In cooperation with the U.S. Environmental Protection Agency,
Region 6, Superfund Division

Occurrence of and Trends in Selected Sediment-Associated Contaminants in Caddo Lake, East Texas, 1940–2002

Water-Resources Investigations Report 03–4253

U.S. Department of the Interior
U.S. Geological Survey

Cover:

Left, Carter Lake; right, collecting box core at Carter Lake.

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By Jennifer T. Wilson

U.S. GEOLOGICAL SURVEY
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Occurrence of and Trends in Selected Sediment-Associated Contaminants in Caddo Lake, East Texas, 1940–2002

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Abstract

Bottom-sediment cores were collected from four sites in Caddo Lake in East Texas during May 2002 for analyses of radionuclides (for age dating), organochlorine pesticides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and major and trace elements, and to describe the occurrence and trends of these sediment-associated contaminants. The Goose Prairie Creek and Harrison Bayou sites receive drainage from an area that includes parts of the now-closed Longhorn Army Ammunitions Plant. The mid-lake site is relatively close to dense oil and gas operations in the lake. The Carter Lake site receives minimal discharge from developed areas.

Sediment age (deposition) dates represented in the cores ranged from 1940 to 2002. The only organochlorine compounds detected in all core samples were the DDT degradation products DDE or DDD, and PCB Aroclors 1242, 1254, and 1260 were detected only at the Goose Prairie Creek site. One or more of the DDE concentrations at all sites exceeded a consensus-based threshold effect concentration (on benthic biota), but none exceeded a consensus-based probable effect concentration. The Goose Prairie Creek site had significant downward trends in concentrations of organochlorine compounds, except for no trend in DDE concentrations. The Ammunitions Plant is a possible historical source of the few organochlorine compounds detected at the Goose Prairie Creek and Harrison Bayou sites.

PAH concentrations at all sites were below respective threshold effect concentrations. Highest PAH concentrations at all four sites were of C2-alkylated naphthalenes. Nearly all statistically significant PAH trends in the cores were downward. On the basis of PAH source-indicator ratios, the majority of PAH compounds appear to have originated from uncombusted sources such as leaks or spills from oil and gas operations or vehicles (automobiles, boats, aircraft) in the Caddo Lake area.

Concentrations of several of the eight trace elements with threshold effect concentrations and probable effect concentrations (among 26 analyzed) were above the respective threshold effect concentrations, but all, except one lead concentration at the Goose Prairie Creek site (deposited about 1961), were below respective probable effect concentrations. Among trace element concentrations at the four sites, lead and mercury were consistently relatively high at the Goose Prairie Creek site. Again the Ammunitions Plant, because of its proximity and history of industrial activities, is the suspected primary source. Statistically significant trends in trace element concentrations were mixed, but more were downward than upward.

Computations to indicate the dominant source (atmospheric fallout or drainage area) of mercury to the Caddo Lake sediment core sites (except Carter Lake) indicate that about one-third of the mercury at the Goose Prairie Creek site might result from drainage area sources. No drainage area sources were indicated for the Harrison Bayou and mid-lake sites. Arsenic, cadmium, and zinc concentrations were highest at the Carter Lake site. No relation between the relatively higher trace element concentrations and any potential source of contamination in the Carter Lake drainage area (for example, oil and gas operations, a road, a boat ramp) is indicated.

INTRODUCTION

Caddo Lake in East Texas (and Louisiana) (fig. 1) is designated a Wetlands of International Importance by the Ramsar Convention on Wetlands (an intergovernmental treaty to promote the wise use of wetlands) and is home to numerous and unique species of fish, birds, and plants (Ramsar Sites Database, 2003). Caddo Lake serves as the primary source of drinking water for several surrounding communities (for example, Uncertain and Karnack) and provides recreational opportunities and lakeshore residential areas. It is the only natural lake in Texas—that is, it was not impounded by humans. The most widely accepted hypothesis regarding the origin of the lake is that the water was pooled in the early 1800s by a logjam. About 1914 the logjam was replaced with a constructed dam on the Louisiana end of the lake to stabilize water levels. The lake was re-dammed in 1971, which increased the surface area of the lake to its current (2003) 103 square kilometers (km^2). The Caddo Lake drainage area is estimated from digital elevation models to be 7,230 km^2 (Toby Welborn, U.S. Geological Survey, written commun., 2002).

The Longhorn Army Ammunitions Plant (LHAAP) operated on the southwestern shore of Caddo Lake for 55 years as a munitions loading and assembly facility. The LHAAP has several areas of known contamination, and surface runoff from the plant drains toward Caddo Lake (fig. 2). Several sediment-associated contaminants are of concern to water-resource managers because of their common occurrence and toxicity. For example, the Agency for Toxic Substances and Disease Registry (ATSDR) releases a priority list of substances as required by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). This list describes, in order of priority, 275 substances that are most commonly detected at facilities on the (Superfund) National Priorities List (NPL) and that have been determined to pose the most substantial potential threat to human health because of their known or suspected toxicity and potential for human exposure at NPL sites (Agency for Toxic Substances and Disease Registry, 2001). The LHAAP was placed on the NPL in August 1990. The analyses of the sediment samples from Caddo Lake yielded 54 of the 275 substances on the ATSDR CERCLA priority list including eight of the top 10 substances.

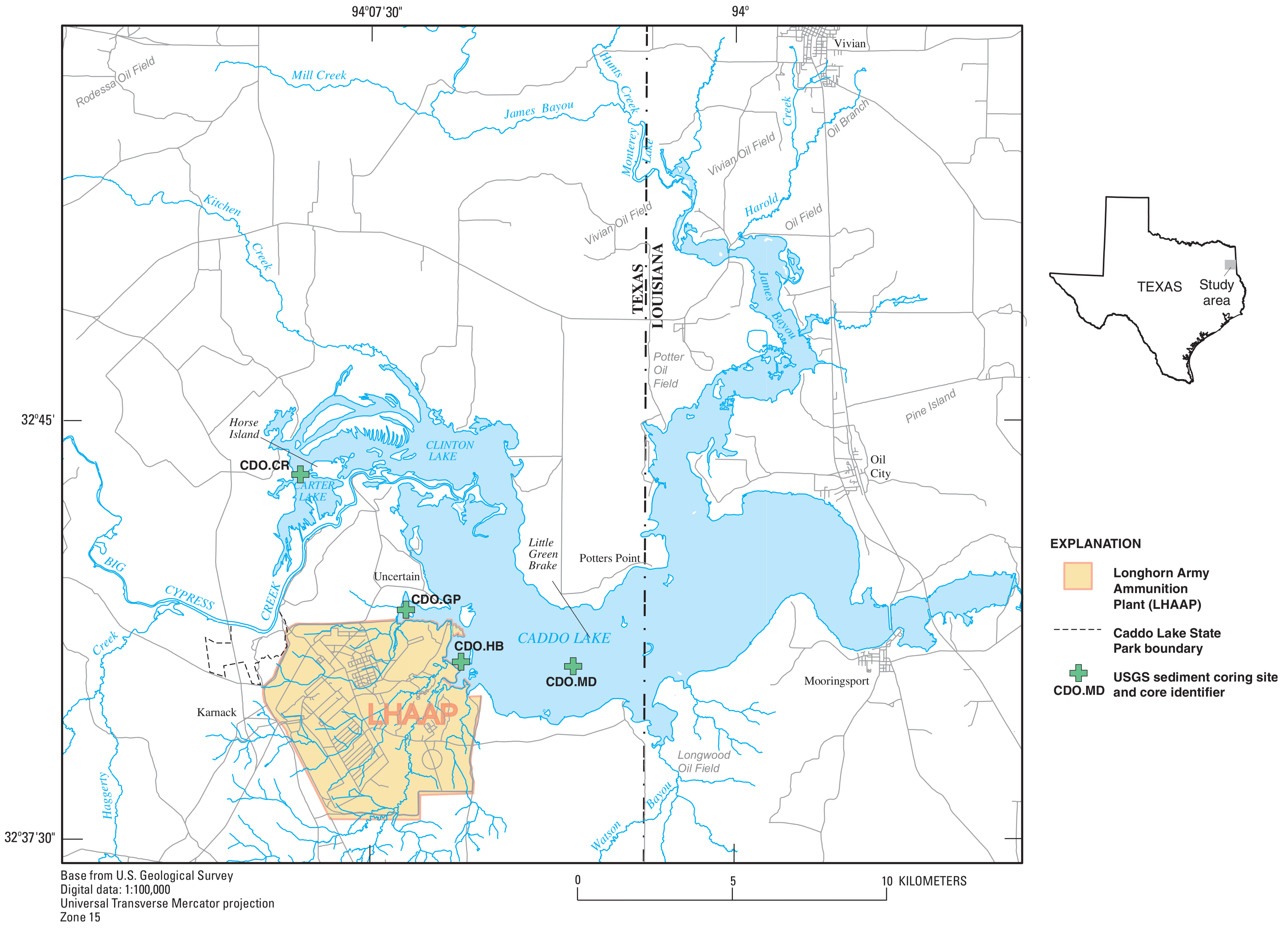
The U.S. Army Corps of Engineers (USACE) and the Texas Commission on Environmental Quality (TCEQ) (formerly the Texas Natural Resource Conservation Commission) have sampled Caddo Lake bottom sediments in Texas extensively (Texas Natural Resource Conservation Commission, 2002a, 2002b; Cliff Murray, U.S. Army Corps of Engineers, unpub. data, 2002). However, both agencies described only the occurrence of contaminants and did not describe contaminant trends over time. The bottom sediments in Caddo Lake (Texas part only) were sampled in May 2002 by the U.S. Geological Survey (USGS), in cooperation with the U.S. Environmental Protection Agency, Region 6, Superfund Division. The evaluation of contaminant trends over time can be used to improve knowledge of possible contaminant sources, the extent of contamination in the sediments, and the potential effects on aquatic biota, and can be used to determine whether conditions are getting better or worse.

Purpose and Scope

The purpose of this report is to describe the occurrence of and trends in selected sediment-associated contaminants in age-dated sediment cores from four sites in the Texas part of Caddo Lake. Radionuclides were used to age date the sediment layers in the cores, which makes possible evaluation of changes in contaminant concentrations over time. The report explains the age-dating process and associates dates of deposition with successive intervals in each core. Concentrations of selected organochlorine compounds, polycyclic aromatic hydrocarbons (PAHs), and trace elements are compared to sediment-quality guidelines that reflect relative sediment toxicity to aquatic biota. Trends based on contaminant concentrations in intervals of core sediment were tested for statistical significance. Potential sources of contaminants, particularly mercury, are discussed.

Background

Caddo Lake can be described as a wetland with bottomland hardwoods and bald cypress swamps. The habitats of the lake support some of the largest populations of wood ducks and mallards in Texas, in addition to migratory birds, restricted wetland species (American alligator and river otter), and other wildlife such as the barred owl, gray squirrel, beaver, and green heron (Cloud and Short, 1993). The lake supports Texas' most diverse fish fauna—one study collected 69 species (Gray, 1955)—and is described by the Texas Parks and Wildlife Department as "the epitome of fish habitat, supporting large populations of largemouth bass, black



Base from U.S. Geological Survey
Digital data: 1:100,000
Universal Transverse Mercator projection
Zone 15

Figure 1. Locations of Longhorn Army Ammunition Plant (LHAAP) and bottom-sediment coring sites sampled in Caddo Lake, Texas, May 2002.

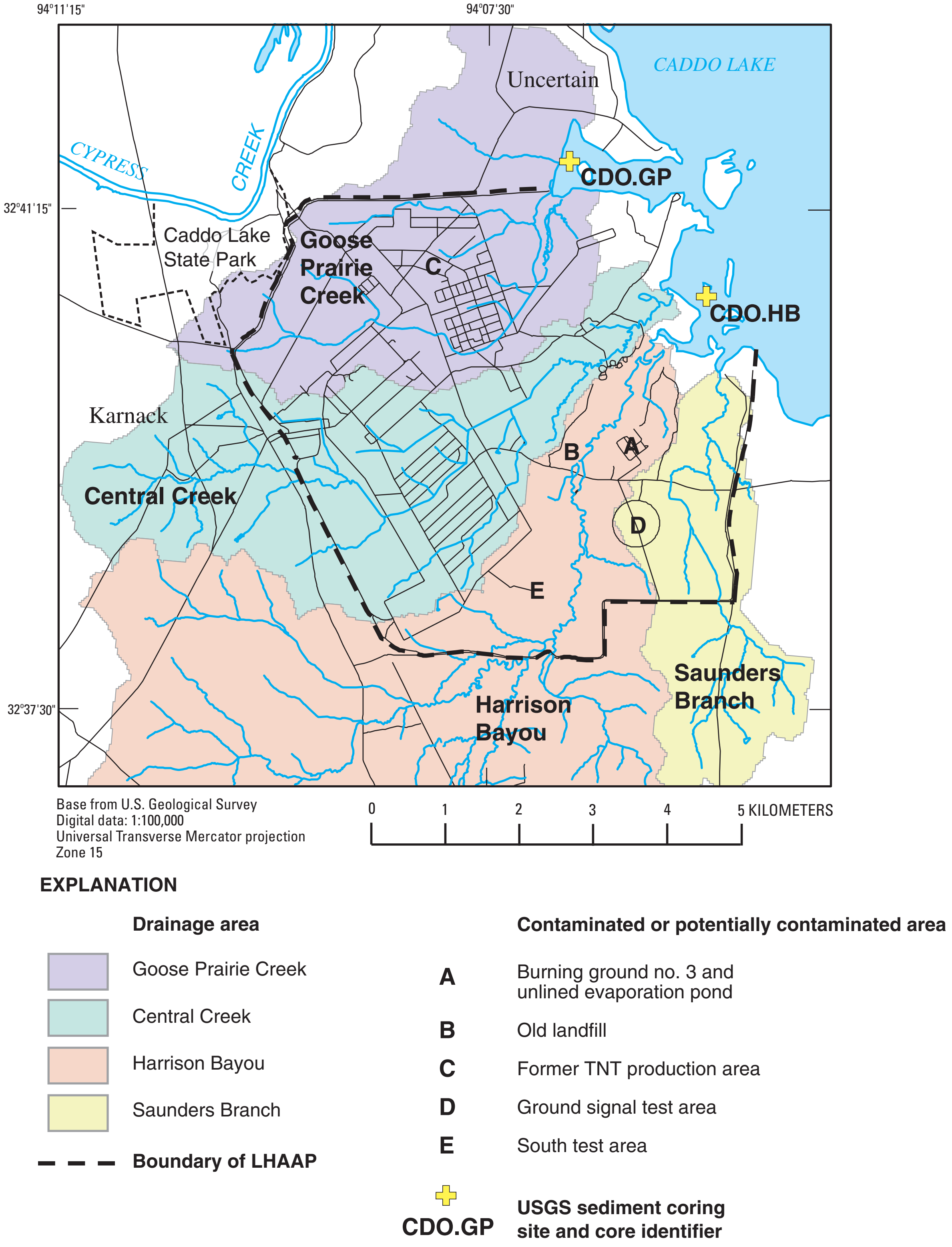


Figure 2. Drainage areas of creeks and contaminated or potentially contaminated areas on Longhorn Army Ammunition Plant (LHAAP), Texas, identified by U.S. Army during a public health assessment of the plant in July 1999 (Texas Department of Health, 1999).

and white crappie, channel catfish, and bluegill" (Toole and Ryan, 1981, *in* Cloud and Short, 1993, p. 5). Several other fish species also are supported by Caddo Lake, for example, bowfin, paddlefish, American eel, southern brook lamprey, and chain pickerel. Caddo Lake and its forested wetlands of mature bald cypress and bottomland hardwoods are considered a unique, fragile, and irreplaceable high-quality habitat for fish and wildlife (Cloud and Short, 1993).

Caddo Lake overlies the Caddo oil and gas field. Oil production in Caddo Lake and its drainage area began in the early 1900s. Gulf Oil Corporation leased the bottom of Caddo Lake for oil development and built the first over-water drilling platform in 1911. Most of the current (2003) producing wells in the lake are in Louisiana.

The LHAAP operated from 1942 to 1997 on the southwestern shore of Caddo Lake near the towns of Karnack and Uncertain (fig. 1). The plant manufactured trinitrotoluene (TNT) and rocket motors; loaded, assembled, and packed pyrotechnic and signal ammunition; and performed rocket demilitarization (U.S. Environmental Protection Agency, 2002). Five areas on the LHAAP (A–E, fig. 2) have active releases of contaminants to surface water: (active) burning ground no. 3, old landfill, former TNT production area, ground signal test area, and south test area. In addition, 15 other areas are contaminated or possibly contaminated and have the potential for contaminant migration off the LHAAP (U.S. Environmental Protection Agency, 2002). The LHAAP site was designated as the Caddo Lake National Wildlife Refuge in October 2000 and will be transferred to the U.S. Department of the Interior, U.S. Fish and Wildlife Service, as areas are designated appropriate for use as a wildlife refuge (U.S. Environmental Protection Agency, 2002).

Study Design

The study was designed to sample and analyze the bottom sediments in Caddo Lake to indicate whether contaminants associated with historical activities at the now-closed LHAAP are being transported into Caddo Lake and if the amount of contaminants associated with activities at the LHAAP or other potential sources has changed over time. Four natural drainage areas direct runoff and stormwater from the LHAAP into Caddo Lake: Goose Prairie Creek, Central Creek, Harrison Bayou, and Saunders Branch (fig. 2). The estimated percentages of surface water draining from the LHAAP through the four creeks are 30 percent in Goose Prairie Creek, 29 percent in Central Creek, 30 percent in Harrison Bayou, and 11 percent in Saunders Branch (Texas Department of Health, 1999).

Two sediment cores were collected near the shores of the LHAAP, one near the mouth of Goose Prairie Creek (core CDO.GP) and one near the mouths of Central Creek, Harrison Bayou, and Saunders Branch (core CDO.HB) (figs. 1, 2). These two sites were selected to assess the occurrence and trends of contaminants that might enter the lake from the LHAAP and to determine if sediment-bound contaminants presumably from the Goose Prairie Creek drainage area differed from those presumably from the three other drainage areas. Another core was collected from the middle of the main body of the lake (core CDO.MD) (fig. 1). This third site was selected to indicate whether contaminants are being transported into the main body of the lake and whether oil and gas production or industrial activity in Caddo Lake and its drainage area have released contaminants to the lake sediments, such as through improper drilling-mud disposals or oil spills. The CDO.MD core was collected near an abandoned oil pipeline. The fourth core was collected from upper Caddo Lake in an area called Carter Lake (core CDO.CR) (fig. 1). This site was selected to indicate background concentrations of contaminants because the location does not receive runoff from the LHAAP and is far from the area of the lake with dense oil and gas operations. No residential, commercial, or industrial development is near the CDO.CR core site. Industrial activity could be a source of contaminants, however, through atmospheric transport and deposition.

Review of Existing Information

There have been numerous studies of Caddo Lake and its drainage areas because it is such a unique and biologically important area. The topics of studies include local history, habitat, possible contaminant sources (such as on-site studies of the LHAAP), and contaminant concentrations in various media in and around Caddo Lake (for example, Gray, 1955; Cloud and Short, 1993; Crowe, 1996; U.S. Environmental Protection Agency, 2002). This section describes existing information that is readily available.

The ATSDR classifies the LHAAP as a hazardous waste site and has a site contaminants list showing the presence of 233 chemicals in various media on the site (Agency for Toxic Substances and Disease Registry, 2003). Of relevance to this report is the documented presence of arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, nickel, strontium, zinc, 1,1-dichloro-2,2-bis(*p*-chlorophenyl)ethane (*p,p'*-DDE), 1,1,1-trichloro-2,2-bis(*p*-chlorophenyl)ethane (*p,p'*-DDT), dieldrin, and aldrin in the soils or the sediment of the LHAAP, or both. Only three contaminant concentrations for the soils and sediments sampled on-site were reported: 1,290 milligrams per kilogram (mg/kg) of lead and 25.4 mg/kg of dieldrin in the soil and 20,500 mg/kg of barium in the sediment. Mercury was not listed as a contaminant in soil or sediment samples from the LHAAP; however, it does not appear that soil or sediment samples were analyzed for mercury. Mercury was reported at 3.3 micrograms per liter (µg/L) in ground-water samples collected during a health assessment at the plant.

The Texas Department of Health (TDH) released a public health assessment of the LHAAP in July 1999 that stated the site posed no apparent public health hazard (Texas Department of Health, 1999). The public health assessment cites releases of arsenic, barium, chromium, and lead on the LHAAP and their presence in ground water. Production wastes were washed into ponds or buried in landfills until about 1984. Areas identified by the Army as contaminated or potentially contaminated during the Installation Restoration Program by the Department of Defense included burning ground no. 3 where flammable wastes were burned beginning in the early 1950s; the unlined evaporation pond, which received about 61 cubic meters per day (16,000 gallons per day) of waste containing arsenic, barium, chromium, lead, and zinc during 1972–84; the old landfill where TNT wastes were disposed during 1942–44; the former TNT production area; and the ground signal and south test areas where rocket motors and ammunition were tested (fig. 2).

The USACE sampled sediment in Caddo Lake three times. The first round of sampling was in August 2000 and included samples from Caddo Lake near the inlets of Goose Prairie Creek, Harrison Bayou, Central Creek, and Saunders Branch. Samples were analyzed for major and trace elements, organochlorine pesticides, organophosphorus pesticides, herbicides, polychlorinated biphenyls (PCBs), explosives, volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs) that included several PAHs, and dioxins and furans. The second round of sampling was in May 2001 in Clinton Lake (northwestern part of Caddo Lake; fig. 1) to determine background concentrations. The samples were analyzed for major and trace elements and dioxins and furans. The final round of sampling was in March 2002 and focused on sediments near the inlets of Goose Prairie Creek and Harrison Bayou, and in Clinton Lake. Samples were analyzed for eight trace elements (arsenic, cadmium, chromium, copper, lead, nickel, silver, and zinc), PCBs, perchlorate, and dioxins and furans. During all sampling events the bottom-sediment samples were collected to a depth of 0.46 meter (m), and three 0.15-m sediment intervals were analyzed.

The USACE compared sample concentrations to the sediment-quality guidelines (SQGs) of Long and others (1995) to determine which constituents were at levels of concern in regard to adverse biological effects (Cliff Murray, U.S. Army Corps of Engineers, written commun., 2002). The two SQGs recommended by Long and others (1995) were based on data obtained from 89 reports that contained chemical data and simultaneous measures of biological effects: (1) the effects range-low (ER–L) and (2) the effects range-median (ER–M) concentrations. Sediment concentrations below the ER–L were “rarely” associated with adverse biological effects. Concentrations above the ER–L but below the ER–M were “occasionally” associated with adverse biological effects; those above the ER–M were “frequently” associated with adverse biological effects. Several trace elements had concentrations that exceeded the SQGs—that is, concentrations that were above threshold concentrations associated with “occasional” or “frequent” adverse biological effects. Lead, mercury, and silver concentrations exceeded the ER–Ms in the upper two sampled depth intervals (0 to 0.15 and 0.15 to 0.3 m) from the near Goose Prairie Creek inlet site. The samples from Harrison Bayou had one concentration of mercury and one concentration of nickel that exceeded the ER–Ls, both in the 0.15- to 0.3-m interval in two different cores. The top interval from one core collected near Central Creek had concentrations of mercury and nickel that exceeded the ER–Ls. Arsenic, nickel, and mercury concentrations exceeded the ER–Ls in the upper two intervals of the samples from Clinton Lake. The cores collected near Saunders Branch had no trace element concentrations that exceeded the SQGs of Long and others (1995).

There were a few detections of VOCs and SVOCs in the cores from near Goose Prairie Creek. Commonly detected compounds were acetone, carbon disulfide, and phenol. The cores from near Goose Prairie Creek had several PAHs at concentrations of 1,000 to 3,000 micrograms per kilogram (µg/kg). Acetone and carbon disulfide also were detected in the cores from near Harrison Bayou, Central Creek, and Saunders Branch. VOCs were not analyzed in the Clinton Lake cores (U.S. Army Corps of Engineers, written commun., 2002). SQGs are not included for VOCs and SVOCs (Long and others, 1995).

A fish consumption advisory was issued by the TDH in November 1995 that included Caddo Lake and Big Cypress Creek (fig. 1), discouraging the consumption of largemouth bass and freshwater drum because of elevated mercury concentrations (Texas Department of Health, 1995). Caddo Lake is one of five East Texas lakes for which fish consumption advisories have been issued because of elevated mercury concentrations in largemouth bass and freshwater drum. The TDH and TCEQ have sampled several species of fish from Caddo Lake for mercury and have detected concentrations ranging from 0.14 to 1.63 parts per million (ppm) (Crowe, 1996). The mercury is thought to be from atmospheric deposition of nonpoint-source emissions (Texas Department of Health, 1999). Possible sources of emissions of mercury in the Big Cypress Creek Basin include a lignite-fired power plant, a coal-fired power plant, a chicken-processing plant, and a steel company, but emissions distant from the basin could be sources of mercury as well. Mercury was not used at the LHAAP and has not been detected in its permitted discharge (Crowe, 1996). Caddo Lake has favorable conditions for mercury methylation and subsequent uptake into the aquatic food chain, such as low pH, low alkalinity, low dissolved calcium, high dissolved organic carbon, high sulfate, and seasonally flooded wetlands (Lange and others, 1993; Wiener, 1995; Crowe, 1996; Krabbenhoft and others, 2002).

The TCEQ currently (2003) samples Caddo Lake as part of its Surface Water Quality Monitoring Program. A draft version of the 2002 Water Quality Inventory (Texas Natural Resource Conservation Commission, 2002a, p. 11) listed sediment contaminants of concern at two sites (mid-lake and Carter Lake) and their possible sources. Two TCEQ sampling sites were near two of the coring sites of the study documented in this report (CDO.MD and CDO.CR). TCEQ data from March 1996 through February 2001 listed lead, manganese, mercury, and zinc as metals of concern in the samples collected from the mid-lake site. Barium, manganese, mercury, selenium, and zinc were listed as contaminants of concern at the Carter Lake site. At least two possible sources were identified by the TCEQ: industrial activity for lead, manganese, mercury, and zinc at the mid-lake site and for manganese, mercury, selenium, and zinc at the Carter Lake site; oil and gas drilling for barium at the Carter Lake site (because barium is used in drilling mud to increase specific gravity) (Texas Natural Resource Conservation Commission, 2002b).

Crowe (1996) summarized the mercury concentrations in water, sediment, soil, and fish samples collected from the Big Cypress Creek Basin during May–October 1994. Sediment samples were collected from two sites in Caddo Lake using an Eckman dredge. The sites were identical to the TCEQ sampling locations, one in mid-lake and the other in Carter Lake. Mercury concentrations in the sediment samples collected from the mid-lake site ranged from less than 0.01 to 1.57 ppm, and those from the Carter Lake site did not exceed the reporting level of 0.01 ppm.

Possible sources of contamination (PSOCs) were identified by various State agencies and compiled by the USGS as part of the Source Water Assessment Program (SWAP) of the TCEQ. SWAP was developed to comply with 1996 amendments to the Safe Drinking Water Act that require States to develop and implement programs to analyze existing and potential threats to the quality of public drinking water in each State (U.S. Environmental Protection Agency, 2003). The PSOC database included the location of the PSOC, the source of the data, a list of regulated contaminants associated with the PSOC, and the method of introduction of the contaminant into the environment. The majority of PSOCs in the Caddo Lake area are oil and gas wells, but other PSOCs in the area include private and industrial wastewater outfalls, mined land, abandoned municipal solid-waste sites, an airport, roads, a marina, and boat ramps (fig. 3). About 50 aircraft per year use two oiled-dirt runways of the private airport (AirNav.com, 2003). Boating is popular in the lake; however, dense vegetation and numerous obstructions such as trees and oil wells in the lake require careful navigation of the waters. Thus, most probably follow the channels or “boat roads” that are dredged throughout the lake to provide a pathway clear of the thick aquatic vegetation.

The National Atmospheric Deposition Program collected weekly precipitation samples from a site at Longview, Tex. (site TX21), in cooperation with the TCEQ (National Atmospheric Deposition Program, 2003). The Longview site is part of the Mercury Deposition Network, which has been providing precipitation samples for total mercury analysis since 1995. Longview is about 72 kilometers southwest of Caddo Lake.

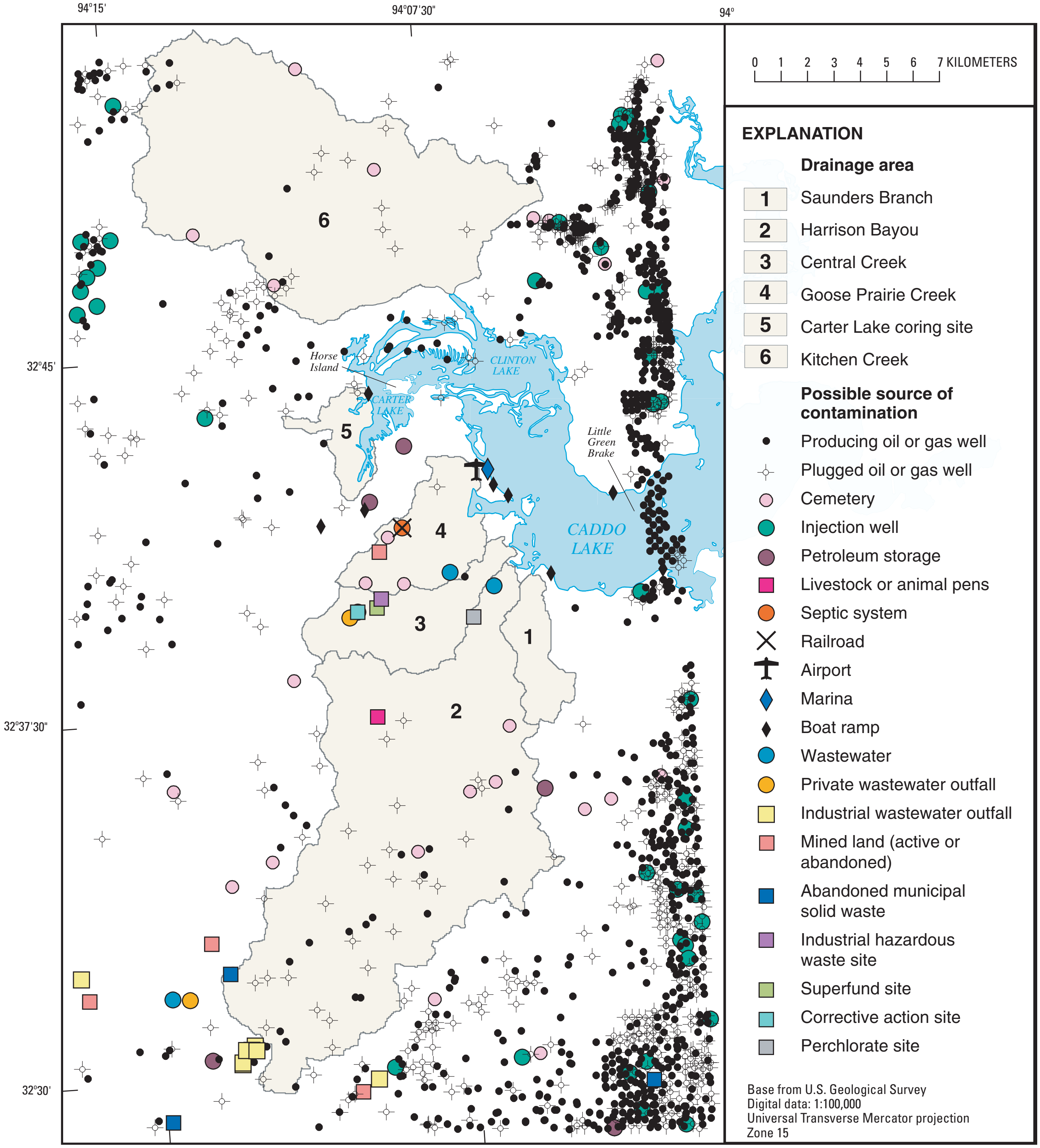


Figure 3. Drainage areas of interest defined using digital elevation models and possible sources of contamination (PSOCs) identified by U.S. Geological Survey Source Water Assessment Program (R.L. Ulery, U.S. Geological Survey, written commun., 2002) in the Caddo Lake, Texas, area.

Table 1. Description of sediment box cores collected from Caddo Lake, Texas, May 2002

[m, meters; cm, centimeters]

Site name	Core identifier (fig. 1)	Latitude and longitude of site	USGS site ID	Water depth (m)	Box core length (cm)
Caddo Lake near Goose Prairie Creek inlet	CDO.GP	N 32°42'08.0" W 94°07'34.6"	324208094073500	0.86	30
Caddo Lake near Harrison Bayou inlet	CDO.HB	N 32°41'13.2" W 94°06'18.7"	324113094061900	1.2	20
Caddo Lake mid-lake (main stem near Little Green Brake)	CDO.MD	N 32°41'02.2" W 94°04'13.6"	324102094041400	2.4	13
Carter Lake near Horse Island	CDO.CR	N 32°44'24.5" W 94°09'45.7"	324424094094600	1.95	25

Complete mercury data were available for 1996–2001. The annual deposition of mercury at the Longview site increased from 9,000 nanograms per square meter (ng/m^2) in 1996 to 15,000 ng/m^2 in 2001 (National Atmospheric Deposition Program, 2003). Sulfate concentrations, a constituent important for mercury methylation (Krabbenhoft and others, 2002), were available for precipitation samples collected during 1983–2001. The average annual sulfate deposition was 17.6 kilograms per hectare (kg/ha) (1.76 grams per square meter [g/m^2]), with peak sulfate deposition occurring in 1991 at 23.7 kg/ha (2.37 g/m^2) (National Atmospheric Deposition Program, 2003).

Acknowledgments

The author thanks Dr. Roy Darville of East Texas Baptist University for taking the USGS sampling team on his boat during lake reconnaissance and sediment coring. Thanks to Dwight Shellman, president of the Caddo Lake Institute, for the use of his dock during USGS sampling activities. Thanks also to Cliff Murray, USACE, for assistance during reconnaissance of the LHAAP and sediment-core collection.

METHODS OF SEDIMENT COLLECTION AND ANALYSIS

This section describes the methods used in the sediment study at Caddo Lake. The methods descriptions will be in the order in which they were used—first, a description of how the sediment cores were collected from Caddo Lake and subsampled for analyses; second, the analytical methods used by the several laboratories to analyze the sediment samples; third, an overview of the methods used to compute and estimate deposition dates for each sample interval; fourth, the criteria used to assess the degree of sediment contamination; then, the trend test used to assess temporal trends is briefly discussed; and finally, sediment-core burdens and focusing factors.

Sediment-Core Collection Method

Sediment box cores were collected by the USGS in May 2002 for chemical analyses of radionuclides (for age dating), organochlorine pesticides, PCBs, PAHs, petroleum biomarkers, forms of carbons, and major and trace elements. Cores were collected from Caddo Lake near the inlet of Goose Prairie Creek (CDO.GP), near the inlet of Harrison Bayou (CDO.HB), in the main stem of the lake (mid-lake) near Little Green Brake (CDO.MD), and in Carter Lake near Horse Island (CDO.CR) (fig. 1; table 1). The samples were collected using a custom-built 14- by 14- by 30-centimeter (cm) aluminum Wildco box corer. An extension rod was attached to the top of the box corer and used to lower the corer onto the bottom and gently press it through the sediment until 20 to 30 cm of sediment was penetrated or the corer could not be pushed deeper because consolidated material was encountered. After a box core was obtained at a sampling site, the core was transported back to shore and subsampled to minimize sediment disturbance.

The box cores were subsampled by placing the box core liner containing the sample onto a piston and pushing the sample up through the core liner. Small intervals of the sediment were sliced off the top of the core and placed into sample containers for analysis. The Goose Prairie Creek and Harrison Bayou cores were subsampled at 1-cm intervals throughout their lengths. The mid-lake box core was subsampled at 0.5-cm intervals from 0 to 10 cm and at 1-cm intervals below 10 cm. The Carter Lake core was subsampled at 0.5-cm intervals from 0 to 5 cm and at 1-cm intervals below 5 cm. Samples for organic analysis were placed into prebaked glass jars, and samples for inorganic analysis were placed in polypropylene jars. All samples were immediately stored on ice and frozen upon arrival at the USGS Austin office. Sample identifiers include the site and depth interval; for example, CDO.GP 1–2 is the 1- to 2-cm depth interval of the Goose Prairie Creek box core.

Push cores also were collected at each sampling site for description. A push core was collected by pushing a hollow cylinder (6-cm diameter) through the sediment until it could not be pushed farther. The push cores were capped and stored in the upright position until the following day when they were extruded, and their color and texture were described (appendix 1). No further analyses were done on the push core samples.

Sediment-Core Analytical Methods

Box core samples from the four coring sites were analyzed for radionuclides (table 2) by Severn Trent Laboratories, Inc., Richland, Wash. (STL Richland), under contract with the USGS National Water Quality Laboratory (NWQL), Denver, Colo. Samples were freeze-dried and ground to a fine powder. Cesium-137 (^{137}Cs) was analyzed by gamma spectroscopy using a high-purity germanium (HPGe) photon detector. Lead-210 (^{210}Pb) and radium-226 (^{226}Ra) were analyzed using a low-energy photon spectrometer (LPES) (Jackie Waddell, Severn Trent Laboratories, Inc., oral commun., 2002).

Box core samples from the four coring sites were analyzed for organochlorine pesticides, PCBs, and PAHs (table 2) by the NWQL. Additionally, selected samples from the mid-lake site were analyzed by the NWQL for petroleum biomarkers (appendix 2.5). A laboratory reporting level (LRL) for each constituent was established by the NWQL for a 25-gram (g) dry sediment weight sample (table 2). The LRL is increased when less than 25 g is submitted to the lab for analysis. Several surficial samples had low dry sediment weights, which resulted in high LRLs. The sample mass available was insufficient for analysis of organochlorine compounds and PAHs in surficial sample CDO.CR 0–0.5.

Samples for analysis of organochlorine pesticides, PCBs, PAHs, and petroleum biomarkers were extracted, isolated, and analyzed using a variation of the procedures of Foreman and others (1995) and Furlong and others (1996). Briefly, sediment was extracted overnight with dichloromethane in a Soxhlet apparatus. Two aliquots of the sample extract were injected into a polystyrene-divinylbenzene gel permeation column (GPC) and eluted with dichloromethane to remove sulfur and partially isolate the target analytes from co-extracted high-molecular-weight interferences such as humic substances. The first aliquot was analyzed for PAHs, alkyl-PAHs, and petroleum biomarkers by capillary-column gas chromatography (GC) with detection by mass spectrometry (MS). The second aliquot was further split into two fractions by combined alumna/silica adsorption chromatography prior to analysis of the organochlorine pesticides and PCBs by dual capillary-column gas chromatography with electron capture detection (GC–ECD).

Variations on the procedures of Furlong and others (1996) for the analysis of PAHs and petroleum biomarkers include the addition of a silica column cleanup step following the GPC step and the use of selected ion monitoring (SIM) MS to reduce chemical interferences and improve detection limits. Eighteen parent PAHs, 10 specific alkyl-PAHs, and the homologues series of alkyl-PAHs were identified for this report (table 2). Concentrations of n-alkanes, three individual biomarkers, and three biomarker classes were reported by the NWQL. The petroleum biomarker results are provided in appendix 2.5; however, the results are not discussed in this report. Comparison of the petroleum biomarker concentrations in the mid-lake core to those of a local crude oil sample was planned, but a crude oil sample was not collected, which made the biomarker results less useful.

Box core samples were analyzed for forms of carbon and major and trace elements (table 2) by the USGS Geologic Discipline laboratory in Denver, Colo. Samples were freeze-dried and ground to a fine powder. Total carbon was analyzed by combustion with an automatic carbon analyzer, inorganic carbon was determined as carbon dioxide by coulometric titration, and organic carbon was computed by difference (Arbogast, 1996). Samples for major and trace elements were

Table 2. Constituents and laboratory reporting levels for samples collected from Caddo Lake, Texas, May 2002

[STL Richland, Severn Trent Laboratories, Inc.; pCi/g, picocuries per gram; NWQL, USGS National Water Quality Laboratory; µg/kg, micrograms per kilogram; GD, USGS Geologic Discipline laboratory; µg/g, micrograms per gram]

Constituent (units)	Laboratory reporting level
Radionuclides (STL Richland)	
Cesium-137 (pCi/g)	0.20
Lead-210 (pCi/g)	.10
Radium-226 (pCi/g)	.40
Organochlorine compounds (NWQL)	
Lindane (µg/kg)	.5
Heptachlor (µg/kg)	.5
Aldrin (µg/kg)	.5
Heptachlor epoxide (µg/kg)	.5
Technical chlordane (µg/kg)	5.0
Endosulfan I (µg/kg)	.5
Dieldrin (µg/kg)	.5
Endrin (µg/kg)	.5
p,p'-DDE (µg/kg)	.5
p,p'-DDD (µg/kg)	.5
p,p'-DDT (µg/kg)	.5
p,p'-Methoxychlor (µg/kg)	2.0
Mirex (µg/kg)	.5
Toxaphene (µg/kg)	50
PCB Aroclor 1242 (µg/kg)	5.0
PCB Aroclor 1254 (µg/kg)	5.0
PCB Aroclor 1260 (µg/kg)	5.0
Polycyclic aromatic hydrocarbons (NWQL)	
Parent compounds:	
Naphthalene (µg/kg)	5
Acenaphthylene (µg/kg)	5
Acenaphthene (µg/kg)	5
9H-Fluorene (µg/kg)	5
Phenanthrene (µg/kg)	5
Anthracene (µg/kg)	5
Polycyclic aromatic hydrocarbons (NWQL)—Cont.	
Parent compounds:—Cont.	
Fluoranthene (µg/kg)	5
Pyrene (µg/kg)	5
Benz(a)anthracene (µg/kg)	5
Chrysene (µg/kg)	5
Benzo(b)fluoranthene (µg/kg)	5
Benzo(k)fluoranthene (µg/kg)	5
Benzo(e)pyrene (µg/kg)	5
Benzo(a)pyrene (µg/kg)	5
Benzo(g,h,i)perylene (µg/kg)	5
Indeno(1,2,3-c,d)pyrene (µg/kg)	5
Dibenzo(a,h)anthracene (µg/kg)	5
Coronene (µg/kg)	5
Alkylated compounds:	
2-Ethylnaphthalene (µg/kg)	5
2,6-Dimethylnaphthalene (µg/kg)	5
1,6-Dimethylnaphthalene (µg/kg)	5
1,2-Dimethylnaphthalene (µg/kg)	5
2,3,6-Trimethylnaphthalene (µg/kg)	5
1-methyl-9H-Fluorene (µg/kg)	5
2-Methylanthracene (µg/kg)	5
4,5-Methylenephenanthrene (µg/kg)	5
1-Methylphenanthrene (µg/kg)	5
1-Methylpyrene (µg/kg)	5
C1-128 isomers (µg/kg)	5
C2-128 isomers (µg/kg)	5
C3-128 isomers (µg/kg)	5
C4-128 isomers (µg/kg)	5
C5-128 isomers (µg/kg)	5

Table 2. Constituents and laboratory reporting levels for samples collected from Caddo Lake, Texas, May 2002—Continued

Constituent (units)	Laboratory reporting level	Constituent (units)	Laboratory reporting level
Polycyclic aromatic hydrocarbons (NWQL)—Cont.		**Major elements (GD)—Cont.**	
Alkylated compounds:—Cont.		Potassium (µg/g)	20
C1-178 isomers (µg/kg)	5	Sodium (µg/g)	6
C2-178 isomers (µg/kg)	5	Titanium (µg/g)	40
C3-178 isomers (µg/kg)	5	**Trace elements (GD)**	
C4-178 isomers (µg/kg)	5	Antimony (µg/g)	.02
C5-178 isomers (µg/kg)	5	Arsenic (µg/g)	.1
C1-202 isomers (µg/kg)	5	Barium (µg/g)	.5
C2-202 isomers (µg/kg)	5	Beryllium (µg/g)	.001
C3-202 isomers (µg/kg)	5	Cadmium (µg/g)	.003
C4-202 isomers (µg/kg)	5	Cerium (µg/g)	.5
C5-202 isomers (µg/kg)	5	Chromium (µg/g)	.2
C1-228 isomers (µg/kg)	5	Cobalt (µg/g)	.1
C2-228 isomers (µg/kg)	5	Copper (µg/g)	.5
C3-228 isomers (µg/kg)	5	Gallium (µg/g)	.006
C4-228 isomers (µg/kg)	5	Lanthanum (µg/g)	.3
C5-228 isomers (µg/kg)	5	Lead (µg/g)	.2
C1-252 isomers (µg/kg)	5	Lithium (µg/g)	.2
C2-252 isomers (µg/kg)	5	Manganese (µg/g)	.2
C3-252 isomers (µg/kg)	5	Mercury (µg/g)	.02
C4-252 isomers (µg/kg)	5	Molybdenum (µg/g)	.1
C5-252 isomers (µg/kg)	5	Nickel (µg/g)	1
Forms of carbon (GD)		Niobium (µg/g)	2
Carbon, carbonate (inorganic) (percent)	.01	Scandium (µg/g)	.3
Carbon, organic (percent)	.01	Selenium (µg/g)	.2
Carbon, total (percent)	.01	Silver (µg/g)	3
Major elements (GD)		Strontium (µg/g)	.05
Aluminum (µg/g)	8	Thorium (µg/g)	.03
Calcium (µg/g)	20	Uranium (µg/g)	.02
Iron (µg/g)	50	Vanadium (µg/g)	.4
Magnesium (µg/g)	.3	Zinc (µg/g)	5
Phosphorus (µg/g)	8		

completely digested using a mixture of hydrochloric-nitric-perchloric-hydrofluoric acids and analyzed for 32 elements by inductively coupled plasma/mass spectrometry (ICP/MS) (Briggs and Meier, 1999). Mercury was analyzed separately by cold vapor atomic absorption spectrometry (Arbogast, 1996).

Sediment-Core Age Dating

Sediment-core deposition dates were based primarily on radionuclide concentrations with supporting evidence for the deposition dates provided by the concentrations of DDD, DDE, and elemental lead. Selected samples from the Goose Prairie Creek and Harrison Bayou sites were analyzed for ^{137}Cs, and those from the mid-lake and Carter Lake sites were analyzed for ^{137}Cs, ^{210}Pb, and ^{226}Ra for age dating purposes. The samples from the Goose Prairie Creek and Harrison Bayou cores were analyzed for only ^{137}Cs because the cores were expected to consist of young sediment deposits (post-1950). The sites were located adjacent to creek inlets where thick sediment deposits tend to accumulate rapidly. Conversely, the samples from the mid-lake and Carter Lake cores were not located adjacent to the mouths of incoming creeks where sediment deposits accumulate rapidly; so the cores were expected to consist of both old (pre-1950) and young (post-1950) sediment deposits, which limited the effectiveness of ^{137}Cs alone for age dating purposes. These samples therefore were analyzed for ^{210}Pb (in addition to ^{137}Cs), which, as will be explained later in this section, can be used to compute the date at which the ^{210}Pb was at the sediment surface. ^{137}Cs (half-life of 30.8 years) was released to the atmosphere through nuclear weapons testing beginning about 1952, and atmospheric concentrations peaked during 1963–64. Wet atmospheric fallout and strong and rapid sorption of ^{137}Cs to soil particles make it useful for studying sediment accumulation rates in lakes and reservoirs (Bolt and others, 1976; Ritchie and McHenry, 1990).

Peak ^{137}Cs activities in the cores were assigned a date of 1964.0, the time at which atmospheric concentrations were highest. The top of each core also was used as a date marker matching the date of sample collection (2002.4). An average mass accumulation rate (MAR) in grams per square centimeter per year was computed using the two depth-date markers, and the porosity and density of each sample were computed using the wet and dry sediment weights. The computation of an average MAR assumed a constant sedimentation rate for the core. The average MAR of the core was used to compute deposition dates for samples between the date markers.

The deposition dates computed for the samples were compared to concentrations of lead and total DDT in the cores for corroboration. The concentration of lead typically peaks in lake sediments deposited during the mid-1970s (Callender and Van Metre, 1997) when the Clean Air Act prompted the phasing out of leaded gasoline. In this report, total DDT is defined as the sum of the para, para (*p,p')* isomers of DDT and its degradates, *p,p'*-DDD and *p,p'*-DDE. The ortho, para (*o,p'*) isomers of the compounds were not analyzed by the laboratory. Total DDT concentrations in lake sediments generally peaked during the mid-1960s (Van Metre and others, 1997), coincident with peak DDT use in the United States (Smith and others, 1988).

The mid-lake and Carter Lake cores were analyzed for ^{210}Pb and ^{226}Ra in addition to ^{137}Cs. ^{210}Pb is a naturally occurring radionuclide in the uranium-238 (^{238}U) decay series:

$$^{238}\mathrm{U} \xrightarrow{4.51\ \mathrm{X}\ 10^{9}\ \mathrm{yr}} {}^{226}\mathrm{Ra} \xrightarrow{1602\ \mathrm{yr}} {}^{222}\mathrm{Rn} \xrightarrow{3.82\ \mathrm{d}} {}^{210}\mathrm{Pb}, \qquad (1)$$
$$\xrightarrow{22.26\ \mathrm{yr}} {}^{206}\mathrm{Pb}$$

(simplified decay sequence from Appleby and Oldfield [1992]). The daughter product of ^{226}Ra, radon-222 (^{222}Rn), is a gas that moves from the soil into the atmosphere where it quickly decays to produce ^{210}Pb. The residence time of ^{210}Pb in the atmosphere is less than 1 month, after which it is removed from the atmosphere through precipitation or dry deposition and adsorbed onto surficial soils. The amount of atmospheric fallout of ^{210}Pb is nearly equal to the amount of ^{222}Ra moving into the atmosphere, and the atmospheric fallout of ^{210}Pb is considered reasonably constant at any given locality (Appleby and Oldfield, 1992; Durrance, 1992). The amount of atmospherically derived ^{210}Pb is called excess or unsupported ^{210}Pb and is the amount of ^{210}Pb present in the soil excluding that contributed by the in-place decay of ^{226}Ra. The unsupported ^{210}Pb in any given layer of a sediment deposit reduces exponentially over time following the radioactive decay law and thus can be used to compute the date at which it was first adsorbed onto the surface of the deposit. The unsupported ^{210}Pb was computed by subtracting the concentration of ^{226}Ra from the concentration of ^{210}Pb.

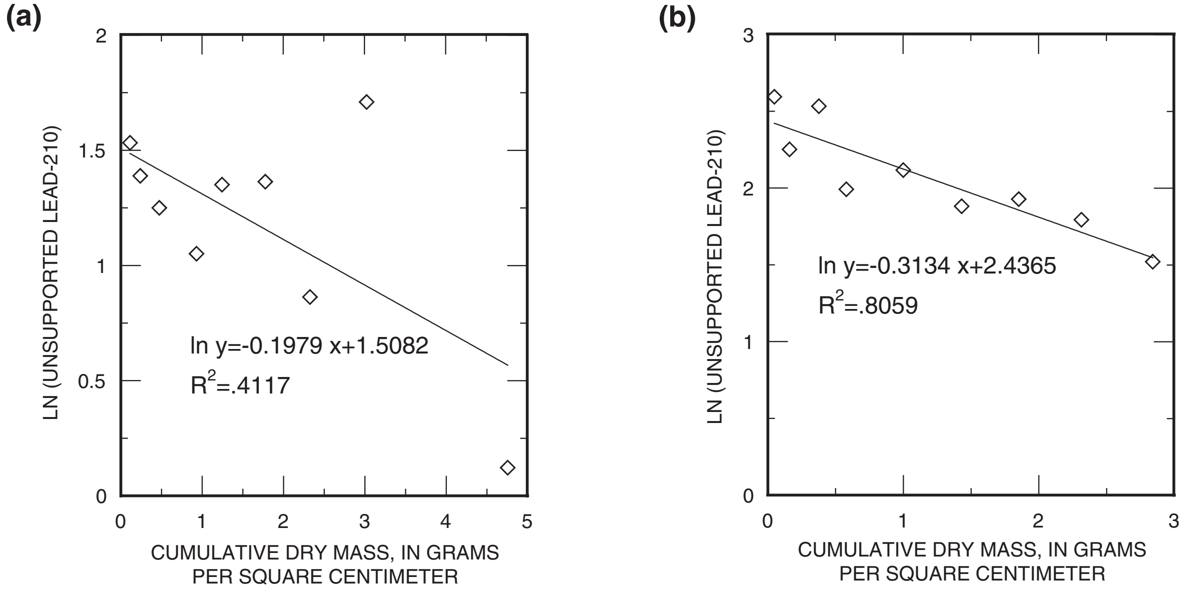


Figure 4. Relation between logarithm of unsupported lead-210 and cumulative dry mass in cores from (a) mid-lake and (b) Carter Lake, Caddo Lake, Texas, May 2002.

The unsupported ^{210}Pb and cumulative dry sediment mass[1] were used in the constant accumulation rate model (which assumes that there is a constant rate of accumulation of unsupported ^{210}Pb and that each layer of the sediment deposited has the same initial unsupported ^{210}Pb) to compute the age of each sample collected from the mid-lake and Carter Lake sites, following the method of Appleby and Oldfield (1992). They define the unsupported ^{210}Pb concentration (activity) in sediments of age *t* as

$$C = C_0 e^{-\lambda_{210} t}, \qquad (2)$$

where

C = concentration in sediments of age *t*, in picocuries per gram;
C_0 = current (2002) surficial concentration, in picocuries per gram;
λ_{210} = ^{210}Pb radioactive decay constant (about 0.03114 per year);
t = cum (grams per centimeter square)/MAR (grams per centimeter square per year), in years; and
cum = cumulative dry sediment mass, in grams per square centimeter.

Using this first-order decay equation, the natural log of unsupported ^{210}Pb was graphed as a function of the cumulative dry mass, and a least-squares line was fit to the data from each of the two sites (fig. 4). The slope of the least-squares line on the graph equals $-\lambda_{210}$/MAR; thus the MAR was obtained by dividing $-\lambda_{210}$ by the computed slope from the graph (actually from the least-squares equation [fig. 4]). The age was then computed as the cumulative mass divided by the MAR. A poor fit to the line can be caused by variable sedimentation rates, variable initial unsupported ^{210}Pb in the layers, or post-depositional sediment mixing (Appleby and Oldfield, 1992).

Caution is needed regarding age dating of the Caddo Lake cores. A thick clay was encountered at the bottom of the cores collected from Goose Prairie Creek, Harrison Bayou, and mid-lake (appendix 1), and age dating indicated that the Goose Prairie Creek and Harrison Bayou cores did not penetrate lacustrine sediment deposited before 1948. Historical records of local precipitation, stream discharge, and lake altitude were investigated for evidence of extremely dry conditions associated with low lake levels around 1948 that could have resulted in the lacustrine sediments of Caddo Lake being exposed, dried, and compacted to form the thick clay layer encountered by the three cores. A record of annual precipitation during 1943–97 for the National Weather Service station in Karnack, Tex.

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[1] Computed by multiplying effective density times thickness of sample interval and summing results for each sample interval over the length of the core.

Table 3. Selected consensus-based sediment-quality guidelines used to assess contaminant concentrations in cores collected from Caddo Lake, Texas, May 2002

[TEC, threshold effect concentration (MacDonald and others, 2000); PEC, probable effect concentration (MacDonald and others, 2000); μg/kg, micrograms per kilogram; μg/g, micrograms per gram]

Constituent	TEC	PEC
Organochlorine compounds (μg/kg)		
DDE	3.16	31.3
DDD	4.88	28.0
Total PCBs	59.8	676
Polycyclic aromatic hydrocarbons (μg/kg)		
Anthracene	57.2	845
Fluorene	77.4	536
Naphthalene	176	561
Phenanthrene	204	1,170
Benz(a)anthracene	108	1,050
Benzo(a)pyrene	150	1,450
Chrysene	166	1,290
Fluoranthene	423	2,230
Polycyclic aromatic hydrocarbons (μg/kg)—Cont.		
Pyrene	195	1,520
Total SQG PAH[1]	1,610	22,800
Trace elements (μg/g)		
Arsenic	9.79	33.0
Cadmium	.99	4.98
Chromium	43.4	111
Copper	31.6	149
Lead	35.8	128
Mercury	.18	1.06
Nickel	22.7	48.6
Zinc	121	459

[1] Sum of 13 PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, and benzo(a)pyrene [Swartz, 1999]).

(fig. 1) showed a drought in the early 1950s (fig. 5). The lowest annual precipitation on record at the weather station was 60.4 cm in 1951. The average annual precipitation over the period of record was 120.7 cm. The monthly mean discharge in Big Cypress Creek near Jefferson, Tex. (USGS station 07346000) was low (near zero) several times around 1950; however, comparably low stream discharge occurred regularly throughout the years of record (fig. 5). Similarly, the lake water level was low a few times around 1950 and was comparably low in other years. Stream discharge and lake water-level records did not support the hypothesis of subaerial exposure of lacustrine sediments in Caddo Lake around 1950; nonetheless, the coring tools could not penetrate the thick clay layer, and sediment deposited at the Goose Prairie Creek and Harrison Bayou sites before 1948 was not collected.

Assessing Contaminant Levels

Sediment concentrations can be compared to SQGs to indicate the degree of contamination of sediments. SQGs are based on numerous field studies and toxicity tests and have been shown to be reasonable predictors of toxicity or other adverse effects on biota from contaminants in sediments (MacDonald and others, 2000). SQGs are derived through a variety of laboratory, field, and theoretical methods and are not definitive for determining sediment toxicity to benthic biota; only sediment toxicity tests can determine this (Long and others, 1995; MacDonald and others, 2000). In addition, SQGs cannot be extrapolated to determine the possibility that the sediments might be harmful to fish, aquatic plants, or humans.

Two consensus-based SQGs are used in this report: the threshold effect concentration (TEC) and the probable effect concentration (PEC). The TEC represents the concentration below which adverse effects to benthic biota rarely occur, and the PEC represents the concentration above which adverse effects are expected to occur frequently (MacDonald and others, 2000). TECs and PECs for a number of substances analyzed for in this report are listed in table 3. The consensus-based SQGs were developed using a number of published SQGs. Some of the SQGs used to develop the consensus-based SQGs were normalized with organic carbon (for example, Swartz, 1999). These organic carbon normalized SQGs were converted to

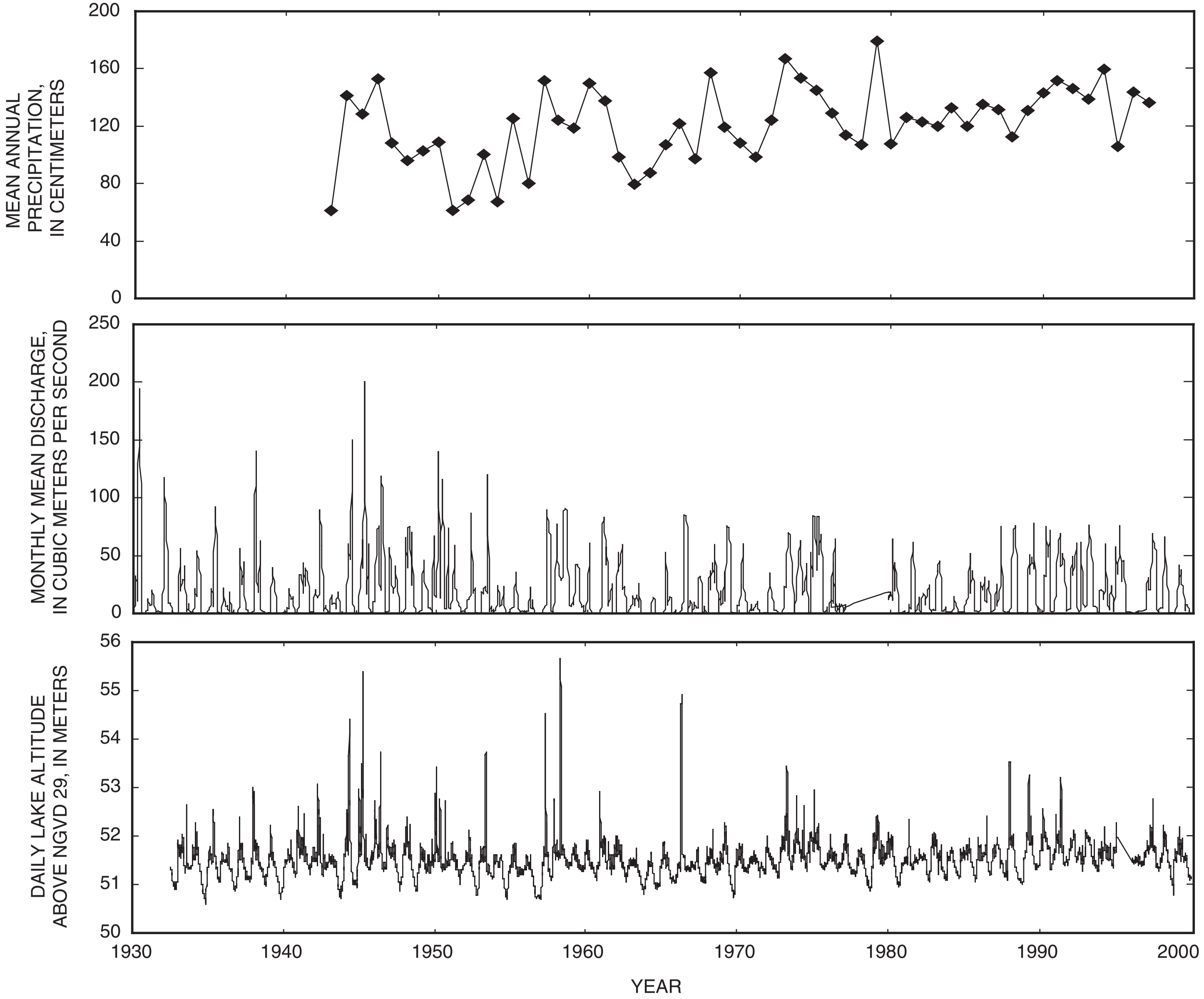


Figure 5. Mean annual precipitation at National Weather Service station in Karnack, Texas, 1943–97 (National Climatic Data Center, 1999); monthly mean discharge in Big Cypress Creek near Jefferson, Texas, 1930–2000 (U.S. Geological Survey, 2003); and daily lake altitude at Caddo Lake dam, Louisiana, 1932–2000 (U.S. Army Corps of Engineers, 1932–98; 1999–2000 data, U.S. Army Corps of Engineers, unpub. data, 2002).

dry-weight normalized values at 1-percent organic carbon (MacDonald and others, 2000). Previous studies have shown that dry-weight normalized SQGs predict sediment toxicity as well or better than organic carbon normalized SQGs in field-collected sediment samples (Barrick and others, 1988; Long and others, 1995; Ingersoll and others, 1996; U.S. Environmental Protection Agency, 1996; MacDonald, 1997).

Trend Testing

Time series of concentrations of selected compounds (those with three or more detections) were tested for statistically significant trends using Kendall's tau (Helsel and Hirsch, 1992). Positive or negative trends were considered significant at the 95-percent confidence level (p-value $\leq$.05). Kendall's tau is a nonparametric (normal distribution of data not required) test based on ranks. Thus the outcome of a trend test is based on the rank order of concentrations rather than actual concentrations.

The NWQL qualifies some organic compound concentrations with an "E" (for estimated), which signifies that although the compounds are qualitatively identified as present, their reported concentrations have greater uncertainty than those reported without qualification (G.L. Cottrell, U.S. Geological Survey, written commun., 2001). In some cases, estimated concentrations of organic compounds reported by the NWQL were less than the LRL. (See Childress and others [1999] for an explanation of conditions under which estimated concentrations can be less than LRLs.) However, the NWQL reports nondetections as "less than the LRL" (Childress and others, 1999). Nondetections were included in trend testing of organic compounds. Nondetections commonly are set equal to LRLs in such tests; but to avoid situations in which nondetections (concentration = LRL) ranked higher than some estimated concentrations (concentration < LRL), nondetections were set equal to values just below the lowest estimated concentration for each compound tested, rather than leaving them at the LRL as reported by the NWQL.

No concentrations of major and trace elements reported by the Geologic Discipline laboratory were less than respective LRLs. Thus, for trend testing, nondetections reported as less than the LRL were set equal to the LRL.

Sediment-Core Burden and Focusing Factor

"Burden" is a term for the amount per unit area of a contaminant in a sediment core. Burdens of ^{137}Cs and mercury were computed as an interim step in estimating what the atmospheric fallout rate of mercury would have to have been to account for the accumulated mercury in a core and thus provide information on whether the predominant source of mercury in a core is atmospheric fallout or point and nonpoint sources in the Caddo Lake drainage area. The ^{137}Cs burden for each sample interval, in picocuries per square centimeter, was computed by multiplying the ^{137}Cs concentration, in picocuries per gram, by the dry mass of sediment, in grams per square centimeter. The interval burdens were summed over the length of the core to yield the ^{137}Cs burden for the core (ϕ_c), in a manner comparable to that of Van Metre and others (1997).

Similarly, the mercury burden for each sample interval in a core, in micrograms per square centimeter, was computed by multiplying the mercury concentration of the interval by the dry mass of sediment and summing the interval burdens over the length of the core to yield the mercury burden for the core (ϕ_{hg}).

"Focusing factor" (FF) is the ratio of the ϕ_c to the model-predicted (from the fallout prediction model of Sarmiento and Gwinn [1986]) atmospheric fallout of ^{137}Cs for the coring site. FF provides an estimate of the degree of focusing of particle-associated contaminants from the drainage area and other parts of a lake to the coring site (Van Metre and others, 1997). An FF near unity implies that the site is influenced only by atmospheric fallout. The ^{137}Cs atmospheric fallout flux (mass per unit area) was computed for the Caddo Lake area using the fallout prediction model of Sarmiento and Gwinn (1986). Precipitation data from the National Weather Service station at Karnack was used in the model. Monthly ^{137}Cs fallout was decay-corrected using the core collection date of the Caddo Lake sampling and summed over the period 1954–74 (the period upon which the prediction model is based) to yield the cumulative ^{137}Cs fallout deposition (ϕ_f). FF for each core was then computed by dividing ϕ_c by ϕ_f.

To estimate what the atmospheric fallout rate of mercury would have to have been to account for the accumulated mercury in a core, ϕ_{hg} is normalized (divided) by FF. ^{137}Cs is supplied to lakes only by atmospheric fallout on the lake and its drainage area—therefore normalized burdens represent the estimated cumulative atmospheric loading of mercury over the drainage area that would be necessary to supply the measured core burdens, assuming no point and nonpoint sources in the drainage area (Van Metre and others, 1997).

QUALITY ASSURANCE OF CHEMICAL DATA

Quality-control (QC) samples are included with each group of environmental samples analyzed to assure the quality of the analytical results. Analytical results from two types of duplicate samples are included in this report—environmental duplicates and laboratory duplicates. An environmental duplicate is a sample that was split into two jars at the time of collection, and both jars were submitted to the laboratory for analysis. A laboratory duplicate is a single sample that was split at the laboratory during preparation and analyzed in duplicate.

Three QC samples were included with each group of environmental samples analyzed for radionuclides: a laboratory duplicate, a blank, and a laboratory control sample (LCS). A blank is a sample of reagent (deionized) water. An LCS is a reference material used by the laboratory to assess the recovery of radionuclides by the analytical instruments. The USGS and STL Richland have established contractually required detection limits for the environmental and QC samples; however, the contractually required detection limits were not met for the environmental samples because the sediment available was insufficient for analysis. STL Richland reports overall method uncertainty estimates with their analytical results, which are listed with sample concentrations in appendix 2.1 (Jackie Waddell, Severn Trent Laboratories, Inc., written commun., 2002).

The analytical results for each group of samples analyzed for radionuclides included blank concentration, percent recovery of the LCS, and relative percent difference (RPD) of the duplicate (appendix 3.1). The RPD is a measure of the variability in the concentrations produced by the analytical method. The RPD was computed for each pair of duplicate samples using the equation

$$\mathrm{RPD} = 100 \times \left| \frac{\mathrm{sample1} - \mathrm{sample2}}{\left(\frac{\mathrm{sample1} + \mathrm{sample2}}{2}\right)} \right| , \qquad (3)$$

where sample 1 and sample 2 are the concentrations of individual compounds in duplicate samples.

The overall median RPD of duplicate analyses for ^{137}Cs is 8.7 percent. There were two duplicate analyses for ^{226}Ra (RPD = 16.10 and 5.65 percent), but ^{210}Pb was not analyzed in duplicate.

Four QC samples were included with each group of environmental samples analyzed for organic compounds: a laboratory duplicate, a blank, a spiked reagent sample, and a certified reference material (CRM). The overall median RPD of duplicate analyses for organochlorine compounds is 15.4 percent and for PAHs, 10.0 percent (appendixes 3.2, 3.3).

There were no detections in the organochlorine blank samples, spike recoveries for four of the five groups analyzed were within acceptable limits, and the CRM concentrations for three of the five groups were within an acceptable range (appendix 3.2). Group number 200221106 had low spike recoveries for mirex and PCB Aroclor 1254 because of a problem in the preparation of the spike at the NWQL. Mirex and PCB Aroclor 1254 were not detected in any of the environmental samples analyzed in group number 200221106. Mirex rarely has been detected in lake-bottom sediments collected as part of the USGS National Water-Quality Assessment Program, but PCB Aroclor 1254 frequently has been detected (P.C. Van Metre, U.S. Geological Survey, unpub. data, 2003). The LRL for PCB Aroclor 1254 in group number 200221106 ranged from 5.0 to 150 µg/kg. Despite the low spike recoveries for this group, the nondetections of PCB Aroclor 1254 were believed to be credible because most samples had low LRLs (5.0 to 15 µg/kg), and the two samples with the highest LRLs (70 and 150 µg/kg for samples CDO.GP 0–1 and CDO.CR 4.5–5, respectively) were not expected to have detectable concentrations of PCB Aroclor 1254. PCB Aroclor 1254 was not expected in CDO.CR 4.5–5 because no other samples from that core had detections of PCBs. The CRM concentrations were below the acceptable limits in group number 200220603 (CDO.HB; CDO.GP) for heptachlor, aldrin, heptachlor epoxide, endosulfan, dieldrin, DDE, and endrin; and in group number 200222605 (CDO.MD) for heptachlor epoxide, endosulfan, and dieldrin. The CRM concentrations were low because of problems in preparation of the sample at the NWQL; however, environmental sample results are not affected by the CRM recoveries.

PAH concentrations were low (estimated [E] values less than LRL) in some blank samples for all groups, PAH spike recoveries were considered normal (the NWQL has not established acceptable recovery ranges), and one compound concentration was below the acceptable CRM limits. Group number 8022R02177

(CDO.HB) had the most detections in its blank sample with low concentrations of naphthalene (E0.15 µg/kg), 2,6-dimethylnaphthalene (E0.44 µg/kg), acenaphthene (E0.18 µg/kg), phenanthrene (E0.24 µg/kg), anthracene (E0.54 µg/kg), fluoranthene (E0.61 µg/kg), pyrene (E0.62 µg/kg), and chrysene (E0.28 µg/kg). The blanks in group number 8022R02171 (CDO.MD) had one detection of naphthalene (E0.23 µg/kg), and group number 8022R02169 (CDO.GP; CDO.HB) had one detection of 2,6-dimethylnaphthalene (E0.46 µg/kg). The LRL for the blank is 5.0 µg/kg, so these detections were considered insignificant. The spike recoveries ranged from 41.4 to 98.7 percent with a median recovery of 70.5 percent. Spike recoveries typically were lowest for coronene and highest for dibenzo(a,h)-anthracene. The NWQL used CRM 362 for groups 8022R02169, 8022R02171, and 8022R02177; and CRM 354 for group number 8022R02165. The CRM is a commercially available organic soil from Environmental Resource Associates Arvada, Colo. The percent recovered of pyrene was 110.2 in group number 8022R02165 (CDO.CR), above the acceptable range of 32 to 90 percent.

QC samples included with samples analyzed for major and trace elements were duplicate and triplicate samples, several blanks, and standard reference materials (SRMs). The blanks were not analyzed for forms of carbon or mercury but were analyzed for major and other trace elements. The SRMs used by the laboratory were the National Research Council Canada Marine Estuarine Sediment (MESS–1), National Institute of Standards and Technology Buffalo River Sediment (NIST 2704), USGS Cody Shale (SCO–1), USGS Marine Sediment (MAG–1), and Chinese Quangxi Province Stream Sediment (GSD–8).

The environmental samples were analyzed in two groups, and two duplicates and one triplicate were included with each group (appendix 3.4). The RPD was computed for a triplicate sample by comparing triplicate sample 1 to triplicate sample 2, triplicate sample 1 to triplicate sample 3, and triplicate sample 2 to triplicate sample 3. The overall median RPD for all duplicate and triplicate samples analyzed for forms of carbon and major and trace elements was 2.4 percent. Four blanks were analyzed in group number 3959: two of the blanks had arsenic concentrations of 0.1 and 0.2 microgram per gram (µg/g), respectively; the LRL for arsenic is 0.1 µg/g. Three of the blanks had beryllium concentrations of 0.003, 0.006, and 0.007 µg/g, respectively; the LRL for beryllium is 0.001 µg/g. The range of arsenic and beryllium concentrations in the environmental samples included in group 3959 was 12 to 15 µg/g and 2.9 to 3.5 µg/g, respectively (appendix 2.4). Group number 3958 included three blank samples, which yielded two detections of beryllium, 0.008 and 0.01 µg/g. The concentration of beryllium in the environmental samples in group 3958 ranged from 1.3 to 2.6 µg/g. The detections of arsenic and beryllium in the blank samples were considered negligible relative to concentrations in environmental samples. SRMs were analyzed with both groups, but MESS–1 was not used in group number 3958. The concentrations obtained by laboratory analysis of the SRMs were compared to the published SRM values (Xuejing and Mingcai, 1985; Potts and others, 1992), and the RPD was computed. The median RPD of the SRMs analyzed in group 3959 was 5.3 percent, and the RPD for group 3958 was 4.7 percent.

OCCURRENCE AND TRENDS

The findings on occurrence and trends are grouped by coring location. Sources of mercury based on burden and FF computations, a comparison of findings to those of other lakes, and implications of the findings relative to drainage area sources are discussed in this section.

Goose Prairie Creek

Age Dating and Sedimentation Rates

The Goose Prairie Creek core was analyzed for ^{137}Cs for age dating. The core has a clearly defined ^{137}Cs peak at 20.5 cm that was used as the 1964.0 date-depth marker (fig. 6a). The ^{137}Cs peak was used to compute a constant average MAR of 0.15 gram per square centimeter per year (g/cm^2-yr) for the core and deposition dates for the remaining samples. Sediment deposition dates were computed back to about 1948 for the Goose Prairie Creek core. DDE concentrations were variable throughout the core, and a clear peak is not well defined. The highest DDE concentration was dated as 1969, which was slightly later than the time of peak historical use of the pesticide but reasonable in the context of possible transport through the drainage area and error associated with age-dating techniques. The lead profile had a pronounced peak, which was dated as 1961 on the basis of the MAR computed from the ^{137}Cs peak. The lead peak in the Goose Prairie Creek core was dated much earlier than lead peaks in the 1970s in most urban lakes (Callender and Van Metre, 1997); however, lead

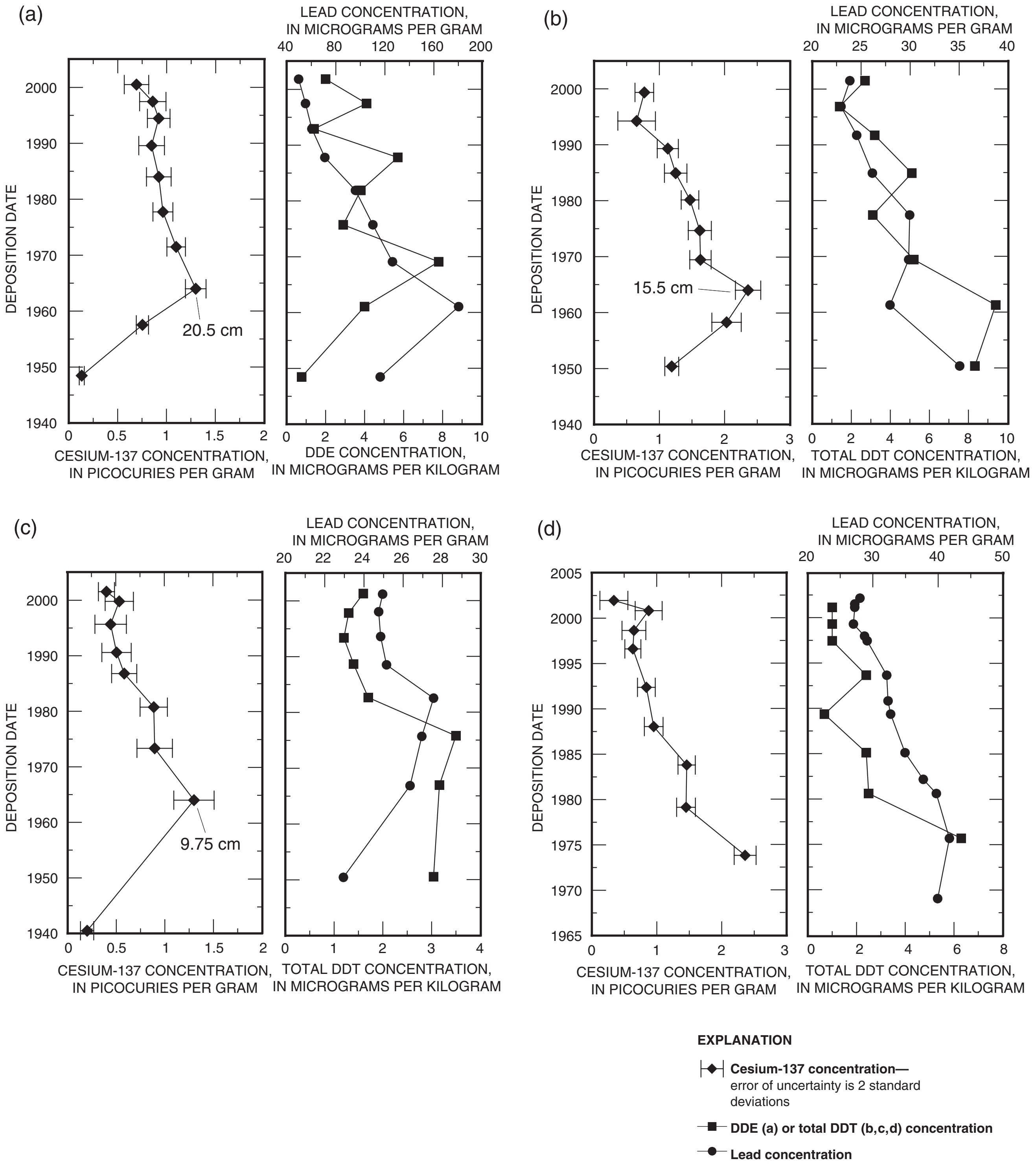


Figure 6. Concentrations of selected constituents used to compute sediment deposition dates in cores collected at (a) Goose Prairie Creek, (b) Harrison Bayou, (c) mid-lake, and (d) Carter Lake sites, Caddo Lake, Texas, May 2002.

concentrations at this site could reflect historical industrial releases and not urban use of leaded gasoline.

Neither DDE nor lead was good corroborating evidence for the ^{137}Cs-based age dates; however the ^{137}Cs profile shows a clear peak and was considered more reliable than the DDE and lead-concentration profiles. It is unlikely that the sediment accumulation rate at the Goose Prairie Creek site was constant over the period of deposition represented in the core, but no additional date-depth markers were in the core (such as a pre-reservoir surface) to use for the computation of more than one MAR; therefore, an uncertainty of ±5 years was estimated for the age dates of the samples in the Goose Prairie Creek core.

Contaminant Occurrence and Trends

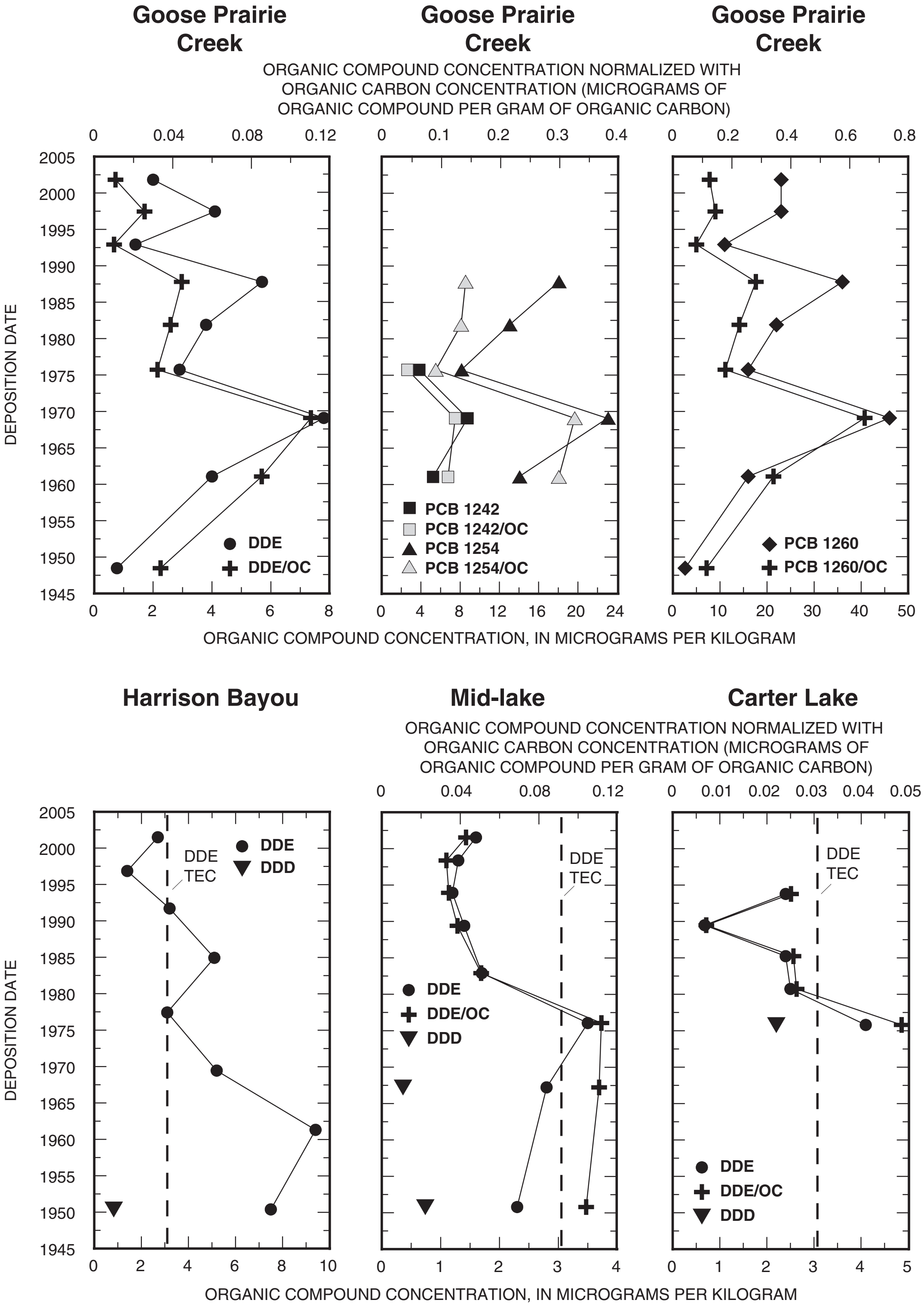
The only organochlorine compounds detected in the Goose Prairie Creek core were DDE and PCB Aroclors 1242, 1254, and 1260 (fig. 7; appendix 2.2). DDE and PCB Aroclor 1260 were detected in all samples analyzed for organochlorines. PCB Aroclor 1242 was detected in samples deposited between 1961 and 1975, and PCB Aroclor 1254 was detected in samples deposited between 1961 and 1987. Fifty-six percent of the sample concentrations of DDE in the Goose Prairie Creek core exceeded the consensus-based SQG TEC for DDE, but they were well below the PEC (table 3; appendix 2.4). An SQG was available for total PCB, which was computed as the sum of PCB Aroclors 1242, 1254, and 1260. Estimated concentrations were included in the summation. Only one sample concentration, deposited about 1969 before restrictions on the use of PCBs, exceeded the TEC for total PCB (table 3; appendix 2.2).

The organic compounds discussed in this report have been shown to preferentially sorb to the organic matter in sediment (Elzerman and Coates, 1987; Schwarzenbach and others, 1993); therefore, a trend in organic carbon could affect the trends of organic compounds. For this reason, the concentrations of organic compounds that were to be trend tested were normalized with (divided by) the concentrations of organic carbon if organic carbon had a statistically significant trend. Organic carbon had a significant upward trend in the Goose Prairie Creek core (Kendall's $\tau = 1$, $p = 0$); therefore, the organochlorine compounds with sufficient numbers of detections for trend testing were normalized with organic carbon before trend testing. Organic carbon normalized PCB Aroclors 1242 and 1254 had significant downward trends (Kendall's $\tau = -.83$ and $-.67$, p-value = .01 and .01, respectively). No other organic carbon normalized organochlorines had significant trends (table 4).

Numerous PAHs were detected, although their concentrations were low (appendix 2.3), comparable to concentrations in lakes in remote areas (P.C. Van Metre, U.S. Geological Survey, unpub. data, 2003) and to those in a core from a forested area of Lake Houston (Van Metre and Sneck-Fahrer, 2002). The naphthalene homologues with two alkyl groups (C2-alkylated naphthalenes), particularly 2,6-dimethylnaphthalene, and fluoranthene had the highest concentrations (fig. 8). No consensus-based SQG has been established for 2,6-dimethylnaphthalene. None of the samples analyzed from the Goose Prairie Creek core had concentrations of individual PAH compounds higher than the respective TEC. Thirty-six of the 53 PAH compounds analyzed (includes parent compounds and alkylated homologues) were detected in the Goose Prairie Creek samples analyzed. Nineteen individual organic carbon normalized PAHs had statistically significant trends—all downward (table 5).

A consensus-based SQG, or TEC, was available for the sum of 13 PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, and benzo(a)pyrene [Swartz, 1999]), which is referred to as "total SQG PAH" in this report. The most recently deposited sample in the Goose Prairie Creek core had the highest total SQG PAH concentration, which was about one-sixth of the TEC. A significant downward trend was indicated for organic carbon normalized total SQG PAH concentrations in the Goose Prairie Creek core (Kendall's $\tau = -.67$, $p = .01$).

Another commonly studied group of PAHs, called "combustion PAH," is the sum of ten 4- and 5-ringed parent compounds (fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(e)pyrene, benzo(a)-pyrene, indeno(1,2,3-c,d)pyrene, and benzo(g,h,i)-perylene) that are produced by high-temperature burning of fuels and are independent of fuel type (Prahl and Carpenter, 1983). The combustion PAH in the Goose Prairie Creek core did not exceed 260 μg/kg, and the organic carbon normalized combustion PAH had a significant downward trend (Kendall's $\tau = -.61$, $p = .02$). As an indicator of general PAH source, the ratio of concentrations of 2- and 3-ringed compounds plus homologues to the 4- and 5-ringed combustion



EXPLANATION

OC, organic carbon

TEC, threshold effect concentration (MacDonald and others, 2000)

Figure 7. Organochlorine pesticide (DDE and DDD) and polychlorinated biphenyl (PCB) concentrations in cores collected from Caddo Lake, Texas, May 2002.

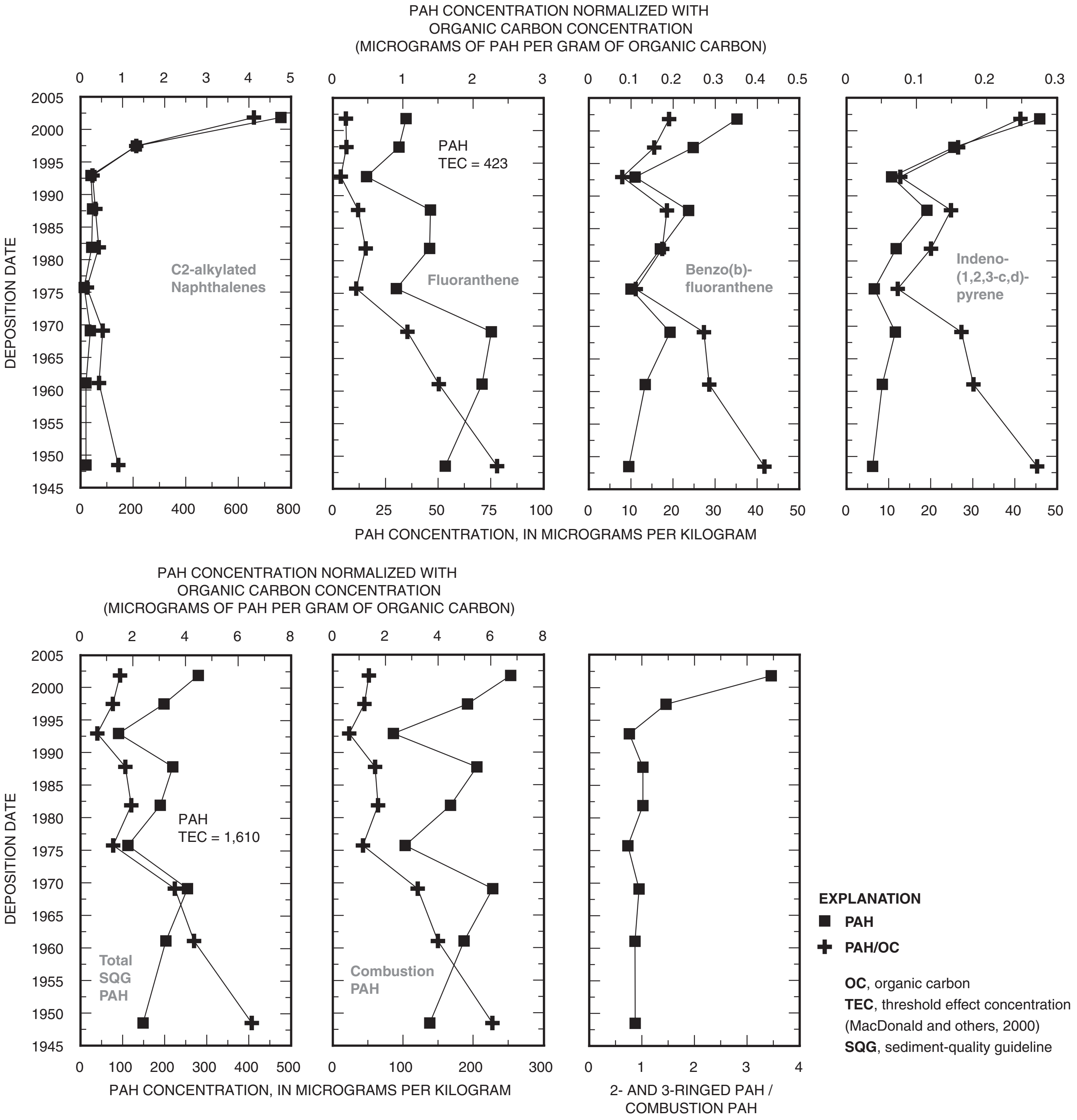


Figure 8. Selected polycyclic aromatic hydrocarbon (PAH) concentrations and trends in core collected near Goose Prairie Creek, Caddo Lake, Texas, May 2002.

PAH was computed for each sample analyzed. The 2- and 3-ringed and alkylated homologue compounds are not typically produced by combustion and are more commonly from uncombusted PAH sources such as crude oil, vehicular fuels, crankcase oil, and organic matter. If the ratio of uncombusted to combusted PAH concentrations is greater than 1, the PAH mixture in the sediments is primarily from uncombusted sources; if the ratio is less than 1, the mixture is primarily from combusted sources. The ratio was about 1 throughout the

Table 4. Summary of trend testing results for organochlorine compounds detected in sediment cores from Caddo Lake, Texas, May 2002—Kendall's tau correlation of concentration with deposition date

[Significant trends at 95-percent confidence level shown in bold; --, not tested]

Organochlorine compound	Goose Prairie Creek inlet				Harrison Bayou inlet	
	Non-normalized concentrations		Organic carbon normalized concentrations		Non-normalized concentrations	
	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value
Organic carbon	**1**	**0**	--	--	.11	.71
DDE	0	1	-.44	.10	**-.71**	**.01**
DDD	--	--	--	--	--	--
Total DDT	0	1	-.44	.10	**-.71**	**.01**
PCB Aroclor 1242	-.47	.08	**-.83**	**.01**	--	--
PCB Aroclor 1254	-.30	.25	**-.67**	**.01**	--	--
PCB Aroclor 1260	.29	.28	-.28	.30	--	--
Total PCB	-.11	.67	-.28	.30	--	--

Organochlorine compound	Mid-lake (main stem near Little Green Brake)				Carter Lake near Horse Island			
	Non-normalized concentrations		Organic carbon normalized concentrations		Non-normalized concentrations		Organic carbon normalized concentrations	
	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value
Organic carbon	**.86**	**0**	--	--	**.73**	**0**	--	--
DDE	-.50	.08	**-.57**	**.048**	**-.85**	**.003**	**-0.86**	**.003**
DDD	--	--	--	--	--	--	--	--
Total DDT	-.50	.08	**-.79**	**.01**	**-.85**	**.003**	**-.86**	**.003**
PCB Aroclor 1242	--	--	--	--	--	--	--	--
PCB Aroclor 1254	--	--	--	--	--	--	--	--
PCB Aroclor 1260	--	--	--	--	--	--	--	--
Total PCB	--	--	--	--	--	--	--	--

core, until about 1995 when the ratio increased (fig. 8), indicating predominance of uncombusted sources in recent years.

The Goose Prairie Creek core was analyzed for eight major elements and 26 trace elements (appendix 2.4). Several major elements compose the bulk of geologic materials, although some also are derived from human activities. For example, phosphorus is a nutrient essential to plant growth and is used in fertilizers. Calcium is a secondary essential plant nutrient and also is used in fertilizers (Hem, 1985; James, 1993).

Several major elements had significant trends in the sediment cores (table 6): potassium (τ = -.89, p = 0), sodium (τ = -.78, p = 0), and titanium (τ = -1, p = 0) had downward trends, and calcium (τ = 1, p = 0) and phosphorus (τ = .89, p = 0) had upward trends.

Aluminum, iron, and titanium are stable elements; significant trends in stable elements often are associated with changes in sedimentation rates. In the Goose Prairie Creek core, no trend was indicated for aluminum or iron concentrations, although titanium concentrations had a significant downward trend. The

Table 5. Summary of trend testing results for polycyclic aromatic hydrocarbon compounds detected in sediment cores from Caddo Lake, Texas, May 2002—Kendall’s tau correlation of concentration with deposition date

[Significant trends at 95-percent confidence level (p-value <.05) shown in bold; --, not tested]

Polycyclic aromatic hydrocarbon	Goose Prairie Creek inlet				Harrison Bayou inlet	
	Non-normalized concentrations		Organic carbon-normalized concentrations		Non-normalized concentrations	
	Kendall’s tau	p-value	Kendall’s tau	p-value	Kendall’s tau	p-value
Organic carbon	**1**	**0**	--	--	.11	.71
Naphthalene	.48	.07	-.44	.10	-.04	.90
2,6-Dimethylnaphthalene	**.78**	**0**	.22	.40	.21	.46
1,6-Dimethylnaphthalene	**.78**	**0**	.11	.68	.14	.62
C2-128 isomers	**.72**	**.01**	.06	.83	.29	.32
Acenaphthylene	**.65**	**.02**	-.33	.21	-.18	.54
1,2-Dimethylnaphthalene	**.83**	**0**	.17	.53	.25	.38
Acenaphthene	.72	.01	-.17	.53	-.04	.90
C3-128 isomers	-.37	.17	**-.72**	**.01**	.49	.09
2,3,6-Trimethylnaphthalene	.03	.92	**-.78**	**0**	.29	.32
9H-Fluorene	-.17	.53	**-.67**	**.007**	**.64**	**.03**
1-methyl-9H-Fluorene	.39	.14	-.50	.06	**-.64**	**.03**
Phenanthrene	.44	.10	-.50	.06	.21	.46
Anthracene	**.54**	**.04**	-.50	.06	.29	.32
2-Methylanthracene	-.26	.33	**-.83**	**0**	--	--
4,5-Methylenephenanthrene	-.17	.53	**-.78**	**.004**	-.55	.06
C1-178 isomers	-.44	.10	**-.89**	**0**	--	--
1-Methylphenanthrene	0	1	**-.67**	**.01**	0	1
C2-178 isomers	**-.67**	**.01**	**-.94**	**0**	-.53	.06
Fluoranthene	-.39	.14	**-.78**	**.004**	.14	.62
Pyrene	-.33	.21	**-.78**	**.004**	0	1
C3-178 isomers	**-.61**	**.02**	**-.83**	**0**	--	--
1-Methylpyrene	.20	.46	**-.61**	**.02**	-.48	.09
C1-202 isomers	**-.65**	**.02**	**-.83**	**0**	--	--
C2-202 isomers	.40	.13	-.33	.21	--	--
Benz(a)anthracene	.44	.10	**-.50**	**.06**	.40	.17
Chrysene	.20	.46	**-.67**	**.01**	.14	.62
C1-228 isomers	-.18	.49	**-.89**	**0**	--	--
Benzo(b)fluoranthene	**.61**	**.02**	-.44	.10	.14	.62
Benzo(k)fluoranthene	.44	.10	-.44	.10	.07	.80
Benzo(e)pyrene	.48	.07	-.39	.14	.14	.62
Benzo(a)pyrene	.61	.02	-.39	.14	-.07	.80
C1-252 isomers	--	--	--	--	--	--
Benzo(g,h,i)perylene	0	1	**-.72**	**.01**	-.14	.62
Indeno(1,2,3-c,d)pyrene	**.72**	**.01**	-.17	.53	.07	.80
Dibenzo(a,h)anthracene	-.03	.91	**-.61**	**.02**	-.53	.06
Coronene	-.20	.45	**-.83**	**0**	.18	.53
Total SQG PAH	.17	.53	**-.67**	**.01**	.14	.62
Combustion PAH	.22	.40	**-.61**	**.02**	.14	.62

Table 5. Summary of trend testing results for polycyclic aromatic hydrocarbon compounds detected in sediment cores from Caddo Lake, Texas, May 2002—Kendall's tau correlation of concentration with deposition date—Continued

Polycyclic aromatic hydrocarbon	Mid-lake (main stem near Little Green Brake)				Carter Lake near Horse Island			
	Non-normalized concentrations		Organic carbon-normalized concentrations		Non-normalized concentrations		Organic carbon-normalized concentrations	
	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value
Organic carbon	**.86**	**0**	--	--	**.73**	**0**	--	--
Naphthalene	-.11	.71	-.29	.32	.32	.26	.07	.80
2,6-Dimethylnaphthalene	.36	.22	.21	.46	.64	.06	.50	.08
1,6-Dimethylnaphthalene	**.57**	**.05**	.36	.22	.50	.08	.50	.08
C2-128 isomers	.36	.22	.21	.46	**.64**	**.03**	.50	.08
Acenaphthylene	**.64**	**.03**	.50	.08	--	--	--	--
1,2-Dimethylnaphthalene	--	--	--	--	-.09	.76	-.43	.14
Acenaphthene	**.37**	**.20**	.07	.80	--	--	--	--
C3-128 isomers	--	--	--	--	--	--	--	--
2,3,6-Trimethylnaphthalene	--	--	--	--	--	--	--	--
9H-Fluorene	-.23	.42	-.36	.22	-.53	.06	**-.71**	**.01**
1-methyl-9H-Fluorene	--	--	--	--	-.53	.06	**-.71**	**.01**
Phenanthrene	.36	.22	.14	.62	.43	.14	.36	.22
Anthracene	**.62**	**.03**	.21	.46	-.11	.71	-.07	.80
2-Methylanthracene	--	--	--	--	--	--	--	--
4,5-Methylenephenanthrene	.19	.51	-.07	.80	**-.73**	**.01**	**-.79**	**.01**
C1-178 isomers	-.48	.09	**-.71**	**.01**	--	--	--	--
1-Methylphenanthrene	--	--	--	--	.29	.32	.21	.46
C2-178 isomers	**-.64**	**.03**	**-.79**	**.01**	--	--	--	--
Fluoranthene	.40	.17	.21	.46	.21	.46	.14	.62
Pyrene	.07	.80	-.36	.22	**.57**	**.05**	.43	.14
C3-178 isomers	--	--	--	--	--	--	--	--
1-Methylpyrene	--	--	--	--	--	--	--	--
C1-202 isomers	--	--	--	--	--	--	--	--
C2-202 isomers	--	--	--	--	--	--	--	--
Benz(a)anthracene	-.08	.78	-.36	.22	--	--	--	--
Chrysene	.40	.17	-.21	.46	**.57**	**.05**	.50	.08
C1-228 isomers	--	--	--	--	--	--	--	--
Benzo(b)fluoranthene	.29	.32	0	1	.43	.14	.36	.22
Benzo(k)fluoranthene	0	1	-.29	.32	.50	.08	.43	.14
Benzo(e)pyrene	.33	.26	-.07	.80	.50	.08	.43	.14
Benzo(a)pyrene	.47	.10	.07	.80	.50	.08	.43	.14
C1-252 isomers	**-.76**	**.01**	**-.86**	**0**	-.49	.09	-.50	.08
Benzo(g,h,i)perylene	**.64**	**.03**	.14	.62	**-.73**	**.01**	**-.79**	**.01**
Indeno(1,2,3-c,d)pyrene	.43	.14	0	1	.21	.46	.21	.46
Dibenzo(a,h)anthracene	.34	.24	-.07	.80	**-.64**	**.03**	**-.79**	**.01**
Coronene	-.47	.10	**-.71**	**.01**	0	1	-.29	.32
Total SQG PAH	.43	.14	-.07	.81	**.79**	**.006**	**.71**	**.01**
Combustion PAH	.43	.14	-.14	.62	**.64**	**.03**	**.64**	**.03**

Table 6. Summary of trend testing results for major and trace elements detected in sediment cores from Caddo Lake, Texas, May 2002—Kendall's tau correlation of concentration with deposition date

[Significant trends at 95-percent confidence level (p-value <.05) shown in bold; nd, not detected]

Constituent	Goose Prairie Creek inlet		Harrison Bayou inlet		Mid-lake (main stem near Green Brake)		Carter Lake near Horse Island	
	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value	Kendall's tau	p-value
Organic carbon	**1**	**0**	.11	.71	**.86**	**0**	**.73**	**0**
Total carbon	**1**	**0**	.22	.44	**.86**	**0**	**.71**	**0**
Aluminum	-.25	.34	**-.71**	**.01**	0	1	**-.69**	**0**
Calcium	**1**	**0**	.07	.80	.11	.71	**.60**	**0**
Iron	.08	.75	**.86**	**0**	**1**	**0**	**-.85**	**0**
Magnesium	.89	.34	-.79	.07	-.14	.62	**-.49**	**.01**
Phosphorus	**.89**	**0**	**.93**	**0**	**1**	**0**	-.32	.11
Potassium	**-.89**	**0**	-.07	.80	**-.93**	**0**	**-.63**	**0**
Sodium	**-.78**	**0**	0	1	**-.86**	**0**	.03	.87
Titanium	**-1**	**0**	-.43	.14	**-.79**	**.01**	**-.61**	**0**
Antimony	-.39	.14	**-.93**	**0**	**-.62**	**.03**	**-.67**	**0**
Arsenic	-.44	.10	-.21	.46	**.57**	**.05**	**-.60**	**0**
Barium	**-.87**	**0**	.36	.22	.47	.10	-.38	.06
Beryllium	.17	.53	-.36	.22	-.25	.38	**-.82**	**0**
Cadmium	.28	.30	.47	.10	.18	.53	**-.54**	**.01**
Cerium	**-.61**	**.02**	.07	.80	**-.57**	**.05**	-.26	.20
Chromium	**-.61**	**.02**	**-.62**	**.03**	-.21	.46	**-.76**	**0**
Cobalt	.42	.11	.50	.08	-.50	.08	-.08	.70
Copper	-.22	.40	**-.64**	**.03**	-.50	.08	.32	.11
Gallium	-.37	.17	**-.69**	**.02**	-.26	.36	**-.75**	**0**
Lanthanum	**-.61**	**.02**	.07	.80	-.55	.06	-.27	.19
Lead	**-.89**	**0**	**-.71**	**.01**	-.14	.62	**-.86**	**0**
Lithium	**-.76**	**0**	**-.71**	**.01**	**-.64**	**.03**	**-.88**	**0**
Manganese	**1**	**0**	**.93**	**0**	**1**	**0**	.02	.91
Mercury	**-.56**	**.04**	**.89**	**0**	**.78**	**.01**	**-.62**	**0**
Molybdenum	**.54**	**.04**	-.43	.14	.36	.22	.36	.08
Nickel	.22	.40	-.50	.08	-.50	.08	**-.80**	**0**
Niobium	**-.67**	**.01**	-.36	.22	**-.71**	**.01**	**-.57**	**0**
Scandium	**-.54**	**.04**	**-.69**	**.02**	-.43	.14	**-.70**	**0**
Selenium	**.99**	**0**	0	1	.18	.53	.05	.82
Silver	-.44	.10	nd	nd	nd	nd	nd	nd
Strontium	.28	.30	-.40	.17	-.40	.17	-.07	.74
Thorium	**-.89**	**0**	-.43	.14	**-.71**	**.01**	**-.61**	**0**
Uranium	**-.83**	**0**	**-.64**	**.03**	**-.79**	**.01**	**-.64**	**0**
Vanadium	-.50	.06	**-.64**	**.03**	-.36	.22	**-.83**	**0**
Zinc	.44	.10	-.14	.62	.04	.90	**-.73**	**0**

significant downward trend in titanium concentrations was not considered strong enough evidence to support a change in the sedimentation rate. Decreasing concentrations of phosphorus with depth have been reported as an effect of early diagenesis (for example, Sundby and others, 1992) and might not be related to changes in fertilizer usage. A significant downward trend in iron was not indicated in the Goose Prairie Creek core.

The eight trace elements with consensus-based SQGs (MacDonald and others, 2000) are arsenic, cadmium, chromium, copper, lead, mercury, nickel, and zinc (table 3). All samples analyzed for trace elements in the Goose Prairie Creek core exceeded the TEC for chromium, lead, mercury, and nickel; and 78 percent of samples were at or higher than the TEC for copper and zinc (fig. 9; table 7). One sample, deposited about 1961, exceeded the PEC for lead with a concentration of 181 μg/g. The highest concentration of cadmium (not graphed in fig. 9) was 55 percent of the TEC. The concentrations of chromium, lead, and mercury have had significant downward trends since about 1961. In sediments deposited after 1980, nickel and zinc had significantly increasing concentrations ($\tau = 1$ and $p = .014$ for both). Seventy percent of the trace elements without SQGs but with significant trends had decreasing concentrations over time in the Goose Prairie Creek core.

Goose Prairie Creek was the only coring site where silver was detected (LRL is 3 μg/g). Silver was detected in all samples in the core except the oldest sample interval analyzed, which had an estimated deposition date of 1948 (fig. 9). The concentration increased to a maximum of 23.8 μg/g in the sample deposited about 1969. Although no consensus-based SQGs exist, Long and others (1995) published recommended SQGs (which included silver). For silver, the ER–L is 1.0 μg/g, and the ER–M is 3.7 μg/g. The concentration of silver (fig. 9) was higher than the ER–M in all samples deposited after 1960. The concentrations did not have a statistically significant trend when all samples were included in the trend test; however, the concentrations decreased significantly over time from the sample deposited in 1969 to the most recently deposited sample ($\tau = -0.86$, $p = .003$).

Harrison Bayou

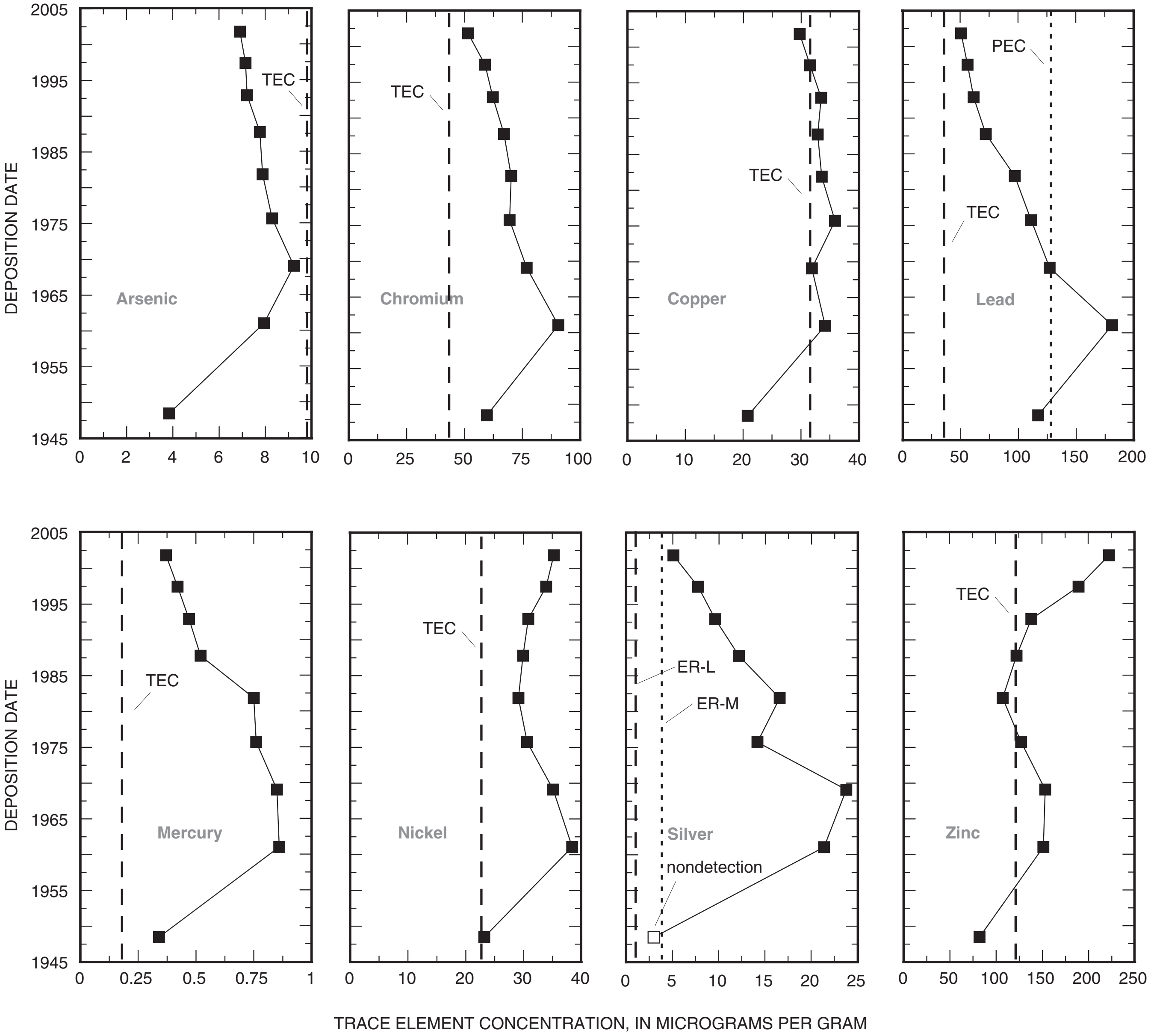
Age Dating and Sedimentation Rates

A clearly defined ^{137}Cs peak at 15.5 cm was used as the 1964.0 date-depth marker (fig. 6b), resulting in an average MAR of 0.063 g/cm^2-yr for the core. Sediment deposition dates were computed back to about 1950 for the Harrison Bayou core. The highest total DDT concentration had a deposition date of 1961, which was compatible with historical DDT use. The highest lead concentration was dated as 1950; however a small “hump” in the lead profile occurred in sediments deposited around the mid- to late-1970s, which was compatible with historical environmental lead concentrations. The ^{137}Cs peak was considered the most reliable age-date marker in the core. The sediment accumulation rate in Harrison Bayou probably was not constant over the period of deposition represented in the core, but no additional date-depth markers were in the core to use for the computation of more than one MAR; therefore, an uncertainty of ±2 years was estimated for the age dates of the samples in the Harrison Bayou core.

Contaminant Occurrence and Trends

DDE was detected in all samples from the Harrison Bayou core that were analyzed for organochlorine compounds. Five of the six samples deposited before 1995 had DDE concentrations higher than the TEC but much lower than the PEC. DDD was detected in the oldest sample only, which was deposited about 1950 (fig. 7). Most organic carbon concentrations were between 9.86 and 11.6 percent and did not indicate a significant trend for the core (Kendall’s $\tau = .11$, $p = .71$); therefore, organic compound concentrations were not normalized with organic carbon before trend testing. DDE concentrations in sediment at the site had a significant downward trend. PCB Aroclors 1242, 1254, and 1260 were not detected in the core.

The PAH mixture was dominated by high concentrations of C2- and C3-alkylated naphthalenes. Concentrations in three of the eight samples exceeded 2,000 and 300 μg/kg for the C2- and C3-alkylated naphthalenes, respectively. Concentrations of all other compounds were less than 50 μg/kg (appendix 2.3). Concentrations of most individual PAH compounds were highest in the most recently deposited sample, but most PAH time series did not show significant upward trends (fig. 10). Individual PAHs and total SQG PAHs were well below the respective TECs.



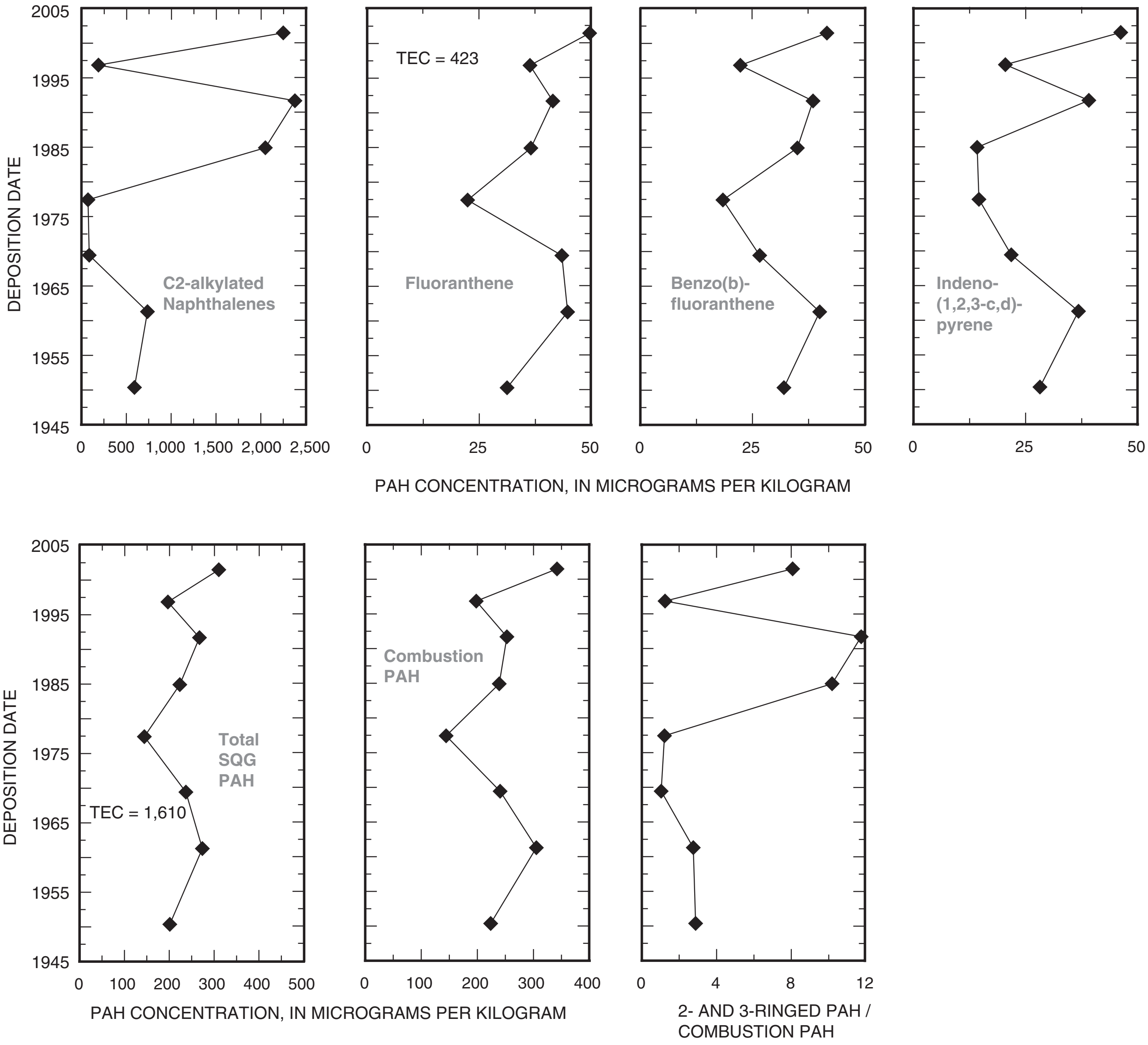
EXPLANATION

TEC, threshold effect concentration
PEC, probable effect concentration
(MacDonald and others, 2000)

ER-L, effects range-low
ER-M, effects range-median
(Long and others, 1995)

Figure 9. Selected trace element concentrations and trends in core collected near Goose Prairie Creek, Caddo Lake, Texas, May 2002.

The ratio of 2- and 3-ringed compounds to combustion PAH was greater than 1 in all samples and substantially greater than 1 in three of four post-1983 samples (fig. 10). Only two of the PAH compounds, 9H-fluorene and 1-methyl-9H-fluorene had significant trends—upward and downward, respectively (table 5). Total SQG PAH and combustion PAH had no trend (fig. 10).



EXPLANATION

SQG, sediment-quality guideline

TEC, threshold effect concentration (MacDonald and others, 2000)

Figure 10. Selected polycyclic aromatic hydrocarbon (PAH) concentrations in core collected near Harrison Bayou, Caddo Lake, Texas, May 2002.

Concentrations of the major elements were tested for significant trends in the Harrison Bayou core (table 6). Aluminum concentrations had a significant downward trend (Kendall's $\tau = -.71$, $p = .01$), iron concentrations had a significant upward trend ($\tau = 0.86$, $p = 0$), and titanium concentrations had no significant trend ($\tau = -.43$, $p = .14$). As with the Goose Prairie Creek data, the lack of similarity among the trends of aluminum, iron, and titanium concentrations provides some evidence that the sedimentation rate over time has not been constant.

Three of the trace elements with SQGs (chromium, nickel, and zinc) had concentrations higher than the respective TECs, although none exceeded the PECs

Table 7. Summary of results of analysis for selected trace elements in sediment cores from Caddo Lake, Texas, May 2002

[TEC, threshold effect concentration (MacDonald and others, 2000); PEC, probable effect concentration (MacDonald and others, 2000); --, not applicable]

Coring site	Trace element	Concentrations greater than TEC (percent)	Concentrations greater than PEC (percent)	Statistically significant trend	If trend, percent change from highest or lowest concentration to present concentration
Goose Prairie Creek (9 samples)	Arsenic	0	0	No trend	--
	Cadmium	0	0	No trend	--
	Chromium	100	0	Downward	43
	Copper	78	0	No trend	--
	Lead	100	11	Downward	72
	Mercury	100	0	Downward	57
	Nickel	100	0	No trend	--
	Zinc	78	0	No trend	--
	Silver	[1]89	[1]89	No trend	--
Harrison Bayou (8 samples)	Arsenic	0	0	No trend	--
	Cadmium	0	0	No trend	--
	Chromium	100	0	Downward	18
	Copper	0	0	Downward	7.5
	Lead	0	0	Downward	32
	Mercury	0	0	Upward	33
	Nickel	100	0	No trend	--
	Zinc	100	0	No trend	--
Mid-lake (8 samples)	Arsenic	25	0	Upward	93
	Cadmium	0	0	No trend	--
	Chromium	100	0	No trend	--
	Copper	0	0	No trend	--
	Lead	0	0	No trend	--
	Mercury	0	0	Upward	50
	Nickel	87.5	0	No trend	--
	Zinc	62.5	0	No trend	--
Carter Lake (14 samples)	Arsenic	100	0	Downward	20
	Cadmium	0	0	Downward	12
	Chromium	100	0	Downward	21
	Copper	7	0	No trend	--
	Lead	29	0	Downward	33
	Mercury	93	0	Downward	9.5
	Nickel	100	0	Downward	16
	Zinc	100	0	Downward	28

[1] Percent samples greater than ER–L (effects range-low) and ER–M (effects range-median) (Long and others, 1995).

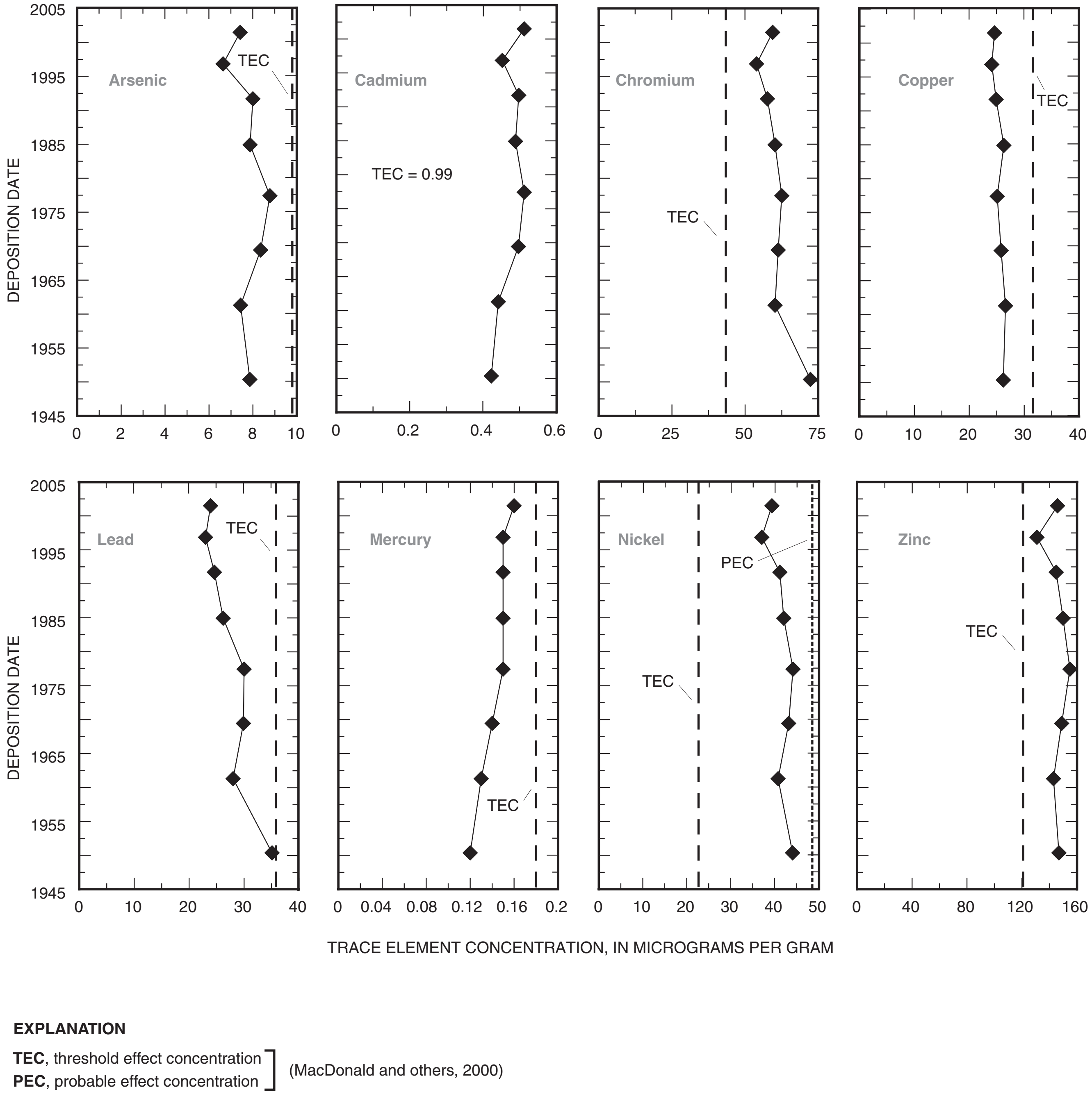


Figure 11. Selected trace element concentrations and trends in core collected near Harrison Bayou, Caddo Lake, Texas, May 2002.

(fig. 11; table 7). The concentrations of nickel were close to, but still lower than, the PEC. Cadmium concentrations were lower than the TEC, and concentrations of arsenic, copper, lead, and mercury were lower than the respective TECs. Chromium, copper, and lead concentrations had significant downward trends, and mercury concentrations had a significant upward trend (fig. 11; table 6). For trace elements without SQGs but with significant trends, 86 percent of the trends were upward.

Mid-Lake

Age Dating and Sedimentation Rates

The mid-lake core was analyzed for ^{137}Cs, ^{210}Pb, and ^{226}Ra. A plot of unsupported ^{210}Pb as a function of cumulative dry mass was scattered and did not follow a linear decrease (when plotted on a logarithmic scale) ($R^2 = .4117$; fig. 4a). It was decided not to use the ^{210}Pb concentrations for age dating the core. The ^{137}Cs concentrations were used instead because they showed a clear peak at 9.75 cm. An average MAR of 0.074 g/cm^2-yr and sediment deposition dates back to about 1940 were computed for the core, which resulted in age dates of 1976.0 for the highest total DDT concentration and 1982.9 for the highest lead concentration. These dates were later than their known historical peak concentrations in cores from other lakes (Callendar and Van Metre, 1997; Van Metre and others, 1997); however, the peak total DDT and lead concentrations in the core might have been missed by the wide sample analysis interval used at the bottom of the core. Only two samples were analyzed for organochlorine compounds and trace elements near the bottom of the core. The estimated deposition dates for those samples were 1950 and 1967. It is unlikely that the sediment accumulation rate in the mid-lake was constant over the period of deposition represented in the core, but no additional date-depth markers were in the core to use for the computation of more than one MAR; therefore, an uncertainty of ±5 years is estimated for the age dates of the samples in the mid-lake core.

Contaminant Occurrence and Trends

DDE was detected in all samples from the mid-lake site analyzed for organochlorine compounds, and DDD was detected in the two oldest samples only, deposited in 1950 and 1967 (fig. 7). Only in the sample deposited in 1976 was a DDE concentration higher than the TEC. Because organic carbon concentrations had a significant upward trend (Kendall's $\tau = .86$, $p = 0$), organic compound concentrations were normalized with organic carbon concentrations before trend testing. DDE had a statistically significant downward trend in the mid-lake core (Kendall's $\tau = -.57$, $p = .048$). PCB Aroclors 1242, 1254, and 1260 were not detected in the core.

PAH concentrations in the mid-lake core were low. The PAH mixture in the mid-lake core was dominated by C2-alkylated naphthalenes, with 2,6-dimethylnaphthalene contributing about one-half to three-fourths of the total C2-alkylated naphthalene concentration. Most individual PAH concentrations were below 20 µg/kg and well below their TECs (fig. 12). Total SQG PAHs did not exceed 120 µg/kg, an order of magnitude lower than the TEC. The ratio of 2- and 3-ringed compounds to combustion PAH was about 1 or greater than 1 in all but the surficial sample. Organic carbon normalized concentrations of C1-178 isomers, C2-178 isomers, C1-252 isomers, and coronene had significant downward trends (table 5). No trends were indicated by concentrations of total SQG PAH and combustion PAH (fig. 12).

Concentrations of the major elements were tested for significant trends in the mid-lake core (table 6). Aluminum concentrations had no trend (Kendall's $\tau = 0$, $p = 1$), iron concentrations had a significant upward trend ($\tau = 1$, $p = 0$), and titanium concentrations had a significant downward trend ($\tau = -.79$, $p = .01$). As with the other core data described, the lack of similarity among the trends of aluminum, iron, and titanium concentrations indicates the possibility of a changing sedimentation rate over time. The organic carbon concentrations in the mid-lake core were noticeably lower than those in the other three cores collected from the lake (appendix 2.4).

Variation in trace element concentrations in the mid-lake core was more subtle than in the other cores—arsenic, manganese, and mercury concentrations had significant upward trends (fig. 13; table 6). Decreasing concentrations of manganese with depth in sediment have been reported as an effect of diagenesis (for example, Gobeil and others, 1997). All chromium concentrations in the core exceeded the TEC (table 7); seven of eight nickel concentrations exceeded the TEC; the five samples deposited between 1976 and 1998 had zinc concentrations that exceeded the TEC; and the two most recently deposited samples had arsenic concentrations that exceeded the TEC. All concentrations of cadmium, copper, lead, and mercury were below the TEC. No trace element concentration exceeded the PEC.

Carter Lake

Age Dating and Sedimentation Rates

The Carter Lake core was analyzed for ^{137}Cs, ^{210}Pb, and ^{226}Ra for age dating. Unsupported ^{210}Pb graphed on a logarithmic scale as a function of cumulative dry mass was almost linear ($R^2 = .8059$; fig. 4b),

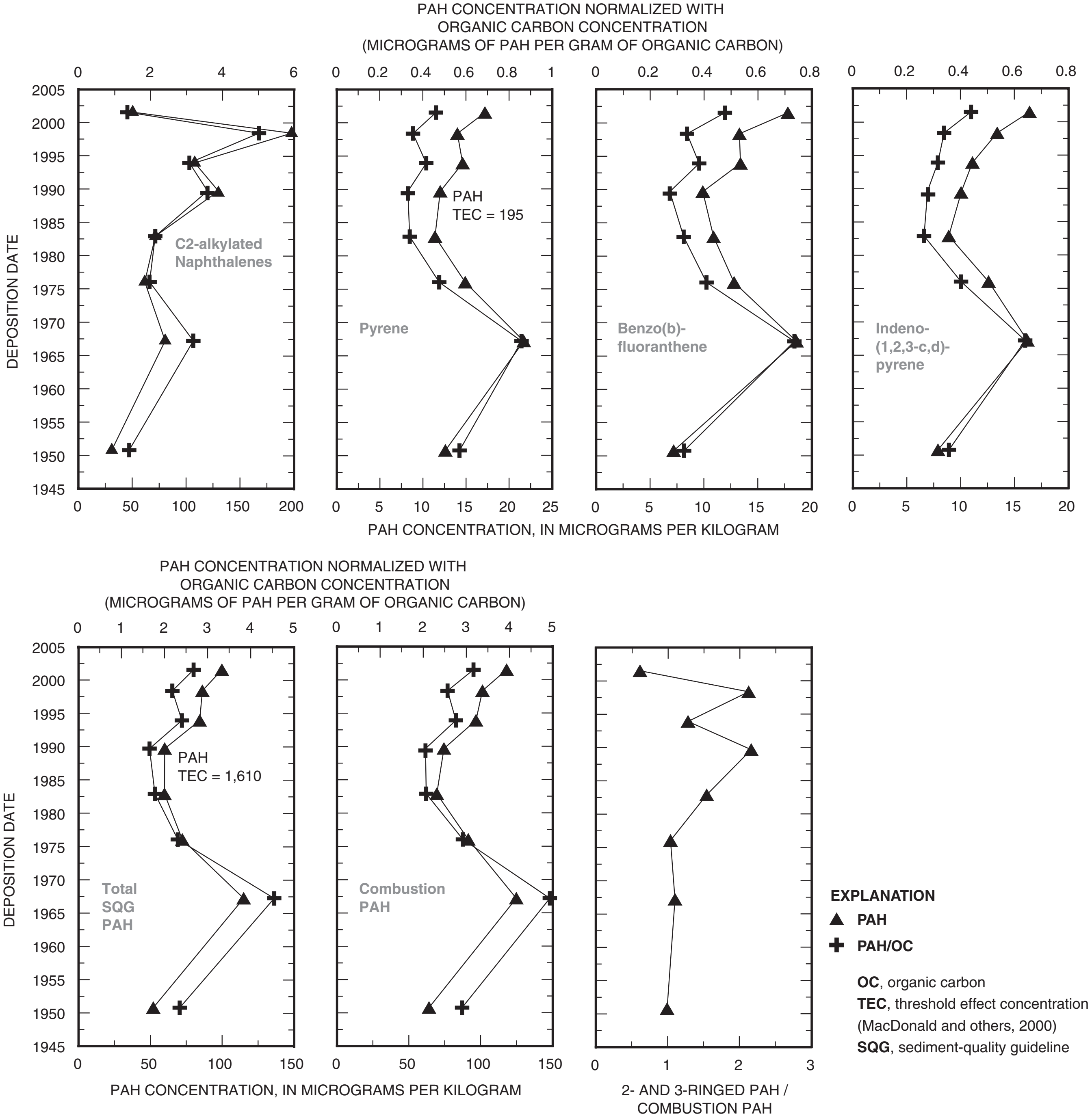
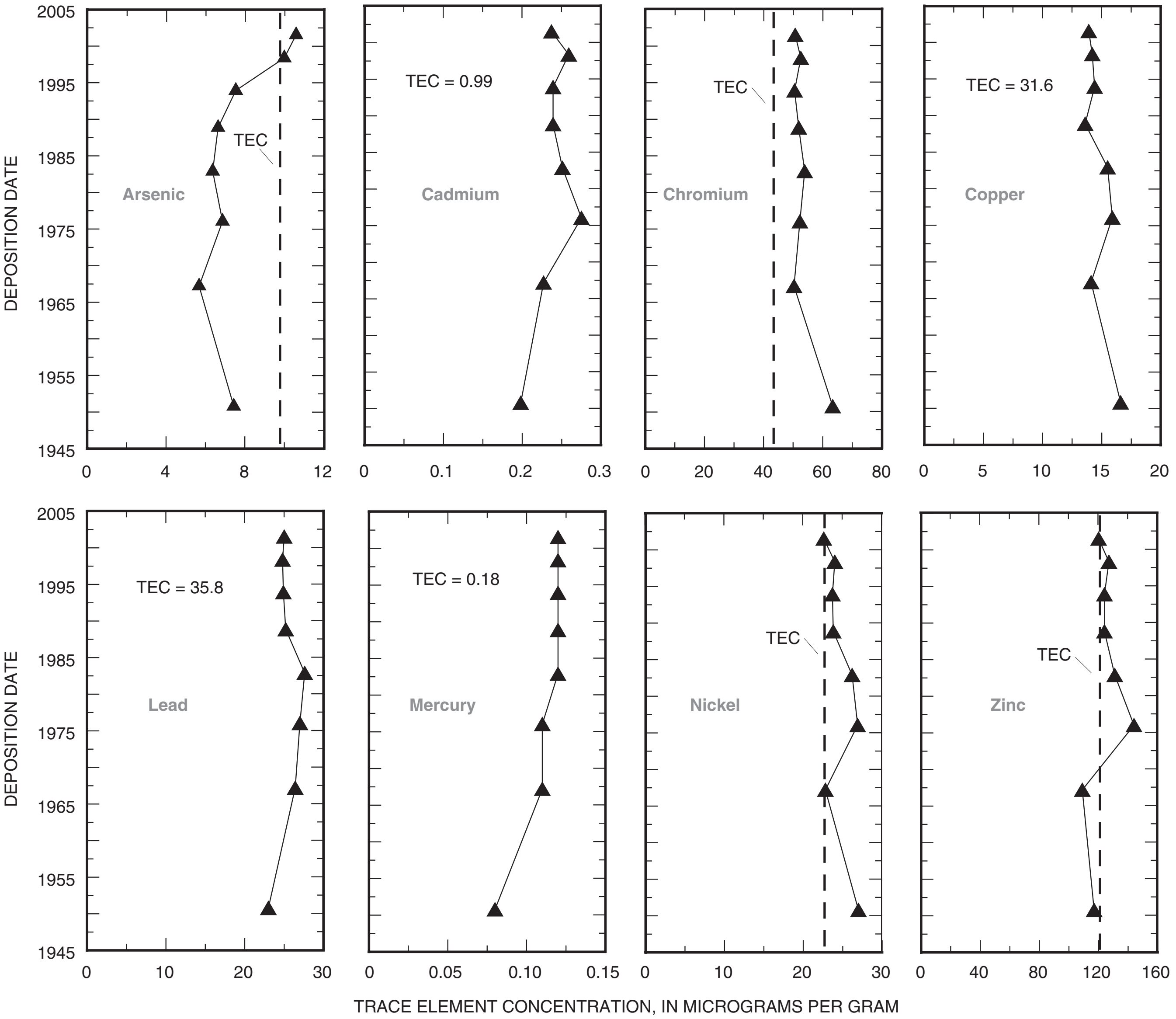


Figure 12. Selected polycyclic aromatic hydrocarbon (PAH) concentrations in core collected in mid-lake of Caddo Lake, Texas, May 2002.

and a clearly defined ^{137}Cs peak for a 1964.0 date-depth marker was not available; thus the slope of the line fit to the points was used to compute age dates back to about 1965 for the samples in the core. A MAR of 0.099 g/cm^2-yr was computed for the core. The highest total DDT and lead concentrations had a deposition date of 1975.8 (fig. 6d). This sample interval was the deepest analyzed for organochlorine compounds, so the total DDT peak might have occurred in sediments deeper than those analyzed at this site. The age dates were



EXPLANATION

TEC, threshold effect concentration, (MacDonald and others, 2000)

Figure 13. Selected trace element concentrations and trends in core collected in mid-lake of Caddo Lake, Texas, May 2002.

consistent with historical lead concentrations in cores. ^{137}Cs concentrations increased with depth in the core back to 1975, which is consistent with the ^{210}Pb deposition dates (fig. 6d). It was unlikely that the sediment accumulation rate in Carter Lake was constant over the period of deposition collected in the core, but as with the other cores, no additional date-depth markers were in the Carter Lake core to use for the computation of more than one MAR; therefore, an uncertainty of ±2 years was estimated for the age dates of the samples in the Carter Lake core.

Contaminant Occurrence and Trends

DDE was detected in samples deposited between 1975 and 1994, and DDD was detected in the oldest sample only, deposited about 1975 (fig. 7). Only the

Table 8. Sediment focusing data and core burdens for mercury, Caddo Lake, Texas, May 2002

[pCi/cm^2, picocuries per square centimeter; µg/cm^2, micrograms per square centimeter]

Sediment core	^{137}Cs burden (ϕ_c) (pCi/cm^2)	Mercury burden (ϕ_{hg}) (µg/cm^2)	Cumulative ^{137}Cs fallout deposition, 1954–74 (ϕ_f) (pCi/cm^2)	Focusing factor (FF) (ϕ_c/ϕ_f)	Mercury burden normalized with focusing factor (µg/cm^2)
Goose Prairie Creek	6.82	5.17	4.65	1.47	3.53
Harrison Bayou	4.36	.49	4.65	.94	.52
Mid-lake	2.45	.52	4.65	.53	1.00

oldest sample had a DDE concentration above the TEC. PCB Aroclors 1242, 1254, and 1260 were not detected in the Carter Lake core. Organic carbon concentrations ranged from 8.06 to 11.2 percent and had a significant upward trend (Kendall's $\tau = .73$, $p = 0$); therefore, organic compound concentrations were normalized with organic carbon concentrations before trend testing. Organic carbon normalized DDE concentrations had a significant downward trend (Kendall's $\tau = -.86$, $p = .003$).

PAH concentrations in the Carter Lake core were less than the TECs (fig. 14). The C2-alkylated naphthalenes had the highest concentrations, particularly 2,6-dimethylnaphthalene. The concentrations of other compounds were about an order of magnitude lower than the C2-alkylated naphthalenes and of similar magnitude to one another. The organic carbon normalized concentrations of 9H-Fluorene, 1-methyl-9H-Fluorene, 4,5-methylenephenanthrene, benzo(g,h,i)perylene, and dibenzo(a,h)anthracene had significant downward trends (table 5), but total SQG PAH and combustion PAH had significant upward trends. The ratio of 2- and 3-ringed PAH to combustion PAH was greater than 1 for all but one sample deposited in 1989, which indicates a predominance of uncombusted PAH sources.

Concentrations of the major elements in the Carter Lake core were trend tested (table 6). Aluminum, iron, and titanium concentrations had significant downward trends (Kendall's $\tau = -.69$, $p = 0$; $\tau = -.85$, $p = 0$; and $\tau = -.61$, $p = 0$, respectively). The downward trends of these three stable elements indicate a decreasing sedimentation rate over time in the Carter Lake core.

Concentrations of several trace elements in the samples from the Carter Lake core exceeded the respective TECs (fig. 15; table 7), but none exceeded the PECs. All sample concentrations of arsenic, chromium, nickel, and zinc exceeded the TECs; all but one mercury concentration exceeded the TEC. Lead concentrations in samples deposited before 1985 were higher than the TEC, but concentrations in samples deposited after 1985 were lower. The copper concentration in one sample, with a deposition date of 1997, exceeded the TEC for copper. The concentrations of seven of the eight trace elements with SQGs had significant downward trends—all except copper. The trends in concentrations of trace elements without SQGs were all downward.

Sediment-Core Burdens, Focusing Factors, and General Sources of Mercury

The burdens of ^{137}Cs (ϕ_c) and mercury (ϕ_{hg}) for the Goose Prairie Creek core, Harrison Bayou core, and mid-lake core, and the FFs for the three coring sites were computed as described in the "Sediment-Core Burden and Focusing Factor" section. The ϕ_{hg} for each core was normalized with the respective FF to compute the amount of cumulative atmospheric mercury fallout necessary over the Caddo Lake region to produce the measured ϕ_{hg}. Similar data for the Carter Lake core were not computed because the oldest Carter Lake sediment analyzed for ^{137}Cs (20- to 21-cm interval) was age dated to 1973.9, essentially at the end of the period 1954–74 upon which the prediction model used to obtain cumulative atmospheric fallout deposition of ^{137}Cs (ϕ_f) for the Caddo Lake region is based. Thus, because ϕ_c and ϕ_f could not be obtained for a common period, a meaningful focusing factor (FF = ϕ_c/ϕ_f) for the Carter Lake site could not be computed.

The ϕ_c ranged from 2.45 pCi/cm^2 for the mid-lake core to 6.82 pCi/cm^2 for the Goose Prairie core (table 8). The ϕ_f for the Caddo Lake region was computed to be 4.65 pCi/cm^2. Comparison of the model-predicted ϕ_f to decay-corrected fallout measured by the U.S. Energy Research and Development Administration, Health and Safety Laboratory (1977), indicated

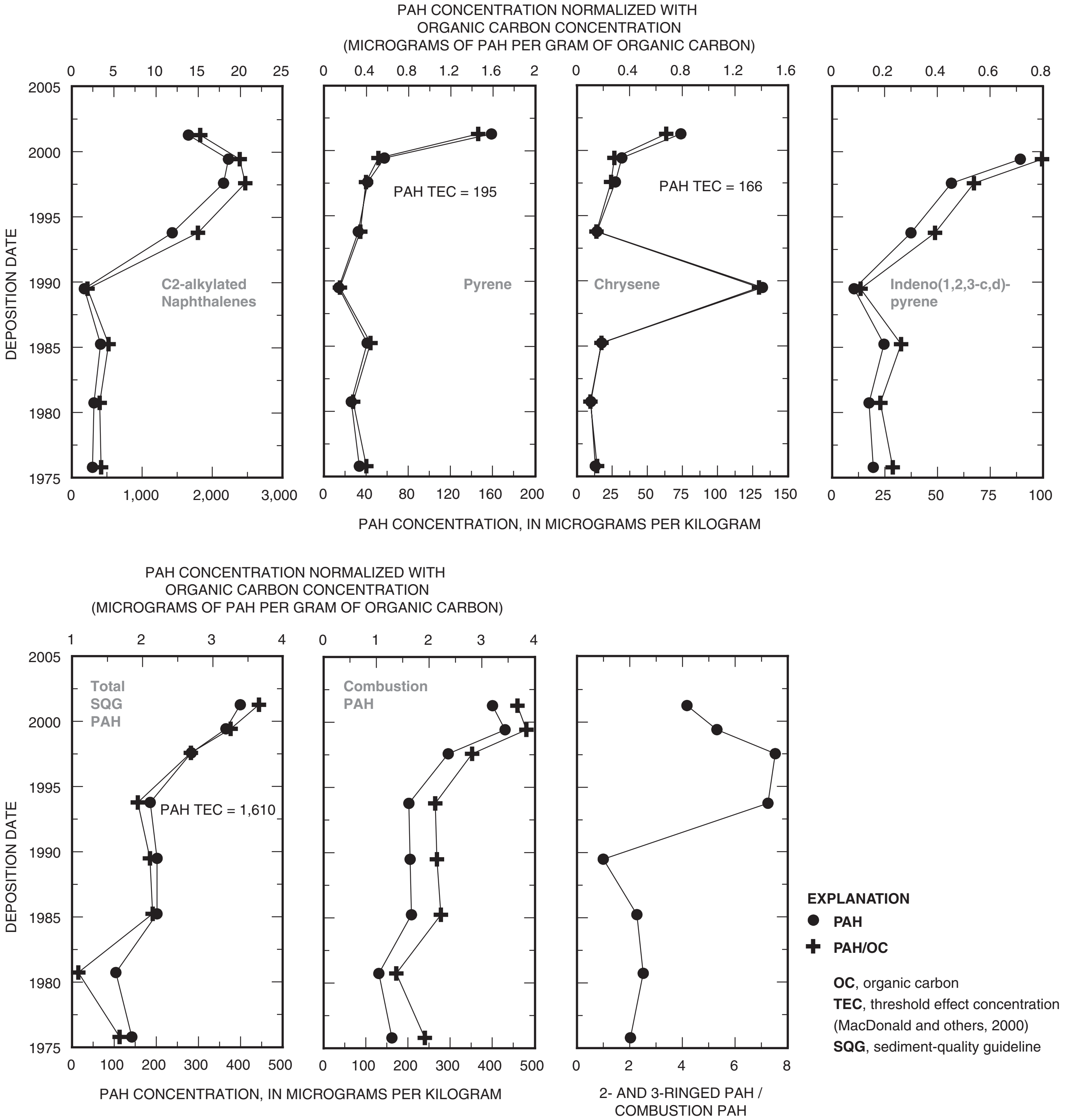


Figure 14. Selected polycyclic aromatic hydrocarbon (PAH) concentrations and trends in core collected from Carter Lake, Caddo Lake, Texas, May 2002.

uncertainty of about 20 percent in the model-predicted ϕ_f and subsequent focusing computations.

The sediment FF was about 1.5 for the Goose Prairie Creek site, about 1.0 for the Harrison Bayou site, and about 0.5 for the mid-lake site (table 8). Comparable FFs have been reported for the Great Lakes (Eadie and Robbins, 1987; Eisenreich and others, 1989), and larger FFs have been reported for reservoirs

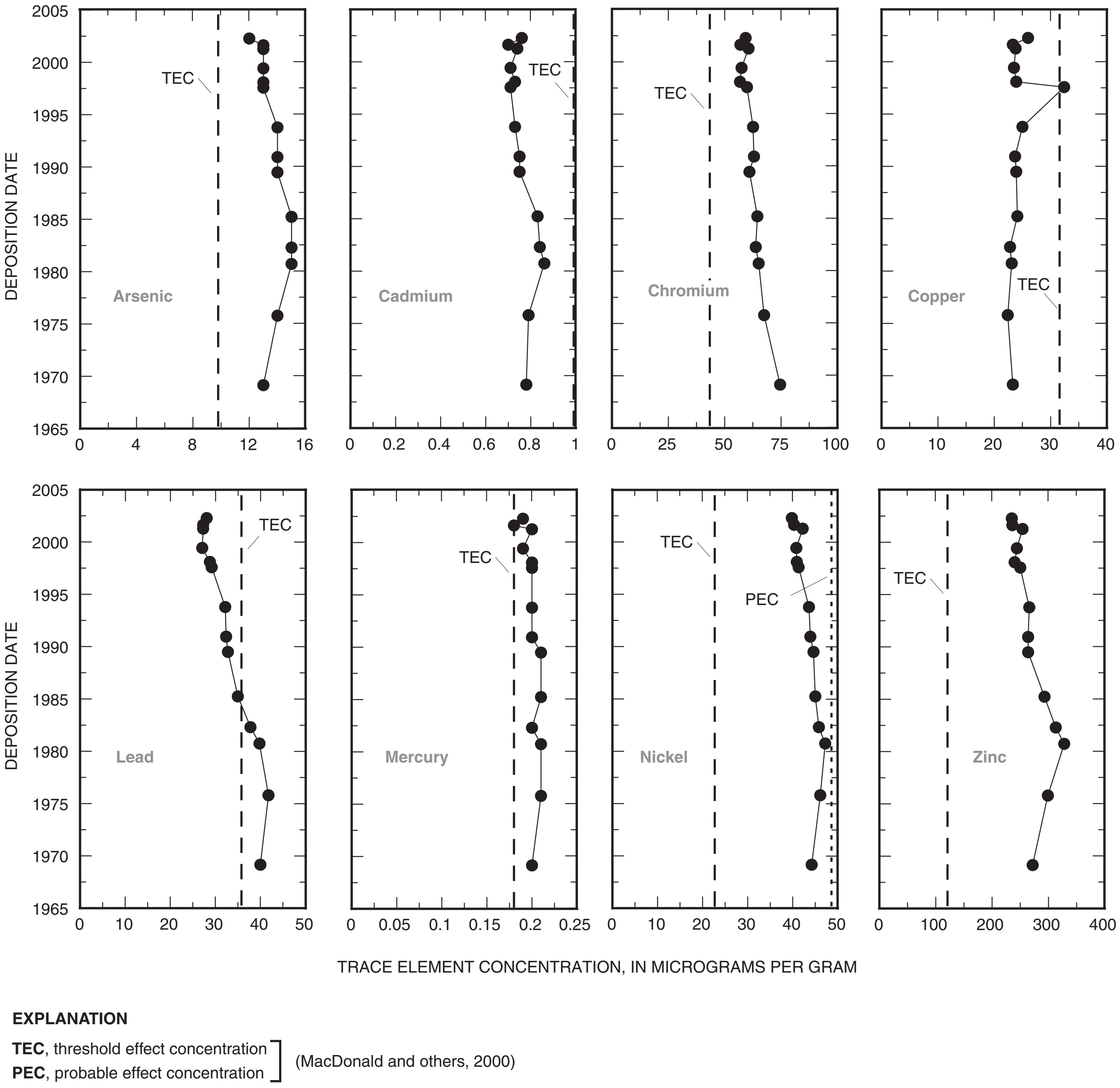


Figure 15. Selected trace element concentrations and trends in core collected from Carter Lake, Caddo Lake, Texas, May 2002.

across the country (for example, Van Metre and others, 1997). The Goose Prairie Creek FF indicates that about one-third of the mercury at the Goose Prairie Creek site might result from drainage area sources. After normalizing for sediment focusing, the Goose Prairie Creek mercury burden is about 3.5 and 7 times larger than the burdens for the Harrison Bayou and mid-lake sites, respectively. No drainage area sources are indicated by the FFs for the Harrison Bayou and mid-lake sites. In a recent statewide inventory of water-quality concerns, the TCEQ (Texas Natural Resource Conservation Commission, 2002b, p. 8–10) reported that the source of mercury to sites near the mid-lake and Carter Lake sites of this report is both industrial point sources and atmospheric deposition.

Comparisons of Findings to Those of Other Texas Lakes

The USGS cored Lake Meredith in the Texas Panhandle to investigate the effects of oil and gas production in the area on the sediments in that reservoir (Mahler and Van Metre, 2001). More than 250 wells are in the Lake Meredith area, and oil and gas exploration has been ongoing since about 1930. Lake Meredith is in a dry, desert-like environment and has high sedimentation rates (MAR 1.1 to 1.7 g/cm^2-yr), whereas Caddo Lake is in a much more humid, swamp-like environment with low sedimentation rates.

Although drainage area characteristics are different, oil and gas production is common to both drainage areas. Concentrations of the trace elements and PAHs were compared for Lake Meredith and Caddo Lake (table 9). Arsenic and cadmium concentrations in the Goose Prairie Creek, Harrison Bayou, and mid-lake cores from Caddo Lake were similar to concentrations in the Lake Meredith cores; concentrations in the Carter Lake core were somewhat higher. Barium concentrations were similar for the four Caddo Lake cores but higher in the Lake Meredith cores. Chromium, copper, and nickel concentrations were similar in sediments from the two water bodies. Lead concentrations were similar, except for the Goose Prairie Creek core in which concentrations were at least three times higher. Mercury concentrations in the Lake Meredith cores did not exceed 0.04 µg/g, considerably less than those in the Caddo Lake cores, which ranged from 0.08 to 0.86 µg/g. Zinc concentrations in the Goose Prairie Creek, Harrison Bayou, and mid-lake cores were about 1.2 to 1.4 times higher than those in the Lake Meredith cores, and concentrations in the Carter Lake core were about 2.5 times higher.

The concentrations of PAHs were of the same order of magnitude in cores from both lakes. Similar to Caddo Lake sediments, Lake Meredith sediments generally contained more 2- and 3-ringed PAHs (uncombusted sources) than 4- and 5-ringed PAHs (combusted sources).

The USGS did another sediment coring study in Mountain Creek Lake in the cities of Dallas and Grand Prairie (Jones and others, 1997). The reservoir is on Mountain Creek, a tributary of the Trinity River, and was constructed to provide cooling water for a utilities power plant. Land use in the Mountain Creek Lake drainage area is a mixture of commercial, residential, and military facilities. The sedimentation rates (MAR 0.53 to 0.96 g/cm^2-yr) in the reservoir are about double the rates computed for Caddo Lake. The Naval Weapons Industrial Reserve Plant was built adjacent to the northeastern part of the reservoir in 1941, with a primary mission of military aircraft manufacturing, and is still in operation today (2003). Box cores collected in Cottonwood Bay (a part of Mountain Creek Lake) near the Naval Weapons Industrial Reserve Plant had mostly higher concentrations of arsenic, chromium, copper, and nickel than the cores from Caddo Lake (table 9). Barium concentrations in the Caddo Lake cores were about two times higher than those in the Mountain Creek Lake cores. The Goose Prairie Creek core had higher concentrations of lead and mercury than those in Mountain Creek Lake cores, but lead concentrations in the three other cores from Caddo Lake were similar to those in Mountain Creek Lake cores. Zinc concentrations in the Carter Lake core were slightly lower than those in Mountain Creek Lake cores, and concentrations in the other Caddo Lake cores were much lower.

The PAH assemblages are different in sediments of the two lakes. Combustion-derived compounds predominate in Mountain Creek Lake sediments, whereas the PAH compounds in Caddo Lake sediments generally are from uncombusted sources.

Among the three lakes, oil and gas production is in the drainage areas of Caddo Lake and Lake Meredith but not Mountain Creek Lake. Barium concentrations in Caddo Lake and Lake Meredith sediments about twice those in Mountain Creek Lake sediments thus could be associated with oil and gas production. Association of other contaminants with drainage-area characteristics, for example weapons production facilities common to the drainage areas of Caddo and Mountain Creek Lakes but not that of Lake Meredith, is less clear.

Implications of Findings Relative to Drainage Area Sources of Contaminants

The organochlorine pesticides DDE and DDD, degradation products of DDT, were detected in all four cores and three of four cores, respectively, collected from Caddo Lake. DDT probably was used by the LHAAP, businesses, and households in the Caddo Lake area during the period of DDT production. The peak concentrations of DDE at the Goose Prairie Creek and Harrison Bayou sites, the two sites closest to points of entry of runoff from the LHAAP, were about twice the peak concentrations at the mid-lake and Carter Lake sites. The Carter Lake core had the fewest detections of

Table 9. Summary of trace element and selected PAH concentrations and ratio of 2- and 3-ringed to 4- and 5-ringed PAH assemblages in sediment cores from Caddo Lake, Texas, May 2002, from Lake Meredith, Texas, May 1999, and from Cottonwood Bay in Mountain Creek Lake, Texas, May–July 1996

[μg/g; micrograms per gram; μg/kg; micrograms per kilogram; na, not analyzed]

Constituent (units)	Caddo Lake - Goose Prairie Creek			Caddo Lake - Harrison Bayou			Caddo Lake - Mid-lake		
	Minimum concen-tration	Maximum concen-tration	Median concen-tration	Minimum concen-tration	Maximum concen-tration	Median concen-tration	Minimum concen-tration	Maximum concen-tration	Median concen-tration
Arsenic (μg/g)	3.8	9.2	7.8	6.6	8.8	7.9	5.7	11	7.2
Barium (μg/g)	373	530	435	462	570	508	484	516	492
Cadmium (μg/g)	.18	.54	.32	.42	.51	.49	.20	.28	.24
Chromium (μg/g)	51.6	90.6	67.1	53.9	72.3	60.2	50.3	63.4	52.1
Copper (μg/g)	20.8	35.9	32.9	24.1	26.6	25.5	13.6	16.6	14.3
Lead (μg/g)	50.4	181	96.8	23.0	35.1	27.1	23.0	27.6	25.1
Mercury (μg/g)	.34	.86	.52	.12	.16	.15	.08	.12	.12
Nickel (μg/g)	23.2	38.4	30.8	37.1	44.2	41.6	22.6	27.0	23.9
Zinc (μg/g)	82.0	222	138	131	155	146	109	144	124
Total SQG PAH (μg/kg)	90.5	280	198	145	311	230	52	115	78.3
C2-alkylated Naphthalenes (μg/kg)	13.9	762	39.8	73.5	2,380	664	31.0	198	76.1
Ratio of 2- and 3-ringed PAH to 4- and 5-ringed PAH	.74	3.5	.95	1.0	11.7	2.8	.61	2.2	1.2

Constituent (units)	Caddo Lake - Carter Lake			Lake Meredith[1]			Mountain Creek Lake[2]		
	Minimum concen-tration	Maximum concen-tration	Median concen-tration	Minimum concen-tration	Maximum concen-tration	Median concen-tration	Minimum concen-tration	Maximum concen-tration	Median concen-tration
Arsenic (μg/g)	12	15	14	3.70	13.7	9.96	7.8	20.7	16.0
Barium (μg/g)	432	507	447	534	845	593	96	334	224
Cadmium (μg/g)	.70	.86	.75	.17	.50	.31	na	na	na
Chromium (μg/g)	56.8	74.6	61.8	27.3	83.1	70.2	117	574	322
Copper (μg/g)	22.4	32.4	23.8	12.9	29.4	27.0	15.2	88.3	63.8
Lead (μg/g)	27.1	41.8	32.3	13.6	26.8	23.1	32.8	93.4	60.0
Mercury (μg/g)	.18	.21	.20	.02	.04	.03	.052	.28	.16
Nickel (μg/g)	39.8	47.2	43.8	13.0	35.8	32.8	31	109	53.2
Zinc (μg/g)	235	328	264	35.4	118	106	127	448	340
Total SQG PAH (μg/kg)	104	399	202	2.2	74.3	14.3	2,037	173,043	14,443
C2-alkylated Naphthalenes (μg/kg)	178	2,230	920	2.4	77.5	26.5	89.9	756	238
Ratio of 2- and 3-ringed PAH to 4- and 5-ringed PAH	1.0	7.5	3.4	.68	4.3	2.1	0.13	1.2	.18

[1] Unpublished data summarized in Mahler and Van Metre, 2001.

[2] From Jones and others, 1997.

DDE, perhaps because adjacent development was sparse and thus DDT use minimal. A public health assessment of the LHAAP found DDD, DDE, aldrin, and dieldrin in soil samples (Agency for Toxic Substances and Disease Registry, 2003). Recently deposited sediments in the four cores had low DDE concentrations (below the TEC) and no detections of DDD, aldrin, or dieldrin. These findings indicate that the LHAAP is a possible source of the detected organochlorines in sediments, although not from recent transport.

The Goose Prairie Creek core was the only core in which PCBs were detected. The former TNT production area of the LHAAP and the town of Uncertain are in the Goose Prairie Creek drainage area (fig. 2). Because PCBs were not detected in the cores from other sites in Caddo Lake and could have been used in industrial activities at the LHAAP, the LHAAP is a potential source of the PCBs to the sediments at the Goose Prairie Creek site. Uncertain would seem an unlikely source of PCBs to the environment.

All PAHs were detected at concentrations below SQGs in the Caddo Lake sediment cores. PAHs in the sediment samples often are from uncombusted sources, as indicated by the dominance of the alkylated naphthalenes in the PAH mixtures and PAH ratio computations. Uncombusted sources are common in the Caddo Lake area, with several oil and gas fields near the lake (fig. 1), oil and gas wells in the lake, and an airport, roads, marina, and numerous boat ramps on or near the shores (fig. 3). Spills or leaks from oil or gas wells, automobiles, aircraft, or boats in or near the lake could contribute PAHs to the sediments in the drainage area and lake. The composition of oil is highly variable depending upon the type of oil, weathering, refinery, and length of use (such as with a lubricant) (for example, Short and others, 1999; Pruell and Quinn, 1988). Likewise, the composition of gasoline is highly variable depending upon the type of gasoline (such as unleaded gasoline for automobiles, aviation gasoline, various grades, octane ratings, and additives) (Irwin and others, 1998). Regardless of their composition, uncombusted sources such as oil and gasoline are characterized by low molecular weight and alkylated PAHs (Eganhouse and Gossett, 1991). However, differentiation among uncombusted sources using the PAH assemblage in the Caddo Lake sediment core samples was not possible.

The only exceedance of a PEC was one lead concentration in a sample from the Goose Prairie Creek site. Lead concentrations at the Goose Prairie Creek site were substantially higher than at the other three sites, although the trend has been steadily downward from a peak about 1961 (fig. 9). As previously described (Review of Existing Information), lead has been detected in soil and sediment samples collected from the LHAAP. Based on this evidence and the sampling site's proximity to the LHAAP, the LHAAP could have been a principal source of the lead detected at the Goose Prairie Creek site. If the 1961–2002 decreasing trend at the Goose Prairie Creek site continues, lead concentrations there should be comparable to concentrations at the other three sites in a few years.

Like lead concentrations, mercury concentrations at the Goose Prairie Creek site were substantially higher than at the other three sites and have been trending downward from a peak about 1961 (fig. 9). Although no reports of mercury use or discharge at the LHAAP have been found, the FF for the Goose Prairie Creek site, unlike FFs for the Harrison Bayou and mid-lake sites (no FF for Carter Lake site), indicates that a substantial part of the mercury at the Goose Prairie Creek site might have come from drainage area sources in addition to atmospheric deposition. The LHAAP remains a possible source of mercury to the site, although no direct link is evident. If the 1961–2002 downward trend at the Goose Prairie Creek site continues, mercury concentrations there should be comparable to concentrations at the other three sites in 10 to 15 years.

Arsenic, cadmium, and zinc concentrations were highest at the Carter Lake site, the site that was selected to represent background conditions because that location does not receive runoff from the LHAAP, is far from the area of the lake with dense oil and gas operations (fig. 3), and has little development within its drainage basin. However, some producing oil and gas wells are in the waters to the north of the Carter Lake site, at least one abandoned (plugged) oil or gas well is in the small Carter Lake drainage area, and a road and boat ramp are near the coring site. No relation between the relatively higher trace element concentrations and any potential source is indicated. The same potential sources of contamination in the Carter Lake drainage area also are in the drainage areas of the three other coring sites in Caddo Lake.

None of the trace element concentrations was salient at the Harrison Bayou or mid-lake sites, despite numerous producing and abandoned oil and gas wells in the Harrison Bayou drainage area and the proximity of dense oil and gas operations in the lake to the east of the mid-lake site. No specific sources of trace elements detected at those sites are indicated by available data.

SUMMARY

Bottom-sediment cores collected from four sites in Caddo Lake, Texas, in May 2002 were analyzed for radionuclides (for age dating), organochlorine pesticides, PCBs, PAHs, and major and trace elements to describe the occurrence and trends of these sediment-associated contaminants. The sediment cores were obtained from sites near the mouth of Goose Prairie Creek; near the mouths of Harrison Bayou, Central Creek, and Saunders Branch; in the main body of the lake (mid-lake); and in a western part of the lake called Carter Lake. The Goose Prairie Creek and Harrison Bayou sites receive drainage from an area that includes parts of the now-closed Longhorn Army Ammunitions Plant (LHAAP). The mid-lake site is the closest to dense oil and gas operations in the lake. The Carter Lake site receives minimal discharge from developed areas. Sediment deposition dates represented in the cores were computed back to about 1948 for the Goose Prairie Creek site, 1950 for the Harrison Bayou site, 1940 for the mid-lake site, and about 1965 for the Carter Lake site on the basis of the concentrations of ^{137}Cs, ^{210}Pb, or both. Contaminant concentrations were compared to sediment-quality guidelines (SQGs)—threshold effect concentrations (TECs) and probable effect concentrations (PECs)—that indicate the degree of potential harm to benthic biota. Samples were tested for statistically significant temporal trends using Kendall's tau.

The only organochlorine compounds detected in the Goose Prairie Creek core were DDE and PCB Aroclors 1242, 1254, and 1260. Fifty-six percent of the sample concentrations of DDE in the Goose Prairie Creek core exceeded the consensus-based TEC for DDE but were well below the PEC. Only one sample concentration, deposited about 1969 before restrictions on the use of PCBs, exceeded the TEC for total PCB. PCB Aroclors 1242 and 1254 had significant downward trends; no other organochlorines had significant trends.

Numerous PAHs were detected in the Goose Prairie Creek core, although concentrations were low, all below the respective TECs. The naphthalene homologues with two alkyl groups (C2-alkylated naphthalenes) had the highest concentrations. Nineteen PAHs had statistically significant trends—all downward. Since about 1995, PAHs appear to have come primarily from uncombusted sources such as petroleum-based fuels, as opposed to combusted sources associated with the high-temperature burning of fuels.

The Goose Prairie Creek core (and other cores) was analyzed for eight major elements and 26 trace elements, eight of which have SQGs. All samples analyzed for trace elements in the Goose Prairie Creek core exceeded the TEC for chromium, lead, mercury, and nickel, and 78 percent of samples were at or above the TEC for copper and zinc. One sample, deposited about 1961, exceeded the PEC for lead. The concentrations of chromium, lead, and mercury had significant downward trends since about 1961. In sediments deposited after 1980, nickel and zinc had significantly increasing concentrations. Seventy percent of the trace elements without SQGs but with significant trends had decreasing concentrations over time in the core.

In the Harrison Bayou core, DDE was detected in all samples analyzed for organochlorine compounds. Five of the six samples deposited before 1995 had DDE concentrations higher than the TEC but much lower than the PEC. DDE concentrations at the site had a significant downward trend. DDD was detected in the oldest sample only, deposited about 1950. PCB Aroclors 1242, 1254, and 1260 were not detected in the core.

The PAH mixture was dominated by high concentrations of C2- and C3-alkylated naphthalenes, some greater than 2,000 µg/kg. Concentrations of all other compounds were less than 50 µg/kg. Concentrations of most individual PAH compounds were highest in the most recently deposited sample, but most PAH concentrations did not show significant upward trends. All PAHs were below the respective TECs. Uncombusted PAH sources dominated.

Among trace element concentrations in the Harrison Bayou core, chromium, nickel, and zinc concentrations were higher than the respective TECs, but none exceeded the PECs. Chromium, copper, and lead concentrations had significant downward trends, and mercury concentrations had a significant upward trend.

DDE occurred in all samples from the mid-lake site analyzed for organochlorine compounds. Only in the sample deposited in 1976 was a DDE concentration higher than the TEC. DDD was detected in the two oldest samples only, deposited in 1950 and 1967. DDE concentrations had a statistically significant downward trend. PCB Aroclors 1242, 1254, and 1260 were not detected in the core.

PAH concentrations in the mid-lake core were low. As in the previously mentioned cores, the PAH mixture in the mid-lake core was dominated by C2-alkylated naphthalenes. Most individual PAH concentrations were below 20 µg/kg and well below their

TECs. The few significant trends in PAHs were all downward. Uncombusted sources dominated.

Variation in trace element concentrations in the mid-lake core was more subtle than in the other cores—arsenic, manganese, and mercury concentrations had significant upward trends. One or more sample concentrations of arsenic, chromium, nickel, and zinc exceeded the respective TEC; no trace element concentration exceeded the PEC.

Similar to organochlorine analyses from the other three cores, DDE was the most commonly detected compound in Carter Lake samples. Only the oldest sample had a DDE concentration above the TEC, and there was a significant downward trend in DDE concentrations. DDD was detected only in the oldest sample, deposited about 1975. No PCBs were detected.

PAH concentrations in the Carter Lake core were less than the TECs. As in the other cores, the C2-alkylated naphthalenes had the highest concentrations, some greater than 2,000 µg/kg and comparable to those at the Harrison Bayou site. The concentrations of other compounds were about an order of magnitude lower than the C2-alkylated naphthalenes. Among the few PAHs with significant trends, almost all were downward. As in the other cores, uncombusted sources dominated.

Concentrations of several trace elements in the samples from the Carter Lake core exceeded the respective TECs, but none exceeded the PECs. The concentrations of seven of the eight trace elements with SQGs had significant downward trends—all except copper.

Burdens (amount per unit area of a contaminant in a sediment core) of ^{137}Cs and mercury and sediment focusing factors (FF) (quantitative measure of the extent to which contaminants from drainage area sources occur at a site) were computed to provide information on the dominant source (atmospheric fallout or drainage area) of mercury to the Caddo Lake sediment core sites (except Carter Lake). Results indicate that about one-third of the mercury at the Goose Prairie Creek site might result from drainage area sources. No drainage area sources were indicated for the Harrison Bayou and mid-lake sites.

Specific drainage area sources of contaminants detected in the lake sediments are difficult to determine with certainty. The fact that peak concentrations of the DDT degradation products at the Goose Prairie Creek and Harrison Bayou sites, the two sites closest to points of entry of runoff from the LHAAP, were about twice the peak concentrations at the mid-lake and Carter Lake sites, and the fact that DDD, DDE, aldrin, and dieldrin have been detected in soil samples from the LHAAP indicate that the LHAAP is a possible historical source of the detected organochlorines. As PCBs were detected only at the Goose Prairie Creek site, the LHAAP is a possible source for those as well.

The majority of PAH compounds appear to have originated from uncombusted fuel sources. Uncombusted fuel sources are common in the Caddo Lake area, with several oil and gas fields near the lake and oil and gas wells in the lake. Oil and gas production in Caddo Lake and its drainage area is a possible source of PAHs in Caddo Lake sediments.

Among trace element concentrations at the four sites, lead and mercury were consistently relatively high at the Goose Prairie Creek site. Again the LHAAP, because of its proximity and history of industrial activities, is a suspected source. Lead has been detected in soil and sediment samples collected from the LHAAP; mercury has not, although burden and FF computations possibly indicate drainage area sources for part of the mercury in sediment at the site.

Arsenic, cadmium, and zinc concentrations were highest at the Carter Lake site. No direct link between these relatively higher trace element concentrations and oil and gas operations is indicated, but the proximity between the site and oil or gas wells and the absence of other potential sources makes oil and gas operations a possible source.

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APPENDIX 1—
Description of Sediment Push Cores

Appendix 1.1. Description of sediment push cores collected at Caddo Lake, Texas, May 2002

[cm, centimeters]

Depth interval (cm)	Color	Comments
		Goose Prairie Creek Inlet
0–5	Olive-gray	Very watery and soft
5–20	Olive-gray with some darker gray areas	More aquatic vegetation than overlying and underlying intervals; lots of roots; one large white root at 15 cm; similar to box core
20–35	Light gray	Much stiffer clay than the overlying material with a lot of root hairs and wood chunks
		Harrison Bayou Inlet
0–9	Medium olive-gray	Very wet, loose combination of sediment and algae
9–22	Medium olive-gray	Still fairly wet; a lot of stringy organics; leaf veins and pine needles; especially dense vegetation at 15 cm
22–30	Light olive-gray	Thick mass of stringy organics that look like roots
30–40	Light greenish-gray	Wet, clayey, more cohesive sediment
40–55	Light greenish-gray	A lot stiffer and dryer than the overlying interval; big black spot at 50 cm that looks like ash; big wood chunk at 53–55 cm; a few root hairs
		Mid-lake (main stem near Little Green Brake)
0–7	Mostly dark olive-gray with some light olive-gray and rusty brown spots	Very soft with a small amount of sand
7–31	Light gray	Dry, stiff clay; pine needles and charred wood bits speckled throughout
		Carter Lake near Horse Island
0–19	Dark olive-gray	Wet at top, becoming more firm and gelatinous with depth
19–35	Light olive-gray	Gelatinous sediment
35–42	Light olive-gray	Large chunks of wood in the sediment
42–45	Light olive-gray	Gelatinous sediment
45–50	Dark olive-gray	Mat of roots and vegetation
50–75	Medium olive-gray	Stiff, sticky clay; especially hard below 62 cm

APPENDIX 2—
Analytical and Trend Testing Results

Appendix 2.1. Analytical results for radionuclides

[In picocuries per gram; uncertainty, two standard deviations; ID, identifier; --, not applicable]

USGS station ID	Sample ID	Cs-137	Cs-137 uncertainty	Pb-210	Ra-226	Ra-226 uncertainty
		Goose Prairie Creek core:				
324208094073500	CDO.GP 1–2	0.693	0.25	--	--	--
324208094073500	CDO.GP 3–4	.86	.27	--	--	--
324208094073500	CDO.GP 5–6	.921	.231	--	--	--
324208094073500	CDO.GP 8–9	.848	.263	--	--	--
324208094073500	CDO.GP 11–12	.922	.252	--	--	--
324208094073500	CDO.GP 14–15	.964	.204	--	--	--
324208094073500	CDO.GP 17–18	1.1	.19	--	--	--
324208094073500	CDO.GP 20–21	1.3	.211	--	--	--
324208094073500	CDO.GP 22–23	.754	.125	--	--	--
324208094073500	CDO.GP 24–25	.133	.0515	--	--	--
		Harrison Bayou core:				
324113094061900	CDO.HB 1–2	.77	.27	--	--	--
324113094061900	CDO.HB 3–4	.652	.231	--	--	--
324113094061900	CDO.HB 5–6	1.13	.263	--	--	--
324113094061900	CDO.HB 7–8	1.25	.252	--	--	--
324113094061900	CDO.HB 9–10	1.47	.204	--	--	--
324113094061900	CDO.HB 11–12	1.62	.19	--	--	--
324113094061900	CDO.HB 13–14	1.63	.211	--	--	--
324113094061900	CDO.HB 15–16	2.36	.125	--	--	--
324113094061900	CDO.HB 17–18	2.03	.0515	--	--	--
324113094061900	CDO.HB 19–20	1.19	.27	--	--	--
		Mid-lake core:				
324102094041400	CDO.MD 0–1	.399	.27	6.29	1.66	0.37
324102094041400	CDO.MD 1–1.5	.531	.231	6.04	2.03	1.1
324102094041400	CDO.MD 2.5–3	.442	.263	2.84	-.65	1.16
324102094041400	CDO.MD 4–4.5	.503	.252	3.93	1.07	1.04
324102094041400	CDO.MD 5–5.5	.582	.204	5.94	2.08	.589
324102094041400	CDO.MD 6.5–7	.886	.19	5.57	1.66	.598
324102094041400	CDO.MD 8–8.5	.896	.211	4.35	1.98	.883
324102094041400	CDO.MD 9.5–10	1.3	.125	6.35	.824	1.1
324102094041400	CDO.MD 12–13	.2	.0515	3.21	2.08	.473
		Carter Lake core:				
324424094094600	CDO.CR 0.5–1	.339	.27	16.6	3.22	1.13
324424094094600	CDO.CR 2–2.5	.875	.231	10.7	1.2	.832
324424094094600	CDO.CR 3.5–4	.648	.263	13	.416	1.55
324424094094600	CDO.CR 5–6	.632	.252	8.23	.904	.622
324424094094600	CDO.CR 8–9	.84	.204	10	1.7	.54
324424094094600	CDO.CR 11–12	.952	.19	8.29	1.73	.444
324424094094600	CDO.CR 14–15	1.46	.211	8.52	1.65	.389
324424094094600	CDO.CR 17–18	1.45	.125	7.83	1.82	.447
324424094094600	CDO.CR 20–21	2.36	.0515	5.65	1.08	.429

Appendix 2.2. Analytical results for organochlorine compounds

[In micrograms per kilogram dry sediment; ID, identifier; <, less than; E, estimated]

USGS site ID	Sample ID	Group number	Deposition date	Lindane	Heptachlor	Aldrin	Heptachlor epoxide	Technical chlordane	Endo-sulfan I	Dieldrin
Goose Prairie Creek core:										
324208094073500	CDO.GP 0–1	200221106	2001.8	<7.0	<7.0	<7.0	<7.0	<70	<7.0	<7.0
324208094073500	CDO.GP 3–4	200220603	1997.5	<3.5	<3.5	<3.5	<3.5	<35	<3.5	<3.5
324208094073500	CDO.GP 6–7	200220603	1992.9	<1.5	<1.5	<1.5	<1.5	<15	<1.5	<1.5
324208094073500	CDO.GP 9–10	200220603	1987.8	<3.0	<3.0	<3.0	<3.0	<30	<3.0	<3.0
324208094073500	CDO.GP 12–13	200220603	1981.9	<2.5	<2.5	<2.5	<2.5	<25	<2.5	<2.5
324208094073500	CDO.GP 15–16	200220603	1975.7	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324208094073500	CDO.GP 18–19	200220603	1969.1	<2.0	<2.0	<2.0	<2.0	<20	<2.0	<2.0
324208094073500	CDO.GP 21–22	200220603	1961.1	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324208094073500	CDO.GP 24–25	200220603	1948.5	<.5	<.5	<.5	<.5	<5.0	<.5	<.5
Harrison Bayou core:										
324113094061900	CDO.HB 0–1	200221211	2001.5	<8.5	<8.5	<8.5	<8.5	<85	<8.5	<8.5
324113094061900	CDO.HB 2–3	200220603	1996.9	<4.0	<4.0	<4.0	<4.0	<40	<4.0	<4.0
324113094061900	CDO.HB 4–5	200221211	1991.7	<6.0	<6.0	<6.0	<6.0	<60	<6.0	<6.0
324113094061900	CDO.HB 7–8	200221211	1984.9	<2.5	<2.5	<2.5	<2.5	<25	<2.5	<2.5
324113094061900	CDO.HB 10–11	200220603	1977.4	<3.5	<3.5	<3.5	<3.5	<35	<3.5	<3.5
324113094061900	CDO.HB 13–14	200220603	1969.4	<4.0	<4.0	<4.0	<4.0	<40	<4.0	<4.0
324113094061900	CDO.HB 16–17	200221211	1961.3	<3.5	<3.5	<3.5	<3.5	<35	<3.5	<3.5
324113094061900	CDO.HB 19–20	200221211	1950.4	<2.5	<2.5	<2.5	<2.5	<25	<2.5	<2.5
Mid-lake core:										
324102094041400	CDO.MD 0–1	200222605	2001.5	<3.0	<3.0	<3.0	<3.0	<30	<3.0	<3.0
324102094041400	CDO.MD 1.5–2	200221106	1998.4	<1.5	<1.5	<1.5	<1.5	<15	<1.5	<1.5
324102094041400	CDO.MD 3–3.5	200221106	1993.9	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324102094041400	CDO.MD 4.5–5	200221106	1988.9	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324102094041400	CDO.MD 6–6.5	200221106	1982.9	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324102094041400	CDO.MD 7.5–8	200221106	1976.0	<1.0	<1.0	<1.0	<1.0	<10	<1.0	<1.0
324102094041400	CDO.MD 9–9.5	200221106	1967.2	<.5	<.5	<.5	<.5	<5.0	<.5	<.5
324102094041400	CDO.MD 11–12	200221106	1950.8	<.5	<.5	<.5	<.5	<5.0	<.5	<.5
Carter Lake core:										
324424094094600	CDO.CR 1.5–2	200219708	2001.3	<6.5	<6.5	<6.5	<6.5	<65	<6.5	<6.5
324424094094600	CDO.CR 3–3.5	200219708	1999.4	<26	<26	<26	<26	<260	<26	<26
324424094094600	CDO.CR 4.5–5	200221106	1997.6	<15	<15	<15	<15	<150	<15	<15
324424094094600	CDO.CR 7–8	200219708	1993.8	<7.0	<7.0	<7.0	<7.0	<70	<7.0	<7.0
324424094094600	CDO.CR 10–11	200219708	1989.5	<2.0	<2.0	<2.0	<2.0	<20	<2.0	<2.0
324424094094600	CDO.CR 13–14	200219708	1985.2	<5.5	<5.5	<5.5	<5.5	<55	<5.5	<5.5
324424094094600	CDO.CR 16–17	200219708	1980.7	<3.0	<3.0	<3.0	<3.0	<30	<3.0	<3.0
324424094094600	CDO.CR 19–20	200219708	1975.8	<2.5	<2.5	<2.5	<2.5	<25	<2.5	<2.5

Appendix 2.2. Analytical results for organochlorine compounds—Continued

Sample ID	*p,p'*-DDE	Endrin	*p,p'*-DDD	*p,p'*-DDT	Methoxy-chlor	Mirex	Toxa-phene	PCB Aroclor 1242	PCB Aroclor 1254	PCB Aroclor 1260	Calculated values	
											Total PCB	Total DDT
						Goose Prairie Creek core:						
CDO.GP 0–1	2.0	<7.0	<7.0	<7.0	<28	<7.0	<700	<70	<70	E23	E23	E2.0
CDO.GP 3–4	4.1	<3.5	<3.5	<3.5	<14	<3.5	<350	<35	<35	E23	E23	E4.1
CDO.GP 6–7	E1.4	<1.5	<1.5	<1.5	<6.0	<1.5	<150	<15	<15	E11	E11	E1.4
CDO.GP 9–10	5.7	<3.0	<3.0	<3.0	<12	<3.0	<300	<30	E18	E36	E54	E5.7
CDO.GP 12–13	E3.8	<2.5	<2.5	<2.5	<10	<2.5	<250	<25	E13	E22	E35	E3.8
CDO.GP 15–16	2.9	<1.0	<1.0	<1.0	<4.0	<1.0	<100	E3.8	E8.1	16	E28	E2.9
CDO.GP 18–19	7.8	<2.0	<2.0	<2.0	<8.0	<2.0	<200	E8.7	E23	46	E78	E7.8
CDO.GP 21–22	4.0	<1.0	<1.0	<1.0	<4.0	<1.0	<100	E5.2	E14	E16	E35	E4.0
CDO.GP 24–25	.77	<.5	<.5	<5.0	<2.0	<.5	<50	<5.0	<5.0	E2.6	E2.6	E.77
						Harrison Bayou core:						
CDO.HB 0–1	E2.7	<8.5	<8.5	<8.5	<34	<8.5	<850	<85	<85	<85	<255	E2.7
CDO.HB 2–3	E1.4	<4.0	<4.0	<4.0	<16	<4.0	<400	<40	<40	<40	<120	E1.4
CDO.HB 4–5	E3.2	<6.0	<6.0	<6.0	<24	<6.0	<600	<60	<60	<60	<180	E3.2
CDO.HB 7–8	5.1	<2.5	<2.5	<2.5	<10	<2.5	<250	<25	<25	<25	<75	E5.1
CDO.HB 10–11	E3.1	<3.5	<3.5	<3.5	<14	<3.5	<350	<35	<35	<35	<105	E3.1
CDO.HB 13–14	5.2	<4.0	<4.0	<4.0	<16	<4.0	<400	<40	<40	<40	<120	E5.2
CDO.HB 16–17	9.4	<3.5	<3.5	<3.5	<14	<3.5	<350	<35	<35	<35	<105	E9.4
CDO.HB 19–20	7.5	<2.5	E.83	<2.5	<10	<2.5	<250	<25	<25	<25	<75	E8.3
						Mid-lake core:						
CDO.MD 0–1	E1.6	<3.0	<3.0	<3.0	<12	<3.0	<300	<30	<30	<30	<90	E1.6
CDO.MD 1.5–2	E1.3	<1.5	<1.5	<1.5	<6.0	<1.5	<150	<15	<15	<15	<45	E1.3
CDO.MD 3–3.5	1.2	<1.0	<1.0	<1.0	<4.0	<1.0	<100	<10	<10	<10	<30	E1.2
CDO.MD 4.5–5	1.4	<1.0	<1.0	<1.0	<4.0	<1.0	<100	<10	<10	<10	<30	E1.4
CDO.MD 6–6.5	1.7	<1.0	<1.0	<1.0	<4.0	<1.0	<100	<10	<10	<10	<30	E1.7
CDO.MD 7.5–8	3.5	<1.0	<1.0	<1.0	<4.0	<1.0	<100	<10	<10	<10	<30	E3.5
CDO.MD 9–9.5	2.8	<.5	E.36	<.5	<2.0	<.5	<50	<5.0	<5.0	<5.0	<15	E3.2
CDO.MD 11–12	2.3	<.5	.74	<.5	<2.0	<.5	<50	<5.0	<5.0	<5.0	<15	E3.0
						Carter Lake core:						
CDO.CR 1.5–2	<6.5	<6.5	<6.5	<6.5	<26	<6.5	<650	<65	<65	<65	<195	<19.5
CDO.CR 3–3.5	<26	<26	<26	<26	<104	<26	<2,600	<260	<260	<260	<780	<78
CDO.CR 4.5–5	<15	<15	<15	<15	<62	<15	<1,500	<150	<150	<150	<450	<45
CDO.CR 7–8	E2.4	<7.0	<7.0	<7.0	<28	<7.0	<700	<70	<70	<70	<210	E2.4
CDO.CR 10–11	E.68	<2.0	<2.0	<2.0	<8.0	<2.0	<200	<20	<20	<20	<60	E.68
CDO.CR 13–14	E2.4	<5.5	<5.5	<5.5	<22	<5.5	<550	<55	<55	<55	<165	E2.4
CDO.CR 16–17	E2.5	<3.0	<3.0	<3.0	<12	<3.0	<300	<30	<30	<30	<90	E2.5
CDO.CR 19–20	4.1	<2.5	E2.2	<2.5	<10	<2.5	<250	<25	<25	<25	<75	E6.3

Appendix 2.3. Analytical results for polycyclic aromatic hydrocarbons

[In micrograms per kilogram dry sediment; ID, identifier; E, estimated; <, less than]

USGS site ID	Sample ID	Group number	Depo-sition date	Naph-thalene	C1-128 isomers, methylated Naph-thalenes	2-Ethyl-naph-thalene	2,6-Di-methyl-naph-thalene	1,6-Dimethyl-naph thalene	C2-128 isomers, C2-alkylated Naph-thalenes	Ace-naph-thylene	1,2-Di-methyl-naph-thalene
					Goose Prairie core:						
324208094073500	CDO.GP 0–1	8022R02169	2001.8	E8.9	<550	<75	604	102	762	E13.3	E62.1
324208094073500	CDO.GP 3–4	8022R02169	1997.5	E6.3	<315	<35	110	E26.5	212	E6.0	E27.8
324208094073500	CDO.GP 6–7	8022R02169	1992.9	E2.6	<80	<20	22.6	E6.8	39.8	E2.9	E7.9
324208094073500	CDO.GP 9–10	8022R02169	1987.8	E5.7	<100	<30	E22.1	E10.6	45.4	E5.6	E15.0
324208094073500	CDO.GP 12–13	8022R02169	1981.9	E4.9	<80	<25	E16.1	E9.0	42.5	E4.2	E6.6
324208094073500	CDO.GP 15–16	8022R02169	1975.7	E2.6	<40	E.87	E7.0	E4.0	13.9	E2.0	E3.4
324208094073500	CDO.GP 18–19	8022R02169	1969.1	E6.5	<60	<20	E10.0	E5.8	37.4	E4.0	E6.3
324208094073500	CDO.GP 21–22	8022R02169	1961.1	E3.0	<40	<10	E8.9	E5.7	20.7	E3.0	E5.0
324208094073500	CDO.GP 24–25	8022R02169	1948.5	E2.0	<25	<5	9.9	E2.8	20.5	E2.0	E2.8
					Harrison Bayou core:						
324113094061900	CDO.HB 0–1	8022R02177	2001.5	<90	<540	<90	1,340	268	2,250	<90	E40.0
324113094061900	CDO.HB 2–3	8022R02169	1996.9	E5.3	<100	<40	108	E17.1	190	E5.7	E12.4
324113094061900	CDO.HB 4–5	8022R02177	1991.7	E18.6	<690	<65	2,100	348	2,380	<65	<65
324113094061900	CDO.HB 7–8	8022R02177	1984.9	<30	<600	<30	1,590	281	2,050	<30	E24.0
324113094061900	CDO.HB 10–11	8022R02169	1977.4	E4.2	E6.4	E2.3	E30.6	E10.1	73.5	E4.4	E5.4
324113094061900	CDO.HB 13–14	8022R02169	1969.4	E7.4	E12.6	<45	47.9	E17.6	86.8	<45	<45
324113094061900	CDO.HB 16–17	8022R02177	1961.3	E6.4	<380	<35	462	112	736	<35	E15.3
324113094061900	CDO.HB 19–20	8022R02177	1950.4	<30	<220	<30	438	75.1	591	E6.1	E11.8
					Mid-lake core:						
324102094041400	CDO.MD 0–1	8022R02171	2001.5	E4.4	<100	<30	E25.3	E9.9	50.4	E4.7	<30
324102094041400	CDO.MD 1.5–2	8022R02171	1998.4	E2.7	<160	<20	106	E16.0	198	E3.3	<20
324102094041400	CDO.MD 3–3.5	8022R02171	1993.9	E2.0	<100	<10	77.4	E8.2	108	E2.2	<10
324102094041400	CDO.MD 4.5–5	8022R02171	1988.9	E2.3	<20	<10	95.1	10.5	130	<10	<10
324102094041400	CDO.MD 6–6.5	8022R02171	1982.9	E2.0	<70	<10	48.4	E6.5	71.5	E2.1	<10
324102094041400	CDO.MD 7.5–8	8022R02171	1976.0	E2.6	<60	<10	44.2	E7.2	61.6	E2.3	<10
324102094041400	CDO.MD 9–9.5	8022R02171	1967.2	E2.9	<90	<10	59.0	E7.8	80.6	<10	<10
324102094041400	CDO.MD 11–12	8022R02171	1950.8	E3.0	<20	<10	21.8	E3.1	31.0	<10	<10
					Carter Lake core:						
324424094094600	CDO.CR 1.5–2	8022R02165	2001.3	<60	<300	<60	1,200	174	1,660	<60	<60
324424094094600	CDO.CR 3–3.5	8022R02165	1999.4	E22.9	<500	<270	1,600	E248	2,230	<270	E62.3
324424094094600	CDO.CR 4.5–5	8022R02165	1997.6	E12.8	<400	<125	1,520	250	2,160	<125	<125
324424094094600	CDO.CR 7–8	8022R02165	1993.8	<60	<300	<60	1,150	189	1,430	<60	<60
324424094094600	CDO.CR 10–11	8022R02165	1989.5	E2.9	<30	<25	134	E17.8	178	E3.6	<25
324424094094600	CDO.CR 13–14	8022R02165	1985.2	E8.5	<60	<60	292	E58.0	409	E7.8	E11.4
324424094094600	CDO.CR 16–17	8022R02165	1980.7	<30	<50	<30	235	40.0	315	<30	<30
324424094094600	CDO.CR 19–20	8022R02165	1975.8	<30	<50	<30	205	E28.3	294	<30	E6.6

Appendix 2.3. Analytical results for polycyclic aromatic hydrocarbons—Continued

Sample ID	Ace-naph-thene	C3-128 isomers, C3-alkylated Naphtha-lenes	2,3,6-Tri-methyl-naph-tha-lene	9H-Fluor-ene	C4-128 isomers, C4-alkylated Naphtha-lenes	1-methyl-9H-Fluor-ene	Phe-nan-threne	Anthra-cene	C5-128 isomers, C5-alkylated Naphtha-lenes	2-Methyl-anthra-cene	4,5-Methyl-ene-phenan-threne	C1-178 isomers, methylated Phenan-threne/ anthra-cenes
Goose Prairie core:												
CDO.GP 0–1	E10.9	<140	<75	E11.8	<75	E22.5	E25.5	E22.8	<75	<75	E15.0	<300
CDO.GP 3–4	E4.4	<70	E4.0	E9.0	<70	E10.8	E18.8	E13.2	<35	<35	E11.0	<130
CDO.GP 6–7	E1.8	<20	<20	E4.4	<20	<20	E8.7	E5.8	<20	<20	E5.2	<20
CDO.GP 9–10	E3.2	E25.7	E3.7	E9.4	<30	E9.0	E23.2	E12.4	<30	E6.8	E13.9	35.3
CDO.GP 12–13	E2.6	E16.6	E3.0	E9.2	<25	E7.1	E20.2	E9.7	<25	E5.0	E14.2	27.0
CDO.GP 15–16	E1.3	<15	E1.6	E5.4	<10	E4.2	10.1	E5.3	<10	E2.7	10.0	16.8
CDO.GP 18–19	E2.4	35.1	E3.5	E14.4	<20	E9.3	E19.6	E11.9	<20	E5.2	25.8	36.1
CDO.GP 21–22	E1.5	23.7	E2.6	11.5	<15	E7.0	11.7	E8.8	<10	E3.5	21.5	30.3
CDO.GP 24–25	E1.2	10.7	E1.7	11.4	<10	E4.5	5.9	5.8	<5	E2.4	13.8	20.2
Harrison Bayou core:												
CDO.HB 0–1	<90	448	E14.5	E11.8	<90	<90	E26.0	E25.5	<90	<90	<90	<400
CDO.HB 2–3	E4.0	<60	<40	E7.0	<40	E9.8	E13.2	E10.7	<40	<40	E11.6	<230
CDO.HB 4–5	<65	514	E13.5	E10.7	<65	<65	E20.7	E22.1	<65	<65	<65	<400
CDO.HB 7–8	E5.1	332	E8.3	E5.9	<75	<30	E19.3	E20.4	<30	<30	E15.2	<360
CDO.HB 10–11	E2.2	E31.9	E3.6	E4.4	<40	E6.8	E10.4	E7.6	<40	E6.8	E9.7	<50
CDO.HB 13–14	E3.5	45.0	E4.9	E6.2	<45	E10.5	E19.1	E12.2	<45	<45	E16.6	<70
CDO.HB 16–17	<35	<160	E5.8	E5.6	<35	E15.2	E23.5	E18.0	<35	<35	E18.8	<100
CDO.HB 19–20	E3.3	<100	E4.4	E4.6	<30	E10.9	E16.1	E12.4	<30	<30	E13.2	<65
Mid-lake core:												
CDO.MD 0–1	<30	<30	<30	<30	<30	<30	E6.4	E6.7	<30	<30	E4.9	<30
CDO.MD 1.5–2	E1.3	<90	<20	<20	<20	<20	E5.0	E5.3	<20	<20	<20	<20
CDO.MD 3–3.5	E1.1	<50	<10	E2.1	<10	<10	E4.9	E4.1	<10	<10	E2.9	<10
CDO.MD 4.5–5	<10	<60	<10	E1.5	<10	<10	E3.2	E3.7	<10	<10	<10	10.9
CDO.MD 6–6.5	E1.1	<40	<10	E1.6	<10	<10	E3.4	E3.5	<10	<10	E2.1	<10
CDO.MD 7.5–8	E.98	<30	<10	E1.6	<10	<10	E3.8	E3.7	<10	<10	<10	E8.2
CDO.MD 9–9.5	<10	<30	<10	E2.0	<10	<10	E6.8	E5.1	<10	<10	E2.8	13.4
CDO.MD 11–12	<10	<50	<10	<10	<10	E2.5	E2.3	E2.3	<10	<10	E1.8	E7.3
Carter Lake core:												
CDO.CR 1.5–2	<60	<340	<60	<60	<60	<60	E13.4	<60	<60	<60	<60	<120
CDO.CR 3–3.5	<270	<550	<270	<270	<270	<270	E34.8	<270	<270	<270	<270	<270
CDO.CR 4.5–5	<125	<500	<125	<125	<125	<125	E24.6	E28.7	<125	<125	<125	<130
CDO.CR 7–8	<60	<300	<60	<60	<60	<60	E17.6	E19.3	<60	<60	<60	<70
CDO.CR 10–11	<25	<60	<25	E3.2	<25	E5.7	E5.5	E5.7	<25	<25	E5.9	<25
CDO.CR 13–14	<60	<140	<60	E7.6	<60	E13.2	E15.4	E12.4	<60	<60	E16.1	<60
CDO.CR 16–17	<30	<100	<30	<30	<30	<30	E8.9	E5.5	<30	<30	E9.6	<40
CDO.CR 19–20	E2.5	<100	<30	E4.7	<30	E8.0	E11.2	E8.4	<30	E6.9	E11.6	<30

Appendix 2.3. Analytical results for polycyclic aromatic hydrocarbons—Continued

Sample ID	1-Methyl-phenan-threne	C2-178 isomers, C2-alkylated Phenan-threne/ anthracenes	Fluor-an-thene	Py-rene	C3-178 isomers, C3-alkylated Phenan-threne/ anthracenes	C4-178 isomers, C4-alkylated Phenan-threne/ anthracenes	1-Methyl-pyrene	C1-202 isomers, methylated Fluoran-thene/ pyrenes	C2-202 isomers, C2-alkylated Fluoran-thene/ pyrenes	C5-178 isomers, C5-alkylated Phenan-threne/ anthracenes	Benz-(a)-anthra-cene
Goose Prairie core:											
CDO.GP 0–1	<75	<75	E34.8	E28.0	<150	<700	E10.4	<1,800	<250	<75	E25.5
CDO.GP 3–4	E7.8	<35	E31.4	E24.9	<35	<400	<35	<730	150	<35	E13.7
CDO.GP 6–7	E4.1	<20	E16.0	E12.0	<20	<20	<20	E7.9	37.8	<20	E6.6
CDO.GP 9–10	E9.4	E19.4	46.4	30.2	E15.3	<30	E5.3	E26.7	<50	<30	E14.3
CDO.GP 12–13	E6.9	E16.1	46.0	29.5	E11.3	<25	E4.1	32.1	54.5	<25	E12.8
CDO.GP 15–16	E4.3	14.2	30.2	19.2	<25	<10	E2.2	10.3	<20	<10	E6.2
CDO.GP 18–19	E8.8	25.0	75.3	44.5	E14.5	<20	E4.0	39.8	<60	<20	E12.9
CDO.GP 21–22	E5.7	27.0	70.9	37.4	14.7	<10	E2.8	66.4	<30	<10	10.5
CDO.GP 24–25	E3.3	19.7	53.4	30.0	17.0	<10	E1.9	23.5	<20	<5	7.2
Harrison Bayou core:											
CDO.HB 0–1	<90	<90	E49.6	E43.8	<90	<90	<90	<90	<90	<90	E24.3
CDO.HB 2–3	E7.6	<40	E36.3	E34.9	<40	<40	<40	E37.3	81.3	<40	E16.2
CDO.HB 4–5	E54.2	<65	E41.4	E36.8	<65	<65	<65	<65	<150	<65	E18.9
CDO.HB 7–8	53.0	<75	36.5	30.8	<30	<30	E6.4	<30	<100	<30	E16.6
CDO.HB 10–11	E8.1	E13.7	E22.4	E25.2	E9.9	<40	E5.4	E21.3	52.2	<40	E13.8
CDO.HB 13–14	E13.1	E26.0	E43.4	45.8	E19.4	<45	<45	<45	<60	<45	E16.6
CDO.HB 16–17	E11.7	35.8	44.7	46.4	<35	<35	E7.7	<35	<35	<35	E18.7
CDO.HB 19–20	E6.7	<30	31.2	31.8	<30	<30	E5.5	<40	<30	<30	E13.3
Mid-lake core:											
CDO.MD 0–1	<30	<30	15.0	17.2	<30	<30	<30	<30	<30	<30	<30
CDO.MD 1.5–2	<20	<20	E12.3	E14.0	<20	<20	<20	<20	<20	<20	E7.2
CDO.MD 3–3.5	<10	<10	13.7	14.6	<10	<10	<10	<10	<10	<10	E7.0
CDO.MD 4.5–5	E1.9	E9.7	E9.5	12.0	<10	<10	<10	E5.3	<10	<10	<10
CDO.MD 6–6.5	<10	E8.1	E8.0	11.4	E8.3	E5.8	<10	<10	<10	<10	<10
CDO.MD 7.5–8	<10	10.4	E9.5	14.9	<10	<10	<10	<10	<10	<10	<10
CDO.MD 9–9.5	<10	13.6	17.0	21.7	<10	13.4	<10	<10	<10	<10	10.1
CDO.MD 11–12	E1.6	E8.2	E6.1	12.6	E7.1	<10	<10	<10	<10	<10	E3.2
Carter Lake core:											
CDO.CR 1.5–2	E21.0	<60	E12.8	159	<60	<1,200	E22.8	<60	<60	<60	78.6
CDO.CR 3–3.5	<270	<270	E60.7	E57.4	<270	<270	<270	<270	<270	<270	<270
CDO.CR 4.5–5	E33.0	<125	E43.0	E41.5	<125	<400	<125	<125	<125	<125	<125
CDO.CR 7–8	E15.7	<60	E28.1	E32.6	<60	<60	<60	<60	<60	<60	<60
CDO.CR 10–11	E5.6	<25	E11.7	E14.1	<25	<25	<25	<25	<25	<25	<25
CDO.CR 13–14	E12.9	<60	E30.3	E41.0	<60	<60	<60	<60	<60	<60	<60
CDO.CR 16–17	E7.0	<30	E16.8	E26.0	<30	<30	<30	<30	<30	<30	<30
CDO.CR 19–20	E6.9	<30	E20.9	33.6	<30	<30	<30	<30	<30	<30	<30

Appendix 2.3. Analytical results for polycyclic aromatic hydrocarbons—Continued

Sample ID	Chrysene	C3-202 isomers, C3-alkylated Fluoranthene/pyrenes	C1-228 isomers, methylated Benzo(a)-anthracene/chrysenes	C4-202 isomers, C4-alkylated Fluoranthene/pyrenes	C5-202 isomers, C5-alkylated Fluoranthene/pyrenes	C2-228 isomers, C2-alkylated Benzo(a)-anthracene/chrysenes	Benzo(b)-fluoranthene	Benzo(k)-fluoranthene	Benzo(e)-pyrene	Benzo(a)-pyrene	C1-252 isomers, C1-methylated Benzo-pyrene/perylenes
Goose Prairie core:											
CDO.GP 0–1	E23.2	<325	<90	<75	<75	<75	E35.2	E20.0	E20.8	E20.2	<75
CDO.GP 3–4	E17.4	<260	<35	<35	<35	<35	E24.8	E15.9	E13.2	E12.6	<35
CDO.GP 6–7	E7.8	<20	<20	<20	<20	<20	E11.0	E5.6	E5.6	E5.3	<20
CDO.GP 9–10	E18.4	<30	E18.4	<30	<30	<30	E23.7	E15.8	E13.1	E11.1	<30
CDO.GP 12–13	E15.2	<75	<25	<25	<25	<25	E17.0	E10.0	E9.3	E8.3	<25
CDO.GP 15–16	10.2	<10	<10	<10	<10	<10	10.0	E6.1	E5.6	E4.2	<10
CDO.GP 18–19	22.1	<40	E15.2	<20	<20	<20	E19.3	E12.4	E12.0	E8.8	<20
CDO.GP 21–22	17.4	<10	11.8	<20	<10	<10	13.4	E8.2	E7.9	E5.5	<10
CDO.GP 24–25	10.9	<20	<10	<5	<5	<5	9.5	6.3	5.7	E3.0	<5
Harrison Bayou core:											
CDO.HB 0–1	E36.6	<90	<90	<90	<90	<90	E41.6	E26.5	E21.4	E24.8	<90
CDO.HB 2–3	E15.3	<100	<40	<40	<40	<40	E22.3	E13.2	E11.9	E13.0	<40
CDO.HB 4–5	E21.6	<65	<65	<65	<65	<65	E38.5	E18.0	E18.3	E20.1	<65
CDO.HB 7–8	E20.0	<50	<30	<30	<30	<30	35.0	E17.7	E16.5	E16.3	<30
CDO.HB 10–11	E10.8	<40	<40	<40	<40	<40	E18.4	E10.4	E8.6	E10.3	<40
CDO.HB 13–14	E20.4	<70	<45	<45	<45	<45	E26.6	E19.6	E14.2	E16.4	<45
CDO.HB 16–17	E25.2	<35	<35	<35	<35	<35	40.0	E24.3	E19.2	E21.0	<35
CDO.HB 19–20	E18.8	<30	<30	<30	<30	<30	32.0	E16.5	E16.3	E15.2	<30
Mid-lake core:											
CDO.MD 0–1	E9.7	<30	<30	<30	<30	<30	E17.8	E7.5	E9.6	E10.4	E22.8
CDO.MD 1.5–2	E7.8	<20	E4.8	<20	<20	<20	13.3	E6.5	E8.4	E7.5	25.2
CDO.MD 3–3.5	E7.5	<10	<10	<10	<10	<10	13.4	E4.8	E7.3	E7.0	25.2
CDO.MD 4.5–5	E6.6	<10	<10	<10	<10	<10	E9.9	E5.7	E6.2	E5.6	26.7
CDO.MD 6–6.5	E6.2	<10	<10	<10	<10	<10	10.9	E4.4	E6.2	E5.2	32.1
CDO.MD 7.5–8	E7.5	<10	<10	<10	<10	<10	12.8	E6.6	E7.9	E6.0	58.6
CDO.MD 9–9.5	11.9	<10	<10	<10	<10	<10	18.6	E8.5	10.5	10.4	54.4
CDO.MD 11–12	E5.3	<10	<10	<10	<10	<10	E7.2	E6.1	E4.9	E3.9	36.3
Carter Lake core:											
CDO.CR 1.5–2	73.9	<1,900	<60	<60	<60	<60	E28.5	E14.9	E14.8	E18.2	<60
CDO.CR 3–3.5	E31.9	<310	<270	<270	<270	<270	E72.7	E30.7	E33.8	E54.1	<270
CDO.CR 4.5–5	E27.3	<440	<125	<125	<125	<125	E48.0	E23.5	E22.2	E33.6	<125
CDO.CR 7–8	E14.2	<400	<60	<60	<60	<60	E33.8	E19.3	E16.3	E20.6	71.1
CDO.CR 10–11	132	<150	<25	<25	<25	<25	E10.4	E5.8	E5.3	E6.9	E14.9
CDO.CR 13–14	E17.5	<240	<60	<60	<60	<60	E29.8	E13.4	E13.6	E17.9	E40.8
CDO.CR 16–17	E9.8	<130	<30	<30	<30	<30	E18.7	E7.5	E9.2	E10.8	32.8
CDO.CR 19–20	E13.0	<130	<30	<30	<30	<30	E25.6	E10.0	E11.0	E12.0	32.9

Appendix 2.3. Analytical results for polycyclic aromatic hydrocarbons—Continued

Sample ID	C3-228 isomers, C3-Benzo(a)-anthracene/chrysenes	C2-252 isomers, C2-alkylated Benzo-pyrene/perylenes	C4-228 isomers, C4-Benzo(a)-anthracene/chrysenes	Benzo-(g,h,i)-perylene	Indeno-(1,2,3-c,d)-pyrene	Dibenzo-(a,h)-anthracene	C3-252 isomers, C3-alkylated Benzo-pyrene/perylenes	C4-252 isomers, C4-alkylated Benzo-pyrene/perylenes	C5-228 isomers, C5-Benzo(a)-anthracene/chrysenes	C5-252 isomers, C5-alkylated Benzo-pyrene/perylenes	Coronene
Goose Prairie core:											
CDO.GP 0–1	<75	<75	<75	<75	E45.9	<75	<75	<75	<75	<75	<75
CDO.GP 3–4	<35	<35	<35	E12.7	E25.5	<35	<35	<35	<35	<35	<35
CDO.GP 6–7	<20	<20	<20	E5.8	E10.7	E5.8	<20	<20	<20	<20	<20
CDO.GP 9–10	<30	<30	<30	E13.4	E19.1	E10.2	<30	<30	<30	<30	E9.0
CDO.GP 12–13	<25	<25	<25	E7.9	E11.8	E7.7	<25	<25	<25	<25	E5.3
CDO.GP 15–16	<10	<10	<10	E4.8	E6.6	E3.6	<10	<10	<10	<10	E3.0
CDO.GP 18–19	<20	<20	<20	E9.1	E11.6	E6.8	<20	<20	<20	<20	E5.1
CDO.GP 21–22	<10	<10	<10	E7.5	E8.5	<10	<10	<10	<10	<10	E3.4
CDO.GP 24–25	<5	<5	<5	6.0	6.2	E2.8	<5	<5	<10	<5	E2.5
Harrison Bayou core:											
CDO.HB 0–1	<90	<90	<90	E27.4	E46.2	<90	<90	<90	<90	<90	E22.5
CDO.HB 2–3	<40	<40	<40	E14.4	E20.5	<40	<40	<40	<40	<40	<40
CDO.HB 4–5	<65	<65	<65	<65	E39.1	<65	<65	<65	<65	<65	E15.6
CDO.HB 7–8	<30	<30	<30	35.4	E14.2	<30	<30	<30	<30	<30	E14.8
CDO.HB 10–11	<40	<40	<40	E9.9	E14.6	E10.6	<40	<40	<40	<40	E7.0
CDO.HB 13–14	<45	<45	<45	E15.9	E21.8	E14.0	<45	<45	<45	<45	<45
CDO.HB 16–17	<35	<35	<35	E29.0	36.8	<35	<35	<35	<35	<35	E17.3
CDO.HB 19–20	<30	<30	<30	E20.4	E28.2	E12.1	<30	<30	<30	<30	E14.1
Mid-lake core:											
CDO.MD 0–1	<30	<30	<30	E14.5	E16.4	E10.8	<30	<30	<30	<30	<30
CDO.MD 1.5–2	<20	<20	<20	E10.9	E13.4	<20	<20	<20	<20	<20	<20
CDO.MD 3–3.5	<10	<10	<10	10.4	11.1	E4.7	<10	<10	<10	<10	<10
CDO.MD 4.5–5	<10	E6.8	<10	E8.9	10.1	E3.9	<10	<10	<10	<10	<10
CDO.MD 6–6.5	<10	<10	<10	E8.3	E8.9	<10	<10	<10	<10	<10	<10
CDO.MD 7.5–8	<10	<10	<10	E13.6	E12.6	E4.2	<10	<10	<10	<10	E4.9
CDO.MD 9–9.5	<10	<10	<10	<15	16.2	<10	<10	<10	<10	<10	<10
CDO.MD 11–12	<10	<10	<10	E6.9	E7.9	E3.1	<10	<10	<10	<10	E3.3
Carter Lake core:											
CDO.CR 1.5–2	<60	79.6	<60	<60	<60	<60	<60	<60	<60	<60	E12.7
CDO.CR 3–3.5	<270	<270	<270	<270	E89.3	<270	<270	<270	<270	<270	<270
CDO.CR 4.5–5	<125	<125	<125	<125	E56.6	<125	<125	<125	<125	<125	<125
CDO.CR 7–8	<60	<60	<60	<60	E37.4	<60	<60	<60	<60	<60	<60
CDO.CR 10–11	<25	<25	<25	E8.6	E10.4	E6.8	<25	<25	<25	<25	E6.5
CDO.CR 13–14	<60	<60	<60	E20.3	E24.6	E16.9	<60	<60	<60	<60	<60
CDO.CR 16–17	<30	<30	<30	E14.7	E17.5	E10.0	<30	<30	<30	<30	<30
CDO.CR 19–20	<30	<30	<30	E16.4	E19.5	E9.3	<30	<30	<30	<30	E8.5

Appendix 2.4. Analytical results for forms of carbon and major and trace elements

[In micrograms per gram dry sediment unless otherwise specified; ID, identifier; <, less than; na, not analyzed]

USGS site ID	Sample ID	Group number	Deposition date	Inorganic carbon, as CO_3 (percent)	Organic carbon (percent)	Total carbon (percent)	Aluminum	Calcium	Iron	Magnesium	Potassium	Phosphorus	Sodium
Goose Prairie Creek core:													
324208094073500	CDO.GP 0–1	3958	2001.8	0.07	18.5	18.6	61,100	4,260	27,000	2,660	6,250	1,780	818
324208094073500	CDO.GP 3–4	3958	1997.5	.04	16.0	16.0	69,100	3,760	25,900	2,740	5,760	1,370	648
324208094073500	CDO.GP 6–7	3958	1992.9	.04	13.9	13.9	71,100	3,330	24,500	2,970	6,310	1,130	760
324208094073500	CDO.GP 9–10	3958	1987.8	.04	12.8	12.8	77,100	3,070	26,000	2,740	6,740	1,050	790
324208094073500	CDO.GP 12–13	3958	1981.9	.03	9.77	9.80	76,500	2,720	25,000	2,760	7,270	791	1,030
324208094073500	CDO.GP 15–16	3958	1975.7	.03	9.00	9.03	69,000	2,410	25,000	3,000	7,130	698	1,130
324208094073500	CDO.GP 18–19	3958	1969.1	.03	7.07	7.10	76,500	2,250	29,700	2,750	7,680	674	1,090
324208094073500	CDO.GP 21–22	3958	1961.1	.02	4.69	4.71	86,700	2,100	29,000	3,240	8,920	739	1,280
324208094073500	CDO.GP 24–25	3958	1948.5	<.01	2.28	2.29	66,900	1,490	20,400	2,450	8,960	372	1,510
Harrison Bayou core:													
324113094061900	CDO.HB 0–1	3958	2001.5	.09	10.4	10.5	72,800	3,660	41,600	3,750	8,440	1,490	1,130
324113094061900	CDO.HB 2–3	3958	1996.9	.08	9.86	9.94	66,900	4,070	40,700	3,900	8,870	1,630	1,960
324113094061900	CDO.HB 4–5	3958	1991.7	.07	11.5	11.6	69,900	3,360	39,800	3,810	8,200	1,340	1,140
324113094061900	CDO.HB 7–8	3958	1984.9	.05	10.3	10.4	73,400	3,650	36,900	4,050	8,540	1,290	1,290
324113094061900	CDO.HB 10–11	3958	1977.4	.04	11.6	11.6	77,400	3,780	37,200	4,230	8,370	990	1,080
324113094061900	CDO.HB 13–14	3958	1969.4	.04	11.3	11.3	76,100	3,860	36,500	4,360	7,950	823	1,110
324113094061900	CDO.HB 16–17	3958	1961.3	.04	10.4	10.4	77,000	3,680	34,500	4,170	8,460	815	1,330
324113094061900	CDO.HB 19–20	3958	1950.4	.03	6.90	6.93	90,700	3,470	36,100	5,080	9,920	693	1,250
Mid-lake core:													
324102094041400	CDO.MD 0–1	3958	2001.5	.05	3.73	3.78	61,800	2,910	56,600	2,410	8,250	1,050	1,160
324102094041400	CDO.MD 1.5–2	3958	1998.4	.05	3.95	4.00	64,000	2,360	56,300	2,380	8,430	1,040	1,180
324102094041400	CDO.MD 3–3.5	3958	1993.9	.04	3.51	3.55	61,600	2,060	52,000	2,310	8,300	796	1,170
324102094041400	CDO.MD 4.5–5	3958	1988.9	.03	3.63	3.66	61,000	2,040	49,500	2,280	8,500	712	1,200
324102094041400	CDO.MD 6–6.5	3958	1982.9	.03	3.36	3.39	65,500	2,190	49,300	2,450	9,000	624	1,260
324102094041400	CDO.MD 7.5–8	3958	1976.0	.03	3.13	3.16	60,200	2,220	42,000	3,000	9,140	549	1,550
324102094041400	CDO.MD 9–9.5	3958	1967.2	.01	2.53	2.54	61,100	2,060	32,600	2,270	9,250	388	1,380
324102094041400	CDO.MD 11–12	3958	1950.8	<.01	2.21	2.22	76,000	2,400	29,200	3,700	10,200	351	1,600
Carter Lake core:													
324424094094600	CDO.CR 0–0.5	3959	2002.3	.09	10.5	10.6	67,900	3,180	46,000	2,960	6,260	1,600	892
324424094094600	CDO.CR 1–1.5	3959	2001.6	.08	10.4	10.5	68,200	3,480	49,000	2,700	6,050	1,700	728
324424094094600	CDO.CR 1.5–2	3959	2001.3	.08	10.9	11.0	71,400	2,970	49,000	2,650	6,190	1,600	697
324424094094600	CDO.CR 3–3.5	3959	1999.4	.08	11.2	11.3	68,100	2,900	48,000	2,700	6,210	1,600	749
324424094094600	CDO.CR 4–4.5	3959	1998.1	.08	10.5	10.6	66,600	2,730	47,000	2,670	5,920	1,500	705
324424094094600	CDO.CR 4.5–5	3959	1997.6	.08	10.2	10.3	72,400	2,880	50,000	2,690	6,390	1,600	746
324424094094600	CDO.CR 7–8	3959	1993.8	.08	9.56	9.64	77,900	2,750	51,000	2,700	6,640	1,700	695
324424094094600	CDO.CR 9–10	3959	1990.9	.09	9.73	9.82	77,300	2,760	52,000	2,770	6,520	1,800	682
324424094094600	CDO.CR 10–11	3959	1989.5	.10	9.55	9.65	73,800	2,660	52,000	2,680	6,170	2,000	671
324424094094600	CDO.CR 13–14	3959	1985.2	.09	9.37	9.46	78,000	3,270	56,000	2,760	6,690	3,000	726
324424094094600	CDO.CR 15–16	3959	1982.3	.08	9.92	10.0	77,800	2,760	56,000	2,780	6,610	1,900	723
324424094094600	CDO.CR 16–17	3959	1980.7	.08	9.51	9.59	77,700	2,720	60,000	2,820	6,650	2,000	707
324424094094600	CDO.CR 19–20	3959	1975.8	.09	8.44	8.53	83,800	2,570	66,000	3,030	7,230	1,600	764
324424094094600	CDO.CR 22–23	3959	1969.1	.07	8.06	8.13	93,200	2,640	64,000	3,390	8,360	1,600	868

Appendix 2.4. Analytical results for forms of carbon and major and trace elements—Continued

Sample ID	Titanium	Antimony	Arsenic	Barium	Beryllium	Cadmium	Cerium	Chromium	Cobalt	Copper	Gallium	Lanthanum	Lead	Lithium
Goose Prairie Creek core:														
CDO.GP 0–1	2,230	0.88	6.9	382	1.9	0.54	61.5	51.6	28.4	29.8	14	30.2	50.4	32.2
CDO.GP 3–4	2,690	.93	7.1	373	1.8	.44	57.6	58.9	24.6	31.6	15	29.2	56.0	37.3
CDO.GP 6–7	2,880	.95	7.2	393	1.6	.27	51.8	62.3	16.4	33.5	16	28.2	61.2	39.0
CDO.GP 9–10	3,020	.98	7.8	416	1.4	.24	56.0	67.1	14.1	32.9	16	28.6	71.8	42.8
CDO.GP 12–13	3,190	.92	7.9	435	1.3	.25	57.9	70.2	14.1	33.6	16	29.3	96.8	44.5
CDO.GP 15–16	3,260	1.01	8.3	435	1.5	.32	64.1	69.5	15.0	35.9	15	32.7	111	42.8
CDO.GP 18–19	3,410	1.02	9.2	445	1.7	.38	70.7	76.9	22.2	31.9	16	35.2	127	48.2
CDO.GP 21–22	3,990	1.28	7.9	530	2.0	.41	80.8	90.6	18.4	34.2	19	40.3	181	54.0
CDO.GP 24–25	4,240	.85	3.8	452	1.5	.18	76.7	59.7	9.59	20.8	14	37.8	117	43.1
Harrison Bayou core:														
CDO.HB 0–1	2,920	.67	7.4	531	2.2	.51	71.4	59.4	26.1	24.6	16	38.9	23.9	40.5
CDO.HB 2–3	2,610	.67	6.6	570	2.0	.45	64.6	53.9	25.1	24.1	15	35.2	23.0	37.1
CDO.HB 4–5	2,810	.71	8.0	507	2.2	.50	69.9	57.6	27.0	24.9	16	38.6	24.6	38.9
CDO.HB 7–8	2,930	.75	7.9	509	2.4	.49	74.3	60.2	27.8	26.3	17	39.8	26.2	41.1
CDO.HB 10–11	3,000	.83	8.8	495	2.4	.51	69.8	62.5	25.5	25.1	18	38.3	30.0	43.0
CDO.HB 13–14	2,960	.87	8.4	462	2.3	.50	65.0	61.3	21.8	25.8	17	36.2	29.9	41.5
CDO.HB 16–17	2,690	.83	7.4	496	2.2	.44	62.7	60.2	19.7	26.6	17	34.5	28.0	41.9
CDO.HB 19–20	3,320	1.06	7.9	519	2.4	.42	78.8	72.3	20.3	26.2	20	41.5	35.1	49.2
Mid-lake core:														
CDO.MD 0–1	3,010	.58	11	516	2.2	.24	82.2	50.7	18.1	13.9	14	42.3	25.0	40.2
CDO.MD 1.5–2	3,420	.64	10	504	2.3	.26	84.3	52.6	18.2	14.2	14	42.9	24.8	42.0
CDO.MD 3–3.5	3,060	.62	7.5	488	2.2	.24	80.6	50.5	18.7	14.4	13	41.4	24.9	42.1
CDO.MD 4.5–5	3,190	.63	6.6	492	2.1	.24	81.9	51.9	19.0	13.6	13	44.5	25.2	41.6
CDO.MD 6–6.5	3,360	.69	6.4	498	2.3	.25	86.8	53.9	19.8	15.5	14	47.2	27.6	44.5
CDO.MD 7.5–8	3,440	.73	6.9	484	2.6	.28	89.4	52.3	21.0	15.9	14	46.1	27.0	42.7
CDO.MD 9–9.5	3,630	.65	5.7	492	2.1	.23	85.0	50.3	17.0	14.1	13	42.9	26.4	42.2
CDO.MD 11–12	4,060	.69	7.4	491	2.6	.20	106	63.4	20.9	16.6	18	57.7	23.0	50.2
Carter Lake core:														
CDO.CR 0–0.5	2,600	.86	12	432	3.1	.76	122	59.3	46.7	26.0	16	52.7	28.1	42.2
CDO.CR 1–1.5	2,800	.80	13	448	2.9	.70	122	57.0	48.8	23.3	16	55.4	27.3	41.4
CDO.CR 1.5–2	2,600	.84	13	432	2.9	.74	122	60.6	52.7	23.8	16	52.0	27.3	42.3
CDO.CR 3–3.5	2,600	.84	13	446	3.0	.71	119	57.5	50.5	23.5	16	51.0	27.1	41.4
CDO.CR 4–4.5	2,500	.85	13	451	3.0	.73	120	56.8	48.2	23.9	15	51.7	28.8	42.5
CDO.CR 4.5–5	2,600	.82	13	447	3.1	.71	123	59.9	48.6	32.4	16	52.8	29.2	43.3
CDO.CR 7–8	3,200	.84	14	440	3.2	.73	130	62.6	49.4	25.0	17	56.0	32.2	45.5
CDO.CR 9–10	2,800	.89	14	436	3.3	.75	132	63.0	50.4	23.7	17	56.4	32.4	45.8
CDO.CR 10–11	3,000	.87	14	449	3.2	.75	128	61.0	51.0	23.9	17	55.3	32.8	44.7
CDO.CR 13–14	3,200	.91	15	481	3.2	.83	134	64.5	53.0	24.1	17	56.9	35.0	46.8
CDO.CR 15–16	2,900	.88	15	439	3.4	.84	125	63.8	51.9	22.8	17	53.8	37.8	46.9
CDO.CR 16–17	3,000	.94	15	443	3.4	.86	122	65.1	51.7	23.1	17	54.0	39.8	47.2
CDO.CR 19–20	3,200	.92	14	458	3.5	.79	125	67.5	44.2	22.4	18	54.5	41.8	51.1
CDO.CR 22–23	3,900	.98	13	507	3.5	.78	122	74.6	37.8	23.3	20	54.0	40.0	57.1

Appendix 2.4. Analytical results for forms of carbon and major and trace elements—Continued

Sample ID	Man-ganese	Mer-cury	Molyb-denum	Nickel	Nio-bium	Scand-ium	Sele-nium	Silver	Stron-tium	Thor-ium	Ura-nium	Vana-dium	Zinc
Goose Prairie Creek core:													
CDO.GP 0–1	1,200	0.37	1.6	35.2	9	9.0	2.0	5.12	62.2	7.7	2.2	80.1	222
CDO.GP 3–4	773	.42	2.0	33.9	11	10.0	1.8	7.79	61.0	8.4	2.4	86.2	189
CDO.GP 6–7	585	.47	2.1	30.8	14	10.7	1.8	9.64	60.9	9.0	2.5	89.6	138
CDO.GP 9–10	479	.52	2.1	29.9	14	11.0	1.6	12.2	61.5	9.2	2.6	93.9	122
CDO.GP 12–13	386	.75	1.7	29.1	13	10.8	1.5	16.6	61.6	9.6	2.7	94.0	107
CDO.GP 15–16	314	.76	1.7	30.6	23	10.8	1.4	14.2	57.9	10.2	3.0	89.0	127
CDO.GP 18–19	309	.85	1.5	35.1	16	11.2	1.3	23.8	58.4	10.1	3.2	96.7	153
CDO.GP 21–22	305	.86	1.4	38.4	19	12.9	1.2	21.4	65.3	11.1	3.3	110	151
CDO.GP 24–25	234	.34	.79	23.2	20	10.1	.67	<3	55.7	11.0	3.0	87.0	82.0
Harrison Bayou core:													
CDO.HB 0–1	1,550	.16	1.1	39.4	13	10.7	1.7	<3	83.9	8.8	2.4	91.1	146
CDO.HB 2–3	1,740	.15	1.3	37.1	13	9.7	1.5	<3	99.2	8.0	2.3	83.7	131
CDO.HB 4–5	1,410	.15	1.6	41.2	15	10.7	1.7	<3	81.4	8.8	2.5	89.4	145
CDO.HB 7–8	1,240	.15	1.8	42.1	13	10.8	1.8	<3	86.6	9.0	2.5	93.8	150
CDO.HB 10–11	966	.15	3.3	44.2	14	11.4	1.8	<3	86.8	9.0	2.7	100	155
CDO.HB 13–14	781	.14	3.1	43.2	14	11.1	1.8	<3	86.8	8.9	2.6	98.8	149
CDO.HB 16–17	573	.13	2.6	40.8	13	11.0	1.7	<3	96.6	8.6	2.5	97.4	143
CDO.HB 19–20	467	.12	1.5	44.1	17	13.2	1.6	<3	93.2	10.2	2.8	117	147
Mid-lake core:													
CDO.MD 0–1	2,200	.12	1.6	22.6	13	8.6	1.3	<3	75.5	9.2	2.6	80.8	120
CDO.MD 1.5–2	1,500	.12	1.3	24.0	15	8.9	1.1	<3	68.0	9.5	2.7	83.3	127
CDO.MD 3–3.5	1,160	.12	1.1	23.7	13	8.6	1.3	<3	63.8	9.2	2.6	80.4	124
CDO.MD 4.5–5	1,020	.12	1.0	23.8	14	8.7	1.2	<3	65.5	9.5	2.7	81.5	124
CDO.MD 6–6.5	954	.12	1.2	26.2	15	9.1	1.1	<3	69.1	9.8	2.8	86.4	131
CDO.MD 7.5–8	788	.11	1.2	26.9	17	9.1	1.5	<3	70.6	10.2	2.9	83.9	144
CDO.MD 9–9.5	608	.11	.92	22.8	15	8.6	1.0	<3	70.6	9.8	2.8	80.5	109
CDO.MD 11–12	561	.08	1.2	27.0	24	11.4	1.3	<3	80.2	11.9	3.5	101	117
Carter Lake core:													
CDO.CR 0–0.5	1,350	.19	3.0	39.8	14	10.1	2.0	na	56.6	9.2	2.5	95.0	235
CDO.CR 1–1.5	1,790	.18	3.1	40.3	13	9.8	2.2	na	58.4	8.6	2.4	93.8	236
CDO.CR 1.5–2	1,440	.20	3.0	42.2	12	10.2	2.2	na	56.3	8.5	2.5	98.5	254
CDO.CR 3–3.5	2,090	.19	2.9	40.8	12	10.0	2.3	na	56.3	8.5	2.5	95.5	244
CDO.CR 4–4.5	1,570	.20	3.0	40.9	13	9.6	2.5	na	61.1	8.8	2.6	94.3	240
CDO.CR 4.5–5	1,660	.20	2.9	41.3	12	10.3	2.3	na	57.8	8.7	2.5	98.7	250
CDO.CR 7–8	1,390	.20	3.0	43.6	14	11.0	2.1	na	56.1	9.1	2.6	105	266
CDO.CR 9–10	1,570	.20	3.1	43.9	13	10.8	2.4	na	55.2	9.1	2.6	105	264
CDO.CR 10–11	2,220	.21	3.3	44.6	13	10.2	2.5	na	55.8	9.2	2.7	102	264
CDO.CR 13–14	1,900	.21	3.2	45.0	14	11.1	2.4	na	61.4	9.5	2.7	106	293
CDO.CR 15–16	1,390	.20	2.8	45.8	14	10.9	2.2	na	56.5	9.0	2.5	106	313
CDO.CR 16–17	1,440	.21	2.5	47.2	14	11.1	2.3	na	56.3	9.2	2.6	107	328
CDO.CR 19–20	1,400	.21	1.8	46.1	15	11.4	1.8	na	57.0	10.1	2.7	110	299
CDO.CR 22–23	1,570	.20	1.7	44.2	18	12.4	1.8	na	61.9	10.5	2.7	117	272

Appendix 2.5. Analytical results for petroleum biomarkers

[In micrograms per kilogram dry sediment; ID, identifier; E, estimated; <, less than]

USGS site ID	Sample ID	Nonane (C9)	Decane (C10)	Undecane (C11)	Dodecane (C12)	Tridecane (C13)	Tetradecane (C14)	Pentadecane (C15)	Hexadecane (C16)	Heptacecane (C17)	Pristane
Mid-lake core:											
324102094041400	CDO.MD 0–1	E17.1	E46.2	E101	E17.3	E15.4	E31.8	E55	139	E864	11.6
324102094041400	CDO.MD 1.5–2	E3.2	E19.4	E44.8	E18.8	E8.0	E16.3	E38.7	88.9	1,880	15.5
324102094041400	CDO.MD 3–3.5	<40	E14.2	E30.7	E11.4	E5.8	E11.6	E33.3	49.5	1,310	14.2
324102094041400	CDO.MD 4.5–5	E6.1	E11.7	E29.0	E7.6	E6.0	E10.6	E16.9	41.5	632	9.3
324102094041400	CDO.MD 6–6.5	E5.3	E9.0	E22.8	E5.8	E5.0	E11.0	E14.8	E32.4	292	6.2
324102094041400	CDO.MD 7.5–8	<40	E9.1	E24.0	E8.1	E4.8	E7.2	E10.2	48.1	190	5.9
324102094041400	CDO.MD 9–9.5	<40	E11.1	E26.7	E8.5	E5.7	E9.0	E14.8	53.9	402	8.8
324102094041400	CDO.MD 11–12	E3.4	E9.5	E24.5	E3.7	E3.3	E7.1	E7.2	E23.2	73.6	3.2

Sample ID	Octadecane (C18)	Phytane	Nonadecane (C19)	Eicosane (C20)	Heneicosane (C21)	Docosane (C22)	Tricosane (C23)	Tetracosane (C24)	Pentacosane (C25)	5B(H) Cholane	Hexacosane (C26)	n-Heptacosane (C27)
Mid-lake core—Continued												
CDO.MD 0–1	E64	115	188	172	320	E206	E744	E309	E752	<135	E318	E1,140
CDO.MD 1.5–2	E70.0	139	190	140	295	E150	E810	E205	E957	<80	E198	E1,220
CDO.MD 3–3.5	49.8	133	198	133	226	E107	E522	E128	E570	<40	E147	E717
CDO.MD 4.5–5	E38.8	100	208	130	203	E92.3	E474	E144	E474	<40	E133	E541
CDO.MD 6–6.5	E26.4	53.5	206	119	187	E91.4	E472	E162	E578	<40	E132	E781
CDO.MD 7.5–8	E24.0	40.8	166	120	152	E103	E400	E320	E541	<40	E139	E734
CDO.MD 9–9.5	E31.6	60.7	258	133	233	E112	E548	E137	E674	<40	E150	E895
CDO.MD 11–12	E12.8	14.8	97.1	58.6	99.6	E65.1	E278	E87.8	E459	<40	E116	E713

Appendix 2.5. Analytical results for petroleum biomarkers—Continued

Sample ID	Choles-tane series	Octaco-sane (C28)	Cholestane + Coprostane	4A-Methyl-sterane series	n-Nona-cosane (C29)	Hopane triter-panoid series	17A(H) 22,29,30 Trisnor-hopane	4A-C28 Methyl-sterane	n-Tria-contane (C30)	n-Hentria-contane (C31)	17B(H) 21A(H) 30 Norho-pane
Mid-lake core—Continued											
CDO.MD 0–1	E110	E344	E6.8	2,990	E1,380	4,210	E29.7	<135	E262	E1,180	<135
CDO.MD 1.5–2	144	E246	E5.4	2,670	E1,320	4,150	E28.7	<80	E164	E1,060	<80
CDO.MD 3–3.5	137	E137	E3.2	2,310	E619	3,110	E17.9	<40	E70.1	E476	<40
CDO.MD 4.5–5	73.6	E129	E2.5	1,880	E604	2,450	E18.9	<40	E76.7	E430	<40
CDO.MD 6–6.5	66.0	E163	<40	1,400	E740	1,860	E23.5	<40	E100	E562	<40
CDO.MD 7.5–8	85.0	E141	E5.0	1,720	E666	3,040	E26.5	<40	E99.9	E473	<45
CDO.MD 9–9.5	101	E174	E7.9	2,120	E816	3,200	E34.8	<40	E100	E582	<55
CDO.MD 11–12	E36.7	E127	E2.1	1,260	E609	2,000	<40	<40	E104	E523	<40

Sample ID	4A-C30 Methyl-sterane	17A(H) 21B Hopane	n-Dotria-contane (C32)	17B(H) 21A Hopane	n-Tritria-contane (C33)	17B(H) 21B(H) Hopane	n-Tetratria-contane (C34)	n-Pentatria-contane (C35)	n-Hexatria-contane (C36)	n-Octatria-contane (C38)	Nonatria-contane (C39)	n-Tetra-contane (C40)
Mid-lake core—Continued												
CDO.MD 0–1	<330	E60.7	E191	<135	E887	E92.4	E111	E528	E95.0	<150	E254	<140
CDO.MD 1.5–2	<160	E57.6	E113	<100	E721	84.1	E45.7	E518	E62.8	<140	E152	E149
CDO.MD 3–3.5	<100	43.7	<40	<80	E89.7	72.2	<40	E256	E33.2	<60	E102	E71.8
CDO.MD 4.5–5	<55	E43.0	E68.4	<80	E270	45.6	<40	E146	E31.7	E39.6	E46.7	E57.2
CDO.MD 6–6.5	<40	47.3	E53.3	<75	E327	64.8	E25.4	E187	E48.0	<75	E103	E87.9
CDO.MD 7.5–8	<50	62.0	E50.6	<80	E233	67.0	E24.4	E141	E32.4	<70	<80	<70
CDO.MD 9–9.5	<55	70.8	E57.7	<90	E404	82.6	E35.5	E220	E35.9	<90	E126	E98.7
CDO.MD 11–12	<40	E27.7	E58.4	<40	E260	E26.9	E32.0	E101	E37.6	<100	E84.8	E56.9

APPENDIX 3—
Quality-Assurance Data

Appendix 3.1. Quality-control samples for radionuclides

[In picocuries per gram unless otherwise specified; ID, identifier; STL, Severn Trent Laboratories, Inc.; na, not analyzed; lab dup, laboratory duplicate; RPD, relative percent difference; env dup, environmental duplicate; LCS, laboratory control sample]

Sample ID	STL lot number	Cesium-137	Radium-226	Lead-210
CDO.MD 12–13	J2G090162	0.200	2.08	na
CDO.MD 12–13 lab dup	J2G090162	.336	1.77	na
	RPD of radionuclide:	50.75	16.10	
CDO.HB 7–8	J2G090162	1.25	na	na
CDO.HB 7–8 lab dup	J2G090162	1.29	na	na
	RPD of radionuclide:	3.15		
CDO.CR 17–18	J2F260205	1.45	1.82	na
CDO.CR 17–18 lab dup	J2F260205	1.78	1.72	na
	RPD of radionuclide:	20.43	5.65	
CDO.GP 3–4	J2F260205	.860	na	na
CDO.GP 3–4 lab dup	J2F260205	.788	na	na
	RPD of radionuclide:	8.74		
CDO.HB 15–16 env dup	J2G110278	3.04	na	na
CDO.HB 15–16 env dup, lab dup	J2G110278	2.83	na	na
	RPD of radionuclide:	7.16		
CDO.GP 14–15	J2F260205	.964	na	na
CDO.GP 14–15 env dup	J2F260205	.994	na	na
	RPD of radionuclide:	3.06		
CDO.HB 15–16	J2G110278	2.36	na	na
CDO.HB 15–16 env dup	J2G110278	3.04	na	na
	RPD of radionuclide:	25.19		
Blank	J2G090162	-.0239	na	na
Percent recovered of LCS	J2G090162	98.40	na	na
Blank	J2G090162	-.0131	.149	-.00460
Percent recovered of LCS	J2G090162	100.47	na	102.77
Blank	J2F260205	-.0107	na	na
Percent recovered of LCS	J2F260205	103.36	na	na
Blank	J2F260205	-.0253	.0832	-.00443
Percent recovered of LCS	J2F260205	109.10	na	107.48
Blank	J2G110278	-.0118	na	na
Percent recovered of LCS	J2G110278	98.41	na	na

Appendix 3.2. Quality-control samples for organochlorine compounds

[In micrograms per kilogram except as noted; ID, identifier; <, less than; env dup, environmental duplicate; RPD, relative percent difference; lab dup, laboratory duplicate; CRM, certified reference material]

Sample ID	Group number	Lindane	Heptachlor	Aldrin	Heptachlor epoxide	Technical chlordane
CDO.CR 7–8	200219708	<7.0	<7.0	<7.0	<7.0	<70
CDO.CR 7–8 env dup	200219708	<6.0	<6.0	<6.0	<6.0	<60
Median RPD of sample = 23.3						
CDO.HB 4–5	200221211	<6.0	<6.0	<6.0	<6.0	<60
CDO.HB 4–5 env dup	200221211	<6.0	<6.0	<6.0	<6.0	<60
Median RPD of sample = 3.2						
CDO.HB 19–20	200221211	<2.5	<2.5	<2.5	<2.5	<25
CDO.HB 19–20 lab dup	200221211	<2.5	<2.5	<2.5	<2.5	<25
Median RPD of sample = 10.0						
Lk Austin Site DC	200222605	<1.0	<1.0	<1.0	<1.0	<10
Lk Austin Site DC lab dup	200222605	<1.0	<1.0	<1.0	<1.0	<10
Median RPD of sample = 23.0						
CDO.MD 11–12	200221106	<.5	<.5	<.5	<.5	<5
CDO.MD 11–12 lab dup	200221106	<.5	<.5	<.5	<.5	<5
Median RPD of sample = 20.2						
CDO.GP 21–22	200220603	<1.0	<1.0	<1.0	<1.0	<10
CDO.GP 21–22 lab dup	200220603	<1.0	<1.0	<1.0	<1.0	<10
Median RPD of sample = 10.1						
Lab blank	200219708	<.5	<.5	<.5	<.5	<5.0
Lab spike, percent recovered (control limits)	200219708	53 (15–112)	63 (25–109)	57 (15–133)	72 (15–155)	<5.0 (not spiked)
CRM 362 (acceptable range, true value)	200219708	78 (50–217, 143)	87 (56–196, 134)	119 (87–253, 186)	92 (78–185, 146)	<5.0 (not spiked)
Lab blank	200221211	<.5	<.5	<.5	<.5	<5.0
Lab spike, percent recovered (control limits)	200221211	65 (15–112)	59 (25–109)	51 (15–133)	65 (15–155)	<5.0 (not spiked)
CRM 362 (acceptable range, true value)	200221211	79 (50–217, 143)	85 (56–196, 134)	131 (87–253, 186)	94 (78–185, 146)	<5.0 (not spiked)
Lab blank	200222605	<.5	<.5	<.5	<.5	<5.0
Lab spike, percent recovered (control limits)	200222605	62 (15–112)	72 (25–109)	76 (15–133)	72 (15–155)	<5.0 (not spiked)
CRM 362 (acceptable range, true value)	200222605	58 (50–217, 143)	62 (56–196, 134)	87 (87–253, 186)	62 (78–185, 146)	<5.0 (not spiked)
Lab blank	200221106	<.5	<.5	<.5	<.5	<5.0
Lab spike, percent recovered (control limits)	200221106	22 (15–112)	30 (25–109)	30 (15–133)	30 (15–155)	<5.0 (not spiked)
CRM 362 (acceptable range, true value)	200221106	83 (50–217, 143)	78 (56–196, 134)	121 (87–253, 186)	102 (78–185, 146)	<5.0 (not spiked)
Lab blank	200220603	<.5	<.5	<.5	<.5	<5.0
Lab spike, percent recovered (control limits)	200220603	57 (15–112)	68 (25–109)	69 (15–133)	63 (15–155)	<5.0 (not spiked)
CRM 362 (acceptable range, true value)	200220603	54 (50–217, 143)	49 (56–196, 134)	66 (87–253, 186)	56 (78–185, 146)	<5.0 (not spiked)

Appendix 3.2. Quality-control samples for organochlorine compounds—Continued

Sample ID	Endosulfan I	Dieldrin	*p,p'*-DDE	Endrin	*p,p'*-DDD	*p,p'*-DDT
CDO.CR 7–8	<7.0	<7.0	E2.4	<7.0	<7.0	<7.0
CDO.CR 7–8 env dup	<6.0	<6.0	E1.9	<6.0	<6.0	<6.0
Median RPD of sample = 23.3			**RPD = 23.3**			
CDO.HB 4–5	<6.0	<6.0	E3.2	<6.0	<6.0	<6.0
CDO.HB 4–5 env dup	<6.0	<6.0	E3.1	<6.0	<6.0	<6.0
Median RPD of sample = 3.2			**RPD = 3.2**			
CDO.HB 19–20	<2.5	<2.5	7.5	<2.5	E.83	<2.5
CDO.HB 19–20 lab dup	<2.5	<2.5	7.6	<2.5	E1.0	<2.5
Median RPD of sample = 10.0			**RPD = 1.3**		**RPD = 18.6**	
Lk Austin Site DC	<1.0	<1.0	E.27	<1.0	<1.0	<1.0
Lk Austin Site DC lab dup	<1.0	<1.0	E.34	<1.0	<1.0	<1.0
Median RPD of sample = 23.0			**RPD = 23.0**			
CDO.MD 11–12	<.5	<.5	2.3	<.5	.7	<.5
CDO.MD 11–12 lab dup	<.5	<.5	2.1	<.5	.5	<.5
Median RPD of sample = 20.2			**RPD = 9.1**		**RPD = 31.3**	
CDO.GP 21–22	<1.0	<1.0	4.0	<1.0	<1.0	<1.0
CDO.GP 21–22 lab dup	<1.0	<1.0	4.2	<1.0	<1.0	<1.0
Median RPD of sample = 10.1			**RPD = 4.9**			
Lab blank	<.5	<.5	<.5	<.5	<.5	<.5
Lab spike, percent recovered (control limits)	65 (25–94)	84 (15–150)	78 (34–140)	98 (33–133)	74 (22–158)	57 (15–150)
CRM 362 (acceptable range, true value)	102 (71–229, 188)	142 (93–265, 191)	104 (74–194, 146)	113 (93–219, 151)	178 (120–401, 271)	160 (94–261, 212)
Lab blank	<.5	<.5	<.5	<.5	<.5	<.5
Lab spike, percent recovered (control limits)	58 (25–94)	62 (15–150)	82 (34–140)	72 (33–133)	67 (22–158)	60 (15–150)
CRM 362 (acceptable range, true value)	91 (71–229, 188)	135 (93–265, 191)	112 (74–194, 146)	118 (93–219, 151)	183 (120–401, 271)	141 (94–261, 212)
Lab blank	<.5	<.5	<.5	<.5	<.5	<.5
Lab spike, percent recovered (control limits)	63 (25–94)	71 (15–150)	89 (34–140)	82 (33–133)	71 (22–158)	70 (15–150)
CRM 362 (acceptable range, true value)	62 (71–229, 188)	84 (93–265, 191)	77 (74–194, 146)	84 (93–219, 151)	124 (120–401, 271)	95 (94–261, 212)
Lab blank	<.5	<.5	<.5	<.5	<.5	<.5
Lab spike, percent recovered (control limits)	28 (25–94)	32 (15–150)	34 (34–140)	36 (33–133)	31 (22–158)	32 (15–150)
CRM 362 (acceptable range, true value)	98 (71–229, 188)	141 (93–265, 191)	99 (74–194, 146)	124 (93–219, 151)	183 (120–401, 271)	153 (94–261, 212)
Lab blank	<.5	<.5	<.5	<.5	<.5	<.5
Lab spike, percent recovered (control limits)	53 (25–94)	70 (15–150)	132 (34–140)	83 (33–133)	85 (22–158)	68 (15–150)
CRM 362 (acceptable range, true value)	53 (71–229, 188)	84 (93–265, 191)	63 (74–194, 146)	90 (93–219, 151)	132 (120–401, 271)	101 (94–261, 212)

Appendix 3.2. Quality-control samples for organochlorine compounds—Continued

Sample ID	Methoxy-chlor	Mirex	Toxaphene	PCB Aroclor 1242	PCB Aroclor 1254	PCB Aroclor 1260
CDO.CR 7–8	<28	<7.0	<700	<70	<70	<70
CDO.CR 7–8 env dup	<24	<6.0	<600	<60	<60	<60
Median RPD of sample = 23.3						
CDO.HB 4–5	<24	<6.0	<600	<60	<60	<60
CDO.HB 4–5 env dup	<24	<6.0	<600	<60	<60	<60
Median RPD of sample = 3.2						
CDO.HB 19–20	<10	<2.5	<250	<25	<25	<25
CDO.HB 19–20 lab dup	<10	<2.5	<250	<25	<25	<25
Median RPD of sample = 10.0						
Lk Austin Site DC	<4.0	<1.0	<100	<10	<10	<10
Lk Austin Site DC lab dup	<4.0	<1.0	<100	<10	<10	<10
Median RPD of sample = 23.0						
CDO.MD 11–12	<2.0	<.5	<50	<5.0	<5.0	<5.0
CDO.MD 11–12 lab dup	<2.0	<.5	<50	<5.0	<5.0	<5.0
Median RPD of sample = 20.2						
CDO.GP 21–22	<4.0	<1.0	<100	E5.2	E14	E16
CDO.GP 21–22 lab dup	<4.0	<1.0	<100	E5.2	E12	20.0
Median RPD of sample = 10.1				**RPD = 0.0**	**RPD = 15.4**	**RPD = 22.2**
Lab blank	<2.0	<.5	<50	<5.0	<5.0	<5.0
Lab spike, percent recovered (control limits)	66 (15–150)	72 (32–120)	<50 (not spiked)	60 (17–145)	80 (53–150)	80 (33–150)
CRM 362 (acceptable range, true value)	168 (144–622, 426)	<.5 (not spiked)	<50 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)
Lab blank	<2.0	<.5	<50	<5.0	<5.0	<5.0
Lab spike, percent recovered (control limits)	84 (15–150)	64 (32–120)	<50 (not spiked)	70 (17–145)	82 (53–150)	85 (33–150)
CRM 362 (acceptable range, true value)	289 (144–622, 426)	<.5 (not spiked)	<50 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)
Lab blank	<2.0	<.5	<50	<5.0	<5.0	<5.0
Lab spike, percent recovered (control limits)	90 (15–150)	74 (32–120)	<50 (not spiked)	90 (17–145)	116 (53–150)	118 (33–150)
CRM 362 (acceptable range, true value)	216 (144–622, 426)	<.5 (not spiked)	<50 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)
Lab blank	<2.0	<.5	<50	<5.0	<5.0	<5.0
Lab spike, percent recovered (control limits)	39 (15–150)	E31 (32–120)	<50 (not spiked)	32 (17–145)	E42 (53–150)	E40 (33–150)
CRM 362 (acceptable range, true value)	259 (144–622, 426)	<.5 (not spiked)	<50 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)
Lab blank	<2.0	<.5	<50	<5.0	<5.0	<5.0
Lab spike, percent recovered (control limits)	46 (15–150)	64 (32–120)	<50 (not spiked)	33 (17–145)	62 (53–150)	65 (33–150)
CRM 362 (acceptable range, true value)	222 (144–622, 426)	<.5 (not spiked)	<50 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)	<5.0 (not spiked)

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons

[In micrograms per kilogram, except as noted; ID, identifier; E, estimated; <, less than; env dup, environmental duplicate; RPD, relative percent difference; lab dup, laboratory duplicate; CRM, certified reference material]

Sample ID	Group number	Naphthalene	C1-128 isomers, methylated Naphthalenes	2-ethyl-Naphthalene	2,6-Dimethyl-naphthalene	1,6-Dimethyl-naphthalene
CDO.HB 4–5	8022R02177	E18.6	<690	<65	2,100	348
CDO.HB 4–5 env dup	8022R02177	<65	<750	<65	1,740	310
Median RPD of sample = 6.7	**RPD of compound =**				**18.8**	**11.6**
CDO.HB 19–20	8022R02177	<30	<220	<30	438	75.1
CDO.HB 19–20 lab dup	8022R02177	E6.4	<160	<30	334	65.2
Median RPD of sample = 4.2	**RPD of compound =**				**26.9**	**14.1**
CDO.MD 11–12	8022R02171	E3.0	<20	<10	21.8	E3.1
CDO.MD 11–12 lab dup	8022R02171	E1.2	<30	<10	16.3	E4.3
Median RPD of sample = 9.1	**RPD of compound =**	**85.7**			**28.9**	**32.4**
CDO.CR 7–8	8022R02165	<60	<300	<60	1,150	189
CDO.CR 7–8 env dup	8022R02165	<60	<200	<60	1,010	148
Median RPD of sample = 18.8	**RPD of compound =**				**13.0**	**24.3**
Lab blank	8022R02165	<5	<5	<5	<5	<5
Lab spike, percent recovered	8022R02165	62.02		61.94	60.94	63.03
CRM 354, percent recovered (acceptable range)	8022R02165	3.85 (15–95)				
Lab blank	8022R02169	<5	<5	<5	E.46	<5
Lab spike, percent recovered	8022R02169	66.69		67.61	66.10	65.22
CRM 362, percent recovered (acceptable range)	8022R02169	38.58 (0–117)				
Lab blank	8022R02171	E.23	<5	<5	<5	<5
Lab spike, percent recovered	8022R02171	E41.42		56.02	57.80	56.02
CRM 362, percent recovered (acceptable range)	8022R02171	32.10 (0–117)				
Lab blank	8022R02177	E.15	<5	<5	E.44	<5
Lab spike, percent recovered	8022R02177	65.46		65.09	66.06	67.48
CRM 362, percent recovered (acceptable range)	8022R02177	44.67 (0–117)				

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	C2-128 isomers, C2-alkylated Naphthalenes	Acenaphthylene	1,2-Dimethylnaphthalene	Acenaphthene	C3-128 isomers, C3-alkylated Naphthalenes	2,3,6-Trimethylnaphthalene	9H-Fluorene	C4-128 isomers, C4-alkylated Naphthalenes
CDO.HB 4–5	2,380	<65	<65	<65	514	E13.5	E10.7	<65
CDO.HB 4–5 env dup	2,190	<65	<65	<65	461	<65	E9.9	<65
Median RPD of sample = 6.7	**8.3**				**10.9**		**7.8**	
CDO.HB 19–20	591	E6.1	E11.8	E3.3	<100	E4.4	E4.6	<30
CDO.HB 19–20 lab dup	516	E6.4	E11.8	E2.9	<125	E4.5	E4.6	<30
Median RPD of sample = 4.2	**13.6**	**4.8**	**0**	**12.9**		**2.2**	**0**	
CDO.MD 11–12	31.0	<10	<10	<10	<50	<10	<10	<10
CDO.MD 11–12 lab dup	25.8	E1.6	<10	E.76	<15	<10	E.97	<10
Median RPD of sample = 9.1	**18.3**							
CDO.CR 7–8	1,430	<60	<60	<60	<300	<60	<60	<60
CDO.CR 7–8 env dup	1,310	<60	<60	E5.6	<70	<60	E7.8	<60
Median RPD of sample = 18.8	**8.8**							
Lab blank	<5	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered		59.00	62.07	62.97		60.44	62.46	
CRM 354, percent recovered (acceptable range)								
Lab blank	<5	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered		65.86	68.05	68.41		70.37	71.93	
CRM 362, percent recovered (acceptable range)		29.32 (28–105)		50.48 (36–113)				
Lab blank	<5	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered		53.77	58.63	58.65		64.72	65.84	
CRM 362, percent recovered (acceptable range)		33.43 (28–105)		55.87 (36–113)				
Lab blank	<5	<5	<5	E.18	<5	<5	<5	<5
Lab spike, percent recovered		63.11	65.24	66.83		66.80	67.77	
CRM 362, percent recovered (acceptable range)		41.99 (28–105)		70.59 (36–113)				

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	1-methyl-9H-Fluorene	Phenanthrene	Anthracene	C5-128 isomers, C5-alkylated Naphthalenes	2-Methyl-anthracene	4,5-Methylene-phenanthrene	C1-178 isomers, methylated Phenanthrene/ anthracenes
CDO.HB 4–5	<65	E20.7	E22.1	<65	<65	<65	<400
CDO.HB 4–5 env dup	<65	E17.8	E21.3	<65	<65	E17.1	<375
Median RPD of sample = 6.7		**15.1**	**3.7**				
CDO.HB 19–20	E10.9	E16.1	E12.4	<30	<30	E13.2	<65
CDO.HB 19–20 lab dup	E10.6	E15.9	E13.0	<30	<30	E12.9	<65
Median RPD of sample = 4.2	**2.8**	**1.3**	**4.7**			**2.3**	
CDO.MD 11–12	E2.5	E2.3	E2.3	<10	<10	E1.8	E7.3
CDO.MD 11–12 lab dup	E2.3	E2.1	E2.2	<10	E1.9	E1.8	E5.3
Median RPD of sample = 9.1	**8.3**	**9.1**	**4.4**			**0**	**31.7**
CDO.CR 7–8	<60	E17.6	E19.3	<60	<60	<60	<70
CDO.CR 7–8 env dup	<60	E13.9	E12.7	<60	<60	<60	<100
Median RPD of sample = 18.8		**23.5**	**41.3**				
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	64.23	67.17	63.00		63.95	67.09	
CRM 354, percent recovered (acceptable range)		75.89 (39–94)	91.51 (18–95)				
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	73.47	70.98	68.97		69.43	71.46	
CRM 362, percent recovered (acceptable range)		59.21 (44–124)	45.99 (38–105)				
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	73.29	72.67	70.38		72.44	76.33	
CRM 362, percent recovered (acceptable range)		80.52 (44–124)	59.86 (38–105)				
Lab blank	<5	E.24	E.54	<5	<5	<5	<5
Lab spike, percent recovered	69.73	70.58	67.94		67.69	69.52	
CRM 362, percent recovered (acceptable range)		83.90 (44–124)	65.01 (38–105)				

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	1-Methyl-phenan-threne	C2-178 isomers, C2-alkylated Phenanthrene/ anthracenes	Fluoran-thene	Pyrene	C3-178 isomers, C3-alkylated Phenanthrene/ anthracenes	C4-178 isomers, C4-alkylated Phenanthrene/ anthracenes	1-Methyl-pyrene
CDO.HB 4–5	E54.2	<65	E41.4	E36.8	<65	<65	<65
CDO.HB 4–5 env dup	E47.7	<75	E38.8	E34.4	<65	<65	<65
Median RPD of sample = 6.7	**12.8**		**6.5**	**6.7**			
CDO.HB 19–20	E6.7	<30	31.2	31.8	<30	<30	E5.5
CDO.HB 19–20 lab dup	E8.0	<30	31.2	32.1	E16.4	<30	E5.5
Median RPD of sample = 4.2	**17.7**		**0**	**0.9**			**0**
CDO.MD 11–12	E1.6	E8.2	E6.1	12.6	E7.1	<10	<10
CDO.MD 11–12 lab dup	<10	E8.1	E6.0	11.8	<10	<10	<10
Median RPD of sample = 9.1		**1.2**	**1.7**	**6.6**			
CDO.CR 7–8	E15.7	<60	E28.1	E32.6	<60	<60	<60
CDO.CR 7–8 env dup	E12.9	<60	E25.5	E29.0	<60	<60	<60
Median RPD of sample = 18.8	**19.6**		**9.7**	**11.7**			
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	70.26		74.28	74.30			76.71
CRM 354, percent recovered (acceptable range)			51.69 (33–90)	110.20 (32–90)			
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	72.05		76.23	76.51			80.19
CRM 362, percent recovered (acceptable range)			67.17 (43–125)	65.57 (32–139)			
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	78.09		86.97	87.47			88.93
CRM 362, percent recovered (acceptable range)			101.14 (43–125)	98.50 (32–139)			
Lab blank	<5	<5	E.61	E.62	<5	<5	<5
Lab spike, percent recovered	70.46		73.63	76.05			74.05
CRM 362, percent recovered (acceptable range)			92.75 (43–125)	89.41 (32–139)			

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	C1-202 isomers, methylated Fluoranthene/ pyrenes	C2-202 isomers, C2-alkylated Fluoranthene/ pyrenes	C5-178 isomers, C5-alkylated Phenanthrene/ anthracenes	Benz(a)-anthra-cene	Chrysene	C3-202 isomers, C3-alkylated Fluoranthene/ pyrenes	C1-228 isomers, methylated Benzo(a)-anthracene/ chrysenes
CDO.HB 4–5	<65	<150	<65	E18.9	E21.6	<65	<65
CDO.HB 4–5 env dup	<65	<100	<65	E17.9	E20.3	<65	<65
Median RPD of sample = 6.7				**5.4**	**6.2**		
CDO.HB 19–20	<40	<30	<30	E13.3	E18.8	<30	<30
CDO.HB 19–20 lab dup	<40	<30	<30	E13.8	E19.6	<30	<30
Median RPD of sample = 4.2				**3.7**	**4.2**		
CDO.MD 11–12	<10	<10	<10	E3.2	E5.3	<10	<10
CDO.MD 11–12 lab dup	<10	<10	<10	<10	E4.7	<10	<10
Median RPD of sample = 9.1					**12.0**		
CDO.CR 7–8	<60	<60	<60	<60	E14.2	<400	<60
CDO.CR 7–8 env dup	<60	<60	<60	<60	E14.3	<300	<60
Median RPD of sample = 18.8					**.7**		
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				80.71	78.60		
CRM 354, percent recovered (acceptable range)					210.60 (42–89)		
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				74.41	69.48		
CRM 362, percent recovered (acceptable range)					64.24 (42–126)		
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				90.00	84.20		
CRM 362, percent recovered (acceptable range)					97.21 (42–126)		
Lab blank	<5	<5	<5	<5	E.28	<5	<5
Lab spike, percent recovered				80.14	77.29		
CRM 362, percent recovered (acceptable range)					84.19 (42–126)		

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	C4-202 isomers, C4-alkylated Fluoranthene/ pyrenes	C5-202 isomers, C5-alkylated Fluoranthene/ pyrenes	C2-228 isomers, C2-alkylated Benzo(a)-anthracene/ chrysenes	Benzo(b)-fluoran-thene	Benzo(k)-fluoran-thene	Benzo(e)-pyrene	Benzo(a)-pyrene
CDO.HB 4–5	<65	<65	<65	E38.5	E18.0	E18.3	E20.1
CDO.HB 4–5 env dup	<65	<65	<65	E34.1	E19.9	E18.1	E19.6
Median RPD of sample = 6.7				**12.1**	**10.0**	**1.1**	**2.5**
CDO.HB 19–20	<30	<30	<30	32.0	E16.5	E16.3	E15.2
CDO.HB 19–20 lab dup	<30	<30	<30	33.1	E18.1	E16.1	E15.0
Median RPD of sample = 4.2				**3.4**	**9.2**	**1.2**	**1.3**
CDO.MD 11–12	<10	<10	<10	E7.2	E6.1	E4.9	E3.9
CDO.MD 11–12 lab dup	<10	<10	<10	E8.2	E3.8	E5.0	E3.8
Median RPD of sample = 9.1				**13.0**	**46.5**	**2.0**	**2.6**
CDO.CR 7–8	<60	<60	<60	E33.8	E19.3	E16.3	E20.6
CDO.CR 7–8 env dup	<60	<60	<60	E27.3	E14.4	E13.5	E18.3
Median RPD of sample = 18.8				**21.3**	**29.1**	**18.8**	**11.8**
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				74.72	79.54	59.28	71.93
CRM 354, percent recovered (acceptable range)					69.88 (42–89)		
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				67.29	77.45	53.99	69.54
CRM 362, percent recovered (acceptable range)				66.75 (37–128)			40.23 (39–104)
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				82.42	94.65	64.58	78.11
CRM 362, percent recovered (acceptable range)				96.93 (37–128)			57.96 (39–104)
Lab blank	<5	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered				71.96	84.07	73.93	75.01
CRM 362, percent recovered (acceptable range)				85.23 (37–128)			56.67 (39–104)

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	C1-252 isomers, C1-methylated Benzopyrene/ perylenes	C3-228 isomers, C3-Benzo(a)- anthracene/ chrysenes	C2-252 isomers, C2-alkylated Benzopyrene/ perylenes	C4-228 isomers, C4-Benzo(a)- anthracene/ chrysenes	Benzo- (g,h,i)- perylene	Indeno- (1,2,3-c,d)- pyrene
CDO.HB 4–5	<65	<65	<65	<65	<65	E39.1
CDO.HB 4–5 env dup	<65	<65	<65	<65	E26.4	E37.4
Median RPD of sample = 6.7						**4.4**
CDO.HB 19–20	<30	<30	<30	<30	E20.4	E28.2
CDO.HB 19–20 lab dup	E7.2	<30	<30	<30	E18.4	E24.8
Median RPD of sample = 4.2					**10.3**	**12.8**
CDO.MD 11–12	36.3	<10	<10	<10	E6.9	E7.9
CDO.MD 11–12 lab dup	61.4	<10	<10	<10	10.2	E7.3
Median RPD of sample = 9.1	**51.4**				**38.6**	**7.9**
CDO.CR 7–8	71.1	<60	<60	<60	<60	E37.4
CDO.CR 7–8 env dup	E53.3	<60	<60	<60	E24.6	E31.3
Median RPD of sample = 18.8	**28.6**					**17.8**
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered					71.61	73.25
CRM 354, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered					70.14	79.70
CRM 362, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered					86.59	95.82
CRM 362, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered					82.99	86.97
CRM 362, percent recovered (acceptable range)						

Appendix 3.3. Quality-control samples for polycyclic aromatic hydrocarbons—Continued

Sample ID	Dibenzo(a,h)-anthracene	C3-252 isomers, C3-alkylated Benzopyrene/perylenes	C4-252 isomers, C4-alkylated Benzopyrene/perylenes	C5-228 isomers, C5-Benzo(a)-anthracene/chrysenes	C5-252 isomers, C5-alkylated Benzopyrene/perylenes	Coron-ene
CDO.HB 4–5	<65	<65	<65	<65	<65	E15.6
CDO.HB 4–5 env dup	E23.6	<65	<65	<65	<65	E15.0
Median RPD of sample = 6.7						**3.9**
CDO.HB 19–20	E12.1	<30	<30	<30	<30	E14.1
CDO.HB 19–20 lab dup	E10.6	<30	<30	<30	<30	E10.0
Median RPD of sample = 4.2	**13.2**					**34.0**
CDO.MD 11–12	E3.1	<10	<10	<10	<10	E3.3
CDO.MD 11–12 lab dup	E3.0	<10	<10	<10	<10	<10
Median RPD of sample = 9.1	**3.3**					
CDO.CR 7–8	<60	<60	<60	<60	<60	<60
CDO.CR 7–8 env dup	E18.1	<60	<60	<60	<60	E19.2
Median RPD of sample = 18.8						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	73.36					E42.03
CRM 354, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	90.49					E45.15
CRM 362, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	98.69					E79.66
CRM 362, percent recovered (acceptable range)						
Lab blank	<5	<5	<5	<5	<5	<5
Lab spike, percent recovered	96.54					E96.58
CRM 362, percent recovered (acceptable range)						

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements

[In micrograms per gram, except as noted; ID, identifier; lab dup, laboratory duplicate; lab tri, laboratory triplicate; RPD, relative percent difference; na, not analyzed; --, not calculated; <, less than; HNO_3 blank, dionized water with nitric acid preservative; MAG–1, USGS Marine Sediment; NIST 2704, National Institute of Standards and Technology Buffalo River Sediment; SCO–1, USGS Cody Shale; GSD–8, Chinese Quangxi Province Stream Sediment; MESS–1, National Research Council Canada Marine Estuarine Sediment; SRM, standard reference material; ?, uncertain]

Sample ID	Group number	Total carbon (percent)	Inorganic carbon, as CO_3 (percent)	Organic carbon (percent)	Aluminum	Calcium	Iron	Magnesium	Potassium	Phosphorus
CDO.GP 9–10	3958	12.8	0.04	12.8	77,100	3,070	26,000	2,740	6,740	1,050
CDO.GP 9–10 lab dup	3958	12.5	.04	12.5	76,100	3,030	26,000	2,800	6,720	1,050
Median RPD of sample = 1.2	**RPD of element =**	**2.37**	**0**	**2.37**	**1.31**	**1.31**	**0**	**2.17**	**.30**	**0**
CDO.GP 15–16	3958	9.03	.03	9.00	69,000	2,410	25,000	3,000	7,130	698
CDO.GP 15–16 lab tri 1	3958	na	na	na	71,900	2,490	25,500	2,930	7,190	754
	RPD of element =	--	--	--	**4.12**	**3.27**	**1.98**	**2.36**	**.84**	**7.71**
CDO.GP 15–16	3958	9.03	.03	9.00	69,000	2,410	25,000	3,000	7,130	698
CDO.GP 15–16 lab tri 2	3958	na	na	na	71,000	2,420	25,300	2,790	7,120	744
	RPD of element =	--	--	--	**2.86**	**.41**	**1.19**	**7.25**	**.14**	**6.38**
CDO.GP 15–16 lab tri 1	3958	na	na	na	71,900	2,490	25,500	2,930	7,190	754
CDO.GP 15–16 lab tri 2	3958	na	na	na	71,000	2,420	25,300	2,790	7,120	744
Median RPD of sample = 3.0	**RPD of element =**	--	--	--	**1.26**	**2.85**	**.79**	**4.90**	**.98**	**1.34**
CDO.HB 4–5	3958	11.6	.07	11.5	69,900	3,360	39,800	3,810	8,200	1,340
CDO.HB 4–5 lab dup	3958	11.2	.07	11.1	70,500	3,360	39,900	3,820	8,190	1,360
Median RPD of sample = 0.9	**RPD of element =**	**3.51**	**0**	**3.54**	**.85**	**0**	**.25**	**.26**	**.12**	**1.48**
CDO.CR 0–0.5	3959	1.2	10.6	10.5	67,900	3,180	46,000	2,960	6,260	1,600
CDO.CR 0–0.5 lab tri 1	3959	1.1	na	na	75,600	3,330	49,000	2,810	6,700	1,700
	RPD of element =	**8.70**	--	--	**10.73**	**4.61**	**6.32**	**5.20**	**6.79**	**6.06**
CDO.CR 0–0.5	3959	1.2	10.6	10.5	67,900	3,180	46,000	2,960	6,260	1,600
CDO.CR 0–0.5 lab tri 2	3959	1.3	na	na	74,700	3,400	49,000	2,840	6,770	1,700
	RPD of element =	**8.00**	--	--	**9.54**	**6.69**	**6.32**	**4.14**	**7.83**	**6.06**
CDO.CR 0–0.5 lab tri 1	3959	1.1	na	na	75,600	3,330	49,000	2,810	6,700	1,700
CDO.CR 0–0.5 lab tri 2	3959	1.3	na	na	74,700	3,400	49,000	2,840	6,770	1,700
Median RPD of sample = 2.8	**RPD of element =**	16.67	--	--	**1.20**	**2.08**	**0**	**1.06**	**1.04**	**0**
CDO.CR 13–14	3959	1.2	9.46	9.37	78,000	3,270	56,000	2,760	6,690	3,000
CDO.CR 13–14 lab dup	3959	1.1	10.0	9.91	72,400	4,020	55,000	2,720	6,430	3,700
Median RPD of sample = 4.7	**RPD of element =**	**8.70**	**5.55**	**5.60**	**7.45**	**20.58**	**1.80**	**1.46**	**3.96**	**20.90**
CDO.CR 19–20	3959	1.2	8.53	8.44	83,800	2,570	66,000	3,030	7,230	1,600
CDO.CR 19–20 lab dup	3959	1.2	8.45	8.37	83,600	2,560	65,000	3,030	7,240	1,600
Median RPD of sample = 0.8	**RPD of element =**	**0**	**.94**	**.83**	**.24**	**.39**	**1.53**	**0**	**.14**	**0**

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Group number	Total carbon (percent)	Inorganic carbon, as CO_3 (percent)	Organic carbon (percent)	Aluminum	Calcium	Iron	Magnesium	Potassium	Phosphorus
HNO_3 blank	3959	na	na	na	<8	<20	<50	<0.3	<20	<8
HNO_3 blank	3959	na	na	na	<8	<20	<50	<.3	<20	<8
HNO_3 blank	3959	na	na	na	<8	<20	<50	<.3	<20	<8
HNO_3 blank	3959	na	na	na	<8	<20	<50	<.3	<20	<8
MAG–1 found	3959	na	na	na	89,600	10,600	50,000	19,200	30,900	760
MAG–1 true (Potts and others, 1992)		na	na	na	86,660	9,790	47,600	18,090	29,500	711
RPD of element		--	--	--	**3.34**	**7.95**	**4.92**	**5.95**	**4.64**	**6.66**
NIST 2704 found	3959	na	na	na	62,800	27,200	42,000	12,600	20,300	1,000
NIST 2704 true (Potts and others, 1992)		na	na	na	61,090	26,000	41,100	12,000	20,000	1,000
RPD of element		--	--	--	**2.76**	**4.51**	**2.17**	**4.88**	**1.49**	**0**
SCO–1 found	3959	na	na	na	76,800	19,600	37,000	17,500	23,200	960
SCO–1 true (Potts and others, 1992)		na	na	na	72,370	18,700	35,900	16,400	23,000	899
RPD of element		--	--	--	**5.94**	**4.70**	**3.02**	**6.49**	**.87**	**6.56**
GSD–8 found	3959	na	na	na	42,200	1,570	16,000	1,530	24,300	120
GSD–8 true (Potts and others, 1992)		na	na	na	40,800	1,790	15,380	1,510	23,500	130
RPD of element		--	--	--	**3.37**	**13.10**	**3.95**	**1.32**	**3.35**	**8.00**
MESS–1 found	3959	na	na	na	57,100	5,010	30,000	8,550	18,200	690
MESS–1 true (Potts and others, 1992)		na	na	na	58,391	4,818	30,490	8,685	18,589	637
RPD of element		--	--	--	**2.24**	**3.91**	**1.62**	**1.57**	**2.12**	**7.94**
MAG–1 found	3959	na	na	na	90,300	10,500	50,000	18,600	31,600	730
MAG–1 true (Potts and others, 1992)		na	na	na	86,660	9,790	47,600	18,090	29,500	711
RPD of element		--	--	--	**4.11**	**7.00**	**4.92**	**2.78**	**6.87**	**2.64**
NIST 2704 found	3959	na	na	na	61,700	27,000	41,000	12,400	20,200	1,000
NIST 2704 true (Potts and others, 1992)		na	na	na	61,090	26,000	41,100	12,000	20,000	1,000
RPD of element		--	--	--	**.99**	**3.77**	**.24**	**3.28**	**1.00**	**0**
Median RPD of SRMs = 5.3										

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Group number	Total carbon (percent)	Inorganic carbon, as CO_3 (percent)	Organic carbon (percent)	Alumi-num	Calcium	Iron	Magne-sium	Potas-sium	Phos-phorus
HNO_3 blank	3958	na	na	na	<8	<20	<50	<0.3	<20	<8
HNO_3 blank	3958	na	na	na	<8	<20	<50	<.3	<20	<8
HNO_3 blank	3958	na	na	na	<8	<20	<50	<.3	<20	<8
MAG–1 found	3958	na	na	na	91,700	10,500	50,500	19,200	32,000	772
MAG–1 true (Potts and others, 1992)		na	na	na	86,660	9,790	47,600	18,090	29,500	711
RPD of element		--	--	--	**5.65**	**7.00**	**5.91**	**5.95**	**8.13**	**8.23**
NIST 2704 found	3958	na	na	na	62,200	26,200	41,500	12,500	20,200	974
NIST 2704 true (Potts and others, 1992)		na	na	na	61,090	26,000	41,100	12,000	20,000	1,000
RPD of element		--	--	--	**1.80**	**.77**	**.97**	**4.08**	**1.00**	**2.63**
SCO–1 found	3958	na	na	na	72,600	18,600	35,700	16,100	22,500	890
SCO–1 true (Potts and others, 1992)		na	na	na	72,370	18,700	35,900	16,400	23,000	899
RPD of element		--	--	--	**.32**	**.54**	**.56**	**1.85**	**2.20**	**1.01**
GSD–8 found	3958	na	na	na	40,300	1,490	15,300	1,450	24,000	125
GSD–8 true (Potts and others, 1992)		na	na	na	40,800	1,790	15,380	1,510	23,500	130
RPD of element		--	--	--	**1.23**	**18.29**	**.52**	**4.05**	**2.11**	**3.92**
MAG–1 found	3958	na	na	na	89,800	10,400	49,900	18,900	31,200	763
MAG–1 true (Potts and others, 1992)		na	na	na	86,660	9,790	47,600	18,090	29,500	711
RPD of element		--	--	--	**3.56**	**6.04**	**4.72**	**4.38**	**5.60**	**7.06**
NIST 2704 found	3958	na	na	na	61,500	26,300	41,300	12,600	20,000	1,000
NIST 2704 true (Potts and others, 1992)		na	na	na	61,090	26,000	41,100	12,000	20,000	1,000
RPD of element		--	--	--	**.67**	**1.15**	**.49**	**4.88**	**0**	**0**
SCO–1 found	3958	na	na	na	70,600	18,300	35,000	16,400	22,200	916
SCO–1 true (Potts and others, 1992)		na	na	na	72,370	18,700	35,900	16,400	23,000	899
RPD of element		--	--	--	**2.48**	**2.16**	**2.54**	**0**	**3.54**	**1.87**
GSD–8 found	3958	na	na	na	40,600	1,500	15,300	1,500	24,100	125
GSD–8 true (Potts and others, 1992)		na	na	na	40,800	1,790	15,380	1,510	23,500	130
RPD of element		--	--	--	**.49**	**17.63**	**.52**	**.66**	**2.52**	**3.92**
Median RPD of SRMs = 4.7										

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Sodium	Titanium	Anti-mony	Ar-senic	Barium	Beryl-lium	Cad-mium	Cerium	Chro-mium	Cobalt
CDO.GP 9–10	790	3,020	0.977	7.76	416	1.40	0.238	56.0	67.1	14.1
CDO.GP 9–10 lab dup	826	3,180	.977	7.98	413	1.42	.233	56.1	67.6	14.1
Median RPD of sample = 1.2	**4.46**	**5.16**	**0**	**2.80**	**.72**	**1.42**	**2.12**	**.18**	**.74**	**0**
CDO.GP 15–16	1,130	3,260	1.01	8.29	435	1.53	.319	64.1	69.5	15.0
CDO.GP 15–16 lab tri 1	1,140	3,380	1.02	8.11	437	1.63	.373	65.5	71.4	15.0
	.88	**3.61**	**.99**	**2.20**	**.46**	**6.33**	**15.61**	**2.16**	**2.70**	**0**
CDO.GP 15–16	1,130	3,260	1.01	8.29	435	1.53	.319	64.1	69.5	15.0
CDO.GP 15–16 lab tri 2	1,080	3,490	1.06	8.24	430	1.61	.344	62.5	70.1	14.9
	4.52	**6.81**	**4.83**	**.60**	**1.16**	**5.10**	**7.54**	**2.53**	**.86**	**.67**
CDO.GP 15–16 lab tri 1	1,140	3,380	1.02	8.11	437	1.63	.373	65.5	71.4	15.0
CDO.GP 15–16 lab tri 2	1,080	3,490	1.06	8.24	430	1.61	.344	62.5	70.1	14.9
Median RPD of sample = 3.0	**5.41**	**3.20**	**3.85**	**1.59**	**1.61**	**1.23**	**8.09**	**4.69**	**1.84**	**.67**
CDO.HB 4–5	1,140	2,810	.711	8.00	507	2.24	.498	69.9	57.6	27.0
CDO.HB 4–5 lab dup	1,100	2,810	.705	8.24	508	2.18	.501	71.1	58.0	27.1
Median RPD of sample = 0.9	**3.57**	**0**	**.85**	**2.96**	**.20**	**2.71**	**.60**	**1.70**	**.69**	**.37**
CDO.CR 0–0.5	892	2,600	.86	12	432	3.1	.76	122	59.3	46.7
CDO.CR 0–0.5 lab tri 1	841	2,800	.90	12	436	3.1	.77	122	62.2	48.1
	5.89	**7.41**	**4.55**	**0**	**.92**	**0**	**1.31**	**0**	**4.77**	**2.95**
CDO.CR 0–0.5	892	2,600	.86	12	432	3.1	.76	122	59.3	46.7
CDO.CR 0–0.5 lab tri 2	855	2,800	.84	13	437	3.2	.78	125	61.8	48.4
	4.24	**7.41**	**2.35**	**8.00**	**1.15**	**3.17**	**2.60**	**2.43**	**4.13**	**3.58**
CDO.CR 0–0.5 lab tri 1	841	2,800	.90	12	436	3.1	.77	122	62.2	48.1
CDO.CR 0–0.5 lab tri 2	855	2,800	.84	13	437	3.2	.78	125	61.8	48.4
Median RPD of sample = 2.8	**1.65**	**0**	**6.90**	**8.00**	**.23**	**3.17**	**1.29**	**2.43**	**.65**	**.62**
CDO.CR 13–14	726	3,200	.91	15	481	3.2	.83	134	64.5	53.0
CDO.CR 13–14 lab dup	781	3,100	.88	14	501	3.2	.78	128	61.5	53.7
Median RPD of sample = 4.7	**7.30**	**3.17**	**3.35**	**6.90**	**4.07**	**0**	**6.21**	**4.58**	**4.76**	**1.31**
CDO.CR 19–20	764	3,200	.92	14	458	3.5	.79	125	67.5	44.2
CDO.CR 19–20 lab dup	782	3,400	.96	14	454	3.4	.81	122	66.3	44.0
Median RPD of sample = 0.8	**2.33**	**6.06**	**4.26**	**0**	**.88**	**2.90**	**2.50**	**2.43**	**1.79**	**.45**

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Sodium	Titanium	Antimony	Arsenic	Barium	Beryllium	Cadmium	Cerium	Chromium	Cobalt
HNO_3 blank	<6	<40	<0.02	<0.1	<0.5	<0.001	<0.003	<0.5	<0.2	<0.1
HNO_3 blank	<6	<40	<.02	.1	<.5	.007	<.003	<.5	<.2	<.1
HNO_3 blank	<6	<40	<.02	.2	<.5	.003	<.003	<.5	<.2	<.1
HNO_3 blank	<6	<40	<.02	<.1	<.5	.006	<.003	<.5	<.2	<.1
MAG–1 found	29,300	4,300	.91	10	508	3.4	.22	89.4	112	22.5
MAG–1 true (Potts and others, 1992)	28,400	4,500	.96	9.2	479	3.20	.20	88.0	97	20.4
RPD of element	**3.12**	**4.55**	**5.35**	**8.33**	**5.88**	**6.06**	**8.53**	**1.58**	**14.35**	**9.79**
NIST 2704 found	5,800	3,400	3.5	22	421	1.8	3.6	59.3	146	13.9
NIST 2704 true (Potts and others, 1992)	5,470	4,580	3.79	23.4	414	na	3.45	72.0	135	14.0
RPD of element	**5.86**	**29.57**	**7.96**	**6.17**	**1.68**	--	**4.26**	**19.35**	**7.83**	**.72**
SCO–1 found	6,890	3,600	2.6	13	588	1.9	.16	55.8	69.7	11.7
SCO–1 true (Potts and others, 1992)	6,670	3,760	2.50	12.4	570	1.84	.14	62.0	68.0	10.5
RPD of element	**3.24**	**4.35**	**3.92**	**4.72**	**3.11**	**3.21**	**13.33**	**10.53**	**2.47**	**10.81**
GSD–8 found	3,220	3,600	.2	2	450	2.0	.01	52.7	5.4	3.4
GSD–8 true (Potts and others, 1992)	3,490	3,660	.24	2.4	480	2.00	.08	54.0	7.60	3.6
RPD of element	**8.05**	**1.65**	**18.18**	**18.18**	**6.45**	**0**	**156.04**	**2.44**	**33.85**	**5.71**
MESS–1 found	17,500	3,500	.67	12	298	2.3	.66	69.7	63.1	11.6
MESS–1 true (Potts and others, 1992)	18,546	5,426	.73	10.6	na	1.9	.59	na	71	10.8
RPD of element	**5.80**	**43.15**	**8.57**	**12.39**	--	**19.05**	**11.20**	--	**11.78**	**7.14**
MAG–1 found	29,100	4,000	.81	9.9	491	3.2	.21	81.3	101	22.2
MAG–1 true (Potts and others, 1992)	28,400	4,500	.96	9.2	479	3.20	.20	88.0	97	20.4
RPD of element	**2.43**	**11.76**	**16.95**	**7.33**	**2.47**	**0**	**3.88**	**7.91**	**4.04**	**8.45**
NIST 2704 found	5,650	3,100	3.2	22	415	2.0	3.5	59.4	143	13.7
NIST 2704 true (Potts and others, 1992)	5,470	4,580	3.79	23.4	414	na	3.45	72.0	135	14.0
RPD of element	**3.24**	**38.54**	**16.88**	**6.17**	**.24**	--	**1.44**	**19.18**	**5.76**	**2.17**
Median RPD of SRMs = 5.3										

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Sodium	Titanium	Anti-mony	Ar-senic	Barium	Beryl-lium	Cad-mium	Cerium	Chro-mium	Cobalt
HNO_3 blank	<6	<40	<0.02	<0.1	<0.5	<0.001	<0.003	<0.5	<0.2	<0.1
HNO_3 blank	<6	<40	<.02	<.1	<.5	.00800	<.003	<.5	<.2	<.1
HNO_3 blank	<6	<40	<.02	<.1	<.5	.0100	<.003	<.5	<.2	<.1
MAG–1 found	29,800	4,350	.926	10.7	509	3.22	.229	86.6	110	22.5
MAG–1 true (Potts and others, 1992)	28,400	4,500	.96	9.2	479	3.20	.20	88.0	97	20.4
RPD of element	**4.81**	**3.39**	**3.61**	**15.08**	**6.07**	**.62**	**12.53**	**1.60**	**12.56**	**9.79**
NIST 2704 found	5,800	3,500	3.60	20.9	423	1.83	3.45	64.5	135	13.6
NIST 2704 true (Potts and others, 1992)	5,470	4,580	3.79	23.4	414	na	3.45	72.0	135	14.0
RPD of element	**5.86**	**26.73**	**5.14**	**11.29**	**2.15**	**--**	**0**	**10.99**	**0**	**2.90**
SCO–1 found	6,630	3,360	2.42	12.1	583	1.81	.142	56.0	71.9	11.1
SCO–1 true (Potts and others, 1992)	6,670	3,760	2.50	12.4	570	1.84	.14	62.0	68.0	10.5
RPD of element	**.60**	**11.24**	**3.25**	**2.45**	**2.25**	**1.64**	**1.42**	**10.17**	**5.58**	**5.56**
GSD–8 found	3,110	3,360	.184	2.73	462	1.93	.068	55.1	5.55	3.34
GSD–8 true (Potts and others, 1992)	3,490	3,660	.24	2.4	480	2.00	.08	54.0	7.60	3.6
RPD of element	**11.52**	**8.55**	**26.42**	**12.87**	**3.82**	**3.56**	**17.45**	**2.02**	**31.18**	**7.49**
MAG–1 found	29,600	4,280	.943	10.4	490	3.25	.225	85.3	107	22.2
MAG–1 true (Potts and others, 1992)	28,400	4,500	.96	9.2	479	3.20	.20	88.0	97	20.4
RPD of element	**4.14**	**5.01**	**1.79**	**12.24**	**2.27**	**1.55**	**10.77**	**3.12**	**9.80**	**8.45**
NIST 2704 found	5,840	3,460	3.70	21.6	409	1.96	3.56	62.4	135	13.6
NIST 2704 true (Potts and others, 1992)	5,470	4,580	3.79	23.4	414	na	3.45	72.0	135	14.0
RPD of element	**6.54**	**27.86**	**2.40**	**8.00**	**1.22**	--	**3.14**	**14.29**	**0**	**2.90**
SCO–1 found	6,760	3,300	2.45	12.0	557	1.86	.149	54.6	70.4	11.0
SCO–1 true (Potts and others, 1992)	6,670	3,760	2.50	12.4	570	1.84	.14	62.0	68.0	10.5
RPD of element	**1.34**	**13.03**	**2.02**	**3.28**	**2.31**	**1.08**	**6.23**	**12.69**	**3.47**	**4.65**
GSD–8 found	3,190	3,500	.231	2.71	448	2.03	.015	53.4	8.04	3.33
GSD–8 true (Potts and others, 1992)	3,490	3,660	.24	2.4	480	2.00	.08	54.0	7.60	3.6
RPD of element	**8.98**	**4.47**	**3.82**	**12.13**	**6.90**	**1.49**	**137.50**	**1.12**	**5.63**	**7.79**
Median RPD of SRMs = 4.7										

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Copper	Gallium	Lantha-num	Lead	Lithium	Manga-nese	Mer-cury	Molyb-denum	Nickel
CDO.GP 9–10	32.9	16.3	28.6	71.8	42.8	479	0.52	2.12	29.9
CDO.GP 9–10 lab dup	33.6	16.2	28.8	72.3	42.3	471	.53	2.19	30.1
Median RPD of sample = 1.2	**2.11**	**.62**	**.70**	**.69**	**1.18**	**1.68**	**1.90**	**3.25**	**.67**
CDO.GP 15–16	35.9	15.4	32.7	111	42.8	314	.76	1.70	30.6
CDO.GP 15–16 lab tri 1	42.4	16.3	33.1	112	45.9	326	na	1.48	30.1
	16.60	**5.68**	**1.22**	**.90**	**6.99**	**3.75**	--	**13.84**	**1.65**
CDO.GP 15–16	35.9	15.4	32.7	111	42.8	314	.76	1.70	30.6
CDO.GP 15–16 lab tri 2	36.8	16.0	31.5	108	44.5	318	na	1.60	29.9
	2.48	**3.82**	**3.74**	**2.74**	**3.89**	**1.27**	--	**6.06**	**2.31**
CDO.GP 15–16 lab tri 1	42.4	16.3	33.1	112	45.9	326	na	1.48	30.1
CDO.GP 15–16 lab tri 2	36.8	16.0	31.5	108	44.5	318	na	1.60	29.9
Median RPD of sample = 3.0	**14.14**	**1.86**	**4.95**	**3.64**	**3.10**	**2.48**	--	**7.79**	**.67**
CDO.HB 4–5	24.9	15.7	38.6	24.6	38.9	1,410	.15	1.57	41.2
CDO.HB 4–5 lab dup	24.0	15.9	38.8	24.7	40.2	1,420	.16	1.52	40.6
Median RPD of sample = 0.9	**3.68**	**1.27**	**.52**	**.41**	**3.29**	**.71**	**6.45**	**3.24**	**1.47**
CDO.CR 0–0.5	26.0	16	52.7	28.1	42.2	1,350	.19	3.0	39.8
CDO.CR 0–0.5 lab tri 1	26.8	16	52.1	27.5	42.7	1,430	na	3.0	41.1
	3.03	**0**	**1.15**	**2.16**	**1.18**	**5.76**	--	**0**	**3.21**
CDO.CR 0–0.5	26.0	16	52.7	28.1	42.2	1,350	.19	3.0	39.8
CDO.CR 0–0.5 lab tri 2	26.7	16	53.6	28.2	43.3	1,430	na	3.0	41.0
	2.66	**0**	**1.69**	**.36**	**2.57**	**5.76**	--	**0**	**2.97**
CDO.CR 0–0.5 lab tri 1	26.8	16	52.1	27.5	42.7	1,430	na	3.0	41.1
CDO.CR 0–0.5 lab tri 2	26.7	16	53.6	28.2	43.3	1,430	na	3.0	41.0
Median RPD of sample = 2.8	**.37**	**0**	**2.84**	**2.51**	**1.40**	**0**	--	**0**	**.24**
CDO.CR 13–14	24.1	17	56.9	35.0	46.8	1,900	.21	3.2	45.0
CDO.CR 13–14 lab dup	23.7	16	54.5	33.5	44.5	1,970	.18	3.1	44.1
Median RPD of sample = 4.7	**1.67**	**6.06**	**4.31**	**4.38**	**5.04**	**3.62**	**15.38**	**3.17**	**2.02**
CDO.CR 19–20	22.4	18	54.5	41.8	51.1	1,400	.21	1.8	46.1
CDO.CR 19–20 lab dup	22.9	18	54.1	40.9	50.7	1,390	.21	1.8	46.0
Median RPD of sample = 0.8	**2.21**	**0**	**.74**	**2.18**	**.79**	**.72**	**0**	**0**	**.22**

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Copper	Gallium	Lanthanum	Lead	Lithium	Manganese	Mercury	Molybdenum	Nickel
HNO_3 blank	<0.5	<0.006	<0.3	<0.2	<0.2	<0.2	na	<0.1	<1
HNO_3 blank	<.5	<.006	<.3	<.2	<.2	<.2	na	<.1	<1
HNO_3 blank	<.5	<.006	<.3	<.2	<.2	<.2	na	<.1	<1
HNO_3 blank	<.5	<.006	<.3	<.2	<.2	<.2	na	<.1	<1
MAG–1 found	30.1	23	43.4	26.3	78.0	777	na	1.1	49.2
MAG–1 true (Potts and others, 1992)	30.0	20.4	43.0	24.0	79.0	760	na	1.60	53.0
RPD of element	**.33**	**11.98**	**.93**	**9.15**	**1.27**	**2.21**	--	**37.04**	**7.44**
NIST 2704 found	94.8	16	30.6	154	45.2	590	na	3.7	41.9
NIST 2704 true (Potts and others, 1992)	98.6	15.0	29.0	161.0	47.5	555	na	na	44.1
RPD of element	**3.93**	**6.45**	**5.37**	**4.44**	**4.96**	**6.11**	--	--	**5.12**
SCO–1 found	29.4	17	33.5	31.6	45.8	409	na	1.2	26.0
SCO–1 true (Potts and others, 1992)	28.7	15.0	29.5	31.0	45.0	410	na	1.37	27.0
RPD of element	**2.41**	**12.50**	**12.70**	**1.92**	**1.76**	**.24**	--	**13.23**	**3.77**
GSD–8 found	5.5	10	27.5	20.6	13.5	347	na	.35	1.6
GSD–8 true (Potts and others, 1992)	4.1	10.8	30.0	21.0	13.2	310	na	.54	2.7
RPD of element	**29.17**	**7.69**	**8.70**	**1.92**	**2.25**	**11.26**	--	**42.70**	**51.16**
MESS–1 found	25.8	14	38.1	32.8	41.3	532	na	2.1	25.8
MESS–1 true (Potts and others, 1992)	25.1	na	na	34	44.9	512	na	na	29.5
RPD of element	**2.75**	--	--	**3.59**	**8.35**	**3.83**	--	--	**13.38**
MAG–1 found	29.1	23	41.2	26.0	77.4	788	na	1.0	48.3
MAG–1 true (Potts and others, 1992)	30.0	20.4	43.0	24.0	79.0	760	na	1.60	53.0
RPD of element	**3.05**	**11.98**	**4.28**	**8.00**	**2.05**	**3.62**	--	**46.15**	**9.28**
NIST 2704 found	93.6	16	31.7	162	44.4	596	na	3.6	41.1
NIST 2704 true (Potts and others, 1992)	98.6	15.0	29.0	161.0	47.5	555	na	na	44.1
RPD of element	**5.20**	**6.45**	**8.90**	**.62**	**6.75**	**7.12**	--	--	**7.04**
Median RPD of SRMs = 5.3									

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Copper	Gallium	Lanthanum	Lead	Lithium	Manganese	Mercury	Molybdenum	Nickel
HNO_3 blank	<0.5	<0.006	<0.3	<0.2	<0.2	<0.2	na	<0.1	<1
HNO_3 blank	<.5	<.006	<.3	<.2	<.2	<.2	na	<.1	<1
HNO_3 blank	<.5	<.006	<.3	<.2	<.2	<.2	na	<.1	<1
MAG–1 found	30.1	22.9	43.6	26.1	79.4	779	na	1.35	49.9
MAG–1 true (Potts and others, 1992)	30.0	20.4	43.0	24.0	79.0	760	na	1.60	53.0
RPD of element	**0.33**	**11.55**	**1.39**	**8.38**	**.51**	**2.47**	--	**16.95**	**6.03**
NIST 2704 found	93.4	15.2	32.0	158	45.8	584	na	3.97	42.2
NIST 2704 true (Potts and others, 1992)	98.6	15.0	29.0	161.0	47.5	555	na	na	44.1
RPD of element	**5.42**	**1.32**	**9.84**	**1.88**	**3.64**	**5.09**	--	--	**4.40**
SCO–1 found	29.3	16.2	30.0	29.8	43.7	384	na	1.30	24.9
SCO–1 true (Potts and others, 1992)	28.7	15.0	29.5	31.0	45.0	410	na	1.37	27.0
RPD of element	**2.07**	**7.69**	**1.68**	**3.95**	**2.93**	**6.55**	--	**5.24**	**8.09**
GSD–8 found	5.10	10.2	28.6	20.8	13.2	334	na	.578	<1
GSD–8 true (Potts and others, 1992)	4.1	10.8	30.0	21.0	13.2	310	na	.54	2.7
RPD of element	**21.74**	**5.71**	**4.78**	**.96**	**0**	**7.45**	--	**6.80**	--
MAG–1 found	30.1	22.8	43.5	26.0	78.7	766	na	1.32	49.7
MAG–1 true (Potts and others, 1992)	30.0	20.4	43.0	24.0	79.0	760	na	1.60	53.0
RPD of element	**.33**	**11.11**	**1.16**	**8.00**	**.38**	**.79**	--	**19.18**	**6.43**
NIST 2704 found	93.8	15.2	32.0	155	45.8	582	na	3.79	42.5
NIST 2704 true (Potts and others, 1992)	98.6	15.0	29.0	161.0	47.5	555	na	na	44.1
RPD of element	**4.99**	**1.32**	**9.84**	**3.80**	**3.64**	**4.75**	--	--	**3.70**
SCO–1 found	29.5	16.2	29.4	28.7	43.3	377	na	1.27	24.7
SCO–1 true (Potts and others, 1992)	28.7	15.0	29.5	31.0	45.0	410	na	1.37	27.0
RPD of element	**2.75**	**7.69**	**.34**	**7.71**	**3.85**	**8.39**	--	**7.58**	**8.90**
GSD–8 found	5.35	10.3	28.2	20.0	13.1	336	na	.586	<1
GSD–8 true (Potts and others, 1992)	4.1	10.8	30.0	21.0	13.2	310	na	.54	2.7
RPD of element	**26.46**	**4.74**	**6.19**	**4.88**	**.76**	**8.05**	--	**8.17**	--
Median RPD of SRMs = 4.7									

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Niobium	Scan-dium	Sele-nium	Silver	Stron-tium	Thorium	Uranium	Vana-dium	Zinc
CDO.GP 9–10	13.6	11.0	1.55	12.2	61.5	9.24	2.61	93.9	122
CDO.GP 9–10 lab dup	14.3	11.0	1.70	11.5	61.2	9.15	2.60	95.2	123
Median RPD of sample = 1.2	**5.02**	**0**	**9.23**	**5.91**	**.49**	**.98**	**.38**	**1.37**	**.82**
CDO.GP 15–16	22.7	10.8	1.37	14.2	57.9	10.2	3.03	89.0	127
CDO.GP 15–16 lab tri 1	15.2	10.6	1.51	14.9	60.2	9.95	3.00	92.8	140
	39.58	**1.87**	**9.72**	**4.81**	**3.90**	**2.48**	**1.00**	**4.18**	**9.74**
CDO.GP 15–16	22.7	10.8	1.37	14.2	57.9	10.2	3.03	89.0	127
CDO.GP 15–16 lab tri 2	16.6	10.6	1.33	18.4	58.6	9.65	2.92	91.3	135
	31.04	**1.87**	**2.96**	**25.77**	**1.20**	**5.54**	**3.70**	**2.55**	**6.11**
CDO.GP 15–16 lab tri 1	15.2	10.6	1.51	14.9	60.2	9.95	3.00	92.8	140
CDO.GP 15–16 lab tri 2	16.6	10.6	1.33	18.4	58.6	9.65	2.92	91.3	135
Median RPD of sample = 3.0	**8.81**	**0**	**12.68**	**21.02**	**2.69**	**3.06**	**2.70**	**1.63**	**3.64**
CDO.HB 4–5	14.7	10.7	1.66	<3	81.4	8.83	2.47	89.4	145
CDO.HB 4–5 lab dup	13.4	10.6	1.86	<3	81.8	8.82	2.46	90.5	142
Median RPD of sample = 0.9	**9.25**	**.94**	**11.36**	--	**.49**	**.11**	**.41**	**1.22**	**2.09**
CDO.CR 0–0.5	14	10.1	2.0	na	56.6	9.2	2.5	95.0	235
CDO.CR 0–0.5 lab tri 1	13	10.5	2.0	na	58.2	8.6	2.4	99.9	250
	7.41	**3.88**	**0**	--	**2.79**	**6.74**	**4.08**	**5.03**	**6.19**
CDO.CR 0–0.5	14	10.1	2.0	na	56.6	9.2	2.5	95.0	235
CDO.CR 0–0.5 lab tri 2	13	10.7	2.3	na	58.2	8.9	2.5	100	249
	7.41	**5.77**	**13.95**	--	**2.79**	**3.31**	**0**	**5.13**	**5.79**
CDO.CR 0–0.5 lab tri 1	13	10.5	2.0	na	58.2	8.6	2.4	99.9	250
CDO.CR 0–0.5 lab tri 2	13	10.7	2.3	na	58.2	8.9	2.5	100	249
Median RPD of sample = 2.8	**0**	**1.89**	**13.95**	--	**0**	**3.43**	**4.08**	**.10**	**.40**
CDO.CR 13–14	14	11.1	2.4	na	61.4	9.5	2.7	106	293
CDO.CR 13–14 lab dup	13	10.5	2.2	na	65.3	9.0	2.6	102	281
Median RPD of sample = 4.7	**7.41**	**5.56**	**8.70**	--	**6.16**	**5.41**	**3.77**	**3.85**	**4.18**
CDO.CR 19–20	15	11.4	1.8	na	57.0	10.1	2.7	110	299
CDO.CR 19–20 lab dup	15	11.4	1.8	na	58.2	9.9	2.6	110	296
Median RPD of sample = 0.8	**0**	**0**	**0**	--	**2.08**	**2.00**	**3.77**	**0**	**1.01**

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Niobium	Scan-dium	Sele-nium	Silver	Stron-tium	Thorium	Uranium	Vana-dium	Zinc
HNO_3 blank	<2	<0.3	<0.2	na	<0.05	<0.03	<0.02	<0.4	<5
HNO_3 blank	<2	<.3	<.2	na	<.05	<.03	<.02	<.4	<5
HNO_3 blank	<2	<.3	<.2	na	<.05	<.03	<.02	<.4	<5
HNO_3 blank	<2	<.3	<.2	na	<.05	<.03	<.02	<.4	<5
MAG–1 found	20	18.8	1.4	.48	146	12.7	2.7	146	141
MAG–1 true (Potts and others, 1992)	12.0	17.2	1.16	.08	146	11.9	2.70	140	130
RPD of element	**50.00**	**8.89**	**18.75**	**142.86**	**0**	**6.50**	**0**	**4.20**	**8.12**
NIST 2704 found	13	12.6	1.2	.42	123	8.8	2.8	95.6	439
NIST 2704 true (Potts and others, 1992)	na	12.0	1.12	na	130	9.2	3.13	95.0	438
RPD of element	--	**4.88**	**6.90**	--	**5.53**	**4.44**	**11.13**	**.63**	**.23**
SCO–1 found	14	12.8	.8	.39	172	9.8	2.9	138	109
SCO–1 true (Potts and others, 1992)	11.0	10.8	.890	.134	174	9.7	3.00	131	103
RPD of element	**24.00**	**16.95**	**10.65**	**97.71**	**1.16**	**1.03**	**3.39**	**5.20**	**5.66**
GSD–8 found	37	5.5	<.2	.75	46.6	13.2	2.9	23.6	47.8
GSD–8 true (Potts and others, 1992)	35.0	5.7	.15?	.062?	52.0	13.4	3.00	26	43.0
RPD of element	**5.56**	**3.57**	--	--	**10.95**	**1.50**	**3.39**	**9.68**	**10.57**
MESS–1 found	15	10.7	.6	.34	88.6	13.3	3.5	73.3	184
MESS–1 true (Potts and others, 1992)	na	na	.34	na	na	na	na	72.4	191
RPD of element	--	--	**55.32**	--	--	--	--	**1.24**	**3.73**
MAG–1 found	16	18.6	1.3	.38	145	11.8	2.5	145	133
MAG–1 true (Potts and others, 1992)	12.0	17.2	1.16	.08	146	11.9	2.70	140	130
RPD of element	**28.57**	**7.82**	**11.38**	**130.43**	**.69**	**.84**	**7.69**	**3.51**	**2.28**
NIST 2704 found	9.8	12.3	1.0	.37	131	9.3	2.8	93.3	428
NIST 2704 true (Potts and others, 1992)	na	12.0	1.12	na	130	9.2	3.13	95.0	438
RPD of element	--	**2.47**	**11.32**	--	**.77**	**1.08**	**11.13**	**1.81**	**2.31**
Median RPD of SRMs = 5.3									

Appendix 3.4. Quality-control samples for forms of carbon and major and trace elements—Continued

Sample ID	Niobium	Scan-dium	Sele-nium	Silver	Stron-tium	Thorium	Uranium	Vana-dium	Zinc
HNO_3 blank	<2	<0.3	<0.2	<3	<0.05	<0.03	<0.02	<0.4	<5
HNO_3 blank	<2	<.3	<.2	<3	<.05	<.03	<.02	<.4	<5
HNO_3 blank	<2	<.3	<.2	<3	<.05	<.03	<.02	<.4	<5
MAG–1 found	22.4	19.3	1.71	<3	150	12.7	2.78	149	139
MAG–1 true (Potts and others, 1992)	12.0	17.2	1.16	.08	146	11.9	2.70	140	130
RPD of element	**60.47**	**11.51**	**38.33**	--	**2.70**	**6.50**	**2.92**	**6.23**	**6.69**
NIST 2704 found	13.0	12.5	1.04	<3	134	9.55	3.05	95.0	417
NIST 2704 true (Potts and others, 1992)	na	12.0	1.12	na	130	9.2	3.13	95.0	438
RPD of element	--	**4.08**	**7.41**	--	**3.03**	**3.73**	**2.59**	**0**	**4.91**
SCO–1 found	13.3	12.3	.951	<3	166	9.72	2.92	134	96.4
SCO–1 true (Potts and others, 1992)	11.0	10.8	.890	.134	174	9.7	3.00	131	103
RPD of element	**18.93**	**12.99**	**6.63**	--	**4.71**	**.21**	**2.70**	**2.26**	**6.62**
GSD–8 found	38.5	5.48	<.2	<3	48.5	14.5	3.52	24.7	44.5
GSD–8 true (Potts and others, 1992)	35.0	5.7	.15?	.062?	52.0	13.4	3.00	26	43.0
RPD of element	**9.52**	**3.94**	--	--	**6.97**	**7.89**	**15.95**	**5.13**	**3.43**
MAG–1 found	22.3	**18.7**	1.54	<3	**148**	12.8	2.78	**147**	**138**
MAG–1 true (Potts and others, 1992)	12.0	17.2	1.16	.08	146	11.9	2.70	140	130
RPD of element	**60.06**	**8.36**	**28.15**	--	**1.36**	**7.29**	**2.92**	**4.88**	**5.97**
NIST 2704 found	13.8	12.4	1.36	<3	133	9.63	2.98	94.4	437
NIST 2704 true (Potts and others, 1992)	na	12.0	1.12	na	130	9.2	3.13	95.0	438
RPD of element	--	**3.28**	**19.35**	--	**2.28**	**4.57**	**4.91**	**.63**	**.23**
SCO–1 found	14.2	12.1	.899	<3	165	9.46	2.80	132	104
SCO–1 true (Potts and others, 1992)	11.0	10.8	.890	.134	174	9.7	3.00	131	103
RPD of element	**25.40**	**11.35**	**1.01**	--	**5.31**	**2.51**	**6.90**	**.76**	**.97**
GSD–8 found	36.7	5.34	<.2	<3	48.9	14.1	3.28	24.8	48.5
GSD–8 true (Potts and others, 1992)	35.0	5.7	.15?	.062?	52.0	13.4	3.00	26	43.0
RPD of element	**4.74**	**6.52**	--	--	**6.14**	**5.09**	**8.92**	**4.72**	**12.02**
Median RPD of SRMs = 4.7									

Appendix 3.5. Quality-control samples for petroleum biomarkers

[In micrograms per kilogram, except as noted; E, estimated; lab dup, laboratory duplicate; <, less than; RPD, relative percent difference; na, not analyzed]

Sample ID	Group number	Nonane (C9)	Decane (C10)	Undecane (C11)	Dodecane (C12)	Tridecane (C13)	Tetradecane (C14)	Pentadecane (C15)	Hexadecane (C16)	Heptacecane (C17)	Pristane
CDO.MD 11–12	8217R02171	E3.4	E9.5	E24.5	E3.7	E3.3	E7.1	E7.2	E23.2	73.6	3.2
CDO.MD 11–12 lab dup	8217R02171	<40	E5.0	E12.4	E5.0	E3.1	E5.8	E7.3	E27.0	65.0	3.2
RPD of sample = 12.9	**RPD of compound =**		**62.1**	**65.6**	**29.9**	**6.2**	**20.2**	**1.4**	**15.1**	**12.4**	**0**
Lab blank	8217R02171	E.76	E5.8	E12.8	E.72	E.57	E1.7	E1.8	E9.5	E1.3	<20
Lab spike, percent recovery	8217R02171	E15.0	47.7	82.7	45.3	56.0	64.3	74.0	125	82.0	70.0

Sample ID	Octadecane (C18)	Phytane	Nonadecane (C19)	Eicosane (C20)	Heneicosane (C21)	Docosane (C22)	Tricosane (C23)	Tetracosane (C24)	Pentacosane (C25)	5B (H) cholane	Hexacosane (C26)	n-heptacosane (C27)
CDO.MD 11–12	E12.8	14.8	97.1	58.6	99.6	E65.1	E278	E87.8	E459	<40	E116	E713
CDO.MD 11–12 lab dup	E12.4	13.0	81.9	51.0	91.5	E59.1	E215	E102	E369	<40	E88.2	E538
RPD of sample = 12.9	**3.2**	**12.9**	**17.0**	**13.9**	**8.5**	**9.7**	**25.6**	**15.0**	**21.7**		**27.2**	**28.0**
Lab blank	E.89	<20	E.92	E1.4	E1.4	E2.1	E4.3	E13.0	E5.3	<20	E5.30	E5.30
Lab spike, percent recovery	82.0	78.8	87.7	102	114	E157	E182	E221	E171	na	E152	E148

Sample ID	Cholestane series	Octacosane (C28)	Cholestane + Coprostane	4A-methyl sterane series	n-nonacosane (C29)	Hopane triterpanoid series	17A(H) 22,29,30 trisnorhopane	4A-C28 methylsterane	n-triacontane (C30)	n-hentriacontane (C31)	17B(H) 21A(H) 30 norhopane
CDO.MD 11–12	E36.7	E127	E2.1	1,260	E609	2,000	<40	<40	E104	E523	<40
CDO.MD 11–12 lab dup	40.0	E112	E2.3	1,440	E530	1,830	<40	<40	E97.6	E472	<40
RPD of sample = 12.9	**8.6**	**12.6**	**9.1**	**13.3**	**13.9**	**8.9**			**6.3**	**10.3**	
Lab blank	<20	E5.2	<20	<20	E6.0	<20	<20	<20	E4.5	E4.1	<20
Lab spike, percent recovery	na	E154	106	na	E145	na	na	na	E135	E135	na

Sample ID	4A-C30 methylsterane	17A(H) 21B hopane	n-dotriacontane (C32)	17B(H) 21A hopane	n-tritriacontane (C33)	17B(H) 21B(H) hopane	n-tetratriacontane (C34)	n-pentatriacontane (C35)	n-hexatriacontane (C36)	n-octatriacontane (C38)	nonatriacontane (C39)	n-tetracontane (C40)
CDO.MD 11–12	<40	E27.7	E58.4	<40	E260	E26.9	E32.0	E101	E37.6	<100	E84.8	E56.9
CDO.MD 11–12 lab dup	<40	E22.2	E57.3	<40	E229	E19.7	E32.6	E101	E31.6	<100	E78.1	E77.1
RPD of sample = 12.9		**22.0**	**1.9**		**12.7**	**30.9**	**1.9**	**0**	**17.3**		**8.2**	**30.1**
Lab blank	<20	<20	E3.1	<20	E2.40	<20	E1.4	E1.3	E1.1	<20	<20	<20
Lab spike, percent recovery	na	na	E125	na	E120	na	E111	E104	E91.3	E90.7	E88.7	E97.3