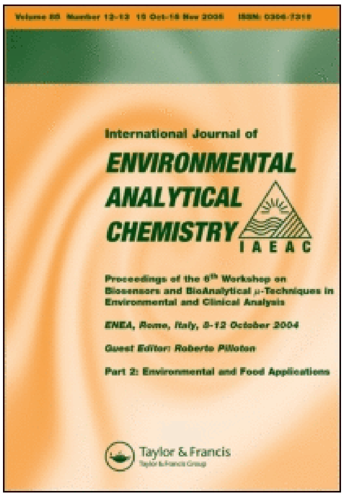

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Purgeable Organics in Great Lakes Raw and Treated Water

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Purgeable Organics in Great Lakes Raw and Treated Water

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The occurrence of 51 volatile organics in water supplies at nine municipalities along the Great Lakes and for a well water supply was examined by a GC-MS technique. Only dichloromethane (max. 19.0 μg/L) and chloroform (17.0 μg/L) were detected at concentrations above 10.0 μg/L and bromodichloromethane, chlorodibromomethane, dichloroacetonitrile, toluene, and styrene were detected at least once above 1.0 μg/L. Aggregate levels of the 19 organics detected at $\geqq 0.1$ μg/L were generally about the same in treated and raw water when values for trihalomethanes (THM) and dichloroacetonitrile were excluded. THM values did not exceed 32 μg/L. The aggregate frequency and the mean of aggregate organics levels were noticeably lower for raw water in winter than in summer or spring. Overall, the water treatment at the sites was not very effective in removal of relatively low levels (0.1 to 5 μg/L) for the volatile organics from raw water. There were no evident trends to the occurrence of organics along the Great Lakes system. Levels of organics in raw and treated water were generally about the same as, or lower than, those previously found in other Canadian surveys.

KEY WORDS: Purgeable organics, Great Lakes, water, raw, treated.

INTRODUCTION

Wide-spread occurrence of organics, many of anthropogenic origin, in North American water supplies has been demonstrated in several surveys.[1-3] Although only a part of the Great Lakes is heavily populated and industrialized, the potential for accumulation of hazardous substances in the water system is considerable. The potential health impact of such pollution is of concern to the many residents who make use of water from the basin. A potential source of exposure to the pollutants is through the drinking water and the Great Lakes Water Quality Board of the International Joint Commission has expressed concern for the protection of the drinking water sources.[4]

Several water quality studies[5] were initiated to aid in a program aimed at the risk assessment on human intake of organic pollutants in Great Lakes water. The present preliminary study was done to determine the levels and trends of selected purgeable organics along a portion of the Great Lakes system, including designated priority areas.[5] Both raw and treated water supplies were analyzed to determine the effect of treatment on levels of organics in drinkiing water and for comparison of results with those from other Canadian water supplies.

EXPERIMENTAL SECTION

Analyses

The preparation of amber glass bottles and control samples at Health and Welfare Canada (HWC) in Ottawa and the detailed procedures at the analytical laboratory in Toronto are described elsewhere.[6] Aliquots (100 mL) of control, raw, and treated water samples were analyzed by means of a Unacon 780B, automatic concentrator and using a purge and trap technique. The concentrator operating conditions were slightly modified to improve analytical results for the final, spring sampling period.[6] A Model 3200 Finnigan gas chromatograph-mass spectrometer (GC-MS) equipped with an INCOS MS 2000 data system was interfaced with the concentrator.

Sampling

Raw and treated water supplies at each of 10 potable water treatment plants (Figure 1), Barrie (A), Sault Ste. Marie (B), Windsor (C), Amherstburg (D), Fort Erie (E), Niagara Falls (F), Burlington (G), Hamilton (H), Kingston (I), and Cornwall (J), were sampled once during each of July–August, 1982, January–February, 1983, and

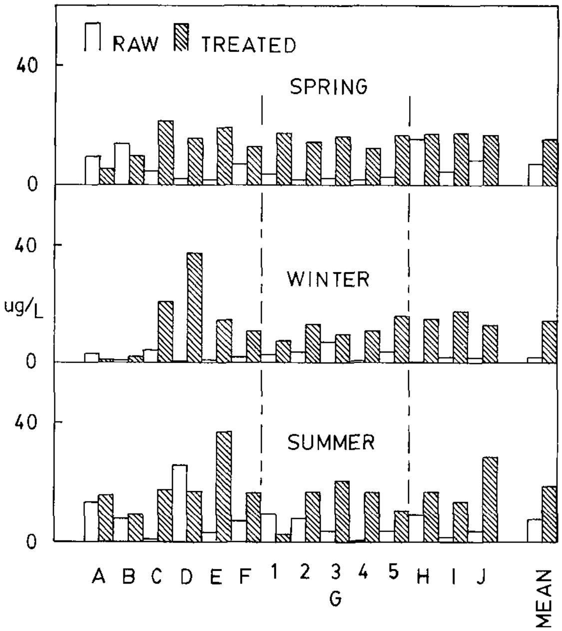

Figure 1 Seasonal aggregate organics levels (μg/L) in water supplies at 10 sites. Mean includes values for five days at Site G.

April–May, 1983. At plant G, water was sampled an additional four days to give five consecutive sampling days during each of the three seasons. Where possible, treated water grab samples were collected at the estimated in-plant detention time after raw water sampling. Raw water grab samples were collected from appropriate taps in the plants except at plants B (spring only) and F where they were collected directly from the St. Mary's and Niagara rivers, respectively, and near the plant intake. During summer and winter, the grab samples were collected near the end of a 24 hr sampling study[7] using XAD-2 cartridges at plants A, B, C, D, E and F. Information on water quality measurements and treatment practices at the 10 plants were recorded.

Empty sampling bottles and identical bottles containing the control samples were packed in coolers containing Freeze-Paks and were shipped, within 24 hr of preparation, by express freight to the sampling firm in Toronto. Blank water and spiked water control samples were shipped with the sampling bottles to and from the sampling sites.[6] At each plant, the first 5 L from a freshly opened tap were discarded and then four bottles each of raw and treated water were filled to a slight overflow. The bottles were immediately sealed, stored in coolers, and delivered within 24 hr to the analysis laboratory. The grab samples and control samples were always stored in inverted bottles in coolers with Freeze-Paks during shipping and at 4°C in the laboratory until about 1 hr before analysis. Usually, only one randomly selected sample from each quadruplicate set of grab samples and control samples was analyzed.

RESULTS AND DISCUSSION

Occurrence of organics

The 51 compounds which were monitored in this water quality survey are listed in Tables I and II. Their selection was based on reports of their occurrence in raw and treated water,[3,8-10] their designation as priority pollutants,[8] and their availability. A detailed evaluation of the purge and trap technique used in determination of these compounds in water is reported elsewhere.[6] However, comment on some of the technique capabilities is necessary for evaluation of these survey results.

Although detection limits[6] were equal to or better than 1 μg/L (usually 0.1 to 0.4 μg/L) for 45 compounds during the initial, summer period of the survey, poor detection limits were obtained for six compounds, i.e. 1,4-dioxane (150 μg/L), dichloroacetonitrile (80 μg/L), 2-chloroethylvinyl ether (80 μg/L), 1,1,2,3-tetrachloropropene (20 μg/L), acrolein (5 μg/L), and acrylonitrile (5 μg/L). Subsequently, the technique operating conditions were modified so that detection limits were generally well below 1 μg/L except for 1,4-dioxane and 2-chloroethylvinylether at 10 μg/L. For all survey samples, when MS analysis results were of sufficient quality, concentrations below the estimated detection limit and as low as 0.1 μg/L were calculated and reported.

Control samples consisting of purified water fortified with different combinations of the target organics were transported and analyzed with the raw and treated water samples to provide an estimate of the reliability of the survey results.[6] Usually, the average recoveries (rec.) ranged 90 ± 15% and the precision of recovery values was <20%

Table I Compounds not detected or found at <0.1 μg/L in 42 raw and 42 treated water samples

Compound formula	Frequency[a]		Compound formula	Frequency[a]	
	Raw	Treated		Raw	Treated
CCl_2F_2	0	0	$C_6H_5(C_2H_5)$	12	14
CCl_3F	0	0	$CHClCHCH_2Cl$[b]	0	0
CH_2CHCl	0	0	CH_2BrCH_2Cl	0	0
CH_3Cl	0	0	C_6H_5Cl	0	0
CH_3CH_2Cl	2	4	$CH_2ClCHCl_2$	0	0
CH_3Br	3	2	CH_2BrCH_2Br	0	0
CH_2CCl_2	0	1	C_6H_5Br	0	0
CH_2CHCH_2Cl	0	0	CCl_2CClCH_2Cl[b]	0	0
CH_2CHCHO	0	0	1,3-$C_6H_4Cl_2$	0	0
$CHClCHCl$[b]	1	0	$CHCl_2CCl_3$	0	1
CH_3CHCl_2	0	0	CCl_3CCl_3	0	0
CCl_4	1	1	$CHCl_2CHCl_2$	0	0
CH_2CHCN	0	0	1,2-$C_6H_4Cl_2$	0	0
$CH_3CHClCH_2Cl$	0	0	$CCl_2CClCClCCl_2$	0	0
CH_2CClCH_2Cl	0	0	1,2,4-$C_6H_3Cl_3$	0	0
$CH_2ClCH_2OCHCH_2$	0	0	1,4-$(CH_2CH_2O)_2$	0	0

[a] Frequency of occurrence at trace levels (see text).
[b] *cis* and *trans* isomers determined (see text).

RSD for individual compounds in replicate control samples and in the nominal concentration range of 5 to 50 μg/L[6]. Compounds which showed precision values >30% RSD and very poor accuracy (average recoveries <55% and >115%) in at least one replicate control sample set were trichlorofluoromethane (386% rec. at 7.0 μg/L), 1,2-dichloroethane (32% RSD, 127% rec. at 11.7 μg/L), 1,3-dichloropropene (43% RSD, 10% rec. at 15.5 μg/L; 38% RSD, 28% rec. at 10.8 μg/L), 1,1-dichloroethylene (124% rec. at 7.9 μg/L), vinyl chloride (50% rec. at 33.8 μg/L), chloroethane (42% rec. at 32.9 μg/L), 2-chloroethylvinylether (0% rec. at 47.4 μg/L), 1,4-dichlorobenzene (123% RSD, 35% rec. at 10.6 μg/L), and bromoform (116% rec. at 15.3 μg/L). The limitations in quantitation of the above compounds at concentrations near the detection limit should be considered during the following discussion of survey results.

Table I lists the 32 compounds which either were not detected or were only found at trace (<0.1 μg/L) levels in water samples from

Table II Detection frequency (f)[a] and mean concentration (μg/L) of 19 compounds at ≧0.1 μg/L

Compound formula	Summer				Winter				Spring			
	Raw		Treated		Raw		Treated		Raw		Treated	
	f	μg/L	f	μg/L	f	μg/L	f	μg/L	f	μg/L	f	μg/L
CCl_2FCClF_2	2^{3}	<0.1	2^{7}	<0.1	3^{1}	<0.1	1^{2}	<0.1	3^{9}	<0.1	2^{11}	<0.1
CS_2	0^{4}	—	0^{4}	—	0^{1}	—	0^{2}	—	2^{11}	—	4^{10}	—
CH_3CCl_3	0	—	0	—	0	—	0	—	7^{7}	<0.1	8^{5}	<0.1
CH_2Cl_2	12^{1}	2.5	13^{1}	1.7	11^{1}	0.7	12^{2}	1.5	14	2.6	14	1.4
C_6H_6	14	0.3	13	0.2	4^{4}	<0.1	3^{5}	<0.1	3^{10}	<0.1	2^{12}	<0.1
$CHClCCl_2$	1	<0.1	1	<0.1	2	<0.1	2	<0.1	6^{7}	0.1	5^{8}	0.1
$CHCl_3$	12	3.4	14	8.5	10^{2}	0.7	14	6.8	14	1.3	14	5.8
CCl_2CCl_2	0	—	0^{1}	—	1	<0.1	0	—	1^{1}	<0.1	1^{1}	<0.1
$C_6H_5(CH_3)$	4^{10}	0.3	7^{7}	<0.1	7^{6}	0.1	10^{4}	0.3	13^{1}	0.5	13^{1}	0.7
CH_2ClCH_2Cl	1^{1}	<0.1	1^{1}	<0.1	0	—	0	—	2	<0.1	2^{1}	<0.1
$1,2\text{-}C_6H_4(CH_3)_2$	0^{1}	—	0^{1}	—	0^{2}	—	0^{3}	—	2^{9}	<0.1	0^{11}	—
$1,3\text{-}C_6H_4(CH_3)_2$	0	—	0^{2}	—	1^{8}	<0.1	1^{7}	<0.1	5^{7}	<0.1	7^{6}	<0.1
$1,4\text{-}C_6H_4(CH_3)_2$	0^{1}	—	0^{1}	—	0^{3}	—	1^{5}	<0.1	1^{7}	<0.1	0^{7}	—
$C_6H_5(CHCH_2)$	4^{4}	<0.1	5^{5}	0.1	8^{6}	0.2	8^{5}	0.3	14	0.5	13^{1}	0.5
$CHBrCl_2$	1	0.2	12	4.4	0	—	12^{1}	2.8	1^{2}	0.3	13	4.1
$CHBr_2Cl$	1	<0.1	12	1.8	0	—	12	1.4	1^{1}	0.1	12^{1}	1.9
$CHBr_3$	0	—	0	—	0	—	0^{1}	—	0	—	7^{4}	0.1
$1,4\text{-}C_6H_4Cl_2$	0^{7}	—	0^{9}	—	2^{12}	<0.1	2^{11}	<0.1	5^{6}	<0.1	4^{8}	<0.1
$CHCl_2CN$	0	—	0	—	0	—	0	—	0	—	7	0.3
Total	52^{34}	>6.7	80^{39}	>16.7	49^{46}	>1.7	78^{48}	>13.1	94^{78}	>5.4	128^{87}	>14.9

[a] Frequency of occurrence at ≧0.1 μg/L and at trace levels (superscript).

the 10 sites and for the three seasons. The frequency of detection includes the occurrences in the total of 15 raw and 15 treated water samples collected at site G (Burlington) over 5 consecutive days during each of the three seasons. Only ethyl benzene was detected frequently at trace levels and then chiefly only in winter and spring, probably due to the somewhat improved detection limits for this period. It should be noted that 5 of the 6 compounds with relatively high detection limits are listed in Table I. The *cis* and *trans* isomers for 1,2-dichloroethylene, 1,3-dichloropropene. and 1,1,2,3-tetrachloropropene were determined separately.

Although 26 of the 51 compounds were detected at least once in the raw and treated water samples, seven of these were found only at trace levels (Table I). The other 19 compounds which are listed in Table II were detected at least once at a concentration $\geqq 0.1\,\mu g/L$, but only seven of these were found at levels $\geqq 1\,\mu g/L$, i.e. dichloromethane, chloroform, bromodichloromethane, chlorodibromomethane, toluene, styrene, and dichloroacetonitrile. The only compounds which were detected at levels $>10\,\mu g/L$ were dichloromethane (max. $19\,\mu g/L$) in one raw and one treated water sample from site D and chloroform (max. $17\,\mu g/L$) in six treated water samples. Toluene, styrene, dichloroacetonitrile, and benzene were the only other compounds with mean concentrations $>0.1\,\mu g/L$ (Table II). The first three were found at levels $>1\,\mu g/L$ in a few water samples. For at least one water type and season, the mean of concentrations of all 10 sites (Table II) was $>1\,\mu g/L$ for only the first four of these seven compounds. Dichloromethane and chloroform were found in almost all raw and treated water samples, although chloroform levels were considerably greater in treated water. Bromodichloromethane and chlorodibromomethane were frequently detected in treated water and seldom in raw water. The trihalomethanes (THM) which include these two compounds, chloroform, and bromoform are known to be formed, often together with dichloroacetonitrile, during chlorination of water.[11,12] Chlorination was used at all 10 treatment plants. Levels of the THM were well below the limits of $100\,\mu g/L$ and $350\,\mu g/L$ recommended by the U.S. E.P.A. and Health and Welfare Canada, respectively, and below the $30\,\mu g/L$ limit for chloroform recommended by the World Health Organization. The maximum THM level was found in treated water during summer at Fort Erie ($32\,\mu g/L$). Dichloroacetonitrile (max. $1.5\,\mu g/L$) was detected only in treated water samples collected at sites C, D, E, G, I and J during the spring and would not have been detected at similar levels during the preceeding summer when the detection limit was worse than 50 μg/L. Concentrations ranging up to 8.1 μg/L were previously detected in Southern Ontario drinking water.[12]

All the organics listed in Table II, except 1,2-dichloroethane and 1,4-xylene, were detected at least once at $\geqq 0.1$ μg/L during extended monitoring at Burlington (G). Similar to the results for all sites (below) six of these compounds, i.e. dichloromethane, chloroform, toluene, bromodichloromethane, chlorodibromomethane, and styrene were detected at least once at >1.0 μg/L. Within each five day monitoring period and water type, the precision of values was usually better than 100% RSD but seldom better than 20% RSD for each compound with a five day mean concentration value $\geqq 0.1$ μg/L. Similarly, there was a relatively wide variation in aggregate organics values over each five day period (Figure 1). However, when its five day mean concentration was >1.0 μg/L, a particular compound was detected in the corresponding water type on each day. Poor precision for values near the detection limit could be expected.

Aggregate values for the organics detected at each site are summarized for each season in Figure 1. Site A (Barrie) was included in this study to allow comparison of results from the nine sites on the Great Lakes with those from a well water supply near the Great Lakes. No trends are evident from examination of the histograms for raw and treated water along the Great Lakes from Sault Ste. Marie (B) to Cornwall (J). When the three season mean of the aggregate levels was calculated for each site, there also were no noticeable trends. However, it was noted that the mean values for raw water were greatest at Amherstburg (D) and Hamilton (H) but these values were reduced, respectively, from 9.1 to 7.0 μg/L and from 8.3 to 1.9 μg/L when THM values were excluded. Somewhat surprisingly, Sault Ste. Marie (7.3 μg/L), which is upstream from many major pollution sources, and Barrie (8.6 μg/L) where water is obtained from a well, had the next highest values. Fort Erie (E, 1.8 μg/L), which is downstream, albeit far, from Detroit/Windsor, had the lowest mean value. For treated water, the three season mean of aggregate values was highest at Windsor (C, 20.0 μg/L), Amherstburg (23.2 μg/L), and Fort Erie (23.4 μg/L) and was lowest at Barrie (7.1 μg/L) and Sault Ste. Marie (6.8 μg/L).

The aggregate levels of identified purgeable organics were generally considerably higher in treated than in raw water. Results from Barrie and Sault Ste. Marie were exceptions in that the mean of aggregate levels for raw and treated water were similar. These were the only two sites where water treatment did not include filtration and consisted only of chlorination and addition of sodium silicate (Barrie) or ammonia (Sault Ste. Marie). When the values for THM and dichloroacetonitrile were excluded, the adjusted aggregate levels of the remaining organics were generally $<4\,\mu g/L$ for each water type and at all the individual sites. The contribution of chlorination to the identifiable purgeable organics load in treated water was emphasized by the observation that the adjusted aggregate levels for treated water were generally about the same as the corresponding raw water values.

The aggregate frequency of occurrences for all detected compounds and the mean values for the aggregate organics levels are listed in Table III. As summarized in Table II, the frequency of occurrences for all compounds was greater in treated than in raw water and the total of frequencies was considerably greater in spring than in summer or winter. However, when the four trihalomethanes (THM) and dichloroacetonitrile were excluded, the frequency of occurrences for the remaining compounds was similar in treated and raw water samples. The mean of aggregate levels of organics in treated water was greater than $10\,\mu g/L$ and considerably greater than the corresponding raw water values. Also, the mean of aggregate levels for raw water was considerably lower in winter than in spring or summer and for treated water was about the same for all

Table III Aggregate frequency (f)[a] and mean of aggregate organics levels ($\mu g/L$) at 10 sites

Water type	Summer		Winter		Spring		3 seasons	
	f	$\mu g/L$	f	$\mu g/L$	f	$\mu g/L$	f	$\mu g/L$
Raw	52^{34}	7.6	49^{46}	1.5	94^{78}	7.1	195^{158}	5.4
Treated	80^{39}	18.4	78^{48}	14.0	128^{87}	15.2	286^{174}	15.9
Raw[b]	38^{32}	3.8	39^{44}	0.8	78^{75}	4.9	155^{151}	3.2
Treated[b]	42^{39}	2.3	40^{47}	2.5	75^{82}	3.1	157^{168}	2.6

[a] Frequency at trace levels (>0 and $<0.1\,\mu g/L$) noted by superscripts.

[b] Excluding values for THM and dichloroacetonitrile (see text).

three seasons. Parallel to the result with frequency of occurrences, when THM and dichloroacetonitrile values were excluded, the mean values for aggregate levels were reduced considerably, i.e. to $<5\,\mu g/L$, and the result was similar raw and treated water values.

As determined by linear regression analysis of the data, there were few correlations between values for raw and treated water for any of the detected organics. The relationship between the occurrence of a few organics in raw and treated water was evident upon visual examination of the data. Dichloromethane, benzene, chloroform, toluene, styrene, and 1,4-dichlorobenzene occurred frequently, although sometimes at trace levels, in matching raw and treated water samples and generally during all three seasons. Also, carbon disulfide, 1,1,2-trichlorotrifluoroethane, trichloroethylene, and the 1,2- and 1,3-xylenes were detected frequently at low levels in matching pairs of raw and treated water samples collected in spring. Some relationships between raw and treated water data were found when linear regression was done for all occurrences at $\geqq 0.1\,\mu g/L$. Correlation coefficients were generally $<+0.75$ when calculations included data for all compounds. Also, poor correlation was usually obtained for a set of data when values were near $0.1\,\mu g/L$. If values for the THM and dichloroacetonitrile, i.e. compounds formed during treatment, were excluded, correlation coefficients $>+0.75$ were often obtained. The strong relationships between the occurrence of organics in raw and treated water during summer and spring was evidence for incomplete treatment at sites A ($r=+0.99$, $+0.94$; slope (treated ÷ raw) $=+0.99$, $+0.25$) and B ($r=+1.00$, $+0.99$; slope $+0.41$, $+0.30$). Some relationships between the occurrence of organics in treated and raw water were observed for the data from all sites during spring ($r=+0.76$, slope $=+0.33$), sites A, B, C, E, F, G, I and J during spring ($r>+0.84$, slope $+0.25$ to $+2.38$), site G, I and J during winter ($r>0.86$, slopes $+0.21$ to $+1.50$) and sites A, B, D, E, F, H and I during summer ($r>+0.98$, slope $+0.02$ to $+4.20$). These results, those for some individual organics, and those for adjusted aggregate values (Table III) indicated that the treatment processes at the plants were not effective in removal of relatively low levels (0.1 to $5\,\mu g/L$) of many of the purgeable organics. An in-depth statistical analysis of the occurrence of volatile organics in 30 Canadian raw and treated water supplies[13] showed similar seasonal trends and relationships as in this study of the Great Lakes basin.

Other Canadian surveys

As in this survey of purgeable organics, no evident trends along the Great Lakes were noted for aggregate levels of organics determined by the XAD-2 sampling technique[7] which was used concurrent with the summer and winter grab sampling at sites A, B, D, E, G and J. With the exception of unusually high levels of polynuclear aromatic hydrocarbons (PAH) at site B, which were also noted during a similar survey in 1980,[14] aggregate levels of PAH, phosphate triesters (TAAP), and organochlorine (OC) compounds were generally lower at sites A and B than at the other four sites. As discussed earlier, this was not the case for the purgeable organics. As compared to purgeable organics levels which were generally higher in treated water and somewhat higher in summer, the aggregate OC levels were generally greater in treated water and OC and TAAP levels were generally greater in summer than in raw water and winter. However, the PAH levels were generally highest in raw water during winter. These trends with PAH, OC, and TAAP can not be readily attributed to water treatment practices as can be done with THM trends.

The 51 purgeable compounds determined in this study included 36 of the 43 organics investigated in a survey of raw and treated water at 30 treatment plants across Canada during August–September and November–December in 1979.[3] Fourteen of the 19 compounds detected at least once at $\geqq 0.1\ \mu g/L$ in this study (Table II) were also detected in the 30 plant survey. From the remaining five compounds, 1,1,2-trichlorotrifluoroethane, carbon disulfide, styrene, and dichloroacetonitrile were not determined in 1979 and 1,1,1-trichloroethane was determined but was not detected. Although 27 compounds were detected at least once during the 30 plant survey, only eight compounds showed a mean concentration $>1\ \mu g/L$ for at least one water type and season. Only dichloromethane, chloroform, and bromodichloromethane from these eight compounds, which also included 1,2-dichloroethane, 1,2-dichloroethylene, trichloroethylene, benzene, and toluene, had mean concentrations $>1\ \mu g/L$ in the present study. Generally, maximum values of organics were greater in the survey of 30 Canadian treatment plants than in this Great Lakes basin survey. Chloroform and bromodichloromethane occurred frequently in treated water in both surveys. Concentrations of THM were considerably greater in the 30 plant survey and in a THM survey at three treatment plants[15] than in this survey. Benzene, toluene, and dichloromethane were detected frequently in both raw and treated water in the Great Lakes and 30 plant surveys. The mean values for aggregate levels of purgeable organics in both raw and treated water from across Canada were considerably higher (ca. 2–5 times) than corresponding values from the Great Lakes basin, with consideration that the sets of detected compounds were not identical for the two surveys. The seasonal variation in levels of THM and other organics, i.e. lowest levels in winter has been more noticeable in the 30 plant study and a 13 month, THM monitoring study.[15] Inefficient removal of organics and an occasional increase in organics levels during water treatment was indicated by the results in both the Great Lakes and the 30 plant studies.

During January 1980, using procedures similar to those in the 30 plant survey, purgeable organics were determined in raw and treated water from 12 Great Lakes municipalities[14] including sites B, D and I. Chloroform, bromodichloromethane, chlorodibromomethane, and toluene were frequently detected in treated water and at levels similar to those found in this survey. Xylene isomers were also detected quite frequently in treated water but the levels were occasionally considerably higher, i.e. 4.4–17 μg/L, than in this study.

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