U. S. Fish and Wildlife Service
Region 2

AN INVESTIGATION OF CONTAMINANT LEVELS IN WHITE-TAILED DEER (*Odocoileus virginianus*) COLLECTED FROM CADDO LAKE NATIONAL WILDLIFE REFUGE, HARRISON COUNTY, TEXAS 2005

Project ID Nos.: DEC No. 200520002; FFS No. 2N53

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ABSTRACT

In January and February, 2005, a contaminants investigation was conducted at Caddo Lake National Wildlife Refuge (CLNWR) by the United States Fish and Wildlife Service (USFWS). The purpose of the investigation was to determine contaminant levels of organochlorine pesticides, metals, and total polychlorinated biphenyls (PCBs) in muscle tissue and livers of white-tailed deer (*Odocoileus virginianus)* on the Refuge. Samples were collected from 20 deer for this investigation. Texas Parks and Wildlife Department personnel collected brain stems samples from five of the harvested deer and analyzed them for the presence of protease-resistant prion protein (PrP-res), the presence of which is diagnostic for chronic wasting disease (CWD). Results from this study were compared to human health screening criteria and will be used to determine if public hunting of white-tailed deer will be allowed on the Refuge and whether consumption of meat from the deer poses a human health risk.

Eleven metals (aluminum, barium, boron, chromium, copper, lead, manganese, nickel, selenium, strontium, and zinc) and two pesticides (mirex and oxychlordane) were detected in one or more muscle samples. Thirteen metals (aluminum, barium, boron, cadmium, chromium, copper, lead, manganese, molybdenum, nickel, selenium, strontium, and zinc) and five pesticides; total dichlorodiphenyl trichloroethane (total DDTs), total hexachlorocyclohexane (total BHC), oxychlordane, o,p'- dichlorodiphenyl dichloroethylene (o,p'-DDE), and gamma hexachlorocyclohexane (gamma-BHC) were detected in one or more liver samples. All contaminants in muscle samples were below their respective health based screening values and all but three contaminants (cadmium, copper, and selenium) in livers were below their respective health based screening values. Levels of contaminants in muscle tissue were not elevated enough to pose a threat to human health. Selenium in one liver tissue sample exceeded the health based screening value for children and adults. Cadmium and copper levels in deer livers may pose adverse health effects and therefore should not be consumed.

Key words: DEC No. 200520002, FFS No. 2N53, Texas Congressional District No. 1, organochlorine pesticides, cadmium, copper, *Odocoileus virginianus*, Caddo Lake NWR, Longhorn Army Ammunition Plant, Selenium, Texas

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TABLE OF CONTENTS

LIST OF FIGURES

AN INVESTIGATION OF CONTAMINANT LEVELS IN WHITE-TAILED DEER (*Odocoileus virginianus*) COLLECTED FROM CADDO LAKE NATIONAL WILDLIFE REFUGE, HARRISON COUNTY, TEXAS 2005
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LIST OF ACRONYMS/ABREVIATIONS

AI	adequate intakes
AMCCOM	Armament, Munitions, and Chemical Command
ATSDR	Agency for Toxic Substances and Disease Registry
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLNWR	Caddo Lake National Wildlife Refuge
CWD	chronic wasting disease
DDD	dichlorodiphenyldichloroethane
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DRI	dietary reference intake
IHC	immunohistochemistry
LHAAP	Longhorn Army Ammunition Plant
MRL	minimal risk levels
NPL	National Priority List
PCBs	Polychlorinated biphenyls
PrP-res	protease-resistant prion protein
RDA	recommended dietary allowance
RfD	reference doses
TDSHS	Texas Department of State Health Services
TNT	trinitrotoluene
TPWD	Texas Parks and Wildlife Department
TVMDLS	Texas Veterinary Medical Diagnostic Laboratory System
UL	tolerable upper intake levels
USDOD	United States Department of Defense
USEPA	United States Environmental Protection Agency
USFWS	United States Fish and Wildlife Service

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INTRODUCTION

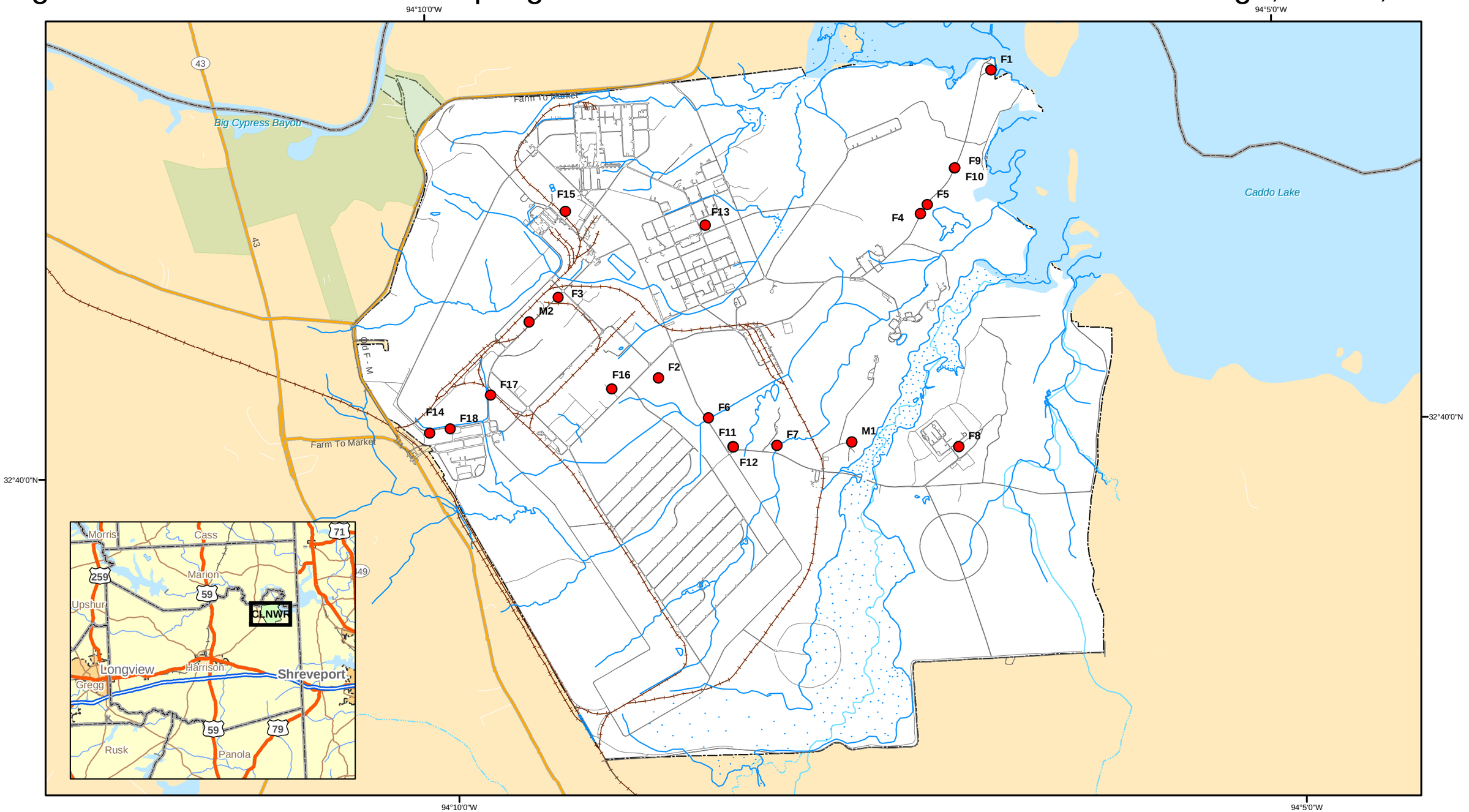
In January and February, 2005, the United States Fish and Wildlife Service (USFWS) conducted a contaminants investigation at Caddo Lake National Wildlife Refuge (CLNWR). The purpose of the investigation was to determine contaminant levels of metals and organochlorine pesticides, and total polychlorinated biphenyls (total-PCBs), in white-tailed deer (*Odocoileus virginianus),* on the Refuge. Texas Parks and Wildlife Department (TPWD) personnel collected samples from a portion of the harvested deer and analyzed the brain stems for the presence of protease-resistant prion protein (PrP-res), the presence of which is diagnostic for chronic wasting disease (CWD). Results will determine if public hunting white-tailed deer will be allowed on the Refuge and whether consumption of meat from the deer poses a human health risk.

STUDY AREA & BACKGROUND

Caddo Lake National Wildlife Refuge (CLNWR) is located on the site of Longhorn Army Ammunition Plant (LHAAP), a former United States Department of Defense (USDOD) ammunition production facility, in Harrison County, Texas (Figure 1). The entire site consists of 8,493 acres (3,437 hectares) of mixed upland pine and bottomland hardwood forests interlaced with remnant structures from the munitions plant. Most of the structures have been demolished leaving only foundations and areas where contaminant investigations are currently being conducted by the USDOD on property still under their primary jurisdiction. The site is drained by four principal lotic systems, Goose Prairie Bayou, Central Creek, Harrison Bayou, and Saunders Branch, all flowing into Caddo Lake which is located on the northeast boundary of the refuge.

The former munitions plant was established by the USDOD under the jurisdiction of the U.S. Army Armament, Munitions, and Chemical Command (AMCCOM) in 1941 to produce trinitrotoluene (TNT) flake through the contract operator Monsanto Chemical Company (TSHA 2002). The plant produced over four hundred million pounds (greater than 180 million kilograms) of TNT between 1942 and 1945 (TSHA, 2002). In late 1945, TNT production ceased and Monsanto suspended all operations at the site, while the facility was placed on standby status by the USDOD (TSHA 2002). The plant remained inactive until 1952, when operations were re-initiated under the contract operator, Universal Match Corporation to produce pyrotechnic and illuminating ammunition such as photoflash bombs, simulators, hand signals, and 40 millimeter tracers (GS 2002; TSHA 2002). By 1956, Morton-Thiokol Incorporated (formerly known as the Thiokol Corporation) had assumed contract operation responsibilities at the facility (GS 2002; TSHA 2002). From 1956 through 1965, the primary mission of the plant was the production of solid propellant rocket motors and fuels for the Nike-Hercules, Falcon, Lacrosse, Honest John,

Figure 1: White-tailed deer sampling locations at Caddo Lake National Wildlife Refuge, Texas, 2005



U.S. Fish & Wildlife Service

Arlington, Texas, Ecological Services Field Office
Projection: UTM Zone 14N, NAD 1983, GRS 1980
Production Date: 3/28/2005

Sample locations
Streams
Roads
Railroads
CLNWR boundary (approximate)

0 0.5 1 Miles
0 0.5 1 Kilometers

Texas

and Sergeant Missile programs (GS 2002). In 1965, the production of pyrotechnic and illuminating ammunition was re-initiated at the plant by Thiokol. The plant continued to produce munitions all during the 1960s and 1970s. At its peak, the facility employed over 2,200 people (Tolbert, personal communication 2002).

In 1987, the LHAAP was selected as one of the sites for the static firing and elimination of Pershing IA and II rocket motors in order to comply with the terms of the Intermediate Nuclear Force Treaty between the U.S. and the Soviet Union (GS 2002). This project was completed by 1991 (TSHA 2002). In 1990, the facility was placed by the United States Environmental Protection Agency (USEPA) on the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) National Priority List (NPL). This listing as a Superfund site was due to groundwater, surface water, sediments, and soil contamination (ATSDR 2002). Contaminants associated with the listing included metals, explosives, semi-volatile organic compounds, and volatile organic compounds (ATSDR 2002). Activities to remediate this contamination were initiated in 1990 and are expected to be completed no earlier than 2030. Thiokol continued operations at the plant, primarily with the production of the plastic explosive CL-20. This continued until 1997 (ATSDR 2002). By 1998, Thiokol had ceased operations at the site and AMCCOM had classified the plant as excess property. In 1999, negotiations were initiated between AMCCOM and USFWS over the possible absorption of the site into the National Wildlife Refuge System. In October, 2000, approximately 7,200 acres (2,914 hectares) of the LHAAP became CLNWR, an overlay refuge, with the U.S. Army maintaining administrative control of the entire property until primary jurisdiction for the site was deemed suitable for transfer to the USFWS.

In 2002, 2003, and 2005, the USFWS conducted multiple contaminants investigations in the overlay portion of CLNWR and portions of the former production area of LHAAP (Giggleman and Lewis 2002; Giggleman and Lewis 2003; Giggleman and Lewis 2005). The purpose of these investigations was to determine contaminant (metals, semi-volatile organic compounds, organochlorine pesticides, total polychlorinated biphenyls, dioxin/furans, and perchlorate) levels in soils and/or sediments in the overlay portions of the Refuge and the portions of the former production area that were not expected to have been impacted by munitions activities. Surficial grab soil and/or sediment samples were collected from 282 sites by USFWS personnel in April and May, 2002, March and April, 2003, and June, 2005. The overall area sampled covered approximately 7,928 acres (3,208 hectares). Data resulting from these investigations were used by the USFWS to determine the suitability of transfer of administrative control of acceptable portions of the Refuge from the U.S. Army to the USFWS. Contaminants associated with past operations detected by USFWS personnel at elevated levels in the overlay area included; metals, mainly cadmium, lead, and mercury; organochlorines; and perchlorate (Giggleman and Lewis 2002; Giggleman and Lewis 2003).

On May 5, 2004, administrative control of approximately 5,032 acres (2,037 hectares) of the overlay refuge was officially transferred from the USDOD to the USFWS. The USDOD still retains administrative control of the remaining 2,500 acres (1,012 hectares) of property within the boundaries of CLNWR pending completion of remedial actions at various CERCLA sites.

Transfer of the portion of the former production area which includes 728 acres (294 hectares) is pending.

As part of their historic resource management strategy, the U.S. Army allowed public deer hunting on an annual basis. The hunting was conducted every deer season and only opened to limited LHAAP personnel. As the Service assumes administrative control of portions of the site, it is projected that public deer hunting will be allowed in the future. However, because of the potential for absorption of contaminants in their diet from contaminated areas the deer were analyzed for contaminants prior to any public deer hunting. Testing the edible muscle tissues from a sub-sample of the deer population (twenty deer) was conducted to address potential human health concerns associated with conducting public hunting at the site.

METHODS & MATERIALS

Twenty white-tailed deer (*Odocoileus virginianus*) were harvested by FWS personnel on the CLNWR following the completion of the Fall Texas hunting season. Deer were harvested, with a rifle, at various times over several weeks starting on January 15th, 2005, and continuing through February 19th, 2005, until 20 deer were collected. Initial plans were to harvest a proportionate number of bucks to does, but eventually it was decided to harvest the deer as they were encountered due to the fact that it was late in the season and bucks were beginning to shed their antlers. Eighteen female deer (does) and two male deer (bucks) were harvested. The ratio of does to bucks (9:1) corresponds to past hunting season records maintained at the facility when the U.S. Army allowed deer hunting by LHAAP personnel.

Immediately after collection, using disposable nitrile gloves and disposable surgical scalpels with stainless steel surgical blades, portions of the muscle tissue in the back strap (loin) and hind quarters were collected from each deer and composited as one tissue sample per deer. Each sample was cut into two portions. One portion was individually vacuum sealed and frozen. The remaining portion was wrapped in foil (shiny side out) then vacuum sealed and frozen. Samples were numbered F1 through F18 for does and M1 and M2 for bucks to differentiate between female and male. The samples were submitted for analyses through the Patuxent Analytical Control Facility to TDI-Brooks International Inc., College Station, Texas and analyzed for organochlorine pesticides; [1,2,3,4-Tetrachlorobenzene, 1,2,4,5-Tetrachlorobenzene, aldrin, alpha chlordane, alpha hexachlorocyclohexane (alpha-BHC), beta hexachlorocyclohexane (beta-BHC), chlorpyrifos, cis-nonachlor, DDMU, delta hexachlorocyclohexane (delta-BHC), dieldrin, endosulfan I, endosulfan II, endosulfan sulfate, endrin, gamma hexachlorocyclohexane (gamma-BHC), gamma chlordane, heptachlor epoxide, hexachlorobenzene (HCB), mirex, o,p'-dichlorodiphenyl dichloroethane (o,p'-DDD), o,p'-dichlorodiphenyl dichloroethylene (o,p'-DDE), o,p'-dichlorodiphenyl trichloroethane (o,p'-DDT), oxychlordane, p,p'-dichlorodiphenyl dichloroethane (p,p'-DDD), p,p'-dichlorodiphenyl dichloroethylene (p,p'-DDE), p,p'-dichlorodiphenyl trichloroethane (p,p'-DDT), pentachloro-anisole, pentachlorobenzene, toxaphene, trans-nonachlor, and total polychlorinated biphenyls (total-PCBs) in micrograms/kilogram (ug/kg). The remaining samples were submitted to Laboratory and Environmental Testing, Inc., Columbia, Missouri and analyzed for percent moisture content and

metals; aluminum, arsenic, barium, beryllium, boron, cadmium, chromium, copper, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, selenium, strontium, vanadium, and zinc, in milligrams/kilograms (mg/kg). Liver samples were collected from 18 of the 20 deer sampled. Whole livers were wrapped in foil, vacuum sealed, and frozen and submitted to the same testing facilities for analyses of the same contaminants (for specific analytical methods see Appendix A). The resulting data were compared to known human health values, where available, as well as with data from comparative studies.

Results were also submitted to the Texas Department of State Health Services (TDSHS) for evaluation. In addition, brain stems were collected from five of the twenty deer and submitted by TPWD to the Texas Veterinary Medical Diagnostic Laboratory System (TVMDLS), College Station, Texas and analyzed for chronic wasting disease immunohistochemistry (CWD IHC).

RESULTS & DISCUSSION

Results for analyses are presented in Appendix B, Tables 1-4. All analytical results, except lead, were compared to the Agency for Toxic Substances and Disease Registry (ATSDR) minimal risk levels (MRLs) or EPA reference doses (RfDs) (ATSDR 2006), and where available, data from comparative studies to determine if there were possible adverse human health effects. For lead, the Texas Department of State Health Services (TDSHS) used the ATSDR "Framework to Guide Public Health Assessment Decisions at Lead Sites" to estimate probable increases in blood lead levels associated with tissue consumption (ATSDR 2006). Contaminants that were below detection limits and whose detection limits were below health based screening values were eliminated from further evaluation. This eliminated six metallic analytes (arsenic, beryllium, cadmium, mercury, molybdenum, and vanadium) and all but two organochlorine pesticides (mirex and oxychlordane) for the muscle tissue samples. For the liver tissue samples, this eliminated four metallic analytes (arsenic, beryllium, mercury, and vanadium) and all but five (gamma-BHC, o,p'-DDE, oxychlordane, total-BHC, and total-DDTs) of the organlchlorine pesticides. This also eliminated total-PCBs from further consideration in all muscle tissue and liver tissue samples. Metallic analytes for muscle tissue samples and liver tissue samples were evaluated using dry weight values while the organochlorine pesticides were evaluated using wet weight values (Bradford; personnel communication 2006, ATSDR 2006).

Metals in Muscle and Liver Tissue Samples

[Aluminum (Al)] Aluminum is a naturally occurring metal that has been utilized by humans for many years. It is readily available for human ingestion through the use of food additives, antacids, buffered aspirin, astringents, nasal sprays, and antiperspirants; from drinking water; from automobile exhaust and tobacco smoke; and from using aluminum foil, aluminum cookware, cans, ceramics, and fireworks (ATSDR 1995). Low-level exposure to aluminum from food, air, water, or contact with skin is not thought to harm your health. Aluminum, however, is not a necessary substance for our bodies and too much may be harmful. Aluminum has been found in at least 489 of the 1416 NPL sites identified by EPA (ATSDR 1995). Puls (1994) indicates that normal muscle tissue aluminum concentrations in sheep and cattle range from 2-

3.8 mg Al/kg dry weight and normal liver tissue aluminum levels range from 1-5 mg Al/kg dry weight while toxic levels for aluminum ranged from 6.3-11 mg Al/kg dry weight. Health based screening values for children were 2000 mg Al/kg dry weight and 4000 mg Al/kg dry weight for adults (ATSDR 2006).

Aluminum levels were detected above the analytical detection limit in 13 of the 20 muscle tissue samples (Appendix B, Table 1). Detected aluminum concentrations ranged from 2 mg Al/kg dry weight in samples F2M, F6M, F7M, F14M, and F18M to 12 mg Al/kg dry weight in sample F10M. In liver tissue samples, aluminum concentrations were detected at the detection limit of 2 mg Al/kg dry weight in samples F6L and F14L (Appendix B, Table 2). All detections are well below the health based screening levels reported by ATSDR (2006).

[Barium (Ba)] Barium compounds are used by the oil and gas industries to make drilling muds. They are also used to make paint, bricks, ceramics, glass, and rubber. Barium sulfate is sometimes used by doctors to perform medical tests and take x-ray photographs of the stomach and intestines (ATSDR 2005). Barium does not accumulate to any great extent in the liver (Puls 1994). Barium and barium compounds have been found in at least 798 of the 1,662 current or former NPL sites (ATSDR 2005). Health based screening values for children are 200 mg Ba/kg dry weight and 400 mg Ba/kg dry weight for adults (ATSDR 2006).

Barium levels were detected above the analytical detection limit in six of the 20 muscle tissue samples (Appendix B, Table 1). Concentrations of barium ranged from 0.3 mg Ba/kg dry weight in sample F1M, F11M, F13M, and M1M to 0.76 mg Ba/kg dry weight in sample F16M. Barium concentrations were detected in 15 liver tissue samples (Appendix B, Table 2). Concentrations of barium ranged from the detection limit of 0.2 mg Ba/kg dry weight in F18L and M2L to 1 mg Ba/kg dry weight in F8L. All detections are well below the health based screening levels reported by ATSDR (2006).

[Boron (B)] Boron is a compound that occurs in nature. It is often found combined with other substances to form compounds called borates. Common borate compounds include boric acid, salts of borates, and boron oxide. Several companies in the United States produce most of the world's borates by processing boron compounds. Borates are used mostly to produce glass. They are also used in fire retardants, leather tanning industries, cosmetics, photographic materials, soaps and cleaners, and for high-energy fuel. Some pesticides used for cockroach control and some wood preservatives also contain borates (ATSDR 1992). This chemical has been found in at least 142 of 1,416 NPL sites identified by the EPA (ATSDR 1992). Toxic levels in goat muscle tissue occur at 308 mg B/kg wet weight (Puls 1994). Health based screening values were 9 mg B/kg dry weight and 20 mg B/kg dry weight for children and adults, respectively (ATSDR 2006).

Boron levels were detected at the analytical detection limit in two of the twenty muscle tissue samples at 2 mg B/kg dry weight (Appendix B, Table 1). The two samples were F1M and F10M. All other muscle tissue samples were below the detection limit. Liver tissue sample concentrations had one sample, F6L, at the detection limit and one sample, F15L, which had a

boron concentration of 3 mg B/kg dry weight (Appendix B, Table 2). All detections are well below the health based screening levels reported by ATSDR (2006).

[Cadmium (Cd)] Food and cigarette smoke are the biggest sources of cadmium exposure for people in the general population. Average cadmium levels in U.S. foods range from 2 to 40 parts of cadmium per billion parts of food (2–40 ppb). Lowest levels are in fruits and beverages, and highest levels are in leafy vegetables and potatoes (ATSDR 1999). Cadmium stays in the body a very long time and can bio-accumulate over time with continued exposure. Long-term exposure to lower levels of cadmium in air, food, or water leads to a buildup of cadmium in the kidneys and possible kidney disease. Other long-term effects are lung damage and fragile bones (ATSDR 1999). Puls (1994) found normal levels in sheep livers to range from 0.02 – 1.4 mg Cd/kg wet weight. Lynch (1973) found that white-tailed deer from Ohio normally had cadmium values of 0.27 mg Cd/kg wet weight while white-tailed deer from Illinois had concentrations of 0.37 mg Cd/kg wet weight (Woolf et al. 1982). Munshower & Neuman (1979) found normal mean cadmium levels in livers of antelope and mule deer to be 0.3 mg Cd/kg freeze dried weight and 0.51mg Cd/kg freeze dried weight, respectively. Mean cadmium concentrations in white-tailed deer livers collected near a zinc smelter in Pennsylvania showed decreasing levels with distance from the smelter. Cadmium levels dropped from 11.6 mg Cd/kg dry weight for deer collected less than eight kilometers from the smelter to 1.9 mg Cd/kg dry weight for deer collected over 100 km from the smelter (Sileo and Beyer 1985). Cadmium has been found in at least 776 of the 1,467 NPL sites identified by the EPA (ATSDR 1999).

Cadmium was not found above the detection limit of 0.1 mg Cd/kg dry weight in any of the muscle tissue samples (Appendix B, Table 1). Cadmium concentrations above the detection limit were found in all 18 liver tissue samples (Appendix B, Table 2) and exceeded the health based screening values for children of 0.2 mg Cd/kg dry weight. Adult health based screening levels of 0.4 mg Cd/kg dry weight were exceeded in 17 of the 18 samples.

[Chromium (Cr)] Chromium is a naturally occurring element found in rocks, animals, plants, soil, and in volcanic dust and gases. Chromium is present in the environment in several different forms. The most common forms are chromium(0), chromium(III), and chromium(VI). Chromium(III) occurs naturally in the environment and is an essential nutrient. Chromium(VI) and chromium(0) are generally produced by industrial processes (ATSDR 2001). Chromium in excessive amounts can be mutagenic, carcinogenic, and teratogenic to many organisms (Eisler 1986). In cattle muscle tissue normal levels of chromium range from 0.1-0.2 mg Cr/kg wet weight (Puls 1984). Health based screening values for chromium range from 1000 mg Cr/kg dry weight in children to 3000 mg Cr/kg dry weight in Adults (ATSDR 2006). Woolf et al (1982) found mean chromium concentrations of 2.7 mg Cd/kg dry weight in white-tailed deer in Illinois. Puls (1984) found that in sheep normal liver concentrations of chromium ranged from 0.09-0.23 mg Cr/kg wet weight. Chromium has been found at 1,036 of the 1,591 NPL sites identified by the EPA (ATSDR 2001).

Chromium was detected above the analytical detection limit in ten of the twenty muscle tissue samples (Appendix B, Table 1). Concentrations ranged from 0.6 mg Cr/kg dry weight in samples F3M, F7M, and F9M to 1.5 mg Cr/kg dry weight in sample M1M. Chromium

concentrations were detected above the analytical detection limit in four liver tissue samples, M2L at a level of 0.6 mg Cr/kg dry weight, F12L at a level of 0.7 mg Cr/kg dry weight, and F18L and M1L at levels of 1 mg Cr/kg dry weight (Appendix B, Table 2). All detections were well below the health based screening values reported by ATSDR (2006).

[Copper (Cu)] Because of its high electrical conductivity copper is used extensively in the manufacturing of electrical equipment, pipe and different metallic alloys. Copper compounds are commonly used in agriculture to treat plant diseases like mildew, for water treatment and, as preservatives for wood, leather, and fabrics (ATSDR 2004). Copper is an essential micronutrient that interacts with other trace elements in animals including iron, zinc, manganese, nickel and selenium (Eisler 1998). Copper also interacts with nonessential elements such as cadmium, mercury, and lead (Eisler 1998, Goyer 1991). Adequate levels of copper in muscle tissue in sheep range from 1.0-1.3 mg Cu/kg wet weight. Adequate levels mean levels sufficient for optimum functioning of all body mechanisms with a small margin of reserve to counteract commonly encountered antagonistic conditions (Puls, 1994). These levels were well above the mean copper levels reported in Montana mule deer (46.3 mg Cu/kg freeze dry weight) and antelope (26.9 mg Cu/kg freeze dry weight) by Munshower & Neuman (1979). Woolf (1982) found mean levels of 109.0 mg Cu/kg dry weight in white-tailed deer from Ohio. Puls (1994) determined that levels from 25-100 mg Cu/kg wet weight in deer livers was adequate, but that levels from 250-1000 mg Cu/kg wet weight in livers of sheep could be toxic, and levels from 250-400 could be toxic in llamas, alpaca, and guanaco. Sileo and Beyer (1985) found that mean copper levels dropped from 190 mg Cu/kg dry weight for deer collected less than eight km from a Pennsylvania zinc smelter to 106 mg Cu/kg dry weight for deer collected over 100 km from the smelter. Copper has been found in at least 906 of the 1,647 National Priority Sites identified by the EPA (ATSDR 2004).

Copper was detected in all 20 muscle tissue samples (Appendix B, Table 1). Concentrations ranged from 5.7 mg Cu/kg dry weight in sample F2M to 8.6 mg Cu/kg dry weight in sample F4M. The copper concentrations for all 18 liver samples exceeded the child health based screening value of 9.0 mg Cu/kg dry weight and the adult health based screening value of 22.0 mg Cu/kg dry weight (ATSDR 2006) (Appendix B, Table 2). Copper concentrations ranged from 22 mg Cu/kg dry weight in F6L to 949 mg Cu/kg dry weight in F17L.

[Iron (Fe)] Iron, one of the most abundant metals on Earth, is essential to most life forms and to normal human physiology. Iron is an integral part of many proteins and enzymes that maintain good health (ODS 2005). Iron is a necessary nutrient that is a constituent of many enzymatic processes and other cellular functions (Horne and Goldman, 1994). Iron is essential for both the maintenance of oxidative systems within the tissue cells and the transport of oxygen to the tissues (Guyton, 1981). Recommendations for iron are provided in the Dietary Reference Intakes (DRIs) developed by the Institute of Medicine of the National Academy of Sciences. *Dietary Reference Intakes* is the general term for a set of reference values used for planning and assessing nutrient intake for healthy people. Three important types of reference values included in the DRIs are *Recommended Dietary Allowances* (RDA), *Adequate Intakes* (AI), and *Tolerable Upper Intake Levels* (UL). The RDA recommends the average daily intake that is sufficient to meet the nutrient requirements of nearly all (97-98%) healthy individuals in each age and gender

group. An AI is set when there is insufficient scientific data available to establish a RDA. AIs meet or exceed the amount needed to maintain a nutritional state of adequacy in nearly all members of a specific age and gender group. The UL, on the other hand, is the maximum daily intake unlikely to result in adverse health effects (IOM 2001). Iron RDA levels in children range from 7-15 mg/day and from 8-18 mg/day in adults (ODS 2005).

Iron was detected above the detection limit in all 20 muscle tissue samples (Appendix B, Table 1). Concentrations ranged from 88 mg Fe/kg dry weight in sample F2M to 170 mg Fe/kg dry weight in samples F4M and F5M. Iron concentrations were above the analytical detection limit in all 18 liver tissue samples (Appendix B, Table 2). Concentrations ranged from 215 mg Fe/kg dry weight in F16L to 612 mg Fe/kg dry weight in F1L. In 2001, the Institute of Medicine of the National Academy of Sciences set a tolerable upper intake level (UL) for iron for healthy people at 40 mg/day for children and 45 mg/day for adults (ODS 2005).

[Lead (Pb)] Lead is the fifth most utilized metal in the U.S. and is used for the production of ammunition, batteries, metal products (solder and pipes), gasoline products, ceramics, and weights, and devices to shield X-rays. Because of health concerns, lead from gasoline, paints and ceramic products, caulking, and pipe solder has been dramatically reduced in recent years (ATSDR 2005). In liver tissues of normal white-tailed deer in Illinois Woolf et al. (1982) found lead values of 4.4 mg Pb/kg dry weight. Munshower & Neuman (1979) found normal mean lead levels in livers of antelope and mule deer to be 0.6 mg Pb/kg freeze dried weight and 0.9 mg Pb/kg freeze dried weight, respectively. Sileo and Beyer (1985) found that normal white-tailed deer in Pennsylvania had mean lead concentrations of 0.2 mg Pb/kg dry weight. The TDSHS uses the ATSDR "Framework to Guide Public Health Assessment Decisions at Lead Sites" to estimate probable increases in blood lead levels associated with tissue consumption. It is estimated that increased blood lead levels in children would be <1.4ug/dL (micrograms per deciliter) and <0.4 ug/dL in adults (ATSDR 2006).

Lead concentration levels were detected above the analytical detection limit in two of the twenty muscle tissue samples (Appendix B, Table 1). Sample F6M had a lead concentration of 0.4 mg Pb/kg dry weight and sample F1M had a lead concentration of 5.2 mg Pb/kg dry weight. Lead levels were detected above the analytical detection limit in one liver tissue sample, F8L, at a concentration of 1.9 mg Pb/kg dry weight (Appendix B, Table 2). Based on the results using the ATSDR "Framework to Guide Public Health Assessment Decisions at Lead Sites" and the maximum lead concentration found in muscle tissue of 5.2 mg/kg, it was estimated that increased blood lead levels were not a health concern (ATSDR 2006).

[Magnesium (Mg)] Magnesium is the fourth most abundant mineral in the body and is essential to good health. Magnesium is needed for more than 300 biochemical reactions in the body (ODS 2005). Magnesium is an essential life nutrient that all living cells require for energy transfer. It is a catalyst that causes the change of adenosine triphosphate (ATP) to adenosine diphosphate (ADP) (Horne and Goldman, 1994). White-tailed deer from Illinois had mean concentrations of 205.0 mg Mg/kg wet weight (Woolf et al. 1982). Puls (1994) found that normal levels of magnesium in sheep muscle tissue range from 218-265 mg Mg/kg wet weight and in sheep liver tissue range from 118-200 mg Mg/kg wet weight. Recommendations for magnesium are

provided in the Dietary Reference Intakes (DRIs) developed by the Institute of Medicine of the National Academy of Sciences. *Dietary Reference Intakes* is the general term for a set of reference values used for planning and assessing nutrient intake for healthy people. Three important types of reference values included in the DRIs are *Recommended Dietary Allowances* (RDA), *Adequate Intakes* (AI), and *Tolerable Upper Intake Levels* (UL). The RDA recommends the average daily intake that is sufficient to meet the nutrient requirements of nearly all (97-98%) healthy individuals in each age and gender group. An AI is set when there is insufficient scientific data available to establish a RDA for specific age/gender groups. AIs meet or exceed the amount needed to maintain a nutritional state of adequacy in nearly all members of a specific age and gender group. The UL, on the other hand, is the *maximum* daily intake unlikely to result in adverse health effects (IOM 1999). Magnesium RDA levels in children range from 80-410 mg/day and from 310-420 mg/day in adults (ODS 2005).

Magnesium levels were detected above the analytical detection limit in all 20 muscle tissue samples (Appendix B, Table 1). Concentrations ranged from 913 mg Mg/kg dry weight in F18M to 1120 mg Mg/kg dry weight in F15M. In the liver tissue samples magnesium concentrations exceeded the analytical detection limit in all 18 samples (Appendix B, Table 2). Concentrations ranged from 514 mg Mg/kg dry weight in F9L to 612 mg Mg/kg dry weight in F1L. In 1999, the Institute of Medicine of the National Academy of Sciences set a tolerable upper intake level (UL) for supplemental magnesium for healthy children at a range of 65-350 mg/day and 350 mg/day for adults (IOM 1999). There is no UL for dietary intake of magnesium; only for magnesium supplements (ODS 2005).

[Manganese (Mn)] Manganese is a necessary nutrient for plants and animals. For the most part it is nontoxic to aquatic biota (Cole, 1983). Puls (1994) found that adequate levels in sheep muscle tissue ranged from 0.24-0.4 mg Mn/kg dry weight and in liver tissue adequate levels ranged from 2.0-4.4 mg Mn/kg wet weight. Munshower & Neuman (1979) found normal mean manganese levels in livers of antelope and mule deer to be 7.3 mg Mn/kg freeze dried weight and 9.4 mg Mn/kg freeze dried weight, respectively. White-tailed deer from Illinois had mean concentrations of 8.4 mg Mn/kg wet weight (Woolf et al. 1982).

Manganese levels were detected at the detection limit of 0.5 mg Mn/kg dry weight in two of the muscle tissue samples F12M and F18M, and above the analytical detection limit in the remaining 18 samples (Appendix B, Table 1). Concentrations ranged from 0.6 mg Mn/kg dry weight in F2M, F5M, F9M, F10M, F14M, F17M, and M1M to 0.8 mg Mn/kg dry weight in F4M, F8M, F11M, F13M, and M2M. In liver tissue samples, manganese concentrations were above analytical detection limits in all 18 samples (Appendix B, Table 2), ranging from 11 mg Mn/kg dry weight in F5L to 19 mg Mn/kg dry weight in F2L. All detections were well below the health based screening values reported by ATSDR (2006).

[Molybdenum (Mo)] Molybdenum is an essential life nutrient for most life forms and is even necessary for fixing atmospheric nitrogen by bacteria in plants; however toxicity can result from excessive exposure in both plants and animals (Goyer 1991). In sheep liver, normal levels of molybdenum range from 1.5-6 mg Mo/kg dry weight (Puls 1994). Puls (1994) also found that molybdenum deficiency and high copper levels (Cu:Mo >20:1) may induce copper toxicity.

Health based screening values for molybdenum ranged from 5 mg Mo/kg dry weight in children and 10 mg Mo/kg dry weight in adults (ATSDR 2006).

In muscle tissue samples there were no detections above the detection limit in any of the 20 samples (Appendix B, Table 1). Molybdenum levels were detected at the analytical detection limit in five of the eighteen liver tissue samples at 2 mg Mo/kg dry weight (Appendix B, Table 2). The five samples were F14M, F15L, F17L, F18L, and M2L and all contained detectable amounts below health based screening levels.

[Nickel (Ni)] Nickel is a very abundant natural element. Pure nickel is a hard, silvery-white metal. Nickel can be combined with other metals, such as iron, copper, chromium, and zinc, to form alloys. These alloys are used to make coins, jewelry, and items such as valves and heat exchangers. Most nickel is used to make stainless steel. Nickel compounds are used for nickel plating, to color ceramics, to make some batteries, and as substances known as catalysts that increase the rate of chemical reactions. The most common harmful health effect of nickel in humans is an allergic reaction (ATSDR 2005). Nickel does not appear to accumulate in fish or in other animals used as food. Levels of nickel in sheep liver tissue, that appear to be adequate, range from 0.05-0.07 mg Ni/kg dry weight (Puls 1994). White-tailed deer from Illinois had mean concentrations of 3.6 mg Ni/kg wet weight (Woolf et al. 1982). Health based screening values for nickel range from 20 mg Ni/kg dry weight in children and 40 mg Ni/kg dry weight in adults (ATSDR 2006). Nickel has been found in at least 882 of the 1,662 NPL sites (ATSDR 2005).

Nickel concentrations were detected at the detection limit of 0.5 mg Ni/kg dry weight in four muscle tissue samples; F11M, F12M, F16M, and M1M, and above the analytical detection limit in six of the twenty muscle tissue samples. Measured concentrations ranged from 0.6 mg Ni/kg dry weight in sample F3M to 2.1 mg Ni/kg dry weight in sample F4M (Appendix B, Table 1). In liver tissue samples, nickel concentrations exceeded the analytical detection limit in three of the samples (Appendix B, Table 2). Levels of nickel ranged from 0.7 mg Ni/kg dry weight in M1L to 3 mg Ni/kg dry weight in F18L, all below the value reported by Woolf et al (1982). All detections were well below the health based screening values reported by ATSDR (2006).

[Selenium (Se)] Selenium is an essential micronutrient. As with many other dietary minerals selenium can be detrimental to organisms at elevated levels. In nature and biotic systems selenium exists as elemental selenium, selenite, selenate, and selenide (Goyer 1991, Eisler 1985). Health based screening values for selenium ranged from 5 mg Se/kg dry weight in children and 10 mg Se/kg dry weight in adults (ATSDR 2006). Selenium levels of 0.09-0.4 mg Se/kg wet weight in muscle tissue are adequate for sheep with toxic levels appearing at 0.4-20 mg Se/kg wet weight. Liver tissue levels of selenium in sheep are adequate at 0.25-1.5 mg Se/kg wet weight and become toxic at 15-30 mg Se/kg dry weight (Puls 1994). Health based screening values for selenium range from 5 mg Se/kg dry weight in children and 10 mg Se/kg dry weight in adults (ATSDR 2006).

Selenium was measured above the analytical detection limit in all 20 muscle tissue samples (Appendix B, Table 1). These concentrations ranged from 0.6 mg Se/kg dry weight in F18M to

1.4 mg Se/kg dry weight in F17M, well below the screening value reported by the ATSDR (2006). Selenium concentrations in liver tissue samples exceeded detection limits in all 18 samples (Appendix B, Table 2). These levels ranged from 0.89 mg Se/kg dry weight in F8L to 20.1 mg Se/kg dry weight in F17L. Only the liver tissue sample F17L exceeded the health based screening level.

[Strontium (Sr)] Strontium is not known to be as essential element for animals. Toxicity depends on the amount of calcium in the diet, if adequate, most species can tolerate 2,000 ppm dietary Strontium. Strontium in sheep muscle tissue at 0.17 mg Sr/kg dry weight is considered normal (Puls 1994). Health based screening values for strontium ranges from 2000 mg Sr/kg dry weight in children and 4000 mg Sr/kg dry weight in adults (ATSDR 2006).

Strontium levels were detected at or above the analytical detection limit in all 20 muscle tissue samples (Appendix B, Table 1) and ranged from 0.2 mg Sr/kg dry weight in F2M, F14M, F15M, F16M, F17M, and F18M to 0.68 mg Sr/kg dry weight in FF4M, well below the screening values reported by the ATSDR (2006). Strontium levels in liver tissue samples were at the detection limit of 0.2 mg Sr/kg dry weight for F1L. Further, ten liver samples contained strontium concentrations above the analytical detection limit. These levels ranged from 0.3 mg Sr/kg dry weight (F6L, F7L, F9L, F11L, F14L, F15L) to 0.620 mg Sr/kg dry weight for F8L (Appendix B, Table 2). All detections are well below the health based screening levels reported by ATSDR (2006).

[Zinc (Zn)] Tissue levels are not a good guide to zinc status since zinc is poorly stored in body tissues and must therefore be present in the diet at all times. Liver zinc levels can be elevated by copper excess. Adequate levels of zinc in sheep muscle tissue range from 75-130 mg Zn/kg dry weight while toxic levels range from 80-130 mg Zn/kg dry weight (Puls 1994). Reported values in livers of normal white-tailed deer in Illinois were reported at 70 mg Zn/kg dry weight (Woolf et al 1982). Munshower & Neuman (1979) found normal mean zinc levels in livers of antelope and mule deer to be 84.8 mg Zn/kg freeze dried weight and 113.3mgZn/kg freeze dried weight, respectively. White-tailed deer from Illinois had mean concentrations of 70 mg Zn/kg wet weight (Woolf et al. 1982). Health based screening values for zinc range from 300 mg Zn/kg dry weight in children and 600 mg Zn/kg dry weight in adults (ATSDR 2006).

Zinc was detected in all 20 muscle tissue samples above the analytical detection limit (Appendix B, Table 1). Levels of zinc ranged from 47 mg Zn/kg dry weight in F9M to 74 mg Zn/kg dry weight in F16M, all well below the screening values reported by the ATSDR (2006). All 18 liver tissue samples exceeded the analytical detection limit for zinc (Appendix B, Table 2). Concentrations ranged from 86.9 mg Zn/kg dry weight in F9L to 162 mg Zn/kg dry weight in F18L, well above the value reported by Woolf et al (1982). All detections are well below the health based screening levels reported by ATSDR (2006).

Organochlorine Pesticides and Total Polychlorinated Biphenyls in Muscle and Liver Tissue Samples

Results for the organochlorine pesticides analyses for the 20 muscle tissue samples and the 18 liver tissue samples are presented in Appendix B, Tables 3 and 4. Two pesticides (mirex and oxychlordane) were detected in one and three muscle tissue samples, respectively (Appendix B, Table 3). In one or more liver tissue samples, five pesticides (Total BHC, gamma-BHC, oxychlordane, Total DDTs, and o,p'-DDE) were detected above the analytical detection limit (Appendix B, Table 4). Total polychlorinated biphenyls were analyzed in all muscle tissue and liver tissue samples. There were no concentrations above the analytical detection limit or health based screening levels for any of the samples analyzed. This eliminated total-PCBs from further consideration in all muscle tissue and liver tissue samples.

[Mirex] Mirex is a manufactured insecticide that does not occur naturally in the environment. Mirex was most commonly used in the 1960s and 1970s, but has not been manufactured or used in the United States since 1978. Mirex was used to control fire ants, and as a flame retardant in plastics, rubber, paint, paper, and electrical goods from 1959 to 1972. Animal studies have shown that ingesting high levels of mirex can harm the stomach, intestine, liver, kidneys, eyes, thyroid, and nervous and reproductive systems (ATSDR 1996). Health based screening values for mirex range from 0.7 mg/kg wet weight in children and 2 mg/kg wet weight in adults (ATSDR 2006).

Mirex was detected above the analytical detection limit in one of the twenty muscle tissue sample, F4M (Appendix B, Table 3). This sample had a mirex concentration of 0.212 ug/mg wet weight well below the screening value reported by the ATSDR (2006). All other muscle tissue samples were below the analytical detection limit. There were no detections for mirex above the analytical detection limit for the 18 liver tissue samples (Appendix B, Table 4). All detections are well below the health based screening levels reported by ATSDR (2006).

[Oxychlordane] Oxychlordane is the major metabolite of the chlordanes and nonachlors. Chlordane was a widely used pesticide until the 1980's and is a toxic and persistent mixture that has accumulated in the food chain. The most abundant constituents of the chlordane mixture are trans-chlordane, cis-chlordane, trans-nonachlor, cis-nonachlor and heptachlor (ATSDR 1994). Because of concern about damage to the environment and harm to human health, the EPA banned all uses of chlordane in 1983 except to control termites. In 1988, EPA banned all uses (RAIS 2002). Chlordane and *trans*-nonachlor are metabolized by the body into oxychlordane and is stored in adipose tissue for long periods of time (ATSDR 1994). Health based screening values for oxychlordane range from 0.6 mg/kg wet weight in children and 1 mg/kg wet weight in adults (ATSDR 2006).

Three of the twenty muscle tissue samples had oxychlordane levels above the analytical detection limit (Appendix B, Table 3). Samples F3M, F5M and F8M had detections of 0.188 ug/kg wet weight, 0.16 ug/kg wet weight, and 4.64 ug/kg wet weight, respectively. Oxychlordane was detected above the analytical detection limit in all 18 liver tissue samples (Appendix B, Table 4). Concentrations ranged from 0.964 ug/kg wet weight in F10L to 22.3

ug/kg wet weight in F13L. All detections are well below the health based screening levels reported by ATSDR (2006).

[gamma-BHC] Hexachlorocyclohexane (HCH), formally known as benzene hexachloride (BHC), is a synthetic chemical that exists in eight chemical forms called isomers. The different isomers are named according to the position of the hydrogen atoms in the structure of the chemical. One of these forms, gamma-HCH (γ-HCH or γ-BHC, commonly called lindane), is produced and used as an insecticide for fruit, vegetables, and forest crops, and animals and animal premises. It is the only isomer in the group of hexachlorocyclohexane with pesticidal properties (ATSDR 2005). Health based screening values for gamma-BHC range from 0.0009 mg/kg wet weight in children and 0.02 mg/kg wet weight in adults (ATSDR 2006).

No muscle tissue samples contained gamma-BHC concentrations above the analytical detection limit (Appendix B, Table 3). In liver tissue samples, nine samples had gamma-BHC concentrations above the analytical detection limit (Appendix B, Table 4). These concentrations ranged from 1.23 ug/kg wet weight in F13L to 3.72 ug/kg wet weight in F14L. All concentrations were below the health based screening levels reported by ATSDR (2006).

[Total-BHC] Hexachlorocyclohexane (HCH), formally known as benzene hexachloride (BHC), is a synthetic chemical that exists in eight chemical forms called isomers. The different isomers are named according to the position of the hydrogen atoms in the structure of the chemical. Total BHC or total hexachlorocyclohexane (HCH) contains several other isomers, including the environmentally significant alpha-, beta-, and delta-HCH isomers. These HCH isomers are persistent, bioaccumulative, toxic, and mobile in the environment (ATSDR 2005). Due to their persistence and ability to migrate long distances through air and water, the HCH isomers travel from sites where HCH is manufactured and lindane is used. A variety of toxicological effects, such as reproductive and neurotoxic impairments, have been recorded for lindane and other isomers of HCH in test animals (EPA 2006). Health based screening values for total BHC ranged from 0.009 mg/kg wet weight in children to 0.02 mg/kg wet weight in adults.

No muscle tissue samples contained total-BHC concentrations above the analytical detection limit (Appendix B, Table 3). In liver tissue samples, total BHC was detected above the analytical detection limit in eight samples, F9L, F11L, F12L, F14L, F15L, F16L, F18L, and M1L (Appendix B, Table 4). The detected total BHC concentrations ranged from 1.98 ug/kg wet weight in M1L to 3.72 ug/kg wet weight in F14L. Detected total BHC concentrations in the liver tissue samples were well below the health based screening values (ATSDR 2006).

[o,p'-DDE] DDE is only found in the environment as a result of contamination or breakdown of DDT. It does not occur naturally in the environment and can build up in plants and in fatty tissues of fish, birds, and other animals (ATSDR 2002). Health based screening values for o,p'-DDE ranged from 0.5 mg/kg wet weight in children to 1 mg/kg wet weight in adults (ATSDR 2006).

There were no concentrations of o,p'-DDE above the analytical detection limit for the 20 muscle tissue samples (Appendix B, Table 3). Seventeen of 18 liver tissue samples had o,p'-DDE

concentrations above the analytical detection limit (Appendix B, Table 4). Concentrations ranged from 1.76 ug/kg wet weight for F10L to 1.77ug/kg wet weight for F2L. These concentrations were well below the health based screening levels reported by the ATSDR (2006).

[Total-DDTs] DDT does not occur naturally in the environment. DDT (dichloro-diphenyl-trichloroethane) is a pesticide that was once widely used to control insects on agricultural crops and insects that carry diseases like malaria and typhus, but is now used in only a few countries to control malaria. Technical-grade DDT is a mixture of three forms, *p,p'*-DDT (85%), *o,p'*-DDT (15%), and *o,o'*-DDT (trace amounts) (ATSDR 2002). Health based screening values for total-DDT range from 0.5 mg/kg wet weight in children to 1 mg/kg wet weight in adults (ATSDR 2006).

Total DDT concentrations were detected at or above the analytical detection limit in two of the 18 liver tissue samples, F2L at a level of 1.77 ug/kg wet weight and F10L at a the detection level of 1.76 ug/kg wet weight (Appendix B, Table 4). Concentrations for both F2L and F10L were well below the health based screening values reported by ATSDR (2006).

Chronic Wasting Disease

Chronic Wasting Disease (CWD) is a transmissible neurological disease of deer and elk that produces small lesions in brains of infected animals. It is characterized by loss of body condition, behavioral abnormalities and death (CWDA, 2002). It is classified as a transmissible spongiform encephalopathy (TSE), and is similar to mad cow disease in cattle and scrapie in sheep. Not much is known about CWD, including its origin, exact mode of transmission, and the causative or etiological agent. Infectious agents of CWD are neither bacteria nor viruses, but are hypothesized to be prions. Prions are infectious proteins without associated nucleic acids according to the CWDA (2002). It may "represent a spontaneous, naturally occurring" form of this disease in cervids thought to be caused by a "low virus infection." A more plausible theory is that CWD is caused by a point mutation of a membrane-bound protein resulting in accumulations of proteinase-resistant proteins called "prions" in the brain (medulla oblongata), tonsils (in deer only), and lymphoid tissue (TPWD, 2005).

Texas Parks and Wildlife Department (TPWD) personnel collected samples from five of the harvested deer and analyzed the brain stems for the presence of protease-resistant prion protein (PrP-res), the presence of which is diagnostic for chronic wasting disease (CWD). A report under separate cover will include results from those deer. Appendix D contains a copy of the analytical results for the five deer that were sampled during this investigation. Four of the five samples (CWD046CLNR1, CWD046CLNR2, CWD046CLNR4, and CWD046CLNR5) indicated that the protease-resistant prion protein (PrP-res) was not detected and the fifth sample (CWD046CLNR3) was determined to be unsuitable for testing (Appendix D).

CONCLUSIONS & RECOMMENDATIONS

Metals, organochlorine pesticides and total PCBs were analyzed from muscle tissue samples from 20 white-tailed deer and liver tissue samples from 18 white-tailed deer collected from the CLNWR. There were no metals analyzed that were a contaminant of concern in the muscle tissue samples. Of the metals analyzed in the liver tissue samples cadmium, copper, and selenium were the only contaminants of concern with concentrations above health based screening levels (ATSDR 2006). Cadmium concentrations in all 18 liver tissue samples exceeded the health based screening values for children of 0.2 mg Cd/kg dry weight. Adult health based screening levels of 0.4 mg Cd/kg dry weight were exceeded in 17 of the 18 liver tissue samples. Copper concentrations for all 18 liver samples exceeded the child health based screening value of 9 mg Cu/kg dry weight and the adult health based screening value of 22 mg Cu/kg dry weight. Selenium concentrations in one liver tissue sample, F17L, exceeded the child health based screening value of 5 mg Se/kg dry weight and the adult health based screening value of 10 mg Se/kg dry weight (ATSDR 2006).

Results for the organochlorine pesticides analyses for the 20 muscle tissue samples and the 18 liver tissue samples indicated that two organochlorine pesticides were detected in muscle tissue above the detection limits. Mirex was detected in one muscle tissue sample, F4M, above the analytical detection limit (Appendix A, Table 3), however this concentration was well below the health based screening value (ATSDR 2006). Three of the twenty muscle tissue samples had oxychlordane levels above the analytical detection limit, while oxychlordane was detected above the analytical detection limit in all eighteen liver tissue samples. All oxychlordane concentrations in the muscle tissue samples and liver tissue samples were well below the health based screening levels of 0.6 mg/kg wet weight in children and 1 mg/kg wet weight in adults. Nine liver tissue samples contained concentrations of gamma-BHC that exceeded the analytical detection limit and eight liver tissue samples had total-BHC concentrations that exceeded analytical detection limits. All detected gamma-BHC and total-BHC levels were well below the health based screening values of 0.5 mg/kg wet weight for children and 1 mg/kg wet weight for adults. Seventeen of eighteen liver tissue samples contained o,p'-DDE concentrations above the analytical detection limit and two of the eighteen liver tissue samples contained total-DDT levels that exceeded the analytical detection limit. Detected concentrations for both o,p'-DDE and total-DDTs were well below the health based screening level of 0.5 mg/kg wet weight for children and 1 mg/kg wet weight for adults. Total polychlorinated biphenyls were analyzed in all muscle tissue and liver tissue samples. There were no concentrations above the analytical detection limit for any of the samples analyzed.

This investigation involved the sampling of twenty white-tailed deer. Although there was a disproportionate number of females to males (9:1) harvested, the ratio corresponds to past hunting season records maintained at the facility. Review of the analytical results did not appear to indicate a difference in contaminant levels between male and female deer. Contaminant concentrations varied widely among both male and female deer. Six liver tissue samples, F3L, F9L and F13L, F16L, F17L, and M2L (Appendix B, Table 2) contained elevated levels of copper. Four of those liver tissue samples, F3L, F9L and F13L, F16L also contained elevated levels of cadmium and selenium. Two of those samples, F3L and M2L were collected at the

former acid storage area (Site 49), while F16L was collected at the former TNT production area (Site 29), and F13L was collected in the Plant 3 production area. These are sites associated with the former LHAAP and are currently being addressed under the CERCLA. Additional investigations in these areas may be necessary to determine if contaminant levels in these deer are associated with former production activities.

In conclusion, the overall results appear to indicate that the contaminant levels in white-tailed deer muscle tissue would not cause adverse health affects to individuals consuming deer muscle tissue and do not appear to pose a public health hazard. Selenium in one liver tissue sample exceeded the health based screening value for children of 5 mg Se/kg dry weight and the adult health based screening value of 10 mg Se/kg dry weight. All other selenium levels were well below these screening values. Both cadmium and copper concentrations exceeded their respective health based screening values in all liver tissue samples. Therefore, consumption of liver tissue from deer from CLNWR could possibly have adverse health affects and should not be consumed by individuals.

It is recommended that a hunting program at the Caddo Lake National Wildlife Refuge be established to help regulate the deer population and that such a program would not pose adverse health affects on those individuals consuming muscle tissue from harvested deer. It is also recommended that all hunters be advised of the results of the deer study that was conducted on the Refuge and advised not to consume livers from harvested deer. ATSDR concluded that if hunting is allowed in the future at CLNWR, and livers are not consumed, then no apparent public health risk would likely exist.

Further investigations should be undertaken, both on refuge and off, to obtain additional baseline data of biota. Off refuge investigations need to be conducted to determine background concentrations of metals, organochlorine pesticides, and total-PCBs in white-tailed deer from other areas around Caddo Lake. This would allow the data already obtained from this investigation to be compared with data from off-site areas. Investigations of other biota such as squirrels and feral hogs will help obtain baseline data that will be needed to determine if other types of hunting will be considered on the Refuge. These investigations will help determine if other game animals could be affected by ingestion of contaminated media and as such pose a human health risk to consumers.

All carcasses collected on the Refuge during this investigation were processed and frozen. Based on the review by the TDSHS, the processed deer meat was turned over to the Caddo Lake Institute for distribution to local churches and other charity organizations.

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APPENDIX A
(ANALYTICAL METHODS)

EXTRACTION OF BIOLOGICAL TISSUES FOR AROMATIC AND CHLORINATED HYDROCARBONS AND POLYBROMINATED FLAME RETARDANTS

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ABSTRACT

Determining organic contaminant levels in tissues require extraction, isolation, and concentration of analytes from the matrix. Tissue extracts require extensive purification procedures to remove lipids causing analytical interferences. Bivalves are shucked and homogenized. Aliquots of homogenized sample are chemically dried using Hydromatrix® and extracted in dichloromethane using a Dionex Acclerated Solvent Extractor. The extracts are purified using alumina/silica gel chromatography columns. The volume of the resultant eluent is further purified using a gel permeation column coupled to a high performance liquid chromatograph. The volume of the resultant eluant is reduced and analyzed for aromatic and chlorinated hydrocarbons and polybrominated flame retardants by gas chromatography/mass spectrometry and gas chromatography/electron capture detection.

1.0 INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), pesticides, polybrominated biphenyls (PBBs) and polybrominated diphenyl ethers (PBDEs) are contaminants of concern in the estuarine environment. The determination of these compounds at low concentrations in tissues is necessary to accurately monitor spatial and temporal changes in U.S. coastal waters. The procedure described is used to extract, isolate, purify, and concentrate aromatic and chlorinated hydrocarbon and polybrominated flame retardant contaminants from tissues. Contaminant concentrations in the parts per billion or parts per trillion can be resolved in lipid rich tissues. Shell length and shell volume are determined for specimens collected at each location. Bivalves are then shucked and multiple organisms are processed as one sample to ensure the sample is representative of a population at a given site and to have sufficient sample to complete the analyses. Tissue samples are homogenized using a stainless steel blender outfitted with titanium blades. Aliquots of approximately 15 g of wet tissue are chemically dried with Hydromatix®. The tissue/Hydromatix® mixtures are extracted with 100% dichloromethane using a Dionex Accelerated Solvent Extractor (ASE200) operated at 100°C and 2,000 psi. The extracts are reduced to 3 mL by evaporative solvent reduction. A 100 µL aliquot is removed and weighed to determine lipid weight (see method entitled “Determination of

Percent Lipid in Tissue"). The remaining sample portion is purified using alumina/silica gel column chromatography and gel permeation column (GPC)/high performance liquid chromatography (HPLC). After HPLC purification, the eluents are reduced to 0.5 mL and analyzed for PAHs, PCBs, pesticides, PCBBs and PBDEs by either gas chromatography/mass spectrometry (GC/MS) or gas chromatography/electron capture (GC/ECD).

2.0 APPARATUS AND MATERIALS

2.1 EQUIPMENT

- Dionex, ASE200 Accelerated Solvent Extractor (ASE) with 33 mL extraction cells
- Water bath, capable of maintaining a temperature of 55-60°C
- Balance, top loading, tare capacity to 300 g, capable of weighing to 1 mg
- Microbalance, capable of weight to 1 μg
- Calibrated weights, certified
- Combustion furnace, electric capable of combusting glassware at 400°C for at least 4 hours
- Oven capable of 40°C temperature maintenance
- Conditioning oven, electric, gravity convection, capable of maintaining a stable temperature of up to 200°C
- Tumbler, Lortone rock tumbler or equivalent
- HPLC system, Water Model 590 programmable solvent delivery module HPLC pump, Waters 717 plus autosampler, Waters UV absorbance detector, Waters 746 data module, Waters Fraction Collector, Phenogel 10μ GPC 100Å size exclusion columns and Phenogel 100Å guard column.
- Glass fiber filter circles, 2.4 cm diameter
- Collection vials, 60 mL certified pre-cleaned with open screw caps and Teflon lined septa
- Micropipettors, calibrated, 1% accuracy, disposable tips
- Zymark®, 50 mL concentration tubes
- 250 mL flat bottom, boiling flasks
- Borosilicate glass chromatography columns, 300 mm x 19 mm, with Teflon stopcock
- Kurderna-Danish (K-D) tubes, 25 mL, slow dry concentrator tubes
- Synder columns, 3-ball
- Boiling chips, Teflon
- Glass wool

18.R. . REAGENTS

- Water (CAS 7732-18-5), gas chromatography/HPLC grade or equivalent purity
- Acetone (CAS 67-64-1), pesticide grade or equivalent purity
- Dichloromethane (CAS 75-09-2), pesticide grade or equivalent
- Hexane (CAS 110-54-3), pesticide grade or equivalent
- Pentane (CAS 109-66-0), pesticide grade or equivalent
- Hydromatrix® (CAS 68855-54-9/14464-46-1), conditioned by combustion at 400°C for at least 4 hours and stored at 120°C

- Sodium sulfate (CAS 7757-82-6), anhydrous granular powder, A.C.S. reagent grade, purified by combusting at 400°C for at least 4 hours and stored at 120°C.
- Alumina (CAS 1344-98-2), 80-325 mesh, basic, purified by combusting at 400°C for at least 4 hours and stored at 120°C
- Silica gel (CAS 1343-98-2), grade 923, 100-200 mesh, purified in an oven at 170°C for at least 16 hours and store at 170°C
- Nitrogen (CAS 7727-37-9), 99.8% purity

18.R. . **PROCEDURE**

Shell length and volume are determined for all bivalves collected at each sampling site. The bivalves are then shucked and the soft tissue homogenized using a stainless steel Waring® blender. Homogenized tissue samples are frozen at –20ºC until extraction. Prior to extraction, tissue samples are thawed and re-homogenized using a stainless steel spatula. A subsample is removed for percent moisture determination (see Dry Weight Determination of Tissues).

Approximately 15 g of tissue are thoroughly mixed and ground with a sufficient quantity (approximately 40 g) of prepared (combusted) Hydromatrix® to "dry" the sample. The tissue samples must be thoroughly dry to optimize the extraction efficiency. Hydromatrix® chemically dries samples by binding moisture. The amount of Hydromatrix® necessary to dry a sample depends upon the amount of sample and the percent moisture in that sample.

Tissues are extracted with dichloromethane using an ASE200. The tissue/Hydromatix® mixture is loaded into 33 mL ASE extraction cells. Appropriate surrogate and spikes are added to the top of the samples. The ASE extractor tubes are sealed and place in the ASE cell carousel. The ASE conditions are: 100% dichloromethane as the extraction solvent; 2,000-psi solvent pressure; 100°C cell temperature; and 2 static cycles for 2 minutes each. Extracts are collected in 60 mL collection vials. The extracts are reduced to approximately 10 mL in the 60 mL collection vials in a 55-60°C water bath. Extracts are then quantitatively transferred to Kurderna-Danish (K-D) tubes and the volume reduced to 3 mL in a 55-60°C water bath. A 100 µL aliquot is removed and weighed to determine lipid content (see method entitled "Determination of Percent Lipid in Tissue"). Quality control samples (e.g., blanks, duplicates, matrix spikes and standard reference materials) are prepared and extracted in the same manner as samples.

Extracts are initially purified using alumina/silica gel chromatography columns. Combusted and cooled alumina is deactivated by adding 1% (w/w) HPLC water and tumbled for at least 1 hour using a Lortone rock tumbler. Combusted and cooled silica gel is deactivated by adding 5% (w/w) HPLC water and tumbling for at least 1 hour using a Lortone rock tumbler. Borosilicate glass columns (300 mm x 19 mm) are filled with dichloromethane and packed from the bottom with: glass wool; 1-2 g of sodium sulfate; 10 g of deactivated alumina; 20 g of deactivated silica gel; and another 1-2 g of sodium sulfate. The dichloromethane is drained to the top of the column followed by the addition of 50 mL of pentane. The pentane is drained to the top of the upper sodium sulfate layer and discarded. The sample extract (approximately 3 mL) is added to the top of the column and eluted with 200 mL of a 50:50 mixture of pentane and dichloromethane at a flow rate of 1 mL/min. The eluent is collected in a 250 mL flat-bottom

flask. The eluent is reduced to approximately 10 mL in a 55-60°C water bath. The extract is transferred to 25 mL K-D tubes and reduced to1-2 mL. The concentrate is transferred to 4 mL amber HPLC vials and brought up to 4 mL with dichloromethane.

The extract is further purified using HPLC. The extract is injected using a Waters, Model 717 Plus autosampler and eluted through one Phenogel 100Å guard column and two Phenogel 10µ GPC 100Å size exclusion columns with 100% dichloromethane at a flow rate of 7 mL per minute. Elution times for compounds of interest are monitored using standards and an UV absorbance detector (254 nm). The appropriate fraction is collected using a Waters Fraction Collector. The sample is collected in 50 mL Zymark tubes and reduced to 10 mL in a 50-60ºC water bath. The extract is transferred to K-D tubes and reduced to 1.0 mL. The dichloromethane is exchanged with hexane and reduced to a final volume of 0.5 mL. The concentrate is transferred to 2 mL amber vials and stored at -20°C until analysis. Figure 1 shows a flow chart of the extraction and purification procedure.

18.R. . QUALITY CONTROL (QC)

Solvents are verified to be contaminant-free by lot tests prior to use. All equipment and glassware used to extract samples are thoroughly cleaned by solvent rinsing or combustion at 400°C. The calibration and accuracy of balances, weights, pipettors and thermometers are checked daily using certified weights and thermometers with calibrations traced to the National Institute of Standards and Technology (NIST). The calibration and accuracy of balances, weight, pipettors and thermometers are verified yearly by an independent source. A series of quality control samples are processed with each batch of 20 samples or less. The following quality controls are used to ensure the accuracy and precision of tissue data.

- Surrogates. Solutions containing analytes that do not interfere with the analytes of interest are prepared at concentrations approximately 5 to 10 times the method detection limit (MDL). Specified surrogates are added to each sample extracted, including QC samples, at a specified volume (typically 100 µL) immediately prior to extraction.
- Method Blank. Method blanks are extractions of all support material used for extraction of samples, with the exception of tissue. A method blank is analyzed with each extraction batch of 20 or fewer samples. The method blank is extracted and analyzed in a manner identical to samples.
- Matrix Spike. Matrix spikes are extractions of sample matrix fortified with spikes of selected target analytes. Spikes are prepared at concentrations approximately 10 times the MDL. A matrix spike and matrix spike duplicate are analyzed with each extraction batch of 20 or fewer samples. Matrix spikes are extracted and analyzed in a manner identical to samples.
- Laboratory Duplicates. A sample is analyzed in duplicate with each extraction batch of 20 or fewer samples.
- Standard Reference Material (SRM). A standard reference material from the NIST (SRM 1974a) is analyzed with each extraction batch of 20 or fewer samples for aromatic and chlorinated hydrocarbons.

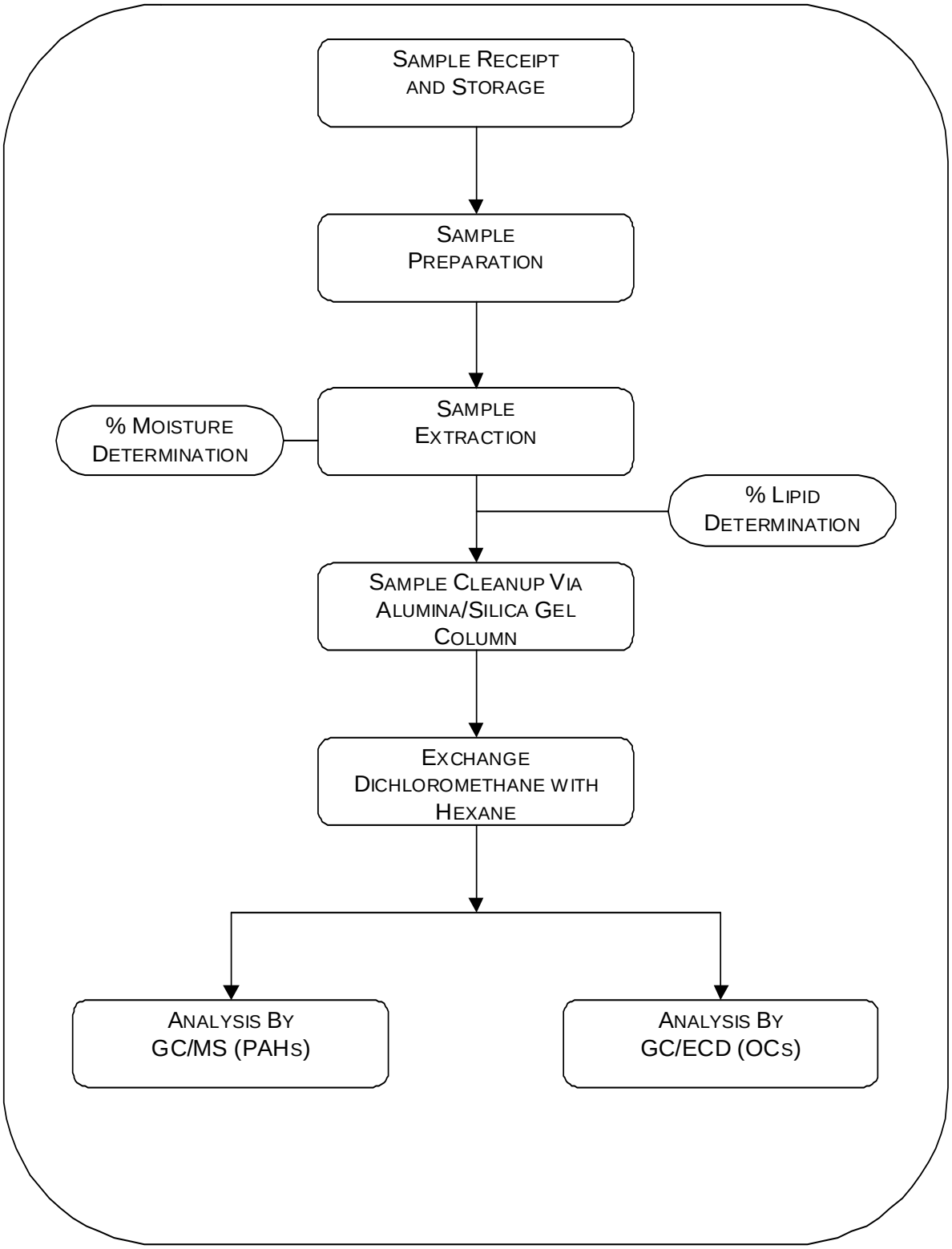

Figure 1. Methodology for Extraction, Isolation and Quantification of Tissue Samples for Aromatic and Chlorinated Hydrocarbons and Polybrominated Flame Retardants.

QUANTITATIVE DETERMINATION OF CHLORINATED HYDROCARBONS USING GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTION

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ABSTRACT

Selected chlorinated hydrocarbons, including polychlorinated biphenyls and pesticides, are detected using gas chromatograph/electron capture detector. This method is capable of detecting low concentration of chlorinated hydrocarbons in complex matrices such as tissues and sediments.

1.0 INTRODUCTION

A gas chromatograph/electron capture detector (GC/ECD), coupled to two capillary columns, is used to resolve and detect chlorinated hydrocarbons (polychlorinated biphenyls and pesticides) in tissues and sediments. Samples are injected into a temperature-programmed GC/ECD, operated in splitless mode. The capillary columns are DB-5 (30 m x 0.25 mm ID and 25 μm film thickness) and DB-17HT (30 m x 0.25 mm ID and 0.15 μm film thickness). The DB-17HT column is used for analyte confirmation. A data acquisition system continuously acquires and stores all data for quantitation. This method is capable of producing data at parts-per billion and parts-per trillion concentrations.

2.0 APPARATUS AND MATERIALS

2.1 EQUIPMENT

- Gas chromatograph, split/splitless injection port and electronic pressure control, dual electron capture detectors, Agilent Techologies 5890-II
- Data acquisition system, Agilent Technologies ChemStation, capable of continuous acquisition and storage of all data during analysis
- Autosampler, capable of making 1 to 5 μL injections
- Capillary columns, J&W DB-5®(30 m x 0.25 mm ID and 0.25 μm film thickness) or equivalent, and J&W DB-17HT®(30 m X 0.25 mm ID and 0.15 μm film thickness)
- Micropipetters, calibrated, 1% accuracy, disposable tips

18.R. . REAGENTS

- Hexane (CAS 110-54-3), pesticide grade or equivalent
- Helium (CAS 7440-59-7), 99.8% purity
- 95% Argon/5% Methane, 99.8% purity

2.3 STANDARDS

2.3.1 Surrogate Spiking Solution

A surrogate spiking solution is prepared from a commercially available solution (Ultra Scientific) that is diluted with hexane to a concentration of 1,000 pg/μL. The surrogate spiking solution includes 4,4'-dibromooctaflurobiphenyl (DBOFB), 2,2',4,5',6 pentachlorobiphenyl (PCB 103), and 2,2',3,3',4,5,5'6 octachlorobiphenyl (PCB 198). Surrogate solution (100 μL) is added to all samples and quality control samples prior to extraction. Surrogate compounds are resolved from, but elute in close proximity to, the analytes of interest. The recovery of PCB 103 is used to correct analyte concentrations.

2.3.2 Internal Standard Solution

The internal standard solution is prepared from a commercially available solution (Ultra Scientific) of tetrachloro-m-xylene (TCMX) diluted with hexane to a final concentration of 1,000 pg/μL. The internal standard compound is resolved from, but elutes in close proximity to, the analytes of interest. The internal standard solution (100 μL) is added to all samples and quality control samples just prior to analysis. Internal standards are used to calculate relative response factors and specific analyte concentrations based on retention time.

2.3.3 Matrix Spiking Solution

To prepare the matrix spiking solution, a certified solution (Accustandard) containing analytes of interest is purchased from commercial vendors and diluted with hexane (Table 1). The matrix spike solution is diluted to a concentration approximately 10 times the MDL and is added to all matrix spike samples.

18.R.M. ***Calibration Solution***

Calibrations solutions are prepared at 5 concentrations ranging from approximately 5 to 200 pg/μL (Table 2) by diluting a commercially prepared solutions (Ultra Scientific and Accustandard) containing the analytes of interest.

Table 1. Chlorinated Hydrocarbons Contained in Matrix Spike Solution .

Pesticide	CAS	Spiking Solution Concentration (pg/µL)
1,2,4,5-Tetrachlorobenzene	95-94-3	40
1,2,3,4-Tetrachlorobenzene	634-66-22	40
Pentachlorobenzene	608-93-5	40
Pentachloroanisole	1825-21-4	40
Chlorpyrifos	2921-88-2	40
Hexachlorobenzene	118-74-1	40
α-HCH	319-84-6	40
β-HCH	319-85-7	40
γ–HCH (Lindane)	55-89-9	40
δ-HCH	319-86-8	40
Heptachlor	76-44-8	40
Heptachlor epoxide	1024-57-3	40
α-Chlordane (cis-)	5103-71-9	40
γ -Chlordane (trans-)	5103-74-2	40
Trans-Nonachlor	39765-80-5	40
Cis-Nonachlor	5103-73-1	40
Aldrin	309-00-2	40
Dieldrin	60-57-1	40
Endrin	72-20-8	40
Mirex	2385-85-5	40
2,4’ DDE	3424-82-6	40
4,4’ DDE	72-55-9	40
2,4’ DDD	53-19-0	40
4,4’ DDD	72-54-8	40
2,4’ DDT	789-02-6	40
4,4’ DDT	50-29-3	40
Endosulfan II	33213-65-9	40
Oxychlordane	27304-13-8	40
Endosulfan Sulfate	1031-07-8	40
PCB 8	34883-43-7	40
PCB 18	37680-65-2	40
PCB 28	7012-37-5	40
PCB 44	41464-39-5	40
PCB 52	35693-99-3	40
PCB 66	32598-10-0	40
PCB 101	37680-73-2	40
PCB 105	32598-14-4	40
PCB 118	31508-00-6	40
PCB 128	38380-07-3	40
PCB 138	35065-28-2	40
PCB 153	35065-27-1	40
PCB 170	35065-30-6	40
PCB 180	35065-29-3	40
PCB 187	52663-68-0	40
PCB 195	52663-78-2	40
PCB 206	40186-72-9	40
PCB 209	2051-24-3	40

Table 2. Chlorinated Hydrocarbons Contained in Calibration Solutions and their Approximate Concentrations.

Compounds Contained in Calibration Solutions	CAS	Level 1 (pg/µl)	Level 2 (pg/µl)	Level 3 (pg/µl)	Level 4 (pg/µl)	Level 5 (pg/µl)
Internal Standard						
TCMX	877-9-8	100	100	100	100	100
Surrogates						
DBOFB	10386-84-2	5	20	40	80	200
PCB 103	60145-21-3	5	20	40	80	200
PCB 198	68194-17-2	5	20	40	80	200
Analytes						
1,2,4,5-Tetrachlorobenzene	95-94-3	5	20	40	80	200
1,2,3,4-Tetrachlorobenzene	634-66-22	5	20	40	80	200
Pentachlorobenzene	608-93-5	5	20	40	80	200
Pentachloroanisole	1825-21-4	5	20	40	80	200
Chlorpyrifos	2921-88-2	5	20	40	80	200
Hexachlorobenzene	118-74-1	5	20	40	80	200
α–HCH	319-84-6	5	20	40	80	200
β–HCH	319-85-7	5	20	40	80	200
γ–HCH	319-86-6	5	20	40	80	200
δ–HCH	58-89-9	5	20	40	80	200
Heptachlor	76-44-8	5	20	40	80	200
Heptachlor epoxide	1024-57-3	5	20	40	80	200
Oxychlordane	27304-13-8	5	20	40	80	200
α-Chlordane (cis-)	5103-71-9	5	20	40	80	200
γ-Chlordane (trans-)	5103-74-2	5	20	40	80	200
Trans-Nonachlor	39765-80-5	5	20	40	80	200
Cis-Nonachlor	5103-73-1	5	20	40	80	200
Aldrin	309-00-2	5	20	40	80	200
Dieldrin	60-57-1	5	20	40	80	200
Endrin	72-20-8	5	20	40	80	200
Mirex	2385-85-5	5	20	40	80	200
2,4'-DDE	3424-82-6	5	20	40	80	200
4,4'-DDE	75-55-9	5	20	40	80	200
2,4'-DDD	53-19-0	5	20	40	80	200
4,4'-DDD	72-54-8	5	20	40	80	200
2,4'-DDT	789-02-6	5	20	40	80	200
4,4'-DDT	50-29-3	5	20	40	80	200
Endosulfan II	33213-65-9	5	20	40	80	200
Endosulfan Sulfate	1031-07-8	5	20	40	80	200
PCB 8	34883-43-7	5	20	40	80	200
PCB 18	37680-65-2	5	20	40	80	200
PCB 28	7012-37-5	5	20	40	80	200
PCB 44	41464-39-5	5	20	40	80	200
PCB 52	35693-99-3	5	20	40	80	200
PCB 66	32598-10-0	5	20	40	80	200
PCB 101	37680-73-2	5	20	40	80	200
PCB 105	32598-14-4	5	20	40	80	200
PCB 118	31508-00-6	5	20	40	80	200
PCB 128	38380-07-3	5	20	40	80	200
PCB 138	35065-28-2	5	20	40	80	200

Compounds Contained in Calibration Solutions	CAS	Level 1 (pg/µl)	Level 2 (pg/µl)	Level 3 (pg/µl)	Level 4 (pg/µl)	Level 5 (pg/µl)
PCB 153	35065-27-1	5	20	40	80	200
PCB 170	35065-30-6	5	20	40	80	200
PCB 180	35065-29-3	5	20	40	80	200
PCB 187	52663-68-0	5	20	40	80	200
PCB 195	52663-78-2	5	20	40	80	200
PCB 206	40186-72-9	5	20	40	80	200
PCB 209	2051-24-3	5	20	40	80	200

3.0 QUANTITATIVE DETERMINATION OF CHLORINATED HYDROCARBONS BY GC/ECD

3.1 CALIBRATION

An ECD exhibits limited linearity, particularly for low concentrations. Consequently, a calibration must be established for each analytical run. An analytical run consists of samples and 5 calibration standards (approximately 5 to 200 pg/µL or 5 to 200 ng/mL) that are interspersed throughout the run. A calibration curve is established by analyzing the 5 interspersed calibration standards and fitting the data to the following quadratic equation.

$$x = \frac{-b_1 + \sqrt{b_1^2 - 4b_2(b_0 - Y)}}{2b_2}$$

Where:

x = the concentration of the analyte (ng/ml)
Y = the ratio of the area of the analyte to the area of the internal standard multiplied by the amount of the internal standard (ng)
b2, b1, b0 = the coefficients for the quadratic equation

The data generated for each analyte in the calibration standards are subjected to the method of least squares to determine the coefficients for the corresponding quadratic equation. Each analyte has different coefficients based on the relative response of the analyte compared to the internal standard, and as a function of the amount of the analyte. The injected concentration of the internal standard analyte is held constant for each set of calibration standards. In order for the calibration to be valid, each analyte must have a correlation coefficient greater than 0.997.

18.R. . GC/ECD ANALYSIS

Sample analyses are completed only if the calibration meets previously described criteria. Samples are analyzed in analytical sets that consist of standards, samples and quality control samples. Quality control (QC) samples are method blanks, laboratory duplicates, matrix spikes, and standard reference material (SRM). An autosampler is used to inject 1 or 5 µL of all samples, standards and QC samples into the capillary column of the GC using the following instrument conditions. Slight modifications may be necessary depending upon the analysis.

Inlet: Splitless
Carrier gas: Helium, 1 mL/min

Temperatures:
- Injection port: 275°C
- Detector: 325°C

Oven program:
- Initial oven temp: 100°C
- Initial hold time: 1 minute
- Ramp rate: 5°C/min to 140°C
- Hold time: 1 minute
- Ramp rate: 1.5°C/min to 250°C
- Hold time: 1 minute
- Final oven rate: 10°C/min to 300°C
- Final hold time: 5 minutes

18.R. . ANALYTE IDENTIFICATION

The retention time of a sample analyte must fall within 15 seconds of the retention time for that analyte in a calibration standard or a retention index solution.

Chromatographic interferences may limit the ability to quantify peaks correctly and these data are reported but qualified to indicate interference.

18.R. . QUANTITATION CALCULATIONS

Sample analyte concentrations are calculated based on the concentration and response of the internal standard (Table 2). The concentration I of each target analyte in the sample (ng/g) is calculated using the following equation:

$$C = \left(\frac{X}{W}\right)(V_e DF)$$

Where:

Ve = the final volume of the extract (mL)
X = the concentration of the analyte (ng/mL) as found from solving the quadratic equation
W = the sample weight (g)
DF = the dilution factor

$$DF = \frac{\text{Volume of Extract (uL)}}{\text{Volume of extract used to make dilution (uL)}}$$

Analyte concentrations are reported as corrected for surrogate recoveries. Percent surrogate recoveries (Surecovery) for each surrogate are calculated using the following equation:

$$SU_{Recovery} = \frac{C_{ESU}}{C_{SU}} x100$$

Where:

CESU = calculated surrogate concentration in the extract
CSU = known concentration of surrogate added to extract

Anayte concentration corrections (Ccorrected) for surrogate recovery are calculated using the following equation:

$$C_{Corrected} \frac{C}{SU_{Recovery}} x\,100$$

18.R. . QUALITY CONTROL (QC)

Samples are analyzed in analytical batches consisting of 19 samples or fewer and QC samples. The QC samples are a method blank, laboratory duplicate, matrix spike, matrix spike duplicate and SRM. A method blank is a reagent blank prepared in the laboratory. A duplicate is a sample for which a second aliquot is analyzed. Matrix spikes are samples that are spiked with known concentrations of known analytes. The SRM used depends upon availability, matrix and analytes. All SRMs are certified and traceable to National Institute of Standards and Testing (NIST).

The validity of the data is monitored using defined QC criteria. The following QC criteria are used to evaluate analytical batches.

1). Calibration

- The calibration criteria (Section 3.1) must be met prior to data analyses. If the calibration criteria are not met, then the run is aborted and the instrument re-calibrated before further sample analysis.

2). Method Blank

- No more than two target analytes many exceed 3 times the concentration of the MDL.
- Exceptions are that if an analyte detected in the method blank exceeds 3 times the concentration of the MDL, but is not present in the associated samples or if a sample analyte concentration is greater than 10 times that analyte concentration in the method blank, the result is qualified and reported.
- If a method blank exceeds these criteria then the source of contamination is determined and corrective action is taken before further sample analysis.

3). Matrix Spikes

- Analytes spiked into a matrix are considered valid only if they are spiked at concentrations equivalent to levels found in the sample.
- The average recovery for all valid spiked analytes in a matrix spike is between 60% and 120%. No more than two individual spiked analyte (valid) recoveries may exceed 40%-120%, with the exception of chlorpyrifos and endosulfan sulfate.

- If the QC criteria are not met then the matrix spike sample failing the criteria will be re-analyzed and if the re-analyzed spike meets the criteria then the data are reported.
- If an analyte exceeds the criteria and is not present in the associated samples analyzed with the analytical batch, the result is qualified and reported.
- If upon re-analysis, QC criteria are still not met, the entire batch of samples is reanalyzed. If sufficient sample is unavailable to re-extract the matrix-spike, another sample may be selected or a blank-spike may be substituted.
- The average RPD for a valid matrix spike/matrix spike duplicate or blank spike/blank spike duplicate pair is 30%. No more than two individual analyte RPDs may exceed 35%.

4). Duplicate

- The average relative percent difference (RPD) between the duplicate and original sample, for analytes greater than 10 times the concentration of the MDL, is 30%. The RPD for no more than two individual analytes may exceed 35%.
- If the QC criteria are not met then the sample pair failing the criteria will be re-analyzed and if the re-analyzed samples meet the criteria then the data are reported.
- If an analyte exceeds the criteria and is not present in the associated samples analyzed with the analytical batch, the result is qualified and reported.
- If upon re-analysis, QC criteria are still not met, the entire batch of samples is reanalyzed. If sufficient sample is unavailable to re-extract the duplicate pair, another sample may be selected.

5). Standard Reference Material

- The average recovery for target analytes in a SRM should not exceed 30% of the upper and lower bounds of the mean certified values. No more than two target analytes should deviate more than 35% from the upper or lower bounds of the mean certified values.
- If the QC criteria are not met then the SRM failing the criteria will be re-analyzed and if the re-analyzed SRM meets the criteria then the data are reported.
- If an analyte exceeds the criteria and is not present in the associated samples analyzed with the analytical batch, the result is qualified and reported.
- If upon re-analysis, QC criteria are still not met, the entire batch of samples is reanalyzed.

6). Surrogates

- The average recovery of surrogate compounds is between 50% and 150%.
- Exceptions are analytical interferences with the surrogates and diluted samples.
- If the average recovery of surrogates exceeds the criteria, and calculation and analytical errors are eliminated, the sample is re-analyzed. If sufficient sample is unavailable for re-extraction, the data are qualified and reported.

7). Method Detection Limit

- The method detection limit (MDL) is determined following the procedures outlined in Federal Register (1984), Vol. 49, No. 209: 198-19.

Metals

ECDMS Analytical Results Report
1/25/2006

Catalog Number	Purchase Order Number	Lab ID	Catalog Submitter	ECDMS User ID
2100002	94420-05-Y519	TDI	Bruckwicki, Paul – Karnack , TX	r2clnwr

Catalog Title	Caddo Lake NWR Deer Study
Lab Name:	TDI – Brooks International, Inc.
DEQ Project ID:	200520003
DEQ Project Title:	An Investigation of Contaminant Levels in White-tailed Deer (Odocoileus virginianus) Collected from Caddo Lake National Wildlife Refuge, Harrison County, Texas

Notes, Symbols and Abbreviations Used
Based on the report options selected the report should be **printed in landscape mode**
The following may appear before a reported result (e.g. < 1234). < - Less than symbol indicates that the actual result is less than the reported detection limit. > - Greater than symbol indicates that the actual result is greater than the reported result.
All results are reported as 3 significant digits.
All results are reported as parts per million (ppm), or percent, unless otherwise noted.

Lab Integrity Report

Lab Receipt Date	04/13/2005	Lab Approval Date:	04/13/2005

Catalog Problems
No problems reported.
Problem Resolution

Bulk Data

Sample Number	Sample Matrix	Sample Weight (grams)	Percent Lipid	Percent Moisture
F10L	Liver	426.26	4.41	70.1
F11L	Liver	759.87	3.83	75.5
F12L	Liver	837.31	4.87	73.1
F13L	Liver	772.46	6.35	69.2
F14L	Liver	774.32	2.98	69.3
F15L	Liver	538.4	4.94	73.5
F16L	Liver	765.25	6.89	70.3
F17L	Liver	894.56	4.16	73.0
F18L	Liver	850.15	5.21	71.7

F1L	Liver	436.6	3.70	70.4
F2L	Liver	612.39	3.04	71.4
F3L	Liver	862.51	3.42	69.5
F6L	Liver	897.6	2.87	71.4
F7L	Liver	825.47	3.89	72.8
F8L	Liver	767.1	3.61	71.5
F9L	Liver	528.8	3.86	71.7
M1L	Liver	923.18	4.59	73.0
M2L	Liver	837.65	4.80	71.8
F10M	Muscle	340.51	3.69	73.8
F11M	Muscle	375.18	2.86	72.4
F12M	Muscle	317.14	3.37	71.8
F13M	Muscle	231.37	2.60	72.3
F14M	Muscle	343.18	3.57	70.3
F15M	Muscle	308.12	1.22	74.4
F16M	Muscle	351.21	2.37	72.3
F17M	Muscle	348.2	1.75	73.3
F18M	Muscle	455.09	3.62	69.5
F1M	Muscle	532.24	1.49	74.1
F2M	Muscle	519.22	2.55	70.2
F3M	Muscle	406.63	3.47	71.7
F4M	Muscle	209.18	4.25	71.3
F5M	Muscle	237.32	3.02	71.7
F6M	Muscle	337.2	3.42	71.3
F7M	Muscle	460.45	2.34	75.0
F8M	Muscle	264.6	2.76	72.3
F9M	Muscle	495.87	7.91	69.9
M1M	Muscle	246.29	1.78	73.8
M2M	Muscle	351.6	0.910	76.1

QA/QC Anomalies

Blank Frequency Anomalies

The required number of blank analyses were performed.

Duplicate Frequency Anomalies

The required number of duplicate analyses were performed.

Spike Frequency Anomalies

The required number of spike analyses were performed.

Reference Material Frequency Anomalies

The required number of Standard Reference Material analyses were performed.

Limit of Detection Anomalies

Limits of Detection were within the contract requirements.

Blank Anomalies

Procedural Blank analyses were acceptable.

Duplicate Anomalies

All duplicate results were within normal limits.

Spike Anomalies

All spike sample results were within normal limits.

S.R.M. Anomalies

All SRM results were within normal limits.

Analytical Methods

Below are the analytical methods used by LET to produce the results included in this report.

Method Codes:	001	002

Lab Matrix	**Analyte**
Animal Tissue	% Moisture

Method Code: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For samples weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or

if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with a blender, followed if necessary by grinding in a Spex mixer mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

Method Code: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 – Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 – 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance.

Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the – sign.

Notes:

1. If the samples do not require % Moisture, then all of the weighing steps can be eliminated.

Method Codes:	001	002	007	012

Lab Matrix	Analyte
Animal Tissue	Arsenic
	Selenium

Method Code: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For samples weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with a blender, followed if necessary by grinding in a Spex mixer

mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

Method Code: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 – Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 – 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance. Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the – sign.

Notes:

1. If the samples do not require % Moisture, then all of the weighing steps can be eliminated.

Method Code: 007

LABORATORY: Laboratory and Environmental Testing, Inc.

L5 – Magnesium Dry Ash

1. Weigh 0.5 g. of sample on a three-place balance and transfer to a cleaned 100 ml. glass beaker with etched numbers. Record the beaker number as well as the sample weight.

2. Wet with 3 ml. of methanol. Then add 5 drops of anti-foam agent, 10 ml. of 40% (W/V) Magnesium Nitrate Hexahydrate, 10 ml. of concentrated trace metal grade HNO3 and 2 ml. of concentrated trace metal grade HCl.

3. Cover with a watch glass and reflux on a hot plate overnight (8-12 hours) at low heat (70-80 C).

4. After reflux increase temperature to 200 C. Slide the watch glass to the side to allow for faster evaporation and cook to complete dryness. This may take 8-12 hours.

5. When no moisture is visible, cover fully with the watch glass and allow to cool.

6. Transfer samples to the cold muffle furnace and use the following program: Start at 250 C and ramp to 500 C at a rate of 1 degree per minute. When 500 C is reached hold for 3 hours then turn off and allow samples to cool to room temperature.

7. Place the cooled samples on a hot plate and add 20 ml. of 50% trace metal grade HCl. Allow the samples to gently boil for 1 hour. After 1 hour readjust volume to 20 ml. with 50 % HCl. Do not allow the samples to go dry. If necessary add more 50 % HCl during the heating.

8. Allow the samples to cool. Then dilute to 50.0 ml. with D.I. water and transfer to a clean 2 oz. labeled bottle.

Notes:

1. This digestion can be used for As or Se by Hydride Generation AA.

2. This digestion must be used on fish for As by Hydride Generation AA.

Method Code: 012

LABORATORY: Laboratory and Environmental Testing, Inc.

Hydride Generation AA

Turn on the computer, printer, 3100, FIAS 200.and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on either the Se-Fias or As-Fias method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the element and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 0.2% NaBH4-0.05% NaOH, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for this element. Change the position of the tubing or new tubes, if both positions have been used.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Borohydride. Run a 5 or 10 PPB standard until the sensitivity has stabilized and consecutive readings vary by less than 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 15.0 PPB. QC checks are 10.0 and a known Reference sample (Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn off the EDL lamp, the Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for As or Se FIAS. Then Browse and find the file name given the data. Place a 3.5” disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

Method Codes:	001	002	009	013

Lab Matrix	Analyte
Animal Tissue	Mercury

Method Code: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For samples weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with a blender, followed if necessary by grinding in a Spex mixer mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

Method Code: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 – Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 – 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance. Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the – sign.

Notes:

1. If the samples do not require % Moisture, then all of the weighing steps can be eliminated.

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the

sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using ½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 013

LABORATORY: Laboratory and Environmental Testing, Inc.

Cold Vapor AA

Turn on the computer, printer, 3100, FIAS 200,and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on the Hg-CV method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the Hg and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 5% Stanous Chloride-10% HCl, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for Mercury. Change the position of the tubing or new tubes, if both positions have been used or determining a different element.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Stanous Chloride. Run a 10 or 20 PPB standard until the sensitivity has stabilized and consecutive readings vary by less the 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 30.0 PPB. QC checks are 10.0, 20.0 and a known Reference Sample(Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn off the EDL lamp, the Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for Hg-CV. Then Browse and find the file name given the data. Place a 3.5” disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

Method Codes:	001	002	009	016

Lab Matrix	Analyte
Animal Tissue	Lead

Method Code: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For samples weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with

a blender, followed if necessary by grinding in a Spex mixer mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

Method Code: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 – Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 – 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance. Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the – sign.

Notes:

1. If the samples do not require % Moisture, then all of the

weighing steps can be eliminated.

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using ½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 016

LABORATORY: Laboratory and Environmental Testing, Inc.

Graphite Furnace using the 5100 Zeeman

Turn on the Furnace Coolant, Argon, Computer, Furnace, Zeeman Power Supply and 5100 in that order. Make sure the lamp is in the right position in the turret, and if using an EDL turn on the lamp to the proper power.

Double click on the AA WinLab Analyst icon. After the ZHGA-600 and 5100 icons have a green check by them, click on workspace. Double click on LET.fm, then double click on the method. Choose the method to be run and double click on the name. Click on Browse by the Sample Information File, and then pick one for standards. (Example: Pbstds.sif) Click on sample information icon to edit the file with sample names, dilutions, etc. Click Browse for the Results Data Set and enter the name of the file to store data. (Example: Pb010101) Click on Analyze. When samples have been prepared and ready for analysis, click on Analyze All if doing calibration or Analyze Samples if just running samples. The instrument is usually calibrated with a zero and one standard. Then a zero and 3-5 standards are run to check the calibration, followed by an instrument check standard and detection limit. If this is acceptable then the samples are run. Be sure to check that the correct modifier is being used for the element being run. Some samples may require the method of Standard Additions.

After the analysis is completed, close AA WinLab Analyst, turn off furnace coolant, Argon, 5100, Zeeman Power supply, Furnace and EDL power supply. Double click on the WinLab Reformat icon. Click on Open Design and choose the design for your element and double click on the name. Click on Browse and find the data file you want to reformat. Double click on the name. Make sure there is a floppy disk in the disk drive and click on Save Results. Transfer to another computer and calculate using Excel.

Method Codes:	001	002	009	018

Lab Matrix	Analyte
Animal Tissue	Aluminum
	Boron
	Barium
	Beryllium
	Cadmium
	Chromium
	Copper
	Iron
	Magnesium
	Manganese
	Molybdenum
	Nickel
	Strontium
	Vanadium
	Zinc

Method Code: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

1. Sample homogenization will depend on the sample type and size.

2. Water samples will not need to be homogenized.

3. For samples weighing less than 100 grams the whole sample will be freeze-dried first, and then homogenized, unless aliquots are being sent for Organic determination, then the sample would be homogenized first and an aliquot taken for freeze-drying.

4. Larger animal samples will be homogenized with a meat grinder. Then an aliquot of approximately 100 grams will be freeze-dried and then further homogenized using a blender, or if necessary, a Spex mixer mill with a Tungsten Carbide vial and ball.

5. Soil and Sediment samples will be mixed and aliquots of 100-200 grams taken for freeze-drying. After freeze-drying, soils will be sieved with a 20 mesh sieve and sediments will be sieved with a 10 mesh sieve followed by grinding with a Spex mixer mill, using a Tungsten Carbide vial and ball.

6. Plant samples will be freeze-dried and then homogenized with a blender, followed if necessary by grinding in a Spex mixer mill with a Tungsten Carbide vial and ball. If aliquots are being sent for Organic determinations, then the samples will be homogenized first, followed by freeze-drying, and further homogenization.

Method Code: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 – Freeze drying and % Moisture

1. Choose an appropriately sized container for the sample. Usually a Whirl-Pak works best for tissue samples. If the sample weighs less than 50 grams and is not being split for organics then use the whole sample.

2. Weigh and record the weight of the bag. If the sample weighs more than 2 grams then a three-place balance should be used. Small samples may require the use of a four or five-place balance.

3. Weight the bag, record the weight and transfer the sample to the bag. Weigh the bag and sample and record the weight. Seal the container or bag and place in a freezer at least overnight or until frozen solid.

4. After the samples are frozen, they are ready to place in the freeze-drier. Turn on the freeze-drier and start the refrigeration. When the temperature reaches -50 C open the container or Whirl-Pak and place in the chamber of the freeze-drier. Close the chamber and start the vacuum pump.

5. Depending on the number of samples and the amount of water present freeze-drying may take 1 – 5 days. When the pressure stops going lower, the samples may be done. If, upon removal, the samples are still cold, place back in the freeze-drier for a longer period of time.

6. After the samples are dry, remove them from the chamber. Then seal the container and weigh on the same balance. Record the weight of the bag and dry sample.

7. Calculate the weight of the dry sample and the weight of the wet sample. To calculate % Moisture divide the weight of the dry sample by the weight of the wet sample, subtract 1 and multiply by 100. Ignore the – sign.

Notes:

1. If the samples do not require % Moisture, then all of the weighing steps can be eliminated.

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the

microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using ½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 018

LABORATORY: Laboratory and Environmental Testing, Inc.

ICP on Perkin-Elmer 4300 DV

Make sure the instrument, Chiller, Air compressor, and gases are on, and at the proper temperatures and pressures. Turn on the computer and double click on the WinLAb32 icon.

Prepare standards and check samples to match the acid matrix of the samples to be analyzed. Change the pump tubing.

Click on “file”, then “Open”, and then “Method”. Click on the method to be used and then click “OK”, TO start the ICP program and call up the Method with the elements to be determined.

Click on the Plasma icon, and click on pump to start the pump and make sure the tubes are in the pump properly. Start the plasma by clicking the “On” icon. Click on the X in the upper right corner to close the Plasma Control. Allow the instrument to warm-up while the samples and standards are loaded into the auto-sampler racks. If the Sample Info table was not filled out previously, then fill in the sample information and save the table using the Batch ID.

Before starting the run, check the Hg wavelength by clicking on “Tools”, and then “Spectrometer Control”. Click on Hg Realign. When that is complete, aspirate a 10.0 Mn Standard and click on “Align View”. After Align View is completed, close the box.

When ready to start analysis, click on the “Auto” icon, make sure that the data is being stored in a file with

the correct name for the Batch, and that the right method is being used. Click the “Analyze” icon and click on “Analyze All”.

When the run is completed, click on “File”, then “Utilities”, then “Data Manager”. Highlight the file, and then click on “Export” icon. Click “Use Existing Design”. Click “Browse” and choose the appropriate template (usually LET-ICP). Click “Open”, place a disk in the “A” drive, and click “Finish”. Click on “Export Data” to transfer data to disk in Drive “A”.

Transfer data to the main computer and calculate the final Concentrations.

[redacted]

Method Codes:	007	012

Lab Matrix	Analyte
Animal Tissue	Arsenic
	Selenium

Method Code: 007

LABORATORY: Laboratory and Environmental Testing, Inc.

L5 – Magnesium Dry Ash

1. Weigh 0.5 g. of sample on a three-place balance and transfer to a cleaned 100 ml. glass beaker with etched numbers. Record the beaker number as well as the sample weight.

2. Wet with 3 ml. of methanol. Then add 5 drops of anti-foam agent, 10 ml. of 40% (W/V) Magnesium Nitrate Hexahydrate, 10 ml. of concentrated trace metal grade HNO3 and 2 ml. of concentrated trace metal grade HCl.

3. Cover with a watch glass and reflux on a hot plate overnight (8-12 hours) at low heat (70-80 C).

4. After reflux increase temperature to 200 C. Slide the watch glass to the side to allow for faster evaporation and cook to complete dryness. This may take 8-12 hours.

5. When no moisture is visible, cover fully with the watch glass and allow to cool.

6. Transfer samples to the cold muffle furnace and use the following program: Start at 250 C and ramp to 500 C at a rate of 1 degree per minute. When 500 C is reached hold for 3 hours then turn off and allow samples to cool to room temperature.

7. Place the cooled samples on a hot plate and add 20 ml. of 50% trace metal grade HCl. Allow the samples to gently boil for 1 hour. After 1 hour readjust volume to 20 ml. with 50 % HCl.

Do not allow the samples to go dry. If necessary add more 50 % HCl during the heating.

8. Allow the samples to cool. Then dilute to 50.0 ml. with D.I. water and transfer to a clean 2 oz. labeled bottle.

Notes:

1. This digestion can be used for As or Se by Hydride Generation AA.

2. This digestion must be used on fish for As by Hydride Generation AA.

Method Code: 012

LABORATORY: Laboratory and Environmental Testing, Inc.

Hydride Generation AA

Turn on the computer, printer, 3100, FIAS 200.and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on either the Se-Fias or As-Fias method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the element and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 0.2% NaBH4-0.05% NaOH, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for this element. Change the position of the tubing or new tubes, if both positions have been used.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Borohydride. Run a 5 or 10 PPB standard until the sensitivity has stabilized and consecutive readings vary by less than 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 15.0 PPB. QC checks are 10.0 and a known Reference sample (Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn

off the EDL lamp, the Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for As or Se FIAS. Then Browse and find the file name given the data. Place a 3.5” disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

Method Codes:	009	013

Lab Matrix	Analyte
Animal Tissue	Mercury

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using

½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 013

LABORATORY: Laboratory and Environmental Testing, Inc.

Cold Vapor AA

Turn on the computer, printer, 3100, FIAS 200,and Argon. Place the appropriate lamp in the instrument and if an EDL turn to its required power. Place the furnace in the burner compartment if it is not already present.

When the computer is ready double click on the WinLab Analyst icon. If the technique is not already FI-Hydride then click on technique and change to FI-Hydride. After the computer has confirmed the IEEE connections are OK, click on Workspace and double click fias.fms. When the screens come up double click on the method and double click on the Hg-CV method. Click on FIAS and turn on the cell.

When the lamp has had time to warm up click on lamps and enter the Hg and click on EDL. Check lamp alignment and wavelength to give the maximum signal. Close lamps.

Prepare the 10% HCl, 5% Stanous Chloride-10% HCl, Calibration standards, and check standards. Change the FIAS tubing and mixing cell if it is not already the set for Mercury. Change the position of the tubing or new tubes, if both positions have been used or determining a different element.

Check the alignment of the furnace in the light path by clicking on Tools and Continuous graphics. Autozero, then check all three positional knobs to get the lowest reading. Autozero whenever necessary.

Start the pumps and place the tubes in the HCl and Stanous Chloride. Run a 10 or 20 PPB standard until the sensitivity has stabilized and consecutive readings vary by less the 2%.

Enter the samples to be run into the Sample Information File. Enter a name for the Data file, and make sure that print log and store data are checked. When the instrument is ready click on Analyze All.

Calibration is done with 0, 1.0, 5.0, 30.0 PPB. QC checks are 10.0, 20.0 and a known Reference Sample(Usually ERA). The 5.00 PPB standard is checked every 10 tubes and if is more than 5% from 5.00 the instrument is recalibrated. If the value is more than 10% from 5.00, then the last 10 samples must be rerun.

After the analysis is finished, rinse system with D.I. water, turn off the pumps (release the pressure), turn off the EDL lamp, the Argon, FIAS and 3100. Click on File then Exit to close the WinLabs Analyst.

Click on WinLab Reformat Icon. Click on Open Design. Pick the design for Hg-CV. Then Browse and find the file name given the data. Place a 3.5” disk in the computer and click on Save Results.

Transfer disk to computer and using Excel calculate the results.

Method Codes:	009	016

Lab Matrix	Analyte
Animal Tissue	Lead

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using ½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 016
LABORATORY: Laboratory and Environmental Testing, Inc. Graphite Furnace using the 5100 Zeeman Turn on the Furnace Coolant, Argon, Computer, Furnace, Zeeman Power Supply and 5100 in that order. Make sure the lamp is in the right position in the turret, and if using an EDL turn on the lamp to the proper power. Double click on the AA WinLab Analyst icon. After the ZHGA-600 and 5100 icons have a green check by them, click on workspace. Double click on LET.fm, then double click on the method. Choose the method to be run and double click on the name. Click on Browse by the Sample Information File, and then pick one for standards. (Example: Pbstds.sif) Click on sample information icon to edit the file with sample names, dilutions, etc. Click Browse for the Results Data Set and enter the name of the file to store data. (Example: Pb010101) Click on Analyze. When samples have been prepared and ready for analysis, click on Analyze All if doing calibration or Analyze Samples if just running samples. The instrument is usually calibrated with a zero and one standard. Then a zero and 3-5 standards are run to check the calibration, followed by an instrument check standard and detection limit. If this is acceptable then the samples are run. Be sure to check that the correct modifier is being used for the element being run. Some samples may require the method of Standard Additions. After the analysis is completed, close AA WinLab Analyst, turn off furnace coolant, Argon, 5100, Zeeman Power supply, Furnace and EDL power supply. Double click on the WinLab Reformat icon. Click on Open Design and choose the design for your element and double click on the name. Click on Browse and find the data file you want to reformat. Double click on the name. Make sure there is a floppy disk in the disk drive and click on Save Results. Transfer to another computer and calculate using Excel.

Method Codes:	009	018

Lab Matrix	Analyte
Animal Tissue	Aluminum
	Boron
	Barium
	Beryllium
	Cadmium
	Chromium
	Copper
	Iron
	Magnesium
	Manganese
	Molybdenum
	Nickel

	Strontium
	Vanadium
	Zinc

Method Code: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 – Microwave Digestion

1. Weigh 0.5 g of dry sample into a clean Teflon digestion vessel. Record the weight to three decimal places.

2. Add 5.0 ml. of concentrated trace metal grade HNO3.

3. Loosely seal to allow release of pressure from the initial acid reaction with the sample.

4. After a few minutes open the vessel and add 1.0 ml of high purity H2O2.

5. Loosely seal the vessel to allow release of pressure.

6. Cap the vessel at the recommended pressure and place in the microwave. Run the program set up for this type of sample.

7. After the microwave heating is complete and the samples have cooled to room temperature, open the vessels and dilute the sample to 50.0 ml. with D.I. water and transfer to a clean 2 oz. plastic bottle. Any vessels that vented during the digestion will need to have the sample redigested and either use less sample or a longer ramp at the lower temperatures.

Notes:

1. Different sample types will require different heating programs to prevent losses due to exceeding the maximum vessel pressure.

2. To keep the same sample dilution, as little as 0.25 g of sample can be weighed and diluted to a final volume of 25.0 ml. using ½ of the HNO3 and H2O2.

3. This digestion can be used for Flame AA, HGA, CV, and ICP.

4. If Mercury is to be run, remove a 10 ml aliquot immediately after dilution and place in a plastic tube and add 100 microliters of concentrated Trace Metal grade Hydrochloric Acid.

Method Code: 018

LABORATORY: Laboratory and Environmental Testing, Inc.

ICP on Perkin-Elmer 4300 DV

Make sure the instrument, Chiller, Air compressor, and gases are on, and at the proper temperatures and pressures. Turn on the computer and double click on the WinLAb32 icon.

Prepare standards and check samples to match the acid matrix of the samples to be analyzed. Change the pump tubing.

Click on “file”, then “Open”, and then “Method”. Click on the method to be used and then click “OK”, TO start the ICP program and call up the Method with the elements to be determined.

Click on the Plasma icon, and click on pump to start the pump and make sure the tubes are in the pump properly. Start the plasma by clicking the “On” icon. Click on the X in the upper right corner to close the Plasma Control. Allow the instrument to warm-up while the samples and standards are loaded into the auto-sampler racks. If the Sample Info table was not filled out previously, then fill in the sample information and save the table using the Batch ID.

Before starting the run, check the Hg wavelength by clicking on “Tools”, and then “Spectrometer Control”. Click on Hg Realign. When that is complete, aspirate a 10.0 Mn Standard and click on “Align View”. After Align View is completed, close the box.

When ready to start analysis, click on the “Auto” icon, make sure that the data is being stored in a file with the correct name for the Batch, and that the right method is being used. Click the “Analyze” icon and click on “Analyze All”.

When the run is completed, click on “File”, then “Utilities”, then “Data Manager”. Highlight the file, and then click on “Export” icon. Click “Use Existing Design”. Click “Browse” and choose the appropriate template (usually LET-ICP). Click “Open”, place a disk in the “A” drive, and click “Finish”. Click on “Export Data” to transfer data to disk in Drive “A”.

Transfer data to the main computer and calculate the final Concentrations.

APPENDIX B
(ANALYTICAL RESULTS)

Table 1. Results of metals analyses in mg/kg dry weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1M = Female #1, Muscle sample, etc; Res Dry = Result dry weight; DL Dry = Detection limit dry weight).

Analyte		Units	F1M	F2M	F3M	F4M	F5M	F6M	F7M	F8M	F9M	F10M
Moisture		%	74.3	72.8	71.9	71.6	71.2	71.9	73.2	71.7	69.8	72
Aluminum	Res Dry	mg/kg	7.4	2	< 2	4	3	2	2	4	4	12
Aluminum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Arsenic	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Arsenic	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Boron	Res Dry	mg/kg	2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	2
Boron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Barium	Res Dry	mg/kg	0.3	< 0.2	< 0.2	0.63	0.4	0.2	0.2	0.2	< 0.2	< 0.2
Barium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Beryllium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Beryllium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cadmium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cadmium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Chromium	Res Dry	mg/kg	< 0.5	< 0.5	0.6	< 0.5	1	0.7	0.6	0.7	0.6	< 0.5
Chromium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Copper	Res Dry	mg/kg	8.3	5.7	7.6	8.6	7	7.6	7.5	8	6.3	7.5
Copper	DL Dry	mg/kg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Iron	Res Dry	mg/kg	130	88	120	170	170	140	160	110	96	110
Iron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Mercury	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Mercury	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Magnesium	Res Dry	mg/kg	1110	1050	1000	961	946	999	1040	1050	983	1050
Magnesium	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Manganese	Res Dry	mg/kg	0.7	0.6	0.7	0.8	0.6	0.7	0.7	0.8	0.6	0.6
Manganese	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Molybdenum	Res Dry	mg/kg	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Molybdenum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Nickel	Res Dry	mg/kg	< 0.5	< 0.5	0.6	2.1	0.8	< 0.5	< 0.5	< 0.5	< 0.5	1
Nickel	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Lead	Res Dry	mg/kg	5.2	< 0.2	< 0.2	< 0.2	< 0.2	0.4	< 0.2	< 0.2	< 0.2	< 0.2
Lead	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Selenium	Res Dry	mg/kg	0.88	0.95	1.2	0.83	1	0.99	0.97	0.62	0.72	0.69
Selenium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Strontium	Res Dry	mg/kg	0.3	0.2	0.3	0.68	0.4	0.3	0.3	0.4	0.3	0.4
Strontium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Vanadium	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Vanadium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc	Res Dry	mg/kg	52.8	57.5	71.9	63.3	55.9	69	66.2	55.1	47	60.9
Zinc	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

Table 1 (concluded). Results of metals analyses in mg/kg dry weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.

(Note: F11M = Female #11, Muscle sample, M1M = Male #1, Muscle sample, etc; Res Dry = Result dry weight; DL Dry = Detection limit dry weight).

Analyte		Units	F11M	F12M	F13M	F14M	F15M	F16M	F17M	F18M	M1M	M2M
Moisture		%	73.3	71.1	72	71.3	73	71.6	73.2	70.8	73.5	74.2
Aluminum	Res Dry	mg/kg	9.7	5	4	2	3	4	3	2	6.4	< 2
Aluminum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Arsenic	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Arsenic	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Boron	Res Dry	mg/kg	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Boron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Barium	Res Dry	mg/kg	0.3	0.2	0.3	< 0.2	< 0.2	0.76	< 0.2	< 0.2	0.3	< 0.2
Barium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Beryllium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Beryllium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cadmium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cadmium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Chromium	Res Dry	mg/kg	< 0.5	< 0.5	1	0.7	< 0.5	0.8	< 0.5	< 0.5	1.5	< 0.5
Chromium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Copper	Res Dry	mg/kg	7.9	7.5	7.7	6.5	8	8	8.2	6	7.4	7.8
Copper	DL Dry	mg/kg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Iron	Res Dry	mg/kg	140	130	130	130	100	130	130	110	120	110
Iron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Mercury	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Mercury	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Magnesium	Res Dry	mg/kg	1010	939	1020	1030	1120	983	1020	913	1040	1110
Magnesium	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Manganese	Res Dry	mg/kg	0.8	0.5	0.8	0.6	0.7	0.7	0.6	0.5	0.6	0.8
Manganese	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Molybdenum	Res Dry	mg/kg	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Molybdenum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2	2
Nickel	Res Dry	mg/kg	0.5	0.5	1.7	< 0.5	< 0.5	0.5	< 0.5	< 0.5	0.5	1
Nickel	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Lead	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Lead	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Selenium	Res Dry	mg/kg	1.3	0.85	0.76	0.74	0.63	1.1	1.4	0.6	0.61	1.3
Selenium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Strontium	Res Dry	mg/kg	0.4	0.3	0.5	0.2	0.2	0.4	0.2	< 0.2	0.4	0.3
Strontium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Vanadium	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Vanadium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc	Res Dry	mg/kg	64.3	68.7	50	57.1	50.4	74.4	61.7	59.4	59.1	67.1
Zinc	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

Table 2. Results of metals analyses in mg/kg dry weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1L = Female #1, Liver sample, etc; Res Dry = Result dry weight; DL Dry = Detection limit dry weight).

Analyte		Units	F1L	F2L	F3L	F6L	F7L	F8L	F9L	F10L	F11L
Moisture		%	68.4	69.1	67.4	70.8	70.9	69.7	68.2	68.2	72.3
Aluminum	Res Dry	mg/kg	< 2	< 2	< 2	2	< 2	< 2	< 2	< 2	< 2
Aluminum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Arsenic	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Arsenic	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Boron	Res Dry	mg/kg	< 2	< 2	< 2	2	< 2	< 2	< 2	< 2	< 2
Boron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Barium	Res Dry	mg/kg	0.5	0.4	< 0.2	0.6	0.6	1	0.3	0.5	0.67
Barium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Beryllium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Beryllium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cadmium	Res Dry	mg/kg	2	1.6	4.4	2.5	3.3	1.3	6.2	3.1	3.6
Cadmium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Chromium	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Chromium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Copper	Res Dry	mg/kg	38	46	150	22	56.6	38.1	155	109	59.4
Copper	DL Dry	mg/kg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Iron	Res Dry	mg/kg	230	264	209	519	559	336	610	319	331
Iron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Mercury	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Mercury	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Magnesium	Res Dry	mg/kg	612	600	576	542	543	573	514	592	610
Magnesium	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Manganese	Res Dry	mg/kg	16	19	14	14	11	13	13	16	13
Manganese	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Molybdenum	Res Dry	mg/kg	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
Molybdenum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Nickel	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	0.8	< 0.5
Nickel	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Lead	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	1.9	< 0.2	< 0.2	< 0.2
Lead	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Selenium	Res Dry	mg/kg	1.1	1.6	2.1	1.3	1.5	0.89	2.5	1.4	1.8
Selenium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Strontium	Res Dry	mg/kg	0.2	< 0.2	< 0.2	0.3	0.3	0.62	0.3	0.4	0.3
Strontium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Vanadium	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Vanadium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc	Res Dry	mg/kg	98.6	122	116	106	93.7	99	86.9	114	129
Zinc	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

Table 2 (concluded). Results of metals analyses in mg/kg dry weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.

(Note: F12L = Female #12, Liver sample, M1L = Male #1, Liver sample, etc; Res Dry = Result dry weight; DL Dry = Detection limit dry weight).

Analyte		Units	F12L	F13L	F14L	F15L	F16L	F17L	F18L	M1L	M2L
Moisture		%	71.7	67.9	68.6	71.5	68.7	70.1	68.6	72.9	68.1
Aluminum	Res Dry	mg/kg	< 2	< 2	2	< 2	< 2	< 2	< 2	< 2	< 2
Aluminum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Arsenic	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Arsenic	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Boron	Res Dry	mg/kg	< 2	< 2	< 2	3	< 2	< 2	< 2	< 2	< 2
Boron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Barium	Res Dry	mg/kg	0.66	0.5	0.3	0.5	< 0.2	< 0.2	0.2	0.9	0.2
Barium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Beryllium	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	0.1
Beryllium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Cadmium	Res Dry	mg/kg	3.3	7.7	1.1	0.34	4.1	3.3	0.96	2	0.75
Cadmium	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Chromium	Res Dry	mg/kg	0.7	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	1	1	0.6
Chromium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Copper	Res Dry	mg/kg	97.6	166	104	129	147	949	75.7	68.2	217
Copper	DL Dry	mg/kg	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Iron	Res Dry	mg/kg	271	270	315	232	215	479	278	387	281
Iron	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Mercury	Res Dry	mg/kg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Mercury	DL Dry	mg/kg	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Magnesium	Res Dry	mg/kg	599	571	558	608	564	546	591	548	558
Magnesium	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Manganese	Res Dry	mg/kg	13	14	13	14	12	12	14	12	13
Manganese	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Molybdenum	Res Dry	mg/kg	< 2	< 2	2	2	< 2	2	2	< 2	2
Molybdenum	DL Dry	mg/kg	2	2	2	2	2	2	2	2	2
Nickel	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	3	0.7	0.5
Nickel	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Lead	Res Dry	mg/kg	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Lead	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Selenium	Res Dry	mg/kg	1.6	1.9	1.5	2	2.2	20.1	1.1	0.9	2.5
Selenium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Strontium	Res Dry	mg/kg	0.4	< 0.2	0.3	0.3	< 0.2	< 0.2	< 0.2	0.5	< 0.2
Strontium	DL Dry	mg/kg	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Vanadium	Res Dry	mg/kg	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Vanadium	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Zinc	Res Dry	mg/kg	118	131	115	123	118	111	162	144	104
Zinc	DL Dry	mg/kg	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5

Table 3. Results of organochlorine pesticides analyses in ug/kg wet weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1M = Female #1, Muscle sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F1M	F2M	F3M	F4M	F5M	F6M	F7M	F8M	F9M	F10M
Lipid ccontent		%	1.49	2.55	3.47	4.25	3.02	3.42	2.34	2.76	7.91	3.69
Moisture content		%	74.13	70.16	71.68	71.3	71.67	71.32	75	72.31	69.92	73.79
Aldrin	Res Wet	ug/kg	< 0.097	< 0.096	< 0.102	< 0.096	< 0.095	< 0.099	< 0.1	< 0.097	< 0.099	< 0.101
Aldrin	DL Wet	ug/kg	0.097	0.096	0.102	0.096	0.095	0.099	0.1	0.097	0.099	0.101
BHC (Total)	Res Wet	ug/kg	< 0.753	< 0.748	< 0.791	< 0.746	< 0.735	< 0.771	< 0.777	< 0.755	< 0.767	< 0.786
BHC (Total)	DL Wet	ug/kg	0.753	0.748	0.791	0.746	0.735	0.771	0.777	0.755	0.767	0.786
DDMU	Res Wet	ug/kg	< 0.129	< 0.128	< 0.136	< 0.128	< 0.126	< 0.132	< 0.133	< 0.13	< 0.132	< 0.135
DDMU	DL Wet	ug/kg	0.129	0.128	0.136	0.128	0.126	0.132	0.133	0.13	0.132	0.135
HCB	Res Wet	ug/kg	< 0.247	< 0.246	< 0.26	< 0.245	< 0.241	< 0.253	< 0.255	< 0.248	< 0.252	< 0.258
HCB	DL Wet	ug/kg	0.247	0.246	0.26	0.245	0.241	0.253	0.255	0.248	0.252	0.258
Heptachlor	Res Wet	ug/kg	< 0.08	< 0.079	< 0.084	< 0.079	< 0.078	< 0.082	< 0.082	< 0.08	< 0.081	< 0.083
Heptachlor	DL Wet	ug/kg	0.08	0.079	0.084	0.079	0.078	0.082	0.082	0.08	0.081	0.083
PCB-TOTAL	Res Wet	ug/kg	< 3.36	< 3.34	< 3.54	< 3.33	< 3.28	< 3.45	< 3.47	< 3.38	< 3.43	< 3.51
PCB-TOTAL	DL Wet	ug/kg	3.36	3.34	3.54	3.33	3.28	3.45	3.47	3.38	3.43	3.51
Total DDT's	Res Wet	ug/kg	< 0.712	< 0.708	< 0.748	< 0.705	< 0.695	< 0.729	< 0.735	< 0.714	< 0.725	< 0.744
Total DDT's	DL Wet	ug/kg	0.712	0.708	0.748	0.705	0.695	0.729	0.735	0.714	0.725	0.744
alpha BHC	Res Wet	ug/kg	< 0.263	< 0.262	< 0.276	< 0.261	< 0.257	< 0.27	< 0.272	< 0.264	< 0.268	< 0.275
alpha BHC	DL Wet	ug/kg	0.263	0.262	0.276	0.261	0.257	0.27	0.272	0.264	0.268	0.275
alpha chlordane	Res Wet	ug/kg	< 0.092	< 0.092	< 0.097	< 0.091	< 0.09	< 0.095	< 0.095	< 0.093	< 0.094	< 0.096
alpha chlordane	DL Wet	ug/kg	0.092	0.092	0.097	0.091	0.09	0.095	0.095	0.093	0.094	0.096
beta BHC	Res Wet	ug/kg	< 0.242	< 0.241	< 0.254	< 0.24	< 0.236	< 0.248	< 0.25	< 0.243	< 0.247	< 0.253
beta BHC	DL Wet	ug/kg	0.242	0.241	0.254	0.24	0.236	0.248	0.25	0.243	0.247	0.253
cis-nonachlor	Res Wet	ug/kg	< 0.127	< 0.126	< 0.134	< 0.126	< 0.124	< 0.13	< 0.131	< 0.128	< 0.13	< 0.133
cis-nonachlor	DL Wet	ug/kg	0.127	0.126	0.134	0.126	0.124	0.13	0.131	0.128	0.13	0.133
delta BHC	Res Wet	ug/kg	< 0.267	< 0.266	< 0.281	< 0.265	< 0.261	< 0.274	< 0.276	< 0.268	< 0.272	< 0.279
delta BHC	DL Wet	ug/kg	0.267	0.266	0.281	0.265	0.261	0.274	0.276	0.268	0.272	0.279
dieldrin	Res Wet	ug/kg	< 0.354	< 0.352	< 0.372	< 0.351	< 0.346	< 0.363	< 0.366	< 0.356	< 0.361	< 0.37
dieldrin	DL Wet	ug/kg	0.354	0.352	0.372	0.351	0.346	0.363	0.366	0.356	0.361	0.37
endosulfan I	Res Wet	ug/kg	< 0.377	< 0.375	< 0.396	< 0.374	< 0.368	< 0.386	< 0.389	< 0.378	< 0.384	< 0.394
endosulfan I	DL Wet	ug/kg	0.377	0.375	0.396	0.374	0.368	0.386	0.389	0.378	0.384	0.394
endosulfan II	Res Wet	ug/kg	< 0.359	< 0.357	< 0.377	< 0.356	< 0.35	< 0.368	< 0.371	< 0.36	< 0.366	< 0.375
endosulfan II	DL Wet	ug/kg	0.359	0.357	0.377	0.356	0.35	0.368	0.371	0.36	0.366	0.375
endosulfan sulfate	Res Wet	ug/kg	< 0.395	< 0.393	< 0.415	< 0.392	< 0.386	< 0.405	< 0.408	< 0.397	< 0.403	< 0.413
endosulfan sulfate	DL Wet	ug/kg	0.395	0.393	0.415	0.392	0.386	0.405	0.408	0.397	0.403	0.413
endrin	Res Wet	ug/kg	< 0.431	< 0.429	< 0.453	< 0.427	< 0.421	< 0.442	< 0.445	< 0.433	< 0.439	< 0.451
endrin	DL Wet	ug/kg	0.431	0.429	0.453	0.427	0.421	0.442	0.445	0.433	0.439	0.451

Table 3 (continued). Results of organochlorine pesticides analyses in ug/kg wet weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F11M = Female #11, Muscle sample and M1M = Male #1, Muscle sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F11M	F12M	F13M	F14M	F15M	F16M	F17M	F18M	M1M	M2M
Lipid ccontent		%	2.86	3.37	2.6	3.57	1.22	2.37	1.75	3.62	1.78	0.91
Moisture content		%	72.41	71.76	72.32	70.31	74.38	72.26	73.26	69.47	73.78	76.09
Aldrin	Res Wet	ug/kg	< 0.096	< 0.095	< 0.1	< 0.097	< 0.095	< 0.096	< 0.1	< 0.096	< 0.098	< 0.099
Aldrin	DL Wet	ug/kg	0.096	0.095	0.1	0.097	0.095	0.096	0.1	0.096	0.098	0.099
BHC (Total)	Res Wet	ug/kg	< 0.743	< 0.739	< 0.776	< 0.754	< 0.736	< 0.742	< 0.776	< 0.748	< 0.763	< 0.768
BHC (Total)	DL Wet	ug/kg	0.743	0.739	0.776	0.754	0.736	0.742	0.776	0.748	0.763	0.768
DDMU	Res Wet	ug/kg	< 0.128	< 0.127	< 0.133	< 0.129	< 0.126	< 0.127	< 0.133	< 0.128	< 0.131	< 0.132
DDMU	DL Wet	ug/kg	0.128	0.127	0.133	0.129	0.126	0.127	0.133	0.128	0.131	0.132
HCB	Res Wet	ug/kg	< 0.244	< 0.242	< 0.255	< 0.247	< 0.242	< 0.243	< 0.255	< 0.246	< 0.25	< 0.252
HCB	DL Wet	ug/kg	0.244	0.242	0.255	0.247	0.242	0.243	0.255	0.246	0.25	0.252
Heptachlor	Res Wet	ug/kg	< 0.079	< 0.078	< 0.082	< 0.08	< 0.078	< 0.079	< 0.082	< 0.079	< 0.081	< 0.081
Heptachlor	DL Wet	ug/kg	0.079	0.078	0.082	0.08	0.078	0.079	0.082	0.079	0.081	0.081
PCB-TOTAL	Res Wet	ug/kg	< 3.32	< 3.3	< 3.47	< 3.37	< 3.29	< 3.31	< 3.47	< 3.34	< 3.41	< 3.43
PCB-TOTAL	DL Wet	ug/kg	3.32	3.3	3.47	3.37	3.29	3.31	3.47	3.34	3.41	3.43
Total DDT's	Res Wet	ug/kg	< 0.703	< 0.699	< 0.734	< 0.713	< 0.696	< 0.701	< 0.734	< 0.708	< 0.721	< 0.727
Total DDT's	DL Wet	ug/kg	0.703	0.699	0.734	0.713	0.696	0.701	0.734	0.708	0.721	0.727
alpha BHC	Res Wet	ug/kg	< 0.26	< 0.258	< 0.271	< 0.264	< 0.257	< 0.259	< 0.271	< 0.262	< 0.267	< 0.269
alpha BHC	DL Wet	ug/kg	0.26	0.258	0.271	0.264	0.257	0.259	0.271	0.262	0.267	0.269
alpha chlordane	Res Wet	ug/kg	< 0.091	< 0.091	< 0.095	< 0.092	< 0.09	< 0.091	< 0.095	< 0.092	< 0.093	< 0.094
alpha chlordane	DL Wet	ug/kg	0.091	0.091	0.095	0.092	0.09	0.091	0.095	0.092	0.093	0.094
beta BHC	Res Wet	ug/kg	< 0.239	< 0.238	< 0.249	< 0.242	, 0.237	< 0.238	< 0.249	< 0.241	< 0.245	, 0.247
beta BHC	DL Wet	ug/kg	0.239	0.238	0.249	0.242	0.237	0.238	0.249	0.241	0.245	0.247
cis-nonachlor	Res Wet	ug/kg	< 0.125	< 0.125	< 0.131	< 0.127	< 0.124	, 0.125	< 0.131	< 0.126	< 0.129	< 0.13
cis-nonachlor	DL Wet	ug/kg	0.125	0.125	0.131	0.127	0.124	0.125	0.131	0.126	0.129	0.13
delta BHC	Res Wet	ug/kg	< 0.264	< 0.262	< 0.276	< 0.268	< 0.262	< 0.263	< 0.276	< 0.266	< 0.271	< 0.273
delta BHC	DL Wet	ug/kg	0.264	0.262	0.276	0.268	0.262	0.263	0.276	0.266	0.271	0.273
dieldrin	Res Wet	ug/kg	< 0.35	< 0.348	< 0.365	< 0.355	< 0.347	< 0.349	< 0.365	< 0.352	< 0.359	< 0.362
dieldrin	DL Wet	ug/kg	0.35	0.348	0.365	0.355	0.347	0.349	0.365	0.352	0.359	0.362
endosulfan I	Res Wet	ug/kg	< 0.372	< 0.37	< 0.389	< 0.378	< 0.369	< 0.372	< 0.389	< 0.375	< 0.382	< 0.385
endosulfan I	DL Wet	ug/kg	0.372	0.37	0.389	0.378	0.369	0.372	0.389	0.375	0.382	0.385
endosulfan II	Res Wet	ug/kg	< 0.354	< 0.352	< 0.37	< 0.36	< 0.351	< 0.354	< 0.37	< 0.357	< 0.364	< 0.366
endosulfan II	DL Wet	ug/kg	0.354	0.352	0.37	0.36	0.351	0.354	0.37	0.357	0.364	0.366
endosulfan sulfate	Res Wet	ug/kg	< 0.39	< 0.388	< 0.407	< 0.396	< 0.387	< 0.389	< 0.407	< 0.393	< 0.4	< 0.404
endosulfan sulfate	DL Wet	ug/kg	0.39	0.388	0.407	0.396	0.387	0.389	0.407	0.393	0.4	0.404
endrin	Res Wet	ug/kg	< 0.426	< 0.423	< 0.444	< 0.432	< 0.422	< 0.425	< 0.444	< 0.429	< 0.437	< 0.44
endrin	DL Wet	ug/kg	0.426	0.423	0.444	0.432	0.422	0.425	0.444	0.429	0.437	0.44

Table 4. Results of organochlorine pesticides analyses in ug/kg wet weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1L = Female #1, Liver sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F1L	F2L	F3L	F6L	F7L	F8L	F9L	F10L	F11L
Lipid Content		%	3.7	3.04	3.42	2.87	3.89	3.61	3.86	4.41	3.83
Moisture Content		%	70.42	71.35	69.47	71.35	72.78	71.5	71.71	70.13	75.54
Aldrin	Res Wet	ug/kg	< 0.259	< 0.236	< 0.254	< 0.184	< 0.225	< 0.193	< 0.242	< 0.24	< 0.231
Aldrin	DL Wet	ug/kg	0.259	0.236	0.254	0.184	0.225	0.193	0.242	0.24	0.231
BHC (Total)	Res Wet	ug/kg	< 2.01	< 1.83	< 1.97	< 1.43	< 1.75	< 1.49	2.99	< 1.86	3.39
BHC (Total)	DL Wet	ug/kg	2.01	1.83	1.97	1.43	1.75	1.49	1.88	1.86	1.79
DDMU	Res Wet	ug/kg	< 0.344	< 0.315	< 0.338	< 0.246	< 0.3	< 0.256	< 0.322	< 0.319	< 0.308
DDMU	DL Wet	ug/kg	0.344	0.315	0.338	0.246	0.3	0.256	0.322	0.319	0.308
HCB	Res Wet	ug/kg	< 0.659	< 0.602	< 0.646	< 0.469	< 0.573	< 0.49	< 0.616	< 0.61	< 0.588
HCB	DL Wet	ug/kg	0.659	0.602	0.646	0.469	0.573	0.49	0.616	0.61	0.588
Heptachlor	Res Wet	ug/kg	< 0.213	< 0.194	< 0.209	< 0.152	< 0.185	< 0.158	< 0.199	< 0.197	< 0.19
Heptachlor	DL Wet	ug/kg	0.213	0.194	0.209	0.152	0.185	0.158	0.199	0.197	0.19
PCB-TOTAL	Res Wet	ug/kg	< 8.97	< 8.19	< 8.79	< 6.39	< 7.81	< 6.68	< 8.38	< 8.31	< 8.01
PCB-TOTAL	DL Wet	ug/kg	8.97	8.19	8.79	6.39	7.81	6.68	8.38	8.31	8.01
Total DDT's	Res Wet	ug/kg	< 1.9	1.77	< 1.86	< 1.35	< 1.65	< 1.41	< 1.77	1.76	< 1.7
Total DDT's	DL Wet	ug/kg	1.9	1.73	1.86	1.35	1.65	1.41	1.77	1.76	1.7
alpha BHC	Res Wet	ug/kg	< 0.701	< 0.641	< 0.688	< 0.5	< 0.61	< 0.522	< 0.656	< 0.65	< 0.627
alpha BHC	DL Wet	ug/kg	0.701	0.641	0.688	0.5	0.61	0.522	0.656	0.65	0.627
alpha chlordane	Res Wet	ug/kg	< 0.246	< 0.225	< 0.241	< 0.175	< 0.214	< 0.183	< 0.23	< 0.228	< 0.22
alpha chlordane	DL Wet	ug/kg	0.246	0.225	0.241	0.175	0.214	0.183	0.23	0.228	0.22
beta BHC	Res Wet	ug/kg	< 0.645	< 0.589	< 0.633	< 0.46	< 0.561	< 0.48	< 0.603	< 0.598	< 0.576
beta BHC	DL Wet	ug/kg	0.645	0.589	0.633	0.46	0.561	0.48	0.603	0.598	0.576
cis-nonachlor	Res Wet	ug/kg	< 0.339	< 0.31	< 0.332	< 0.242	< 0.295	< 0.252	< 0.317	< 0.314	< 0.303
cis-nonachlor	DL Wet	ug/kg	0.339	0.31	0.332	0.242	0.295	0.252	0.317	0.314	0.303
delta BHC	Res Wet	ug/kg	< 0.713	< 0.651	< 0.699	< 0.508	< 0.62	< 0.531	< 0.667	< 0.66	< 0.637
delta BHC	DL Wet	ug/kg	0.713	0.651	0.699	0.508	0.62	0.531	0.667	0.66	0.637
dieldrin	Res Wet	ug/kg	< 0.945	< 0.863	< 0.926	< 0.673	< 0.822	< 0.703	< 0.883	< 0.875	< 0.844
dieldrin	DL Wet	ug/kg	0.945	0.863	0.926	0.673	0.822	0.703	0.883	0.875	0.844
endosulfan I	Res Wet	ug/kg	< 1.01	< 0.919	< 0.986	< 0.717	< 0.875	< 0.749	< 0.94	< 0.931	< 0.898
endosulfan I	DL Wet	ug/kg	1.01	0.919	0.986	0.717	0.875	0.749	0.94	0.931	0.898
endosulfan II	Res Wet	ug/kg	< 0.957	< 0.874	< 0.938	< 0.682	< 0.833	< 0.712	< 0.895	< 0.886	< 0.855
endosulfan II	DL Wet	ug/kg	0.957	0.874	0.938	0.682	0.833	0.712	0.895	0.886	0.855
endosulfan sulfate	Res Wet	ug/kg	< 1.05	< 0.963	< 1.03	< 0.751	< 0.917	< 0.785	< 0.985	< 0.976	< 0.942
endosulfan sulfate	DL Wet	ug/kg	1.05	0.963	1.03	0.751	0.917	0.785	0.985	0.976	0.942
endrin	Res Wet	ug/kg	< 1.15	< 1.05	< 1.13	< 0.82	< 1	< 0.856	< 1.08	< 1.06	< 1.03
endrin	DL Wet	ug/kg	1.15	1.05	1.13	0.82	1	0.856	1.08	1.06	1.03

Table 4 (continued). Results of organochlorine pesticides analyses in ug/kg wet weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F12L = Female #12, Liver sample and M1L = Male #1, Liver sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F12L	F13L	F14L	F15L	F16L	F17L	F18L	M1L	M2L
Lipid Content		%	4.87	6.35	2.98	4.94	6.89	4.16	5.21	4.59	4.8
Moisture Content		%	73.13	69.17	69.27	73.51	70.32	73.04	71.68	73.05	71.83
Aldrin	Res Wet	ug/kg	< 0.26	< 0.216	< 0.23	< 0.241	< 0.257	< 0.24	< 0.255	< 0.236	< 0.24
Aldrin	DL Wet	ug/kg	0.26	0.216	0.23	0.241	0.257	0.24	0.255	0.236	0.24
BHC (Total)	Res Wet	ug/kg	2.81	< 1.67	3.72	2.52	2.89	< 1.86	3.31	1.98	< 1.86
BHC (Total)	DL Wet	ug/kg	2.02	1.67	1.78	1.87	2	1.86	1.98	1.83	1.86
DDMU	Res Wet	ug/kg	< 0.346	< 0.287	< 0.306	< 0.321	< 0.343	< 0.319	< 0.339	< 0.315	< 0.319
DDMU	DL Wet	ug/kg	0.346	0.287	0.306	0.321	0.343	0.319	0.339	0.315	0.319
HCB	Res Wet	ug/kg	< 0.662	< 0.549	< 0.586	< 0.613	< 0.655	< 0.61	< 0.649	< 0.602	< 0.61
HCB	DL Wet	ug/kg	0.662	0.549	0.586	0.613	0.655	0.61	0.649	0.602	0.61
Heptachlor	Res Wet	ug/kg	< 0.214	< 0.177	< 0.189	< 0.198	< 0.212	< 0.197	< 0.21	< 0.194	< 0.197
Heptachlor	DL Wet	ug/kg	0.214	0.177	0.189	0.198	0.212	0.197	0.21	0.194	0.197
PCB-TOTAL	Res Wet	ug/kg	< 9.01	< 7.48	< 7.98	< 8.35	< 8.92	< 8.31	< 8.84	< 8.19	< 8.31
PCB-TOTAL	DL Wet	ug/kg	9.01	7.48	7.98	8.35	8.92	8.31	8.84	8.19	8.31
Total DDT's	Res Wet	ug/kg	< 1.91	< 1.58	< 1.69	< 1.77	< 1.89	< 1.76	< 1.87	< 1.73	< 1.76
Total DDT's	DL Wet	ug/kg	1.91	1.58	1.69	1.77	1.89	1.76	1.87	1.73	1.76
alpha BHC	Res Wet	ug/kg	< 0.705	< 0.585	< 0.624	< 0.653	< 0.698	< 0.65	< 0.691	< 0.641	< 0.65
alpha BHC	DL Wet	ug/kg	0.705	0.585	0.624	0.653	0.698	0.65	0.691	0.641	0.65
alpha chlordane	Res Wet	ug/kg	< 0.247	< 0.205	< 0.219	< 0.229	< 0.245	< 0.228	< 0.242	< 0.225	< 0.228
alpha chlordane	DL Wet	ug/kg	0.247	0.205	0.219	0.229	0.245	0.228	0.242	0.225	0.228
beta BHC	Res Wet	ug/kg	< 0.648	< 0.538	< 0.574	< 0.6	< 0.642	< 0.598	< 0.636	< 0.589	< 0.598
beta BHC	DL Wet	ug/kg	0.648	0.538	0.574	0.6	0.642	0.598	0.636	0.589	0.598
cis-nonachlor	Res Wet	ug/kg	< 0.341	< 0.283	< 0.301	< 0.315	< 0.337	< 0.314	< 0.334	< 0.31	< 0.314
cis-nonachlor	DL Wet	ug/kg	0.341	0.283	0.301	0.315	0.337	0.314	0.334	0.31	0.314
delta BHC	Res Wet	ug/kg	< 0.717	< 0.595	< 0.634	< 0.663	< 0.709	< 0.66	< 0.702	< 0.651	< 0.66
delta BHC	DL Wet	ug/kg	0.717	0.595	0.634	0.663	0.709	0.66	0.702	0.651	0.66
dieldrin	Res Wet	ug/kg	< 0.95	< 0.788	< 0.84	< 0.879	< 0.94	< 0.875	< 0.931	< 0.863	< 0.875
dieldrin	DL Wet	ug/kg	0.95	0.788	0.84	0.879	0.94	0.875	0.931	0.863	0.875
endosulfan I	Res Wet	ug/kg	< 1.01	< 0.839	< 0.894	< 0.936	< 1	< 0.931	< 0.991	< 0.919	< 0.931
endosulfan I	DL Wet	ug/kg	1.01	0.839	0.894	0.936	1	0.931	0.991	0.919	0.931
endosulfan II	Res Wet	ug/kg	< 0.962	< 0.798	< 0.851	< 0.891	< 0.952	< 0.886	< 0.943	< 0.874	< 0.886
endosulfan II	DL Wet	ug/kg	0.962	0.798	0.851	0.891	0.952	0.886	0.943	0.874	0.886
endosulfan sulfate	Res Wet	ug/kg	< 1.06	< 0.879	< 0.937	< 0.981	< 1.05	< 0.976	< 1.04	< 0.963	< 0.976
endosulfan sulfate	DL Wet	ug/kg	1.06	0.879	0.937	0.981	1.05	0.976	1.04	0.963	0.976
endrin	Res Wet	ug/kg	< 1.16	< 0.959	< 1.02	< 1.07	< 1.14	< 1.06	< 1.13	< 1.05	< 1.06
endrin	DL Wet	ug/kg	1.16	0.959	1.02	1.07	1.14	1.06	1.13	1.05	1.06

Table 4 (continued). Results of organochlorine pesticides analyses in ug/kg wet weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1L = Female #1, Liver sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F1L	F2L	F3L	F6L	F7L	F8L	F9L	F10L	F11L
gamma BHC	Res Wet	ug/kg	< 0.347	< 0.317	< 0.34	< 0.247	< 0.302	< 0.258	2.99	< 0.321	3.39
gamma BHC	DL Wet	ug/kg	0.347	0.317	0.34	0.247	0.302	0.258	0.324	0.321	0.31
gamma chlordane	Res Wet	ug/kg	< 0.282	< 0.258	< 0.277	< 0.201	< 0.246	< 0.21	< 0.264	< 0.262	< 0.252
gamma chlordane	DL Wet	ug/kg	0.282	0.258	0.277	0.201	0.246	0.21	0.264	0.262	0.252
heptachlor epoxide	Res Wet	ug/kg	< 0.752	< 0.687	< 0.738	< 0.536	< 0.655	< 0.56	< 0.703	< 0.697	< 0.672
heptachlor epoxide	DL Wet	ug/kg	0.752	0.687	0.738	0.536	0.655	0.56	0.703	0.697	0.672
mirex	Res Wet	ug/kg	< 0.243	< 0.222	< 0.238	< 0.173	< 0.211	< 0.181	< 0.227	< 0.225	< 0.217
mirex	DL Wet	ug/kg	0.243	0.222	0.238	0.173	0.211	0.181	0.227	0.225	0.217
o,p'-DDD	Res Wet	ug/kg	< 0.486	< 0.444	< 0.477	< 0.346	< 0.423	< 0.362	< 0.454	< 0.45	< 0.434
o,p'-DDD	DL Wet	ug/kg	0.486	0.444	0.477	0.346	0.423	0.362	0.454	0.45	0.434
o,p'-DDE	Res Wet	ug/kg	0.909	1.77	1.72	1.03	1.49	1.02	1.65	1.76	0.589
o,p'-DDE	DL Wet	ug/kg	0.23	0.211	0.226	0.164	0.201	0.172	0.215	0.213	0.206
o,p'-DDT	Res Wet	ug/kg	< 0.363	< 0.331	< 0.356	< 0.259	< 0.316	< 0.27	< 0.339	< 0.336	< 0.324
o,p'-DDT	DL Wet	ug/kg	0.363	0.331	0.356	0.259	0.316	0.27	0.339	0.336	0.324
oxychlordane	Res Wet	ug/kg	15.4	6.5	4.03	3.27	5	1.79	2.16	0.964	4.14
oxychlordane	DL Wet	ug/kg	0.367	0.336	0.36	0.262	0.32	0.273	0.343	0.34	0.328
p,p'-DDD	Res Wet	ug/kg	< 0.488	< 0.446	< 0.478	< 0.348	< 0.424	< 0.363	< 0.456	< 0.452	< 0.436
p,p'-DDD	DL Wet	ug/kg	0.488	0.446	0.478	0.348	0.424	0.363	0.456	0.452	0.436
p,p'-DDE	Res Wet	ug/kg	< 0.242	< 0.221	< 0.237	< 0.172	< 0.21	< 0.18	< 0.226	< 0.224	< 0.216
p,p'-DDE	DL Wet	ug/kg	0.242	0.221	0.237	0.172	0.21	0.18	0.226	0.224	0.216
p,p'-DDT	Res Wet	ug/kg	< 0.258	< 0.236	< 0.253	< 0.184	< 0.225	< 0.192	< 0.241	< 0.239	< 0.231
p,p'-DDT	DL Wet	ug/kg	0.258	0.236	0.253	0.184	0.225	0.192	0.241	0.239	0.231
pentachloro-anisole	Res Wet	ug/kg	< 0.266	< 0.243	< 0.26	< 0.189	< 0.231	< 0.198	< 0.248	< 0.246	< 0.237
pentachloro-anisole	DL Wet	ug/kg	0.266	0.243	0.26	0.189	0.231	0.198	0.248	0.246	0.237
toxaphene	Res Wet	ug/kg	< 24.9	< 22.7	< 24.4	< 17.7	< 21.6	< 18.5	< 23.3	< 23	< 22.2
toxaphene	DL Wet	ug/kg	24.9	22.7	24.4	17.7	21.6	18.5	23.3	23	22.2
trans-nonachlor	Res Wet	ug/kg	< 0.271	< 0.247	< 0.265	< 0.193	< 0.236	< 0.202	< 0.253	< 0.251	< 0.242
trans-nonachlor	DL Wet	ug/kg	0.271	0.247	0.265	0.193	0.236	0.202	0.253	0.251	0.242
chlorpyrifos	Res Wet	ug/kg	< 0.589	< 0.538	< 0.577	< 0.42	< 0.512	< 0.438	< 0.551	< 0.546	< 0.526
chlorpyrifos	DL Wet	ug/kg	0.589	0.538	0.577	0.42	0.512	0.438	0.551	0.546	0.526
1,2,3,4-Tetrachlorobenzene	Res Wet	ug/kg	< 0.272	< 0.248	< 0.267	< 0.194	< 0.237	< 0.202	< 0.254	< 0.252	< 0.243
1,2,3,4-Tetrachlorobenzene	DL Wet	ug/kg	0.272	0.248	0.267	0.194	0.237	0.202	0.254	0.252	0.243
1,2,4,5-Tetrachlorobenzene	Res Wet	ug/kg	< 0.54	< 0.494	< 0.53	< 0.385	< 0.47	< 0.402	< 0.505	< 0.501	< 0.483
1,2,4,5-Tetrachlorobenzene	DL Wet	ug/kg	0.54	0.494	0.53	0.385	0.47	0.402	0.505	0.501	0.483
Pentachlorobenzene	Res Wet	ug/kg	< 0.259	< 0.237	< 0.254	< 0.185	< 0.226	< 0.193	< 0.243	< 0.24	< 0.232
Pentachlorobenzene	DL Wet	ug/kg	0.259	0.237	0.254	0.185	0.226	0.193	0.243	0.24	0.232

Table 4 (concluded). Results of organochlorine pesticides analyses in ug/kg wet weight for liver tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F12L = Female #12, Liver sample and M1L = Male #1, Liver sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F12L	F13L	F14L	F15L	F16L	F17L	F18L	M1L	M2L
gamma BHC	Res Wet	ug/kg	2.81	1.23	3.72	2.52	2.89	< 0.321	3.31	1.98	< 0.321
gamma BHC	DL Wet	ug/kg	0.348	0.289	0.308	0.322	0.345	0.321	0.341	0.317	0.321
gamma chlordane	Res Wet	ug/kg	< 0.284	< 0.236	< 0.251	< 0.263	< 0.281	< 0.262	< 0.278	< 0.258	< 0.262
gamma chlordane	DL Wet	ug/kg	0.284	0.236	0.251	0.263	0.281	0.262	0.278	0.258	0.262
heptachlor epoxide	Res Wet	ug/kg	< 0.756	< 0.628	< 0.669	< 0.7	< 0.749	< 0.697	< 0.741	< 0.687	< 0.697
heptachlor epoxide	DL Wet	ug/kg	0.756	0.628	0.669	0.7	0.749	0.697	0.741	0.687	0.697
mirex	Res Wet	ug/kg	< 0.244	< 0.203	< 0.216	< 0.226	< 0.242	< 0.225	< 0.239	< 0.222	< 0.225
mirex	DL Wet	ug/kg	0.244	0.203	0.216	0.226	0.242	0.225	0.239	0.222	0.225
o,p'-DDD	Res Wet	ug/kg	< 0.489	< 0.405	< 0.432	< 0.452	< 0.484	< 0.45	< 0.479	< 0.444	< 0.45
o,p'-DDD	DL Wet	ug/kg	0.489	0.405	0.432	0.452	0.484	0.45	0.479	0.444	0.45
o,p'-DDE	Res Wet	ug/kg	1.43	0.229	1.17	0.866	1.43	1.43	0.975	1.21	< 0.213
o,p'-DDE	DL Wet	ug/kg	0.232	0.192	0.205	0.214	0.229	0.213	0.227	0.211	0.213
o,p'-DDT	Res Wet	ug/kg	< 0.365	< 0.303	< 0.323	< 0.338	< 0.361	< 0.336	< 0.357	< 0.331	< 0.336
o,p'-DDT	DL Wet	ug/kg	0.365	0.303	0.323	0.338	0.361	0.336	0.357	0.331	0.336
oxychlordane	Res Wet	ug/kg	10.2	22.3	3.75	7.68	4.21	2.59	4.03	6.97	6.93
oxychlordane	DL Wet	ug/kg	0.369	0.306	0.327	0.342	0.366	0.34	0.362	0.336	0.34
p,p'-DDD	Res Wet	ug/kg	< 0.49	< 0.407	< 0.434	< 0.454	< 0.485	< 0.452	< 0.481	< 0.446	< 0.452
p,p'-DDD	DL Wet	ug/kg	0.49	0.407	0.434	0.454	0.485	0.452	0.481	0.446	0.452
p,p'-DDE	Res Wet	ug/kg	< 0.243	< 0.202	< 0.215	< 0.225	< 0.24	< 0.224	< 0.238	< 0.221	< 0.224
p,p'-DDE	DL Wet	ug/kg	0.243	0.202	0.215	0.225	0.24	0.224	0.238	0.221	0.224
p,p'-DDT	Res Wet	ug/kg	< 0.259	< 0.215	< 0.23	< 0.24	< 0.257	< 0.239	< 0.254	< 0.236	< 0.239
p,p'-DDT	DL Wet	ug/kg	0.259	0.215	0.23	0.24	0.257	0.239	0.254	0.236	0.239
pentachloro-anisole	Res Wet	ug/kg	< 0.267	< 0.221	< 0.236	< 0.247	< 0.264	< 0.246	< 0.262	< 0.243	< 0.246
pentachloro-anisole	DL Wet	ug/kg	0.267	0.221	0.236	0.247	0.264	0.246	0.262	0.243	0.246
toxaphene	Res Wet	ug/kg	< 25	< 20.7	< 22.1	< 23.1	< 24.8	< 23	< 24.5	< 22.7	< 23
toxaphene	DL Wet	ug/kg	25	20.7	22.1	23.1	24.8	23	24.5	22.7	23
trans-nonachlor	Res Wet	ug/kg	< 0.272	< 0.226	< 0.241	< 0.252	< 0.269	< 0.251	< 0.267	< 0.247	< 0.251
trans-nonachlor	DL Wet	ug/kg	0.272	0.226	0.241	0.252	0.269	0.251	0.267	0.247	0.251
chlorpyrifos	Res Wet	ug/kg	< 0.592	< 0.491	< 0.524	< 0.548	< 0.586	< 0.546	< 0.58	< 0.538	< 0.546
chlorpyrifos	DL Wet	ug/kg	0.592	0.491	0.524	0.548	0.586	0.546	0.58	0.538	0.546
1,2,3,4-Tetrachlorobenzene	Res Wet	ug/kg	< 0.273	< 0.227	< 0.242	< 0.253	< 0.271	< 0.252	< 0.268	< 0.248	< 0.252
1,2,3,4-Tetrachlorobenzene	DL Wet	ug/kg	0.273	0.227	0.242	0.253	0.271	0.252	0.268	0.248	0.252
1,2,4,5-Tetrachlorobenzene	Res Wet	ug/kg	< 0.543	< 0.451	< 0.481	< 0.503	< 0.538	< 0.501	< 0.532	< 0.494	< 0.501
1,2,4,5-Tetrachlorobenzene	DL Wet	ug/kg	0.543	0.451	0.481	0.503	0.538	0.501	0.532	0.494	0.501
Pentachlorobenzene	Res Wet	ug/kg	< 0.261	< 0.216	< 0.231	< 0.241	< 0.258	< 0.24	< 0.256	< 0.237	< 0.24
Pentachlorobenzene	DL Wet	ug/kg	0.261	0.216	0.231	0.241	0.258	0.24	0.256	0.237	0.24

Table 3 (continued). Results of organochlorine pesticides analyses in ug/kg wet weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.
(Note: F1M = Female #1, Muscle sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F1M	F2M	F3M	F4M	F5M	F6M	F7M	F8M	F9M	F10M
gamma BHC	Res Wet	ug/kg	< 0.13	< 0.129	< 0.137	< 0.129	< 0.127	< 0.133	< 0.134	< 0.13	< 0.132	< 0.136
gamma BHC	DL Wet	ug/kg	0.13	0.129	0.137	0.129	0.127	0.133	0.134	0.13	0.132	0.136
gamma chlordane	Res Wet	ug/kg	< 0.106	< 0.105	< 0.111	< 0.105	< 0.103	< 0.109	< 0.109	< 0.106	< 0.108	< 0.111
gamma chlordane	DL Wet	ug/kg	0.106	0.105	0.111	0.105	0.103	0.109	0.109	0.106	0.108	0.111
heptachlor epoxide	Res Wet	ug/kg	< 0.282	< 0.281	< 0.297	< 0.28	< 0.275	< 0.289	< 0.291	< 0.283	< 0.288	< 0.295
heptachlor epoxide	DL Wet	ug/kg	0.282	0.281	0.297	0.28	0.275	0.289	0.291	0.283	0.288	0.295
mirex	Res Wet	ug/kg	< 0.091	< 0.091	< 0.096	0.212	< 0.089	< 0.093	< 0.094	< 0.091	< 0.093	< 0.095
mirex	DL Wet	ug/kg	0.091	0.091	0.096	0.09	0.089	0.093	0.094	0.091	0.093	0.095
o,p'-DDD	Res Wet	ug/kg	< 0.182	< 0.181	< 0.192	< 0.181	< 0.178	< 0.187	< 0.188	< 0.183	< 0.186	< 0.19
o,p'-DDD	DL Wet	ug/kg	0.182	0.181	0.192	0.181	0.178	0.187	0.188	0.183	0.186	0.19
o,p'-DDE	Res Wet	ug/kg	< 0.086	< 0.086	< 0.091	< 0.086	< 0.084	< 0.089	< 0.089	< 0.087	< 0.088	< 0.09
o,p'-DDE	DL Wet	ug/kg	0.086	0.086	0.091	0.086	0.084	0.089	0.089	0.087	0.088	0.09
o,p'-DDT	Res Wet	ug/kg	< 0.136	< 0.135	< 0.143	< 0.135	< 0.133	< 0.139	< 0.14	< 0.137	< 0.139	< 0.142
o,p'-DDT	DL Wet	ug/kg	0.136	0.135	0.143	0.135	0.133	0.139	0.14	0.137	0.139	0.142
oxychlordane	Res Wet	ug/kg	< 0.138	< 0.137	0.188	< 0.136	0.16	< 0.141	< 0.142	4.64	< 0.14	< 0.144
oxychlordane	DL Wet	ug/kg	0.138	0.137	0.145	0.136	0.134	0.141	0.142	0.138	0.14	0.144
p,p'-DDD	Res Wet	ug/kg	< 0.183	< 0.182	< 0.192	< 0.181	< 0.179	< 0.187	< 0.189	< 0.184	< 0.186	< 0.191
p,p'-DDD	DL Wet	ug/kg	0.183	0.182	0.192	0.181	0.179	0.187	0.189	0.184	0.186	0.191
p,p'-DDE	Res Wet	ug/kg	< 0.091	< 0.09	< 0.095	< 0.09	< 0.088	< 0.093	< 0.094	< 0.091	< 0.092	< 0.095
p,p'-DDE	DL Wet	ug/kg	0.091	0.09	0.095	0.09	0.088	0.093	0.094	0.091	0.092	0.095
p,p'-DDT	Res Wet	ug/kg	< 0.097	< 0.096	< 0.102	< 0.096	< 0.095	< 0.099	< 0.1	< 0.097	< 0.099	< 0.101
p,p'-DDT	DL Wet	ug/kg	0.097	0.096	0.102	0.096	0.095	0.099	0.1	0.097	0.099	0.101
pentachloro-anisole	Res Wet	ug/kg	< 0.1	< 0.099	< 0.105	< 0.099	< 0.097	< 0.102	< 0.103	< 0.1	< 0.101	< 0.104
pentachloro-anisole	DL Wet	ug/kg	0.1	0.099	0.105	0.099	0.097	0.102	0.103	0.1	0.101	0.104
toxaphene	Res Wet	ug/kg	< 9.33	< 9.28	< 9.8	< 9.24	< 9.11	< 9.56	< 9.63	< 9.36	< 9.51	< 9.75
toxaphene	DL Wet	ug/kg	9.33	9.28	9.8	9.24	9.11	9.56	9.63	9.36	9.51	9.75
trans-nonachlor	Res Wet	ug/kg	< 0.102	< 0.101	< 0.107	< 0.101	< 0.099	< 0.104	< 0.105	< 0.102	< 0.103	< 0.106
trans-nonachlor	DL Wet	ug/kg	0.102	0.101	0.107	0.101	0.099	0.104	0.105	0.102	0.103	0.106
chlorpyrifos	Res Wet	ug/kg	< 0.221	< 0.22	< 0.232	< 0.219	< 0.216	< 0.226	< 0.228	< 0.222	< 0.225	< 0.231
chlorpyrifos	DL Wet	ug/kg	0.221	0.22	0.232	0.219	0.216	0.226	0.228	0.222	0.225	0.231
1,2,3,4-Tetrachlorobenzene	Res Wet	ug/kg	< 0.102	< 0.101	< 0.107	< 0.101	< 0.1	< 0.104	< 0.105	< 0.102	< 0.104	< 0.107
1,2,3,4-Tetrachlorobenzene	DL Wet	ug/kg	0.102	0.101	0.107	0.101	0.1	0.104	0.105	0.102	0.104	0.107
1,2,4,5-Tetrachlorobenzene	Res Wet	ug/kg	< 0.203	< 0.202	< 0.213	< 0.201	< 0.198	< 0.208	< 0.209	< 0.203	< 0.206	< 0.212
1,2,4,5-Tetrachlorobenzene	DL Wet	ug/kg	0.203	0.202	0.213	0.201	0.198	0.208	0.209	0.203	0.206	0.212
Pentachlorobenzene	Res Wet	ug/kg	< 0.097	< 0.097	< 0.102	< 0.096	< 0.095	< 0.1	< 0.1	< 0.098	< 0.099	< 0.102
Pentachlorobenzene	DL Wet	ug/kg	0.097	0.097	0.102	0.096	0.095	0.1	0.1	0.098	0.099	0.102

Table 3 (concluded). Results of organochlorine pesticides analyses in ug/kg wet weight for muscle tissue samples from white-tailed deer (*Odocoileus virginianus*) from Caddo Lake National Wildlife Refuge during January-February, 2005.

(Note: F11M = Female #11, Muscle sample and M1M = Male #1, Muscle sample, etc; Res Wet = Result wet weight; DL Wet = Detection limit wet weight).

Analyte		Units	F11M	F12M	F13M	F14M	F15M	F16M	F17M	F18M	M1M	M2M
gamma BHC	Res Wet	ug/kg	< 0.128	< 0.128	< 0.134	< 0.13	< 0.127	< 0.128	< 0.134	< 0.129	< 0.132	< 0.133
gamma BHC	DL Wet	ug/kg	0.128	0.128	0.134	0.13	0.127	0.128	0.134	0.129	0.132	0.133
gamma chlordane	Res Wet	ug/kg	< 0.105	< 0.104	< 0.109	< 0.106	< 0.104	< 0.104	< 0.109	< 0.105	< 0.107	< 0.108
gamma chlordane	DL Wet	ug/kg	0.105	0.104	0.109	0.106	0.104	0.104	0.109	0.105	0.107	0.108
heptachlor epoxide	Res Wet	ug/kg	< 0.279	< 0.277	< 0.291	< 0.283	< 0.276	< 0.278	< 0.291	< 0.281	< 0.286	< 0.288
heptachlor epoxide	DL Wet	ug/kg	0.279	0.277	0.291	0.283	0.276	0.278	0.291	0.281	0.286	0.288
mirex	Res Wet	ug/kg	< 0.09	< 0.089	< 0.094	< 0.091	< 0.089	< 0.09	< 0.094	< 0.091	< 0.092	< 0.093
mirex	DL Wet	ug/kg	0.09	0.089	0.094	0.091	0.089	0.09	0.094	0.091	0.092	0.093
o,p'-DDD	Res Wet	ug/kg	< 0.18	< 0.179	< 0.188	< 0.183	< 0.178	< 0.18	< 0.188	< 0.181	< 0.185	< 0.186
o,p'-DDD	DL Wet	ug/kg	0.18	0.179	0.188	0.183	0.178	0.18	0.188	0.181	0.185	0.186
o,p'-DDE	Res Wet	ug/kg	< 0.085	< 0.085	< 0.089	< 0.087	< 0.085	< 0.085	< 0.089	< 0.086	< 0.088	< 0.088
o,p'-DDE	DL Wet	ug/kg	0.085	0.085	0.089	0.087	0.085	0.085	0.089	0.086	0.088	0.088
o,p'-DDT	Res Wet	ug/kg	< 0.134	< 0.134	< 0.14	< 0.136	< 0.133	< 0.134	< 0.14	< 0.135	< 0.138	< 0.139
o,p'-DDT	DL Wet	ug/kg	0.134	0.134	0.14	0.136	0.133	0.134	0.14	0.135	0.138	0.139
oxychlordane	Res Wet	ug/kg	< 0.136	< 0.135	< 0.142	< 0.138	< 0.135	< 0.136	< 0.142	< 0.137	< 0.14	< 0.141
oxychlordane	DL Wet	ug/kg	0.136	0.135	0.142	0.138	0.135	0.136	0.142	0.137	0.14	0.141
p,p'-DDD	Res Wet	ug/kg	< 0.181	< 0.18	< 0.189	< 0.183	< 0.179	< 0.18	< 0.189	< 0.182	< 0.185	< 0.187
p,p'-DDD	DL Wet	ug/kg	0.181	0.18	0.189	0.183	0.179	0.18	0.189	0.182	0.185	0.187
p,p'-DDE	Res Wet	ug/kg	< 0.089	< 0.089	< 0.093	< 0.091	< 0.089	< 0.089	< 0.093	< 0.09	< 0.092	< 0.093
p,p'-DDE	DL Wet	ug/kg	0.089	0.089	0.093	0.091	0.089	0.089	0.093	0.09	0.092	0.093
p,p'-DDT	Res Wet	ug/kg	< 0.096	< 0.095	< 0.1	< 0.097	< 0.095	< 0.095	< 0.1	< 0.096	< 0.098	< 0.099
p,p'-DDT	DL Wet	ug/kg	0.096	0.095	0.1	0.097	0.095	0.095	0.1	0.096	0.098	0.099
pentachloro-anisole	Res Wet	ug/kg	< 0.098	< 0.098	< 0.103	< 0.1	< 0.097	< 0.098	< 0.103	< 0.099	< 0.101	< 0.102
pentachloro-anisole	DL Wet	ug/kg	0.098	0.098	0.103	0.1	0.097	0.098	0.103	0.099	0.101	0.102
toxaphene	Res Wet	ug/kg	< 9.21	< 9.16	< 9.62	< 9.35	< 9.12	< 9.19	< 9.62	< 9.28	< 9.45	< 9.52
toxaphene	DL Wet	ug/kg	9.21	9.16	9.62	9.35	9.12	9.19	9.62	9.28	9.45	9.52
trans-nonachlor	Res Wet	ug/kg	< 0.1	< 0.1	< 0.105	< 0.102	< 0.099	< 0.1	< 0.105	< 0.101	< 0.103	< 0.104
trans-nonachlor	DL Wet	ug/kg	0.1	0.1	0.105	0.102	0.099	0.1	0.105	0.101	0.103	0.104
chlorpyrifos	Res Wet	ug/kg	< 0.218	< 0.217	< 0.228	< 0.221	< 0.216	< 0.218	< 0.228	< 0.22	< 0.224	< 0.225
chlorpyrifos	DL Wet	ug/kg	0.218	0.217	0.228	0.221	0.216	0.218	0.228	0.22	0.224	0.225
1,2,3,4-Tetrachlorobenzene	Res Wet	ug/kg	< 0.101	< 0.1	< 0.105	< 0.102	< 0.1	< 0.1	< 0.105	< 0.101	< 0.103	< 0.104
1,2,3,4-Tetrachlorobenzene	DL Wet	ug/kg	0.101	0.1	0.105	0.102	0.1	0.1	0.105	0.101	0.103	0.104
1,2,4,5-Tetrachlorobenzene	Res Wet	ug/kg	< 0.2	< 0.199	< 0.209	< 0.203	< 0.198	< 0.2	< 0.209	< 0.202	< 0.205	< 0.207
1,2,4,5-Tetrachlorobenzene	DL Wet	ug/kg	0.2	0.199	0.209	0.203	0.198	0.2	0.209	0.202	0.205	0.207
Pentachlorobenzene	Res Wet	ug/kg	< 0.096	< 0.096	< 0.1	< 0.097	< 0.095	< 0.096	< 0.1	< 0.097	< 0.099	< 0.099
Pentachlorobenzene	DL Wet	ug/kg	0.096	0.096	0.1	0.097	0.095	0.096	0.1	0.097	0.099	0.099

APPENDIX C
(HEALTH CONSULTATION REPORT)

Health Consultation

CONSUMPTION OF DEER TISSUE COLLECTED AT
CADDO LAKE NATIONAL WILDLIFE REFUGE

KARNACK, HARRISON COUNTY, TEXAS

EPA FACILITY ID: TX6213820529

JANUARY 24, 2006

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES
Public Health Service
Agency for Toxic Substances and Disease Registry
Division of Health Assessment and Consultation
Atlanta, Georgia 30333

Health Consultation: A Note of Explanation

An ATSDR health consultation is a verbal or written response from ATSDR to a specific request for information about health risks related to a specific site, a chemical release, or the presence of hazardous material. In order to prevent or mitigate exposures, a consultation may lead to specific actions, such as restricting use of or replacing water supplies; intensifying environmental sampling; restricting site access; or removing the contaminated material.

In addition, consultations may recommend additional public health actions, such as conducting health surveillance activities to evaluate exposure or trends in adverse health outcomes; conducting biological indicators of exposure studies to assess exposure; and providing health education for health care providers and community members. This concludes the health consultation process for this site, unless additional information is obtained by ATSDR which, in the Agency's opinion, indicates a need to revise or append the conclusions previously issued.

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HEALTH CONSULTATION

CONSUMPTION OF DEER TISSUE COLLECTED AT CADDO LAKE NATIONAL WILDLIFE REFUGE

KARNACK, HARRISON COUNTY, TEXAS

EPA FACILITY ID: TX6213820529

Prepared by:

Texas Department of State Health Services
Under a Cooperative Agreement with the
Agency for Toxic Substances and Disease Registry

Table of Contents

Purpose and Statement of Issues

The United States Fish and Wildlife Service (USFWS) asked the Texas Department of State Health Services (DSHS) to review analyses of muscle and liver samples collected from white-tailed deer (*Odocoileus virginianus*) on the Caddo Lake National Wildlife Refuge (NWR). The Caddo Lake NWR is located on part of the former Longhorn Army Ammunition Plant (LAAP), a National Priorities List (NPL) "Superfund" site. Metals, volatile and semi-volatile organic compounds, and explosives have been found in surface water, sediment, surface soil, and groundwater on the former LAAP site.

The USFWS would like to offer limited deer hunting on Caddo Lake NWR to the public. Prior to doing this, the agency asked DSHS for assistance in determining whether eating deer from Caddo Lake NWR could present a health risk to the public (Note: Appendix A lists abbreviations and acronyms used in this report).

Background

Site Description and History

Caddo Lake NWR is located in northeast Texas along the southwestern portion of Caddo Lake. The NWR is in Harrison County (population 62,110) [1] and is surrounded by the communities of Karnack (population 775) [2] and Uncertain (population 150) [1]. About 1,500 people live within a one-mile radius of the site [3].

Caddo Lake is the only naturally occurring lake in Texas. The western part of the lake is in Texas and the eastern portion is in Louisiana (Appendix B). The lake covers 26,810 acres of cypress swamp [4]. The Caddo Lake habitat supports the largest populations of several duck species and the most diverse fish fauna in Texas [5]. Studies of the refuge have listed up to 224 species of birds, 22 species of amphibians, 46 species of reptiles, and 93 fish species [3]. On November 2, 1995, the DSHS issued a fish consumption advisory (ADV-12) due to elevated levels of mercury in largemouth bass (*Micropterus salmoides*) and freshwater drum (*Aplodinotus grunniens*) from Caddo Lake. The advisory states that eating largemouth bass or freshwater drum should be limited to minimize potential exposure to mercury [6].

Caddo Lake NWR is located on property of the former LAAP. The LAAP was an 8,943 acre U.S. Department of Defense (DOD) facility that operated intermittently from 1942 to 1997. The LAAP produced 2,4,6-trinitrotoluene (TNT), pyrotechnic ammunition, rocket motors, and plastic explosives [7]. At peak production, the facility had over 2,200 employees. Metals, volatile and semi-volatile organic compounds, and explosives were previously found in on-site surface water, sediment, surface soil, and groundwater [7]. The LAAP was placed on the U.S. Environmental Protection Agency's (EPA) NPL on August 30, 1990. The Agency for Toxic Substances and Disease Registry (ATSDR) reviewed environmental data for the site and prepared a public health assessment. In July 1999, ATSDR concluded that the LAAP site posed no apparent public health hazard because people were not likely to come into contact with site-related contaminants or because institutional controls were sufficient to protect human health [7].

Administrative control of about 5,000 acres of the LAAP site was granted to USFWS as the Caddo Lake NWR on May 5, 2004. The DOD retains control of the remaining LAAP property until remediation is completed at several areas on the site [8]. The USFWS plans to eventually allow 300 individuals to hunt deer on the refuge property annually. The estimated hunter success rate is 30%, and about 100 deer are expected to be harvested per season. Each hunter will be allowed to harvest two deer (either sex) per season. Seasonal harvest limits may change depending on the total deer population on the refuge [9].

Environmental Sampling

The USFWS collected muscle and liver samples from 20 white-tailed deer (*Odocoileus virginianus*) during January - February 2005. Eighteen female deer (does) and two male deer (bucks) were collected from the Caddo Lake NWR area and the remaining LAAP property. They ranged from 1½ to 6½ years of age. The sampling ratio of does to bucks (9:1) was similar to harvest results of past seasons when the U.S. Army allowed deer hunting on the LAAP facility by a limited number of personnel [10].

Average live weight for the 18 does was 45-50 kilograms (kg) or approximately 100-110 pounds (lbs). Live weights of the bucks were 59 and 64 kg (130 and 140 lbs) [11]. For muscle tissue, samples of backstrap (loin) and hind quarters were pooled as one muscle tissue sample per deer. Liver samples were also collected from each deer [10].

Samples were sent to TDI - Brooks International, Inc., College Station, Texas for pesticide analyses and to Laboratory and Environmental Testing, Inc., Columbia, Missouri for metals analyses [12]. The DSHS and ATSDR relied on information provided in the referenced documents and assumed adequate quality assurance/quality control (QA/QC) procedures were followed with regard to data collection, chain-of-custody, laboratory procedures, and data reporting. Procedural blank analyses were acceptable, analyses of duplicate samples were within normal limits, and limits of detection were within laboratory contract requirements.

Data on background concentrations of metals and pesticides in white-tailed deer from other areas around Caddo Lake were not collected; thus, it was not possible to compare the risks associated with eating deer from the area of concern to those associated with eating deer from other areas.

Discussion

Health-based screening values specifically for deer tissues were not available. Therefore, for all contaminants except lead, we used ATSDR minimal risk levels (MRLs)[1] and EPA reference doses (RfDs)[2] to develop non-cancer screening values. We used standard assumptions for ingestion rate (4 ounces (oz) per week for children; 8 oz per week for adults) and body weight (15 kg for children; 70 kg for adults). For lead, we used the ATSDR "Framework to Guide

[1] An MRL is a contaminant specific exposure dose below those which might cause adverse health effects in the people most sensitive to such chemical-induced effects. MRLs generally are based on the most sensitive chemical-induced end point considered to be of relevance to humans.

[2] An RfD is an estimate, with uncertainty spanning perhaps an order of magnitude, of a daily exposure to the human population (including sensitive groups) that is likely to be without appreciable risk of deleterious effects during a lifetime.

Public Health Assessment Decisions at Lead Sites" to estimate probable increases in blood lead levels associated with tissue consumption [13].

For contaminants classified as human carcinogens, probable human carcinogens, or possible human carcinogens, we used EPA chemical specific cancer slope factors (CSF) and an estimated excess lifetime cancer risk of one-in-one-million persons exposed to develop cancer-screening values. Arsenic and polychlorinated biphenyls (PCBs) were the only contaminants for which CSFs were available. Concentrations of these contaminants in the liver and muscle samples were below detection limits.

Exceeding either a non-cancer or a cancer screening value does not necessarily mean that the contaminant will cause harm; however, it does suggest that potential exposure to the contaminant warrants further consideration. Factors that influence whether exposure to a contaminant could or would result in adverse health effects include: how much of the contaminant an individual is exposed to, how often and how long they are exposed, and the manner in which the contaminant enters or contacts the body. Once exposure occurs, characteristics such as age, sex, nutritional status, genetics, lifestyle, and health status all may influence how well the individual absorbs, distributes, metabolizes, and excretes the contaminant.

We assessed the public health significance of contaminants that exceeded screening values by reviewing and integrating relevant toxicological information with plausible exposure scenarios. We used a weight-of-evidence approach to determine the public health significance of the contaminants that exceeded the screening values.

Public Health Implications

For many samples, contaminant concentrations were below detection limits. We eliminated from further consideration those contaminants which were below detection limits in all samples if the respective detection limit was less than the health-based screening value. For muscle samples, this eliminated arsenic, beryllium, cadmium, mercury, molybdenum, vanadium, and all pesticides tested, except mirex and oxychlordane, as contaminants of concern. For liver samples, this eliminated arsenic, beryllium, mercury, vanadium, and most organochlorine pesticides.

For the remaining contaminants found in one or more tissue samples, we compared the maximum levels found to the respective calculated health-based screening values. For muscle samples, all contaminants were below their respective health-based screening values (Table C-1). For liver samples, all but three contaminants were below their respective health-based screening values (Table C-2). Cadmium, copper and selenium exceeded their respective health-based screening values in liver samples (Table C-3). In the absence of background samples for comparison, we cannot determine with any degree of certainty whether the presence of these contaminants in deer liver poses an excess health risk.

For lead, we used the maximum concentration found in muscle tissue, 5.2 milligrams per kilogram (mg/kg), and ATSDR's "Framework to Guide Public Health Assessment Decisions at Lead Sites" to estimate probable increases in blood lead levels associated with tissue consumption [13]. We estimated that increased blood lead levels in children would be <1.4

µg/dL (micrograms per deciliter) and < 0.4 µg/dL in adults. Based on these findings, lead was eliminated as a contaminant of concern.

Throughout the world, studies have been conducted to determine the risk to hunters of eating wild game contaminated with heavy metals. One metal of particular concern has been cadmium. In wild game, cadmium levels may be elevated in liver and kidney tissues but low in muscle tissues [14]. Because of this, health advisories on the consumption of liver and kidney tissues have been issued in many areas, including Maine and New Hampshire, as well as in Canada (Ontario, New Quebec, and New Brunswick) [15].

Child Health Considerations

In communities faced with air, water, or food contamination, children could be at greater risk than are adults from certain kinds of exposure to hazardous substances. A child's lower body weight and higher intake rate results in a greater dose of hazardous substance per unit of body weight. Sufficient exposure levels during critical growth stages can sustain permanent damage to the developing body systems of children. Children are dependent on adults for access to housing, for access to medical care, and for risk identification. Thus, adults need as much information as possible to make informed decisions regarding their children's health.

Health risks to children consuming white-tailed deer collected at Caddo Lake NWR were evaluated in this health consultation. Assuming levels of contaminants in samples of deer tissue collected at Caddo Lake NWR are representative of concentrations to which children could be exposed; children may consume deer muscle tissue collected from the site without appreciable risk of adverse health affects.

Conclusions

The conclusions reached in this report only apply to deer taken from the Caddo Lake National Wildlife Refuge (NWR). Based on the information available for this report:

1. Using a plausible exposure scenario, none of the contaminants exceeded their respective health-based screening values in muscle. We therefore conclude that if deer hunting is allowed, then eating muscle from deer taken from Caddo Lake NWR would pose **no apparent public health hazard**.

2. Selenium exceeded the health-based screening value in only one liver sample. We think it unlikely that people eating deer liver would be exposed to selenium at levels of concern on a regular basis. Given the available information, we conclude that if deer hunting is allowed, then exposure to selenium in deer liver taken from Caddo Lake NWR would pose **no apparent public health hazard**.

3. Cadmium and copper were found at levels above their respective health-based screening values in all liver samples. Because deer hunting is presently not allowed on the Caddo Lake NWR, these levels currently pose **no public health hazard**. If hunting is allowed in the future, and livers from the Caddo Lake NWR deer are not consumed, then no apparent public health hazard would likely to exist.

Recommendations

1. If deer hunting is allowed on Caddo Lake NWR, the USFWS should retain and dispose of the livers. Hunters should be informed that eating the liver of deer taken from the Caddo Lake NWR may result in adverse health effects due to elevated levels of copper and cadmium.

2. Given the previous land use for this refuge, collecting data from a suitable background area could help determine if the contaminants found in the liver pose an excess risk.

3. If other types of hunting (squirrel, turkey, and wild hog) are considered for Caddo Lake NWR, the USFWS should sample these biota to help determine whether environmental contaminant levels are a concern.

Public Health Action Plan

Actions Completed

1. The USFWS collected and analyzed tissue (muscle and liver) samples from 20 white-tailed deer inhabiting the Caddo Lake NWR.

2. The DSHS and ATSDR evaluated contaminant data from deer collected at Caddo Lake NWR.

Actions Planned

1. Based on the conclusions of this Public Health Consultation, the USFWS will decide whether to allow public hunting on the Caddo Lake NWR.

2. This health consultation will be provided to the public, the local government, and state and federal health/environmental agencies.

3. Additional wild game sampling data from Caddo Lake NWR will be evaluated by DSHS and ATSDR as it becomes available.

Authors, Technical Advisors, and Organizations

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Certification

This public health consultation was prepared by the Texas Department of State Health Services (DSHS) under a cooperative agreement with the Agency for Toxic Substances and Disease Registry (ATSDR). It is in accordance with approved methodology and procedures existing at the time this public health consultation was initiated. Editorial review was completed by the Cooperative Agreement partner.

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Technical Project Officer, CAT, CAPEB, DHAC, ATSDR

The Division of Health Assessment and Consultation, ATSDR, has reviewed this public health consultation and concurs with its findings.

Alan W. Yarbrough

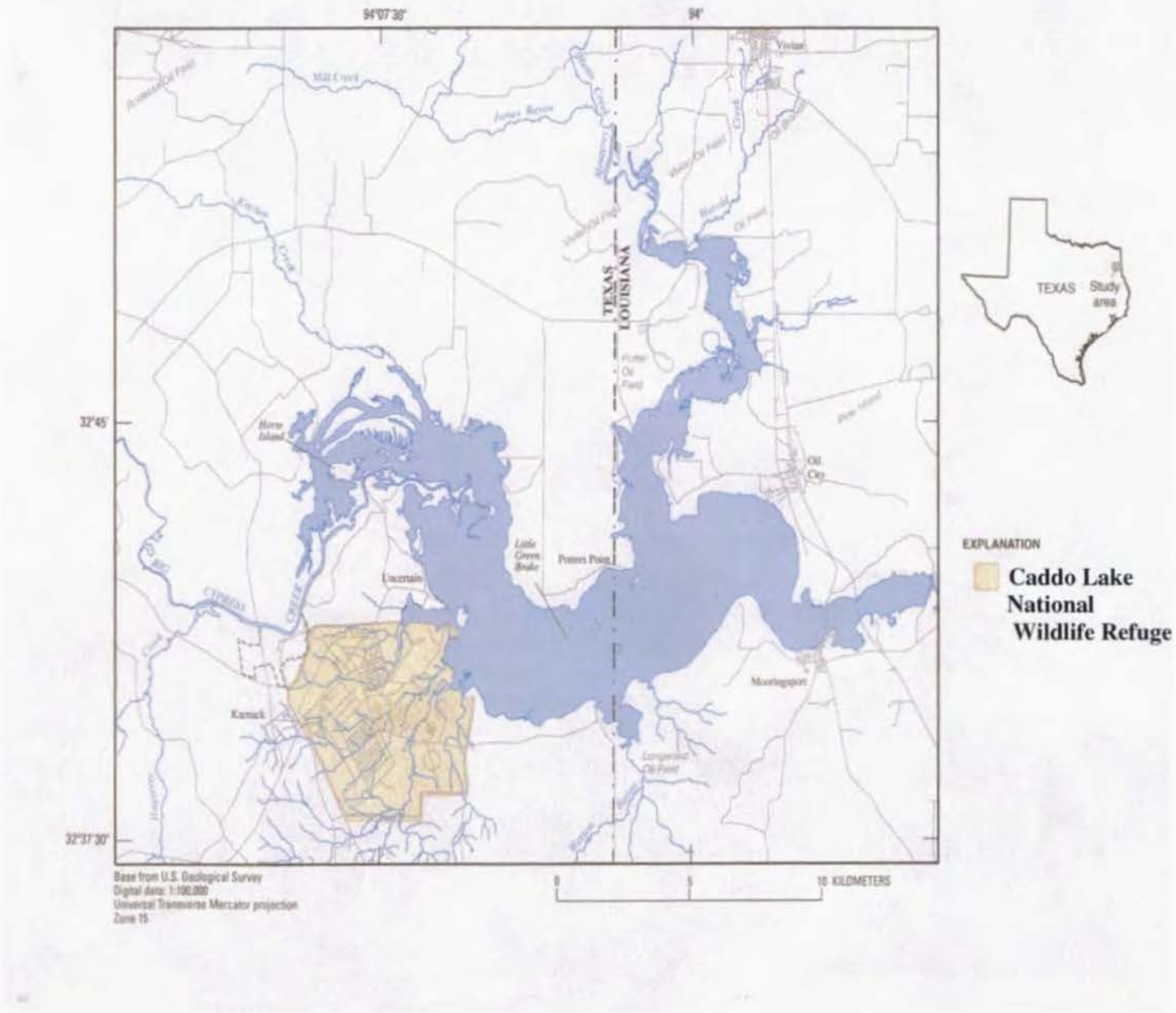
Team Lead, CAT, CAPEB, ATSDR

Appendix A - Acronyms and Abbreviations

ATSDR	Agency for Toxic Substances and Disease Registry
BHC	benzenehexachloride
CSF	Cancer Slope Factor
DDE	dichlorodiphenyldichloroethylene
DDT	dichlorodiphenyltrichloroethane
DL	detection limit
DOD	Department of Defense
DSHS	Department of State Health Services
EPA	Environmental Protection Agency
kg	kilogram
LAAP	Longhorn Army Ammunition Plant
lbs	pounds
MRL	Minimal Risk Level
mg/kg	milligrams per kilogram
µg/dL	micrograms per deciliter
NA	not applicable
NPL	National Priorities List
NWR	National Wildlife Refuge
oz	ounces
PCB	polychlorinated biphenyls
QA/QC	Quality Assurance/Quality Control
RfD	Reference Dose
TNT	trinitrotoluene
USFWS	United States Fish and Wildlife Service

Appendix B - Map

Caddo Lake and surrounding area, including Caddo Lake National Wildlife Refuge (NWR).



Appendix C - Tables

Table C-1. Summary data for contaminants found in one or more **muscle** samples (n=20) that did not exceed health-based screening values. No contaminants of concern were identified.

Contaminant	Concentration Range (mg/kg) Muscle[†]	Screening Value (mg/kg)[‡] Child	Screening Value (mg/kg)[‡] Adult
Aluminum	<DL-12	2000	4000
Barium	<DL-0.76	200	400
Boron	<DL-2	9	20
Chromium	<DL-1.5	1000	3000
Copper	5.7-8.6	9	22
Lead	<DL-5.2	NA	NA
Manganese	0.5-0.8	50	100
Nickel	<DL-2.1	20	40
Selenium	0.6-1.4	5	10
Strontium	<DL-0.68	2000	4000
Zinc	47-74.4	300	600
Mirex	<DL-0.000212	0.7	2
Oxychlordane	<DL-0.004640	0.6	1

[†] DL = detection limit of the analytical instrument.

[‡] Derived from the MRL or RfD for non-cancerous adverse health effects using standard assumptions of body weight for children (15 kg or 33 lbs) and adults (70 kg or 154 lbs). An intake rate of 0.113 kg per week (0.25 lb) for children and 0.227 kg per week (0.5) for adults and an exposure frequency of one day per week for six months out of the year were used in this analysis.

NA = Not applicable, no MRL or RfD was available for lead, thus a screening value could not be determined. We estimated a < 1.4 µg/dL increase in blood lead levels for children and < 0.4 µg/dL blood lead increase for adults, thus lead is not a contaminant of concern.

Table C-2. Summary data for contaminants found in one or more **liver** samples (n=18[#]) that did not exceed health-based screening values.

Contaminant	Concentration Range (mg/kg) Liver[†]	Screening Value (mg/kg)[‡] Child	Screening Value (mg/kg)[‡] Adult
Aluminum	<DL-2	2000	4000
Barium	<DL-1	200	400
Boron	<DL-3	9	20
Chromium	<DL-1	1000	3000
Lead	<DL-1.9	NA	NA
Manganese	11-19	50	100
Molybdenum	<DL-2	5	10
Nickel	<DL-3	20	40
Strontium	<DL-0.62	2000	4000
Zinc	86.9-162	300	600
γ-BHC[*]	<DL-0.003720	0.009	0.02
o,p'-DDE[*]	<DL-0.001770	0.5	1
Oxychlordane	0.000964-0.022300	0.6	1
Total-BHC	<DL-0.003720	0.009	0.02
Total-DDTs[*]	<DL-0.001770	0.5	1

[#] During one of the sampling events, the livers were not collected from two does, thus only 18 liver samples were analyzed [16].

[†] DL = detection limit of the analytical instrument.

[‡] Derived from the MRL or RfD for non-cancerous adverse health effects using standard assumptions of body weight for children (15 kg or 33 lbs) and adults (70 kg or 154 lbs). An intake rate of 0.113 kg per week (0.25 lb) for children and 0.227 kg per week (0.5) for adults and an exposure frequency of one day per week for six months out of the year were used in this analysis.

NA = Not applicable, no MRL or RfD was available for lead, thus a screening value could not be determined. We estimated a < 1.4 µg/dL increase in blood lead levels for children and < 0.4 µg/dL blood lead increase for adults, thus lead is not a contaminant of concern.

[*] BHC=benzenehexachloride, DDE=dichlorodiphenyldichloroethylene, and DDT=dichlorodiphenyltrichloroethane.

Table C-3. Summary data for contaminants of concern in white-tailed deer **liver** tissues taken from Caddo Lake NWR. There were no contaminants of concern in **muscle** tissues.

Contaminant	Concentration Range (mg/kg)	Screening Value (mg/kg)[‡] Child	Adult	# Samples Exceeded[§] Child	Adult
Cadmium	0.34 – 7.7	0.2		18	
		0.4		17	
Copper	22 – 949	9		18	
		22		18	
Selenium	0.89 – 20.10	5		1	
		10		1	

[‡] Derived from the MRL or RfD for non-cancerous adverse health effects using standard assumptions of body weight for children (15 kg or 33 lbs) and adults (70 kg or 154 lbs). An intake rate of 0.113 kg per week (0.25 lb) for children and 0.227 kg per week (0.5) for adults and an exposure frequency of one day per week for six months out of the year were used in this analysis.

[§] Number of samples that exceeded the screening value.

APPENDIX D
(TEXAS PARKS & WILDLIFE DEPARTMENT CHRONIC WASTING DISEASE ANALYTICAL REULTS)

Texas Veterinary Medical Diagnostic Laboratory System

Final Report

P.O. Drawer 3040, College Station, TX 77841-3040
Phone: (979)845-3414 Fax: (979)845-1794 http://tvmdlweb.tamu.edu/

Owner's Name: (409)384-6894
Gary Calkins
1342 S. Wheeler
JASPER, TX 75951

(512)389-4581
Veterinarian/Submitter: **Account#: 23683**
Texas Parks and Wildlife Dept-CWD
Attention: Clayton Wolf
111 East Travis Ste 201
La Grange, TX 78945

Date specimens received: 4/6/05

Preliminary reports:
Phone reports:
Final report: Mail 4/28/05

Species: Exotic, multiple animals/specimens, see lab results for animal/specimen ID's

Tests Requested: CWD IHC

Specimens Submitted: 26 brain stems

Clinical History: Harrison County. /bw

Clinical Diagnosis:

Previous Cases:
Treatment:

Conclusion/Interpretation of Lab Findings:
Laboratory results as listed.

LABORATORY TEST STATUS:	Ordered	Current Status
Immunohistochemistry CWD test, TPW(26) (H)	4/14/05	Completed 4/28/05

Histopathology

Immunohistochemistry CWD test, TPW
Date Completed: 4/28/05 **Pathologist:** Eugster, Konrad

Animal ID	# Tests	County	Breed	OBEX
CWD046CDM7	1	Harrison	White Tail Deer	Not detected
CWD046CDM10	1	Harrison	White Tail Deer	Not detected
CWD046CDM27	1	Harrison	White Tail Deer	Not detected
CWD046CDM39	1	Harrison	White Tail Deer	Not detected
CWD046CDM40	1	Harrison	White Tail Deer	Not detected
CWD046CDM41	1	Harrison	White Tail Deer	Not detected
CWD046CDM69	1	Harrison	White Tail Deer	Not detected
CWD046CDM86	1	Harrison	White Tail Deer	Not detected
CWD046CDM88	1	Harrison	White Tail Deer	Not detected
CWD046CDM89	1	Harrison	White Tail Deer	Not detected
CWD046CDM94	1	Harrison	White Tail Deer	Not detected
CWD046CDM96	1	Harrison	White Tail Deer	Not detected
CWD046CDM105	1	Harrison	White Tail Deer	Not detected
CWD046CDM117	1	Harrison	White Tail Deer	Not detected
CWD046CDM118	1	Harrison	White Tail Deer	Not detected
CWD046KJF45	1	Harrison	White Tail Deer	Not detected
CWD046KJF80	1	Harrison	White Tail Deer	Not detected
CWD046KJF92	0	Harrison	White Tail Deer	See comment
CWD046KJF93	1	Harrison	White Tail Deer	Not detected
CWD046KJF95	1	Harrison	White Tail Deer	Not detected
CWD046KJF108	1	Harrison	White Tail Deer	Not detected
CWD046CLNR1	1	Harrison	White Tail Deer	Not detected
CWD046CLNR2	1	Harrison	White Tail Deer	Not detected
CWD046CLNR3	0	Harrison	White Tail Deer	See comment
CWD046CLNR4	1	Harrison	White Tail Deer	Not detected
CWD046CLNR5	1	Harrison	White Tail Deer	Not detected

COMMENT:
NOT DETECTED - Specifically protease-resistant prion protein (PrP-res) was not detected in the sample examined (see disclaimer below).

CWD046KJF80 & CWD046CLNR3: No test was performed as either a sample was not received or the sample received was determined to be unsuitable for testing (some examples include advanced autolysis, freeze/thaw artifact, or fragmentation/compression, etc). There is no charge as no test was performed.

DISCLAIMER: These immunohistochemistry tests utilize a USDA

approved protocol to test for the presence of protease-resistant prion protein (PrP-res), the presence of which is diagnostic for chronic wasting disease. However, it is possible to have PrP-res present in tissues at levels below the sensitivity of this test. The test is applied to one or more tissue sites that have been shown to consistently have PrP-res in diseased animals. However, it should be noted that PrP-res may be present in tissues other than those that were examined.

/pjt