TX- Contamination in Aquatic and Sediment at Trinity River National Wildlife Refuge, Texas

Project ID: 200220005-2N50

EXECUTIVE SUMMARY

The Trinity River National Wildlife Refuge (Refuge) was established in January 1994, and subsequently Champion Lake was acquired as part of the Refuge. This 700 acre lake receives water from the Trinity River. The river drains the moderately industrialized Dallas - Fort Worth, Texas metroplex, and flows southward for 250 miles (402 km) through several small urban areas and ranch land. Prior contaminant investigations (Irwin 1988, Ulery and Brown 1994, and Moring 1997) upstream of the refuge indicated several organochlorine compounds and heavy metals which may pose a hazard to fish eating birds and human consumption of aquatic resources from Champion Lake.

On request of the Refuge Manager, in June, 2002, personnel from the U.S. Fish and Wildlife Service (Service) Ecological Services Field Office in Clear lake, TX initiated an investigation to determine levels of contaminants in sediments and fish inhabiting Champion Lake on the Refuge and to assess risks to humans from consuming fish from Champion Lake. To accomplish this, biological samples, consisting of fillet and whole body samples from largemouth bass, catfish and crappie, edible portions of blue crab, and sediment samples were collected from Champion Lake and analyzed for 29 organochlorine compounds and 19 heavy metals. The sediment samples were also analyzed for 44 polycyclic aromatic hydrocarbons (PAHs).

Results of the PAH analyses indicated no elevated concentrations in the sediments. Additionally, polychlorinated biphenyls (PCB-TOTAL) and 22 other organochlorine compounds either the whole body or the skinless fish fillet samples were below human health risk levels (FDA 1992). Blue crabs had very low levels of only seven organochlorines, and none were at ecological or human health concern concentrations.

Nineteen heavy metals were included in the analysis of sediment, bass, catfish, crappie and blue crabs. Several of these heavy metals such as arsenic, cadmium, chromium, nickel and lead are known or suspected carcinogens, however none were detected in the sediment at levels of

ecological concern. Mercury was not detected in the sediment samples, but was detected in all of the fish and crab samples. Mercury concentrations were elevated in the edible portions of all three fish species.

Results from the preliminary human health risk assessment indicate that fish from Champion Lake contain elevated levels of mercury. Mercury in fish tissue may represent a potential health concern to fisherman and others consuming fish from Champion Lake. The Service recommends taking a conservative approach based on these preliminary findings and posting a fish consumption advisory for mercury for bass, catfish and crappie suggesting that fishermen limit consumption to no more than 3 8-ounce fish meals per month. In addition, to more thoroughly assess risk, a creel survey be conducted to examine species consumed and frequency of consumption. Further studies should be conducted involving the collection of fish, crawfish, avian eggs, and water samples to determine the extent and availability of mercury to fish and other wildlife resources at the Refuge.

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U.S. Fish and Wildlife Service
Region 2
Contaminants Program

Contamination in Aquatics and Sediment at Trinity River National Wildlife Refuge, Texas

Prepared by
Environmental Contaminants Program
Region 2

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Project ID: 200220005-2N50

EXECUTIVE SUMMARY

Located northeast of Houston in Liberty County, Texas, the Trinity River National Wildlife Refuge (Refuge) was established in January 1994, and soon thereafter Champion Lake was acquired as part of the Refuge. This 700 acre lake receives water from the Trinity River. The river drains the moderately industrialized Dallas - Fort Worth, Texas metroplex, and flows southward for 250 miles (402 km) through several small urban areas and ranch land. Prior contaminant investigations of sites upstream of the refuge indicated several organochlorine compounds and heavy metals which may pose a hazard to fish eating birds and human consumption of aquatic resources from Champion Lake (Irwin 1988, Ulery and Brown 1994, and Moring 1997).

On request of the Refuge Manager, in June, 2002, personnel from the U.S. Fish and Wildlife Service (Service) Ecological Services Field Office in Clear Lake, TX initiated an investigation to determine levels of contaminants in sediments and fish inhabiting Champion Lake on the Refuge and to assess risks to people that eat fish from Champion Lake. To accomplish this, tissue samples, consisting of fillet and whole body samples from largemouth bass (*Micropterus salmoides*), channel catfish (*Ictalurus punctatus*), blue catfish (*Ictalurus furcatus*) and white crappie (*Pomoxis annularis*), edible portions of blue crab (*Callinectes sapidus*), and sediment samples were collected from Champion Lake and analyzed for 29 organochlorine compounds and 19 heavy metals. Additionally, sediment samples were also analyzed for 44 polycyclic aromatic hydrocarbons (PAHs).

PCB, mercury, and dieldrin fish tissue concentrations showed exceedences of screening values. Exceedences of the recreational fisher PCB screening value (0.02 ppm) were found in four out of five of the bass fillet samples, three out of five of the catfish fillet samples, and two out of five of the crappie fillet samples. All the remaining samples exceed the subsistence fisher PCB screening value (0.00245 ppm).

Exceedences of the recreational fisher mercury screening value (0.4 ppm) were found in one of the five crab samples and one of the five catfish fillet samples. All remaining samples exceed the subsistence fisher mercury screening value (0.049 ppm).

The process detailed in USEPA's Guidance for Assessing Chemical Contamination Data for Use in Fish Advisories was followed in determining fish consumption recommendations. Results indicate that fish and crab from Champion Lake contain elevated levels of mercury and PCBs. The Service recommends taking a conservative approach based on these preliminary findings and posting a fish consumption guideline for blue crab, bass, catfish and crappie. Utilizing EPA's default assumptions it is recommended that individuals should restrict their consumption of crab to one eight ounce meal per month, largemouth bass to three eight ounce meals per month, and catfish and crappie to two eight ounce meals per mouth.

These data are based on preliminary sampling. To more thoroughly assess risk, a creel survey should be conducted to examine species consumed and frequency of consumption. Further studies should be conducted involving the collection of fish, crawfish, avian eggs, and water samples to determine the extent and availability of mercury and PCBs to fish and other wildlife resources at the Refuge.

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INTRODUCTION

The Trinity River National Wildlife Refuge (Refuge) was established in January, 1994, with the first tract comprising 4,400 acres. By the year 2001, 15 tracts, that now comprise nearly 11,000 acres in the Trinity River flood plain, had been acquired under the authority of the Emergency Wetlands Resources Act of 1986. The Refuge is about 45 miles (72.4 km) east of Houston, Texas, and although located in a very rural setting of Liberty County, it is within 65 miles (105 km) of over 4,000,000 people.

The primary purpose of establishing this Refuge was to protect a remnant of the bottomland hardwood forest ecosystem along the Trinity River. It is a priority-one bottomland site identified for protection in the Texas Bottomland Protection Plan. Additionally, the Refuge is located within the Lower Mississippi Joint Venture Project Area of the North American Waterfowl Management Plan and, as such, is valuable habitat for a diversity of waterfowl species. This type of habitat is used during migration or as nesting habitat by nearly 50 percent of the neotropical migratory bird species listed by the U. S. Fish and Wildlife Service (Service). Bottomland hardwood forests also support abundant populations of white-tailed deer, squirrels, numerous other furbearers, freshwater turtles, alligators, snakes, river otters and the federally listed bald eagle.

The recently purchased 700 acre Champion Lake addition to the Trinity River NWR is connected to the Trinity River and is now open to public fishing. With the headwaters of the river beginning to the west of the moderately industrialized Dallas-Fort Worth, Texas area and traveling south for some 250 miles (402 km) through heavily modified agricultural and ranching wetland habitats, it was hypothesized that there may be some level of contamination in bottom sediments, and in the fish and crustacea inhabiting Champion Lake. Although not fully surveyed, the Refuge is known to contain more than 610 plant species and more than 400 vertebrate species, including 46 species of fish. A list of fish and crustacea commonly found in Champion Lake is presented in Table 1. Within the Champion Lake tract, several active rookeries provide an important nesting area for egrets, herons, cormorants, anhingas and other colonial nesting species. Additionally, bald eagles frequent the lake, and as a predatory species, could be accumulating contaminants present in fish.

The Refuge Manager has observed a number of fish with lesions and tumors during collections at Champion Lake (Stuart Marcus, personal communication). The Texas Commission on Environmental Quality (TCEQ), formerly the Texas Natural Resource Conservation Commission (TNRCC), has designated this section of the Trinity River as Segment 0802 which extends from 1.9 miles downstream of US 90 northward through much of the Refuge to Livingston Dam. Segment 0802 is classified as water quality impaired due to water quality standards violations. Its designated water uses include contact recreation, high aquatic life use and public water supply. However, dissolved cadmium concentrations exceed the chronic aquatic life criterion in the lower 25 miles (40.2 km), therefore the aquatic life use is not supported (TNRCC 1996). Fecal coliform bacteria levels and orthophosphorus concentrations are also exceeded.

The U. S. Geological Survey (USGS) published results that indicate the dichloro-diphenyl-trichloroethane (DDT) metabolite p,p'- dichloro-diphenyl-dichloroethylene (DDE) was present in common carp tissue (73 ppb wet weight) in the Trinity River at the Romayor, Texas site, just

upstream of the Refuge. At this same location, PCBs (100 ppb wet weight) were found in carp tissue (Moring 1997). In addition, at the same sampling location, a number of pesticides and herbicides have been detected, including diazinon in ~55% of water samples, 2,4-D chlorophenoxy in ~60% of water samples, 2,4,5-T chlorophenoxy in ~60% of water samples and dieldrin in ~10% of water samples (Ulery and Brown 1994).

The purpose of this contaminant investigation was to assure the public that fish and crustacea caught in Champion Lake are safe for human consumption, and to assess potential contamination of fish eating birds. In addition, an evaluation of sediments was warranted to determine if unsafe levels of contaminants are found in this ecosystem.

Table 1. Fish and crustacea species common to Champion Lake, Trinity National Wildlife Refuge.

FISH			
Common Name	**Scientific Name**	**Common Name**	**Scientific Name**
Alligator Gar	*Lepisosteus spatula*	Longear Sunfish	*Lepomis megalotis*
Black Bullhead	*Ameiurus melas*	Mosquitofish	*Gambusia affinis*
Black Crappie	*Pomoxis nigromaculatus*	Pirate Perch	*Aphredoderus sayanus*
Blue Catfish	*Ictalurus furcatus*	Pugnose Minnow	*Opsopeodus emiliae*
Bluegill	*Lepomis macrochirus*	Redear Sunfish	*Lepomis microlophus*
Bowfin	*Amia calva*	Smallmouth Buffalo	*Ictiobus bubalus*
Channel Catfish	*Ictalurus punctatus*	Spotted Gar	*Lepisosteus oculatus*
Common Carp	*Cyprinus carpio*	Spotted Sunfish	*Lepomis punctatus*
Dollar Sunfish	*Lepomis marginatus*	Stirped Mullet	*Mugil cephalus*
Freshwater Drum	*Aplodinotus grunniens*	Threadfin Shad	*Dorosoma petenense*
Gizzard Shad	*Dorosoma cepedianum*	Warmouth	*Lepomis gulosus*
Golden Shiner	*Notemigonus chrysoleucas*	White Crappie	*Pomoxis annularis*
Inland Silverside	*Menidia beryllina*	Yellow Bass	*Morone mississippiensis*
Largemouth Bass	*Micropterus salmoides*	Yellow Bullhead	*Ameiurus natalis*

CRUSTACEA			
Common Name	**Scientific Name**	**Common Name**	**Scientific Name**
Blue Crab	*Callinectes sapidus*	Glass Shrimp	*Palaeomonetes sp.*
Crawfish	*Procambarus sp.*		

OBJECTIVES

The objectives of this study, as stipulated in the project proposal, were to determine (1) levels of contaminants in fish, crustacea, and sediment, and (2) risks to wildlife and humans from these potentially contaminated resources in Champion Lake.

STUDY AREA

General Setting

Prior to the Service's acquisition of Champion Lake into the Trinity River NWR complex in 1994, a corporate hunting club operated at this site for the exclusive use of its members. Outdoor and wildlife-oriented recreational activities available to the public at Champion Lake include bird watching, hunting and fishing, photography and general nature observation. The

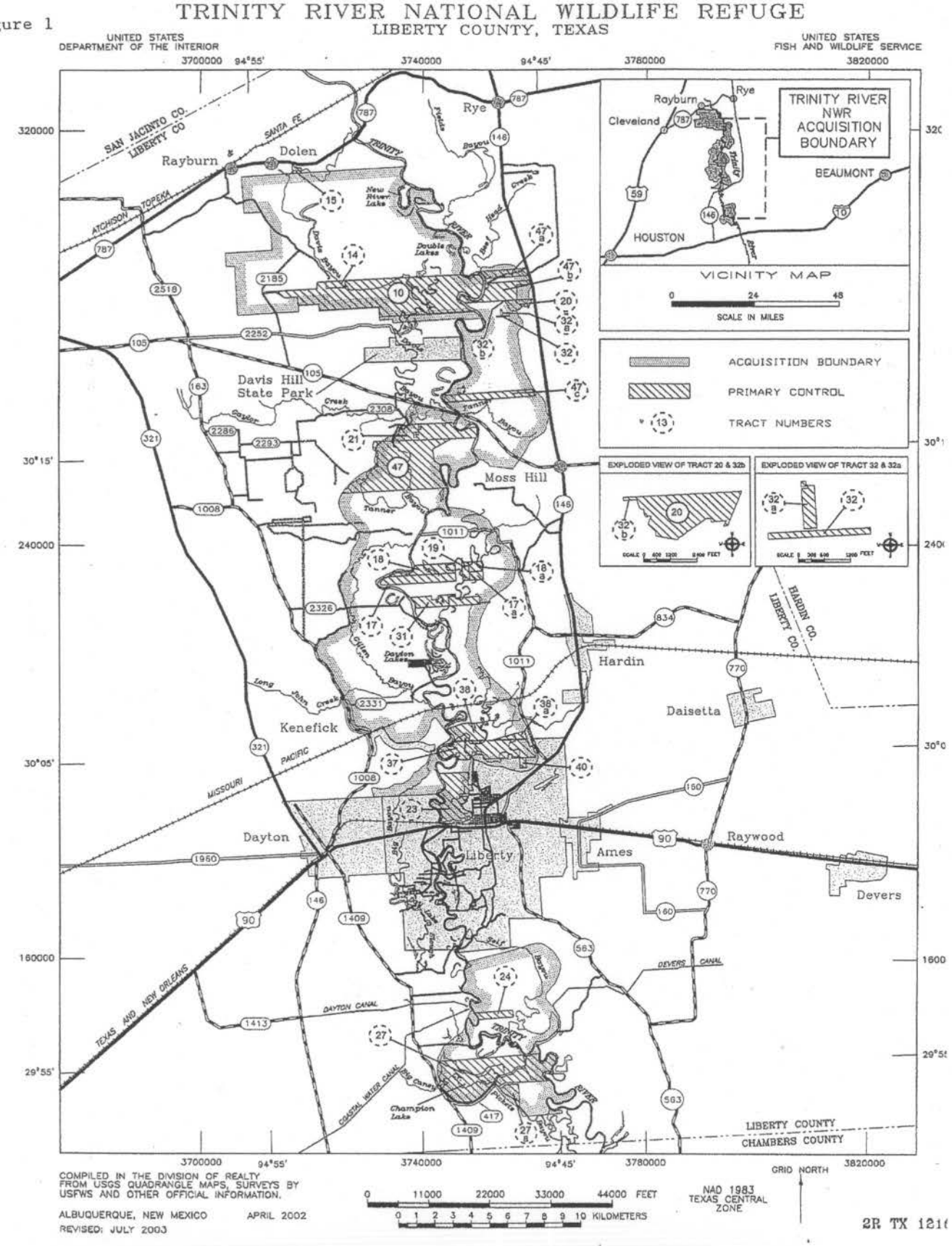
Trinity River NWR and surrounding area attract numerous species of waterfowl and neotropical migrants by providing important wintering, breeding and resting habitat during migration. However, fishing is the most popular public activity at this site and the Refuge has provided several amenities including a fishing pier, maintained waterfront fishing areas and an enhanced boat ramp to facilitate the various fishing preferences of Refuge visitors. Game fish commonly caught in Champion Lake include largemouth bass (*Micropterus salmoides*), channel catfish (*Ictalurus punctatus*), blue catfish (*Ictalurus furcatus*), white crappie (*Pomoxis annularisi*) and black crappie (*Pomoxis nigromaculatus*). The Refuge has established conservation guidelines limiting boat motors to no greater than 10 horsepower, and prohibiting fishing or entry within 200 yards (183 meters) of an established bird rookery during certain times of the year. A general site map of the Champion Lake portion of Trinity River NWR is presented in Figure 1.

The main body of Champion Lake is dominated by mature baldcypress trees (*Taxodium distichum*). Old stands of deadwood occur along the channel edges where Big Caney Creek historically transected the site. Several islands are noticeable during low water conditions. Many of these areas are active rookeries and shallow water areas are commonly utilized by wading birds such as egrets, herons and ibis. Open water areas are typically utilized by ducks, coots, cormorants, anhingas and grebes. Aquatic vegetation is common in the shallow, open areas of the site.

Champion Lake is heavily influenced by the Trinity River via Picketts Bayou, and is subjected to frequent flooding during periods of excessive rainfall or by waters being released from dams upstream. Typically, water depth ranges from one to six feet (0.3-1.8 meters). Water conditions are generally turbid during periods of moderate to heavy ingress due to the sediment load entering the site via storm water drainage, or from Big Caney Creek, Picketts Bayou and the Trinity River. The Trinity River is a major waterway with its headwaters beginning just south of the Oklahoma border and crossing through Texas to the Gulf coast. During periods of drought or low ingress, water conditions can become less turbid and even clear, especially in shallower, more protected areas of the site.

The study area included representative sample points within Champion Lake, as well as a sample point near its confluence with Picketts Bayou, a component of the Trinity River flood plain system. This flood plain system is comprised of many diverse habitats such as bottomland hardwood forests, cypress swamps, vegetated wetlands and open waters, all of which occur in or around Champion Lake. In general, the Trinity River NWR is a remnant of what was once a much larger natural area, comprised of a broad, flat flood plain made up of numerous sloughs, oxbows, artesian wells and tributaries. The surrounding upland areas consists of mixed pine-hardwood forest, pine forest, agricultural crop and pastureland.

Figure 1


TRINITY RIVER NATIONAL WILDLIFE REFUGE
LIBERTY COUNTY, TEXAS
UNITED STATES
DEPARTMENT OF THE INTERIOR
UNITED STATES
FISH AND WILDLIFE SERVICE
TRINITY RIVER NWR ACQUISITION BOUNDARY
VICINITY MAP
SCALE IN MILES
ACQUISITION BOUNDARY
PRIMARY CONTROL
TRACT NUMBERS
EXPLODED VIEW OF TRACT 20 & 32b
EXPLODED VIEW OF TRACT 32 & 32a
Rayburn
Dolen
Rye
Cleveland
BEAUMONT
HOUSTON
Davis Hill State Park
Moss Hill
Hardin
Daisetta
Kenefick
Dayton
Liberty
Ames
Raywood
Devers
HARDIN CO.
LIBERTY CO.
LIBERTY COUNTY
CHAMBERS COUNTY
COMPILED IN THE DIVISION OF REALTY FROM USGS QUADRANGLE MAPS, SURVEYS BY USFWS AND OTHER OFFICIAL INFORMATION.
ALBUQUERQUE, NEW MEXICO APRIL 2002
REVISED: JULY 2003
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Climate

Champion Lake occurs in the southern portion of Liberty County, Texas. The climate in this part of the state is considered subtropical and humid. Daily temperatures range from a minimum of 40° F (4°C) in January to a maximum of 94° (34°C) in July. This area receives an average annual rainfall of approximately 51 inches (130 cm).

METHODS

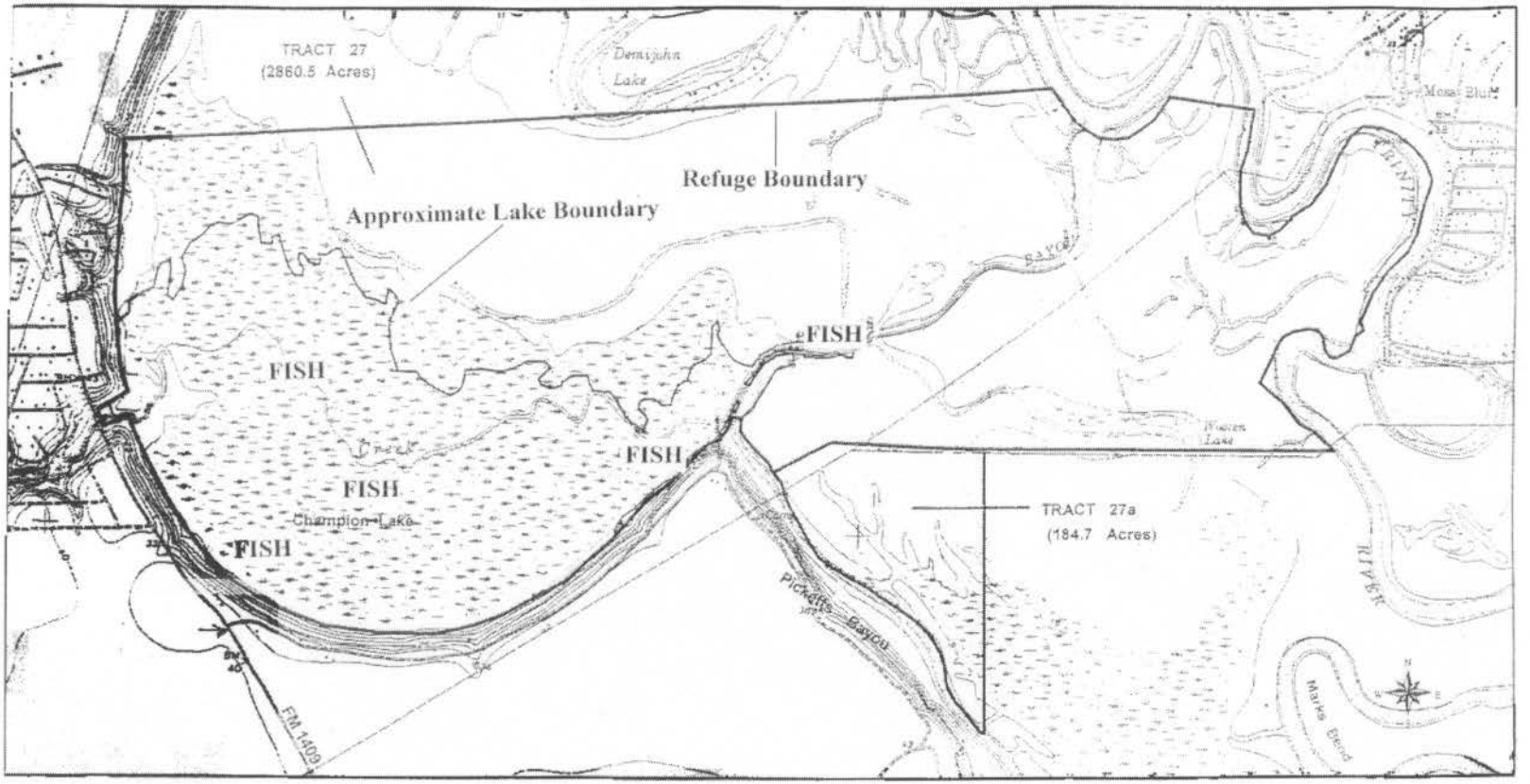
Fish, crustacea and sediment samples were collected from Champion Lake from March to June, 2002. A change was necessary due to the unavailability of the proposed targeted crustacea, crawfish, in Champion Lake at the time of sampling. Attempts were made to capture crawfish, but it was apparent that an insufficient number of crawfish were present in the study area to satisfy the sampling requirements. However, area drought conditions contributed to saltwater intrusion up the Trinity River which provided suitable conditions for an abundant population of blue crab (*Callinectes sapidus*) in Champion Lake. This change in target crustacea species was made since crawfish and crab are both bottom dwellers with similar feeding and habitat requirements.

All samples collected were submitted for analyses to Geochemical & Environmental Research Group, Texas A&M (GERG) in August, 2002. Analytical methods used by the testing laboratory to obtain the organic analytical data are detailed in Appendix B. Analytical methods to obtain inorganic analytical data are detailed in Appendix C.

Fish

Fish samples were collected from areas used by the public in Champion Lake (Figure 2). These areas are accessible to the public either by boat, or from the shoreline at or near the boat ramp and beyond the pedestrian gate leading to Picketts Bayou. Collection techniques included the use of an electro-shocking boat, gill net, hoop nets, trotline, and conventional fishing tackle. Texas A&M University graduate students used an electro-shocking boat to assist during a single weekend sampling event that resulted in three fish samples. A Refuge sponsored fishing event on May 25, 2002, yielded many of the bass and crappie samples submitted. A trotline was deployed near the confluence of Picketts Bayou within Champion Lake which yielded most of the catfish specimens. A gill net and hoop nets deployed in other areas proved unsuccessful in collecting any of the targeted fish samples. Throughout the sampling events, a total of ten largemouth bass, white crappie and catfish (both channel and blue catfish) were collected by the aforementioned methods. Of the fish captured, larger sized specimens were selected to obtain skinless fillets (boneless, edible portions) and whole body (completely intact specimen) samples to assess the potential for bioaccumulation of contaminants. Fish collected during each sampling event were immediately placed on ice. A hand-held Global Positioning System (GPS) unit was used to obtain coordinates for fish sample locations when the equipment was available.

Figure 2
Fish Sample Locations
Trinity River National Wildlife Refuge



Base Map: USGS 7.5' Topographic Quadrangle Moss Bluff, TX.

Scale 1: 19,000

Feet

0 1000 2000 3000 4000

Immediately following each sampling trip, fish collected were removed from the ice, weighed and measured. Skinless fillets from five of the larger fish of the three species collected (15 samples total) were removed, weighed and placed individually in analytical-grade sample containers (16 to 32 ounce pre-cleaned, clear glass, wide-mouth jar with a Teflon-lined lid). This was accomplished by placing each fish on a clean piece of plastic sheeting and using clean Nitrile gloves to handle the specimen. A stainless steel fillet knife was used to remove boneless, edible portions of meat from each fish. The fillet knife was decontaminated using a pH-neutral, biodegradable detergent (Liquinox) and subsequent clean water rinse. Clean gloves were used between samples to further prevent cross-contamination. Five additional fish of each species (15 samples total) were individually wrapped in aluminum foil (dull side toward the specimen) for whole body analysis. Clean gloves were used to handle each individual specimen to prevent cross-contamination. All samples were properly labeled following preparation. Information recorded on the sample container labels included the sample date, time, discrete identification (ID), type of sample and collector's name. The aluminum foil wrapped samples were also labeled in a similar manner.

Following each sampling trip and respective preparation, all fish samples were placed in a freezer and maintained in a frozen state pending delivery to the analytical laboratory where they were kept frozen until analyzed. All skinless fillet and whole body fish samples were submitted for the analyses of heavy metals (including mercury) and organochlorines.

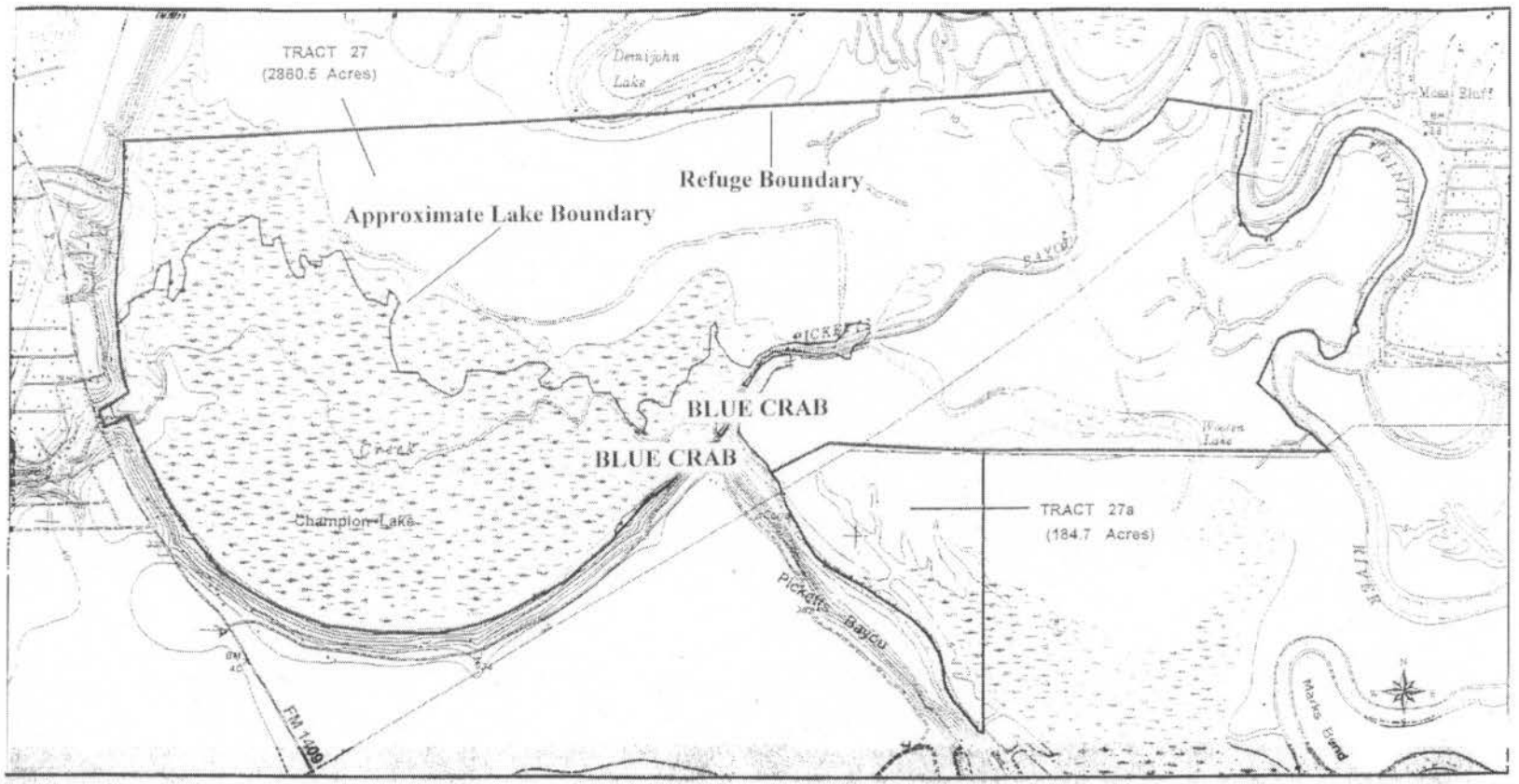
Crustacea

Blue crabs were collected from shoreline areas accessible by the public; primarily at the boat ramp and beyond the pedestrian gate at Champion Lake, by using a baited line and dip net during a single sampling event (Figure 3). Only crabs of legal-size crab were retained as samples, and they were immediately placed on ice.

Immediately following the sampling trip, the crabs were removed from the ice, placed in a freezer in bulk and maintained in a frozen state pending further processing. Prior to delivery to the analytical laboratory, the crabs were removed from the freezer and allowed to partially thaw to allow separation and processing of the individual specimens. Five composite samples, comprised from two crabs each, were prepared by removing the carapace, gills, claws, legs and exposed internal organs of each crab. The main bodies and claws containing edible portions of meat from each crab in the composite sample were weighed together and wrapped in aluminum foil for edible tissue analysis. Five additional crabs were individually wrapped in aluminum foil for whole body (completely intact specimen) analysis. Each sample was properly labeled with the sample date, time and discrete ID. Clean gloves were used to handle each sample to prevent cross-contamination during sample processing.

Following this sample preparation, all crab samples were then re-frozen and maintained in a frozen state pending delivery to the analytical laboratory where the samples were kept frozen until analyzed. All edible portion and whole body crab samples were submitted for the analyses of heavy metals and organochlorines.

Figure 3
Blue Crab Sample Locations
Trinity River National Wildlife Refuge



Base Map: USGS 7.5' Topographic Quadrangle Moss Bluff, TX.

Sediment

Surficial sediment samples were collected from four sites in Champion Lake (Figure 4). These sample locations are representative of depositional or inflow areas where possible accumulation or introduction of potential contaminants within the study area would most likely occur. A brief description of each collection site follows.

Sediment sample location SED 1 is located approximately 50 to 70 yards (46 - 64 meters) out from the existing public boat ramp. Water depth at the time of sampling was approximately 4 feet (1.2 meters). The sample media consisted of soft, fine sediment comprised of unconsolidated silts. This sample point was selected due to its close proximity to the boat ramp where boat usage most frequently occurs. Boating operational and maintenance activities such as (1) immersing boat trailers and tow vehicle rear axles in the water, (2) mixing engine fuel oils, (3) idling boat motors, (4) rinsing equipment and (5) draining boat hulls all contribute to the cumulative potential contamination of this high use area.

Sediment sample location SED 2 is located approximately 20 yards (18 meters) offshore from a storm water drainage culvert situated near the waterfront residences occurring along the southwestern shore of the study area. Water depth at the time of sampling was approximately 3.5 feet (1 meter). The sample media consisted of a soft sediment comprised of unconsolidated silts and organic detritus. This drainage culvert is one of the most prominent man-made inflow areas discharging runoff from the adjacent uplands and rural areas.

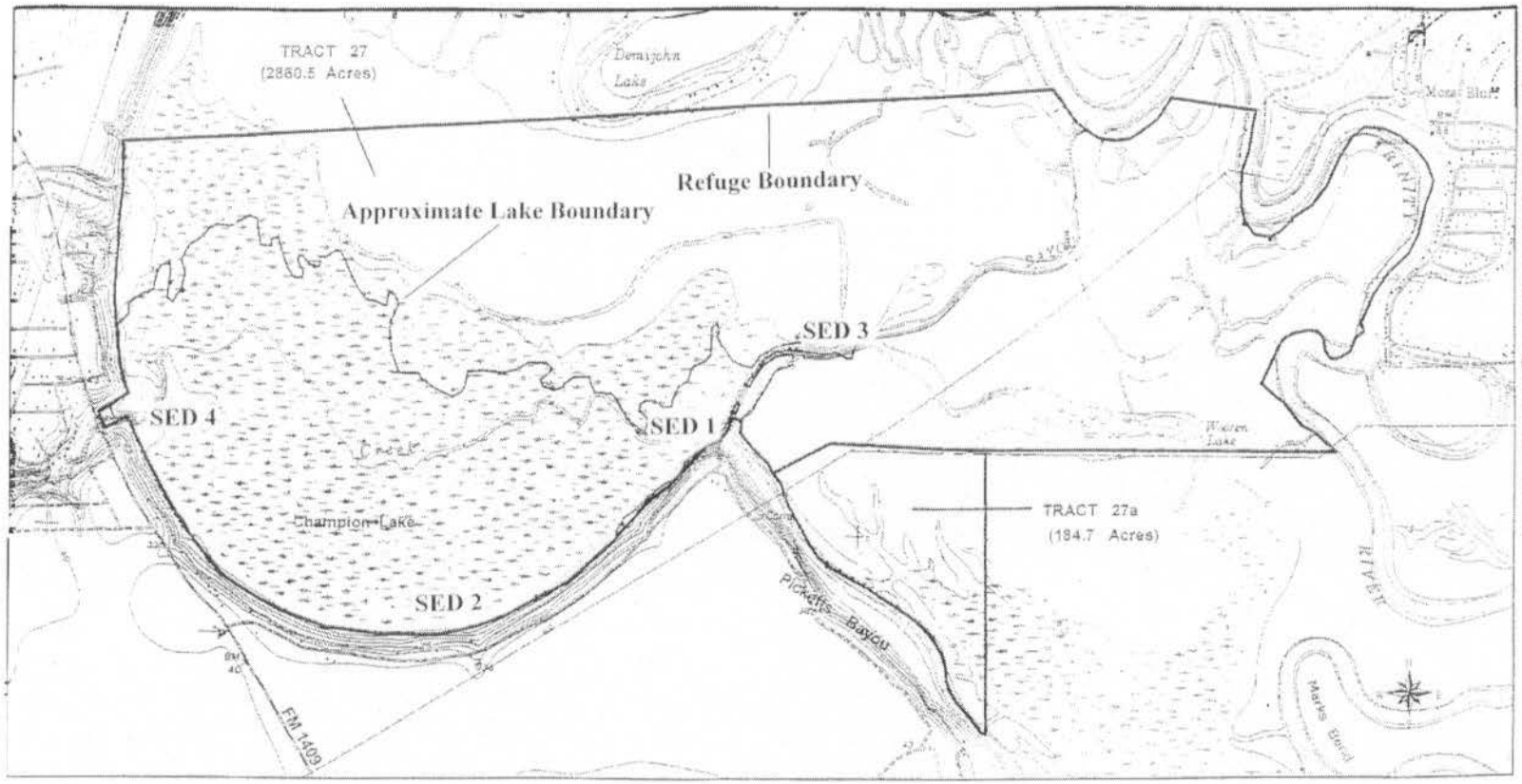
Sediment sample location SED 3 is located near the confluence of Picketts Bayou within Champion Lake on the east side of the study area. Water depth at the time of sampling was approximately 2 feet (0.6 meters). The sample media consisted of medium soft and fine sediment comprised of unconsolidated silts. This sample location is the initial point of entry of the sediment load deposited in Champion Lake from the Trinity River via Picketts Bayou. The river is subjected to potential contamination from urban, municipal, industrial and agricultural runoff from the metroplex of Dallas-Fort Worth and several small cities as it flows south to Trinity Bay.

Sediment sample location SED 4 is located near the confluence of Big Caney Creek within Champion Lake. A delta occurs at the mouth of the creek as it enters the west side of the study area. Water depth at the time of sampling was only a few inches. The sample point is a high deposition area and the sample media consisted of sandy silt and organic detritus. Big Caney Creek is the most significant drainage pathway flowing into Champion Lake from the adjacent rural development.

A petite ponar dredge was used to collect bottom sediment at each sample location. Three discrete grabs of sediment were collected from each location and mixed in a stainless steel bowl. From this homogenous mixture, a composite sample was placed in an analytical-grade sample container (8-ounce, pre-cleaned, clear glass, wide-mouth jar with a Teflon-lined lid) using clean gloves and a new disposable scoop for each sample. In addition, an ambient rinse of the petite ponar dredge and stainless steel bowl was performed between sample locations to prevent cross-contamination. Each sample container was properly labeled and then placed on ice. Information recorded on the sample container labels included the sample date, time, discrete ID, type of sample and collector's name. A hand-held GPS unit was used to obtain coordinates for each sample location (Figure 4). Following the sampling trip, the four sediment samples were placed in a freezer and maintained in a frozen state pending delivery to the analytical laboratory where the samples were kept frozen until analyzed. All sediment samples were submitted for the

analyses of heavy metals, PAHs and organochlorines.

Figure 4
Sediment Sample Locations
Trinity River National Wildlife Refuge



Base Map: USGS 7.5' Topographic Quadrangle Moss Bluff, TX

Scale 1: 19,000

Feet

0 1000 2000 3000 4000

SED 1	N 29° 55' 17.2"	W 94° 47' 58.7"	SED 2	N 29° 54' 46.8"	W 94° 48' 58.0"
SED 3	N 29° 55' 29.9"	W 94° 47' 33.8"	SED 4	N 29° 55' 32.8"	W 94° 49' 35.0"

RESULTS AND DISCUSSION

Organic Contaminants

A suite of 29 organochlorine compounds and 44 petroleum hydrocarbons (i. e. PAHs) were included in the analysis of the four sediment samples collected from Champion Lake. PAHs were not included in the analysis of fish or crab tissue samples because these compounds rapidly accumulate at exposure time in animal tissue, and are then rapidly metabolized and depurated (Nava and Englehardt 1980, Lawrence and Weber 1984). Due to this metabolic process, tissue samples generally do not yield petroleum compounds unless there is a continual source of contamination.

Sediment Samples - Organochlorines

Eleven of the 29 organochlorine compounds were detected in the four sediment samples (Table 2). Only two compounds (PCB-TOTAL and 1,2,4,5-tetrachlorobenzene) were detected in all four sediment samples. PCB-TOTAL concentrations varied between 0.04 ppm and 0.023 ppm on a dry weight basis. None of the other 10 organochlorine compounds, including the 1,2,4,5-tetrachloro-benzene, were detected above the 0.002 ppm dry weight concentration in any sample. One sediment sample (SED 2) collected near the storm water drain that empties into Champion Lake contained 10 of the eleven organochlorine compounds detected, whereas the other three samples contained only 3 -5 organochlorine compounds (Table 2). None of these sediment samples were heavily impacted by organochlorine compounds. The sum of all organochlorine compounds in SED 2 is only 0.030 ppm and does not appear to represent a serious threat to infaunal or benthic organisms based upon the Accumulation Factor (AF) derived by Lee (1992). Similar evaluations of sediment contaminants (Lake et al. 1990, Ferraro et al. 1990) support this evaluation.

Sediment Samples - PAHs

Fourteen of the 44 PAH compounds included in the analysis of sediment samples were detected in the sediment (Table 2). Twelve of the 14 detected PAH compounds were in the SED 2 sample, whereas the other three sediment samples only contained between three and seven PAH compounds. SED 4, collected from a high sediment depositional area, had seven PAHs detected at less than 0.1 ppm dry weight. Johnson et al. (in press) suggest that sediment PAH concentrations at or below 1.0 ppm, liver lesions, DNA adduct levels and growth indicators are similar to levels in English sole (*Pleuronectes vetulus*) from reference sites with minimal sediment contamination, and that the fish exhibit little or no toxicopathic injury. Other research (Malins, et al. 1985, and Eisler 1987) has reported that PAH sediment levels above 1.0 ppm are associated with hazards to fish.

Sediment standards are not available for most PAH compounds. However, the U.S. Environmental Protection Agency (USEPA) established a freshwater sediment criteria of 180 ppm phenanthrene per gram of organic carbon (USEPA 1993a), and 620 ppm fluoranthene per gram of organic carbon (USEPA 1993b) as the limits where no toxic effect should be expected to benthic organisms. This means that the higher the organic carbon is in the sediment, the higher the PAH concentration in whole sediment can be without harmful effects to aquatic life as a result of a particular PAH compound. The TNRCC (2001) lists ecological benchmarks for freshwater sediments as 0.04 ppm dry weight for phenanthrene and 0.11 ppm for fluoranthene. PAH residues in sediment samples collected from Champion Lake (Table 2) were below the

USEPA criteria stated above, and well below these TNRCC benchmark values. Because the PAH residues from Champion Lake were not parent material but showed evidence of biodegradation (i.e. a C group attached), these compounds are not considered to be an ecological hazard to benthic organisms at this time.

Tissue Samples- Organochlorine

Twenty- two organochlorine compounds (excluding PCB-TOTAL) were detected in either the skinless fillet or whole body samples of bass, crappie and catfish collected from Champion Lake (Table 3). PCB-TOTAL was detected in all five samples representing both skinless fillet and whole body samples from all three fish species, however, the highest concentration in crappie whole body averaged only 0.059 ppm. Fillets from the bass, crappie and catfish averaged 0.03, 0.02 and 0.03 ppm wet weight for PCB-TOTAL, respectively. The other 22 organic contaminants in all of the fish samples averaged at least 10 times lower in concentration (Table 3).

PCB-TOTAL is a group of 209 synthetic halogenated aromatic hydrocarbons used in products such as lubricants, dielectric agents, flame retardants, plasticizers and water proofing materials (Roberts et al. 1978). Some of the isomers (i.e. Aroclor 1260) are more toxic than other isomers, however, they generally represent only a small fraction of a PCB mixture. Total PCB residue in sensitive teleosts (fish) in excess of 0.50 ppm in their diet, 0.40 ppm in whole body and 0.30 ppm in their eggs should be considered as presumptive evidence of significant PCB contamination (Eisler 1986).

Blue crabs had detectable levels of seven organochlorine compounds (Table 3) out of the 23 included in this analytical screen. Blue crabs are not usually present in Champion Lake on the Trinity River NWR and most likely migrated into the area during 2002, perhaps in response to the severe drought that was present during the spring and summer months.

Table 2. Organic and Inorganic Contaminant Concentration in Sediment Samples from Champion Lake, Trinity River National Wildlife Refuge.

		Analyte	SED 1 (ppm Dry Wt.)	SED 2 (ppm Dry Wt.)	SED 3 (ppm Dry Wt.)	SED 4 (ppm Dry Wt.)
ORGANIC	Polycyclic Aromatic Hydrocarbons	1-methylphenanthrene	--	0.010	--	--
		C1-Fluoranthenes & Pyrenes	--	0.030	0.010	0.050
		C1-Phenanthrenes & Anthracenes	--	0.010	--	--
		C1-chrysenes	--	0.013	0.010	--
		C1-naphthalenes	--	0.010	--	--
		C2-chrysenes	--	--	0.010	--
		C2-fluorenes	0.010	0.031	--	0.037
		C2-naphthalenes	--	0.010	--	0.011
		C3-fluorenes	--	--	--	0.016
		C3-naphthalenes	0.010	0.010	--	0.015
		benzo(b)fluoranthene	--	0.010	--	--
		fluoranthene	--	0.013	--	0.015
		naphthalene	--	0.010	--	--
		perylene	0.334	0.122	--	0.082
	Organochlorine Pesticides	1,2,4,5-Tetrachlorobenzene	0.001	0.002	0.001	0.002
		HCB	--	0.000	--	--
		PCB-TOTAL	0.010	0.022	0.004	0.023
		endosulfan II	--	--	--	0.002
		endrin	--	0.001	--	0.002
		gamma BHC	--	0.000	--	--
		gamma chlordane	--	0.001	--	--
		mirex	--	0.000	--	--
		o,p'-DDD	0.000	0.001	0.001	--
		o,p'-DDE	--	0.001	--	--
		p,p'-DDE	0.001	0.002	--	0.001
INORGANIC	Heavy Metals	Al	41300.0	67500.0	47200.0	60500.0
		As	7.3	6.6	6.3	6.1
		B	10.0	20.0	10.0	20.0
		Ba	148.0	235.0	143.0	238.0
		Be	1.7	2.4	2.0	2.1
		Cd	1.1	0.5	--	--
		Cr	39.0	62.0	45.0	55.0
		Cu	13.0	24.0	14.0	19.0
		Fe	27900.0	37500.0	29200.0	33100.0
		Mg	4380.0	6230.0	4940.0	5500.0
		Mn	522.0	371.0	453.0	433.0
		Ni	22.0	30.0	22.0	27.0
		Pb	24.0	31.0	21.0	25.0
		Se	1.0	1.0	--	0.7
		Sr	69.0	93.0	85.0	77.0
		V	56.0	85.0	58.0	80.0
		Zn	84.0	120.0	89.0	110.0

** (PAHs)

-- Analytical Results Below Detection Limit (Appendix C).

Table 3. Mean Organic Contaminant Concentrations in Fish Samples from Champion Lake, Trinity National Wildlife Refuge.

	Analyte	Bass (ppm Wet Wt.)				Catfish (ppm Wet Wt.)				Crappie (ppm Wet Wt.)			
		Skinless Fillet	(#)	Whole Body	(#)	Skinless Fillet	(#)	Whole Body	(#)	Skinless Fillet	(#)	Whole Body	(#)
Organochlorine Pesticides	1,2,3,4-Tetrachlorobenzene	--	(0)	0.001	(2)	--	(0)	0.000	(1)	--	(0)	0.001	(3)
	1,2,4,5-Tetrachlorobenzene	0.003	(5)	0.002	(4)	0.002	(3)	0.003	(5)	0.002	(3)	0.002	(5)
	Aldrin	0.001	(1)	--	(0)	--	(0)	0.001	(1)	--	(0)	0.000	(1)
	HCB	--	(0)	--	(0)	0.000	(1)	--	(0)	--	(0)	0.000	(1)
	PCB-TOTAL	0.030	(5)	0.045	(5)	0.033	(5)	0.035	(5)	0.021	(5)	0.059	(5)
	alpha chlordane	0.001	(2)	0.001	(3)	0.000	(1)	0.001	(2)	0.001	(5)	0.000	(4)
	beta BHC	0.001	(1)	0.000	(1)	--	(0)	0.001	(1)	--	(0)	0.001	(2)
	chlorpyrifos	--	(0)	0.001	(1)	--	(0)	--	(0)	--	(0)	0.000	(2)
	cis-nonachlor	0.001	(4)	0.001	(5)	0.001	(4)	0.001	(3)	0.000	(4)	0.001	(4)
	dieldrin	0.001	(3)	--	(0)	0.001	(3)	0.001	(3)	0.001	(2)	--	(0)
	endosulfan II	0.001	(1)	0.001	(2)	--	(0)	0.001	(1)	--	(0)	0.001	(3)
	endrin	0.001	(4)	--	(0)	0.001	(3)	0.001	(2)	0.000	(1)	--	(0)
	gamma chlordane	0.001	(1)	--	(0)	0.000	(2)	--	(0)	0.000	(1)	0.000	(1)
	heptachlor epoxide	0.000	(1)	--	(0)	--	(0)	0.000	(1)	--	(0)	--	(0)
	mirex	--	(0)	0.000	(3)	0.002	(2)	0.000	(2)	--	(0)	0.000	(3)
	o,p'-DDD	0.001	(4)	0.001	(4)	0.001	(3)	0.000	(4)	0.002	(3)	0.001	(5)
	o,p'-DDT	--	(0)	--	(0)	--	(0)	0.000	(1)	--	(0)	--	(0)
	oxychlordane	0.001	(2)	0.001	(2)	0.001	(3)	0.001	(2)	0.001	(2)	0.001	(5)
	p,p'-DDD	0.001	(4)	0.001	(4)	0.001	(5)	0.001	(5)	0.001	(5)	0.001	(4)
	p,p'-DDE	0.004	(5)	0.008	(5)	0.007	(5)	0.009	(5)	0.003	(5)	0.009	(5)
	p,p'-DDT	--	(0)	0.000	(1)	0.000	(1)	--	(0)	--	(0)	0.001	(2)
	pentachloro-anisole	--	(0)	--	(0)	0.000	(3)	--	(0)	0.000	(1)	0.001	(3)
	trans-nonachlor	0.001	(5)	0.002	(5)	0.001	(5)	0.001	(4)	0.001	(5)	0.002	(4)

(#) Number of Samples Averaged having Detectable Analyte
-- Analytical Results Below Detection Limit

Table 4. Mean Organic Contaminant Concentrations in Blue Crab (*Callinectes sapidus*) samples from Champion Lake, Trinity River National Wildlife Refuge.

	Analyte	(ppm Wet Wt.)			
		Edible Portion	(#)	Whole Body	(#)
Organochlorine Pesticide	1,2,3,4-Tetrachlorobenzene	0.001	(2)	0.001	(3)
	1,2,4,5-Tetrachlorobenzene	0.002	(4)	0.002	(5)
	PCB-TOTAL	0.007	(5)	0.006	(5)
	alpha chlordane	0.001	(4)	0.001	(3)
	o,p'-DDD	0.001	(3)	0.001	(4)
	p,p'-DDD	0.000	(1)	0.000	(3)
	p,p'-DDE	0.000	(4)	0.001	(4)

(#) Number of Samples Averaged having Detectable Analyte

Inorganic Contaminants

A suite of 19 inorganic elements with their analytical detection limits included in the analysis of all sediment, bass, crappie, catfish and blue crab samples collected from Champion Lake. These elements are referred to as heavy metals, trace elements, micronutrients or macronutrients in various reports, articles or books. In this report they will be referred to as heavy metals or metals. Some of these contaminants were not detected in various samples and will not be discussed further. Mercury and molybdenum were not detected in the four sediment samples. Beryllium and molybdenum were not detected in any fish sample (Table 5). Thirteen of 19 heavy metals were detected in the blue crab samples (Table 6).

Several factors that were not measured in this study, such as the presence of metal binding sites in soil and sediment, and the metal speciation (e.g. carbonate, oxides or sulfate), influence the bioavailability of heavy metals (Linder and Grillitsch 2000). Binding of metals in soil and sediment is attributed to organic carbon content, clay type, cation exchange capacity, redox potential, pH and surface area of the soil or sediment particles (Alloway 1990). All metals may be toxic above certain levels and the health risk is determined by the metal itself, its concentration, its form and duration of exposure to the receptor. Only the bioavailable proportion of a metal in the environment is of toxicological relevance (Linder and Grillitsch 2000).

Aluminum - Al

Sediment concentration of aluminum ranged from 41,300 to 67,500 ppm dry weight (Table 3) from Champion Lake. Catfish collected from the lake had a mean whole body aluminum residue of 50.4 ppm wet weight, while bass and crappie mean whole body residues were 6.22 and 6.50 ppm, respectively (Table 5). Blue crabs averaged 16.9 ppm aluminum on a whole body analysis (Table 6). The high aluminum concentration in catfish whole body is probably related to gut content because this fish species feeds on benthic organisms and usually ingests sediment while feeding. Aluminum residues in blue crabs from Champion Lake were much lower than the amount reported from blue crabs collected from an urban (162 ppm) and an industrial (571 ppm) runoff area (Cain and Albe 1997).

Aluminum is ubiquitous in soils from around the world and varies from 20,000 to 94,000 ppm (Sparling and Lowe 1996). The environmental chemistry and thus its toxicity is driven by pH in soil and water. Its bioavailability may be enhanced by acidification, but is reduced in organically rich soil due to numerous binding sites (Sparling and Lowe 1996). In general, concentrations less than 1000 ppm in diets of birds are not considered harmful because only a fraction of the metal is assimilated by birds and their fecal excretion is relatively efficient.

Arsenic-As

Arsenic residues in four sediment samples from Champion Lake ranged from 6.1 to 7.3 ppm on a dry weight basis (Table 2) which are similar to the TNRCC (2001) published freshwater ecological sediment benchmark of 5.9 ppm dry weight for arsenic at remediation sites. The threshold effects level (TEL) and probable effects level (PEL) published in the USEPA National Sediment Quality Survey (USEPA 1997) are 7.24 and 41.6 ppm, respectively. The highest Champion Lake concentration was just slightly elevated over the USEPA TEL.

Arsenic was detected in several fish tissue samples with the highest average concentration of 0.30 ppm being in the whole body crappie samples (Table 5). Skinless fillets from crappie contained an average of 0.10 ppm wet weight arsenic. Blue crab edible portion and whole body samples averaged 0.15 and 0.14 ppm, respectively, on a wet weight basis (Table

6). Arsenic levels in all fish and crab samples were similar to aquatic samples collected from the Trinity River near Dallas, Texas, by Irwin (1988).

In nature, arsenic exists in four oxidation states and may be in either inorganic or organic forms (DOI 1998), and is considered ubiquitous in the environment. Arsenic concentrations in soil range from 1.0 to 50 ppm and are less than 10 ppb in water (Eisler 1988a). Arsenic is not an essential element for most animal species and at certain levels it has been considered as teratogenic and carcinogenic in many mammal species (Eisler 1988a). Arsenic in forms such as sodium arsenate and arsenic trioxide are toxic to plants, and have been used to defoliate cotton plants and as an aquatic herbicide. In bird studies, the dietary no-observed-adverse-effect levels (NOAEL) were estimated at 3.4 to 22.0 ppm depending on the arsenic form and the bird species (DOI 1998). All of the fish and crab samples from Champion Lake had arsenic concentrations less than one-tenth these NOAELs. Arsenic does not appear to be a contaminant of concern in Champion Lake at this time.

Table 5. Mean Inorganic Contaminant Concentrations in Fish Samples from Champion Lake, Trinity River National Wildlife Refuge.

	Analyte	Bass (ppm Wet Wt.)				Catfish (ppm Wet Wt.)				Crappie (ppm Wet Wt.)			
		Skinless Fillet	(#)	Whole Body	(#)	Skinless Fillet	(#)	Whole Body	(#)	Skinless Fillet	(#)	Whole Body	(#)
Heavy Metals	Al	1.15	(4)	6.22	(5)	1.32	(5)	50.40	(5)	0.83	(3)	6.50	(5)
	As	0.10	(4)	0.09	(4)	--	(0)	0.08	(3)	0.10	(5)	0.30	(5)
	B	--	(0)	--	(0)	--	(0)	0.75	(2)	--	(0)	--	(0)
	Ba	0.12	(5)	1.28	(5)	--	(0)	2.73	(5)	0.22	(5)	2.22	(5)
	Cd	0.04	(4)	0.07	(5)	--	(0)	0.05	(5)	0.03	(5)	0.05	(5)
	Cr	--	(0)	--	(0)	--	(0)	0.20	(2)	--	(0)	0.30	(1)
	Cu	0.35	(5)	1.55	(5)	0.72	(5)	1.25	(5)	0.32	(5)	1.64	(5)
	Fe	2.98	(5)	16.20	(5)	3.28	(5)	64.96	(5)	2.94	(5)	12.00	(5)
	Hg	0.29	(5)	0.15	(5)	0.26	(5)	0.05	(5)	0.22	(5)	0.11	(5)
	Mg	306.00	(5)	533.40	(5)	275.60	(5)	406.20	(5)	340.40	(5)	530.00	(5)
	Mn	0.27	(5)	3.30	(5)	0.24	(5)	14.90	(5)	0.81	(5)	4.90	(5)
	Ni	--	(0)	0.41	(3)	0.10	(2)	0.20	(1)	--	(0)	3.85	(2)
	Pb	0.06	(1)	0.07	(1)	0.08	(1)	0.07	(2)	--	(0)	0.08	(2)
	Se	0.23	(5)	0.27	(5)	0.12	(5)	0.21	(5)	0.24	(5)	0.31	(5)
	Sr	3.16	(5)	27.12	(5)	0.30	(5)	21.48	(5)	3.88	(5)	33.52	(5)
	V	--	(0)	--	(0)	--	(0)	0.22	(5)	--	(0)	--	(0)
	Zn	5.26	(5)	15.92	(5)	6.22	(5)	22.44	(5)	6.62	(5)	18.98	(5)

(#) Number of Samples Averaged having Detectable Analyte

-- Analytical Results Below Detection Limit

Table 6. Mean Inorganic Contaminant Concentrations in Blue Crab Samples from Champion Lake, Trinity River National Wildlife Refuge.

Analyte		Blue Crab (ppm Wet Wt.)			
		Edible Portion	(#)	Whole Body	(#)
Heavy Metals	Al	15.92	(5)	16.90	(5)
	As	0.15	(5)	0.14	(5)
	Ba	1.80	(5)	1.32	(5)
	Cd	0.05	(5)	0.04	(5)
	Cu	7.18	(5)	7.57	(5)
	Fe	10.38	(5)	14.30	(5)
	Hg	0.08	(5)	0.07	(5)
	Mg	280.00	(5)	271.00	(5)
	Mn	2.54	(5)	1.72	(5)
	Pb	0.08	(2)	--	(0)
	Se	0.23	(5)	0.21	(5)
	Sr	11.61	(5)	8.46	(5)
	Zn	33.42	(5)	29.16	(5)

(#) Number of Samples Averaged having Detectable Analyte
-- Analytical Results Below Detection Limit

Boron-B, Barium-Ba, Beryllium-Be, Cadmium-Cd, Chromium-Cr

Boron, barium, beryllium and chromium were all detected in the four sediment samples collected from Champion Lake (Table 2) at levels that are within the background levels for soils of the United States (Shacklette and Boerngen 1984). Cadmium was detected in only two of the four sediment samples (Table 2). Similar levels in soil or sediment samples from Buffalo Lake NWR (Baker et al. 1998) and from industrial runoff sites in Texas City, Texas (Cain and Albe1997) indicate there is no ecological concern from these five metals in the sediments from Champion Lake.

Only two catfish samples contained any residue of boron, barium was detected at low levels in all three species of fish and the blue crab samples, and beryllium was not detected in any of the fish or blue crab samples. Cadmium was detected at low levels in most of the fish samples (Table 5) and the blue crab samples (Table 6). These levels were at least one-tenth the levels in blue crabs collected from Galveston Bay and Texas City urban runoff ditches (Cain and Albe 1997) and are not at any ecological concern level. Based on these findings, no further discussion of the potential effects of these metals to wildlife resources is warranted.

Copper-Cu

Copper is one of the most abundant heavy metals having a concentration of approximately 50 ppm in the earth's crust, and it is an essential element for all living plants and animals (DOI 1998). In sediment samples from Champion Lake, copper concentrations were detected from 13 to 24 ppm dry weight (Table 2). The USEPA sediment screening criteria TEL and PEL are 18.7 and 108 ppm, respectively (USEPA 1997). Sediment concentrations are below

the TNRCC (2001) ecological screening benchmark value of 35.7 ppm dry weight for freshwater sediments at remediation sites. These levels are also below 40 ppm dry weight, which is the statewide 90th percentile level (Irwin 1988). In soil, copper has been reported to be toxic to sensitive plants at 25 to 50 ppm (Demayo et al. 1982).

Copper was detected in all fish samples collected from Champion Lake (Table 5). Mean copper residues ranged from 1.25 to 1.64 ppm wet weight in whole body samples, and 0.32 to 0.72 ppm wet weight in skinless fillet samples. These mean whole body levels are above 90 % of all fish species collected in a national survey (Lowe et al. 1985), but do not pose an ecological risk to fish-eating birds. Birds retain very little copper from their diet and dietary toxicity levels for birds is greater than 100 ppm (Eisler 1997, DOI 1998).

Copper residues in blue crabs averaged 7.18 and 7.57 ppm wet weight in the edible portion and whole body samples (Table 6), respectively. These levels were four to five times higher than levels detected in the three fish species collected from the same area (Table 5). Copper residues in blue crabs from Galveston Bay and an urban rainfall runoff area of Texas City, Texas, however, had two to three times higher concentrations of copper (Cain and Albe 1997). These copper levels do not pose a risk since mammals, like birds, are also relatively tolerant to copper and the maximum recommended tolerable level in diets of domestic mammals is from 100 to 800 ppm (DOI 1998). Although copper in sediment is slightly elevated over EPA screening levels, there does not appear to be an ecological, nor human consumption, concern for copper levels detected in the sediment and aquatic resources collected from Champion Lake at this time.

Iron-Fe, Magnesium-Mg, Manganese-Mn

Iron, magnesium and manganese were all detected at high levels in the four sediment samples collected from Champion Lake (Table 2). These heavy metals were also present in all of the fish (Table 5) and blue crab (Table 6) samples. Iron is the most abundant heavy metal in nature and manganese is the second most abundant (Forstner and Whittmann 1979). Iron is very important to the oxygen carrying function of hemoglobin in red blood; manganese is involved in glucose utilization in biologic systems; and magnesium is necessary for nerve impulse transmission, muscle contraction and metabolic functions (Forstner and Whittmann 1979). These heavy metals were present in sediment and fish samples from the Champion Lake area at levels similar to those reported in several contaminant assessments (Cain and Albe 1997, Irwin 1988 and Baker et al. 1998) and not considered as a threat to fish and wildlife resources. These are essential elements to biologic systems and were not present in the fish and blue crabs from Champion Lake at levels indicative of polluted waters or a threat to the aquatic resources.

Mercury-Hg

Mercury concentrations were below the 0.10 ppm detection limit (Appendix D) in the four sediment samples collected from Champion Lake. The USEPA freshwater sediment screening criteria TEL and PEL are 0.13 ppm and 0.696 ppm, respectively. Canada has proposed a probable effect level of 0.48 ppm dry weight for sediments (DOI 1998). Mercury has no known biological function and the elemental form is usually converted via bacterial activity in sediments and water to the methylmercury form. Mercury in the sediment from this section of Champion Lake is not likely a serious contaminant at this time.

Methylmercury is the most toxic and bioavailable form of mercury and results in neurological disorders (DOI 1998). Whereas, most mercury in sediment is typically inorganic Hg (II), mercury in fish tissue is almost entirely methylmercury. Within a population of fish, concentrations of mercury in tissue or whole body usually increase with size and age (Wiener et

al. 2003). Legal size bass, crappie, catfish and blue crabs did not contain residues of detected heavy metals at the "action level" of either the Texas Department of Health (0.7ppm, 1997) or the USFDA (1.0 ppm). USEPA revised its methylmercury fish tissue residue criterion ("action level") for freshwater and estuarine fishes to 0.30 ppm for a 70kg adult person (USEPA 2000a). Skinless fillets had much higher concentrations of mercury than whole body samples for all three fish species (Table 5) as would be expected since mercury tends to accumulate in the muscle tissue of fish.

Nickel-Ni

Nickel was detected in all sediment samples and ranged between 22.0 and 30.0 ppm dry weight (Table 2). This level is similar to the sediment criteria of 25.0 ppm dry weight used by the Texas Commission on Environmental Quality (TNRCC 1996) for risk analysis of potentially contaminated sites. However, nickel levels are elevated over the latest TNRCC (2001) ecological benchmark of 18.0 ppm for freshwater sediment at remediation sites in Texas and the USEPA (1997) freshwater sediment criteria TEL for nickel of 15.9 ppm. The USEPA PEL is 42.8 ppm. Average nickel residue was 3.85 ppm wet weight in two crappie whole body samples (Table 5), but was not detected in crappie skinless fillet samples. Bass and catfish from Champion Lake had low levels of nickel (<0.50 ppm) in six samples. Blue crabs did not have detectable levels of nickel in edible portions or whole body samples. Nickel does not appear to be bioaccumulating into the aquatic food chain from the sediment at Champion Lake.

There is little known information about the effects of nickel residue in fish and wildlife species (Irwin 1988) even though nickel is one of the 65 priority pollutants recognized by USEPA. Cain and Pafford (l981) indicated that dietary levels of 200 ppm nickel had no effect on growth and development of mallard ducklings, however, some adverse effects did occur at 800 ppm dietary exposure for more than 30 days. Levels of nickel detected in fish and crab samples from Champion Lake appear to be below any published ecological concern level for fish eating birds.

Lead-Pb

Lead residue averaged approximately 25 ppm on a dry weight basis for the four sediment samples (Table 2) collected from Champion Lake. Soils in the U. S. generally have a background lead level of up to 55 ppm dry weight (Baker et al. 1998). The sediment lead criterion for Texas is 61.5 ppm dry weight (TNRCC 1996), and the ecological benchmark established for remediation sites by the TNRCC (2001) is 35.0 ppm dry weight. The USEPA sediment screening level TEL and PEL are 30.2 and 112 ppm, respectively (USEPA 1997). Sediment residues of lead from Champion Lake are below all of these levels of concern.

Lead was detected in 7 of the 30 fish tissue samples collected from Champion Lake at concentrations between 0.06 to 0.08 ppm wet weight (Table 5), and at 0.08 ppm wet weight in only 2 crab samples (Table 6). These levels were at least one-tenth the level detected in crab samples from Galveston Bay (Cain and Albe 1997). Lead is known to be toxic to aquatic organisms (Eisler 1988b) and there does not appear to be a "no-effect" level for lead in animals (Beyer et al. 1996).

The affinity of lead for thiol and phosphate-containing ligands inhibits the synthesis of heme, necessary for hemoglobin, and it affects the membrane permeability of kidney, liver and brain cells (Forstner and Wittmann 1979). Lead ingestion of shotgun pellets by waterfowl and the resulting toxicological effects was extensively studied in the 1950s (Bellrose 1959). Recent literature suggests that waterfowl with no history of lead poisoning usually have detectable (less than 2 ppm wet weight) liver lead concentration (Beyer et al. 1996). Lead can enter water bodies through aerial deposition and roadway runoff, which may account for the lead detected in Champion Lake. The lead concentrations detected in the sediment and aquatic resources of Champion Lake do not appear to be at an ecological level of concern at this time.

Selenium-Se

Selenium was detected in three of the four sediment samples (Table 2) at levels below the Texas State criterion of 1.73 ppm dry weight (TNRCC 1996). Seleniferous bedrock does not occur in Texas east of the Edwards Plateau (DOI 1998) and input of selenium to coastal river sediments may occur from industrial sources. All of the fish samples (Table 5) contained selenium below 1.0 ppm wet weight and the blue crab samples all contained equally low selenium levels (Table 6). Selenium, in low concentrations, is considered an essential nutrient for growth and fertility in animals (Forstner and Wittmann 1979), however, at elevated levels in food items it has caused the disappearance of fish species from several water bodies (DOI 1998) and wildlife mortalities (Ohlendorf et al. 1988). In these studies, selenium concentrations were reported between 5.0 to 12.0 ppm dry weight in the sediments (DOI 1998).

An important feature of selenium is the narrow range between the nutritionally optimum level for animals and the toxicity dietary concentration. Selenium is generally reported as an essential nutrient at concentrations of 0.1 to 0.3 ppm and dietary toxicity occurs between 2.0 to 5.0 ppm (DOI l998). Selenium deficiency or toxicity apparently causes similar effects in animals such as white muscle disease, weight loss, anemia, immune dysfunction and reproductive depression. Levels of selenium in the fish (Table 5) and blue crab (Table 6) samples are within the range of essential nutrients for fish eating birds and are not an ecological concern for Champion Lake.

Strontium-Sr, Vanadium-V, Zinc-Zn

Strontium, vanadium and zinc were detected in each of the four sediment samples (Table 2), but at levels that are within the range for U. S. soils background concentrations (i. e. 43 to930 ppm, 18 to 270 ppm and 17 to 180 ppm, respectively) reported in Baker et al. (1998). Of the aquatic resources sampled, vanadium was only detected in the five catfish whole body samples (Table 5) and will not be discussed further in this report. Strontium was detected at higher levels in the whole body samples of all three fish species than in the edible portion samples (Table 5). Blue crabs also had strontium residues (Table 6), but not at the levels previously reported for Galveston Bay and other sites in Texas (Cain 1992, Cain1993, Cain and Albe 1997, and Roach et al. 1992). Since strontium is not recognized as a toxic heavy metal, there is very little published information on the toxicological effects of strontium to aquatic organisms or its ability to bioaccumulate. Strontium has been reported at 8.1 ppm in seawater and ranges between 0.05 to

0.31 ppm in freshwater (Forstner and Whittmann 1979). This element repeatedly shows up at high sediment concentrations in areas that receive produced water from oil and gas production facilities (Cain 1992, 1993, and Roach et al. 1992). Levels of strontium in the sediment and aquatic resource samples collected from Champion Lake do not appear to be bioaccumulating, and are not considered to be present at any ecological concern level or represent a hazard for fish eating birds.

Zinc was detected in all four sediment samples from Champion Lake and ranged from 84.0 to 120.0 ppm dry weight (Table 2). All of the fish samples contained zinc and the whole body concentrations were generally three times higher than that of the skinless fillets (Table 5). This is likely due to the zinc incorporated in the gut from the whole body samples. Blue crabs in general, contained higher levels of zinc (Table 6) than did fish.

Zinc is one of the most abundant of the essential heavy metals required by animals and it functions as a component of many enzymes. Zinc is readily bioaccumulated by all organisms, and both a deficient or excessive amount may cause adverse effects in all species (DOI 1998). In the toxicity classification of Forstner and Wittmann (1979), zinc is listed as a very toxic and relatively accessible element. However, zinc appears to be relatively non toxic to mammals and birds (Eisler 1993) and is homeostatically controlled. Zinc poisoning may occur in birds if liver or kidney levels exceed 2,000 ppm dry weight (Eisler 1993).

The mean concentration of zinc in worldwide soils is estimated at 65 ppm and background levels in soils or sediments seldom exceed 200 ppm (Eisler 1993). Sediment concentrations of 150 ppm dry weight are at the "no effect" level according to Long et al. (1995) whereas zinc concentrations between 150 ppm to 410 ppm are at a threshold level of concern. Above 410 ppm dry weight, adverse effects on benthic organisms are common (Long et al. 1995). Sulfides in sediment combine with zinc to reduce its toxicity and as long as the sediments are not fully oxidized, acute toxicity to benthic organisms are unlikely (DOI 1998). Levels of zinc in the sediment samples from Champion Lake do not indicate a pollution potential that would (1) affect fish reproduction, (2) cause adverse effects in the fish, or (3) be a hazard to humans or fish eating birds.

FISH CONSUMPTION RECOMMENDATIONS

The contaminant levels in individual crab edible tissue and individual fish fillet samples are listed in Appendix A. These data were used as the starting point for the development of fish consumption recommendations. Methodology for deriving fish consumption recommendations follows EPAs Guidelines for Assessing Chemical Contaminant Data for Use in Fish Advisories (USEPA 2000a,b).

The data was first grouped by species (blue crab – *Callinectes sapaidus*, largemouth bass – *Micropterus salmoides*, blue and channel catfish – *Ictalurus furcatus* and *I. punctatus*, and white crappie-*Pomoxis annularisi*), each species represented by five individual samples. A proxy value of one-half the detection limit was used for non-detected contaminant concentrations for which 50 % or greater (three or more of the five samples) of the samples had quantifiable

contaminant concentrations. The data which was used in the subsequent steps of this analysis are shown in Table 7. Concentrations which exceed EPA's screening values for recreational and subsistence fishers are identified by red and yellow shading, respectively. These screening values are contained in Table 8. The arsenic screening value, which is based on inorganic arsenic, was not used. The arsenic analysis conducted on the fish tissue samples reported total arsenic. Generally arsenic in fish and shellfish is predominantly in an organic form (USEPA, 2000). Since inorganic arsenic values were not available for this analysis, the total arsenic concentrations found in the crab and fish tissue samples were not compared to the arsenic screening values.

PCB, mercury, and dieldrin fish tissue concentrations showed exceedences of screening values. Exceedences of the recreational fisher PCB screening value (0.02 ppm) were found in four out of five of the bass fillet samples, three out of five of the catfish fillet samples, and two out of five of the crappie fillet samples. All the remaining samples exceed the subsistence fisher PCB screening value (0.00245 ppm).

Exceedences of the recreational fisher mercury screening value (0.4 ppm) were found in one of the five crab samples and one of the five catfish fillet samples. All remaining samples exceed the subsistence fisher mercury screening value (0.049 ppm).

Three of the five bass fillet samples, three of the five catfish fillet samples, and two of the five crappie fillet samples exceed the subsistence fisher screening value for dieldrin (0.000397 ppm) for. All other contaminant concentrations were below the subsistence fisher screening values.

Species sample averages were compared to the screening values to determine if the sample averages were significantly higher than the screening values. Student t values were calculated using the equation:

$$t_s = (x_{average} - SV)/s$$

where

t_s = Student's t value, (n-1 degrees of freedom, n=5)

$x_{average}$ = average contaminant concentration by species

SV = Screening Values (recreational or subsistence fisher)

s = Standard Deviation

These comparisons are shown in Table 9. The screening value exceedences for dieldrin are not shown to be significantly higher than the subsistence fisher screening value. All species PCB average concentrations are significantly higher than the subsistence fisher screening value, but not significantly above the recreational fisher screening value. The mercury species average values for bass, catfish, and crappie were significantly above the subsistence fisher screening value, but not significantly above the recreational fisher screening value.

The risk-based consumption limits development using USEPA's guidance (USEPA 2000b) for the average and maximum edible tissue concentrations are shown in Table 10. The most restrictive limit is used to set the species consumption recommendation. An individual should limit their consumption of blue crab from Champion Lake to one eight ounce meal a month based on the maximum mercury concentration in crab tissue. The recommended consumption frequency for an eight ounce meal of largemouth bass is three meals per month based on the mercury concentration found in the fillet. The recommended consumption frequency for an eight ounce meal of catfish and crappie is two meals per month based on fillet mercury concentrations.

These data are based on preliminary sampling. To more thoroughly assess risk, a creel survey should be conducted to examine species consumed and frequency of consumption. Further studies should be conducted involving the collection of fish, crawfish, avian eggs, and water samples to determine the extent and availability of mercury and PCBs to fish and other wildlife resources at the Refuge.

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Appendix A

Analytical results for crab meat and fish fillets in ppm.

Sample Number	C1-P	C2-P	C3-P	C4-P	C5-P
Sample Type	Crab w/o shell	Crab w/o shell	Crab w/o shell	Crab w/o shell	Crab w/o shell
1,2,3,4-Tetrachlorobenzene	0.00119	< 0.000352	< 0.000366	0.00062	< 0.000353
1,2,4,5-Tetrachlorobenzene	< 0.000357	0.0016	0.00313	0.00243	0.000755
Aldrin	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
HCB	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Heptachlor	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
PCB Total	0.0076	0.0061	0.0075	0.0062	0.007
alpha BHC	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
alpha Chlordane	< 0.000357	0.000542	0.00068	0.000535	0.000529
beta BHC	< 0.000357	< 0.00352	< 0.000366	< 0.000363	< 0.000353
Chlorpyrifos	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
cis-Nonachlor	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
delta BHC	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Dieldrin	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Endosulfan II	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Endrin	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
gamma BHC	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
gamma Chlordane	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Heptachlor epoxide	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Mirex	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
o,p'-DDD	< 0.000357	0.000395	0.00139	0.000865	< 0.000353
o,p'-DDE	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
o,p'-DDT	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Oxychlordane	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
p,p'-DDD	< 0.000357	< 0.000352	< 0.000366	0.000375	< 0.000353
p,p'-DDE	0.000376	0.000539	0.000472	0.000436	< 0.000353
p,p'-DDT	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Pentachloro-Anisole	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Toxaphene	< 0.00446	< 0.00440	< 0.00458	< 0.00454	< 0.00441
trans-Nonachlor	< 0.000357	< 0.000352	< 0.000366	< 0.000363	< 0.000353
Aluminum	11	14	12	16	26.5
Arsenic	0.2	0.14	0.14	0.13	0.14
Boron	< 0.300	< 0.300	< 0.300	< 0.300	< 0.200
Barium	1	2.3	2	2.4	1.3
Beryllium	< 0.200	< 0.0100	< 0.0100	< 0.0100	< 0.0100
Cadmium	0.05	0.057	0.047	0.046	0.052
Chromium	< 0.0800	< 0.0700	< 0.0700	< 0.0700	< 0.0600

Copper	6.72	7.35	8.52	6.58	6.74
Iron	8.1	8	7.8	11	17
Mercury	0.07	0.077	0.11	0.08	0.52
Magnesium	263	289	287	302	260
Manganese	2.2	3.1	2.7	2.9	1.8
Molybdenum	< 0.300	< 0.300	< 0.300	< 0.300	< 0.200
Nickel	< 0.800	< 0.700	< 0.700	< 0.700	< 0.600
Lead	< 0.300	< 0.300	0.11	0.05	< 0.200
Selenium	0.24	0.23	0.24	0.21	0.22
Strontium	8.13	13	12.9	14.1	9.92
Vanadium	< 0.0800	< 0.0700	< 0.0700	< 0.0700	< 0.0600
Zinc	35.5	32.1	38	36.4	25.1

Sample Number	**B1-F**	**B2-F**	**B3-F**	**B4-F**	**B5-F**
Sample Type	**Bass Fillet**	**Bass Fillet**	**Bass Fillet**	**Bass Fillet**	**Bass Fillet**
1,2,3,4-Tetrachlorobenzene	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
1,2,4,5-Tetrachlorobenzene	0.00224	0.003	0.003	0.0032	0.00234
Aldrin	< 0.000341	0.000787	< 0.000385	< 0.000391	< 0.000392
HCB	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
Heptachlor	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
PCB Total	0.0153	0.0391	0.037	0.0259	0.0244
alpha BHC	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
alpha Chlordane	0.000751	< 0.000387	< 0.000385	0.000618	< 0.000392
beta BHC	< 0.000341	< 0.000387	0.0014	< 0.000391	< 0.000392
Chlorpyrifos	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
cis-Nonachlor	< 0.000341	0.000844	0.000546	0.000622	0.000505
delta BHC	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
Dieldrin	0.000813	0.000541	< 0.000385	0.000547	< 0.000392
Endosulfan II	< 0.000341	0.00105	< 0.000385	< 0.000391	< 0.000392
Endrin	< 0.000341	0.00104	0.000809	0.000475	0.000996
gamma BHC	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
gamma Chlordane	< 0.000341	< 0.000387	0.00062	< 0.000391	< 0.000392
Heptachlor epoxide	< 0.000341	< 0.000387	0.00044	< 0.000391	< 0.000392
Mirex	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
o,p'-DDD	0.000605	0.000785	0.001	< 0.000391	0.00102
o,p'-DDE	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
o,p'-DDT	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
Oxychlordane	< 0.000341	0.00065	0.00114	< 0.000391	< 0.000392
p,p'-DDD	< 0.000341	0.000985	0.00076	0.000413	0.000586
p,p'-DDE	0.00101	0.00484	0.00351	0.0067	0.00358
p,p'-DDT	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
Pentachloro-Anisole	< 0.000341	< 0.000387	< 0.000385	< 0.000391	< 0.000392
Toxaphene	< 0.00427	< 0.00484	< 0.00482	< 0.00488	< 0.00490

trans-Nonachlor	0.00132	0.00251	0.000822	0.00128	0.000742
Aluminum	2.9	0.6	< 0.500	0.6	0.5
Arsenic	0.07	0.17	< 0.500	0.09	0.7
Boron	0.5	< 0.500	< 0.500	< 0.500	< 0.500
Barium	0.07	0.09	0.17	0.18	0.1
Beryllium	< 0.0200	< 0.0200	< 0.0200	< 0.0300	< 0.0200
Cadmium	< 0.0200	0.03	0.04	0.05	0.03
Chromium	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Copper	0.37	0.33	0.44	0.2	0.43
Iron	4.3	3	2.4	3.4	1.8
Mercury	0.37	0.28	0.24	0.3	0.24
Magnesium	305	296	312	327	290
Manganese	0.2	0.2	0.34	0.3	0.3
Molybdenum	< 0.400	< 0.500	< 0.500	< 0.500	< 0.500
Nickel	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Lead	< 0.400	< 0.0500	<0.0500	0.06	< 0.0500
Selenium	0.23	0.26	0.23	0.25	0.19
Strontium	9.9	2.3	4.4	5.32	2.9
Vanadium	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Zinc	4.5	4.8	6.4	5.5	5.1

Sample Number	**CA1-F**	**CA2-F**	**CA3-F**	**CA4-F**	**CA5-F**
Sample Type	**Catfish Fillet**	**Catfish Fillet**	**Catfish Fillet**	**Catfish Fillet**	**Catfish Fillet**
1,2,3,4-Tetrachlorobenzene	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
1,2,4,5-Tetrachlorobenzene	0.0031	0.00265	< 0.000372	< 0.000394	0.00136
Aldrin	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
HCB	< 0.000366	< 0.000368	< 0.000372	< 0.000394	0.000403
Heptachlor	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
PCB Total	0.0128	0.0479	0.0163	0.0406	0.0412
alpha BHC	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
alpha Chlordane	< 0.000366	< 0.000368	0.000372	< 0.000394	< 0.000395
beta BHC	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
Chlorpyrifos	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
cis-Nonachlor	< 0.000366	0.000648	0.000449	0.000751	0.000586
delta BHC	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
Dieldrin	< 0.000366	0.000714	0.000425	< 0.000394	0.000916
Endosulfan II	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
Endrin	< 0.000366	0.000746	0.000518	< 0.000394	0.000964
gamma BHC	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
gamma Chlordane	< 0.000366	0.000446	< 0.000372	0.000398	< 0.000395
Heptachlor epoxide	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
Mirex	< 0.000366	0.00174	< 0.000372	< 0.000394	0.00232
o,p'-DDD	0.00148	0.00112	0.000395	< 0.000394	< 0.000395

o,p'-DDE	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
o,p'-DDT	< 0.000366	< 0.000368	< 0.000372	< 0.000394	< 0.000395
Oxychlordane	< 0.000366	0.00125	< 0.000372	0.000541	0.00122
p,p'-DDD	0.000508	0.000622	0.000708	0.00138	0.00271
p,p'-DDE	0.00179	0.0118	0.00266	0.00763	0.012
p,p'-DDT	< 0.000366	< 0.000368	< 0.000372	0.00043	< 0.000395
Pentachloro-Anisole	< 0.000366	0.000379	0.000372	< 0.000394	0.000509
Toxaphene	< 0.00457	< 0.00460	< 0.00466	< 0.00492	< 0.00494
trans-Nonachlor	0.000418	0.00117	0.000676	0.00119	0.00161
Aluminum	0.6	1.9	1	1	2.1
Arsenic	< 0.0400	< 0.0400	< 0.0400	< 0.0400	< 0.0400
Boron	< 0.0400	< 0.0400	< 0.0400	< 0.0400	< 0.0400
Barium	< 0.0400	< 0.0400	< 0.0400	< 0.0400	< 0.0400
Beryllium	< 0.0200	< 0.0200	< 0.0200	< 0.0200	< 0.0200
Cadmium	< 0.0200	< 0.0200	0.02	< 0.0200	< 0.0200
Chromium	< 0.100	< 0.100	< 0.100	< 0.0900	< 0.100
Copper	1.5	0.26	0.34	0.67	0.82
Iron	2.4	4.5	3.2	3.9	2.4
Mercury	0.15	0.23	0.12	0.43	0.35
Magnesium	280	288	279	257	274
Manganese	0.2	0.39	0.33	0.1	0.2
Molybdenum	< 0.400	< 0.400	< 0.400	< 0.400	< 0.400
Nickel	0.1	< 0.100	< 0.100	0.1	< 0.100
Lead	0.08	< 0.0400	< 0.0400	< 0.0400	< 0.0400
Selenium	0.13	0.13	0.1	0.12	0.1
Strontium	0.15	0.17	0.91	0.08	0.21
Vanadium	< 0.100	< 0.100	< 0.100	< 0.0900	< 0.100
Zinc	6	5.9	5.8	8.2	5.2

Sample Number	**CR1-F**	**CR2-F**	**CR3-F**	**CR4-F**	**CR5-F**
Sample Type	**Crappie Fillet**	**Crappie Fillet**	**Crappie Fillet**	**Crappie Fillet**	**Crappie Fillet**
1,2,3,4-Tetrachlorobenzene	< 0.000354	< 0.000362	<0.000394	< 0.000400	< 0,000389
1,2,4,5-Tetrachlorobenzene	0.00273	0.00246	< 0.000394	0.000394	0.00126
Aldrin	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
HCB	< 0.000354	<0.000362	< 0.000394	< 0.000400	< 0.000389
Heptachlor	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
PCB Total	0.0235	0.0155	0.0215	0.0198	0.0166
alpha BHC	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
alpha Chlordane	0.000601	0.000505	0.000488	0.000709	0.000567
beta BHC	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Chlorpyrifos	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
cis-Nonachlor	0.00055	0.000368	0.000495	0.000467	< 0.000389

delta BHC	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Dieldrin	0.000862	< 0.000362	0.00119	< 0.000400	< 0.000389
Endosulfan II	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Endrin	< 0.000354	< 0.000362	0.000421	< 0.000400	< 0.000389
gamma BHC	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
gamma Chlordane	0.000451	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Heptachlor epoxide	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Mirex	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
o,p'-DDD	0.0018	0.00143	< 0.000394	< 0.000400	0.00154
o,p'-DDE	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
o,p'-DDT	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Oxychlordane	0.000557	< 0.000362	0.000762	< 0.000400	< 0.000389
p,p'-DDD	0.000618	0.000429	0.000572	0.000614	0.000429
p,p'-DDE	0.00392	0.00232	0.00303	0.00332	0.00166
p,p'-DDT	< 0.000354	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Pentachloro-Anisole	0.000399	< 0.000362	< 0.000394	< 0.000400	< 0.000389
Toxaphene	< 0.00442	< 0.00453	< 0.00493	< 0.00500	< 0.00486
trans-Nonachlor	0.000937	0.000428	0.000825	0.000518	0.000421
Aluminum	< 0.500	< 0.400	0.6	1.4	0.5
Arsenic	0.07	0.1	0.1	0.14	0.08
Boron	< 0.500	< 0.400	< 0.500	< 0.400	< 0.500
Barium	0.22	0.34	0.23	0.14	0.108
Beryllium	< 0.0200	< 0.0200	< 0.300	< 0.0200	< 0.0200
Cadmium	0.03	0.04	0.03	0.04	0.03
Chromium	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Copper	0.2	0.39	0.27	0.25	0.49
Iron	5.3	2.2	2.1	2.3	2.8
Mercury	0.33	0.21	0.33	0.1	0.15
Magnesium	322	358	357	326	339
Manganese	0.74	1.4	0.61	0.53	0.77
Molybdenum	< 0.500	< 0.400	< 0.500	< 0.400	< 0.500
Nickel	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Lead	< 0.0500	< 0.0400	< 0.0500	< 0.0400	< 0.0500
Selenium	0.23	0.19	0.29	0.22	0.27
Strontium	3.6	6.51	2.7	2.9	3.7
Vanadium	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
Zinc	6.5	7.2	7.2	5.5	6.7

Appendix B

Laboratory Analytical Methods for Organic Analytes
Catalog: 2040044 PO Number: 92223-0-Y562E Lab: GERG Page: 62

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001

Lab Matrix	Analyte(s)
Animal Tissue	1,2,3,4-Tetrachlorobenzene
	1,2,4,5-Tetrachlorobenzene
	Aldrin
	HCB
	Heptachlor
	PCB-TOTAL
	alpha BHC
	alpha chlordane
	beta BHC
	chlorpyrifos
	cis-nonachlor
	delta BHC
	dieldrin
	endosulfan II
	endrin
	gamma BHC
	gamma chlordane
	heptachlor epoxide
	mirex
	o,p'-DDD
	o,p'-DDE
	o,p'-DDT
	oxychlordane
	p,p'-DDD
	p,p'-DDE
	p,p'-DDT
	pentachloro-anisole
	toxaphene

trans-nonachlor

METHOD CODE: 001

LABORATORY: Geochemical & Environmental Research Group, Texas A&M

Tissue Organics

I. The tissue samples were extracted by the NOAA Status and Trends Method (MacLeod et al., 1985) with minor revisions (Brooks et al., 1989; Wade et al., 1988). Briefly, the tissue samples were homogenized with a Teckmar Tissumizer. A 1 to 10-gram sample (wet weight) was extracted with the Teckmar Tissumizer by adding surrogate standards, Na2SO4, and

ANALYTICAL METHODS
(Cont.)

methylene chloride in a centrifuge tube. The tissue extracts were purified by silica/alumina column chromatography to isolate the aliphatic and PAH/pesticide/PCB fractions. The PAH/pesticide/PCB fraction was further purified by HPLC in order to remove interfering lipids.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988).

There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide

Endosulfan I and the PCB congeners 114 and 157. In these cases, the samples will be analyzed by CGC with a mass spectrometer detector in the SIM mode.

References

1. Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

2. MacLeod, W.D., D.W. Brown, A.J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R.G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS, NOAA Tech. Memo. NMFS F/NWRC-92.

3. Wade, T.L., E.L. Atlas, J.M. Brooks, M.C. Kennicutt II, R.G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oyster. Estuaries 11, 171-179.

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ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 003

Lab	Matrix	Analyte(s)
Animal Tissue		% Lipid

% Moisture
Soil/Sediment % Moisture

METHOD CODE: 003

LABORATORY: Geochemical & Environmental Research Group, Texas A&M

% Dry Weight

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

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

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 004

Lab Matrix	Analyte(s)
Soil/Sediment	1,2,3,4-Tetrachlorobenzene
	1,2,4,5-Tetrachlorobenzene
	1,6,7-Trimethyl-naphthalene
	1-methylnaphthalene

1-methylphenanthrene
2,6-dimethylnaphthalene
2-methylnaphthalene
Aldrin
Benzo(a)anthracene
C1-Fluoranthenes & Pyrenes
C1-Phenanthrenes & Anthracenes
C1-chrysenes
C1-dibenzothiophenes
C1-fluorenes
C1-naphthalenes
C2-Phenanthrenes & Anthracenes
C2-chrysenes
C2-dibenzothiophenes
C2-fluorenes
C2-naphthalenes
C3-Phenanthrenes & Anthracenes
C3-chrysenes
C3-dibenzothiophenes
C3-fluorenes
C3-naphthalenes
C4-Phenanthrenes & Anthracenes
C4-chrysenes
C4-naphthalenes
Dibenz(a,h)anthracene
HCB
Heptachlor
PCB-TOTAL
acenaphthalene
acenaphthene
alpha BHC
alpha chlordane
anthracene
benzo(a)pyrene

ben
zo(
b)fl
uor
ant
hen
e

benzo(e)pyrene
benzo(g,h,i)perylene

benzo(k)fluoranthene
beta BHC

ANALYTICAL METHODS (Cont.)

This section describes the methods used for analysis by analyte.

Method Codes: 004

Lab	Matrix	Analyte(s)
		biphenyl
		chlorpyrifos
		chrysene
		cis-nonachlor
		delta BHC
		dibenzothiophene
		dieldrin
		endosulfan II
		endrin
		fluoranthene
		fluorene
		gamma BHC
		gamma chlordane
		heptachlor epoxide
		indeno(1,2,3-cd)pyrene
		mirex
		naphthalene
		o,p'-DDD
		o,p'-DDE
		o,p'-DDT
		oxychlordane
		p,p'-DDD
		p,p'-DDE
		p,p'-DDT
		pentachloro-anisole

per
yle
ne

phenanthrene
pyrene
toxaphene
trans-nonachlor

METHOD CODE: 004

LABORATORY: Geochemical & Environmental Research Group, Texas A&M

Sediments Organic/Pesticide

IV. The sediment samples were freeze-dried and extracted in a Soxhlet extraction apparatus. Briefly, the freeze-dried sediment samples were homogenized and a 10-gram sample was weighed into the extraction thimble. Surrogate standards and methylene chloride were added and

ANALYTICAL METHODS (Cont.)

the samples extracted for 12 hrs. The extracts were treated with copper to remove sulfur and were purified by silica/alumina column chromatography (MacLeod et al., 1985; Brooks et al., 1989) to isolate the aliphatic and aromatic/pesticide/PCB fractions.

The quantitative analyses were performed by capillary gas chromatography (CGC) with a flame ionization detector for aliphatic hydrocarbons, CGC with electron capture detector for pesticides and PCB's, and a mass spectrometer detector in the SIM mode for aromatic hydrocarbons (Wade et al., 1988).

There are specific cases where analytes requested for the pesticide and PCB analyses and are known to co-elute with other analytes in the normal CGC with electron capture. These include the pesticide Endosulfan I and the PCB congeners 114 and 157. In these cases, the samples will be analyzed by CGC with a mass spectrometer detector in the SIM mode.

References:

1. Brooks, J.M., T.L. Wade, E.L. Atlas, M.C. Kennicutt II, B.J. Presley, R.R. Fay, E.N. Powell, and G. Wolff (1989) Analysis of Bivalves and Sediments for Organic Chemicals and Trace Elements. Third Annual Report for NOAA's National Status and Trends Program, Contract 50-DGNC-5-00262.

2. MacLeod, W.D., D.W. Brown, A.J. Friedman, D.G. Burrow, O. Mayes, R.W. Pearce, C.A. Wigren, and R.G. Bogar (1985) Standard Analytical Procedures of the NOAA National Analytical Facility 1985-1986. Extractable Toxic Organic Compounds. 2nd Ed. U.S. Department of Commerce, NOAA/NMFS, NOAA Tech. Memo. NMFS F/NWRC-92

3. Wade, T.L., E.L. Atlas, J.M. Brooks, M.C. Kennicutt II, R.G. Fox, J. Sericano, B. Garcia, and D. DeFreitas (1988) NOAA Gulf of Mexico Status and Trends Program: Trace Organic Contaminant Distribution in Sediments and Oyster. Estuaries 11, 171-179.

Appendix C

Laboratory Analytical Methods for Inorganic Analytes
Catalog: 2040044 PO Number: 94420-02-Y109 Lab: LET Page: 34

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001 002

Lab Matrix	Analyte(s)
Animal Tissue	% Moisture
Soil/Sediment	% Moisture

METHOD CODE: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

I. 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.

ANALYTICAL METHODS

(Cont.)

METHOD CODE: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 - Freeze drying and % Moisture

II. 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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001 002 007 012

Lab	Matrix	Analyte(s)
Animal Tissue		As Se
Soil/Sediment		As Se

METHOD CODE: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

I. 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.

ANALYTICAL METHODS

(Cont.)

METHOD CODE: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 - Freeze drying and % Moisture

II. 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.

ANALYTICAL METHODS
(Cont.)

METHOD CODE: 007

LABORATORY: Laboratory and Environmental Testing, Inc.

L5 - Magnesium Dry Ash

VI. 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.**

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ANALYTICAL METHODS (Cont.)

METHOD CODE: 012

LABORATORY: Laboratory and Environmental Testing, Inc.

Hydride Generation AA

XII. 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.

ANALYTICAL METHODS (Cont.)

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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001 002 009 013

Lab Matrix	Analyte(s)
Animal Tissue	Hg
Soil/Sediment	Hg

METHOD CODE: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

I. 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.

ANALYTICAL METHODS
(Cont.)

METHOD CODE: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 - Freeze drying and % Moisture

II. 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.

ANALYTICAL METHODS
(Cont.)

METHOD CODE: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 - Microwave Digestion

IX. 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 1/2 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.

ANALYTICAL METHODS (Cont.)

METHOD CODE: 013

LABORATORY: Laboratory and Environmental Testing, Inc.

Cold Vapor AA

XIII. 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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001 002 009 016

Lab Matrix	Analyte(s)
Animal Tissue	Pb

METHOD CODE: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

I. 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.

ANALYTICAL METHODS (Cont.)

METHOD CODE: 002

LABORATORY: Laboratory and Environmental Testing, Inc.

L9 - Freeze drying and % Moisture

II. 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.

ANALYTICAL METHODS (Cont.)

METHOD CODE: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 - Microwave Digestion

IX. 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 1/2 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.

ANALYTICAL METHODS (Cont.)

METHOD CODE: 016

LABORATORY: Laboratory and Environmental Testing, Inc.

Graphite Furnace using the 5100 Zeeman

XVI. 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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 001 002 009 018

Lab Matrix	Analyte(s)
Animal Tissue	Al

B
Ba
Be
Cd
Cr
Cu
Fe
Mg
Mn
Mo
Ni
Sr
V
Zn

Soil/Sediment Al
B
Ba
Be
Cd
Cr
Cu
Fe
Mg
Mn
Mo
Ni
Pb
Sr
V
Zn

METHOD CODE: 001

LABORATORY: Laboratory and Environmental Testing, Inc.

Homogenization

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

ANALYTICAL METHODS
(Cont.)

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

II. 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.

ANALYTICAL METHODS (Cont.)

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

IX. 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.

ANALYTICAL METHODS
(Cont.)

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 1/2 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

XVIII. 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

ANALYTICAL METHODS (Cont.)

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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 007 012

Lab Matrix	Analyte(s)
Animal Tissue	As
	Se
Soil/Sediment	As
	Se

METHOD CODE: 007

LABORATORY: Laboratory and Environmental Testing, Inc.

L5 - Magnesium Dry Ash

VI. 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.

ANALYTICAL METHODS (Cont.)

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

XII. 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

ANALYTICAL METHODS (Cont.)

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.

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ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 009 013

Lab Matrix	Analyte(s)
Animal Tissue	Hg
Soil/Sediment	Hg

METHOD CODE: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 - Microwave Digestion

IX. 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 1/2 of the HNO_3 and H_2O_2.

ANALYTICAL METHODS (Cont.)

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

XIII. 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

ANALYTICAL METHODS (Cont.)

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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 009 016

Lab Matrix	Analyte(s)
Animal Tissue	Pb

METHOD CODE: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 - Microwave Digestion

IX. 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 1/2 of the HNO3 and H2O2.

ANALYTICAL METHODS (Cont.)

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

XVI. 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.

ANALYTICAL METHODS

This section describes the methods used for analysis by analyte.

Method Codes: 009 018

Lab	Matrix	Analyte(s)
Animal Tissue		Al
		B
		Ba
		Be
		Cd
		Cr
		Cu
		Fe
		Mg
		Mn
		Mo
		Ni
		Sr
		V
		Zn
Soil/Sediment		Al
		B

Ba
Be
Cd
Cr
Cu
Fe
Mg
Mn
Mo
Ni
Pb
Sr
V
Zn

METHOD CODE: 009

LABORATORY: Laboratory and Environmental Testing, Inc.

L10 - Microwave Digestion

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

ANALYTICAL METHODS (Cont.)

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 1/2 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

XVIII. 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.

ANALYTICAL METHODS (Cont.)

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.