

**Historical Impact of Industrial Development on Groundwater and Surface Water  
Quality in the American Bottoms**

**Progress Report**

**Year 2**

July 15, 2002 to July 15, 2003

Illinois Groundwater Consortium

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**Abstract**

Industrial development over the last 110 years has contaminated many parts of the American Bottoms, an extensive floodplain of the Mississippi River just east of St. Louis, MO. Water resources in this region have been severely impacted by long-term mismanagement of hazardous waste disposal by local industries. A record of metal contamination exists in the sediment of Horseshoe Lake, a 3,000 year-old natural oxbow lake in the most industrialized portion of the American Bottoms. We examined 4 dated sediment cores and 84 surface sediment samples from Horseshoe Lake to reconstruct the historical record of environmental contamination. Pb, Cd, and Zn concentrations increased in the sediment after the 1940's. Stable isotopes of Pb show that this increase is due to anthropogenic contamination. Sediment with the highest concentrations of lead are located in the SW basin of the lake near the NL Industries site, a potential source of contamination. We used isotopes of nitrogen to track the history of sewage contamination finding that sediment  $\delta^{15}\text{N}$  increased to values  $> 10 \text{ ‰}$  in the 1920's. Because such high values of  $\delta^{15}\text{N}$  are only associated with the presence of human or animal wastes, we deduce that major contamination of the lake by sewage began at that time. Increases in biogenic  $\text{CaCO}_3$  and in  $\delta^{13}\text{C}$  indicate that this input of waste raised the trophic status of the lake. Our results provide a physical record of contamination that is consistent with Colten's (1988) description of hazardous waste disposal in the American Bottoms.

## Introduction

The American Bottoms is a floodplain of the Mississippi River just east of St. Louis, MO, in Illinois. Because of cheap transportation, water and fuel, the American Bottoms has been a favored location for development since the beginning of the industrial revolution, however, with industrial development came environmental pollution. Colten (1988, 1990) documented the historical record of environmental contamination in the American Bottoms. He reports that during the early part of the 20<sup>th</sup> century industrial waste was simply poured on the ground, released into local waterways, or stored permanently on-site.

Lake sediments from Horseshoe Lake, a large, 3,000 year-old (Ollendorf 1993) oxbow lake in the American Bottoms, hold a physical record of environmental pollution that can supplement the historical record described by Colten (1988, 1990). Heavy metals are strongly adsorbed to sediment particles allowing the sediment column to retain a record of past pollution (von Gunten *et al.* 1997; Smol 2002). Investigators around the world have found increased concentrations of heavy metals in recently deposited sediment from many lakes indicating widespread environmental contamination from industrial processes (Johansson, 1989; Norton *et al.* 1992; Graney *et al.* 1995; Boyle *et al.* 1997; Brännvall *et al.* 2001).

Isotopes of carbon and nitrogen can be used to reconstruct the history of contamination by sewage which can result in the eutrophication of lakes. Increasing contamination with human and animal waste results in enrichment in <sup>15</sup>N, the heavy isotope of nitrogen (Roadcap *et al.* 2001). As trophic status increases because of increased nutrient inputs, the <sup>13</sup>C content of organic matter also increases (Hodell *et al.* 1998).

Because different lead (Pb) ore bodies have different isotopic "signatures", changes in stable isotopes of Pb can also be used to indicate contamination from identifiable pollution sources. Human inputs of Pb mined outside the lake watershed change the isotopic ratio of the Pb sequestered in lake sediment. (Graney *et al.* 1995; Brännvall *et al.* 1999; Marcantonio *et al.* 2000; Brännvall *et al.* 2001).

Our goal is to reconstruct the history of environmental pollution in the American Bottoms using the sedimentary record from Horseshoe Lake (Fig. 1).

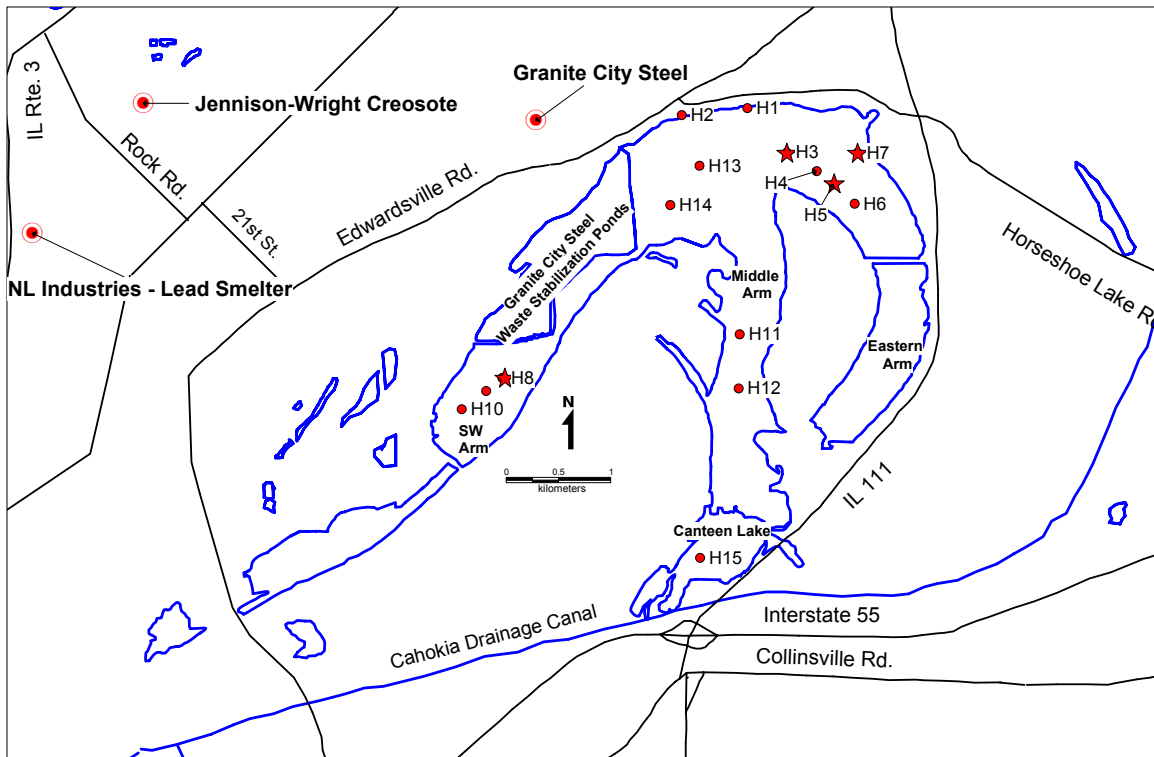


Figure 1: Map of Horseshoe Lake showing major industries. Core sites are shown with red circles. The cores discussed in this report are indicated by red stars. Industrial sites are shown with red bull's eyes. Water bodies are outlined in blue.

### History of Industrial Development in the Horseshoe Lake Watershed

The first permanent European-American homestead on Horseshoe Lake was established in 1808 by Jacques Germain (Anonymous 1882). Industrial development began in 1866 with the relocation of St. Louis Stamping Company to Granite City, IL. Granite City Steel was established on the northwest shore of the Horseshoe Lake in 1895 (Crayne 1999). The steel company grew into a large integrated steel mill, which included a coking facility. In 1903 National Lead Company built a Pb smelter about 5 km from the western shore of the lake. The National Lead smelter refined new Pb from ore and recycled old Pb from automobile batteries. The company eventually accumulated a 250,000 ton pile of Pb waste at this site (Bosworth 1982). In 1920, Jennison-Wright Corporation began treating railroad ties in Granite City using creosote from the Granite City Steel coking process.

National Lead closed its Granite City facility in 1983 (USEPA 2003a). By that time Pb from the National Lead site contaminated nearby groundwater wells. Twenty-five percent of the children living near the plant had  $>10 \mu\text{g/dL}$  of Pb in their blood and numerous residential sites were contaminated with Pb. As part of a superfund clean up, at the National Lead site, the Pb waste pile was capped and sealed. Sixteen hundred homes were decontaminated (USEPA 2003a). The clean-up was complete by 2000. Jennison-Wright ceased operation in 1990 (USEPA 2003b), but the site has not yet been fully decontaminated (Lambrecht 2002).

## Methods

Fifteen sediment cores were taken from Horseshoe Lake using a 7.5 cm diameter polycarbonate tube attached to vanadium steel rods (Fig 1). Coring was done manually from a boat (Wright 1980). Cores were taken to the laboratory where they were extruded vertically and sliced into 1 cm intervals. Each centimeter was stored in 4 oz. Whirl-Pak<sup>®</sup> plastic bags at 4°C.

In addition to the cores, 84 surface sediment samples were taken from the lake using an Ekman Dredge. Samples were located at 0.1 mi. (0.16 km) intervals across the lake. Sample locations were determined using a geographic positioning system. Small aliquots of sediment were removed from the dredge and stored in 4 oz. Whirl-Pak<sup>®</sup> bags at 4°C until analyzed.

The St. Croix Watershed Research Center in Marine-on-St. Croix, MN dated cores H3 and H8 using the polonium alpha method (Eakins and Morrison 1978) for <sup>210</sup>Pb. Cores H5 and H7 were dated at Southern Illinois University Edwardsville using the bismuth ingrowth method for <sup>210</sup>Pb (Krishnaswami *et al.* 1971).

Samples from cores H3 and H5 for metals analysis were prepared by grinding with an alumina mortar and pestle followed by borate fusion. Metals were analyzed using X-ray Fluorescence Analysis at the Department of Earth and Planetary Sciences, Washington University, St. Louis, MO. (Brugam *et al.* In Press). Surface sediment samples and samples from cores H7 and H8 were dried and sent to Activation Laboratories, Ancaster, Ontario for metals analysis using ICP-MS.

Samples for carbon and nitrogen stable isotope analysis were first dried and then ground in a Spex Certiprep<sup>®</sup> (6750 Freezer Mill) cryogenic impact grinder cooled by liquid nitrogen. The entire grinding process took about three minutes for each sample. After the samples were ground in the Freezer Mill they were transferred into 2.0 ml microcentrifuge tubes with attached O-Ring Seal Caps. The microcentrifuge tubes were sealed and sent to CoBSIL (Cornell-Boyce Thompson Stable Isotope Laboratory, Ithaca, NY) for isotopic analysis. There was no pre-treatment of samples before analysis.

For analysis of stable Pb isotopes approximately 0.5 cm<sup>3</sup> of sediment was dried overnight. The samples were then refluxed overnight using 25ml of H<sub>2</sub>O with 5 ml of HCl and HNO<sub>3</sub>. After refluxing, 1 ml of 30 % H<sub>2</sub>O<sub>2</sub> was added and the samples were evaporated to dryness. The samples were brought back into solution using 5 ml HNO<sub>3</sub> and approximately 25 ml of H<sub>2</sub>O. Residues present in the solution were filtered using Whatman P5 filters. The filtrate was collected and diluted to 50 ml.

Principal Components Analysis (PCA) included all core levels in which we analyzed sediment chemistry. PCA was completed using the computer program, PCord<sup>®</sup> v. 4.0.

## Results

Although 57 chemical elements were analyzed in the sediment cores and surface samples only those of special interest are examined here. We used PCA to estimate the similarity among samples from the cores to discover statistical relationships among chemical elements. Factor scores from the analysis were used to group samples with similar chemical characteristics (Fig. 2). Eigenvectors reveal chemical elements that vary together in concentration (Fig 3).

PCA produces two large groupings of samples (Fig. 2). Samples older than 1939 cluster to the right on the factor scores diagram and samples younger than that date cluster to the left of the Factor 1 axis showing that there is a major change in sediment composition around 1939.

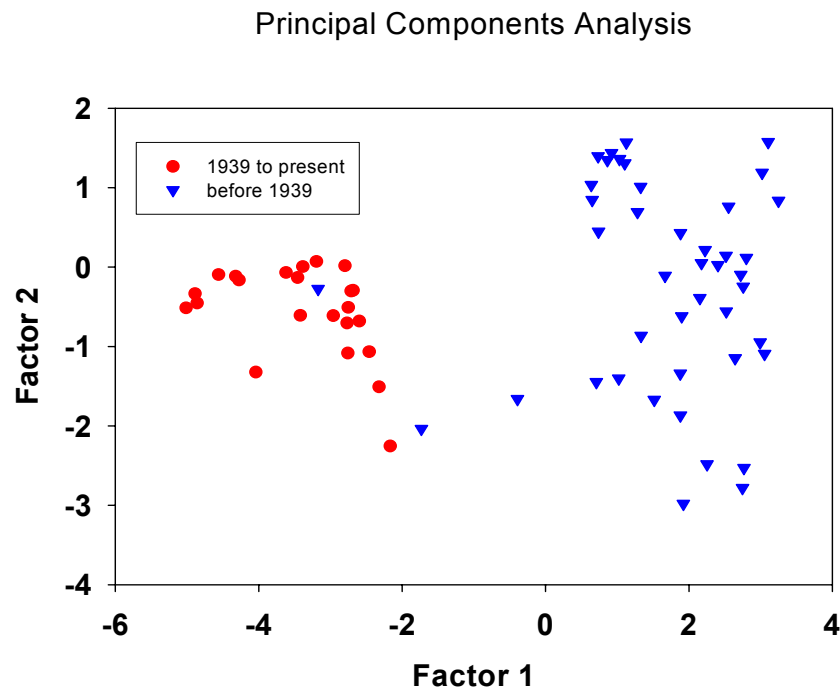


Figure 2: Principal Components Analysis (PCA) of sediment core data from Horseshoe lake, Madison County, IL. Blue triangles indicate samples dated before 1939.

The eigenvalues of the PCA show which chemical elements cause the distributions of the samples in factor space (Fig. 3). Ca, Pb, Zn, and Cd have negative eigenvectors on Factor 1 and Si, Ti, Al, and Fe have positive eigenvectors. From the PCA results, we can infer that the changes in sediment composition in the most recently deposited sediment result from increases in Ca, Pb, Zn, and Cd, and decreases in Si, Ti, Al, and Fe.

### Eigenvectors of Principal Components Analysis

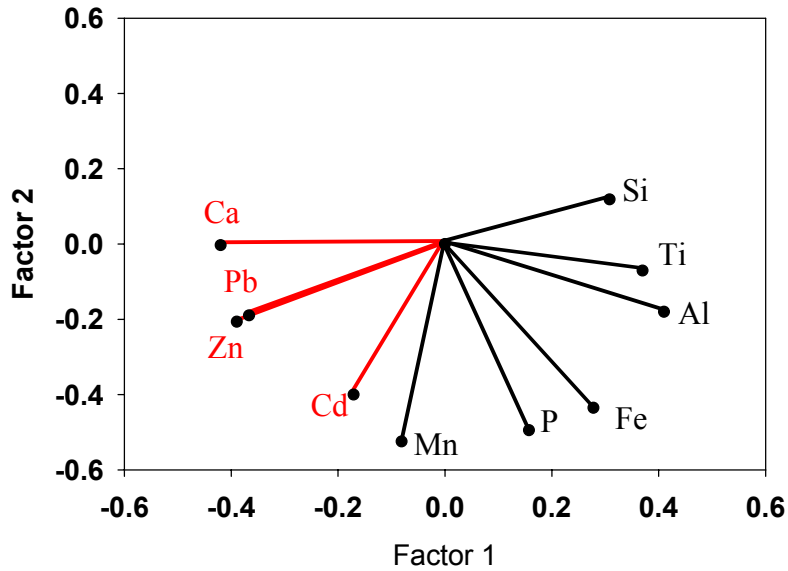


Figure 3: Eigenvectors from the PCA of sediment core data from Horseshoe Lake, Madison County, IL (see Fig. 2). Black lines indicate elements that decrease in recent sediment. Red lines indicate elements that increase in recent sediment.

Major elements are components of sediment present in the gm/kg range. The major elements found in most sediment are Si, Al, Fe, Mn, Ca, Mg, Na, K, Ti, and P. The PCA analysis suggests that there is a shift in major element composition in the more recently deposited sediments of Horseshoe Lake. Si is a representative major element, which declines, in recent sediment (Fig. 4). In contrast with the other major elements, Ca increases in recent sediments (Fig. 5).

### Silicate Stratigraphy

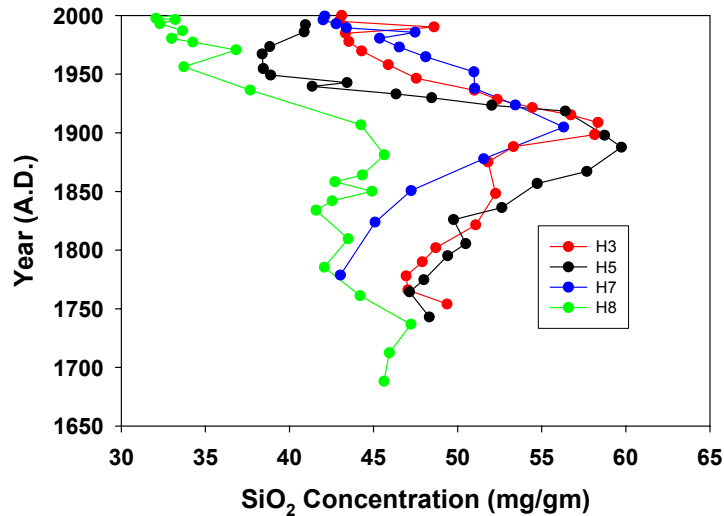


Figure 4: Silicate (Si) in sediment cores from Horseshoe Lake, Madison County, IL.

## Ca Stratigraphy

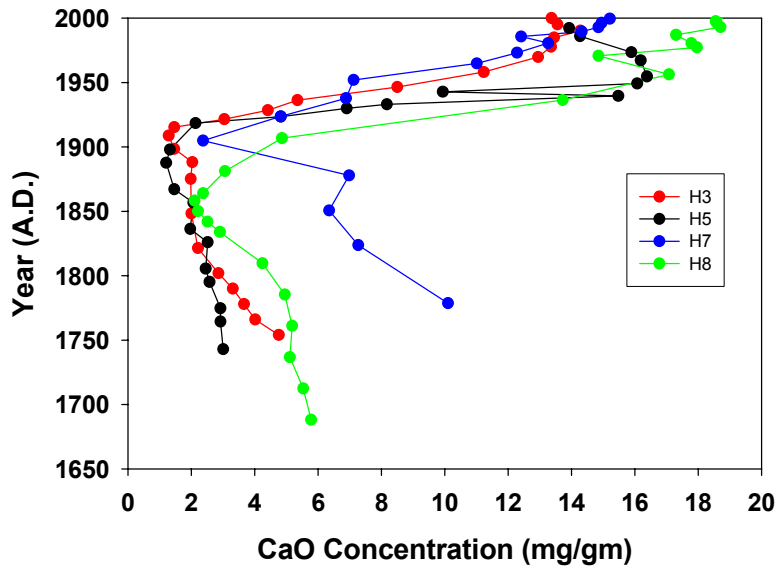


Figure 5: Calcium (Ca) in sediment cores from Horseshoe Lake, Madison County, IL.

Potential contaminants in Horseshoe Lake are Pb and Zn. Both of these elements increase by a factor of 10 after 1939 in all sediment cores (Fig. 6 and 7).

## Pb Stratigraphy

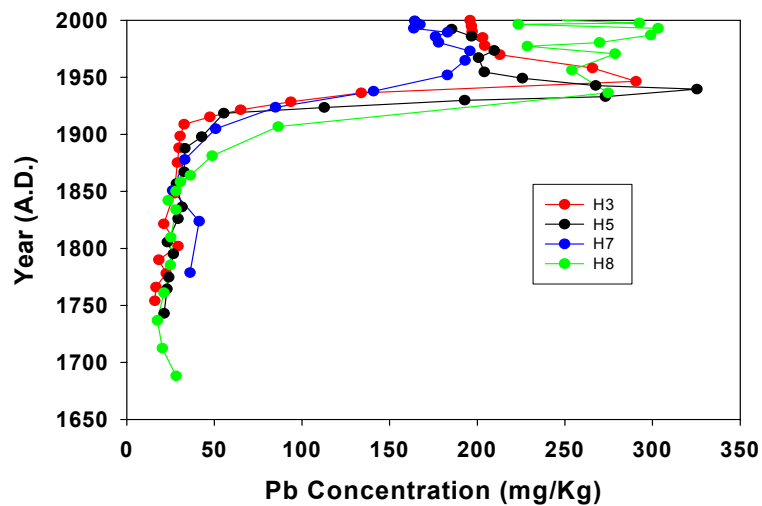


Figure 6: Lead (Pb) in the sediment cores from Horseshoe lake, Madison County, IL.

## Zn Stratigraphy

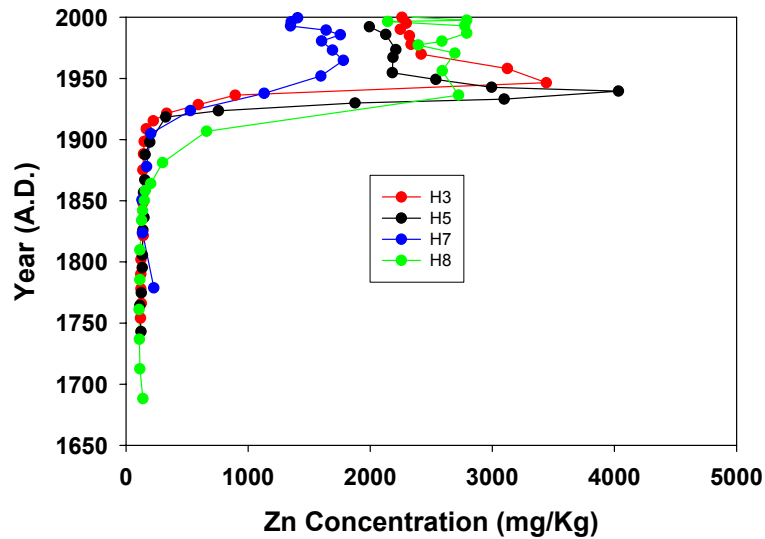


Figure 7: Zinc (Zn) in sediment cores from Horseshoe Lake, Madison County, IL.

Surface sediment concentrations of Pb and Si show significant differences across the lake bottom. Pb concentrations are highest in the southwest corner of the lake (Fig 8). Si concentrations increase in the middle arm of the lake (Fig. 9).

$\delta^{15}\text{N}$  increases in the recent sediment of cores H3, H4, and H5. The rise in  $\delta^{15}\text{N}$  begins about 1920 and continues to the present reaching +10 ‰ in cores H3 and H5 but +16 ‰ in core H8 (Fig 10). Forty three of the surface samples have  $\delta^{15}\text{N}$  greater than +10 ‰. These samples are located in the main basin of the lake and in the southwest arm. The samples with the lowest  $\delta^{15}\text{N}$  are in Canteen Lake and the east arm of the lake (Fig. 11).

$\delta^{13}\text{C}$  increases at the same core levels as  $\delta^{15}\text{N}$  reaching values of -16 ‰ (Fig. 12). The highest values of  $\delta^{13}\text{C}$  occur in the main basin of the lake, but the lowest values are in Canteen Lake and the east arm (Fig. 13).

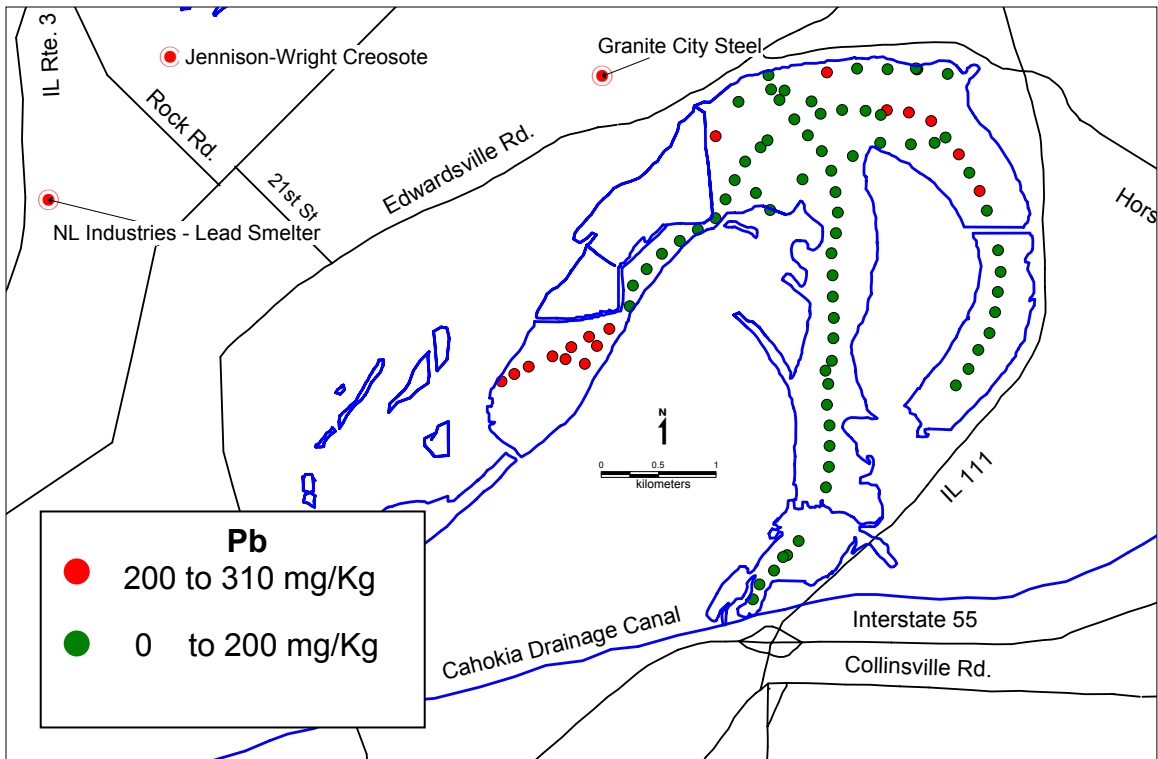


Figure 8: Surface sample Pb concentrations from Horseshoe lake, Madison County, IL.

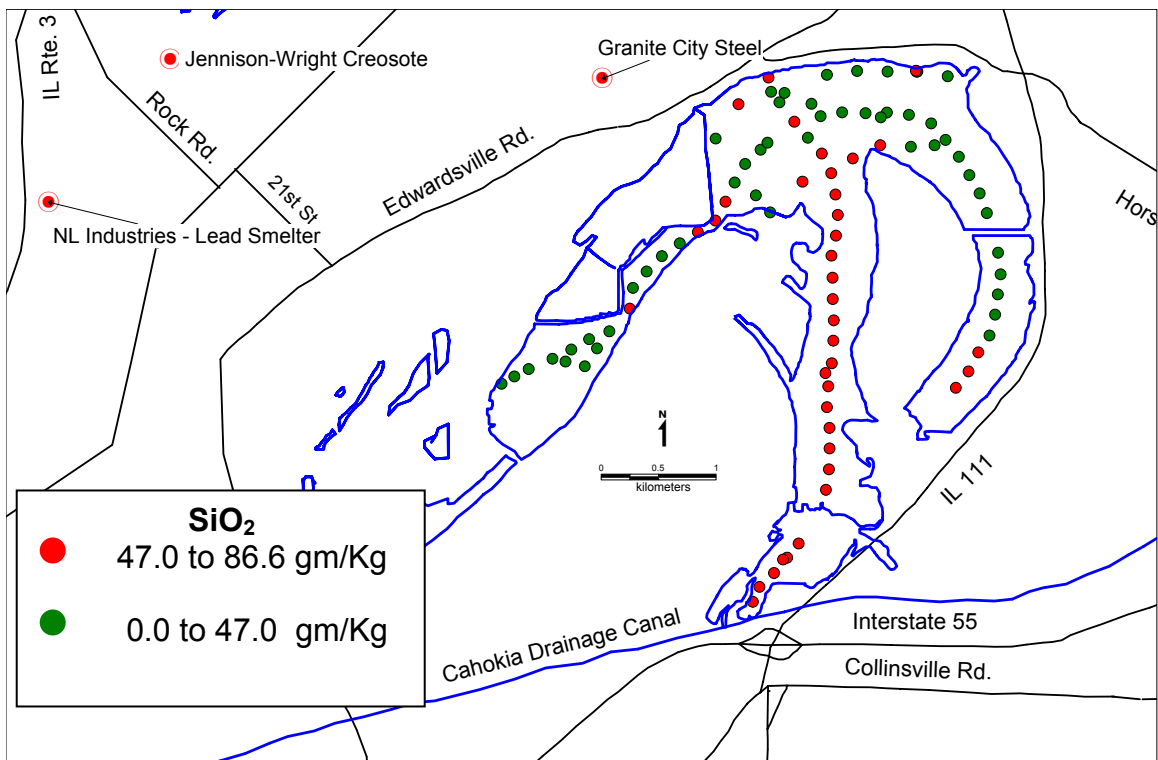


Figure 9: Surface Sample Si concentrations (as gm SiO<sub>2</sub>/Kg) from Horseshoe lake, Madison County, IL.

## Nitrogen Isotopes

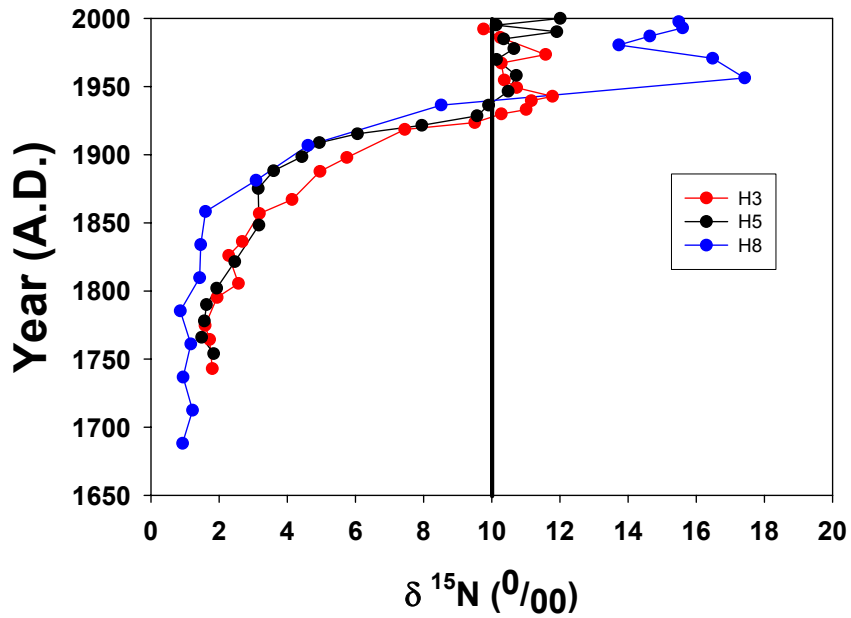


Figure 10:  $\delta^{15}\text{N}$  values in sediment cores from Horseshoe Lake, Madison County, IL. Vertical line indicates  $\delta^{15}\text{N}$  of +10 ‰.

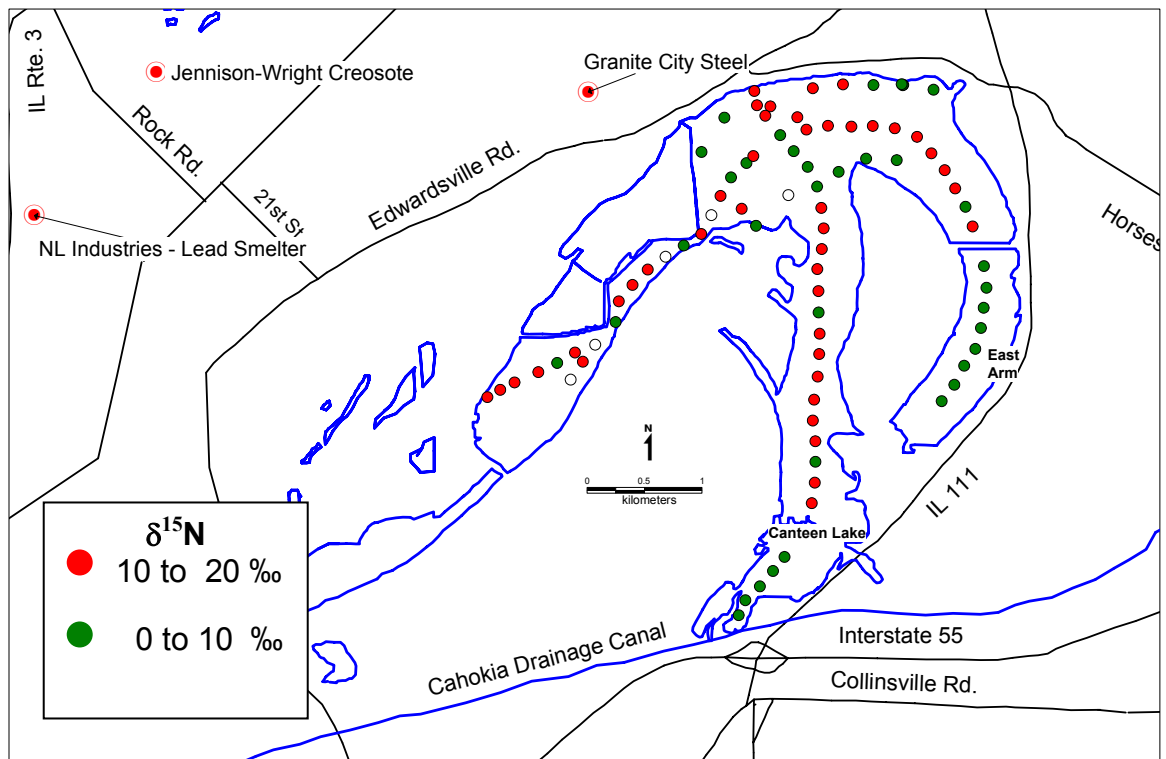


Figure 11:  $\delta^{15}\text{N}$  values in surface sediment samples from Horseshoe Lake Madison County, IL.

# Carbon Isotopes

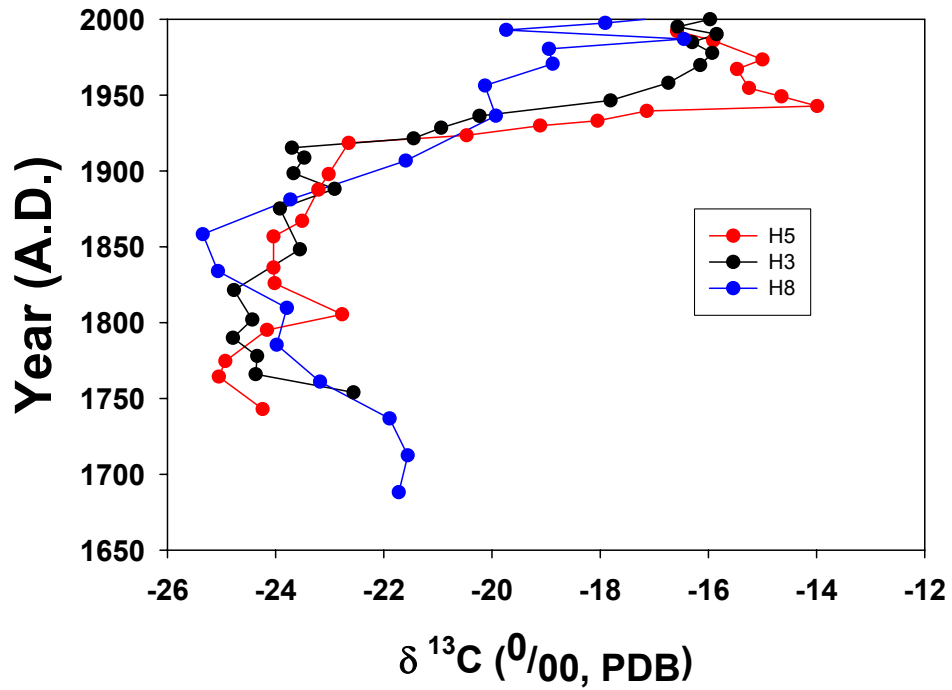


Figure 12:  $\delta^{13}\text{C}$  in sediment cores from Horseshoe Lake, Madison County, IL.

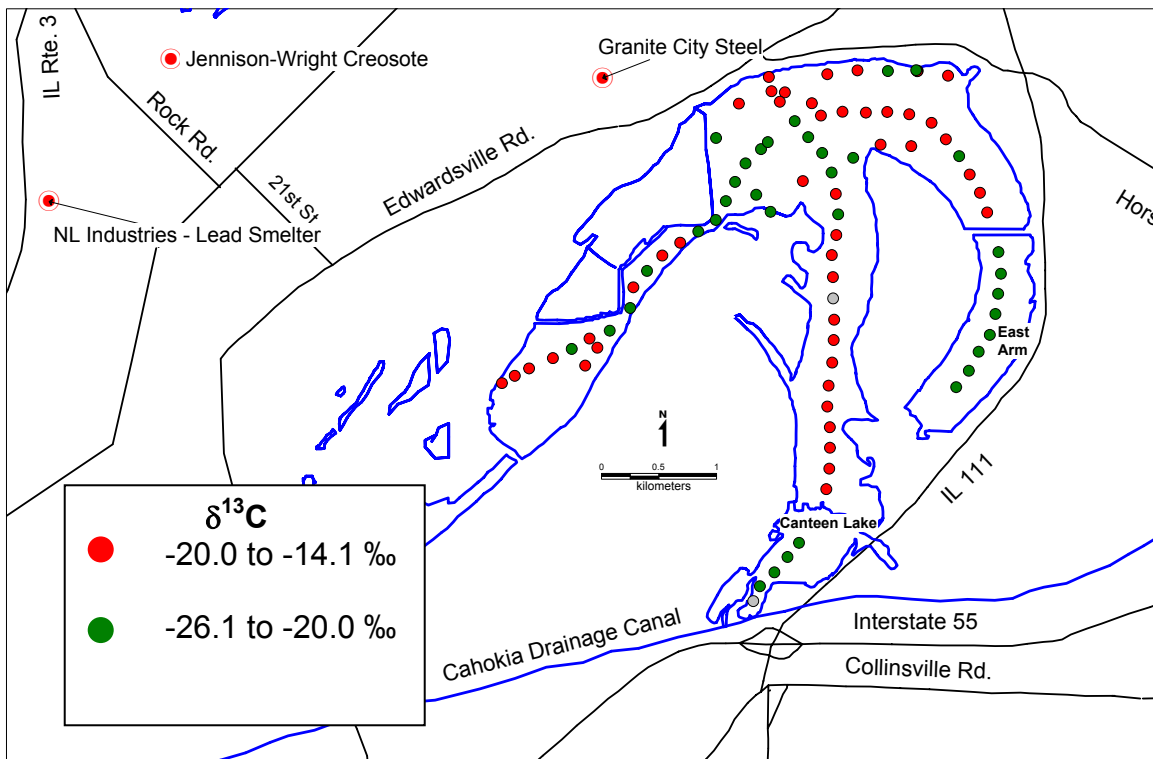


Figure 13:  $\delta^{13}\text{C}$  values in surface sediment samples from Horseshoe Lake

The  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio varies over a broad range in core H8 (Fig. 14). It rises slowly from the bottom of the core reaching a peak of 1.25 % in 1850 and falling abruptly in the 1860's. As the Pb concentration rises the  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio declines Fig 14). The sharpest decline in this period occurs around 1900. The ratio reaches about 1.22 % in the early 1940's.

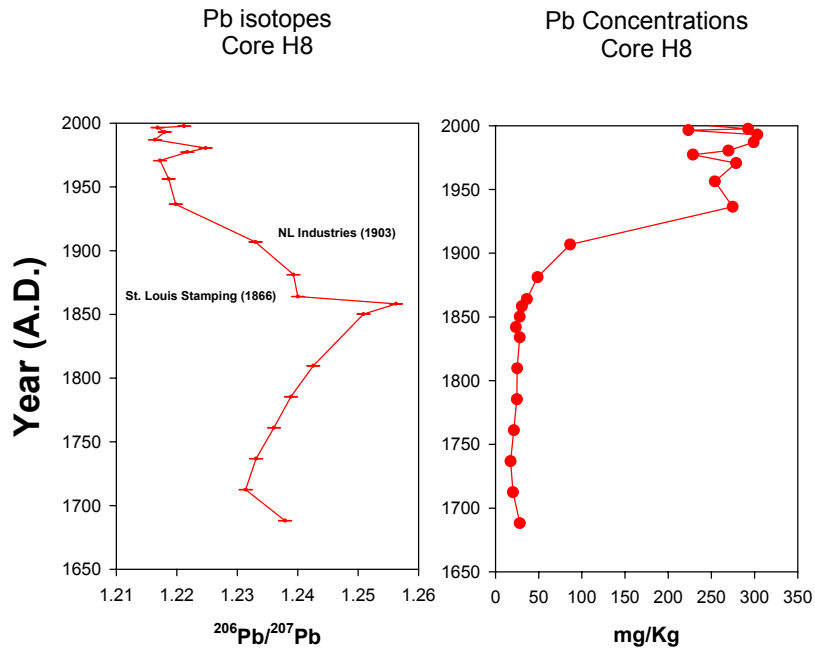


Figure 14: Lead isotopes in core H8 (left panel) from Horseshoe Lake Madison County, IL. Lead concentration in core H8 (right panel).

## Discussion

The sedimentary record of Horseshoe Lake shows clear evidence of contamination from human activities. Ca, Pb, and Zn concentrations increase in the sediment along with changing isotopic ratios of contaminants indicates increasing contamination from industrial activities and sewage pollution.

### Pb Contamination of Horseshoe Lake

PCA shows a shift in sediment composition around 1939 when Pb and Zn concentrations reach their peak. We hypothesize that these metals come from the metal processing industries in Granite City, IL. The shift in the  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio at different levels in the core supports our hypothesis. Each Pb ore body has a particular isotopic composition based on its geologic history (Marcantonio *et al.* 2002). The background concentrations of Pb in a lake's watershed also has a particular isotopic composition (Brännvall *et al.* 1999). If there is no human disturbance of a lake, one might expect that the isotopic ratio of Pb in the accumulating sediment might remain unchanged for a long

period as it does in long sediment cores from Europe (Brännvall *et al.* 1999; Eades *et al.* 2002). However, when humans transport Pb from outside a lake watershed and release it into a lake, they change the isotopic composition of the Pb accumulating in the lake sediment. Core H8 shows strong changes in  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio that seem to correspond to changes in watershed history. The slow rise in the  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio from the bottom of the core to 1850 suggests that there was a low, but increasing contamination of the lake before major industrialization of the watershed. We suggest that this signal represents low-level contamination from colonial French towns in the American bottoms. Cahokia, IL, was founded 17 km southwest of Horseshoe Lake in 1699. Pb mines were established on the west side of the Mississippi River as early as 1720 when Pb deposits were found on the Big and Meremec Rivers just south of St. Louis, MO. Pb was smelted on site and shipped from Cahokia south to New Orleans. The mines closed in 1742 (March 1967). This history suggests thriving local colonial settlements long before Jacques Germain began farming the lake in 1808. In addition to Pb processing, these communities would also have supported simple metalworking industries like black smithing that would likely release metals into the environment.

The abrupt decline in the  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio in the 1860's suggests that the establishment of St. Louis Stamping Co. changed the sources of Pb to the lake. The construction of the National Lead smelter changed the source of Pb again in 1910. The declines in  $^{206}\text{Pb}/^{207}\text{Pb}$  ratio as industrial development proceeded are consistent with the hypothesis that metalworking industries brought Pb from sources outside the Lake watershed inadvertently releasing it to the environment as industrial waste. The Pb isotopes support the hypothesis that most of the rise in Pb in the recent sediment of the lake comes from human input. It is reasonable to suppose that the main source of this Pb was the National Lead smelter, because the highest concentrations of Pb in the surface sediment are found in the southwestern arm of the Lake near the National Lead site. It is likely that the contamination of Horseshoe Lake is part of the broader contamination of Granite City, IL, by the smelter discovered by the U.S. Environmental Protection Agency (USEPA 2003a).

### Eutrophication of Horseshoe Lake

Horseshoe Lake is currently extremely eutrophic. Between 1959 and 1981, there were 14 documented fish kills. The likely cause of these fish kills was deoxygenation under ice during the winter (Hill *et al.* 1981). The sedimentary record can trace the history of eutrophication at the lake.

$^{15}\text{N}$  is an indicator of contamination by human and animal wastes, which are potential causes of eutrophication. Processing of N compounds through animal metabolism raises the  $\delta^{15}\text{N}$  values (Roadcap *et al.* 2001). Values greater than  $+10\text{‰}$  indicate contamination by animal wastes. The sedimentary record of nitrogen isotopes can, therefore, be used as an indicator of sewage pollution. Increases in  $\delta^{15}\text{N}$  values of recent sediment have been found in the Baltic (Voss *et al.* 2000) and in Florida lakes (Brenner *et al.* 1998). In both locations, the increased  $\delta^{15}\text{N}$  values were interpreted to indicate increased nutrient inputs from sewage contamination. All cores analyzed from

Horseshoe Lake show similar increases in  $\delta^{15}\text{N}$  values beginning in the 1920's, which we interpret to indicate sewage contamination. It is unlikely that the contamination results from Granite City because its sewage has always been released into the Mississippi River (Colten 1988). More likely, the increased nutrient inputs come from communities along the eastern edge of the American Bottoms whose wastewater treatment plants discharge into streams feeding the Cahokia Drainage Canal, the main water source for the lake in modern times.

Carbon isotopic ratios provide clear evidence of the effects of nutrient addition to the lake. Normally, phytoplankton discriminate against  $^{13}\text{C}$  during photosynthesis. However, as primary production increases, C limitation becomes important and phytoplankton are unable to discriminate against  $^{13}\text{C}$  (Hodell *et al.* 1998). The result of phytoplankton C limitation will be an increase in  $\delta^{13}\text{C}$  values in the organic matter generated by the phytoplankton and archived in the sedimentary record. Hodell *et al.* (1998) found increases in  $\delta^{13}\text{C}$  values in the recently deposited sediment of Lake Ontario. Brenner *et al.* (1998) found high  $\delta^{13}\text{C}$  values in the recent sediment of Florida lakes, which they explained by recent eutrophication. Voss *et al.* (2000) also found increases in  $\delta^{13}\text{C}$  values in the Baltic Sea, which they attributed to eutrophication.

Our  $\delta^{13}\text{C}$  values from Horseshoe Lake increase at the same time that  $\delta^{15}\text{N}$  increases. Our interpretation is that the  $\delta^{15}\text{N}$  ratio is recording the input of nutrients from sewage and the  $\delta^{13}\text{C}$  is recording the resulting eutrophication. The results from the surface samples are difficult to reconcile with this interpretation. The lowest  $\delta^{15}\text{N}$  and  $\delta^{13}\text{C}$  are found in sediment from Canteen Lake and the eastern arm of the lake. The main input from Cahokia Drainage Canal enters the lake at Canteen Lake. The eastern arm is drained for about 3 months every autumn to allow wetland plants to grow. Both basins are very shallow with lots of terrestrial vegetation so a significant fraction of the organic matter in the sediment may come from terrestrial plants – not carbon limited phytoplankton. In the open basin, the organic matter in the sediment may contain a higher fraction of phytoplankton C allowing it to reflect changes in C availability in the water.

The change in major elements also reflects the eutrophication of the Horseshoe Lake because of biogenic precipitation of  $\text{CaCO}_3$  under conditions of high phytoplankton productivity in hard water lakes (Wetzel 1983; Hodell *et al.* 1998). The increase in Ca in the sediment occurs at the same time our postulated increase in primary production caused by sewage contamination. We hypothesize that the precipitation of  $\text{CaCO}_3$  has been rapid enough to dilute Si, Al, and Ti-containing clay minerals.

The PCA emphasizes the change of sediment composition at 1939. Although Pb, Zn, and Ca are strongly correlated in the lake sediment, they do not increase at exactly the same time. Ca increases with  $\delta^{15}\text{N}$  and  $\delta^{13}\text{C}$  in the 1920's while Pb and Zn begin their increase in the early 1900's. Both reach their maximums in the late 1930's and early 1940's.

## Conclusions

The sedimentary record of Horseshoe Lake, Madison County, Illinois shows strong impacts of watershed development on the lake. Contamination of the lake with heavy metals probably began even in the earliest colonial times, but reached its maximum during the period when the National Lead smelter was in operation. A separate source of contamination was sewage effluents from local communities, which started in the 1920's and caused an increase in primary production in the lake, which may have resulted in the precipitation of CaCO<sub>3</sub> and fish kills.

## Acknowledgements

We thank the Illinois Groundwater Consortium, the Department of Biological Sciences, the Environmental Sciences Program, and the Graduate School of Southern Illinois University Edwardsville for support. We thank Christopher Hamm for preparing <sup>210</sup>Pb samples for counting and for drawing maps. We thank the Illinois Department of Natural Resources and the American Bottoms Conservancy for permission to work on the site.

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