

Atomic Absorption Spectrometric Determination of Chromium, Copper, Lead, Mercury, and Zinc in Sediments Collected in Bayou d'Inde, Southwestern Louisiana

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The concentrations of the metals chromium (Cr), copper (Cu), lead (Pb), mercury (Hg), and zinc (Zn) were determined in sediment cores taken from Bayou d'Inde in southwestern Louisiana. Previous studies had reported elevated concentrations of these trace metals in sediments along this waterway. The current study, conducted more than a decade later, was designed to determine if these metals had migrated from the areas of highest concentrations. Concentrations as high as 400 mg/kg for Cr, 1100 mg/kg for Cu, 10 mg/kg for Hg, and 600 mg/kg for Pb and Zn were found. Areas of high concentrations of metals were isolated within a distance of about 1 km from the most highly contaminated areas in the bayou. Low concentrations of metals were found at the mouth of the bayou. Estimated sedimentation rates of 0.67–1.2 cm/yr were based on the burial of the sediments originally studied. The concentrations of the metals studied remained high and were also highly localized, both spatially and temporally.

Bayou d'Inde, a major tributary of the Calcasieu River, is located in southwestern Louisiana near the city of Lake Charles and drains a sizable metropolitan and industrial area. A bayou is defined as a small, sluggish inlet, with currents that are typically 5 cm/s. This sluggishness plays an important role in the redistribution of pollutants introduced to the bayou. During the early 1940s, the area surrounding Bayou d'Inde became heavily industrialized and populated and, ever since, it has been used as a discharge point for both industrial and municipal waste. Several studies (1–7) of

the Calcasieu River watershed have identified Bayou d'Inde and Bayou Verdine as major sources of pollutants that could ultimately negatively impact the entire watershed.

Concentrations of the metals chromium (Cr), copper (Cu), lead (Pb), mercury (Hg), and zinc (Zn) were determined in sediment samples located across the Calcasieu River/Lake riverine system during the period from November 1983 to July 1984 (1). Uncontaminated sediments within the watershed were reported to have baseline concentrations of 25 mg/kg for Cr, 10 mg/kg for Cu, 15 mg/kg for Pb, 0.10 mg/kg for Hg, and 40 mg/kg for Zn. Baseline concentrations determined only in Calcasieu Lake were previously reported to be 18 mg/kg for Cr, 12 mg/kg for Cu, 10 mg/kg for Pb, 0.10 mg/kg for Hg, and 36 mg/kg for Zn (2). Similar concentrations are generally found throughout the watershed except in the vicinity of and within Bayou d'Inde, Contraband Bayou, and Bayou Verdine. Contraband Bayou drains the city of Lake Charles and carries mainly municipal waste, whereas Bayou Verdine carries industrial wastes almost exclusively. This previous study focused on variations in metal concentrations found along the lower portions of Bayou d'Inde. Metal concentrations in sediments along Bayou d'Inde were reported to be as high as 600 mg/kg for Cr, 650 mg/kg for Cu, 225 mg/kg for Pb, 4.0 mg/kg for Hg, and 250 mg/kg for Zn, which far exceeded baseline concentrations (1). The previous study also showed that these metals were highly localized (1), were bound strongly to sediments, and did not migrate from the bayou into the estuary as a whole (8).

Later studies (2–6) conducted from 1989 to 1994 also showed that metal contamination in sediments was highly localized in the northern portion of the estuary, mainly around the 3 major bayous. For example, trace metals determined in sediments collected in the northern reaches of the Calcasieu River (Figure 1) were found to have concentrations as high as 710 mg/kg for Cr, 346 mg/kg for Zn, 60 mg/kg for Cu, and 129 mg/kg for Pb, but they decreased to baseline levels in the Ship Channel, near Calcasieu Lake (4). These trends in concentrations, which were confirmed in all subsequent studies, also

Guest edited as a special report on "Environmental Analysis" by Joseph Sherma.

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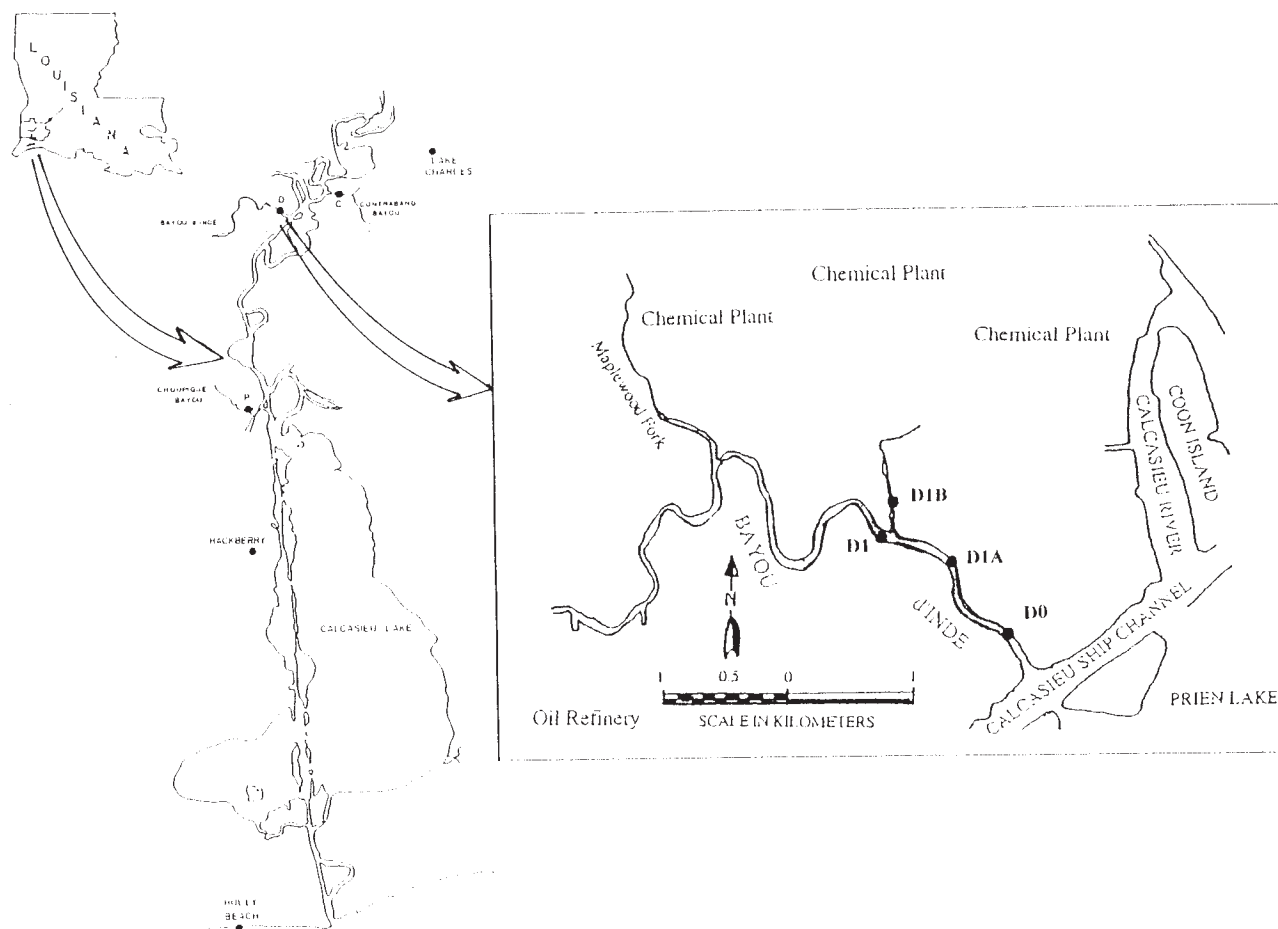


Figure 1. Map of the study area in southwestern Louisiana, including the location of Bayou d'Inde within the Calcasieu estuary along with the relative locations of the sampling sites. The notations C (Contraband Bayou), D (Bayou d'Inde), and P (Choupique Bayou) on the regional map show the locations of major tributaries of the Calcasieu River in the vicinity of the sample area in Bayou d'Inde.

reveal that the highest concentrations of pollutants are found along and within Bayou d'Inde and Bayou Verdine (2–6).

The current study was undertaken to determine if metal concentrations within Bayou d'Inde remain as high and as localized as reported previously (1). Core samples were collected in Bayou d'Inde to coincide with the sampling stations established in the previous study (1). This approach allowed a determination of the extent of migration as well as the current sedimentation rates within the bayou. Metal concentration profiles determined could then be correlated with previous results to determine concentration changes with time and location. This current study was conducted about 12 years after the previous comprehensive study of the bayou.

Experimental

Sample Sites

All sample sites were established within Bayou d'Inde, were selected to coincide with sampling stations established

previously, and were focused on areas where the highest concentrations of metals were reported. Figure 1 shows a map of the study area and gives the relative locations of the sampling stations. The bayou is a narrow waterway with a width of 70 m and a depth of 5 m. Sample station D1 was located 100 m upstream from an industrial drainage canal. Station D1A was located ca 0.7 km downstream from D1; D0 was located at the bayou mouth, ca 2 km from D1. D1B was located ca 200 m upstream from the mouth of a drainage canal. D1 and D1B represented areas of high concentrations of metals; D1A, intermediate concentrations; and D0, low concentrations.

Sample Collection and Handling

Core samples were collected by using a core sampler developed for soft sediments (9). The core tube and sediment were collected by pushing a thin-walled polyvinyl chloride (PVC) pipe, with 10 cm diameter, into the sediment to a depth of 70 cm. The core tube was extracted from the sediment,

Table 1. Concentrations of Cr, Cu, Pb, Hg, and Zn measured in sediments collected in Bayou d'Inde, southwestern Louisiana^a

Station	Depth, cm ^b	Chromium, mg/kg	Copper, mg/kg	Lead, mg/kg	Mercury, mg/kg	Zinc, mg/kg
D0	4	10 (9.1)	7.6 (7.0)	18 (18)	0.06 (0.07)	12 (14)
	17	12 (9.0)	6.9 (6.4)	14 (20)	0.04 (0.04)	15 (14)
	29	14 (14)	8.0 (8.1)	15 (16)	0.05 (0.03)	17 (16)
D1	6	76 (70)	360 (360)	67 (64)	2.2 (2.7)	170 (160)
	29	98 (110)	310 (340)	120 (140)	1.9 (2.0)	150 (210)
	50	400 (360)	380 (390)	160 (110)	5.7 (6.2)	400 (310)
D1A	4	36 (36)	120 (120)	68 (73)	4.3 (3.5)	200 (210)
	24	45 (46)	110 (120)	71 (68)	3.9 (3.5)	180 (190)
	37	50 (47)	130 (130)	69 (68)	4.8 (4.0)	220 (220)
	50	65 (62)	150 (180)	80 (79)	—	210 (200)
D1B	6	32 (33)	110 (110)	89 (110)	5.3 (4.5)	160 (150)

^a Each value in parentheses is the concentration measured in a duplicate sediment sample taken from the same sample bag as the original sample.

^b Depth of the core segment analyzed.

capped on each end, and labeled by location, core number, date, and time. The collected sediment was then stored in the upright tube until it was sent to the laboratory. The sediment was then extruded from the core tube and sectioned into 2.54 cm lengths. The core segments labeled by core number and depth were weighed and then dried at 95 °C for 48 h or until completely dry. The segments were then reweighed to determine moisture content, ground to a powder that passed a 20 mesh sieve, and stored in pre-labeled plastic bags.

Sample Digestion

The digestion method developed earlier (10) was used as modified (11–13). Each sample of 0.2 g sediment was weighed to 0.1 mg in a Teflon digestion vessel. A 5 mL portion of American Chemical Society (ACS) certified TraceMetal grade concentrated nitric acid was added to the sample, and the bomb was sealed. The bomb was then placed in a microwave oven for heating for 1 min. The bomb was allowed to cool and opened and, if the sample contained significant organic material, hydrogen peroxide was added and the microwave heating was repeated. After cooling, the sample was filtered through Whatman No. 40 filter paper, and the filtrate was diluted to 25 mL with deionized water.

Sample Analysis

The concentrations of metals in digested sediment samples were determined by using a Model 5000 flame atomic absorption spectrometer (Perkin-Elmer, Inc., Norwalk, CT) under basic conditions given in the Perkin-Elmer procedures manual (14). The following flame atomic absorption methods of the U.S. Environmental Protection Agency (EPA; 15) were closely followed: 218.1 (Cr), 220.1 (Cu), and 289.1 (Zn). Mercury was determined by the cold vapor method (16) that corresponds to EPA Method 7471A (15). A Model 50B analyzer system (Bacharach, Inc., Pittsburgh, PA) was used to de-

termine the concentration of Hg in the samples. Lead was determined by using a Model 372 graphite furnace atomic absorption spectrometer (Perkin-Elmer, Inc.) and EPA Method 239.2 (15).

Because bomb digestion methods did not completely dissolve the silica matrix, it was necessary to determine recovery rates for the analytes. The recovery rates were measured for a Standard Reference Material (SRM; National Institute of Science and Technology [NIST] Rocky Flats Soil No. 1, SRM 4353). The recovery rates were determined to be 90% for Zn, 80% for Cu, 70% for Cr and Pb, and 100% for Hg. Recoveries were found to vary by less than 5% between 5 replicate determinations. Recovery rates were found to be relatively constant and thus would not be expected to affect observed variations in metal concentrations with sediment depth.

A duplicate sediment sample was analyzed for each set of 10 samples. The results of the duplicate sample analyses are presented in Table 1. Each number in parentheses is the value for the duplicate sample analysis, and depth refers to the depth of the core segment analyzed. The segments were selected randomly for each core sample analyzed. There is general agreement between the metal concentrations found for each sample and its duplicate, with most differing by less than 15%. The accuracy, precision, and reproducibility of the analytical method were established previously (1, 3) by using SRMs and statistical data analyses. Duplicate sample analyses and sample and method blanks were used to ensure that the analytical procedures were operating properly. No metals were detected in any of the blank samples.

Results

Results for the determination of metals in core samples are presented in Figure 2 for Cr, Cu, Pb, and Zn and in Figure 3 for Hg. The results are plotted as concentration (in mg/kg) in a core segment versus collection depth. The following results

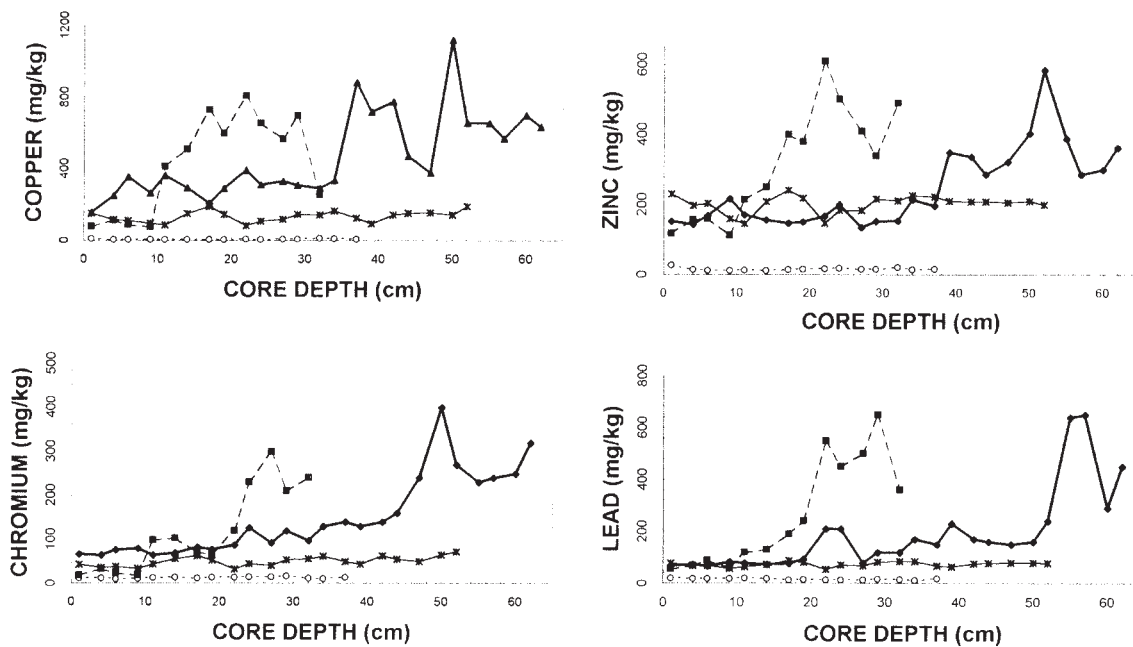


Figure 2. Variations in concentration (mg/kg) of Cr, Cu, Pb, and Zn with sediment core depth at the 4 sampling stations. The lines plotted represent data collected at the following sampling stations: D0 (dashed line with open circles), D1 (solid line with closed diamonds), D1A (solid line with stars), and D1B (dashed line with closed squares).

compare metal distributions with depth at sample stations D0, D1, D1A, and D1B.

Chromium

Peak Cr concentrations were found at stations D1 and D1B (Figure 2). At D1, peak concentrations were observed at a depth of 35–40 cm and continued to be high to the maximum depth of the core at 62 cm. The concentration of Cr peaked at 425 mg/kg at a depth of 49 cm. In contrast, Cr began to increase in sediments at D1B at a depth of 20 cm and remained high at the maximum core depth of 34 cm. The pattern of variation was similar at both stations. Cores taken at the remaining stations did not show evidence of peak concentrations in Cr. At both stations, metal concentrations remained essentially constant with depth, but Cr concentrations at D1A were found to be about 5 times higher than those measured at D0.

Copper

Peak Cu concentrations were also found at stations D1 and D1B (Figure 2). At D1, peak concentrations were observed at depths of 35–40 cm and at 49 cm, and continued to be high to the maximum depth of the core at 62 cm. The concentration of Cu peaked at 1125 mg/kg at a depth of 49 cm. In contrast, Cu began to increase in sediments at D1B at a depth of 10 cm, remained high to a depth of 30 cm, and then began to decrease. Peak concentrations remained high for 20 cm of core length. Cores taken at the remaining stations did not show evidence of peak concentrations in Cu, with the concentrations remaining essentially constant with depth. The concentration of Cu at station D1A was greater by a factor of 10 than the concentration at D0.

Zinc

Peak Zn concentrations were found at stations D1 and D1B (Figure 2). At D1, peak concentrations were observed at depths of 35–40 cm and at a depth of 50 cm, and continued to be high to the maximum depth of the core at 62 cm. The concentration of Zn peaked at 570 mg/kg at a depth of 50 cm. In contrast, Zn began to increase in sediments at D1B at a depth of 10 cm and remained high to a depth of 32 cm, peaking in concentration at a depth of 21 cm. Peak concentrations remained high for about 25 cm of core length. Cores taken at the remaining stations did not show evidence of peak concentrations in Zn, with the concentrations remaining essentially constant with depth. The concentration of Zn at station D1A was greater by a factor of 10 than the concentration at D0.

Lead

Peak Pb concentrations were also found at stations D1 and D1B (Figure 2). At D1, peak concentrations were observed at depths of 50 cm and continued to be high to the maximum depth of the core at 62 cm. The concentration of Pb peaked at 660 mg/kg at a depth of 56 cm. In contrast, Pb began to increase in the sediment core at D1B at a depth of 15 cm, remained high to a depth of 30 cm, and then began to decrease. Peak concentrations remained high at both stations for more than 10 cm of core length. Cores taken at the remaining stations did not show evidence of peak concentrations in Pb, with concentrations remaining essentially constant with depth. The concentration of Pb at station D1A was greater by a factor of 5 than the concentration at D0.

Mercury

Peak Hg concentrations were also found at stations D1 and D1B (Figure 3). At D1, peak concentrations were observed to occur at depths of 55 cm and continued to be high to the maximum depth of the core. The concentration of Hg peaked at 10 mg/kg at a depth of 56 cm. In contrast, Hg began to increase in sediments at D1B at a depth of 8 cm and remained high to a depth of 32 cm. Peak concentrations remained high for 20 cm of core length at D1B and about 10 cm of core length at D1. Cores taken at the remaining stations did not show evidence of peak concentrations in Hg, with the concentrations remaining essentially constant with depth. The concentration of Hg at station D1A was greater by a factor of 40 than the concentration at D0.

Discussion

It is evident from Figures 2 and 3 that concentrations of metals in sediment cores collected at the mouth of the bayou were low throughout the core sample. In fact, the concentrations measured at D0 were similar to those established as baseline values for the estuary as a whole (1, 2). However, slightly higher concentrations of Hg (0.3 mg/kg), compared with values found in the remainder of the core (0.1 mg/kg), were found in core segments at 2 and 29 cm. These findings may represent events that carried Hg-laden sediments to the mouth of the bayou, but they could also represent natural variations in sediment loading or analyses.

The highest concentrations of all metals were found in and around the drainage canal, as demonstrated by the peak concentrations at D1 and D1B. The similarity in the patterns of the variations in metal concentration with depth at stations D1 and D1B is striking, although the patterns differ by several centimeters of depth. These differences may suggest that the metals were introduced at these stations at similar rates and concentrations but over different periods of time (1). The differences in the concentration profiles may also be due to the fact that core samples were taken at D1 on the inside of a bend in the bayou. The sample site originally selected was directly across the bayou. The location was changed because construction of a bulwark on the outside of the bend had disturbed the sediments. Shoaling on the inside of the bend may have buried contaminated sediments. Peak concentrations of the metals were 400 mg/kg for Cr, 1100 mg/kg for Cu, 10 mg/kg for Hg, 600 mg/kg for Pb, and 600 mg/kg for Zn. The high values determined agree well with the high concentrations previously found (1–4).

It is noteworthy that each metal sampled at station D1A gave a generally constant concentration with depth. These concentrations were significantly higher than the values measured at D0 and were similar to those found near the surface at stations D1 and D1B. It appears that the area within about 1 km of station D1 was uniformly contaminated up to the present time. This contamination may have resulted from the construction at D1 and the resulting mobilization and displacement of contaminated sediments.

Figures 4 and 5 compare the distributions of the concentrations of Cr, Cu, Pb, Zn, and Hg in core samples taken in 1983–1984 (1) at sampling station D1 and in 1995 at D1 and D1B. Figure 4 shows that the distributions of 4 metals have similar trends in metal concentrations, except for variations in the depth at which increases in metal concentrations lead to peak concentrations. Previous sampling (dashed line) reveals a peak in concentration for Cr beginning at about 6 cm of depth, which contrasts with the peak concentrations at D1B and D1 in 1995 at depths of 20 and about 50 cm, respectively. The depth of 6 cm was determined by radiochronological dating to correspond to the year 1975, which suggested sedimentation rates of about 0.75 cm/yr (1). If the depth of 20 cm measured in the current study at D1B also corresponds to the year 1975, then about 14 cm of sediment has been added since 1983. This would correspond to a sedimentation rate of 1.2 cm/yr, somewhat higher than reported earlier. The sedimentation rate at D1, by the same analogy, would be significantly higher (3.2 cm/yr).

As previously stated, construction of a bulwark at D1 at the time of sampling may have redistributed contaminated sediment along the bayou. This redistribution would be expected to result in significant increases in sedimentation rates at this location. This activity may not have affected D1B because of its location upstream, within the drainage canal. Peak concentrations of Cr varied from about 600 mg/kg (1983) to about 400 mg/kg (1995); however, this trend was reversed for the other metals. In each case, the highest concentrations were found in cores taken in 1995, suggesting sample site differences in metal contamination. Calculating estimates of sedimentation rates in the manner described above by using the vertical distribution of Cu, Pb, and Zn in cores yields lower sedimentation rates at D1B, ranging from 0.67 (Zn) to

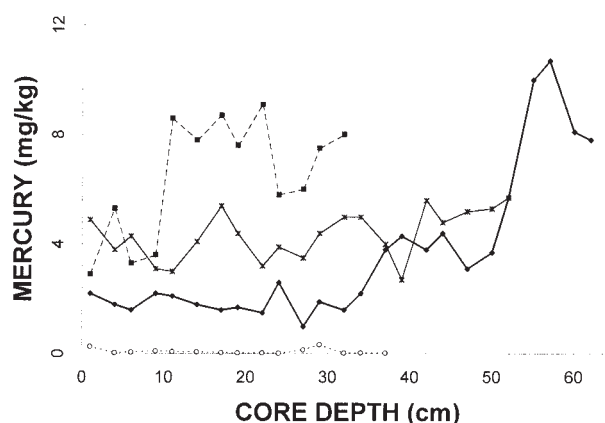


Figure 3. Variations in concentration (mg/kg) of Hg with sediment core depth at the 4 sampling stations. The lines plotted represent data collected at the following sampling stations: D0 (dashed line with open circles), D1 (solid line with closed diamonds), D1A (solid line with stars), and D1B (dashed line with closed squares).

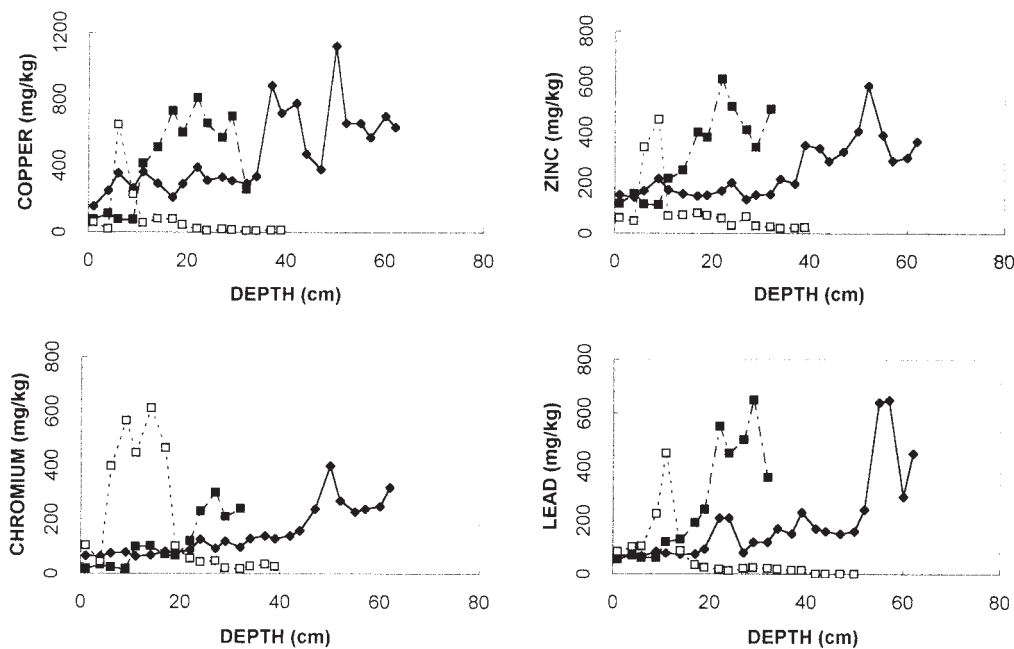


Figure 4. Variations in concentration (mg/kg) of Cr, Cu, Pb, and Zn with sediment core depth at the 4 sampling stations. The dashed line with open squares represents data collected in 1983–1984 at station D1. The dashed line with closed squares represents sampling station D1B, and the solid line with closed diamonds represents station D1, for samples collected in 1995.

0.83 cm/yr (Cu, Pb). These rates agree well with previous estimates (1) but cannot be taken as definitive sedimentation rates.

The distribution of mercury with respect to depth is presented in Figure 5 for station D1 as determined in 1983–1984 and for stations D1 and D1B as determined in 1995. The observed variation at each station is similar to those for previously described metal distributions with respect to depth. The

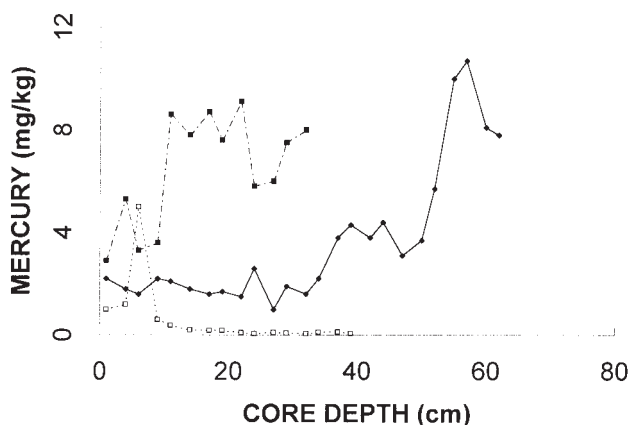


Figure 5. Variations in concentration (mg/kg) of Hg with sediment core depth at the 4 sampling stations. The dashed line with open squares represents data collected in 1983–1984 at station D1. The dashed line with closed squares represents sampling station D1B, and the solid line with closed diamonds represents station D1, for samples collected in 1995.

onset of peak Hg concentrations at D1 occurred at about 4 cm in cores taken in 1983–1984 and at about 40 cm in cores taken in 1995, whereas the beginning of the peak concentration at D1B is at about 12 cm. With the assumption that core samples collected at D1 in 1983–1984 and at D1B represent undisturbed sediments, a sedimentation rate of 0.67 cm/yr would be estimated. The highest concentrations of Hg measured at these stations would be about 5 mg/kg in 1983–1984 at D1 and, during the current study, 9 mg/kg at D1B and 10 mg/kg at D1. This variation in concentration by a factor of 2 may suggest that contamination is patchy across the sampling area.

The highest concentrations of metals occurred in the vicinity of the drainage canal, which carries cooling water with temperatures that are 5–10 C higher than those of ambient bayou water (3). It may be possible that metals carried to this area of the bayou from upstream encounter the warmer, sometimes more saline, water and precipitate there. The fact that high concentrations of metals are found in the drainage canal suggests that significant amounts of these metals were once carried with the cooling waters and precipitated within or near the drainage canal.

Mean concentrations of the metals studied were calculated from the concentrations of individual metals in all core segments. These mean concentrations of metals are presented in Figure 6 for each sampling station. It is obvious from Figure 6 that the concentrations of metals were highest at sampling stations D1 and D1B, decreased rapidly to station D1A, and then became essentially characteristic of the uncontaminated estuary at the mouth of the bayou. The concentration of Hg remained essentially constant at all stations except D0, where the concentrations became similar to values found across the

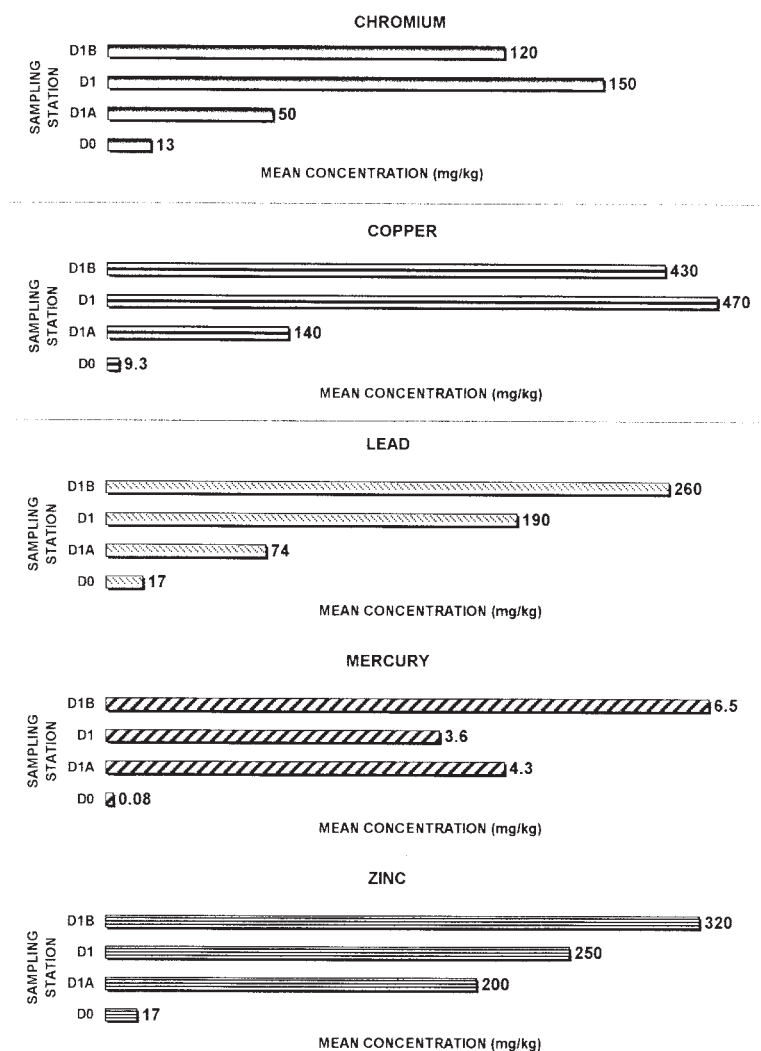


Figure 6. A histogram of mean metal concentrations determined for the entire core collected at each of the 4 sampling stations (D1B, D1, D1A, and D0) showing the decrease in metal concentration from D1 to the mouth of the bayou. Mean metal concentrations (mg/kg) are given at the right of each bar.

state of Louisiana (17, 18) and the Calcasieu estuary as a whole (19). It is clear that metal contamination has remained highly localized within the bayou as reported earlier (1) and has not severely affected sediments near the mouth of the bayou. However, it appears that metal contamination of sediments has continued to be a problem within the bayou, as evidenced by elevated metal concentrations at the surface at sampling stations within 1 km of the drainage canal. There is evidence that construction near the canal may have redistributed possibly contaminated sediments to nearby areas and thereby contributed to the high concentrations of metals at the sediment surface. It may be possible to confirm this in later sampling, after a few more years have passed.

Conclusions

Sediments in Bayou d'Inde in southwestern Louisiana were reported as contaminated by the metals studied, and this

problem was confirmed in the present study. The highest concentrations found in Bayou d'Inde sediments were 400 mg/kg for Cr, 1100 mg/kg for Cu, 10 mg/kg for Hg, and 600 mg/kg for Pb and Zn. These metal concentrations agree well with those found in previous studies of sediments collected in the bayou. The high concentrations of metals measured about 1 km from the mouth of the bayou have not significantly affected metal concentrations found in sediment samples collected there. In fact, metal concentrations in sediments collected near the mouth of the bayou yielded values that are considered baseline concentrations for the Calcasieu estuary.

The variations in depth within the core samples for the 2 sampling periods for which peak metal concentrations were found were compared to allow an estimate of the amount of sediment deposited between 1983–1984 and 1995. Recent sampling revealed that peak concentrations are currently located at a greater depth in the core, as expected. Sedimentation

rates for the bayou were reported to range from 0.7 to 1.0 cm/yr (1). Comparing the depth to the onset of peak concentrations in the core profiles determined in the 2 sampling periods allowed us to estimate sedimentation rates over the 12 years since the initial study. Sedimentation rates were estimated to range from 0.67 to 1.2 cm/yr.

Acknowledgments

We thank the Louisiana Educational Quality Support Fund (LEQSF; Baton Rouge, LA) for financial support under its graduate fellowship program. This work was also supported, in part, by EPA Grant R-824-143-01-00.

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