

Organic Substances in the Rainfall of Lake Superior: 1983. NRBibKey # 88131

W. M. J. Strachan
Environmental Contaminants Division
National Water Research Institute
Canada Centre for Inland Waters
Burlington, Ontario, Canada, L7R 4A6.

Lake Superior is uninfluenced by man relative to the lower Great Lakes but despite this, concentrations of persistent organochlorine substances in fishes are near actionable levels by one or another of the jurisdictions. Known local sources are considered small for these compounds relative to the indicated atmospheric inputs.

A rainfall sampling program for organic substances was undertaken for two "open-lake" sites during 1983. Triplicate samples were collected from Caribou Island in Canada and from Isle Royale National Park in the U. S. Concentrations observed ranged from an annual average of 17.4 ng/L for alpha-BHC to 0.075 ng/L for HCB. Based upon these concentrations, annual loadings of the identified chemicals were estimated. These ranged from 860 kg/annum for alpha-BHC to 3.7 kg/annum for HCB; PCBs were calculated to input 300 kg/annum. The west side of the lake received an average of twice the concentrations found on the east and springtime rain contained elevated levels relative to other times.

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Abstract

In 1983, sampling for organic substances in rainfall was undertaken at two, open-lake locations at opposite ends of Lake Superior - Isle Royale and Caribou Island. Triplicate samples averaging 22 litres were collected for each time period (approximately six weeks) and these were analysed for alpha-BHC, lindane, heptachlor epoxide, dieldrin, endrin, DDT residues, methoxychlor, PCBs and HCB. Alpha- and gamma-chlordane, mirex and toxaphene were also investigated but were not found in any of the samples.

Concentrations of these compounds ranged from 65 ng/L to 0.01 ng/L. The east side of the lake received considerably lower concentrations in rainfall (on average roughly 65% less) and the levels of most of the observed substances were higher at both locations in the spring/early summer. Estimates of direct loadings to the lake were made using volume weighted concentrations. The highest was for alpha-BHC at 860 kg/annum and the lowest for HCB at 3.7 kg/annum; the loading estimate for PCBs was 330 kg/annum.

Keywords: Lake Superior, rain, precipitation, toxaphene, BHC, PCB, methoxychlor, organochlorine, pesticide, loading, atmosphere

Introduction

The presence of toxic organic substances in the atmosphere has been well documented (eg. Harvey and Steinhauer, 1974; Bengtson and Sodergren, 1974; Nisbet and Sarofim, 1972). In the Great Lakes region, the Canada-U.S. Great Lakes Water Quality Agreement (1978) calls for "the more accurate definition of wet and dry deposition rates" of airborne pollutants. The Science Advisory Board of the International Joint Commission reviewed this subject as it related to the Great Lakes (SAB, 1979 and 1980) and made strong recommendations to the effect that agencies of the two governments should investigate the role of this medium in the loading of toxic organic substances to this ecosystem. In 1982, they re-iterated this recommendation and called for additional research to identify indicators of such pollution (SAB, 1982).

The mechanism of transfer of such material to surface waters including the Great Lakes is poorly understood. While theoretical models exist to estimate the vapour-phase and particulate dryfall transfer of chemicals to water (Slinn et al., 1978; Smith et al., 1981; Wolff and van der Heijde, 1982; Mackay and Yeun, 1981), they have not been well-established mainly because of the paucity of reliable verification data. For wetfall deposition of organics, the theoretical problems are apparently less although good data on the vapour-phase concentrations of organics severely hinder the determination of their washout ratios. It is only recently that reliable field data for rain and snow have been collected for the Great Lakes region (Murphy and Rzeszutko, 1977; Swain, 1978 and Strachan and

Huneault, 1979) and in most of these studies, the focus has largely been on the PCBs. Estimates using such data (SAB Appendix, 1980; Eisenreich et al., 1981) have indicated that thousands of kilograms of many of these materials are entering each lake annually and that the atmosphere is now conceded to be the major source of such contamination, at least for the international upper Great Lakes. To date, however, the data have been single values pertaining to single events or at best to short periods and the statistical base has not been suitable for the development of reliable estimates for annual loadings.

A particular problem associated with Lake Superior and toxic organic substances in rainfall has been the group of compounds called toxaphene. This pesticide, like the PCB's with which they have common analytical difficulties, is a collection of nearly 200 compounds formed by the chlorination of camphene. They have been widely found in fishes from the lake (WQB, 1984) and up until recently, they were extensively used in the southeast U.S. in aerial spray programs (Seiber et al., 1979). There is no known use of these materials within the system, and hence atmospheric transport is indicated. They have also been found in fish from Lake Siskiwit on Isle Royale (WQB, 1984) which has no known sources of such contamination other than the atmosphere. Rice and Evans (1984) have summarized much of the toxaphene data relevant to the Great Lakes. In their review, they report these compounds in air and rain of the Lake Michigan and Huron areas — at <5.5 ng/m^3 and 7.3 - 168.3 ng/L respectively. Levels for Lake Superior have not been reported although they might be expected to be

similar.

These considerations — the need for more precise estimates of the loadings and the question of toxaphene in the system — led to the study reported here. Open-lake sites were selected to more accurately reflect precipitation on the lake as a whole although data would seem to indicate that there is little difference (<15%) in the volume of the precipitation (Jones and Meredith, 1972) between on-shore and off-shore sites.

Experimental

Sample Collection

Three rain samplers were installed at each of two "open-lake" locations in the Lake Superior area; their locations are shown in Figure 1. Isle Royale (Mott Island) is a U.S. National Park and has been so since 1946. It remains in a relatively pristine state approximately 45 km west of mainland U.S. and 50 km south of Thunder Bay, Canada; Mott Island is a small island to the southeast of the main island and serves as the park headquarters. Caribou Island lies in the eastern part of the lake about 100 km west of the Canadian mainland. It serves as a manned lighthouse station for the Canadian Coast Guard. At both sites, cleared areas around the samplers were sufficient to allow 3 m between each sampler and approximately 10 m from the nearest tall obstruction.

The rain collectors have been previously described (Strachan and Huneault, 1984). Each consists of a 0.2 m² teflon coated funnel with a lid which opens only during rainfall and a teflon column containing XAD-2 resin. Volumes collected were determined

from the funnel area and the precipitation measured with a tipping bucket rain gauge which was also installed (Mott Island) or available (Caribou Island) at each location. The columns were removed periodically (the goal was 100 mm rainfall) and capped with teflon caps. The three columns thus sealed were sent via regular mail and, in all cases, were received at the laboratory full of water and in excellent condition. Replacement columns were shipped the same way.

Analysis

The samples were removed from the columns and provided to the analyst along with resin blanks and "spiked" resin samples — all in a randomly numbered order. In all cases, the samples were processed within two weeks of receipt.

The XAD-2 resin and residual rainwater were drained in 30 cm x 1.5 cm glass columns and extracted with 200 ml of diethyl ether after equilibration with that solvent. The ether was dried by passage through a 7 - 8 cm column of Na₂SO₄ (previously fired in a muffle furnace) and reduced to 1 - 2 mL. The extract was cleaned and fractionated by passing it through a 10 cm column of deactivated silica gel which had been pre-equilibrated with hexane; elution was with 60 mL hexane and with 50 mL benzene. Iso-octane (1 mL) was added to each fraction and the fractions reduced to 1.0 mL by a combination of rotoevaporation and nitrogen blow-down. Analysis was on a 30 m x 0.25 mm SE54 capillary column operating under a temperature programme of: 80 °C for 2 min., 8 °C/min. to 160 °C then 4 °C/min. to 260 °C after which the temperature was held for 8 minutes. Splitless mode injection

was employed and detection was with an electron capture detector. All samples were processed in batches of six together with separate organochlorine and PCB/toxaphene standards. Occasional samples were run on a Finnigan 4500 GC/MS using a similar column and temperature program with detection in the negative ion mode.

Quality Control

Blank samples were processed on two occasions (each time in triplicate as with the rain samples); these samples corresponded to the two batches of the XAD-2 prepared as described. Alpha-BHC was observed on both occasions - at 9.2 ($\pm$ 5.0 s.d.) and at 1.5 ($\pm$ 0.5 s.d.) ng/sample. Both of these levels were well below the levels observed with any sample (lowest found was 73. ng in the April 21-May 22 sample from Caribou Is.). PCB's were observed at 38.5 ($\pm$ 18.4 s.d.) ng/sample only in the first batch and this compares to a mean observed level of 134. ng/sample; the lowest level found was 20. ng/sample. Further, the first batch of XAD-2 corresponded to samples up to July and these generally contained well above the observed background level. No other substances were detected in either of the blanks.

Triplicate resin samples were spiked at realistic levels using several of the investigated samples; recoveries and precisions of analyses were determined and are presented in Table 1 along with the corresponding mean precisions for all of the samples. The precisions obtained, both in the laboratory and with the field samples were comparable to those obtained during trials of the sampler (Strachan and Huneault, 1984); recoveries were somewhat lower, however. Data presented in the balance

of this report are uncorrected for these recoveries and therefore represent minimum concentrations and loadings for the rain.

Results

A total of three samples were collected for the Isle Royale site covering the period May 19, 1983 to October 15, 1983. This corresponded to the park season during which time personnel were available to operate the equipment. At Caribou Island, the lighthouse location was open from April 21, 1983 to November 22, 1983 because of the shipping season. Results for these sample periods are presented in Table 2 (Isle Royale) and Table 3 (Caribou Island). Also included in these tables are the standard deviations for the triplicate samples collected.

A number of compounds were looked for but not found. These were (detection levels in ng/sample): o,p'-DDT (0.4), alpha- and gamma-chlordane (1.0 each), mirex (0.4) and toxaphene (5.0/50.0). The minimum detectable concentrations to which these levels correspond vary with the volumes of rain sampled. In general, except for toxaphene, these corresponded to approximately 0.05 ng/L. None of the preceding compounds were found in any of the replicate samples nor were compounds for which a "nd" (not detected) is given in Tables 2 and 3.

Toxaphene's limit was 5.0 ng/sample for samples from Caribou during July 10-September 5 and from Isle Royale during July 2-October 15; the 50.0 ng/sample limit in other samples was due to large amounts of interfering material collected at the same time and presently unidentified. Even with the higher

quantification level, the concentrations that would have been detected were approximately 3 ng/L; with the lower limit, the detectable concentration was 0.2 ng/L. As with the other unobserved substances mentioned, no toxaphene was noted in any sample despite the fact that it was readily determined in spiked resin material which was analysed in the same batches. Examination of occasional samples concentrated to approximately 0.1 mL (instead of the usual 1.0 mL) using negative ion GC/MS also failed to detect these compounds. These observations must be considered in the light of the widely held view (SAB, 1980 and 1982; Rice and Evans, 1984) that this group of substances arrives in this lake via atmospheric transport. It is difficult to conceive of such materials being present in the air and not being scrubbed out in the rain; nevertheless, this is the conclusion to be drawn from the preceding evidence unless the atmospheric route is not involved. It should be noted that the same analyst is also responsible for much of the Canadian data on toxaphene in Lake Superior fishes -- data which are comparable to U.S. data from the same area (WQB, 1984).

Discussion

There are several aspects of the data from Tables 2 and 3 which can be evaluated. The first is the concentrations and loadings of organic substances in the rain. Alpha-BHC, lindane, methoxychlor and PCB's occurred at substantial levels in all samples, generally well above the 1 - 5 ng/sample that were their detection limits. Other compounds found at concentrations closer to their limits (0.2 - 1 ng/sample) were less frequently

observed; nonetheless, where reported, those were present at real and significant levels.

The collection period for the samples corresponded to the period when the bulk of the wetfall occurred on this lake. For the period 1951-1980, an annual average of 805 mm of precipitation fell on two nearby Lake Superior shore sites (AES, 1983) of which 72% or 580 mm was rain; similar data exist elsewhere in the literature (GLBC, 1975; O'Connor and Mueller, 1970; Jones and Meredith, 1972) and collectively indicate an annual variability of approximately 20%. The average snowmelt from the two lake perimeter sites (Thunder Bay and Sault Ste. Marie) was 28% of the total precipitation for 1951-1980 (AES, 1983) and a similar proportion is assumed for the two sample sites. Studies have indicated that concentrations of these compounds in this medium are approximately 10% of the levels in rainfall (Strachan et al., 1980); PCBs are an exception where comparable levels are probable.

The volume weighted concentrations of these substances in the rain is presented in Table 4 together with the annual loadings which they represent and previous atmospheric loading estimates which have been developed for this lake (SAB Appendix, 1980; Eisenreich et al., 1981). It is apparent from Table 4 that the loading estimates from this study are lower than those previously estimated, most of them considerably so. They may be lower because the concentrations in the atmosphere have decreased in the 4 - 6 years between the earlier collections and those of this study but this trend is far from being established. It is

also noted that these loadings include only a rough estimate of the contributions from snowfall although these are generally less than the variability in annual precipitation and errors are therefore likely to be small.

Loadings via other input routes are also difficult to quantify. The report of the Upper Lakes Reference Group (ULRG, 1977) attempted to quantify inputs from tributaries (which presumably include some atmospheric contributions) and from direct municipal and industrial discharges. Data were presented only for PCBs and "pesticides" with the largest loadings being via the tributaries (>99%). For PCBs, a lakewide loading of 1800 kg/annum was noted while for the pesticides, the value was 1100 kg/annum. Eisenreich et al. (1981) calculated 1400 kg PCBs/annum for the same sources, again mainly via the tributaries. They also reported total atmospheric estimations for several of the individual compounds investigated in this study. The differences between the data in that study and that in SAB, Appendix (1980) give the results noted under Dryfall in Table 4.

It is considered in this study that the figures in Table 4 represent statistically more significant loading estimates than have previously been available. Atmospheric wetfall loadings probably represent the major known inputs of identified compounds to Lake Superior other than PCBs; the major unknown loading is probably the dryfall (particulate and/or vapour phase) which is estimated as being at least as great as the contribution from wetfall.

A second aspect of the data which can be examined is the

differences between the two sites. Examination of the dates for the middle three samples from Caribou (Table 2) shows that they correspond very closely to the dates of the samples collected from Isle Royale (Table 1). Comparison of the results, therefore, is considered to be valid in evaluating the differences at the two ends of the lake - a distance of some 250 km. Both locations may be considered "open-lake" with the major differences presumed to arise from the fact that the prevailing air movement is west to east.

Comparisons using the Student t-Test showed that for virtually all of the instances where comparisons between the two locations can be made, there were significant differences between the data sets. All data for HCB, endrin, DDE and one set for PCBs did not meet a $p < 0.1$ criterion for significance and for HCB, endrin and DDE, the values for Caribou Island were low and near their detection limits. Examination of the direction of those differences which were significant (14 cases) showed that, except for one DDT and one methoxychlor set, Caribou Island received lower concentrations of all compounds in its rainfall. This reduction was an average of 65% (range 25 - 93%) although the figures were not consistent for the different compounds. The lower Caribou Island results probably reflect washout by rainfall or other removal mechanisms in the area between these two sites since comparable amounts of rain fell at the two locations themselves and the number of events was even slightly higher at Caribou Island. Isle Royale values may also have been higher due to contributions from particulate matter in the rain since this

location is closer downwind from the land mass than is Caribou Island.

Finally, the data can be looked at from a seasonal point of view. The data from Caribou Island in particular showed elevated concentrations of the two BHC isomers and of heptachlor epoxide during the period May 22-July 10; PCB's were highest in the April 21-May 22 period. A similar although less pronounced pattern was observed for the same compounds in the Isle Royale samples. Methoxychlor also showed increased springtime concentrations at Isle Royale although the behaviour was not apparent at Caribou Island. Other compounds did not have any distinct pattern of concentrations in the rain. The pattern of elevated springtime levels may be the result of increased usage of pesticides and their release to the atmosphere (globally, continentally and locally) during the spring and early summer months although this explanation would not serve for PCB's.

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Table 1 : Precision and Recoveries for Rain Sampling.

Compound	Recoveries			Mean Sampling Precision (% r.s.d.)
	Spike Level (ng)	Recovery (%)	Precision (% r.s.d.)	
a-BHC	61	46	8	20
lindane	26	65	9	16
Hept.Ep.	18	78	2	25
Dieldrin	—	—	—	23
Endrin	—	—	—	30
pp'-DDE	31	48	30	74
pp'-DDT	—	—	—	53
pp'-DDD	—	—	—	29
Toxaphene	150	73	24	—
M'ychlor	51	47	14	31
PCB's	170	44	18	48
HCB	—	—	—	54

Table 2 : Rain Data for Isle Royale, Lake Superior, 1983.

Mean values are ng/L; numbers in parentheses are percent relative standard deviations for triplicate samples.

Compound	My19-Jy2	Jy2-Sep4	Sep4-Oct15
a-BHC	65. (21)	40. (15)	19. (11)
Lindane	16. (26)	8.2 (14)	4.6 (13)
Hept.Ep.	1.3 (28)	nd*	0.4 (39)
Dieldrin	nd	1.3 (32)	0.6 (27)
Endrin	nd	nd	0.3 (43)
pp'-DDE	nd	0.7 (95)	nd
pp'-DDT	0.1 (50)	nd	nd
pp'-DDD	nd	nd	0.5 (50)
M'ychlor	7.4 (36)	1.5 (38)	1.3 (41)
PCB's	16. (28)	5.4 (62)	1.4 (53)
HCB	0.1 (60)	0.01 (100)	nd
Rain (L)	17.6	17.1	34.5
(mm)	84.	82.	165.

* - not detected.

Table 3 : Rain Data for Caribou Island, Lake Superior, 1983.

Mean values in ng/L; numbers in parentheses are percent relative standard deviations for triplicate samples.

Compound	Apr21-May22	May22-July10	July10-Sep5	Sep5-Oct10	Oct10-Nov22
a-BHC	9.2 (16)	46. (3)	3.9 (75)	5.8 (3)	4.6 (16)
Lindane	4.2 (16)	12. (5)	1.6 (27)	1.8 (9)	1.6 (14)
Hept.Ep.	0.5 (21)	0.6 (10)	0.1 (40)	nd*	0.1 (11)
Dieldrin	0.7 (22)	nd	0.3 (26)	0.2 (25)	0.3 (6)
Endrin	nd	nd	nd	0.09(16)#	nd
pp'-DDE	1.0 (53)	nd	0.02(75)	nd	nd
pp'-DDT	nd	0.2 (20)	0.2(110)	0.2 (40)	0.1 (43)
pp'-DDD	nd	nd	nd	0.05(29)#	0.33(9)
M'ychlor	3.3 (26)	4.1 (17)	0.4 (41)	2.6 (15)	1.4 (35)
PCB's	48. (21)	6.1 (56)	0.6 (50)	2.0 (75)#	2.1 (51)
HCB	nd	0.1 (12)	0.02(69)	0.4 (28)#	nd
Rain(L)	7.9	26.3	33.6	19.7	21.7
(mm)	38.	126.	161.	94.	104.

* - not detected.

- duplicate samples only.

Table 4 : Estimates of Rainfall Loadings of Organics to Lake Superior

Compound	Volume Weighted Rain Conc'ns* (ng/L)	Loadings via Rain & Snow@ (kg/yr)	Previous Estimates\$ Rain (kg/yr)	Dryfall (kg/yr)
a-BHC	17.4	260.	1000.	2300.
Lindane	5.9	290	330.	15600.
Hept.Ep.	0.35	17.	—	—
Dieldrin	0.56	28.	130.	410.
Endrin	0.085#	4.2	—	—
pp'-DDE	0.12#	5.9)	(	(
pp'-DDT	0.11	5.4) 17.	(330.	(250.
pp'-DDD	0.11#	5.4)	(	(
M'ychlor	2.4	120.	520.	7800.
PCB's	6.0	300.	2000.	7800.
HCB	0.075	3.7	130.	1600.

* - half the detection limit was used where no compound was detected.

@ - rain 580 mm; snowmelt 225 mm with concentrations per text; surface area of lake 82100 km².

\$ - reference SAB Appendix, 1980, table 27 for rain and Eisenreich et al., 1981, table 7 for total.

- less than half the samples reported this compound.

