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SEWAGE TREATMENT PLANT INFLUENTS,
EFFLUENTS AND SLUDGES ACROSS
CANADA IN 2003**

R.J. Maguire and S.P. Batchelor

NWRI Contribution No. 04-391



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**Occurrence of Organotin Species in Sewage Treatment
Plant Influent, Effluents and Sludges across Canada in
2003**

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Abstract

Influents, effluents and sludges from eleven Canadian sewage treatment plants (STPs) in 2003 were analyzed for fourteen organotin species. Butyltin species (tributyltin, dibutyltin and monobutyltin) were found frequently in STP influents, likely because of a combination of pesticidal and non-pesticidal uses. The butyltin species were fairly efficiently removed in STPs, rarely appearing in effluents. However, they were concentrated in sewage sludges, to which they had adsorbed. Methyltin species (dimethyltin and monomethyltin) were only found infrequently in STP influents, effluents and sludges. Octyltin species (dioctyltin and monoctyltin) were only found in sludges. No other organotin species were found. It is concluded that organotin species are fairly well eliminated from effluents of Canadian STPs, largely through adsorption to sludge. More intensive study is required to derive mass balances and removal efficiencies of organotin species in Canadian STPs with confidence. The persistence, fate and toxicological significance of organotin residues in sewage sludge are other areas that deserve attention.

Résumé

Nous avons analysé les influents, les effluents et les boues dans onze stations canadiennes d'épuration d'eaux usées, en 2003, en vue d'y déceler la présence de quatorze composés organostanniques. Des butylétains (tributylétain, dibutylétain et monobutylétain) ont été décelés fréquemment dans les influents des stations d'épuration; ils provenaient vraisemblablement de produits pesticides et non pesticides. L'élimination de ces substances par les stations d'épuration est passablement efficace, puisqu'elles ne se trouvent que rarement dans les effluents. Elles se concentrent toutefois dans les boues d'épuration, où elles se fixent par adsorption. Les méthylétains (diméthylétain et monométhylétain) ne se trouvent que rarement dans les influents, les effluents et les boues. Les octylétains (dioctylétain et monoctylétain) ne se trouvent que dans les boues. Aucun autre organostannique n'a été détecté. Nous en concluons que l'élimination des organostanniques par les stations d'épuration canadiennes est passablement efficace, et qu'elle dépend de l'adsorption dans les boues d'épuration. Il convient toutefois d'entreprendre des études plus poussées pour établir les bilans massiques et mesurer avec certitude l'efficacité de l'élimination des composés organostanniques dans les stations canadiennes d'épuration d'eaux usées. La persistance, le devenir et l'importance au plan toxicologique des résidus d'organostanniques dans les boues d'épuration méritent également d'être étudiés.

Introduction

Organotin compounds are an important class of organometallic chemicals that have found extensive use as pesticides and industrial chemicals. The most notable organotin species from an environmental viewpoint is tributyltin (TBT). TBT compounds are primarily used as antifouling pesticides, wood and material preservatives, and slimicides (for reviews on TBT and other organotin compounds, see Maguire, 1991; Environment Canada and Health and Welfare Canada, 1993; Fent, 1996; de Mora, 1996; Champ and Seligman, 1996; World Health Organization, 1999a,b; Maguire, 2000; Meador, 2000; Champ, 2000; Omae, 2003). TBT is perhaps the most toxic chemical that has ever been deliberately introduced into the aquatic environment (through its antifouling use). As a consequence of its extremely high aquatic toxicity, many countries regulated antifouling uses of TBT in the 1980s or early 1990s (for a detailed discussion see Chau et al., 1997 and Maguire, 2000). Later, the International Maritime organization proposed a prohibition on the application of organotin antifouling paints by January 1, 2003, and a prohibition on their presence on ship hulls by 2008 (see Maguire, 2000). Canada supported the resolution, and put in place a similar regulation (Pest Management Regulatory Agency, 2000).

It was of interest to determine whether TBT enters the Canadian environment through pesticidal uses other than antifouling uses, e.g., as slimicides in cooling towers, or wood and material preservatives. Over the past decade our group has investigated the presence and trends of TBT and non-pesticidal organotin species in sewage treatment plant (STP) influents, effluents and sludges. This contribution adds to our database by reporting on concentrations of 14 organotin species in influents, effluents and sludges from 11 STPs across Canada in 2003. In addition to TBT, the following 13

organotin species were determined using our multi-residue method: monomethyltin (MMT), dimethyltin (DMT), trimethyltin (TMT), tripropyltin (TPrT), monobutyltin (MBT), dibutyltin (DBT), mono-octyltin (MOT), dioctyltin (DOT), monophenyltin (MPT), diphenyltin (DPT), triphenyltin (TPT), dicyclohexyltin (D-c-HT), and tricyclohexyltin (T-c-HT). All these compounds in aqueous media are present as cations or in complex forms, depending upon the nature and concentration of other solutes (e.g., Laughlin et al. 1986; Maguire 1987, 1991). For brevity, they are referred to in this report as though they exist only in cationic form.

Materials and Methods

Reagents

The carrier gas for the gas chromatograph - flame photometric (GC-FPD) system used in the organotin analyses was high purity helium, 99.999%, from Canox Ltd. (Mississauga, Ontario). The reagent gases, zero air (<0.1 ppm hydrocarbon) and hydrogen (99.99999 + %) were provided by generators in the laboratory (Whatman 76-803 and Whatman 75-34, respectively), (Parker Hannifin Corporation, Tewksbury, MA, U.S.A.). All monobutyltin trichloride, dibutyltin dichloride, tributyltin chloride, tripropyltin chloride, dicyclohexyltin dichloride and triphenyltin chloride (which was used as the internal standard) standards were obtained from Alfa Products (Ward Hill, MA, U.S.A.). Mono-octyltin trichloride, dioctyltin dichloride, monomethyltin trichloride, dimethyltin dichloride, trimethyltin chloride, triphenyltin chloride, tropolone (2-hydroxy-2,4,6-cycloheptatrien-1-one) and ethylmagnesium bromide (3.0 M in diethylether) were obtained from Aldrich Ltd. (Milwaukee, WI, U.S.A.). Diphenyltin dichloride and monophenyltin trichloride were obtained from Gelest, Inc. (Tullytown, PA, U.S.A.). All solvents, acids and

common laboratory reagents were of analytical grade. Distilled water, further purified by passage through a NANOpure system (Barnstead Thermolyne, Dubuque, IA, U.S.A.), was used throughout. Stock solutions of organotin compounds (1000 mg/L as Sn) were prepared in methanol or in toluene. All glassware was solvent-rinsed before use. The sodium sulfate and aluminum foil used to line the tops of the sediment jars were fired at 450 °C before use.

Sample Collection

Samples of influent, effluent and sludge were collected from 11 sewage treatment plants (STP) across Canada in 2003. Influent and effluent samples (3 L) were acidified immediately to pH 1 with hydrochloric acid that had been solvent-washed to remove organic contaminants. All liquid samples were shipped to our laboratory where they were kept at 4 °C in the dark before extraction. Sludge samples (2 L) were not pretreated prior to shipment.

Sample Extraction

The determination of organotin species in STP samples basically follows methods established at the National Water Research Institute in the early 1980s. Since then some changes have been made to improve the methods, or simply for convenience. Brief procedures are described here for each type of sample matrix.

Influent and effluent samples (1 L volume) previously acidified to pH 1 were extracted by shaking in 2 L Teflon separatory funnels with 50 mL of 0.1% (w/v) tropolone in toluene after the addition of 200 μ L of a tripropyltin chloride solution (1.019 mg Sn/L, MeOH) as surrogate standard (Chau et al. 1994). After separation of the solvent phase, the water was again extracted

with 25 mL of 0.1% tropolone/toluene. The two toluene fractions were combined and dried by passage through anhydrous sodium sulfate, evaporated under reduced pressure in a rotary evaporator at 30 °C to 2-3 mL, transferred to a test tube, and finally evaporated just until dry by a stream of nitrogen, and reconstituted with 1 mL of hexane. Ethylmagnesium bromide solution (0.3 mL, 3.0 M) was added and the mixture was allowed to stand for about 10 min. The excess ethylmagnesium bromide was destroyed by shaking for 1 min. with 2 mL of 0.5 M H₂SO₄. Interfering substances were removed by a silica gel clean-up with a Pasteur pipette mini-column (15 cm x 5 mm i.d., containing approximately 1 g of silica gel). The extract was applied to the column and eluted with 5 mL of hexane, then reduced under nitrogen at room temperature to 1 mL before analysis (Chau et al. 1996). Spike recovery experiments were not done for the influent and effluent samples. However, spike recovery experiments showed that the recoveries of all species spiked into natural waters at 1 µg Sn/L each were in the range 87-107% (Chau et al. 1994).

Sludge samples were extracted by a slightly modified procedure. After the addition of 200 µL of a solution of triphenyltin chloride (1.019 mg Sn/L) as internal standard and 20 mL of concentrated acetic acid to 100 mL of sludge sample, the mixture was left inside a fume cupboard overnight for digestion and evolution of gases. After the addition of 40 g of NaCl and 20 mL of a 0.5% (w/v) solution of tropolone in toluene, the mixture was magnetically stirred for 1 hr. and then transferred to a Teflon bottle for centrifugation at 3000 r.p.m. for 15 min. An aliquot (10 mL) of the extract was transferred to a test tube and evaporated almost to dryness at 35 °C with a stream of nitrogen. The volume was adjusted to 1 mL with hexane for ethylation with ethylmagnesium bromide as described in the preceding section. The clean-up procedure for sludge required slight modifications in that a larger column was

used. The column (15 cm x 1.5 cm i.d.), containing about 6 g of silica gel was packed from bottom to top in the following order: glass wool, 1 cm layer of anhydrous sodium sulfate, silica gel bed, and 1 cm layer of anhydrous sodium sulfate. All 14 organotin species sought could be quantitatively eluted with 40 mL of hexane. After reduction of the volume to 2 - 4 mL under reduced pressure in a rotary evaporator at 30 °C, the extract was transferred to a test tube and evaporated at room temperature to 1 mL in a stream of nitrogen, and stored in an autosampler vial. The overall recoveries of these compounds spiked into sludge at 1 µg/g dry weight were satisfactory (74-102%) except for the three methyltin species (9-56%) and DPT (56%) (Chau et al. 1994). Concentrations reported in this study were not corrected for recovery.

Analysis

Sample extracts after derivatization were analyzed for organotin species with a GC-FPD/MSD system from Hewlett-Packard (HP - Avondale, PA, U.S.A.), consisting of a gas chromatograph (HP 5890, Series II) equipped with a split/splitless injection port, flame photometric and mass selective detectors, and an autosampler (HP 7673A). The system was controlled with HP G1701AA ChemStation software. Operating parameters for the GC-FPD/MSD system are listed in Table 1. Standard mixtures of the ethyl derivatives of all 15 organotin species (including the triphenyltin surrogate standard) in the expected concentration ranges were prepared and used to calibrate detector responses. Quantitation was by peak height response vs. external standards. All concentrations of organotin species in this article are expressed as Sn. Chromatographic "windows" were typically 0.04 min. at most at 15 min. retention time. The presence of an organotin species was taken to be tentatively confirmed if (i) it occurred within the appropriate chromatographic

window, and (ii) the concentrations were above the limit of quantitation (LOQ) for the particular sample, defined here as three times the noise level. LOQ values for the organotin species were in the range 5-12 ng Sn/L for a 1 L sample of influent or effluent, and in the range 50-120 ng Sn/L for a 100 mL sample of liquid sludge (100 mL of liquid sludge was roughly equivalent to 2 g of dry sludge). Reagent blanks were analyzed according to the methods given above. No contamination was evident.

Results and Discussion

Table 2 shows concentrations of the butyltin species in influent, effluent and sludge at 11 sewage treatment plants (STP) across Canada in 2003. The butyltin species were found frequently in influent samples at concentrations up to 29, 61 and 55 ng Sn/L for TBT, DBT and MBT, respectively. It is difficult to comment definitively on the effectiveness of the sewage treatment processes because the influent and effluent samples were grab samples, and no provision was made for time of travel through the STP. However, if the influents and effluents represent a steady state, it can be stated that there is practically complete elimination of butyltin species from the effluent. The butyltin species were all found at relatively high concentrations in sewage sludge, indicating strong partitioning to the solid phase. The highest concentrations of butyltin species were found in influent and sludge from a Toronto STP. Although there was apparently complete removal of butyltin species from the effluent of that STP, the ultimate fate and effects of butyltin species in sludge are not clear. Concentrations of the butyltin species in sludge from the Toronto STP were similar to those observed by Lee et al. (2004). These results are also in general similar to those observed for samples collected in 1993 and 1994 (Chau et al. 1997) although there is significant variability in concentrations of butyltin species in particular STPs at particular

times. The concentrations are, however, much lower than concentrations found in Canadian STPs in the early 1990s (Chau et al. 1992a,b). The reason for this discrepancy is not known. Further intensive study of STPs is required in order to derive mass balances and removal efficiencies with confidence.

The source of the butyltin species in STP influents is not clear. The presence of TBT may be ascribed to non-antifouling pesticidal uses, e.g., in material preservation. It is known that DBT and MBT compounds are used as stabilizers in PVC, and that they can be leached from PVC pipes by water (Environment Canada and Health and Welfare Canada 1993). However, their presence could also be ascribed to the degradation of TBT.

Dimethyltin and monomethyltin were found infrequently in STP influents, effluents and sludges in 2003 (see Table 3), in contrast to findings from the 1993-1994 survey, and trimethyltin was not found at all. The highest concentrations were found in Edmonton in the 2003 and 1993-1994 surveys, but the DMT concentration in Edmonton STP influent in 2003 (62 ng Sn/L) was much lower than the figure of 454 ng Sn/L found in 1993-1994. Its presence may be due to leaching from DMT-stabilized PVC tubing (or from chemical industries manufacturing PVC with methyltin stabilizers). There was in general a significant, but not complete, reduction in concentrations of each methyltin species in STP effluents. The DMT and MMT species were found in sludges at much lower concentrations than the butyltin species. Further intensive study of STPs is required in order to derive mass balances and removal efficiencies of methyltin species with confidence.

DOT and MOT compounds are commonly used as stabilizers for PVC for food and beverage packaging (Environment Canada and Health and Welfare Canada 1993). DOT and MOT were not found in STP influent or effluent in

this survey (in 1993-1994 they were found infrequently). However, they were found frequently in STP sludges, as shown in Table 4. The frequency of occurrence and concentrations of DOT and MOT in 2003 were similar to the 1993-1994 survey. DOT and MOT concentrations in sludge were generally much lower than concentrations of butyltin species.

Other Organotin Species

No other organotin species analyzed for were found in STP influents effluents and sludges.

In conclusion, it appears that organotin species are fairly well eliminated from effluents of Canadian STPs, largely through adsorption to sludge. More intensive study is required to derive mass balances and removal efficiencies of organotin species in Canadian STPs with confidence. The persistence, fate and toxicological significance of organotin residues in sewage sludge are other areas that deserve attention.

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Table 1. Operating conditions for GC-FPD/MSD

GC parameters

injection port	splitless
injection port temperature	250 °C
injection volume	1 µL
injection purge time	1 min.
carrier gas	He, constant flow mode, 10 p.s.i. at 50 °C
temperature program	50 °C for 1 min., then 20 °C/min to 280 °C, with 2.5 min. final hold

FPD parameters

column	DB5, 30m x 0.25 mm i.d., film thickness 0.25 µm
combustion gases	H ₂ and air, each at 100 mL/min.
detector temperature	200 °C

MSD SIM parameters

column	HP -5MS, 30m x 0.25 mm i.d., film thickness 0.25 µm
detector temperature	280 °C
electron impact mode	
ionization potential	70 eV
source temperature	190 °C

species (ethyl derivatives)	ions monitored
MMT	135, 165, 193
DMT	151, 135, 179
TMT	165, 135, 151
MBT	121, 149, 179
TPrT	121, 149, 193
DBT	121, 149, 179
MPT	120, 197
TBT	121, 149, 177
MOT	179, 149, 121
DPT	121, 197
D-c-HT	149, 179
DOT	149, 121, 179
TPT	120, 197
T-c-HT	149, 233

Table 2. Concentrations of butyltin species in influent, effluent and sludge from some Canadian sewage treatment plants in 2003*																
Location	Influent (ng Sn/L)				Effluent (ng Sn/L)				Sludge (as liquid) (ng Sn/L)				Sludge (dried) (ng Sn/g dry weight)			
	TBT	DBT	MBT	TOTAL	TBT	DBT	MBT	TOTAL	TBT	DBT	MBT	TOTAL	TBT	DBT	MBT	TOTAL
Burnaby	d	d	12.5 ± 0.7	12.5			d		477.4 ± 42.0	1316.4 ± 509.6	1181.4 ± 99.6	2975	21.6 ± 1.9	59.4 ± 23.0	53.3 ± 4.5	134.3
Edmonton (Gold Bar)	d	8.4 ± 0.2	15.5 ± 1.9	23.9			d		1298.7 ± 157.6	1855.2 ± 370.5	1732.3 ± 516.1	4886	65.5 ± 8.0	93.6 ± 18.7	87.4 ± 26.0	246.5
Regina	10.6± 1.6	20.7 ± 7.0	21.9 ± 0.3	53.2			d		544.3 ± 77.0	977.2 ± 54.2	1036.0 ± 88.7	2558	20.2 ± 2.9	36.3 ± 2.0	38.5 ± 3.3	95.0
Winnipeg (North End)	10.2± 1.2	23.5 ± 0.5	36.5 ± 0.6	70.2			6.6 ± 1.0	6.6	1577.2 ± 102.1	3823.8 ± 388.7	3442.7 ± 233.7	8844	49.5 ± 3.2	120.1 ± 12.2	108.1 ± 7.3	277.7
Sarnia	11.3± 0.9	d	d	11.3			d		1310.2 ± 31.9	1088.1 ± 1458.3	1211.1 ± 968.8	14509	53.4 ± 1.3	44.4 ± 59.5	494.0 ± 39.5	591.8
Hamilton	d	10.3 ± 3.5	16.8 ± 5.7	27.1					2705.2 ± 264.8	3351.7 ± 516.1	2373.0 ± 344.7	8430	79.8 ± 7.8	98.8 ± 15.2	69.5 ± 10.2	248.1
Toronto (Main)	28.7± 2.8	61.4 ± 13.3	55.2 ± 20.5	145.3		d	d		3058.1 ± 128.9	8746.1 ± 840.3	4129.6 ± 137.2	15934	146. ± 6.2	418.1 ± 40.2	197.4 ± 6.6	761.7
Montréal	8.1 ± 0.6	8.2 ± 1.3	23.1 ± 6.9	39.4	d	d	11.5 ± 0.2	11.5	1485.3 ± 104.7	1789.2 ± 140.7	1826.5 ± 208.6	5101	50.4 ± 3.6	60.7 ± 4.8	62.0 ± 7.1	173.1
Saint John (Millidgeville)	d	d	12.0 ± 0.6	12.0		d			519.4 ± 35.8	1829.8 ± 96.8	1309.9 ± 122.9	3659	32.7 ± 2.3	115.2 ± 6.1	82.5 ± 26.6	230.4
Charlottetown	5.0 ± 0.5	d	12.0 ± 1.8	17.0	d	d	d		314.8 ± 46.1	589.3 ± 257.7	578.9± 317.0	1483	12.1 ± 1.8	22.7 ± 9.9	22.3 ± 12.2	57.1
Bedford	d	d	12.3 ± 1.6	12.3	d		d		1245.9 ± 92.3	1672.5 ± 96.8	2702.2 ± 368.1	5621	50.6 ± 3.7	67.9 ± 3.9	109.6 ± 14.9	228.1

*TBT - tributyltin, DBT - dibutyltin, MBT - monobutyltin; d - detected, but below limit of quantitation; ± values are standard deviations. Influent, effluent and sludge samples were divided into three subsamples and analyzed separately.

Table 3. Concentrations of dimethyltin and monomethyltin in influent, effluent and sludge from some Canadian sewage treatment plants in 2003*								
Location	Influent (ng Sn/L)		Effluent (ng Sn/L)		Sludge (as liquid) (ng Sn/L)		Sludge (dried) (ng Sn/g dry weight)	
	DMT	MMT	DMT	MMT	DMT	MMT	DMT	MMT
Burnaby	d	d						
Edmonton	62.1 ± 1.9	32.1 ± 5.9	31.7 ± 6.1	45.0 ± 9.5	213.9 ± 30.3	204.2 ± 65.3	10.8 ± 1.5	10.3 ± 3.3
Regina	26.2 ± 2.6		d					
Winnipeg	17.2 ± 2.5		8.0 ± 0.1		d		d	
Sarnia	d		8.4 ± 0.4	d	147.6 ± 43.0	140.6 ± 28.7	6.0 ± 1.8	5.7 ± 1.2
Hamilton	d							
Toronto	10.2 ± 2.4	d	d	d	d		d	
Montréal	8.1 ± 0.6	d	d	d				
Saint John		d						
Charlottetown	d	d	d					
Bedford	d	d			d		d	

*DMT – dimethyltin, MMT – monomethyltin; d – detected, but below limit of quantitation; ± values are standard deviations. Influent, effluent and sludge samples were divided into three subsamples and analyzed separately.

Table 4. Concentrations of dioctyltin and monoctyltin in sludge from some Canadian sewage treatment plants in 2003*				
Location	Sludge (as liquid) (ng Sn/L)		Sludge (dried) (ng Sn/g dry weight)	
	DOT	MOT	DOT	MOT
Burnaby	85.8 ± 16.1	119.8 ± 12.3	3.9 ± 0.7	5.4 ± 0.6
Edmonton	98.2 ± 25.9	110.9 ± 45.3	5.0 ± 1.3	5.6 ± 2.3
Regina				d
Winnipeg	94.5 ± 14.6	139.0 ± 29.1	3.0 ± 0.5	4.4 ± 0.9
Sarnia		477.4 ± 65.4		19.5 ± 2.7
Hamilton			d	
Toronto	114.4 ± 23.3	197.8 ± 33.2	5.5 ± 1.1	9.5 ± 1.6
Montréal	116.7 ± 27.5	157.4 ± 2.3	4.0 ± 0.9	5.3 ± 0.1
Saint John	140.3 ± 3.6	148.4 ± 5.7	8.8 ± 0.2	9.3 ± 0.4
Charlottetown			d	d
Bedford	174.9 ± 15.7	424.4 ± 100.2	7.1 ± 0.6	17.2 ± 4.1

*DOT - dioctyltin, MOT - monoctyltin; d - detected, but below limit of quantitation; ± values are standard deviations. Sludge samples were divided into three subsamples and analyzed separately.

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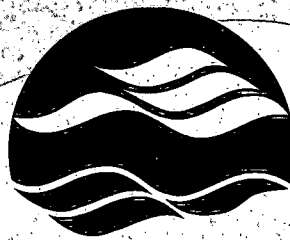
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