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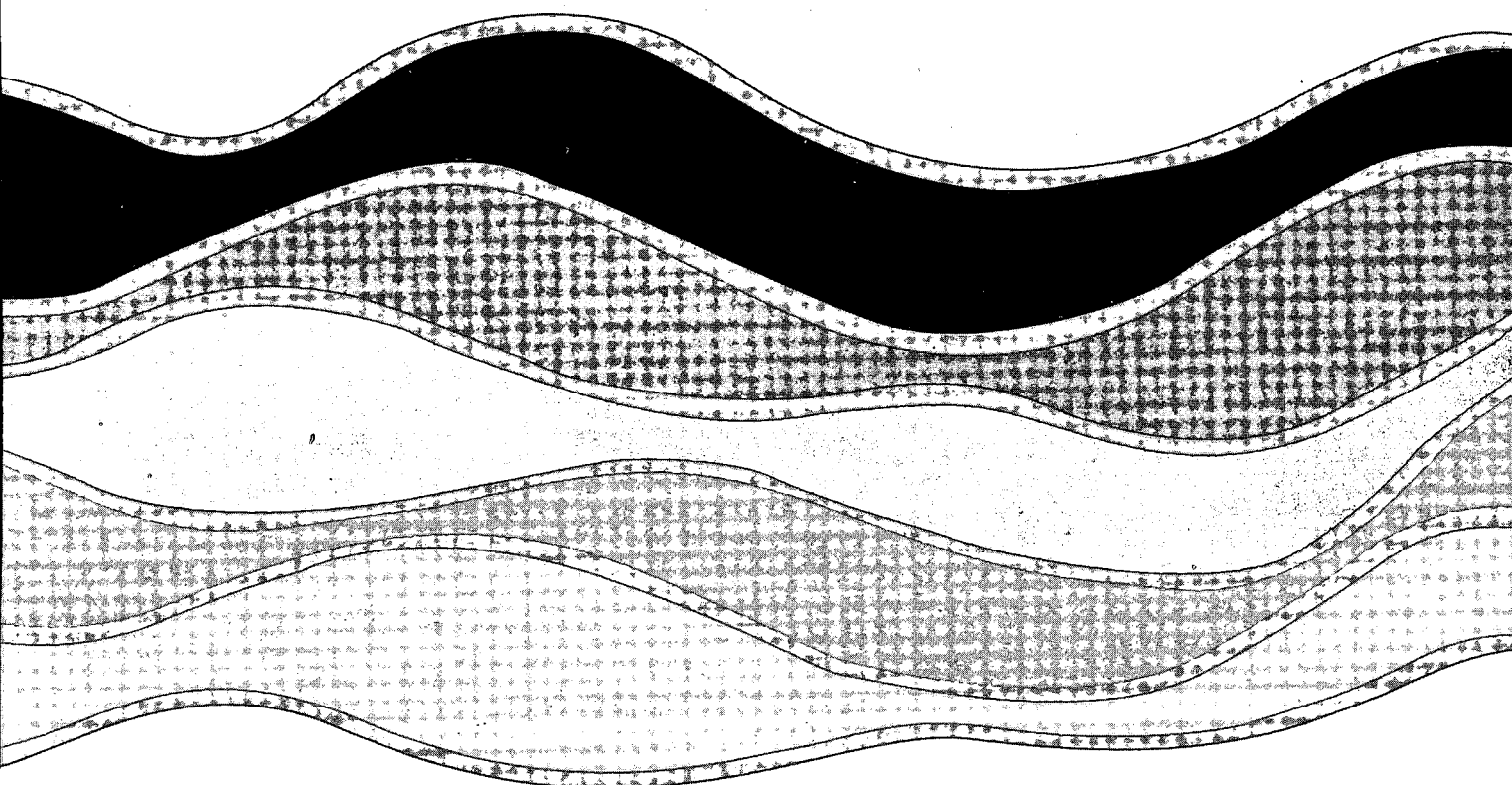
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**OCCURRENCE OF BUTYLTIN SPECIES IN
SEWAGE AND SLUDGE IN CANADA**

Y.K. Chau, S. Zhang
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NWRI Contribution No. 91-74

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OCCURRENCE OF BUTYLTIN SPECIES IN SEWAGE AND SLUDGE IN CANADA

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MANAGEMENT PERSPECTIVE

The overall objectives of this program are (i) to assess the occurrence of tributyltin in sewage treatment plant influent, effluent and sludges as a result of uses other than antifouling use, and (ii) to assess the occurrence of non-pesticidal organotins in sewage treatment plant samples. The former objective contributes to DOE's pesticide responsibilities, while the latter objective is work identified in the CEPA assessment of non-pesticidal organotins. This study was supported in part by PESTFUND and CEPA funding.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Les objectifs généraux du présent programme sont i) l'évaluation de la présence de tributylétain dans les affluents, les effluents et les boues de l'usine de traitement des eaux usées due à l'emploi d'autres agents antisalissures, et ii) l'évaluation de la présence de composés organostanniques autres que des pesticides dans les échantillons d'une usine de traitement des eaux usées. Le premier objectif fait partie des responsabilités du ministère de l' Environnement en ce qui concerne les pesticides, tandis que le deuxième objectif se rapporte aux travaux définis dans l'évaluation des composés organostanniques autres que des pesticides dans le cadre de la LCPE. La présente étude a été subventionnée en partie par le PESTFUND et des fonds provenant de la LCPE.

ABSTRACT

Samples of sewage treatment influent, effluent and sludges collected monthly from five Canadian cities over the period of July 1990 to January 1991 were analyzed for butyltin and octyltin species. Monobutyltin was found in all influent samples, but dibutyltin and tributyltin were found only infrequently, methyltin and octyltin species were not found at all. In the case of monobutyltin, there was significant reduction in its concentration by degradation and adsorption to sludge during passage through the sewage treatment plant. The average reduction was 40%. The monobutyltin found in the effluent likely came from its use as a poly(vinyl chloride) stabilizer, and from the degradation of tributyltin, which is used as a slimicide. No methyltins, butyltin or octyltin species was found in five landfill leachate samples in southern Ontario during the same period.

RÉSUMÉ

Des échantillons provenant des affluents, des effluents et des boues d'usines de traitement des eaux usées recueillis mensuellement dans cinq villes canadiennes, entre juillet 1990 et janvier 1991, ont été analysés afin de déceler la présence d'espèces de butylétain et d'octylétain. Tous les échantillons des affluents contenaient du monobutylétain, mais rarement du dibutylétain et du tributylétain, et jamais d'espèces de méthylétain et d'octylétain. Dans le cas du monobutylétain, on a relevé une baisse importante de sa concentration par dégradation et adsorption à des boues lors du passage dans l'usine de traitement des eaux usées. La réduction moyenne était de 40%. Le monobutylétain présent dans les effluents provenait probablement de son utilisation comme stabilisateur du polychlorure de vinyle, et de la dégradation du tributylétain utilisé comme myxobactéricide. Aucun des échantillons de lixiviat prélevés dans cinq décharges du sud de l'Ontario pendant la même période ne contenait de méthylétain, d'espèces de butylétain ou d'octylétain.

INTRODUCTION

Organotin compounds are important industrial commodities which are used as poly(vinyl chloride) stabilizers, industrial catalysts, industrial and agricultural biocides, and wood preserving and antifouling agents (Thompson *et al.*, 1985). The increasing use of organotin compounds, some of which are very toxic, attracted the attention of environmental agencies in a number of countries in the 1970s. Organotin compounds as a class were placed on Canada's Environmental Contaminants Act Category III list in 1979 (Canada Department of Environment and Department of Health and Welfare, 1979), which meant that further information was required on their occurrence, persistence and toxicity in order to make environmental and human health risk assessments. The main organotin compounds that are likely to be currently released to the environment in Canada are those of methyltin, dimethyltin, n-butyltin, di-n-butyltin, tri-n-butyltin, n-octyltin, di-n-octyltin and hexakis(β,β -dimethylphenylethyl)stannoxane. The mono- and di-methyltins, butyltins and octyltins are used mainly as poly(vinyl chloride) stabilizers. Tri-n-butyltin is an antifouling agent whose use in Canada is now strictly regulated under the Canadian Pest Control Products Act. It can also be used as a general lumber preservative and as a slimicide in cooling towers, but its use as a net preservative is not allowed. Hexakis(β,β -dimethylphenylethyl)stannoxane is a miticide. In addition to these compounds, triphenyltin may be introduced to coastal and harbour areas in Canada through its use as an antifouling agent on vessels painted in other countries. It is not a registered antifoulant in Canada.

Tributyltin has been the organotin compound of most concern because its use as an antifouling agent results in direct contact with aquatic environments, and because of its high toxicity to aquatic organisms. Lethal concentrations are in the range 0.04 - 16 $\mu\text{g Sn/L}$ for short-term exposures of aquatic organisms (*e.g.*, Maguire, 1987; World Health Organization, 1990). Over the past ten years an extensive investigation has been made of the occurrence and persistence of tributyltin and its less toxic degradation products dibutyltin and monobutyltin in water and sediment in Canada (Maguire, 1987, and references therein). Tributyltin was mainly found in areas of heavy boating or

shipping traffic, which was consistent with its use as an antifouling agent. In about 8% of the 269 locations across Canada at which samples were collected, tributyltin was found in water at concentrations which could cause chronic toxicity in a sensitive species, rainbow trout. It was also found in about 30% of sediment samples collected across Canada. Biological degradation in water and sediment appears to be the most important factor limiting the persistence of tributyltin in aquatic ecosystems. Estimates of the half-life of biological degradation of tributyltin in fresh water and sediment in Canada are in the range of a few weeks to 4-5 months, respectively.

In 1989 the Canadian government regulated tributyltin under the Pest Control Products Act (Canada Department of Agriculture, 1989). There is a maximum daily release rate of 4 μg of tributyltin per square cm of hull surface, and tributyltin is prohibited on vessels of less than 25 m in length, with the exception of those with aluminum hulls. These regulations should minimize the environmental impact of antifouling uses of tributyltin in Canada. As indicated above, other registered uses for tributyltin in Canada are as a general lumber preservative and as a slimicide in cooling towers. It was therefore of interest to determine if significant amounts of tributyltin were being released to the environment through these uses. Accordingly, this article reports the results of an investigation of the occurrence of tributyltin, dibutyltin, monobutyltin and other non-pesticidal organotins (mono- and dimethyl, mono- and dioctyltin) in landfill leachates and in sewage treatment plant influent, effluent and sludge from several Canadian cities, and the effectiveness of their removal in sewage treatment plants. Recent reports from Switzerland have shown substantial concentrations of tributyltin in sewage treatment plant influents (Fent, 1989; Fent and Muller, 1991).

EXPERIMENTAL

Materials and Methods

Monobutyltin trichloride, dibutyltin dichloride and tributyltin chloride were obtained from Alfa Products, Ward Hill, MA. Tropolone and ethylmagnesium bromide (2.0 M in tetrahydrofuran) were from Aldrich, Milwaukee, WI. All solvents were of pesticide grade; water was distilled and deionized. Standard butyltin solutions (1000 $\mu\text{g/mL}$ as Sn) were prepared by dissolving appropriate amounts of butyltin compounds in water. The internal standard addition technique was used for the determination of the concentration of butyltin.

The sewage samples (acidified to pH 1 and cooled to 4 °C) were allowed to warm to room temperature inside a fume cupboard. After complete gas evolution a 200 mL aliquot was extracted as described below. Sludge samples (100 mL) were taken from a well-mixed suspension. After addition of 10 mL of conc. HCl, the samples were placed inside a fume cupboard until gas evolution was complete, and the pH was adjusted to 1-2.

The butyltin and Sn(IV) compounds in sewage and sludge were extracted in a mechanical shaker for 4 hr with 20 mL of 0.5% tropolone in toluene after the addition of 60 g of NaCl. The extracted analytes were derivatized to their ethyl derivatives ($\text{R}_4\text{Et}_4\text{Sn}$) and cleaned up on a 5% water-deactivated silica gel glass column (15 cm x 1.5 cm dia.). The organotin compounds were eluted with 35 mL of hexane at 0.7 mL/min. After reduction of the eluent volume to 0.5 mL in a rotary evaporator and a vortex evaporator, the ethyl derivatives of the various butyltin and octyltin were determined by gas chromatography-atomic absorption spectrometry (GC-AAS). Pasteur pipets (15 cm x 5 mm dia.) packed with the same silica gel could be conveniently used as columns for sewage samples which contained relatively less amounts of organic matter. In this case, the column was eluted with 25 mL of hexane at a rate of 0.7 mL/min. As sludge contains a more complex matrix, the capacity of the clean-up column and the volume of the eluent must be investigated for a particular type of sludge.

Samples were analyzed in duplicate. Detection limits for sewage and sludge are 40 ng Sn/L and 2 ng Sn/g (dry wt.), respectively. Details of the analysis have been given previously (Chau *et al.*, 1991).

Sewage influent, effluent and sludge samples were collected from five municipal treatment plants. The influent samples were taken from the sedimentation tanks and the effluent samples were taken after the final treatment step. The Toronto (Humber Plant) and Hamilton plants are equipped with secondary treatment, whereas the plants at Montreal, Vancouver, and Sarnia are primary treatment plants. Samples were collected at monthly intervals from July 1990 to January 1991. Sewage samples were acidified with 2 mL of conc. HCl/L immediately after collection and stored in brown-colored glass bottles. Sludge samples were not treated, but stored in brown-colored glass bottles, loosely capped, and transported on ice. Samples received in the laboratory were stored in the dark at 5°C before analysis. Analyses were normally carried out within one week after receipt.

Landfill leachate samples were all collected in Ontario in the holding lagoons of the following municipal landfill sites:

- Sample 1. Essex Sanitary Landfill Site. No. 1, located in part of lots 14, concession 7, Colchester North.
- Sample 2. Essex County Sanitary Landfill Site No. 2, located in parts of lots 12 and 13, concession 2, Eastern Division and Gosfield South.
- Sample 3. Essex County Sanitary Landfill Site No. 3, located in parts of lots 14 and 15, concession 4 in Maidstone.
- Sample 4. Landfill site in Guelph, Ontario.
- Sample 5. Landfill site in Muskoka, Ontario.

Samples were stored in brown-colored glass bottles in the dark at 5°C until analysis. They were analyzed in the same manner as for sewage.

RESULTS

Table 1 shows concentrations of the three butyltin species and acid-extractable inorganic Sn in the influent and effluent of sewage treatment plants in the five Canadian cities in the period July 1990 to January 1991. No methyltin or octyltin species were found. Tributyltin was only found in 1 of 36 influent samples, which was taken in Montreal in July 1990, at 5.2 $\mu\text{g Sn/L}$. It was not found in the plant effluent sampled the same day, or in of the other 35 effluent samples. The concentration of 5 $\mu\text{g Sn/L}$ is significantly higher than the highest concentration (0.2 $\mu\text{g/L}$) observed in sewage treatment plant influent in Zurich, Switzerland (Fent and Muller, 1991), a city of population 350,000. Dibutyltin was found in only 3 influent samples and 1 effluent samples, at concentrations of 1 - 2 $\mu\text{g Sn/L}$, concentrations equal to or slightly higher than the range observed for the Zurich sewage treatment plant. In contrast to tributyltin and dibutyltin, monobutyltin was found in all 36 influent and effluent samples. Concentration ranges were 1.9 -20.6 $\mu\text{g Sn/L}$ for influent and 0.7 - 14.5 $\mu\text{g Sn/L}$ for effluent. In 25 of the 36 cases, there was a significant reduction in concentration in passing through the sewage treatment plant, but in no case was there total elimination of monobutyltin.

Table 2 shows concentrations of the three butyltin species and inorganic tin in sludge from the sewage treatment plants collected at the same time as the influent and effluent samples. No octyltin species were found. Tributyltin was found in 9 of the 36 samples, with a range of 5 - 278 $\mu\text{g Sn/kg}$. This range is a little lower than that observed for sludge from the Zurich sewage treatment plant (280 - 1500 $\mu\text{g/kg}$, Fent and Muller, 1991). Tributyltin was not found in any of the sludge samples from Vancouver and Hamilton, but was found in 2 of 7 samples each from Toronto and Montreal, and 5 of 7 samples from Sarnia. The higher frequency of occurrence of tributyltin in sludge from the Sarnia sewage treatment plant may reflect its use as a slimicide in cooling towers of this heavily-industrialized city, the petrochemical center for Canada. Dibutyltin was found in 7 of 36 sludges, with a range of 11 - 305 $\mu\text{g/kg}$. Monobutyltin was found in 6 of 36 sludges, with a range of 16 - 440 $\mu\text{g/kg}$. Both of these ranges are equal to or lower

than those observed for sludge from the Zurich sewage treatment plant (Fent and Muller, 1991). Acid-extractable inorganic Sn (IV) was found in every sludge sample, at much higher concentrations than the butyltin species, in the range 1 - 222 mg Sn/kg.

There were no methyltin, butyltin or octyltin species in any of the five landfill leachates analyzed, but the Essex (1) and Essex (3) samples contained 3.7 and 3.0 μg Sn/L of acid extractable Sn(IV), respectively.

DISCUSSION

None of the organotin compounds sought in this study was found in the landfill leachate samples analyzed. Octyltin species were not found in sewage treatment plant influents and effluents either, but tributyltin and dibutyltin were found infrequently, and monobutyltin was found in every influent and effluent sample analyzed. Its removal in the sewage treatment process did not appear to be as efficient as was observed in the Zurich sewage treatment plant, whose primary, secondary and tertiary effluents showed averages of 73, 90 and 98% butyltin species removal, respectively (Fent and Muller, 1991). In this study, of the 25 cases in which there was significant reduction in monobutyltin concentration in passing through the sewage treatment plant, the average reduction was 40%, with a range of 19 - 75%. However, effluent concentrations of monobutyltin less than 20 μg Sn/L will probably pose no hazard to aquatic organisms since the effluent will be diluted greatly, and toxicity of monobutyltin to aquatic organisms has only been observed at much higher concentrations. For example, EC_{50} values are 15 mg/L for algae (Wong *et al.*, 1982), 25 mg/L for bacteria (Dooley and Kenis, 1987) and 49 mg/L for *Daphnia magna* (Vighi and Calamari, 1985), and the 48-hr LC_{50} value for red killifish is 44 mg/L (Nagase *et al.*, 1991).

Monobutyltin found in the sewage treatment plant influent likely comes from two sources: (i) its use as a PVC stabilizer, along with dibutyltin, and (ii) the use of tributyltin as a slimicide. Monobutyltin has been shown (Boettner *et al.*, 1981) and inferred (Schebek *et al.*, 1991) to be a product of the aqueous leaching of PVC pipe. In addition, the finding of monobutyltin and dibutyltin in areas where tributyltin is used as

an antifoulant is well documented (World Health Organization, 1990). The finding of monobutyltin in all influent samples in which very little dibutyltin or tributyltin were found suggests that its origin in these sewage treatment plants is in PVC stabilization. However, the finding of tributyltin, dibutyltin and monobutyltin species together in most of the sludge samples taken from Sarnia is strong evidence for tributyltin use in this city, perhaps as a slimicide. The fact that dibutyltin and tributyltin were found in sludge in Sarnia, but not in sewage influent and effluent samples collected on the same day, may be explained by the fact that these species are concentrated by adsorption on sludge (Fent and Muller, 1991).

Even though tributyltin was rarely found in this study, it should be noted that the discharge of untreated municipal wastewater can be a source of tributyltin in aquatic ecosystems. Even with efficient removal of tributyltin by adsorption onto sludge, little is known of its biological availability or effects if the sludge is spread on land. Further research is necessary in this regard.

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Table 1. Concentration of Butyltin and Sn(IV) in Sewage Samples

Sample	Sn ⁴⁺		BuSn ³⁺		Bu ₂ Sn ²⁺		Bu ₃ Sn ⁺	
	Infl.	Effl.	Infl.	Effl.	Infl.	Effl.	Infl.	Effl.
Montreal								
Jul 90	-	-	8.2±0.2	5.8±0.7	1.4±0.4	-	5.2±0.1	-
Aug 90	-	-	1.9±0.3	0.7±0	-	-	-	-
Sep 90	-	-	4.0±0.3	2.8±0.4	-	-	-	-
Oct 90	-	-	13.3±0.6	5.5±0.9	-	-	-	-
Nov 90	3.2±0.6	-	9.6±1.4	8.0±0.1	-	-	-	-
Jan 91	3.0±0	-	7.0±0.1	4.5±0.1	-	-	-	-
Feb 91	1.3±0	1.8±0.1	20.6±0.8	12.2±0.4	2.1±0.1	-	-	-
Toronto								
Jul 90	-	-	8.8±0.1	9.1±0	-	-	-	-
Aug 90	-	-	10.7±0.4	2.7±0.3	-	-	-	-
Sep 90	-	-	10.7±0.7	8.7±0	2.4±0.1	2.0±0.3	-	-
Oct 90	-	-	9.1±0.7	6.0±1.0	-	-	-	-
Nov 90	2.1±0.4	-	6.8±0.5	6.6±0	-	-	-	-
Dec 90	-	-	10.3±0.6	5.2±0.4	-	-	-	-
Jan 91	4.1±0.1	-	9.1±0.6	5.6±0.2	-	-	-	-
Hamilton								
Jul 90	-	-	8.8±0.1	5.8±0.7	-	-	-	-
Aug 90	-	-	8.4±0.7	6.3±0	-	-	-	-
Sep 90	-	-	13.7±0.9	6.9±0.1	-	-	-	-
Oct 90	2.5±0.2	-	13.8±0.1	13.1±0.8	-	-	-	-
Nov 90	5.2±0.5	2.1±0.1	13.6±0.3	10.4±1.4	-	-	-	-

Dec 90	27.5±2.1	9.2±1.2	5.7±1.1	9.5±0.2	-	-	-	-
Jan 91	4.0±0.5	2.2±0.2	11.4±0.4	7.0±0.1	-	-	-	-

Sarnia

Jul 90	-	-	9.4±0.4	5.0±0.4	-	-	-	-
Aug 90	-	-	5.3±0.3	3.2±0.2	-	-	-	-
Sep 90	-	-	3.4±0.3	3.3±0.3	-	-	-	-
Oct 90	-	-	14.0±0.4	14.5±0.9	-	-	-	-
Nov 90	5.6±1.0	-	10.7±0.4	11.3±0.5	-	-	-	-
Dec 90	-	-	5.0±0.4	1.3±0.3	-	-	-	-
Jan 91	1.8±0	-	5.5±1.1	2.3±0.1	-	-	-	-

Vancouver

Jun 90	-	-	11.3±0.7	7.7±0.1	-	-	-	-
Jul 90	-	-	4.4±0.1	2.9±0.2	-	-	-	-
Aug 90	-	-	4.7±0.6	5.3±0	-	-	-	-
Sep 90	-	-	4.8±0.5	2.8±0.1	-	-	-	-
Oct 90	-	-	6.7±0.6	6.9±0.1	-	-	-	-
Nov 90	-	-	8.3±0.1	6.6±0.6	-	-	-	-
Jan 91	-	-	6.3±0.5	6.4±0.3	-	-	-	-
Feb 91	-	-	11.1±0.2	10.7±0.7	-	-	-	-

Results are average of duplicate analysis, given in µg/L as Sn; detection limit for sewage, 40 ng/L; (-) not detected.

Table 2. Concentration of Butyltin and Sn(IV) in Sludge Samples

Location	Time	Sn ⁴⁺ (µg/g)	BuSn ³⁺ (µg/Kg)	Bu ₂ Sn ²⁺ (µg/Kg)	Bu ₃ Sn ⁺ (µg/Kg)
Montreal	July 90	4.4 ± 0	-	-	-
	Aug 90	7.2 ± 0.2	-	-	-
	Sept 90	3.3 ± 0.6	-	-	-
	Oct 90	26.2 ± 2.4	-	-	234.5 ± 24.2
	Nov 90	5.5 ± 0.2	-	-	-
	Jan 91	2.6 ± 0.2	28.8 ± 4.2	-	174.3 ± 2.9
	Feb 91	11.9 ± 0.2	-	-	-
Toronto	July 90	4.4 ± 0.5	-	-	-
	Aug 90	6.5 ± 0.1	-	-	-
	Sept 90	9.3 ± 0.3	-	-	-
	Oct 90	10.3 ± 0.3	-	-	-
	Nov 90	15.0 ± 1.0	-	-	-
	Dec 90	24.0 ± 0	440.0 ± 30	210.0 ± 35	277.5 ± 22.5
	Jan 91	19.5 ± 0.5	-	305.0 ± 5	245.0 ± 10
Hamilton	July 90	144.5 ± 33.4	-	-	-
	Aug 90	222.2 ± 3.7	-	-	-
	Sept 90	192.6 ± 7.4	-	-	-
	Oct 90	205.6 ± 5.6	-	-	-
	Nov 90	118.5 ± 7.4	-	-	-
	Dec 90	40.7 ± 0	-	-	-
	Jan 91	57.5 ± 5.6	-	-	-

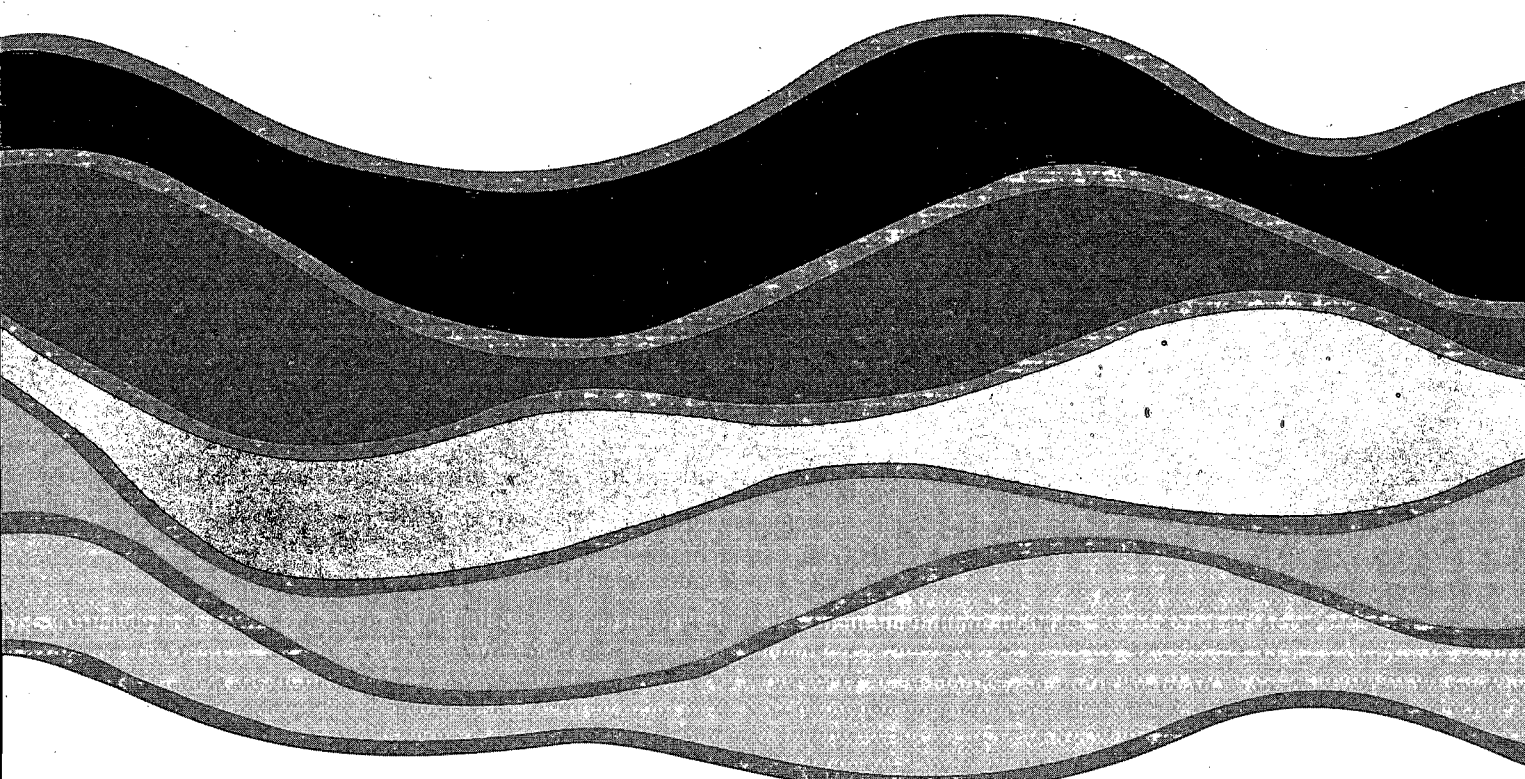
Sarnia	July 90	5.5 ± 0	31.0 ± 0	15.9 ± 2.3	5.0 ± 0.1
	Aug 90	2.9 ± 0	-	-	-
	Sept 90	5.8 ± 0.3	-	20.5 ± 0.5	130.5 ± 2.3
	Oct 90	3.7 ± 0.1	22.7 ± 0.9	61.0 ± 4.6	175.0 ± 2.3
	Nov 90	2.1 ± 0.2	18.2 ± 1.8	10.5 ± 0.5	49.6 ± 1.4
	Dec 90	3.2 ± 0.4	16.4 ± 0	35.0 ± 0.5	45.5 ± 2.8
	Jan 91	1.1 ± 0.1	-	-	-
Vancouver	June 90	12.5 ± 0	-	-	-
	July 90	5.2 ± 0	-	-	-
	Aug 90	6.4 ± 0.6	-	-	-
	Sept 90	3.7 ± 0.3	-	-	-
	Oct 90	5.5 ± 0.3	-	-	-
	Nov 90	9.2 ± 0.9	-	-	-
	Jan 91	2.0 ± 0.1	-	-	-
	Feb 91	5.9 ± 0	-	-	-

Results are average of duplicate analysis, expressed as Sn on dry wt. basis; detection limit for sludge, 2 ng/g; (-) - not detected.

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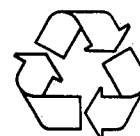


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