

CONCENTRATIONS OF INORGANIC AND ORGANIC CONTAMINANTS IN SEDIMENTS OF  
SIX HARBORS ON THE NORTH AMERICAN GREAT LAKES

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#### ABSTRACT

Samples of surficial sediments were collected from six different harbors on the North American Great Lakes in May of 1989. Concentrations of 12 metals, 17 pesticides, total PCBs, chlorinated naphthalenes, cyanide and total and unionized ammonia were determined. The sediments were also characterized for moisture content, particle size, total organic carbon content, pH, COD, total solids, total volatile solids.

**Keywords:** Great Lakes, Sediments, Metals, Pesticides, PCBs, Ammonia, Cyanide, chlorinated naphthalenes

#### INTRODUCTION

The sediments of the Great Lakes are known to have been contaminated with a variety of organic and inorganic substances due to human activities. The present study was undertaken to establish the current concentrations of both inorganic and organic contaminants in sediments from six harbors in the North American Great Lakes.

Concentrations of contaminants have been determined in the sediments of the Great Lakes (Fallon and Horvath 1985, Anon 1985). Studies have reported the concentrations of metals (Handy and Post 1985), the concentrations of synthetic, organic contaminants (Chau et al. 1985, Kaiser et al. 1985, Platford et al. 1985) and several have reported concentrations of both inorganic and organic contaminants (Maguire et al. 1985, Pranckevicius 1986).

#### METHODS

##### Sample Collection

Samples were collected from six locations (Table 1) by the U.S. Army Corps of Engineers, St. Paul, MN and shipped to the Michigan State University Aquatic Toxicology Laboratory in E. Lansing, MI by Thermo Analytical Inc. on May 25, 1989.

Table 1. Locations of Sample Collections.

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Duluth, MN Harbor, 21st Street Channel
St. Joseph, MI #1
St. Joseph, MI #2
Rouge River, MI
Saginaw Bay, MI
Sebewaing (Reference)

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### Sediment Characterization

#### Physical:

All six samples were mechanically homogenized under an argon atmosphere and stored in plastic buckets at 4°C. The sediments from six locations were characterized for particle size distribution, total solids (TS), total volatile solids (TVS), total organic carbon (TOC), and percent moisture. The particle size distribution was determined by the wet sieve and pipet method (Plumb 1981). Total solids content was determined by heating wet samples to 103°C until constant weight was achieved which also gives the percent moisture (% H<sub>2</sub>O). Volatile solids were determined by ashing at 550°C. Total organic carbon was determined using a Leco organic analyzer. All characterization methods were those of Plumb (1981).

#### Inorganic:

Concentrations of chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), nickel (Ni), and zinc (Zn) were determined by inductively coupled plasma atomic emission spectroscopy (ICP) following an acid digestion of the sediment (Plumb 1981). Concentrations of arsenic were determined by arsine generation followed by colorimetric detection with silver diethyldithio-carbamate as an indicator. Sediment samples were prepared for arsenic analysis with successive hydrogen peroxide and a HNO<sub>3</sub>/H<sub>2</sub>SO<sub>4</sub> digestions of a 2.5-3.5 g sample of moist sediment (E.P.A. Methods 3010 and 7061, APHA 1985). Cadmium was determined using flame atomic absorption spectrometry (AAS) on samples digested in the same way as described for ICP analysis (Plumb 1981). Mercury was determined using the cold vapor technique with a 4.0-5.0 g wet sample (Plumb 1981).

Selenium was determined fluorometrically. A 1 g sample (dry weight equivalent of moist sediment) was added to a flask with 5 ml of HNO<sub>3</sub> and 3 ml of H<sub>3</sub>PO<sub>4</sub> and heated at 180°C until no brown fumes were evident. Three milliliters of H<sub>2</sub>O<sub>2</sub> were then added and heated first at 150°C and then at 230°C until a weight which equalled the mass of the flask, sediment, and H<sub>3</sub>PO<sub>4</sub> plus 1.0 g was attained. Flasks were cooled and 6 ml of 50% HCl and 2 ml of 96% formic acid were added and the flasks heated at 105-180°C for 10 min. Five milliliters of a solution containing 20 g/L EDTA and 24 g/L hydroxylamine were added along with one drop of cresol red. The samples were titrated with 30% NH<sub>4</sub>OH to an amber color (pH 2.3-2.8). The flasks were cooled and 5 ml of diamino-naphthalene hydrochloride (0.005 g/flask in a 1% aqueous solution of HCl) was added. After heating to 100°C for 20 min, samples were cooled to room temperature and 5 ml of cyclohexane were added and stirred for 5 min. Samples were extracted with cyclohexane, collected and quantified with an excitation wavelength of 376 nm and emission wave length 518 nm (Per. com., Dr. Howard Stowe, Animal Health Diagnostic

Laboratory, Michigan State University, E. Lansing, MI). Standards contained 0.5 ml of  $\text{HNO}_3$ , 3 ml of  $\text{H}_3\text{PO}_4$ , and 3 ml of  $\text{H}_2\text{O}_2$  and were digested in the same manner as samples. Concentrations of metals in NBS (National Bureau of Standards, Gathersburg, MD) reference sediments (numbers SRM 1605 and SRM 2704) were measured and the results compared to the certified values and ranges.

Ammonia-nitrogen was determined following distillation of a 10 g wet weight sample (APHA, 1985). The concentration of ammonia was determined using an Orion ionanalyzer model #701A and  $\text{NH}_3$  probe model #95-12 (Orion Inst. Co., Inc., Cambridge, MA.). Chemical oxygen demand was determined with a potassium dichromate digestion technique using 40-90 mg of wet sediment (APHA 1985) followed by a reflux distillation from a 5 g sample (Method SW846, USEPA 1986).

### Organic

#### Extraction:

Organic compounds were separated from sediments by Soxhlet extraction with methylene chloride according to US EPA method SW846 (USEPA 1986) with several modifications. Samples were mixed with 20 g sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), dried over night and thoroughly ground to a powder. PCB congener number 30 (IUPAC) was added (2.85  $\mu\text{g}$ ) prior to extraction to allow for the correction of extraction efficiencies. Volumes of samples were reduced with a rotoevaporator at 34°C.

#### Clean-up:

Concentrated extracts were initially cleaned by gel permeation chromatography (GPC) (USEPA 1986). The GPC eluent was further cleaned and components separated by florisil and silica gel chromatography (Schmitt et al. 1985) in 10 mm(I.D.) columns. The GPC eluent was transferred to a florisil (5 g) column and eluted with 40 ml 6% diethylether in petroleum ether, followed by 40 ml 35% diethylether in petroleum ether and finally with 50 ml toluene. The first fraction was further separated on a silica gel (5 g) column with 40 ml 0.5% benzene in hexane, followed by 40 ml 25% diethylether in hexane. The first fraction contained sulfur and was decontaminated with TBA-sulfite reagent (USEPA 1986). Lindane (0.5  $\mu\text{g}$ ) was added as an internal standard for quantification of pesticides. The volumes of the four fractions were reduced and transferred to 1 ml of isooctane for quantification by gas-liquid chromatography (GLC).

#### Quantification:

Concentrations of individual compounds were quantified by gas chromatography with a 30 m DB-1 column, electron capture detection (ECD-GC), and internal standard methods (USEPA 1986). Aldrin (0.6  $\mu\text{g}$ ) was added to the fractions which contained pesticides for internal standard calculations. Pesticides which occurred in the first fraction eluted from the silica gel column were quantified by comparison to an internal lindane standard (Appendix I). Concentrations of polychlorinated biphenyls (PCBs) which were collected in the first fraction which was eluted from the silica gel column were quantified vs. the internal standard (PCB congener IUPAC #30) by the use of a statistical pattern recognition system (Comstar; Burkhard

and Weiniger 1987) and standard mixtures of Aroclors 1242, 1248, 1254 and 1260. Concentrations of polychlorinated naphthalenes (PCNs) were quantified by comparison to Halowax 1014 by using the total peak area from peaks in the second fraction eluted from the Florisil column.

#### QA/QC:

A method blank was analyzed to check for interferences. PCB analyses were conducted in duplicate for the Duluth site using hexane:acetone 1:1 (v:v) and methylene chloride extractions. Triplicate analyses were conducted to evaluate possible matrix interferences and determine background responses. The recovery efficiency was determined by analyzing a sediment spiked with an Aroclor mixture. The linear range for analyses of Aroclor mixtures was found to be sufficient for all concentrations of PCBs quantified in this study. Calibration curves and relative response factors were developed for the pesticide analysis. A PCN mixture, Halowax 1014, was chromatographed on Florisil and silica gel to determine the elution pattern.

## RESULTS

### Characteristics of Sediments

The particle size distribution was similar among the locations (Table 2). The total solids and % water contents varied considerably among locations. Sediment from the Rouge River and Sebawaing Harbor (Reference) had the least water content (Table 3). The organic carbon contents varied from 1.7% in the Sebawaing sediment to 3.9% in the sediment from Duluth Harbor (Table 3). The least COD (chemical oxygen demand) was observed in sediments from Sebawaing (Reference) with the greatest CODs observed in sediment from St. Joseph Harbor and the Rouge River (Table 4). Cyanide concentrations were low in sediments from all locations (Table 4). Concentrations of ammonia ranged from a minimum of 47.6  $\mu\text{g/g}$  in sediments from Duluth to a maximum of 103.6  $\mu\text{g/g}$  in sediment from location #1 in St. Joseph Harbor (Table 4).

Concentrations of metals varied greatly among locations (Table 5). In general, the greatest concentrations were observed in sediments from the Rouge River while the least concentrations were observed in sediments from Sebawaing Harbor.

Concentrations of total PCBs ranged from 30 ppb to 930 ppb on a wet weight basis (Table 6). The least concentration of PCBs which was observed in sediment from the Reference site and was near the quantitation limit of the Comstar program due to the multiple peak analysis nature of the program. The coefficient of determination ( $R^2$ ) for the regression fitting the observed pattern to Aroclor was 0.872 (Table 7), which is considered to be a poor fit compared to technical mixtures of Aroclor. Concentrations of PCBs were above the quantification limits at all other locations. The large coefficient of determination ( $R^2$ ) observed for the pattern fit for PCB in sediments from Duluth indicates a recent, uncontaminated, unweathered Aroclor mixture was present at that location.

Table 2. Particle size distribution of sediments as determined by the sieve and pipet method.

$\phi$ Size	LOCATION					
	DUL	ST.JOE 1	ST.JOE 2	ROUGE	SAG	REF
	( _____ % Smaller than $\phi$ Size _____ )					
-5	100	100	100	100	100	100
-4	100	100	100	100	100	100
-3	100	100	100	98.0	100	99.9
-2	100	100	100	91.1	100	99.5
-1	99.6	99.6	99.4	87.3	99.5	98.9
0	98.0	98.7	98.7	82.1	98.5	98.1
1	94.7	97.5	97.3	73.4	94.5	96.9
2	88.7	95.4	91.8	58.6	80.1	89.8
3	76.3	91.6	76.8	43.0	58.7	70.3
4	62.9	85.3	59.2	28.0	40.4	42.2
5	58.5	65.5	43.5	24.8	38.4	37.8
6	54.1	45.6	33.5	21.7	36.3	33.4
7	47.4	39.5	25.1	21.7	34.9	29.3
8	40.8	30.2	23.4	20.2	31.8	24.9
9	31.1	25.2	14.6	13.8	25.3	18.7

DUL - Duluth, MN, 21 st. St., Channel  
 ST.JOE 1 - St. Joseph, MI, #1  
 ST.JOE 2 - St. Joseph, MI, #2  
 ROUGE - Rouge River, MI  
 SAG - Saginaw Bay, MI  
 REF - Reference (Sebawaing)

Concentrations of pesticides were less than 100 ppb, wet weight basis, in sediments from all locations (Tables 8a and 9b). All pesticides are reported uncorrected for recovery (Appendix I). If a peak was located within the retention time window, but its value was less than the reportable limit (DL), the concentration of that compound was reported as a trace with the measured concentration given in parentheses. In some cases, a peak bordered the 0.05 min retention window. These peaks were accepted, though absolute identification is uncertain (see footnote 1 in Table 8a). Endosulfan-I and pp'-DDT coelute on the DB-1 GC

Table 3. Physical characteristics of sediments.

LOCATION	TS ( $\mu\text{g/g}$ )	TVS ( $\mu\text{g/g}$ )	TOC ( $\mu\text{g/g}$ )	% H <sub>2</sub> O
DUL	508,600	44,200	38,500	60.2
ST. JOE 1	231,200	32,500	36,600	76.9
ST. JOE 2	348,800	17,600	29,700	65.1
ROUGE	555,600	96,400	32,600	44.4
SAG	520,100	25,100	21,000	48.0
REF.	543,200	22,100	17,500	45.7

TS - Total solids.

VS - Volatile solids.

TOC - Total organic carbon.

% H<sub>2</sub>O - Percent moisture.

See Table 2 for key to location codes.

Table 4. Total ammonia, cyanide and chemical oxygen demand of sediments.

LOCATION	NH <sub>3</sub> -N ( $\mu\text{g/g}$ )	HCN ( $\mu\text{g/g}$ (wet weight))	COD
DUL	11.6	< 0.5	45,240
ST. JOE 1	103.6	< 0.5	34,020
ST. JOE 2	63.8	0.7	89,680
ROUGE	59.0	2.7	78,150
SAG	50.3	< 0.5	21,540
REF	47.6	< 0.5	15,880

NH<sub>3</sub>-N - Ammonia nitrogen reported as N.

HCN - Total hydrogen sulfide.

COD - Chemical oxygen demand.

See Table 2 for description of location codes.

column. However, if the peak located at the retention time is taken as pure pp' DDT or Endosulfan-I with 100% recovery, the calculated concentrations are very small (Table 8b). Thus, it was concluded that this error would be negligible. Chlordane was represented by its components, cis- and trans-chlordane and trans-nonachlor and by the primary metabolite oxychlordane. Quantifiable concentrations of chlordane, DDD, and pp'-DDE were observed in sediments from all of the locations while quantifiable concentrations of aldrin, endrin, heptachlor, and op'-DDE were not observed in any sediments. All peaks were identified in sediment extracts from all of the locations except those from the Rouge River, which contained great concentrations of unknown organic compounds in the second fraction eluted

from the silica gel column. These compounds had long retention times and contained multiple peaks.

Table 5. Total concentration of acid extractable metals in sediments.

LOCATION	METAL									
	As	Cd	Cr	Cu	Pb	Mn	Hg	Ni	Se	Zn
	( $\mu\text{g/g}$ (wet weight))									
DUL <sup>1</sup>	7.8	3.2	51	101	133	380	4.24	36	0.3	427
ST. JOE #1	32.0	2.9	45	77	103	763	<0.05	59	0.6	452
ST. JOE #2	16.3	2.8	22	32	29	848	<0.05	10	0.5	153
ROUGE	13.0	3.3	53	129	147	328	0.25	35	0.8	332
SAG	13.2	0.5	22	23	15	368	<0.05	10	0.4	201
REF	6.4	0.3	9	12	3	200	<0.05	<5	0.5	52

<sup>1</sup> See Table 2 for location codes.

Table 6. Concentrations of total Aroclor in sediments from six locations as quantified by Comstar.

LOCATION	CONCENTRATION ( $\mu\text{g/kg}$ )	
	WET WEIGHT	DRY WEIGHT
ROUGE <sup>1</sup>	923.6	1,626
SAG	682.6	1,274
ST. JOE #1	110.4	434.7
ST. JOE #2	88.6	237.9
DUL	873.7	2,123
REF	31.6	55.7

<sup>1</sup> See Table 2 for location codes.

Table 7. Comstar statistics.

	Confidence Levels		R <sup>2</sup>		F-Value
	Contamination	Weathering	Initial	Final	
ROUGE <sup>1</sup>	95%	95%	0.900	0.986	1573
SAG	95%	95%	0.736	0.978	1261
ST. JOE #1	90%	90%	0.305	0.951	338.0
ST. JOE #2	90%	90%	0.224	0.982	566.0
DUL	90%	90%	0.942	0.989	1751
REF	80%	99%	0.205	0.872	122.0

See Table 2 for location codes.

Table 9. Metals exceeding the WDNR sediment quality guidelines are designed by + (Sullivan et al., 1985).

Metal	DUL	LOCATION			RR	SAG
		St. Joe #1	St. Joe #2			
As		+	+	+	+	
Cd	+	+	+	+		
Cr						
Cu	+			+		
Pb	+	+		+		
Hg	+			+		
Ni						
Zn	+	+	+	+	+	

See Table 2 for code to locations.

Polychlorinated naphthalenes (PCN) were not detected in sediments from any of the locations. The PCN screen consisted of 14 peaks in the last half of the Halowax 1014 chromatogram. These compounds were eluted in the second fraction through the florisil column. With a minimum PCN recovery of 50%, the minimum detection limit, based on the # of peaks x (background + 2σ) vs. the total relative response factor ([ΣRel peak areas in florisil frac #2]/total Halowax 1014 concentration) was found to be 30 ppb. Also, if significant concentrations of PCNs had been present in the sediment samples analyzed they would have caused poorer Comstar fits (i.e., greater elimination of contaminated peaks).



Table 8a. Concentrations of pesticides in sediments from six locations, reported on a wet weight basis.

Location	Lindane		α-chlordane		γ-chlordane		γ-nanachlor	op'-DDD	pp'-DDD
	α-BHC	Lindane	Concentration μg/kg (wet weight)		Concentration μg/kg (wet weight)				
ROUGE <sup>1</sup>	0.69	0.99 <sup>1</sup>	4.27	2.39	1.45	7.72	17.8		
SAG	< D.L.	0.25	1.33	0.41	trace (0.26)	3.73	5.28		
ST. JOE #1	< D.L.	< D.L.	6.22	3.62	5.52	2.13	6.37		
ST. JOE #2	< D.L.	70.1 <sup>1</sup>	2.65	1.38	1.09	1.32	3.36		
DUL	2.56	< D.L.	8.10	6.24	4.80	21.8	21.3		
REF	trace (0.08)	trace (0.10)	trace 0.31	trace (0.31)	(0.11)	trace < D.L.	(0.40)		

<sup>1</sup> Probably not Lindane but occurs within 0.05 min of retention time window for specific compound, no mass-spec confirmation.

<sup>2</sup> Trace = peak observed but below reportable limit.

Table 8b. Concentrations of pesticides in sediments from six locations, reported on a wet weight basis.

Location	Concentration $\mu\text{g}/\text{kg}$ (wet weight)					
	pp'-DDT	pp'-DDE	Dieldrin	Endosulfan	Methoxychlor	Heptachlor Epoxide
ROUGE	0.32	19.6	trace (0.26)	0.26	15.2	< D.L.
SAG	< D.L.	5.07	trace (0.12)	< D.L.	< D.L.	< D.L.
ST. JOE #1	< D.L.	12.5	trace (0.14)	< D.L.	trace (1.34)	trace (0.23)
ST. JOE #2	0.47	7.26	0.45	0.38	5.19 <sup>2</sup>	< D.L.
DUL	trace 0.01	14.2	trace (0.17)	trace (0.01)	trace (1.15)	1.74
REF	trace (0.07)	1.05	< D.L.	(0.06)	< D.L.	< D.L.

<sup>1</sup> Probably not Lindane but occurs within 0.05 min of retention time window for specific compound, no mass-spec confirmation.

<sup>2</sup> Peak borders the 0.05 min retention time window.

This was not observed, therefore, it is concluded that the concentrations of PCN in the sediments analyzed were small.

Several sets of sediment quality criteria have been reported (Anon 1973, Anon 1977a, Sullivan et al. 1985, Persaud et al. 1989, Anon 1988, Hart 1988). There are wide ranges in these criteria and guidelines and few of these compilations are comprehensive (Giesy and Hoke 1990). Most of the available criteria are for metals, pesticides or industrial chemicals, such as PCBs. One of the most recently compiled and comprehensive listings is that prepared by the Wisconsin Department of Natural Resources (WDNR) (Sullivan et al. 1985). We compared the results of our analyses to these guidelines. Of the pesticides measured in our study, only concentrations of chlordane were found to exceed the Wisconsin guidelines. Concentrations of chlordane exceeded the guidelines of 0.01  $\mu\text{g/g}$  at all locations. Concentrations of at least one metal exceeded the WDNR guidelines at all locations, except the Sebewaing (Table 9). The proportions of the metals exceeding the WDNR guidelines were 63%, 50%, 37%, 75% and 25% at Duluth, St. Joseph #1, St. Joseph #2, Rouge River and Saginaw Bay, respectively. The WDNR guidelines were exceeded by concentrations of Zn at all locations except for Sebewaing while concentrations of As exceeded the guidelines at four locations (Table 9).

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