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Organochlorine contaminants in sediments of the Tauranga Harbour, New Zealand

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Abstract DDT, DDE, and DDD (up to 0.5 ng g^{-1} per compound) and traces of dieldrin ($< 0.1 \text{ ng g}^{-1}$), were detected in surficial sediments collected from four sites in the greater Tauranga Harbour. An elevated level of PCBs (19.9 ng g^{-1}) was identified in a Waikareao Estuary sample. PCB and DDT levels of up to 24.1 ng g^{-1} and 5.38 ng g^{-1} , respectively, were subsequently detected in other eastern shore Waikareao Estuary sediment samples. Higher levels of PCBs (68.6 and 73.8 ng g^{-1}) and DDTs (total 7.52 and 19.2 ng g^{-1}) were detected in sediments from two eastern shore stormwater drains identified as the likely sources of the PCB and DDT burden entering the Waikareao Estuary. Black, carbonaceous, coal- and asphalt-like, particulate material (98% loss of organic carbon on ignition), isolated by sieving a representative sediment sample, exhibited DDT and PCB levels which were respectively 85 and 6 times higher than was the case for other sieved fractions.

Keywords organochlorine compounds (OCCs); polychlorinated biphenyls (PCBs); DDT; DDE; DDD; gas chromatography; Tauranga Harbour; Waikareao Estuary.

INTRODUCTION

Organochlorine compounds (OCCs) are a major class of organic environmental contaminants that

are routinely detected in almost all environmental compartments, including: human adipose tissue and milk (Smith et al. 1990; Kutz et al. 1991), sea water and sediment (Colombo et al. 1990), fish and shellfish (Swackhammer & Hites 1988; Niimi & Oliver 1989) and birds (Bishop & Weseloh 1990) around the world. Their toxicity, environmental persistence, and bioaccumulation potential, are the major factors warranting their study in the environment.

DDT, its degradation products (DDE, DDD), and polychlorinated biphenyls (PCBs) are OCCs over which much controversy has arisen. In recent years, national and regional authorities, and the public in general, have become increasingly concerned about the levels of OCCs in the New Zealand aquatic environment. The recent discovery of significant levels of pentachlorophenol (PCP) in sediments collected from timber treatment sites throughout New Zealand (Szabo 1993) demonstrates that this concern is with good cause.

In 1988 a comprehensive survey of levels of OCCs in the sediments of the Manukau Harbour (Fox et al. 1988) found levels of PCBs, DDTs, hexachlorocyclohexanes (HCHs), dieldrin, endrin, aldrin, and chlordanes, comparable to heavily populated areas outside New Zealand (The Environmental Agency, Japan 1983; Phillips & Spies 1988). The Manukau Harbour findings prompted the Bay of Plenty Regional Council to have sentinel oyster samples from four sites within Tauranga Harbour analysed for several organochlorine contaminants. Trower & Holland (1991) subsequently reported the presence of low levels of a range of OCCs throughout the harbour.

We now report the results of an investigation of the OCC levels of some sediment samples from Tauranga Harbour. This harbour is a large tidal estuary with an area of approximately 200 km^2 . It is located within the Bay of Plenty on the east coast of the North Island, at latitude $37^\circ 39'S$, longitude $176^\circ 11'E$. The harbour has a varied hinterland of industrial, urban, and rural catchments.

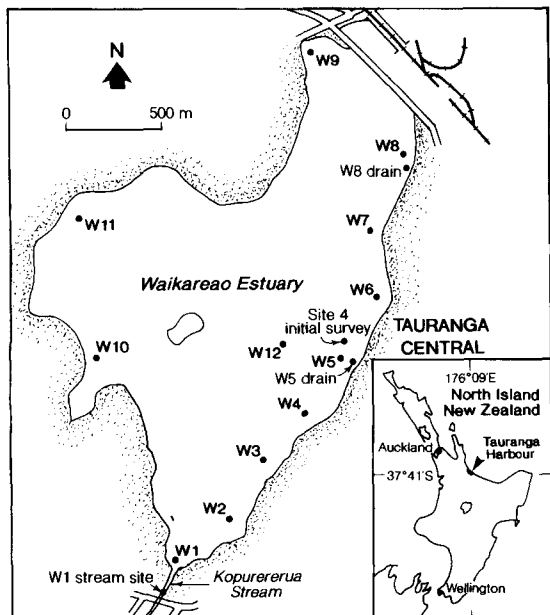


Fig. 1 Location of survey sites in the Waikareao Estuary, Tauranga Harbour.

Sediment analysis is a convenient means of investigating estuarine OCC levels since the concentrations of xenobiotic agents in sediment are generally significantly higher than concentrations in the overlying water. Sediment analysis can also provide an effective method of distinguishing between point and diffuse sources of contamination.

MATERIALS AND METHODS

Sample collection and extraction

Four sites were selected for the initial sediment analyses. These included the most common sub-catchment types likely to cause organochlorine contamination of a typical harbour or estuary (Table 1). In subsequent work a detailed survey of the Waikareao Estuary was undertaken (Table 1, Fig. 1).

Sediments were collected and extracted according to Fox et al. (1988) and Pridmore et al. (1991). At each site, three sediment samples were collected, generally at about the mid-tide level, combined in polyethylene bags, and frozen at -20°C until analysis. Before analysis, each sediment sample was thawed, mixed well and a sub-sample (c. 150 g) removed and air dried at 40°C . The dried sediment sample was ground and sieved (2 mm) to remove stones and shells and a subsample (40 g) was extracted with chloroform for 6 h in a Soxhlet apparatus. 2-chlorononachlor (Smith 1991) was used as an internal standard in the initial survey. No lindane was detected in the environmental samples, therefore lindane was used as an internal standard in subsequent work. The internal standard was added immediately before Soxhlet extraction. The chloroform extract was concentrated to approximately 2 ml under reduced pressure on a rotary evaporator and chromatographed on a florisil mini-column using light petroleum ether (fraction A) and diethyl ether-light petroleum ether (1:19) (fraction B) as eluent. Sulphur was removed from the eluent solutions by the addition of bright copper wire. Approximately 2 ml of heptane was added and the extract evaporated under N_2 to c. 1 ml, before gas chromatographic analysis with electron

Table 1 Description of the Tauranga Harbour sites sampled.

Sample Site	Date	Sub-catchment type
(1) Airport	24 Nov 91	Industrial/Tauranga Port facilities
(2) Karikari Point	24 Nov 91	Rural/agricultural and possible leachate from the Mount Maunganui Tip
(3) Wairoa River Mouth	24 Nov 91	Rural/agricultural and a small amount of horticulture
(4) Waikareao Estuary	24 Nov 91	Urban/light industry, rural/agricultural and possible leachate from the Tauranga City Tip/Oxidation Ponds viz Kopurererua Stream.
(5)–(16) Site W1 - W12 Waikareao Estuary	17 Feb 92	as (4) above
(17) Drain at site W5	14 Mar 92	as (4) above
(18) Drain at site W8	14 Mar 92	as (4) above
(19) Stream site near W1	14 Mar 92	as (4) above

capture detection (GC/ECD). Each fraction was analysed separately. Fraction A contained the PCBs, DDTs, chlordanes, and aldrin. Fraction B contained dieldrin.

GC/ECD analysis

GC/ECD analysis were performed using a 25 m × 0.25 mm DB5 fused silica capillary column (J & W Limited) installed in a Hewlett Packard (HP) 5890 GC instrument equipped with an HP 7636A automatic injector. Total PCBs were quantified by comparison with the nine major peaks of an Aroclor 1260 PCB standard (a technical PCB mixture which closely resembled the PCB residue profiles in the environmental samples). Other OCCs were identified and quantified by comparison with crystalline standards. Chlordane levels are presented as Technical Chlordane equivalent, defined as the sum of *cis*- and *trans*-chlordane × 2.33 (Trower & Holland 1991). *Cis*- and *trans*-chlordane comprise c. 43% of the Technical Chlordane formulation (Sovocool et al. 1977). The presence of DDT, DDE, DDD and PCBs in the sediment extracts was verified using combined gas chromatography mass spectrometry (GC/MS) while the identification of dieldrin and chlordane isomers was confirmed using a second, more polar DB17 column (20 m × 0.53 mm, J & W Limited). Ortho DDTs were only minor components in the samples analysed and were not quantified. The detection limit for a 40 g sediment extraction was typically 0.06 ng g⁻¹ for all OCCs except p,p'-DDT for which the detection limit was 0.1 ng g⁻¹. Only OCCs at levels greater than 0.1 ng g⁻¹ are reported (quantification limit).

Method validation

Mean recoveries of PCBs, DDTs, chlordanes, lindane, dieldrin and aldrin spiked onto sediment samples at a range of different concentrations, were 96%, 100%, 101%, 95%, 95%, and 100% respectively. Procedural blanks were routinely carried out with each batch of samples to ensure the experimental procedure remained free of contamination. Five replicate GC/ECD analyses of the same sediment gave relative standard deviations (RSDs) of 9% and 13% for total PCB and DDT (= p,p'-DDT + p,p'-DDE + p,p'-DDD), respectively.

Extraction times of 2, 6, and 24 h were chosen to test the extraction technique on sediments shown to be contaminated with PCBs and DDTs

(Waikareao Estuary). Levels of OCCs determined for both 6 and 24 h extraction times were similar to the 2 h extraction time. Since the nature of all sediments is not likely to be identical, a 6 h extraction time was adopted for this project. Others have also used a 6 h extraction time (Pridmore et al. 1991; Wilcock et al. 1993).

Depth and particle size analysis

Horizontal sediment layers (0–2 cm, 2–5 cm and 5–10 cm) were taken at 3 points around site W5 using a 13 cm diameter sediment corer. Equivalent layers from each core were bulked before analysis. Dried sediment was fractionated into different particle size fractions using dry sieving techniques described by Barrett & Brooker (1989) to obtain five size fractions (Table 5). Each fraction was examined for aggregates, which were removed, lightly ground, and re-sieved. An estimate of the organic content of the fractions was determined using the ignition loss procedure of Sansoni & Panday (1981; modified by the sample being heated to 450°C for 6 h) and measurement of readily oxidisable carbon (Gaudette et al. 1974).

RESULTS AND DISCUSSION

To determine how widespread OCCs were within the harbour, sediments were initially collected from four well separated mud-flats (Table 1). The levels of organochlorine compounds detected in these samples are given in Table 2. DDT and its degradation products (DDE and DDD) were detected in each of the four samples. The Wairoa River site had the highest Σ DDT level (2.02 ng g⁻¹), which can be ascribed to the proximity of this site to a large agricultural catchment. Although traces of dieldrin were also detected in each of the four samples, the levels of aldrin and lindane were below the respective detection limits. Oxidation of aldrin to dieldrin in the environment may account for the absence of aldrin in the samples. Traces of chlordanes were detected in the Waikareao Estuary sample only.

An elevated level of PCBs (19.9 ng g⁻¹) was detected in the Waikareao Estuary sample. This result suggested that PCBs, which had a wide variety of uses in industry including dielectric fluids for electrical equipment, paint thinners and hydraulic fluids may have escaped from the light industrial sites along the eastern foreshore and alongside the Kopurererua Stream which enters

the estuary at its southern end. The elevated PCB level determined for the Waikareao Estuary sample prompted a more extensive investigation of sediments from this part of the Tauranga Harbour.

Surficial sediment samples were therefore collected from 12 sites within the Waikareao Estuary (Fig. 1). The levels of organochlorine compounds detected in these samples are presented in Table 3. A typical GC/ECD trace is shown in Fig. 2. Sediment from four sites (W1, W2, W5, and W8) had elevated levels of OCCs in comparison to the other sites surveyed in the estuary. The downward trend in the OCC levels determined for the site W1 to W3 samples (Table 3) suggests the Kopurererua Stream may be the predominant source of the OCCs detected in the southern portion of the Waikareao Estuary. However, the elevated OCC levels determined for the W5 and W8 sites (but not

the W4 and W6 sites) suggest that other point sources may exist in the vicinity of the W5 and W8 sites.

Two major storm-water drain outfalls are located adjacent to sites W5 and W8, analyses of sediment samples from each of these drains, and the Kopurererua Stream, were undertaken. Appreciable levels of DDTs (7.52 and 19.2 ng g⁻¹) and PCBs (68.6 and 73.8 ng g⁻¹) were detected in each of the drain sediment samples, while much lower levels of DDTs (0.85 ng g⁻¹) and PCBs (3.38 ng g⁻¹) were detected in the Kopurererua Stream sediment sample. The higher levels of DDTs (2.41 ng g⁻¹) and PCBs (10.2 ng g⁻¹) detected in the W1 site sample (adjacent to the mouth of the Kopurererua stream) compared to those determined for the stream sample itself, could be due to salt water induced flocculation, or settling of suspended

Table 2 Concentrations of some organochlorine compounds (ng g⁻¹ dry wt) in surficial sediment samples from the Tauranga Harbour.

Site	p,p-DDT	p,p-DDE	p,p-DDD	DDT	Chlordanes	PCBs	Lindane	Dieldrin	Aldrin
Airport	0.11	0.32	0.29	0.72	nd	nd	nd	tr	nd
Karikari Point	0.35	0.38	0.10	0.83	nd	nd	nd	tr	nd
Waikareao Estuary	tr	0.21	0.48	0.69	tr	19.9	nd	tr	nd
Wairoa River Mouth	0.51	1.19	0.32	2.02	nd	nd	nd	tr	nd

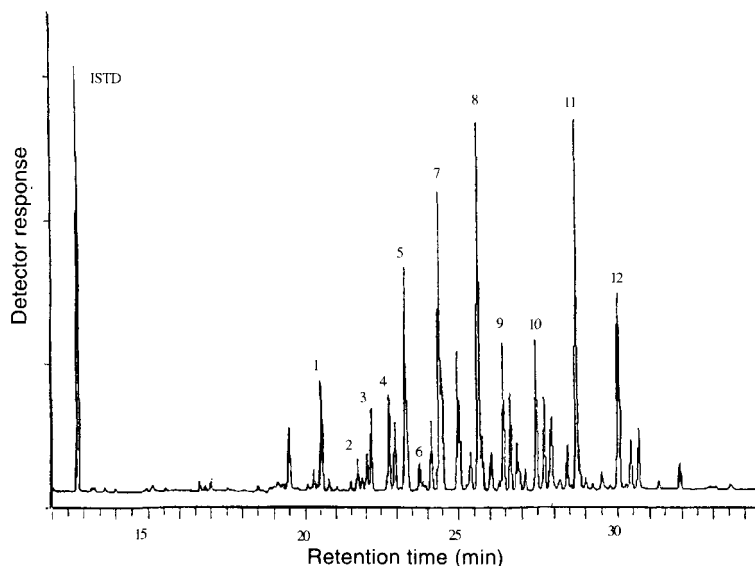
DDT = p,p-DDT + p,p-DDE + p,p-DDD; chlordanes = Technical Chlordane equivalent; nd = not detected (< 0.06 ng g⁻¹); tr = trace (< 0.1 ng g⁻¹); PCBs = total PCB expressed as an Aroclor 1260 equivalent.

Table 3 Concentration of some organochlorine contaminants (ng g⁻¹) in surficial sediments from the Waikareao Estuary, Tauranga Harbour, and from potential source sites.

Site	DDT	DDE	DDD	DDT	Chlordanes	PCBs	Dieldrin
W1	0.68	1.08	0.65	2.41	tr	10.2	0.11
W2	0.54	0.21	0.21	0.96	tr	4.24	tr
W3	tr	tr	tr	tr	tr	2.36	tr
W4	nd	0.11	tr	0.11	nd	2.74	tr
W5	0.35	1.19	1.31	2.85	tr	24.1	tr
W6	nd	nd	nd	nd	nd	0.18	tr
W7	nd	tr	tr	tr	nd	tr	tr
W8	0.86	1.96	2.54	5.36	tr	14.2	tr
W9	nd	tr	tr	tr	nd	0.83	tr
W10	nd	tr	nd	tr	nd	tr	nd
W11	nd	tr	nd	tr	nd	tr	nd
W12	nd	tr	tr	tr	nd	0.87	tr
Drain by site W5	2.43	3.5	1.59	7.52	tr	68.6	tr
Drain by site W8	3.62	6.1	9.45	19.2	tr	73.8	tr
Stream sample	0.30	0.33	0.23	0.85	tr	3.38	tr

DDT = p,p-DDT + p,p-DDE + p,p-DDD; chlordanes = Technical Chlordane equivalent; nd = not detected (< 0.06 ng g⁻¹); tr = trace (< 0.1 ng g⁻¹); PCBs = total PCB expressed as an Aroclor 1260 equivalent.

Fig. 2 A typical gc/ecd chromatogram for a sediment sample from the Waikareao Estuary. The major OCCs in the peaks marked are: (1) PCB 101, (2) p,p'-DDE, (3) PCB 110, (4) PCB 151, (5) PCB 149, (6) p,p'-DDD, (7) PCB 153, (8) PCB 138, (9) PCB 187, (10) PCB 174, (11) PCB 180, (12) PCB 170. Congener chlorine number was confirmed by GC/MS. Note the absence of earlier eluting PCBs and a predominance of later eluting more persistent penta-, hexa- and hepta-chlorinated congeners.



matter in the stiller water of the estuary. It is also likely that loss by erosion of surficial sediments will occur to a greater extent in the stream bed.

Table 3 shows that the eastern margin surficial sediments are more contaminated than those of open areas of the estuary (compare sites W12 and W5). This may be due to the effects of physical processes such as water circulation and the greater influence of the estuarine channel in open areas, which would tend to remove contaminated sediment. The western perimeter sites (e.g., sites W10 and W11) were found to be substantially less affected, presumably because of their distance from the suspected OCC entry points.

The depth dependency of the OCC levels determined for a sediment sample from site W5 is given in Table 4. The average level of PCBs in the top sediment layers (0–5 cm) was found to be approximately three times greater than that of the lower layers (5–10 cm). No obvious differences

were observed between the PCB residue profiles at the depths analysed. The lower levels of DDT and PCBs in the 0–2 cm layer when compared to the 2–5 cm layer appear to reflect the high mobility of the top layers of sediment and/or a decrease in OCC inputs into the estuary.

Only traces of DDTs were detected below 5 cm. Assuming that the first significant usage of DDT in the catchment was c. 1950, as was the case for New Zealand in general (Hume et al. 1989), that the time lag between its first use and its occurrence in runoff was short, and that sediment has not been significantly disturbed over the intervening years, then the 5 cm depth (maximum depth at which significant DDTs are detected) corresponds to an uncorrected sediment accumulation rate of c. 1 mm yr⁻¹. The maximum DDT concentration is at the 2–5 cm depth interval, which is the same as the maximum PCB concentration. Using a sediment accumulation rate of 1 mm yr⁻¹ the 2–5 cm depth

Table 4 Depth dependency of the concentrations of some organochlorine compounds (ng g⁻¹ dry wt) in sediment at the W5 site, Waikareao Estuary, Tauranga Harbour.

Depth	DDT	DDE	DDD	DDT	Chlordanes	PCBs	dieldrin
0–2 cm	nd	0.16	0.15	0.31	nd	16.4	tr
2–5 cm	nd	0.24	0.35	0.59	nd	22.3	tr
5–10 cm	nd	tr	nd	tr	nd	6.11	tr

DDT = p,p-DDT + p,p-DDE + p,p-DDD; PCBs = total PCB expressed as an Aroclor 1260 equivalent; chlordanes = Technical Chlordane equivalent; nd = not detected (< 0.06 ng g⁻¹); tr = trace (< 0.1 ng g⁻¹).

Table 5 Physical characteristics and concentrations of some organochlorine compounds in fractions from the initial survey site surficial sediment sample, Waikareao Estuary, Tauranga Harbour.

Particle size (mm)	Sediment type	DDT (ng g ⁻¹)	PCBs (ng g ⁻¹)	Ignition loss (% by wt)	Readily oxidisable carbon (mg g ⁻¹ dry wt)	Fraction frequency (% by wt)
> 0.500	Coarse sand	2.02 ^a	44.5 ^a	3.6	5.40	6.6
0.500–0.250	Medium sand	1.61 ^a	30.9 ^a	3.2	4.25	28.8
0.250–0.125	Fine sand	0.34	9.9	2.7	3.90	48.4
0.125–0.0625	Very fine sand	0.61	18.8	3.3	4.25	13.5
< 0.0625	Silt	0.73 ^a	28.4 ^a	6.1	11.30	2.8

^aAverage of two determinations; DDT = p,p-DDT + p,p-DDE + p,p-DDD; PCBs = total PCB expressed as an Aroclor 1260 equivalent; Mass balance calculations: e.g., contribution of PCBs in coarse sand fraction to total PCBs in an unfractionated sample = 44.5 (PCB concentration in fraction) × 6.6/100 (fraction frequency/100); PCB and DDT contributions from other fractions were calculated similarly. Total contributions from 5 fractions for PCBs and DDT are 19.9 and 0.85 respectively compared to 19.9 and 0.69 for the unfractionated sediment (see Waikareao Estuary - Table 2).

interval corresponds to the period 1950 to 1970 which covers the period of maximum DDT use. The data indicates that patterns of PCB usage have been similar to those of DDT. It is recognised that a number of factors can perturb depth profiles (Hume et al. 1989), including sediment reworking by bioturbation and deep water currents.

Surficial sediment from site 4 (initial survey - Waikareao Estuary) was sieved to afford fractions which were separately extracted and analysed for OCCs. Mass balance calculations (footnote Table 5) for the separated fractions compared favourably with the analysis of the unfractionated sample. The values obtained for the fine sand, very fine sand, and silt fractions (Table 5) exhibited the expected increase in the levels of OCCs with decreasing particle size (increasing surface area). An unexpected result was the high levels of OCCs identified in the coarse sand and medium sand fractions. Since it is widely acknowledged (Karickhoff 1984) that sorption of neutral (non-

ionic) organic compounds depends on the amount of organic matter in the sediment, ignition loss experiments and measurements of readily oxidisable carbon were conducted to gain an estimate of the organic content of the different size fractions (Table 5). The values determined for the fine sand, very fine sand, and silt fractions increase with decreasing particle size. However the values for the coarse sand and medium sand fractions increase with increasing particle size. This increase is not sufficient to account for the large increase in OCC residues observed in the coarser sand fractions.

Upon closer inspection the coarsest sand fraction was found to be a heterogeneous mixture containing shell fragments, pumice, stones, quartz material, and small black particles of unknown origin. That part of the black particulate material which could be separated easily, when extracted and analysed using the GC/ECD protocol, yielded levels of the DDTs and PCBs much higher than the mean value for the fraction as a whole (Table 6). Extraction of the shell fragments, pumice and stones afforded only low levels of OCCs. The black particulate material was readily oxidised and appeared to be comprised mainly of carbonaceous components such as charcoal, coal, or asphalt-like material associated with roading.

Although the low levels of OCCs detected in most of the sediments surveyed supported the popular view that chemical pollution of the New Zealand marine environment is lower than is the situation for more industrialised and highly populated overseas countries, the concentrations of OCCs at some sites within the Waikareao

Table 6 Concentrations of some organochlorine compounds in sub-fractions from the coarse sand fraction isolated from the Waikareao Estuary, Tauranga Harbour.

Sub-fraction description	DDT (ng g ⁻¹)	PCBs (ng g ⁻¹)	Ignition loss (% by wt)
Black particulates	205	265	98
Shells	nd	2.01	nq
Pumice and stones	nd	3.21	nq

DDT = p,p-DDT + p,p-DDE + p,p-DDD; PCBs = total PCB expressed as an Aroclor 1260 equivalent; nq = not quantified; nd = not detected.

Estuary, (e.g., W1, W5, and W8) had OCC levels similar to those reported in sediment from San Francisco Bay, California (Phillips & Spies 1988) and Rio de La Plata, Argentina (Colombo et al. 1990). Other studies outside New Zealand have, however, also recorded levels much higher than those determined in the Waikareao sediments (Turk 1980; Sarkar & Sen Gupta 1988).

Levels of OCCs at some of the Waikareao Estuary sites (e.g., W1, W5, and W8) are comparable to those determined in the Mangere Inlet and Pukaki Creek of the Manukau Harbour (Fox et al. 1988); however, the levels at the other sites in the greater Tauranga Harbour (see Table 1) were not substantially different from background levels established in the Hauraki Gulf (Roper & Wilcock 1991).

The bulk of the PCB burden appears to have entered the Waikareao Estuary via the storm-water drains close to the W5 and W8 sites, and to a lesser extent also the Kopurererua Stream. New Zealand urban storm water is kept separate from domestic and industrial sewer systems and untreated storm water is usually discharged into the closest waterway (Smith 1986) such as the Waikareao Estuary. Although PCBs can be transported as dissolved material in water, their solubility is very low and generally they pose little threat if contained. However, if surface material containing these contaminants is susceptible to erosion by wind, rain, and surface water they can be easily transported as dust or as sediment in run-off and so find their way into stormwater and streams. The industrialised areas along the eastern foreshore of the Waikareao Estuary appear to be the most likely sources of the PCBs.

DDT inputs into the Waikareao Estuary probably arise from a combination of domestic, agricultural, and industrial uses. The high DDT levels identified in the W5 and W8 sediment sample sites may also be a consequence of previous grass management practices, since DDT was once a popular grass grub (*Costelytra zealandica*) control agent. The high proportion of DDD and DDE in relation to the parent compound DDT is indicative of the historical entry of DDT into the environment. It is likely that the inputs of OCCs into the Waikareao Estuary have declined in recent years due to legislative restrictions and the adoption of wiser environmental practices.

Estuarine areas adjacent to cities are often affected by pollution. The current study has confirmed that urbanised zones of the Tauranga

Harbour such as the Waikareao Estuary are vulnerable to this problem.

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