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ANTHROPOGENIC, POLYHALOGENATED, ORGANIC COMPOUNDS IN NON-MIGRATORY FISH FROM THE NIAGARA RIVER AREA AND TRIBUTARIES TO LAKE ONTARIO

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ABSTRACT. Non-migratory fish from the mouths of tributaries to Lake Ontario and from the Niagara River and its tributaries were analyzed for anthropogenic organic compounds by methane enhanced, negative ion, gas chromatographic mass spectrometry. The results indicate that non-migratory fish, sampled at distances of up to 300 km from Niagara Falls, New York, accumulate pollutants originating at a particular hazardous waste site in that city. Several brominated aromatic compounds that apparently come from Rochester, New York, were also identified. A variety of chlorinated pesticides and compounds of industrial origin were found.
ADDITIONAL INDEX WORDS: Toxic substances, gas chromatographic mass spectrometry, waste management, fish, pesticides.

INTRODUCTION

The presence of numerous hazardous waste dumps near the shores of the Niagara River and the industrial nature of the city of Niagara Falls, New York, have created pollution problems in the Niagara River and Lake Ontario (Allen *et al.* 1983). One problem is human exposure to pollutants through the consumption of fish. We have previously shown that one of these dumps, the Hyde Park dump, is the major source of several fluorinated pollutants in Niagara River fish (Jaffe and Hites 1985a). These hydrophobic compounds are probably particle-associated and may accumulate in the food chain. The impact of these compounds (and by implication, of other compounds leaking from the Niagara Falls dumps) on the fish in Lake Ontario is not yet known. Therefore, we have analyzed fish taken from numerous rivermouths and embayments along the southern and eastern shores of Lake Ontario for these compounds. Fish were sampled and analyzed from the Niagara River and from tributaries to the Niagara River to gain a better understanding of the sources of the various compounds. In all cases, only non-migratory, bottom-feeding fish species were collected so as to be representative of the local vicinity. The presence of compounds from a specific point source in non-migratory fish provides information on the bioavailability and transport of these compounds.

The fluorinated compounds, which were the primary analytes of this study, were shown to be leaking from the Hyde Park dump by Elder *et al.* (1981) and by Jaffe and Hites (1985a). They are all by-products of Hooker Chemical's production of 4-chlorobenzotrifluoride; their structures are shown in Figure 1. We have previously shown that these compounds come almost exclusively from the Hyde Park dump (Jaffe and Hites 1985a). If this dump has a long-range impact on the fish of Lake Ontario, these compounds will be present in fish from remote areas of the lake. Our previous results show that sediments from remote areas of the lake have trace levels of these fluorinated compounds (Jaffe and Hites 1985a and b, Kaminsky *et al.* 1983), and based on these data, a transport mechanism for particle adsorbed pollutants entering Lake Ontario from the Niagara River was proposed (Jaffe and Hites 1985b). The intent of this paper is to further demonstrate the long-range transport of certain organic pollutants in Lake Ontario and indicate their bioavailability.

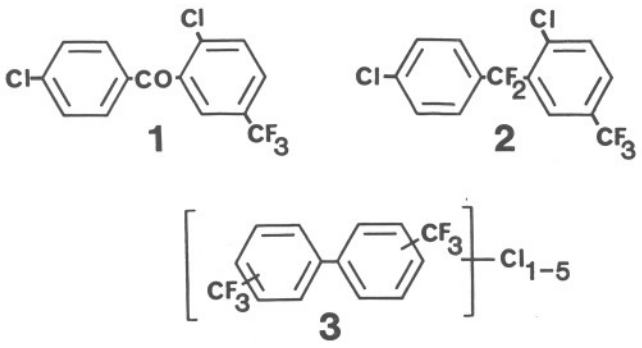

FIG. 1. Structures of the fluorinated compounds. Compound 1: dichloro-trifluoromethylbenzophenone; Compound 2: alpha, alpha-difluoro-dichlorotrifluoromethyldiphenylmethane; Compound 3: bis(tri-fluoromethyl)chlorobiphenyls. We will designate this last group as compounds 3-1, 3-2, 3-3, 3-4, and 3-5 where the second digit indicates the number of chlorines.

The analytical methods were based on gas chromatographic mass spectrometry operating in the negative ion mode. Because this technique responds to other poly-halogenated compounds, pesticides and other industrial compounds were also measured in the fish samples. Thus, we were able to determine a point source of some brominated pollutants.

EXPERIMENTAL SECTION

Fish were sampled in the summer of 1984 by electro-shocking. Sample locations are shown in Figures 2 and 3. Lake Ontario rivermouth and embayment sampling sites were within 1 mile of the lake, except for the Genesee River sample which was taken 2 miles upstream. All of the fish were carp except for the Black River, the Oak Orchard Creek, and the duplicate samples from the Buffalo River and the Eighteen Mile Creek (see Table 1). All fish were non-migratory bottom-feeding fish.

The fish were immediately labeled, weighed, measured, wrapped in clean aluminum foil, and frozen. In the laboratory, fish from the same location, of the same species, and of similar size, were combined into composites (see Table 1). The ground composite material was mixed and reground two additional times before a 500 gram subsample was placed into a clean glass container and frozen until analysis. Extraction and cleanup procedures have been described elsewhere (Jaffe *et al.* 1985).

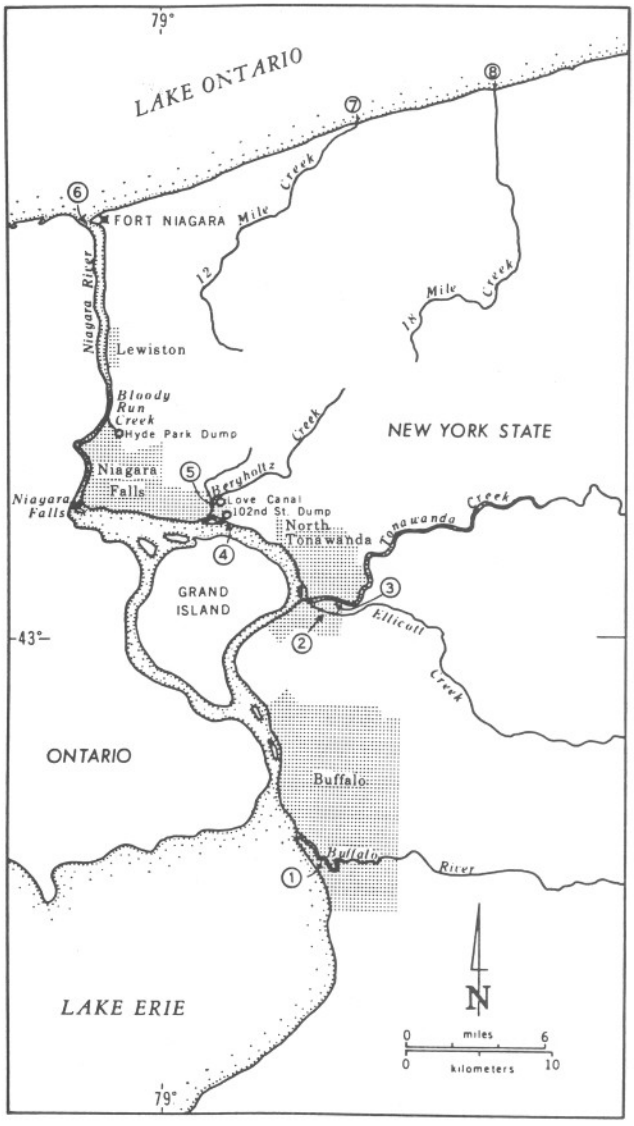

FIG. 2. Map of the Niagara River area indicating the sampling sites and some of the hazardous waste disposal sites.

The fractionated fish extracts were analyzed by methane enhanced, negative ion, GC/MS on a Hewlett Packard 5985B instrument, equipped with a DB-5 30-m capillary column. The ion source pressure and temperature were 0.4 torr and 100°C, respectively. The GC oven temperature was programmed from 40°C for 4 minutes to 280°C for 20 minutes at a rate of 4°C/minute. Injector and transfer line temperatures were set to 285°C.

An internal standard, 2-chloro-5-(trifluoromethyl)-benzophenone, was used for quantitiation purposes. Response factors were determined using

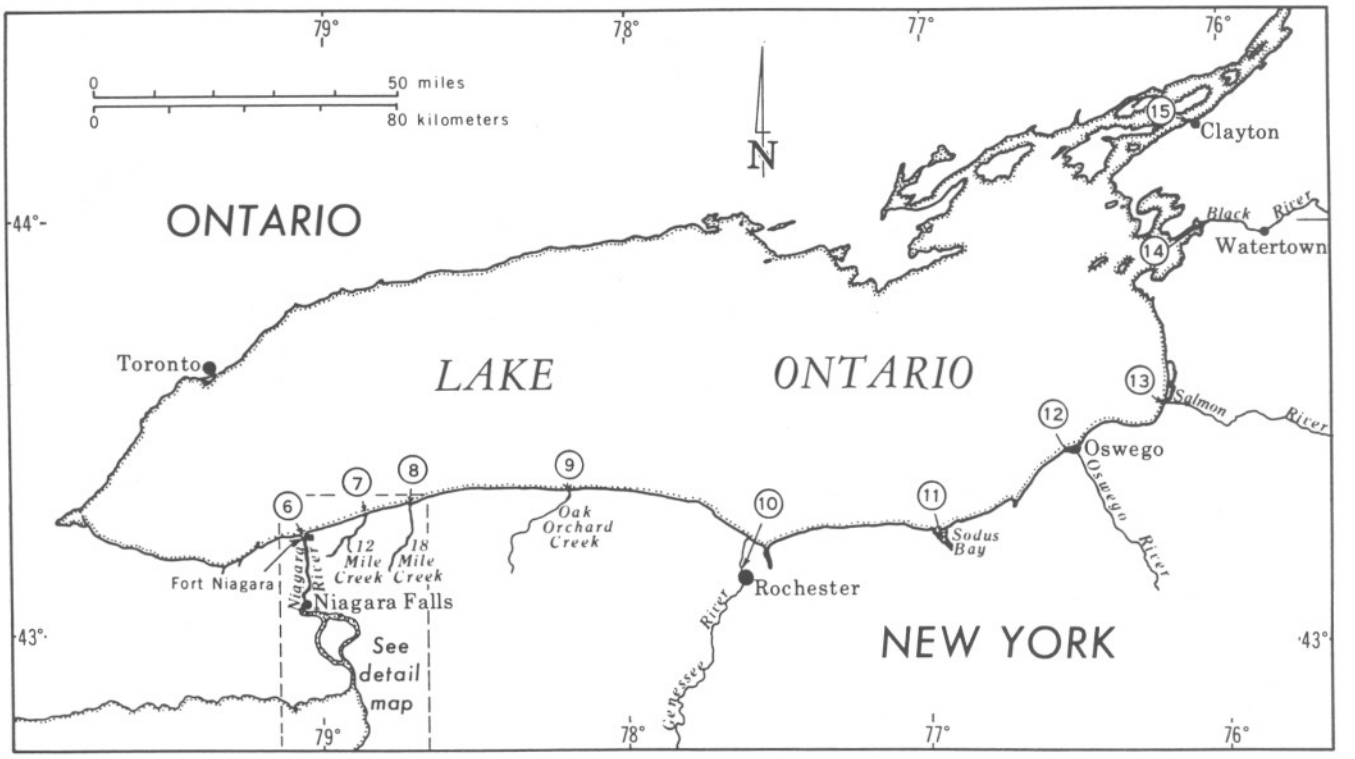

FIG. 3. Map of the Lake Ontario area indicating the sampling sites. All samples (from the tributaries to the lake) were taken within a distance of 1 mile from Lake Ontario, except site 10 which was 2 miles upstream.

TABLE 1. Sampling details on the fish collected from the 15 sites shown in Figures 2 and 3. Key: C, common carp **(Cyprinus carpio)***; GF, goldfish* **(Carassius auratus)***; CF, catfish* **(Ictalurus** *sp.***)***; S. sucker* **(Catostomus commersoni).**

Site #	Location	Species	# Fish	Av.Length cm(range)	Av.Wt.kg (range)	% Fat
1-a	Buffalo River	C	3	45(53–39)	1.5(2.1–0.9)	11.5
1-b	Buffalo River	C	1	33	1.0	20.0
2	Ellicott Creek	C	3	44(46–40)	1.4(1.8–1.0)	5.5
3	Tonawanda Creek	C	4	49(59–40)	1.9(2.6–1.0)	13.8
4	102nd Street Bay	C*	3	55(62–51)	2.8(4.0–1.8)	12.8
5	Bergholtz Creek	C*	3	41(47–35)	1.4(1.8–0.8)	8.0
6	Fort Niagara	C	3	55(58–52)	3.2(4.1–2.6)	16.7
7	12 Mile Creek	C	3	61(66–56)	3.6(4.6–2.7)	8.0
8-a	18 Mile Creek	C	2	56(65–47)	3.1(4.2–1.9)	8.2
8-b	18 Mile Creek	GF	3	29(31–26)	0.5(0.6–0.3)	10.1
9-a	Oak Orchard Cr.	CF	4	33(33–32)	0.7(0.7–0.7)	5.4
9-b	Oak Orchard Cr.	GF	1	33	0.8	5.9
10	Genesee River	C	2	56(67–45)	3.2(4.7–1.7)	13.8
11	Sodus Bay	C	2	67(67–67)	4.7(5.0–4.3)	14.6
12	Oswego River	C*	1	66	4.1	2.0
13	Salmon River	C	1	81	11.0	7.6
14	Black River	S	2	36(42-30)	0.7(0.9–0.4)	4.9
15	St. Lawrence	C	1	69	4.8	12.0

*One fish in this composite showed external tumors.

available standards. Response factors for compounds where standards were not available were estimated from compounds with similar structures. The response factor determined for octachlorostyrene was also used for heptachlorostyrene. The brominated compounds from the Genesee River were quantitated based on the response factor of 2,4,6-tribromophenol. Response factors for the bis (trifluoromethyl)chloro-biphenyls were assumed to be unity relative to the internal standard. PCBs were reported as Aroclor 1254 and were quantitated based on the three consistently most abundant congeners; their response factors were determined relative to Aroclor 1254. Blanks were run through the procedure, and no significant contamination was observed. Replicate analyses of the same sample had errors of ± 15%, and replicate analyses of different samples from the same site had errors of ± 25%.

RESULTS AND DISCUSSION

The percent lipid, species, number of fish per composite, average weight, and average length are shown in Table 1. Table 2 shows the concentrations of the various compounds found in the fish at the locations shown in Figures 2 and 3. Because compounds of this nature tend to concentrate in fish lipids, concentrations are given in ng/g of fish lipid to normalize for differences in lipid content between species.

BY-PRODUCTS OF THE INDUSTRIAL SYNTHESIS OF 4-CHLOROBENZOTRIFLUORIDE

Approximately 8,000 tons of chemical wastes related to the production of 4-chlorobenzotrifluoride have been dumped into the Hyde Park dump in the city of Niagara Falls (Interagency Task Force on Hazardous Waste 1979). Some compounds leaking from this dump (see Fig. 1) have been identified in sediments and fish from this area and from Lake Ontario (Jaffe and Hites 1985a). A transport mechanism for particle absorbed pollutants in Lake Ontario was proposed using these chemicals as marker compounds (Jaffe and Hites 1985b).

As shown in Table 2, this group of fluorinated compounds was detected in most of our fish samples. Since these chemicals are directly related to Hooker Chemical dump sites, it was expected that we would detect them only in those samples taken below the Love Canal area. Indeed, fish from the Buffalo River, Ellicott Creek, and Tonawanda Creek (see Fig. 2) did not show the presence of these compounds. The samples with the highest concentrations were those from the Fort Niagara area and Twelve Mile Creek and Eighteen Mile Creek. These are the closest locations downstream from the Hyde Park dump. Concentrations were lower at sites farther away from the Niagara River. However, concentrations at Sodus Bay, Oak Orchard Creek, Salmon River, and the St. Lawrence River differed from those near the Niagara River by only a factor of 2 to 5. The fact that these compounds were present in fish from the St. Lawrence River at elevated concentrations (30 ppb) is a clear indication of their wide-range transport and bioavailability.

The data shown here demonstrate the bioavailability of chemicals from a specific hazardous waste dump in Niagara Falls to non-migratory fish in Lake Ontario tributaries. Some of these fish were collected at a distance of 200-300 km from the dump. How do these chemicals get there? We have recently discussed the transport mechanism of particle-adsorbed pollutants from the Niagara area to Lake Ontario (Jaffe and Hites 1985b). Once into the lake, these particles are distributed along the southern shore of the lake by counter-clockwise water currents. During biannual turnover, these particles become resuspended and incorporated into the deeper areas of the lake. However, while deposited in the nearshore zone they become available to fish, in particular bottom feeders. This is a likely mechanism by which fish from sites 7 to 14 could become exposed to these compounds. At locations where the suspended sediments from the nearshore zone can get "trapped," such as bays, these pollutants are available to the biota for extended periods of time. This is the case at Sodus Bay and the lower Salmon River (Jaffe and Hites 1985b).

Similar to our previous observations, compound 1 was not detected in most of our samples (Jaffe and Hites 1985a). We believe this is due to its relatively higher water solubility and/or the preferential metabolism of the keto group.

BROMINATED AROMATIC COMPOUNDS

Brominated compounds were found in fish from the Genesee River only. Identities, concentrations, and abbreviated negative ion mass spectra are shown in Table 3. In all cases, the proper isotope patterns were observed. Only the tribromoanisole

was concentrated enough to give a confirming electron impact (EI) mass spectrum. Tribomophenol was confirmed using an authentic 2,4,6 substituted standard. Some of the brominated compounds which were found could not be identified based on their NCI-mass spectra. Often these showed only the presence of the ions at m/z 79 and 81 (Br-79 or Br-81). Some of the brominated compounds found here have been previously identified in fish exposed to waste water effluents that had been disinfected with bromine chloride (Kuehl *et al.* 1978). Another possible source of bromine could be the photographic industry in the Rochester, New York, area. Silver bromide is used extensively in the production of photographic supplies. Brominated aromatic compounds are also used in the manufacture of dyes, as indicators, and as fire retardants (Kirk *et al.* 1980). For example, tribromophenol is used in antiseptics, germicides, and fungicides and is used to prepare fire-retardants, and tribromocresol is a topical fungicide (Merck 1983). The exclusive presence of these brominated compounds in Genesee River fish indicates that this area has a point source of these compounds.

PESTICIDES

A variety of chlorinated pesticides was detected, many of them were derivatives of hexachlorocyclopentadiene. Fish from the Ellicott Creek and Tonawanda Creek showed the highest concentrations of many of these compounds. Endosulfan-I appeared in highest concentrations in the 102nd Street Bay and Fort Niagara samples. Total DDT was lowest at the 102nd Street Bay and the Oswego River. Dacthal, a pre-emergence herbicide, was detected in all fish samples with highest concentrations in the Ellicott Creek and Tonawanda Creek samples.

Hexachlorocyclohexanes (BHCs) were also detected in all samples. The concentrations for the different isomers (alpha, beta, gamma, and delta-BHC) are shown in Table 2. Concentrations of all isomers were highest in the 102nd Street Bay and Bergholtz Creek fish samples. This is not surprising, since over 7,000 tons of this material were dumped in this area (Interagency Task Force on Hazardous Waste 1979). Concentrations in the sediments near the 102nd Street Dump and Bergholtz Creek were found to be as high as 260 ppm and 1 ppm, respectively (Jaffe and Hites 1984). The apparent sources of the BHCs are the Love Canal and 102nd Street Dump.

Chlordene, heptachlor, aldrin, 1-hydroxychlordene, and kepone were not detected in any of our samples. However, mirex and photomirex were. It is interesting to note that photomirex was not found in any of the samples taken above Niagara Falls but was prevalent below the falls. Mirex concentrations were also lower above the falls. The highest concentrations were found in carp from Eighteen Mile Creek. Both mirex and photomirex have been found in Lake Ontario herring gulls (Norstrom *et al.* 1980) and fish (Clark *et al.* 1984, Kaiser 1978, Laseter *et al.* 1978).

Fish from Lake Ontario tributaries contained the same contaminants found in non-migratory fish from Lake Huron and Lake Superior tributaries (Jaffe *et al.* 1985) except that Endosulfan-I, mirex, and photomirex were not detected in Lake Huron and Lake Superior fish and heptachlorepoxide and oxychlordane were not found in Lake Superior fish. Dacthal, BHC, heptachlorepoxide, oxychlordane, dieldrin, and total DDT generally were more concentrated in the Niagara and Lake Ontario areas as compared to the tributaries of Lakes Huron and Superior. Concentrations of chlordane and nonachlor were similar to those found in Lake Huron but were higher compared to Lake Superior.

COMMON INDUSTRIAL POLLUTANTS

Chlorinated benzenes, toluenes, anisoles, styrenes, biphenyls, and diphenylethers have previously been detected in fish from the Great Lakes area (Jaffe *et al.* 1985, Clark *et al.* 1984). Chlorinated benzenes have been produced on an industrial scale in the city of Niagara Falls, and many hazardous waste disposal sites in this area are known to contain large amounts of these compounds (Interagency Task Force on Hazardous Waste 1979). Thus, river sediments adjacent to some of these dumps have high levels of chlorobenzenes (Elder *et al.* 1981, Jaffe and Hites 1984). As shown in Table 2, 1,2,3,4- and 1,2,3,5-tetrachlorobenzenes were found only in fish from the Love Canal area whereas 1,2,4,5-tetrachlorobenzene was detected at many more locations. Penta- and hexachlorobenzene were found in all samples. However, the concentrations of all of the chlorobenzenes were higher (by a factor of 10 to 100) in the fish sampled at the 102nd Street Bay and Bergholtz Creek sites. This is not surprising since very high concentrations of these compounds were detected in sediments at these locations (Jaffe and Hites 1984).

TABLE 2. Concentrations of polyhalogenated organic compounds in fish taken from sites around Lake Ontario. The site locations are given in Figures 2 and 3 and in Table 1. Concentrations are reported as nanograms of compound per gram of fish fat (ppb). A suffix of "D" on the site number indicates duplicate analyses of the same sample. Abbreviations: BTF, benzotrifluoride; BHC, hexachlorocyclohexanes; TCB, tetrachlorobenzene; DPE, diphenylether. Percent fat values are listed in Table 1.

	Site No.									
COMPOUND	1-a	1-b	2	3	4 D	4 D	5	6 D	6 D	7
Fluorinated Aromatics										
1 (See Fig.1)	0	0	0	0	4	3	0	1	0	0
2 (See Fig.1)	0	0	0	0	48	46	26	160	160	62
3-1 (See Fig.1)	0	0	0	0	0	0	0	2	0	0
3-2 (See Fig.1)	0	0	0	0	6	4	4	83	78	44
3-3 (See Fig.1)	0	0	0	0	0	0	0	44	32	55
3-4 (See Fig.1)	0	0	0	0	0	0	0	17	10	23
Tetrachloro-BTF	0	0	0	0	100	70	6	24	23	4
Pesticides										
alpha-BHC	57	33	82	41	320	310	610	65	63	56
beta & gamma-BHC	47	28	66	66	250	270	230	48	42	29
delta-BHC	0	0	13	0	300	170	410	0	0	0
Dacthal	680	880	2,300	2,100	170	190	570	200	180	93
Heptachlorepoxide	30	38	120	64	30	22	38	44	58	28
Oxychlordane	20	13	98	62	27	26	42	70	61	66
gamma-Chlordane	170	85	550	530	63	50	170	180	160	150
misc-Chlordane	71	44	270	230	15	34	91	85	90	110
alpha-Chlordane	220	130	970	290	100	76	240	280	300	38
Endosulfan-I	40	32	39	22	140	180	40	280	290	74
trans-Nonachlor	200	130	940	760	150	180	260	540	530	600
misc-Nonachlor	62	49	330	270	44	51	89	130	160	210
Dieldrin	190	130	560	370	160	140	160	300	310	200
Photomirex	0	0	0	0	0	0	0	230	220	400
Mirex	14	12	100	67	35	37	220	580	540	740
o,p′-DDE	37	0	25	51	0	0	17	88	64	63
p,p′-DDE	1,600	790	3,600	1,000	420	600	1,800	4,900	7,900	5,700
o,p′-DDD	370	182	420	72	0	0	260	170	230	330
p,p′-DDD	1,100	490	1,600	1,100	190	200	930	540	750	1,100
Industrial										
TCB-1234 & 1235	0	0	0	0	400	420	56	0	0	0
TCB-1245	36	32	11	0	7,400	6,700	470	75	82	5
Pentachlorobenzene	260	170	130	43	2,000	3,500	1,100	225	220	55
Hexachlorobenzene	140	130	130	82	1,600	1,100	860	360	360	180
Pentachlorotoluene	4	2	7	3	34	25	11	5	5	2
Trichloroanisole	0	0	0	0	0	0	0	0	0	0
Tetrachloroanisole	11	19	11	7	2	1	8	7	7	9
Pentachloroanisole	110	160	86	72	36	31	35	19	17	29
Heptachlorostyrene	0	0	13	9	15	10	170	150	140	140
Octachlorostyrene	3	0	42	10	63	59	650	410	410	340
Decachloro-DPE	0	0	15	0	200	370	360	120	100	59
PCBs	9,300	10,500	35,600	26,600	17,600	14,200	73,800	25,300	21,400	27,200

TABLE 2. Continued

Site No.										
8-a	8-b	9-a	9-b	10	11	12 D	12 D	13	14	15
0	0	0	0	0	0	0	0	0	0	0
160	21	16	24	5	40	2	0	16	3	33
0	0	0	0	0	0	0	0	0	0	0
65	8	6	8	2	22	0	0	11	1	17
92	13	18	13	1	33	0	0	22	2	29
18	6	8	4	0	7	0	0	6	1	4
2	2	0	1	0	0	0	0	0	0	0
49	49	48	53	37	52	18	0	22	27	64
47	55	20	13	50	20	23	52	21	12	21
0	0	0	0	9	0	0	0	0	0	0
120	170	130	130	270	120	100	97	29	49	77
31	54	24	50	20	36	10	0	25	11	38
65	72	43	58	28	44	17	0	41	40	25
210	97	42	59	130	69	33	23	35	27	95
46	40	40	67	51	57	18	25	33	34	57
330	170	120	170	210	150	64	60	100	110	180
36	44	35	22	17	23	5	0	12	12	6
610	520	360	460	170	310	75	84	310	170	300
210	220	120	180	63	82	25	39	110	68	100
350	380	180	210	190	220	100	66	78	100	150
280	250	240	300	0	230	0	0	240	47	280
1,400	550	410	420	43	560	350	440	390	150	600
61	30	0	0	0	76	0	0	0	0	30
4,800	7,200	4,400	6,400	800	5,100	770	790	3,400	1,900	4,200
350	130	0	57	110	290	0	0	0	120	79
1,200	1,300	840	970	420	2,000	0	0	220	500	490
0	0	0	0	0	0	0	0	0	0	0
0	0	0	0	0	0	0	0	0	0	0
53	65	19	36	18	23	30	88	16	7	9
340	480	69	150	66	100	52	100	54	37	52
2	7	6	11	2	2	12	0	2	3	2
3	0	0	0	13	0	0	0	0	0	0
78	77	0	0	18	3	14	14	2	3	0
61	62	10	17	300	32	130	110	35	78	8
430	85	84	92	170	130	4	0	54	24	210
900	270	210	160	25	310	35	33	140	62	480
170	42	0	0	0	69	41	0	0	0	46

TABLE 3. Identities, concentrations, and mass spectra of brominated compounds found in Genesee River fish. Isotopic ratios indicate the proper number of bromines. Only the lightest ion of a cluster is listed.

Compound	Concentration (ng/g fish fat)	Negative ion mass spectrum
Tribromophenol	130	79(6), 250(4), 328(33)
Tribromoanisole	600	79(50), 261(15), 342(35)
Tribromocresol	220	79(94), 276(6), 356(3)
Tribromoaniline	99	79(18), 251(2), 327(33)
Tribromotoluene	180	79(95), 246(1), 326(1)
Dibromochloroanisole	43	79(95), 218(15), 298(30)
Dibromochloroaniline	29	79(31), 283(45)
Bromodichloroanisole	9	79(95), 174(35), 254(13)
Dibromophenol	76	79(10), 250(53)

Similarly, the highest concentration of pentachlorotoluene was found in the Love Canal area.

Comparisons with fish from tributaries to Lakes Huron and Superior (Jaffe *et al.* 1985) showed that fish from the Niagara River and Lake Ontario area have higher concentrations of chlorobenzenes. Only the Tittabawassee River (close to Saginaw Bay) had equally high levels of these compounds.

Chlorinated anisoles, which may be environmental conversion products of chlorinated phenols (Kimbrough 1980), were detected in all fish samples. The Eighteen Mile Creek fish showed high levels of tetrachloroanisole and the Genesee River high levels of tri- and pentachloroanisole. Thus, possible point sources of these compounds may be present in these areas.

Chlorinated styrenes were present in most samples except for those from the Buffalo River. Highest concentrations were found in carp from Eighteen Mile Creek. Chlorostyrenes have previously been identified in Lake Ontario fish (Kuehl *et al.* 1976) and sediments (Kaminsky and Hites 1984). Concentrations of octachlorostyrene in fish from tributaries to Lake Ontario were similar to those from Lake Huron and Lake Superior (Jaffe *et al.* 1985).

PCBs were found at high levels in all Lake Ontario samples. Concentrations were similar to those found in Lake Huron samples and greater than those measured in Lake Superior samples (Jaffe *et al.* 1985). Decachlorodiphenylether was detected at its highest concentration in samples from the Love Canal area and Eighteen Mile Creek. Some chlorinated diphenylethers with two chlorines per molecule were also present in some samples.

CONCLUSIONS

All of the fish analyzed in this study showed relatively high levels of pollutants. In general, the most common pesticides were found in all of our samples and their concentrations were similar to those found in fish from Lake Huron tributaries. Dacthal and the hexachlorocyclohexanes appear to have point sources in the Ellicott and Tonawanda area and the Love Canal area, respectively. We have also identified the Rochester area as a source of a group of brominated aromatic compounds not yet reported in the Lake Ontario area.

Many compounds from industrial sources were also detected. The bioavailability and long-range transport of compounds from the Hyde Park dump was shown by the presence of the fluorinated aromatic compounds in most of our samples. These data support our previously proposed distribution mechanism of particle-bound contaminants in Lake Ontario (Jaffe and Hites 1985b). It is interesting to realize how extensive the effect of the Niagara Falls pollution problem has been on the biota in areas of the lake relatively remote from this city.

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