for non-industrial soils in the State of Louisiana is 1.5 mg/kg (AEHS, 2002). In Texas, as a measure to protect groundwater, the remedial target value for residential soils is 0.2 mg/kg (TAC, 1993). Chlordane residues (isomers and/or metabolites) were detected above the analytical detection limits at Sites 46 and 48 (Table 4). These detected residues were used following Munn and Gruber (1997) to calculate the technical or total chlordane concentrations (sum of α - and γ chlordane, *cis*- and *trans*-nonachlor, oxychlordane, heptachlor, and heptachlor epoxide) for both of these sites. The calculated total chlordane value for Site 46 was 0.0033 mg/kg dry weight, while the calculated total chlordane value for Site 48 was 0.000164 mg/kg dry weight. Both of these values were below all of the screening criteria cited above (TAC, 1993; EPT, 1999; AEHS, 2002).

Table 4. Results of organochlorine pesticide and total PCB analyses in mg/kg dry weight for soil samples collected from six sites at Caddo Lake National Wildlife Refuge in 2002 (Note - dl is the analytical detection limit; bdl is below the analytical detection limit; and ★ is detected above the analytical detection limit).

Analyte	Site 44	Site 45	Site 46	Site 47	Site 48	Site 49
1,2,3,4-tetrachlorobenzene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
1,2,4,5-tetrachlorobenzene★	0.00119	0.000898	0.00147	0.00156	0.00117	0.000980
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
aldrin	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
HCB★	0.000459	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
heptachlor	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
total PCB★	0.00565	0.0180	0.431	0.00930	0.0140	0.00497
dl	0.00180	0.00186	0.00184	0.00150	0.00154	0.00161
αBHC	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
αchlordane★	bdl	bdl	0.00164	bdl	0.000164	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
βBHC	bdl	bdl	0.00220	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
cis-nonachlor★	bdl	bdl	0.000778	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
δBHC	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
dieldrin	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
endosulfan II★	bdl	bdl	0.00155	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
endrin★	0.000268	0.000280	bdl	0.000241	bdl	0.000551
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
γBHC★	bdl	bdl	bdl	bdl	0.000165	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
γchlordane	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
heptachlor epoxide	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
mirex★	bdl	bdl	0.000824	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
o,p'-DDD★	bdl	0.000174	0.00344	0.000146	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
o,p'-DDE	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
o,p'-DDT★	bdl	0.000198	0.000394	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
oxychlordane	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
p,p'-DDD★	bdl	bdl	0.000314	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
p,p'-DDE★	0.000949	0.00136	0.000593	0.000579	0.00291	0.000444
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
p,p'-DDT★	bdl	bdl	0.00322	bdl	bdl	0.000130
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
pentachloro-anisole★	0.000555	bdl	0.000161	bdl	0.000296	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129
toxaphene	bdl	bdl	bdl	bdl	bdl	bdl
dl	0.00180	0.00186	0.00184	0.00150	0.00154	0.00161
trans-nonachlor★	bdl	bdl	0.000889	bdl	bdl	bdl
dl	0.000144	0.000149	0.000147	0.000120	0.000123	0.000129

[Dichloro-diphenyl-trichloroethane (DDT), isomers, and metabolites] First developed in 1939, dichloro-diphenyl-trichloroethane (DDT) was used extensively throughout the world as an all purpose insecticide (ATSDR, 1995). The metabolites dichloro-diphenyl-dichloroethane (DDD) and dichloro-diphenyl-dichloroethylene (DDE) are microbial degradation products formed by the dehydrohalogenation of DDT (ATSDR, 2000a). Considered a probable human carcinogen by the USEPA, commercial production of DDT was banned in the United States in 1972 because of adverse affects to non-target wildlife species and the potential harm to human health (ATSDR, 1995; ATSDR, 2000a). In wildlife, DDT exposure resulted in birds, alligators, and turtles producing eggs with shells too thin for offspring survival (Baskin, 2002). Dichloro-diphenyl-trichloroethane also exhibits very low solubility in aqueous environments and bio-accumulates in the fatty tissues of fish, birds, and other animals (Baskin, 2002). In soils, DDT binds readily to soil particles and exhibits a half life estimated at 2 to 15 years (ATSDR, 1995). The screening criterion in Ontario (Canada) for DDD, DDE, and DDT in coarse textured agricultural, residential, and parkland soils is 2.2, 1.6, and 1.6 mg/kg, respectively (EPT, 1999). The CCME recommends a soils-DDT screening criterion of 0.7 mg/kg for agricultural, residential, and parkland soils (EPT, 1999). As a measure to protect groundwater in Texas the remedial target value for residential soils is 0.025 mg/kg (TAC, 1993), while the cleanup standard for non-industrial soils in Louisiana is 1.7 mg/kg. (AEHS, 2002). For total-DDT, the USEPA recommends an ecological benchmark value of 0.0025 mg/kg (RAIS, 2002).

One or both of the DDD isomers were detected above the analytical detection limits at Sites 45, 46, and 47 (Table 4), while one of the isomers of the metabolite DDE (p,p'-DDE) was detected above the analytical detection limits at all six sites (Table 4). Isomers of the parent compound DDT were detected above the analytical detection limits at Sites 45, 46, and 49 (Table 4). For screening purposes, the sum of the detected isomer concentrations of DDD (o,p'-DDD + p,p'-DDD), DDE (o,p'-DDE + p,p'-DDE), and/or DDT (o,p'-DDT + p,p'-DDT) were calculated following Munn and Gruber (1997) for each site where detected above the analytical detection limits. In addition, total-DDT concentrations were calculated for Sites 45 (0.00173 mg/kg dry weight) and 46 (0.00796 mg/kg dry weight) by adding the sum of DDD, DDE, and DDT for each of these sites. None of the sites sampled contained individual isomeric DDD, DDE, and/or DDT concentrations that exceeded soil screening criteria or remedial target values (TAC, 1993; EPT, 1999; AEHS, 2002), whereas total-DDT concentrations exceeded the soil benchmark value recommended by the USEPA (RAIS, 2002) only at Site 46. The level of total-DDT measured at Site 46 indicates that further investigations are warranted at this site.

[Endosulfan] The organochlorine pesticide endosulfan was first introduced in the United States in 1954, however it has not been commercially produced in the United States since 1982 (ATSDR, 2000b). This compound exists as two principal isomers, alpha- and beta-endosulfan (ATSDR, 2000b). Endosulfan can degrade in the environment through photolysis, bio-transformation, or oxidation into the metabolite, endosulfan sulfate (ATSDR, 2000b). The screening criterion in Ontario (Canada) for endosulfan concentrations in coarse textured agricultural, residential, and parkland soils is 0.18 mg/kg (EPT, 1999). As a measure to protect groundwater, the remedial target value for residential soils in Texas is 0.183 mg/kg (TAC, 1993). In contrast to these values the cleanup standard for non-industrial soils in the State of Louisiana is 31 mg/kg (AEHS, 2002).

Endosulfan concentrations were detected above the analytical detection limit in soil samples collected from only one site, Site 46 (Table 4). The detected concentration at this site (0.00155 mg/kg dry weight) was below all of the cited screening criterion (TAC, 1993; EPT, 1999; AEHS, 2002).

[Endrin] Endrin is a stereoisomer of dieldrin (ATSDR, 1996). It was first used as an insecticide, rodenticide, and avicide in 1951 (ATSDR, 1996). Manufacturing of this compound discontinued in the United States in 1991 primarily because of its toxicity to non-target populations of raptors and migratory birds (ATSDR, 1996). The ecological screening benchmark recommended by the USEPA for endrin in soils is 0.001 mg/kg (RAIS, 2002). Endrin was detected above the analytical detection limits in samples collected from Sites 44, 45, 47, and 49 (Table 4). The detected concentrations ranged from 0.000241 mg/kg dry weight at Site 47 to 0.000551 mg/kg dry weight at Site 49 (Table 4), all below the reported ecological benchmark value (RAIS, 2002).

[Hexachlorobenzene (HCB)] First introduced in 1945, hexachlorobenzene was widely used in the United States as a fungicide (ATSDR, 1997; EMS 2002a). It was also used in the manufacturing of fireworks, ammunition, and synthetic rubber, and can be produced as a by-product in the waste streams of chloralkali and wood-preserving plants and the incineration of industrial and municipal solid wastes (ATSDR, 1997). Production of HCB as a fungicide ceased in 1965 and currently there are no commercial uses for this compound in the United States (ATSDR, 1997). Once released to a terrestrial system, HCB is highly persistent in soils, with reported half lives ranging from 2.7 to 22.9 years (ETN, 1996; EMS, 2002a). At the surface, evaporation is rapid, but once HCB is mixed into the soil, this process slows down considerably (ETN, 1996). This compound is a suspected carcinogen and is toxic to fish and avian species, while chronic exposure in humans can also lead to liver disease (ATSDR, 1997; EMS, 2002a). The ecological screening benchmark recommended by the USEPA for HCB in soils is 0.0025 mg/kg, while the Oak Ridge National Laboratory considers a soils-HCB concentration of 1000 mg/kg as a benchmark value protective of soil microorganisms (RAIS, 2002). In Canada, a soils-HCB concentration of 0.1 mg/kg is considered background, while an HCB concentration of 1 mg/kg is indicative of moderate soil contamination (Beyer, 1990). Of the six samples collected, only Site 44 contained HCB levels above the analytical detection limits (Table 4). The concentration detected at this site (0.000459 mg/kg dry weight) was below all cited screening criteria (Beyer, 1990; RAIS, 2002).

[gamma-Hexachlorocyclohexane (γ BHC)] Hexachlorocyclohexane represents a group of manufactured chemicals used in pesticides that do not occur naturally in the environment (ATSDR, 1999). Eight isomers are formed from hexachlorocyclohexane of which the four most common are alpha (α -), beta (β -), delta (δ -), and gamma (γ)-BHC (ATSDR, 1999). In the United States, the commercial production of γ -BHC, also known as lindane, began in 1945 (EHP, 2002). This compound was used extensively in the 1950s as an insecticide in the timber industry but is no longer manufactured commercially in the United States (ATSDR, 1999; EHP, 2002). In 1983 the commercial production of all BHCs ceased in the United States (EHP, 2002). Once these compounds are released to soils, they can degrade rapidly under anaerobic conditions but are considered extremely persistent in upland soils (Damborsky *et al.*, 2002). Under aerobic conditions,

bio-degradation mineralizes α -BHC and γ -BHC, whereas β -BHC persists (Middeldorp and McLeish, 2002). Hexachlorocyclohexane and its isomers are reasonably anticipated to be human carcinogens (EHP, 2002). As a measure to address groundwater protection, the remedial target value for residential soils is 0.02 mg/kg in the State of Texas (TAC, 1993), while in Louisiana, the cleanup standard for non-industrial soils is 0.38 mg/kg (AEHS, 2002). The ecological benchmark recommended by the USEPA for γ -BHC in soils is 0.00005 mg/kg (RAIS, 2002). Of the samples analyzed, only Site 48 contained a γ -BHC concentration above the analytical detection limit (Table 4), and the detected concentration at this site (0.000165 mg/kg dry weight) exceeded the recommended benchmark value, but was well below the cited remedial target values (TAC, 1993; AEHS, 2002; RAIS, 2002). Even though the concentration measured at Site 48 was below cleanup levels, further investigations may be warranted at this site to address possible ecological concerns.

[Mirex] First developed in 1946, the pesticide mirex is a highly stable chlorinated hydrocarbon compound that exhibits very low solubility in water and is highly resistant to chemical, thermal, and biochemical degradation (Eisler, 1985). From 1959 to 1972, mirex was used to control fire ants and as a flame retardant in plastics, rubber, paint, paper, and electrical goods (Eco-USA, 2002). It has not been produced or used in the United States since 1978 (Eco-USA, 2002). Because of its resistance to degradation, mirex has a half-life of over 10 years in terrestrial soils (Eisler, 1985; EMS, 2002b). Listed by the USEPA as PBT, mirex is a known endocrine disruptor and suspected carcinogen (Eco-USA, 2002; EMS, 2002b). In the State of Arizona, the soil remediation level for residential soils is 2.5 mg/kg (AAC, 1999). Of the samples analyzed, only Site 46 contained a mirex concentration above the analytical detection limit (Table 4). The concentration detected at this site (0.000824 mg/kg dry weight) was well below the cited criterion (AAC, 1999).

[Pentachloroanisole] A suspected carcinogen, pentachloroanisole is a chlorinated aromatic compound that is widely distributed in the environment (NTP, 2002). It is formed as a degradation product of pentachloronitrobenzene and pentachlorophenol (NTP, 2002). Currently, there are no screening criteria available for pentachloroanisole in soils. However, there are criteria available for its parent compound, pentachlorophenol which is used as a pesticide and wood preservative. The ecological screening benchmark recommended by the USEPA for pentachlorophenol in soils is 0.002 mg/kg, while the Oak Ridge National Laboratory considers a soils-pentachlorophenol concentration of 3 mg/kg as a benchmark value protective of plants (RAIS, 2002). Pentachloroanisole concentrations were detected above the analytical detection limits in soils collected from Sites 44, 46, and 48 (Table 4). These concentrations ranged from 0.000161 mg/kg dry weight at Site 46 to 0.000555 mg/kg dry weight at Site 44 (Table 4). All of the sites where pentachloroanisole was detected contained concentrations below the ecological screening criteria recommended for pentachlorophenol.

Polychlorinated Biphenyls

[Total Polychlorinated Biphenyls (PCBs)] Polychlorinated biphenyls were used extensively in electrical transformers, capacitors, heat transfer fluids, and electrical utilities as lubricants, insulators, and coolants until production was banned in 1979 (USEPA, 1994b; Moring, 1997). Total-PCBs

represents a quantification of approximately 209 individual congeners (Moring, 1997). These congeners are relatively stable compounds that exhibit low water solubilities, high heat capacities, low flammabilities, low electric conductivities, and low vapor pressures (USEPA, 1994b; Moring, 1997). The CCME recommends a total-PCBs concentration of 0.3 mg/kg as the screening criterion for agricultural, residential, and parkland soils (EPT, 1999). According to Buchman (1999), the National Oceanic and Atmospheric Administration (NOAA) considers a total-PCBs concentration of 0.5 mg/kg dry weight as the target value for remedial efforts in agricultural soils and a concentration of 5 mg/kg dry weight as the target value for remedial activities in urban park/residential soils. As a measure to address groundwater protection, the remedial target value for residential soils is 0.05 mg/kg in the State of Texas (TAC, 1993), while in Louisiana, the cleanup standard for non-industrial soils is 0.19 mg/kg (AEHS, 2002). The ecological screening benchmark recommended by the USEPA for total-PCBs in soils is 0.02 mg/kg, while the Oak Ridge National Laboratory considers a total-PCBs concentration of 40 mg/kg as a benchmark value protective of plants (RAIS, 2002).

As previously stated, total-PCBs were detected in all six of the soil samples collected (Table 4). The measured concentrations ranged from 0.00497 mg/kg dry weight at Site 49 to 0.431 mg/kg dry weight at Site 46 (Table 4). All of the sites contained total-PCB concentrations below soil screening criteria with the exception of the concentration measured at Site 46 which exceeded the Texas and Louisiana remedial target values, the soil benchmark value recommended by the USEPA, and the screening criterion suggested the CCME, but was below both of the remedial target values recommended by NOAA, and well below the soil benchmark proposed by Oak Ridge National Laboratory (TAC, 1993; Buchman, 1999; EPT, 1999; AEHS, 2002; RAIS, 2002b). However, considering the bio-accumulative propensity of PCBs in succeeding trophic guilds, the concentration detected at Site 46 warrants further investigation.

Perchlorate

[Perchlorate (ClO₄)] Perchlorate compounds are strong oxidizers that have been widely used as additives in solid rocket propellants and ignitable sources in munitions and fireworks (Smith *et al.*, 2001; York *et al.*, 2001). In the environment, perchlorate is highly soluble in water, readily moves through both groundwater and surface water, and can persist for decades (Nzengung and Wang, 2000; Smith *et al.*, 2001). In humans, perchlorate can interfere with iodine uptake in the thyroid gland and at elevated concentrations interferes with the thyroid's ability to produce hormones and regulate metabolism (Nzengung and Wang, 2000). Nationally, the toxicological and risk characteristics of perchlorate are currently being reviewed by the USEPA under the premise of establishing standards and/or screening criteria. In the interim, the only available comparative value is 4 µg/L which is the current Texas regulatory action level for perchlorate in groundwater (Sher, personal communication, 2002).

Of the six sites sampled, perchlorate was measured above the analytical detection limit at two sites, Site 46 and 47 (Table 2). Considering that recent studies have indicated that perchlorate bio-accumulates in plants with unknown bio-magnification potential (Smith *et al.*, 2001), combined with the lack of soil criteria or standards currently available for comparative purposes, the detected concentrations at these sites (86 µg/kg at Site 46 and 96.5 µg/kg at Site 47) warrant further investigation.

CONCLUSIONS & RECOMMENDATIONS

Of the six sites sampled, Site 45 contained levels of beryllium and lead that were high enough to warrant additional investigations. Site 46 contained levels of semi-volatile organic compounds (three of the eight PAH compounds detected at this site exceeded ecological screening criteria), organochlorine pesticides (primarily total-DDT), total-PCBs, and perchlorate that warrant further investigation, while Site 47 contained levels of perchlorate that also warrant further investigation. Additional investigations may also be necessary at Site 48 because the level of γ -BHC detected at this site exceeded the ecological benchmark value. Neither of the remaining two sites investigated (Sites 44 and 49) contained contaminants at high enough levels where significant adverse impacts to ecological resources would be expected to occur.

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PERSONAL COMMUNICATIONS

Sher, J. 2002. Texas Commission on Environmental Quality. telephone: 512/239-2444

APPENDIX A
(ANALYTICAL METHODS)

Method Code: 003 for % Moisture, % Dry Weight
Laboratory: Geochemical % Environmental Research Group, Texas A&M

Approximately 1 gram of wet sample is weighed into a clean, labeled, pre-weighed 10 ml beaker. The beaker is placed in a forced air oven at approximately 75°Celsius for 24 hours. The beaker with the dry sample is then weighed and the % dry weight is calculated by the formula:

$$\frac{(\text{wt. dry sample and beaker}) - (\text{wt. beaker})}{(\text{wt. wet sample and beaker}) - (\text{wt. beaker})}(100)$$

Method Code: 004 for 1,2,3,4-tetrachlorobenzene, 1,2,4,5-tetrachlorobenzene, aldrin, hexachlorobenzene (HCB), heptachlor, alpha hexachlorocyclohexane (α BHC), alpha (α) chlordane, beta hexachlorocyclohexane (β BHC), cis-nonachlor, delta hexachlorocyclohexane (δ BHC), dieldrin, endosulfan II, endrin, gamma hexachlorocyclohexane (γ BHC), gamma (γ) chlordane, heptachlor epoxide, mirex, o,p'-dichloro-diphenyl-dichloroethane (o,p'-DDD), o,p'-dichloro-diphenyl-trichloroethane (o,p'-DDT), oxychlordane, p,p'-dichloro-diphenyl-dichloroethane (p,p'-DDD), p,p'-dichloro-diphenyl-dichloroethylene (p,p'-DDE), p,p'-dichloro-diphenyl-trichloroethane (p,p'-DDT), pentachloro-anisole, toxaphene, trans-nonachlor, and total polychlorobiphenyls (PCBs)

Laboratory: Geochemical % Environmental Research Group, Texas A&M

The soil/sediment samples were freeze dried and extracted in a Soxhlet extraction apparatus. Briefly, the freeze dried soil/sediment samples were homogenized and a 10 gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and the samples extracted for 12 hours. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod *et al.*, 1985; Brooks *et al.*, 1989) to isolate the pesticide and PCB fractions. The quantitative analyses were performed by capillary gas chromatography (CGC) with electron capture detector for pesticides and PCBs (Wade *et al.*, 1988). There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide endosulfan I and the PCB congeners 114 and 157. In these cases, the samples were analyzed by CGC with mass spectrometer detector in the SIM mode.

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Method Code: 006 for Soil/Sediment Clay, Silt, and Sand Grain Sizes
Laboratory: Geochemical % Environmental Research Group, Texas A&M

A small aliquot of sediment is treated with 30% hydrogen peroxide to remove organic coating from grains. A dispersing agent is then added to the sample. The sand/mud fractions are then separated using a 63 micron sieve. The sand fraction (greater than 63 microns) is retained on the screen and the mud fraction (silt and clay less than 63 microns) is washed into a 1 liter volumetric cylinder. The sand fraction is dried, sieved on a 63 micron screen and weighed. The sediment which passes through the screen a second time is added to the 1 liter cylinder. The mud fraction is analyzed by stirring the cylinder and sampling 20 ml aliquots at 4 and 8 phi intervals. The 4 and 8 phi samples are dried and weighed. The % sand, silt, and clay fractions are determined on a dry weight basis.

Method Code: 031 for 1,2,4-trichlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1-chloronaphthalene, 1-naphthylamine, 2,3,4,6-tetrachlorophenol, 2,4,5-trichlorophenol, 2,4,6-trichlorophenol, 2,4-dichlorophenol, 2,4-dimethylphenol, 2,4-dinitrophenol, 2,4-dinitrotoluene, 2,6-dichlorophenol, 2,6-dinitrotoluene, 2-chloronaphthalene, 2-chlorophenol, 2-methylphenol, 2-naphthylamine, 2-nitroaniline, 2-nitrophenol, 2-picoline, 2-methylnaphthalene, 3,3'-dichlorobenzidine, 3-methylcholanthrene, 3-nitroaniline, 4,6-dinitro-2-methylphenol, 4-aminobiphenyl, 4-bromophenylphenylether, 4-chloro-3-methylphenol, 4-chloroaniline, 4-chlorophenylphenylether, 4-methylphenol, 4-nitrophenol, 7,12-dimethylbenz(a)anthracene, acetophenone, aniline, benzidine, benzo(a)anthracene, benzoic acid, benzyl alcohol, bis(2-chloroethoxy)methane, bis(2-chloroethyl)ether, bis(2-ethylhexyl)phthalate, bis(2chloroisopropyl)ether, butylbenzylphthalate, carbazole, di-n-butylphthalate, di-n-octylphthalate, dibenz(a,h)anthracene, dibenz(a,j)acridine, dibenzofuran, diethyl phthalate, dimethylphthalate, diphenylamine, ethyl methanesulfonate, hexachlorobutadiene, hexachlorocyclopentadiene, hexachloroethane, isophorone, methyl methanesulfonate, n-nitroso-di-n-propylamine, n-nitrosopiperidine, nitrobenzene, pentachlorobenzene, pentachloronitrobenzene, pentachlorophenol, phenacetin, phenol, pronamide, a,a-dimethylphenylamine, acenaphthalene, acenaphthene, anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, benzo(k)fluoranthene, chrysene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, n-nitrosodiphenylamine, naphthalene, p-dimethylaminoazobenzene, phenanthrene, and pyrene
Laboratory: Geochemical % Environmental Research Group, Texas A&M

The soil/sediment samples are dried with sodium sulfate; surrogate standards are added and the soil/sediment sample is extracted with methylene chloride in an Accelerated Solvent Extraction (ASE) apparatus. Copper is added to remove elemental sulfur. The quantitative analyses were

performed by capillary gas chromatography (GC) with a mass spectrometer detector in the SCAN mode for semivolatile hydrocarbons (EPA Contract Laboratory for Organic Analyses OLM04.2 and EPA 8270). The compound list is based on the EPA CLP protocol but analytes from SW846 Method 8270 have been added.

References: Qian, Y., J.L. Sericano, and T.L. Wade. 1998. Extraction Tissues for Trace Organic Analysis. In Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update. G.G. Lauenstein and A.Y. Cantillo, eds. NOAA Technical Memorandum NOS ORCA 130. pp 98-101.

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Method Codes: 001, 004, and 006 for aluminum, barium, beryllium, boron, cadmium, chromium, copper, iron, lead, magnesium, manganese, molybdenum, nickel, strontium, vanadium, and zinc.

Laboratory: Research Triangle Institute

Homogenization (001) - Soil/sediment samples are pre-homogenized using a food processor. A portion of the sample is then freeze dried for determination of moisture content and ground to 100 mesh with a mill.

Digestion for Graphite Furnace and Cold Vapor Atomic Absorption (GFAA) Measurement (004) - Using a CEM microwave oven, 0.25 to 0.5 grams of freeze dried sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 15 minutes at 450 watts. The residue is then diluted to 50 ml with laboratory pure water.

ICP (006) - ICP measurements are made using a Leeman Labs Plasma Spec 1 sequential or ES2000 simultaneous spectrometer.

Method Codes: 001, 004, and 007 for arsenic and selenium.

Laboratory: Research Triangle Institute

Homogenization (001) - Soil/sediment samples are pre-homogenized using a food processor. A portion of the sample is then freeze dried for determination of moisture content and ground to 100 mesh with a mill.

Digestion for Graphite Furnace and Cold Vapor Atomic Absorption (GFAA) Measurement (004) - Using a CEM microwave oven, 0.25 to 0.5 grams of freeze dried sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 15 minutes at 450 watts. The residue is then diluted to 50 ml with laboratory pure water.

GFAA (007) - GFAA measurements are made using a Perkin-Elmer Zeeman 3030 or 4100ZL atomic absorption spectrometer.

Method Codes: 001, 004, and 008 for mercury.
Laboratory: Research Triangle Institute

Homogenization (001) - Soil/sediment samples are pre-homogenized using a food processor. A portion of the sample is then freeze dried for determination of moisture content and ground to 100 mesh with a mill.

Digestion for Graphite Furnace and Cold Vapor Atomic Absorption (GFAA) Measurement (004) - Using a CEM microwave oven, 0.25 to 0.5 grams of freeze dried sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 15 minutes at 450 watts. The residue is then diluted to 50 ml with laboratory pure water.

Cold Vapor Atomic Absorption (CVAA) - mercury measurements are conducted using SnC14 as the reducing agent. A Leeman PS200 Mercury Analyzer is employed for the analysis.

Method Codes: 001, 004, and 039 for silver.
Laboratory: Research Triangle Institute

Homogenization (001) - Soil/sediment samples are pre-homogenized using a food processor. A portion of the sample is then freeze dried for determination of moisture content and ground to 100 mesh with a mill.

Digestion for Graphite Furnace and Cold Vapor Atomic Absorption (GFAA) Measurement (004) - Using a CEM microwave oven, 0.25 to 0.5 grams of freeze dried sample are heated in a capped 120 ml Teflon vessel in the presence of 5 ml of Baker Instra-Analyzed nitric acid for three minutes at 120 watts, three minutes at 300 watts, and 15 minutes at 450 watts. The residue is then diluted to 50 ml with laboratory pure water.

ICP-MS (039) - sample is measured by ICP-MS.

Method Code: Modification of EPA Method 314.0 for perchlorate
Laboratory: Institute of Environmental and Human Health, Texas Tech University

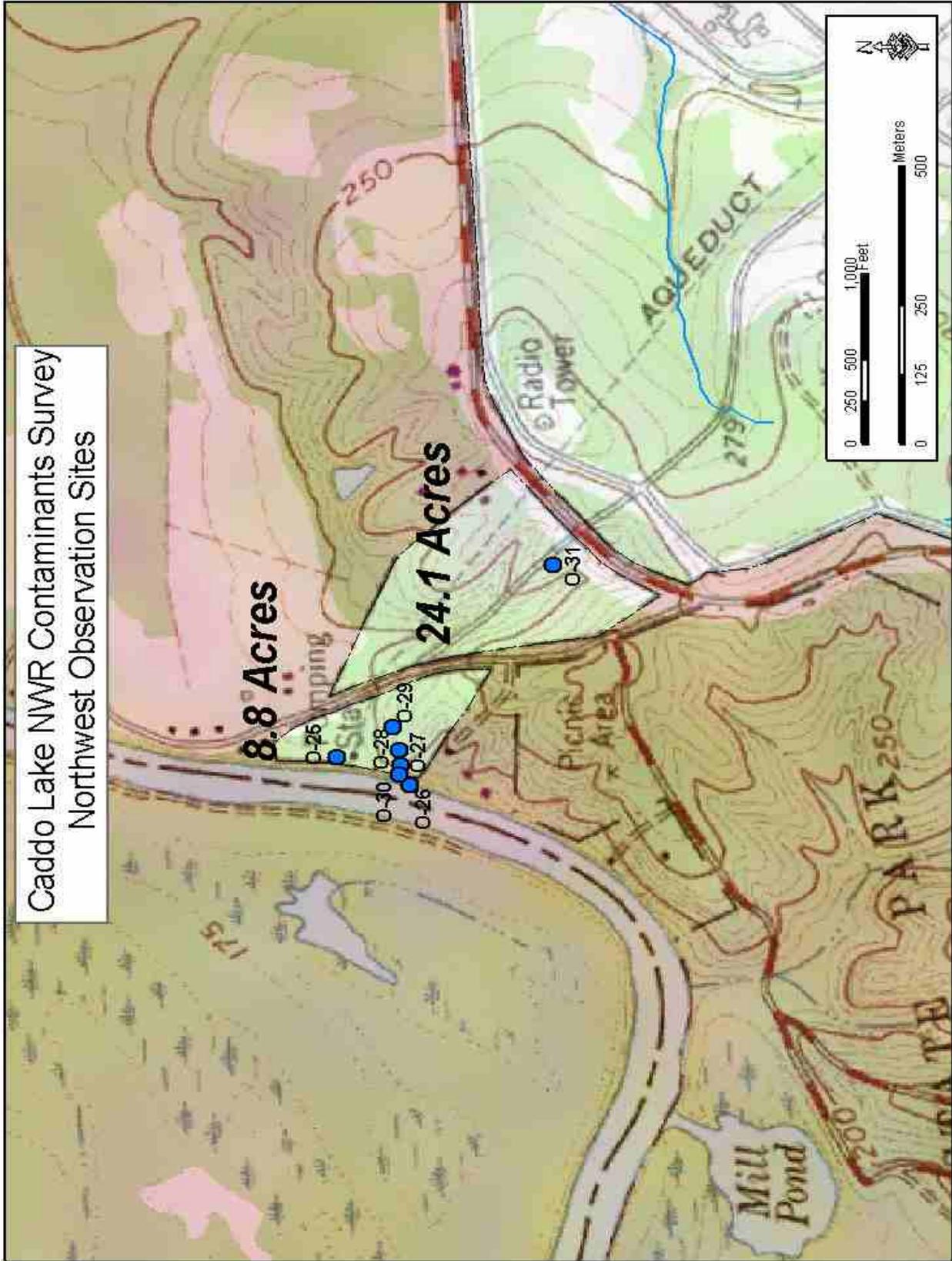
Soil/sediment samples were analyzed by ion chromatography using a modification of EPA Method 314.0 because at the time this study was conducted, the USEPA had not established a method for analyzing soil matrices for perchlorate.

APPENDIX B
(FIELD OBSERVATIONS)

FIELD OBSERVATIONS

Any type of anomalies (i.e., drum, pipeline, solid waste, foundations, etc.) observed during the field sampling conducted in December, 2002, were noted. Coordinates for these sites were entered into a geographic information system database and are presented on the map on page B2. Photographs of these observations are presented on pages B3 through B5.

Caddo Lake NWR Contaminants Survey
Northwest Observation Sites





Observation Point 25 (O-25). Surface Water Pump House adjacent to Big Cypress Bayou.



Observation Point 26 (O-26). Northwest corner of property fence line adjacent to Big Cypress Bayou.



Observation Point 27 (O-27). Portion of concrete foundation.



Observation Point 28 (O-28). Well House.



Observation Point 29 (O-29). Discarded drum.



Observation Point 30 (O-30). Fallen light post.



Observation Point 31 (O-31). Discarded pipe.