

**LABORATORY SEDIMENT
BIOASSAY REPORT ON
WHEATLEY HARBOUR SEDIMENTS
1992**

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BIOASSAY REPORT ON
WHEATLEY HARBOUR SEDIMENTS
1992**

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EXECUTIVE SUMMARY

Sediments were sampled from Wheatley Harbour and Muddy Creek located along the north shore of Lake Erie in 1992 in order to carry out a laboratory biological assessment of contaminated sediments from that Area of Concern. The main objectives of the study were:

- 1) To describe the level of inorganic and organic chemical contamination in surficial sediments at one reference and five test locations;
- 2) To assess the toxicity of whole-sediments in laboratory tests using the mayfly, *Hexagenia limbata* (survival and growth, 21-day exposure), the midge, *Chironomus tentans* (survival and growth, 11-day exposure) and the juvenile fathead minnow, *Pimephales promelas* (survival, 21-day exposure);
- 3) To determine the availability of sediment-bound organic contaminants to the forage fish, *Pimephales promelas* after a 21 day exposure period.

The key findings of the study were as follows:

- 1) The majority of the test locations contained sediments that were nutrient-enriched with total phosphorus and total Kjeldahl nitrogen at levels greater than the Provincial Sediment Quality Guideline Severe Effect Level concentrations. Total organic carbon content also reached moderate levels. Inputs of suspended materials from the nearby wetlands and surrounding agricultural areas are suspected as contributing to this enrichment.
- 2) Trace metal and organic chemical sediment concentrations were low with no exceedences of the PSQG Severe Effect Level concentrations being reported. Concentrations were either at or slightly above the respective Lowest Effect Level concentrations or were comparable to background concentrations as reported for the reference location, which was situated upstream of industrial point discharges.
- 3) Whole-sediment toxicity tests indicated no significant trends in lethal or sublethal responses for the three test species. The occasional positive response was noted at a single station for the mayfly and minnow assays. There was no relationship found between the bioassay results and either sediment physical and chemical parameters.
- 4) Whole organism fish residues were below analytical detection limits for those organic compounds that were measured in the sediment. This suggests the sediment-bound chemicals are not readily available through those routes of exposure associated with a forage fish species.

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1.0 INTRODUCTION

Wheatley Harbour was designated as an Area of Concern (AOC) in the Great Lakes basin by the International Joint Commission (IJC) in the 1980's due to elevated nutrient, suspended solids and bacterial levels in the water, and organic contaminants in the sediment. The Canadian federal and provincial governments are committed to the development of a remedial action plan (RAP) for the AOC as part of the revised Great Lakes Water Quality Agreement (IJC, 1987). Investigations have characterized the environmental conditions and problems that exist for the area (Wheatley Harbour RAP, 1992).

Historical investigations have characterized the sediments in Wheatley Harbour as having been contaminated with polychlorinated biphenyls (PCBs) that were anthropogenically introduced, which over time has resulted in restrictions on dredging activities (Wheatley Harbour RAP, 1992). The PCB contaminant problem has been related primarily to point source discharges into Muddy Creek which has resulted in elevated PCB concentrations within the area of the outfall (Wheatley Harbour RAP, 1992). The Omstead Fisheries Ltd plant owned by Omstead Foods Ltd, has been in operation since the early 1970s and is the main processor of locally-caught fish. Earlier, the process effluent discharges were found to contain appreciable levels of PCBs at a time when the wastewater received minimal treatment prior to release into Muddy Creek. The plant has since undergone a major upgrade in the handling and treatment of the wastewater in 1983 and ceased production of fish meal in 1985. These changes have resulted in a substantial improvement in effluent quality, often resulting in non-detectable concentrations. The PCBs that were found earlier appear to have originated from the PCBs contained in the whole fish that were caught in Lake Erie and were then subsequently concentrated in the fish oil and wastes that were generated from plant operations. Analysis of sediment cores collected in 1986 by Environment Canada, indicated elevated PCB concentrations frequently occurring at depths >6 cm, suggesting historical inputs into the system (Wheatley Harbour RAP, 1992).

Since 1973, field investigations in the area have been ongoing for the purpose of monitoring sediment contamination. In 1987, the Ministry of Environment and Energy (MOEE), Southwestern Region, London Regional Office, carried out a comprehensive study of Wheatley Harbour involving sediment chemical analysis, benthic community structure analysis and laboratory sediment bioassays (OMOE, 1987). PCB concentrations in surficial sediment were found to exceed the MOEE Open-Water Disposal Guidelines (Persaud and Wilkins, 1976) which were used for sediment assessment at that time. The study also helped to better define the background conditions for the area in terms of nutrient, organic and inorganic contamination.

A sediment survey conducted in August 1992 at 16 sites, identified an area of sediments with PCB sediment concentrations ranging from 0.06 to 0.36 µg/g (D. Huber, *pers. comm.*). This prompted an additional survey in November 1992 of Wheatley Harbour and Muddy Creek sediments. The purpose of this study was to determine the distribution and degree of

contamination in sediments and to assess the biological impacts of sediment-associated contaminants using laboratory toxicity tests. The laboratory biological tests encompassed a broader battery of test species and biological effects as compared to the 1987 study. Specifically, surficial sediments were obtained from control and test field sampling sites and submitted to the MOEE Sediment Bioassay Laboratory for standardized laboratory sediment bioassays (Bedard *et al.*, 1992). Whole-sediment toxicity tests were conducted using the mayfly nymph, *Hexagenia* sp. (21-day exposure, survival and growth), the midge larvae, *Chironomus tentans* (11-day exposure, survival and growth) and the juvenile fathead minnow, *Panephales promelas* (21-day exposure, survival and chemical bioaccumulation). The sediment was analyzed for metals, nutrients, PCBs, chlorinated organics and pesticides. The results obtained from the laboratory bioassays are to provide toxicological information on contaminated sediments in order to assist in the development of the Wheatley Harbour RAP.

2.0 MATERIALS AND METHODS

2.1 Sample Collection and Site Description

Surficial sediment was collected at six locations within the Wheatley Harbour AOC by MOEE, Water Resources Branch, for the completion of laboratory sediment bioassays. The test sediments were selected from both Muddy Creek and Wheatley Harbour. Sample locations are shown in Figure 1. Collection was made using a 22 X 22 cm Ekman grab sampler on November 10, 1992. At each sample location, approximately 15 L of composited surficial sediment (top 5 cm) was collected from several grabs. The composited sediment was placed into 20 L plastic buckets lined with food-grade polyethylene bags and transported to the MOEE laboratory in Toronto where they were stored in the dark, at 4°C until required.

Sediments were collected from both the Muddy Creek wetlands and within the harbour. The stations were selected to cover as wide a range of contamination as possible based on available data. An upstream control site (Station #1) was selected near the inflow from the marsh and is referred to as the reference control sediment. The reference control sediment should be representative of naturally occurring background contaminant levels for the study area and should be physically similar to the test sediments to help discriminate effects due to physical or chemical causes. Three of the test sediments (Stations #2, #3 and #4) were situated below the Omstead Foods Ltd discharge points within Muddy Creek (above County Road #1). The remaining two collection sites were located within the harbour, either at the mid-way point within the channel (Station #6) or in a sheltered adjoining basin (Station #5). In addition, sediment collected from Honey Harbour in Georgian Bay served as a 'negative control' for each bioassay. The negative control sediment is a relatively uncontaminated sediment that provides a measure of test acceptability and is a basis for comparing the biological responses from the test sediments.

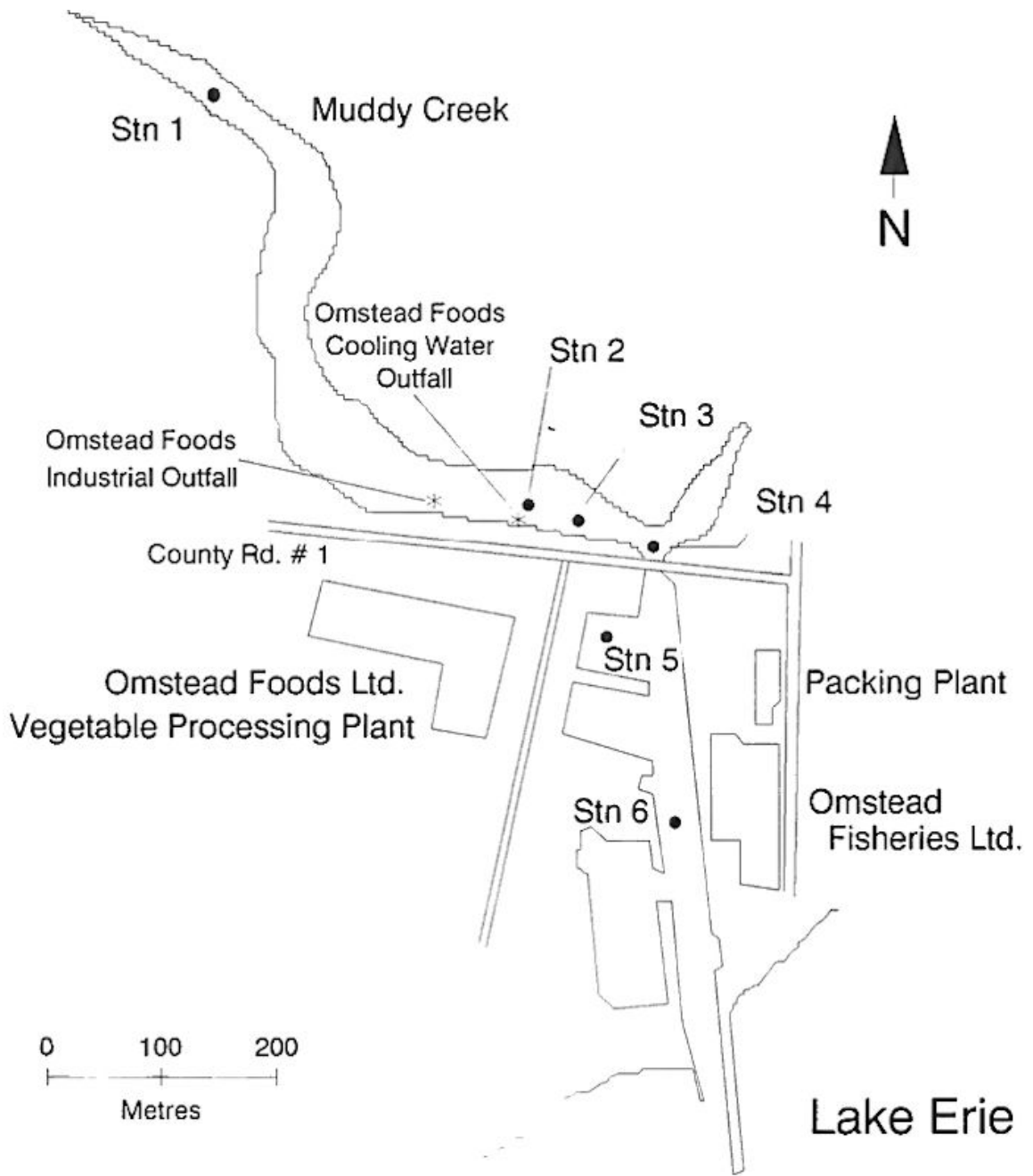


Figure 1: Wheatley Harbour Sediment Sampling Locations.

The following is a detailed description of the sample locations:

Station	Description
# 1	Reference control station located in Muddy Creek about 450 metres upstream of the Omstead Foods Ltd industrial waste outfall. Found in an area surrounded by wetland.
# 2	Situated 20 metres east of the Omstead Foods Ltd cooling water discharge in Muddy Creek.
# 3	Located 100 metres below the Omstead Foods Ltd cooling water discharge, along the south side of Muddy Creek.
# 4	Located at the upstream entrance to Wheatley Harbour, just above the bridge at County Road #1.
# 5	Situated within the first turning and docking basin and found 65 metres below the County Road #1 bridge, 16 metres from the eastern side of the basin.
# 6	Located 300 metres downstream of County Road #1 within the harbour shipping channel across from the Omstead Fisheries Ltd plant.

2.2 Analytical Methods

Chemical analysis of sediment and biota samples was carried out by the MOEE, Laboratory Services Branch, located in Toronto. Detailed methodology is described in the *OMOE Handbook of Analytical Methods for Environmental Samples* (OMOE, 1983). Analytical detection limits for each of the test parameters are listed in Table A1. Quality assurance procedures included method blanks, spikes, duplicates and standard reference materials.

Sediment Nutrients and Particle Size Characterization

Homogenized bulk sediment (<2 mm fraction) was measured for total phosphorus (TP), total Kjeldahl nitrogen (TKN) and percent weight loss on ignition (LOI) which measured approximate organic content. Sediment total organic carbon (TOC) was determined with a LECO carbon analyzer using a dry combustion technique which oxidized carbon to CO₂. Particle size was measured on 50 g, air-dried samples using a Microtrac particle size analyzer for the size range 1.00 mm to 0.1 µm. This was to provide data for % sand (2mm - 62 µm), % silt (62- 3.7 µm) and % clay (3.7 - 0.1 µm) size classes.

Trace Metals in Sediment

Prepared sediment samples were digested using a concentrated aqua-regia acid mixture (1 part HNO₃ to 3 parts HCl). The dissolved trace metals including As, Cd, Cr, Cu, Fe, Pb, Mn, Ni and Zn in the digestates were detected by inductively coupled argon plasma atomic emission spectroscopy (ICP-AES) and Hg by flow injection vapor generated flameless atomic absorption spectroscopy (AAS).

Organic Chemicals in Sediment

Moist sediment samples were extracted with acetone and dichloromethane. The extract was transferred to a rotary evaporator, concentrated and fractionated on a Florisil column. Different solvent combinations were used to elute the extracts into three groups, Fraction A1 contained total PCBs, 5 Aroclor groups, hexachlorobenzene, heptachlor, aldrin, octachlorostyrene, pp-DDE and mirex. Fraction A2 contained α - & β -BHC, α - & β -chlordane, op-DDT, pp-DDD, pp-DDT and fraction A3 included heptachlor epoxide, oxychlordane, dieldrin, endosulfan I & II, endosulfan sulphate, endrin and methoxychlor. Analytes were identified and quantified using capillary gas chromatography equipped with a Ni⁶³ electron capture detector (GLC-ECD).

Organic Chemicals and Percent Lipid in Biota

Pooled whole fish samples (~5g) were thawed, homogenized and acid digested using concentrated HCl acid on duplicate samples per sediment. The digestate was reacted with a mixture of 25% dichloromethane in hexane. The extract was treated with sodium bicarbonate to ensure neutralization and dried with anhydrous sodium sulphate. Sample clean-up and detection followed identical procedures as those described for sediment analysis. Final results are reported on a wet weight basis for 16 compounds (Table 1A). Percent lipid was determined on an aliquot (25 ml) of the final extract obtained prior to clean-up. The solvent was allowed to evaporate by air-drying in a fumehood for 24 hours and lipid residues were measured.

2.3 Laboratory Biological Testing Methods

Basic Experimental Design

Sediment biological tests were conducted according to MOEE standardized procedures (Bedard *et al.*, 1992) and are briefly described below. The bioassays were static, single-species tests using whole sediment. The experimental unit was a 1.8 L test chamber containing prepared sediment and dechlorinated municipal tap water (1:4, v:v). The chambers were randomly placed into a holding tank at ambient room temperature and maintained under a 16:8 hour, light:dark photoperiod and continuous aeration.

Moist field-collected bottom sediment was pressed through a 2-mm stainless-steel sieve to remove existing large biota and debris prior to use. Subsamples of this homogenized sediment were submitted for chemical and physical characterization according to standard MOEE procedures (OMOE, 1989). The sieved sediment was homogenized with a spatula and stored in 4 L acid-rinsed glass jars until required. Three hundred and twenty-five millilitre aliquots of homogenized sediment were placed into the test chamber and overlaid with the test water. After settling overnight, the chambers were aerated continuously until the termination of the test. A clean, negative control sediment that was collected from Honey Harbour, Georgian Bay, was used for each bioassay. Control mortality must not exceed 15% for mayflies and fathead minnows and 25% for chironomids or the test is declared invalid.

Water in the exposure chambers was regularly monitored for pH, conductivity and dissolved oxygen. Dead organisms were removed and the numbers recorded on a daily basis. Any signs of abnormal behaviour of the test organisms or changes in appearance of the test chambers were noted. Water loss due to evaporation was replenished as needed. At the conclusion of the test, fathead minnow test organisms were collected, placed into glass vials and frozen prior to analysis. The whole body tissue samples were measured for total PCBs, hexachlorobenzene, aldrin, pp-DDE, pp-DDD, pp-DDT, mirex, octachlorostyrene, toxaphene, and percent lipid on duplicate samples.

***Hexagenia limbata* Lethality and Growth Assay**

The tests used 4 month old laboratory reared mayfly nymphs with an average wet weight of $14.5 \text{ mg} \pm 1.31$ (s.e.). The nymphs were raised from eggs collected by Dr. J. Ciborowski at the University of Windsor, Windsor, Ontario. Mayflies were reared according to MOEE procedures (Bedard *et al.*, 1992) and methods described in the literature (Friesen, 1981).

The rearing procedure involved the transfer of 600 newly-hatched nymphs to a 6.5 L aquarium which contained 2 cm of autoclaved sediment and 5.6 L dechlorinated tap water. Animals were maintained at ambient room temperature, 16:8 hour, light:dark photoperiod, constant aeration and fed a vegetable diet.

Test organisms were retrieved from the rearing aquaria by sieving small portions of sediment in a 500- μm mesh brass sieve. The nymphs were washed into an enamelled tray which held a fine mesh sieve and aerated, dechlorinated water. A Pasteur pipette (5-mm opening) was used to transfer the mayflies into 100 mL beakers of water and the contents were gently poured into the test chambers. Four replicates were run for each sediment and ten nymphs were added for each replicate, for a period of 21 days. During sorting, 33 individuals were randomly selected and weighed individually to the nearest 0.01 mg to obtain the starting weight and then discarded. Animals were not fed during the length of the test.

At the end of the test, the contents of each test chamber were emptied and rinsed in a sieve bucket. Surviving animals were counted and transferred to 150 mL beakers holding 100 mL dechlorinated water. The nymphs were immobilized with Alka-Seltzer®, blotted dry and individuals weighed to the nearest 0.01 mg, placed in vials and stored in a freezer.

***Chironomus tentans* Lethality and Growth Assay**

The tests used 10-12 day old, cultured chironomid larvae weighing an average wet weight less than 1 mg. The MOEE continuously cultures *C. tentans* larvae from egg to adult following standard methods (Bedard *et al.*, 1992, Mosher *et al.*, 1987, Townsend *et al.*, 1981). Egg masses were acquired from Dr. J. Giesy at Michigan State University, Lansing, Michigan and have been cultured for several generations in our laboratory.

Initially, the midges were reared in enamelled trays for a period of 10 to 12 days and then maintained in a 21 L aquarium containing 1.6 L of silica sand. The cultures were held at ambient room temperature with continuous aeration and under a 16:8 hour, light:dark photoperiod. The larvae were provided a vegetable diet *ad libitum*.

Second instar larvae were directly transferred from the enamelled rearing pans into the test chamber using the 5-mm opening of a Pasteur pipette. A total of 15 animals were added per chamber to each of the four replicates. Animals were fed daily 30 mg of a Cerophyll®:Tetra Conditioning Vegetable® (3:2, w:w) diet. Within 18 hours the jars were checked and "floaters" were removed and replaced.

After 11 days, the contents of the test chambers were emptied and washed in a sieve bucket. Surviving animals were sorted, removed and placed into 150 mL beakers holding 100 mL dechlorinated water and 15 mL silica sand. The larvae were counted, blotted dry and individuals weighed to the nearest 0.01 mg.

***Pimephales promelas* Lethality and Bioaccumulation Assay**

The tests used cultured, juvenile fathead minnows that weighed 336 mg ± 26 (s.e.) (wet weight). The minnows were cultured at the MOEE laboratory and followed techniques which for the most part are US EPA procedures (USEPA, 1987) with minor revisions (Bedard *et al.*, 1992).

Cultures were maintained at 25°C in a flow-through dechlorinated water system and under a 16:8 hour, light:dark photoperiod. Breeders were kept in 60 L glass aquarium and eggs are laid on spawning tiles. The tiles were incubated in a 25°C water-bath and the developing larvae were transferred to 400 L fibreglass holding tanks. Larval fish were fed 48-hour old live

brine shrimp while juveniles and breeders were provided frozen brine shrimp. Each size class was fed *ad libitum*.

Each test chamber received 10 juvenile minnows for each of the three replicates. The minnows were sorted into 250 mL glass beakers in groups of five. The contents of the beakers were emptied into a small net and the minnows released into the test chamber. During sorting a random subsample of 30 animals was separated, weighed individually and frozen.

The minnows were exposed for 21 days and fed Tetra Conditioning Vegetable® diet in an amount equivalent to 1% of the average starting wet weight, on a daily basis. After 21 days the surviving fathead minnows were pooled from each replicate, counted, immobilized with Alka-Seltzer® and placed into 30 mL glass vials and frozen pending chemical analysis.

Bioassay Schedule

Test Organism	Species	Starting Date ('92)	Completion Date ('92)	Test Duration
Mayfly	<i>Hexagenia limbata</i>	Thur. Nov 19	Thur. Dec 10	21 days
Chironomid	<i>Chironomus lentans</i>	Thur. Dec 3	Mon. Dec 14	11 days
Minnow	<i>Pimephales promelas</i>	Thur. Nov 19	Thur. Dec 10	21 days

2.4 Statistical Methods

Statistical analyses were performed using SAS® software package (SAS, 1985). Comparisons were made among the test and control sediments using One-Way Analysis of Variance (ANOVA) and Tukey's studentized range test (HSD) and planned comparisons (Steel and Torrie, 1960). Dunnett's t-test was used solely to compare mortality between the control and test sediments. Analysis was made on arc-sine transformed mortality data. Homogeneity of variance across groups was tested using Bartlett's test. Coefficients of variation (C.V. %) were calculated for each endpoint as a measure of test precision. Spearman Rank Correlation Analysis was used to investigate the correlation among the different biological endpoints for each species and sediment characteristics.

3.0 RESULTS AND DISCUSSION

3.1 Water Quality Test Parameters

Conductivity, pH and dissolved oxygen parameters were periodically measured on the overlying water for each test species and recorded in Table 1. Values are reported as mean \pm standard deviation. Similar water quality measurements were recorded among the control and test sediments, regardless of test duration or test species. Average pH values were 8.3, 8.4 and 8.1 for *H. limbata*, *C. tentans* and *P. promelas*, respectively. Mean conductivity values were consistent among the three test organisms with values of 406, 410 and 427 $\mu\text{mho/cm}$ for the mayfly, midge and minnow bioassays, respectively. Dissolved oxygen within the test jars remained above acceptable levels ($>4 \text{ mg/l}$) throughout the test (OMOE, 1984). Test temperature averaged 18.3°C for all bioassays.

3.2 Sediment Characterization

Physical and Nutrient Properties

Physical and nutrient properties of the control and test sediments are reported in Table 2. Sieved sediments were characterized for % sand (2mm - $62 \mu\text{m}$), % silt ($62\text{-}3.7 \mu\text{m}$), % clay ($3.7\text{-}0.1 \mu\text{m}$), % loss on ignition (% LOI), total organic carbon (TOC), total phosphorus (TP) and total Kjeldahl nitrogen (TKN).

All of the control and test sediments shared similar physical grain-size composition. The sediments were comprised mainly of silt and clay-sized particles. Percent fines ($<62 \mu\text{m}$) ranged from 84% to 100%. The low variability in sediment type may be a result of the selective collection of those sediments indicative of depositional areas where contaminants may accumulate rather than a reflection of the natural variability occurring throughout the study area. Differences in nutrient characteristics were apparent, particularly between the control and test sediments. Generally the negative and reference control sediments contained similar but substantially lower amounts of organic matter, TP and TKN. Several exceedences of PSQG-SEL concentrations for TP and TKN were recorded in each of the test sediments. Values ranged from 1.9 - 2.7 mg/g for TP and 3.9 - 7.7 mg/g for TKN. The respective PSQG-SEL concentrations are TP 2.0 mg/g and TKN 4.8 mg/g. Fairly high amounts of organic matter were detected with TOC levels ranging from 37 to 85 mg/g. The transport of suspended sediments and runoff materials originating from the adjacent Muddy Creek wetlands may act as a continuous source of organically-enriched sediment. In addition, agricultural runoff from nearby farmlands that enters Muddy Creek and eventually accumulates in sheltered areas of the harbour or settles out during periods of low flow, may have contributed to the elevated sediment phosphorus and nitrogen levels. Relative to other wetland areas located along the north shore of Lake Erie, the Wheatley Harbour and Muddy Creek area tend to have similar background sediment nutrient values (Wheatley Harbour RAP, 1992).

TABLE 1. Mean (\pm s.d.) water quality characteristics in sediment bioassays.

<i>Test Organism: Mayfly (Hexagenia limbata)</i> ^a		Test Temperature: 18.3 (0.6) °C ^a	
<i>Station</i>	<i>pH</i>	<i>D.O.</i> mg/L	<i>Conductivity</i> µmho/cm
Control	8.17 (0.17)	8.5 (0.4)	297 (10)
Reference ^b	8.45 (0.12)	8.7 (0.2)	422 (43)
Station #2	8.23 (0.02)	8.5 (0.4)	422 (68)
Station #3	8.27 (0.35)	8.6 (0.5)	382 (22)
Station #4	8.44 (0.27)	8.6 (0.2)	461 (52)
Station #5	8.49 (0.02)	8.7 (0.4)	450 (34)
Station #6	8.49 (0.16)	8.6 (0.4)	409 (19)

<i>Test Organism: Midge (Chironomus tentans)</i> ^c		Test Temperature: 18.3 (0.4) °C ^c	
<i>Station</i>	<i>pH</i>	<i>D.O.</i> mg/L	<i>Conductivity</i> µmho/cm
Control	8.24 (0.14)	8.5 (0.1)	329 (16)
Reference	8.46 (0.27)	8.5 (0.2)	415 (45)
Station #2	8.47 (0.32)	8.6 (0.2)	402 (21)
Station #3	8.45 (0.31)	8.6 (0.2)	406 (13)
Station #4	8.44 (0.07)	8.4 (0.1)	465 (59)
Station #5	8.36 (0.03)	8.5 (0.2)	432 (21)
Station #6	8.47 (0.17)	8.5 (0.1)	423 (40)

<i>Test Organism: Fathead Minnow (Pimephales promelas)</i> ^a		Test Temperature: 18.3 (0.6) °C ^a	
<i>Station</i>	<i>pH</i>	<i>D.O.</i> mg/L	<i>Conductivity</i> µmho/cm
Control	7.75 (0.04)	8.5 (0.4)	319 (10)
Reference	8.28 (0.17)	8.5 (0.4)	415 (33)
Station #2	8.21 (0.29)	8.6 (0.2)	455 (74)
Station #3	8.10 (0.26)	8.6 (0.4)	445 (46)
Station #4	8.38 (0.23)	8.5 (0.3)	499 (68)
Station #5	8.19 (0.23)	8.5 (0.3)	443 (35)
Station #6	8.15 (0.22)	8.6 (0.4)	414 (25)

a Sample size N=3,

b Reference sediment collected at Muddy Creek Station #1;

c Sample size N=2.

TABLE 2. Sediment physical and nutrient characteristics in control(s) and Wheatley Harbour sediment used in sediment bioassays.

<i>Station</i>	<i>% Sand</i> (2mm- 62µm)	<i>% Silt</i> (62 - 3.7µm)	<i>% Clay</i> (3.7 -0.1µm)	<i>% LOI</i>	<i>TOC</i> mg/g	<i>TP</i> mg/g	<i>TKN</i> mg/g
Control	8.0	60.9	31.1	9.5	39	1.2	3.7
Upstream Reference Station #1	9.4	55.6	34.9	9.7	37	1.5	3.9
Muddy Creek Station #2	15.9	60.0	24.2	14	65	1.9	5.2
Muddy Creek Station #3	6.3	66.7	26.9	19	85	2.4	7.7
Muddy Creek Station #4	0.3	65.4	34.3	15	67	2.7	7.0
Wheatley Harbour Station #5	0.2	63.6	36.4	13	55	2.5	5.1
Wheatley Harbour Station #6	1.8	64.0	34.4	8.5	37	2.3	3.9
PSQG SEL Conc. (mg/g dry weight)					100	2.0	4.8

Shading indicates sediment nutrient concentrations that exceed PSOG-SELs.

Trace Metal Sediment Concentrations

Bulk sediment was analyzed for 11 trace metals (Table 3). The sediment metal concentrations were compared to Severe Effect Level (SEL) and Lowest Effect Level (LEL) concentrations as outlined in the Provincial Sediment Quality Guidelines (PSQGs) (Persaud *et al.*, 1992). The SEL is defined as the chemical concentration in the sediment that is considered to be detrimental to the majority of the macrobenthos and the LEL is the sediment contaminant concentration which can be tolerated by most benthic species.

Chemical analysis indicated that the test sediments collected throughout the study area had no reported exceedences of the PSQG-SEL concentrations for any of the trace metals and in fact were well below these levels. Sediment concentrations for arsenic (As), cadmium (Cd), chromium (Cr) and zinc (Zn) were at PSQG-LEL concentrations and were only slightly above for copper (Cu) and nickel (Ni). All the test sediment metal concentrations were representative of upstream background levels. The reference control sediment collected from a site far removed from any influence from industrial discharges, had metal levels generally at or slightly above PSQG-LEL concentrations and within the same range as the test sediments. This pattern of sediment metal concentrations suggests an equal distribution of metals with no indication of point sources. No significant changes in sediment metal concentrations are apparent relative to those measured during a 1987 survey (Table A2; OMOE, 1987).

Organic Chemical Sediment Concentrations

The group of organic chemicals that were analyzed for but not detected, in the test sediments included heptachlor, aldrin, mirex, γ -chlordane, pp-DDT, endosulfan, dieldrin, endrin and octachlorostyrene. Five organic chemicals were measured at detectable levels in at least one of the control and test sediments (Table 4). Sediment concentrations for A-chlordane were at PSQG-LEL concentrations of 0.007 $\mu\text{g/g}$ pp-DDE was measured in all the control and test sediments (Range: 0.005 - 0.035 $\mu\text{g/g}$) and were above the PSQG-LEL concentration but substantially lower than the PSQG-SEL concentration of 19 $\mu\text{g/g}$ organic carbon. The reference control sediment contained only trace amounts of hexachlorobenzene (HCB) and pp-DDD.

Levels of total polychlorinated biphenyls (PCBs) measured in the test sediments were less than twice background levels (Station #1 - 0.18 $\mu\text{g/g}$). Values for the test sediments ranged from 0.20 to 0.33 $\mu\text{g/g}$ and were just above the PSQG-LEL concentration of 0.07 $\mu\text{g/g}$ and were at least 61 X lower than the corresponding PSQG-SEL concentrations (corrected for sediment TOC). Stations #5 and #6 had PCB sediment concentrations closer to their respective PSQG-SEL concentrations (corrected for TOC) due to the lower amounts of organic matter associated with the sediment. In addition, sediment PCB concentrations were found to be significantly correlated with percent fines (see Table 9). For instance, somewhat higher PCB concentrations were noted for those sediments containing > 93% silt and clay, and included Stations #3, #4, #5 and #6.

TABLE 3. Bulk concentrations of trace metals in control(s) and Wheatley Harbour sediment ($\mu\text{g/g}$ dry weight) used in sediment bioassays.

Station	Al %	As	Cd	Cr	Cu	Fe %	Hg	Mn	Ni	Pb	Zn
Control	2.4	<u>6.0</u>	<u>1.5</u>	<u>44</u>	<u>22</u>	<u>3.4</u>	0.06	<u>930</u>	<u>33</u>	<u>43</u>	<u>140</u>
Upstream Reference Station #1	2.7	<u>6.5</u>	<u>1.5</u>	<u>33</u>	<u>34</u>	<u>2.3</u>	0.08	220	<u>33</u>	28	<u>120</u>
Muddy Creek Station #2	1.4	5.7	<u>1.1</u>	22	<u>40</u>	1.6	0.14	190	<u>24</u>	<u>34</u>	<u>200</u>
Muddy Creek Station #3	2.1	<u>6.9</u>	<u>1.4</u>	<u>28</u>	<u>42</u>	1.8	0.10	180	<u>31</u>	28	<u>160</u>
Muddy Creek Station #4	2.6	<u>7.7</u>	<u>1.2</u>	<u>32</u>	<u>43</u>	<u>2.2</u>	0.08	230	<u>34</u>	30	<u>180</u>
Wheatley Harbour Station #5	2.8	<u>9.1</u>	<u>1.4</u>	<u>35</u>	<u>45</u>	<u>2.4</u>	0.08	230	<u>36</u>	<u>33</u>	<u>190</u>
Wheatley Harbour Station #6	2.2	<u>9.0</u>	<u>1.4</u>	29	<u>35</u>	<u>2.1</u>	0.07	310	<u>29</u>	28	<u>130</u>
PSQG LEL Conc. ($\mu\text{g/g}$ dry weight)	NA	6.0	0.6	26	16	2.0	0.20	460	16	31	120

Underlining indicates sediment trace metal concentrations that exceed PSOG-LELs.

NA - Not Available.

TABLE 4. Bulk concentrations of selected chlorinated organics and pesticides in control(s) and Wheatley Harbour sediment ($\mu\text{g/g}$ dry weight) used in sediment bioassays.

Station	<i>Total PCBs</i>	<i>HCB</i>	<i>pp-DDE</i>	<i>pp-DDD</i>	<i>α-Chlordane</i>
Control	ND	ND	<u>0.005 <T</u>	ND	ND
upstream Reference Station #1	<u>0.18 <T</u>	0.003 <T	<u>0.015</u>	0.006 <T	0.005 <T
Muddy Creek Station #2	<u>0.20 <T</u>	ND	<u>0.015</u>	ND	<u>0.010 <T</u>
Muddy Creek Station #3	<u>0.32</u>	ND	<u>0.035</u>	ND	<u>0.015 <T</u>
Muddy Creek Station #4	<u>0.23</u>	ND	<u>0.035</u>	ND	<u>0.010 <T</u>
Wheatley Harbour Station #5	<u>0.33</u>	ND	<u>0.030</u>	ND	<u>0.010 <T</u>
Wheatley Harbour Station #6	<u>0.32</u>	0.002 <T	<u>0.025</u>	ND	<u>0.015 <T</u>
PSQG LEL Conc. ($\mu\text{g/g}$ dry weight)	0.07	0.02	0.005	0.008	0.007

Underlining indicates sediment organic concentrations that exceed PSOG—LELs.

ND — Not Detected;

T — Trace Amount Detected.

During the frequent past dredging operations which occurred in the harbour these areas may have been more subjected to the greater deposition of re-suspended fine sediment particles. In this study, PCB levels were fairly consistent among the sample sites suggesting no active inputs of PCBs and these levels appear to be typical for the area (Wheatley Harbour RAP, 1992). In a 1987 survey, sediment PCB concentrations ranged from a low of 0.090 µg/g to a maximum of 0.455 µg/g (Table A3, OMOE, 1987) and correspond with the 1992 sediment PCB concentrations.

3.3 *Hexagenia limbata* Lethality and Growth Results

The biological data for the two endpoints, mortality and growth, are summarized in Table 5. Organism growth results are depicted in Figure 2. Mayfly survival was statistically similar among the control and test sediments ($p < 0.55$). Average percent mortality was minimal and fell within an identical range for both the control animals and those exposed to the test sediments (Range: 0% - 2.5%).

The majority of the test sediments resulted in mayfly growth that was significantly higher than the controls. Average individual wet weight for Stations #2, #3, #4 and #5 ranged from 34.1 mg to 44.0 mg, as compared to the reference control animals (29.8 mg). Only Station #6 sediment caused a 26% reduction in size relative to the reference control animals. An appropriate rate of growth is considered to be a doubling of the initial starting weight, consequently a target value of > 29 mg would be representative of average growth, and was attained in all sediments with the exception of Station #6. The differences in biomass attained in the two control sediments could be attributed to the quality and quantity of the nutritional value associated with each sediment. The reference control sediment would be more indicative of the type of detrital matter and conditions found within the study area, whereas the negative control sediment was stored several months prior to testing.

Sediment laboratory bioassays were previously performed in 1987 during the OMOE In-Place Pollutants study and followed a different test methodology as described in Lomas and Krantzberg (1988). In the 1987 study, field-collected mayfly nymphs were exposed to eight test sediments and two control sediments for 10 days. Test endpoints were limited to lethal effects. Sediments collected from Muddy Creek and within the harbour proper were found to be non-lethal (0 - 6%) (Table A4). These values are consistent with the 1992 results (this study), even given the longer test exposure (21-d) used in the latter toxicity tests. Sediments collected from one station in 1987 along the shore of Lake Erie, located just outside the harbour mouth, was found to kill 60% of the mayflies. This was later determined to be a result of the unsuitability of the substrate. The substrate was suboptimal in terms of both physical and nutrient characteristics as evident by the high amounts of organically-poor coarse-grained particles (Table A2).

TABLE 5. Summary of biological results on mayfly, midge and minnow sediment bioassays for control(s) and Wheatley Harbour sediments. Mean values (\pm standard deviation).

Test Organism	<i>Hexagenia limbata</i> (Mayfly)		<i>Chironomus tentans</i> (Midge)		<i>Pimephales promelas</i> (Fathead Minnow)
Station	% Mortality (N=4)	Ave. Individual Body Weight (mg wet wt.)	% Mortality (N=4)	Ave. Individual Body Weight (mg wet wt.)	% Modality(N=3)
	A	D	A	BC	A
Control	2.5 (5)	15.73 (2.3)	4.9 (6)	3.80 (0.9)	0 (0)

Upstream Reference	A	C	A	AB	A
Station #1	0 (0)	29.83 (3.1)	26.6 (31)	5.05 (1.2)	0 (0)

Muddy Creek	A	A	A	AB	A
Station #2	0 (0)	44.03 (4.4)	3.3 (4)	4.85 (0.5)	3.3 (6)
Muddy Creek	A	AB	A	BC	A
Station #3	2.5 (5)	38.98 (3.4)	8.3 (8)	3.94 (0.4)	0 (0)
Muddy Creek	A	A	A	A	B*
Station #4	0 (0)	42.79 (0.9)	8.3 (13)	5.82 (1.3)	36.6 (31)

Wheatley Harbour	A	BC	A	BC	A
Station #5	0 (0)	34.13 (1.6)	1.6 (3)	3.87 (0.7)	0 (0)
Wheatley Harbour	A	D	A	C	A
Station #6	0 (0)	21.80 (3.4)	9.9 (12)	2.82 (0.7)	0 (0)

* % Mortality value is significantly different than the control and reference sediment (Dunnett's t-test; $p < 0.05$).

A Means sharing a common letter within a column are not significantly different; Tukey's HSD test for % Mortality ($p < 0.05$) and planned comparisons using LSMEANS for comparing Body Weight ($p < 0.01$).

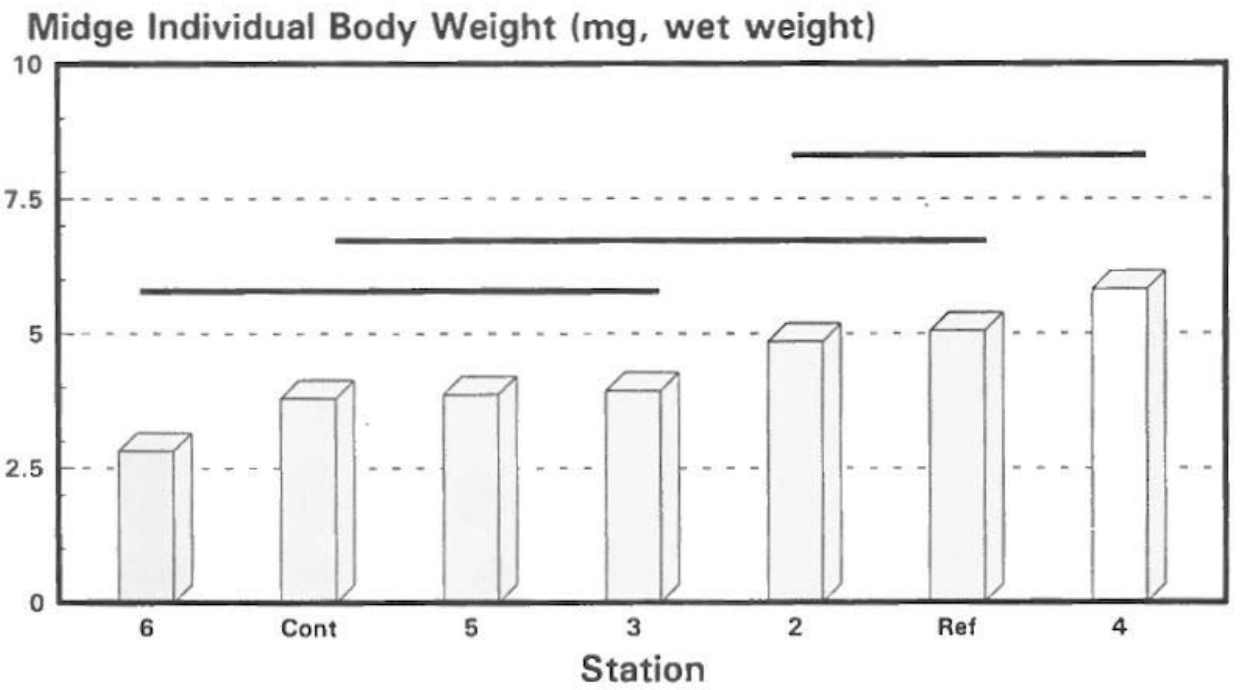
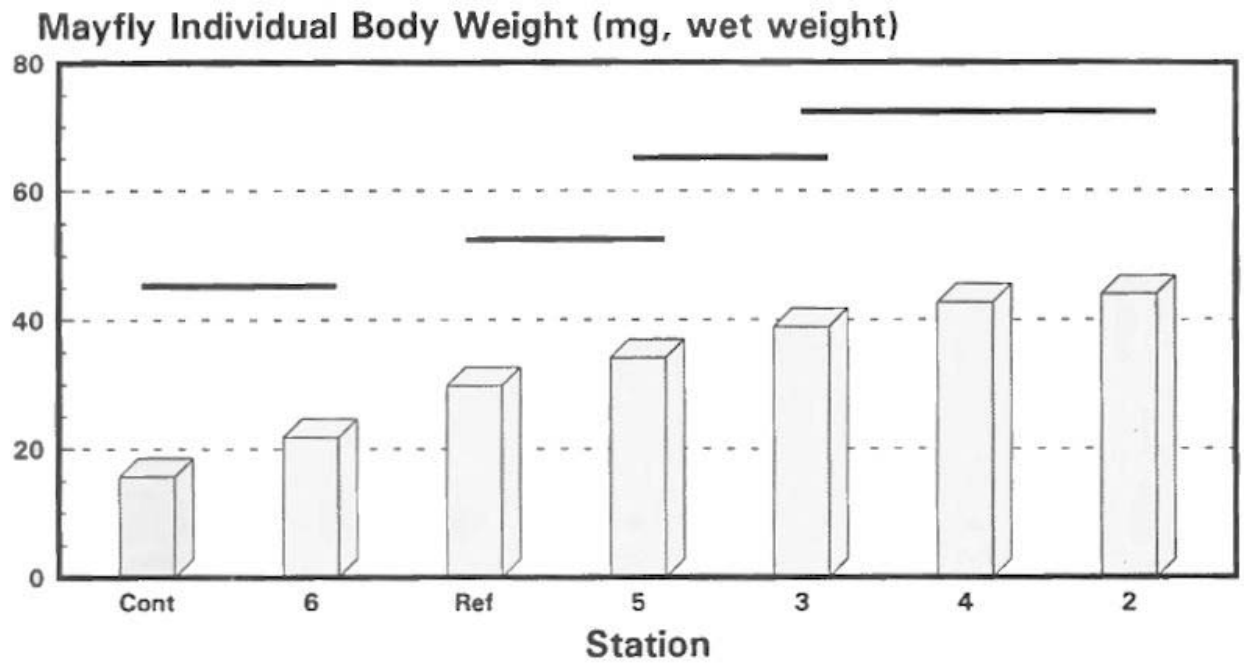


Figure 2. Organism growth response to Wheatley Harbour sediment 1992. Lines indicate significant groups.

3.4 *Chironomus tentans* Lethality and Growth Results

Results for chironomid growth and lethality are reported in Table 5 and growth illustrated in Figure 2. Chironomid mortality for the negative control sediment was well below the acceptable control mortality of 25%. The reference control sediment exhibited higher percent mortality (26%) and a higher degree of variability. Statistical analysis revealed no significant differences in midge mortality among sites ($p < 0.60$) nor between the test sediments (Range: 1.6% to 9.9%) and either of the control sediments (Range: 4.9% to 26%).

The surviving *C. tentans* larvae for each of the test sediments showed comparable growth with at least one of the control groups. The only significant response was noted for Station #6 sediment (2.8 mg), which had lower midge body weights relative to the reference control and Station #2 and #4 sediments (Range: 4.8 mg to 5.8 mg) ($p < 0.0001$).

3.5 *Pimephales promelas* Lethality Results

Juvenile fathead minnow percent mortality data is reported in Table 5. Station #4 sediment was moderately toxic to fathead minnows, resulting in 36% mortality. This value was significantly higher than the control percent mortality of 0% and the remaining test sediments (Range: 0 - 3% mortality) ($p < 0.0008$). Death of fathead minnows exposed to Station #4 sediment initially began on Day 14 and was unequally distributed among the replicates. The final percent mortality for the individual replicates was as follows: 30%, 10% and 70% (Table A5).

For the most part, sediment toxicity to *P. promelas* coincided with bioassay results from 1987 (OMOE, 1987). Bottom sediments collected from Muddy Creek and Wheatley Harbour in 1987 were primarily non-lethal (Table A4) and were comparable to those levels found in this study, despite the differences in exposure duration (10 vs 21 days). Among the 8 test locations from the 1987 study, only one test sediment caused 100% mortality. It was suspected that elevated ammonia levels may have been the cause, since the sediment contained the highest amounts of TKN (6.6 mg/g) relative to the other test sites and may have been a source of nitrogen ions (Table A2). Similarly, in this study, the sediment with relatively high concentrations of TKN (7.0 mg/g) was found to be moderately toxic. Direct measurements for total ammonia in the overlying water were not included in the routine monitoring for either study.

3.6 Comparison Among Test Organisms and Endpoints

Among the lethal endpoints, the fathead minnow assay was the only test species that identified a difference in sediment quality (D.P. = 7.0) (Table 6). Station #4 sediment was found to be moderately toxic in the 21-day test to fish but was not supported by the two

TABLE 6. Test precision and discriminatory power of biological endpoints measured for Wheatley Harbour bioassays.

<i>Endpoint</i>	<i>Sample Size</i>	<i>Average of s.d.'s</i>	<i>Average^a of C.V.'s (%)</i>	<i>Mean Maximum</i>	<i>Mean Minimum</i>	<i>Discriminatory Power^b</i>
<i>H. limbata</i>						
(Mayfly)						
% Mortality	7	1.4	197	2.5	0.0	1.7
Growth	7	2.7	8	44.0	15.7	10.4
<i>C. tentans</i>						
(Midge)						
% Mortality	7	11.0	123	26	1.6	2.2
Growth	7	0.8	18	5.8	2.2	3.7
<i>P. promelas</i>						
(Minnow)						
% Mortality	7	5.2	91	36	0.0	7.0

a C.V.= 100*Ave. s.d./Y

b DP = (Max Y—Min Y)/Ave. s.d.

TABLE 7. Spearman Rank Correlation coefficients among toxicity results for endpoints measured in test and control sediment. Significance level is reported in parenthesis.

	<i>H. limbata</i> % Mortality	<i>H. limbata</i> Growth	<i>C. tentans</i> % Mortality	<i>C. tentans</i> Growth
<i>H. limbata</i> Growth	-.316 (.43)			
<i>C. tentans</i> % Mortality	-.079 (.84)	-.306 (.45)		
<i>C. tentans</i> Growth	-.234 (.55)	.630 (.12)	.200 (.26)	
<i>P. promelas</i> % Mortality	-.394 (.33)	.757 (.06)	-.179 (.65)	.674 (.09)

benthic invertebrate assays (Range D.P.: 1.7 - 2.2). Organism lethality for all three test species had a limited response range as reflected in the poor test precision values (91 - 197 % C.V.).

The sublethal endpoints measured for both of the benthic species had the best test precision (8 - 18% C.V.) and a good response range, particularly for *H. limbata*, which recognized more differences among and between sediments (D.P. = 10.4). However, there were no consistent trends in sediment contamination among each of the test species and test endpoints. The lack of identifiable differences among sediment types was further confirmed using nonparametric Spearman Rank correlation analysis. No significantly important correlations among the endpoints were reported (Table 7).

3.7 Relationships among Biological Endpoints, Sediment Properties and Provincial Sediment Quality Guidelines

Analysis of inorganic and organic contaminants in bottom sediments from Muddy Creek and Wheatley Harbour test locations exhibited a fairly low degree of contamination that was comparable to background values as measured at the reference site (Station #1). In fact, bulk sediment metal concentrations for the test sediments are similar (less than double) to background concentrations that are typically found in lake surficial sediment from the Great Lakes basin (Ankley *et al.*, 1994; Persaud *et al.*, 1992). At these levels, the absence of sediment toxicity and any considerable sublethal impacts to the two benthic invertebrate test organisms in the 11 and 21-day exposures, is not unexpected.

Spearman Rank correlation analysis failed to find any significant correlations between sediment metal concentrations and any of the biological effects (Table 8). Although the midge mortality endpoint was positively correlated with sediment HCB concentrations, neither of these parameters were considered important in the data interpretation. There was only a single occurrence of a significant growth response and this was a minor reduction in mayfly nymph biomass for Station #6 sediment. There was no sufficient explanation for the possible cause for this isolated response. Often chemical concentrations in sediment can be described in terms of sediment physical characteristics due to the binding capacity and depositional behaviour of sediment particles. Sediment grain-size distribution and organic content of the sediment failed to predict the relative distribution of metals and organic chemicals. The only exception was for arsenic and total PCBs with %fines (Table 9). The correlation analysis may have been biased due to the low variability in the chemical and physical data for the reference control and test sediments.

The selective moderate toxic effect to fathead minnows of Station #4 sediment, was likely due to naturally occurring causes. A slow but gradual build-up of ammonia may have been due to the unusually high TKN levels in the sediment. The situation was probably further exacerbated due to the static exposure conditions and the use of minimal water replacements during the test. Also, relative to the benthic species, fish are generally more sensitive to

TABLE 8. Correlation analysis summary for the biological endpoints against sediment physical and chemical parameters. Sample size N=6.

<i>Test Organism / Biological Endpoint</i>	Mayfly Mortality	Mayfly Growth	Midge Mortality	Midge Growth	Minnow Mortality
<i>Sediment Characteristic</i>					
Total organic carbon	n.s.	n.s.	n.s.	n.s.	n.s.
Percent fines	n.s.	n.s.	n.s.	n.s.	n.s.
<i>Bulk sediment chemical concentration</i> ^a			HCB		

n.s. — Not Significant; $p > 0.05$.

^a $p < 0.05$.

TABLE 9. Spearman Rank Correlation coefficients for bulk sediment metal and organic chemical concentrations against total organic carbon and percent fines for the Wheatley Harbour sediments. Sample size N=6.

Metal	TOC	% Fines	Organic	TOC	% Fines
Arsenic	-.173 (.69)	.942 (.03)	Total PCBs	.246 (.58)	.880 (.05)
Cadmium	-.523 (.24)	.091 (.83)	Hexachlorbenzene	-.823 (.06)	-.270 (.54)
Chromium	-.434 (.33)	.657 (.14)	pp- DDD	-.531 (.23)	-.392 (.37)
Copper	.579 (.19)	.714 (.11)	pp- DDE	.701 (.11)	.647 (.14)
Iron	-.434 (.33)	.657 (.14)	α -Chlordane	.328 (.46)	.185 (.67)
Mercury	.646 (.14)	-.576 (.19)			
Manganese	-.588 (.18)	.637 (.15)			
Nickel	.024 (.94)	.771 (.08)			
Lead	.215 (.62)	.030 (.94)			
Zinc	.463 (.29)	.085 (.84)			

TOC vs. % fines: $r=0.029$, $p<0.94$

Shading indicates significant correlation coefficients at $p<0.05$.

changes in ammonia (Hellawell, 1986).

One of the key objectives of this study was to determine the potential for PCBs to elicit organism acute or chronic effects. PCB sediment concentrations (0.20 - 0.33 µg/g) were closer to the PSQG-LEL concentrations, which is suggestive of only minor impacts on sediment-dwelling organisms. This appeared to be the case given the absence of any correlation between sediment and biological data (Table 8) and the generally low PCB sediment concentrations. The 1987 In-Place Pollutants study conducted in Wheatley Harbour included a series of laboratory sediment bioassays using procedures described in Lomas and Krantzberg (1988). The toxicity results are reported in Table A4 (OMOE, 1987). Despite the occasional significant biological response, there was no predictable pattern of toxicity in terms of PCB sediment contamination. Generally, the sediments were non-toxic over a sediment PCB concentration of 0.09 to 0.455 µg/g (Table A3).

Other laboratory studies which have investigated PCB-contaminated inland sites within the province, have found toxic effects at sediment total PCB concentrations substantially higher than those used in this study. For example, five sediment samples collected from Lyons Creek located near Welland, Ontario, in 1992, were primarily contaminated with PCBs (Bedard and Petro, 1992). Using procedures identical to this study (Bedard *et al.*, 1992) the bioassays found for three of the test sites, mayfly, midge and minnow mortality were below their respective acceptable control mortalities. Sediment PCB concentrations varied from 0.1 to 1.04 µg/g (dry weight). It was only at concentrations that surpassed 3.0 µg/g PCB, that a significant degree of toxicity was noted. Mayfly and midge percent mortality ranged from 40% to 66% at the two other sites. Sediment PCB concentration reached a maximum of 6.0 µg/g. In 1987, a biomonitoring program conducted in the Otonabee River and Rice Lake near Peterborough, Ontario showed no acute toxicity in laboratory sediment bioassays (Jaagumagi and Petro, 1991). The highest amount of mortality (only 20% mortality) to both mayfly nymphs and juvenile fathead minnows coincided with a single sample containing 6.1 µg/g PCB, after a 10 day exposure. The other sediments were generally non-lethal and contained 0.20 to 1.37 µg/g PCBs (n=14). It appears that only as the PCB concentration in the sediment approaches the PSQG-SEL (after correction for TOC) does the likelihood of toxicity increase.

Often laboratory sediment toxicity tests are done in conjunction with other types of field biomonitoring studies in order to obtain as complete an overview of ecosystem health as possible (Jaagumagi and Persaud, 1993). Concurrent field surveys were not completed during the 1992 study but information is available from work completed in 1987. The pattern of toxicity observed in the laboratory tests appears to have corresponded with the benthic community structure field assessment (Wheatley Harbour RAP, 1992). The number and type of benthic organisms collected in the field were not unlike those dominating other Lake Erie harbours. Oligochaeta and Chironomidae were present in the largest numbers with a higher density of worms associated with nutrient-enriched sediments located in Muddy Creek. Pockets of high organism diversity and abundance were noted in areas with macrophytes.

3.8 Relationships Between Chemical Bioaccumulation in *Pimephales promelas* and Sediment Properties

The examination of organic chemical availability to aquatic organisms is valuable for assessing the potential for chemical transfer through the food web. The primary objective of this bioassay is to make general observations on whole organism tissue concentrations as they relate to total organic chemical concentrations in the sediment. Whole body tissue concentrations (ng/g, dry weight) were measured for total PCBs, aldrin, α -, β - and γ -BHC, α - and γ -chlordane, DDD, DDE, DDT, heptachlor, hexachlorobenzene, mirex, toxaphene and octachlorostyrene. All organochlorine and pesticide tissue concentrations were non-detectable with the exception of aldrin. Trace amounts of aldrin were reported for the water-only and negative control minnows at the same average concentration of $36.8 \text{ ng/g} \pm 4.7$. These values were similar to background concentrations of 40.2 ng/g measured in a set of pre-exposed minnows and are indicative of levels acquired through culturing practices. Higher aldrin concentrations were found in each of the test sediments and the reference control animals (Range: 73.7 to 157.4 ng/g). Elevated tissue concentrations were found for Station #2, #3, #4 and #5 exposures. Since the reference control and test sediments did not contain detectable levels of aldrin ($<0.001 \text{ } \mu\text{g/g}$, dry weight), this precluded the examination of relative chemical bioavailability among sites.

The only organic compounds measured above trace concentrations in the sediment were pp-DDE and total PCBs. Neither of these chemicals was found in the whole fish samples. Similarly, laboratory bioaccumulation tests completed in 1987 using mayfly nymphs and juvenile fathead minnows with Wheatley Harbour bottom sediments found no appreciable uptake of PCBs (OMOE, 1987). Also, during the same year, field caging studies conducted within Wheatley Harbour in the vicinity of discharge failed to show any PCB availability to mussels after a 21 day exposure period (Wheatley Harbour RAP, 1992). In other circumstances, the same technique has been found useful in the identification of PCB contamination, particularly if there is an active source in the water column (Maude *et al.*, 1992).

There are several plausible explanations for the lack of chemical bioaccumulation observed in the laboratory bioaccumulation tests. Theoretically, the behaviour of neutral organic compounds e.g. PCBs, is dictated by the relative partitioning of the chemical between organic phases. At equilibrium, the organic chemical is distributed between the organic carbon fraction of the sediment (Karickhoff, 1981; Karickhoff *et al.*, 1979) and the lipid fraction in the organism (Gobas *et al.*, 1989). The amount of organic carbon present in the sediment could reduce the availability of neutral organic compounds. This relationship does not entirely apply to the Wheatley Harbour situation because there was no significant correlation found between sediment PCB concentrations and sediment TOC (Table 9). However, given both the relatively high amounts of organic matter in the test sediments (3.7% to 8.5% TOC) and the low PCB sediment concentrations (0.20 to $0.33 \text{ } \mu\text{g/g}$), there could be a reduction in the potential availability of the chemical to the biota. Several authors have noted higher PCB tissue residues

are often associated with sediment with low TOC ($<3\%$) (Boese *et al.*, 1995; Lake *et al.*, 1990). These conditions did not apply to the Wheatley Harbour tests. In addition, the fish used in this study had low levels of lipid (1.2 to 2.1% lipid, wet weight) in which the contaminant could partition into and could further explain the lack of uptake. Another factor that can affect final tissue chemical concentrations is the duration of the experiment. The laboratory exposure duration of 21 days may be insufficient to attain steady-state tissue concentrations and would therefore underestimate the potential for chemical bioaccumulation. Boese *et al.*, (1995), estimated the time to reach steady-state levels in the marine clam, *Mercenaria nasuta*, for 12 of 13 PCB congeners in spiked-sediment was 28 to 42 days. It is plausible that at low PCB sediment concentrations (less than twice trace amounts), the chemical was readily excreted or transformed by the fish or could not be measured accurately in the biota samples. Along the same lines, even though PCBs are fairly persistent, at lower concentrations there may have been a partial loss of PCBs during storage of the sediment prior to testing.

The pathway of uptake of the chemical into the fish is critical in understanding contaminant movement into the food chain. Under laboratory conditions, the sediment acts as the only potential source of PCBs to the organism. As a pelagic species, the transfer of contaminant from the sediment into the water column and into the fish would represent one exposure pathway. The hydrophobic properties of PCBs would limit its desorption from the sediment into the overlying water, thereby, limiting its release into the water column where it can be readily transferred into the minnow through the gills (Gobas *et al.*, 1986). Another route of uptake could be through the direct ingestion of sediment particles while foraging. This can lead to two different scenarios. The fairly high amounts of organic matter associated with the test sediments, combined with sediment resuspension due to feeding and swimming activities, could have led to the formation of a flocculent layer of lighter material settling above the heavier sediment particles which may have been ingested by the minnow but would not be associated with high PCB levels. The distribution of PCBs was better explained by the relative amounts of silt/clay-sized particles (Table 9). The finer-grained sediment particles would be found in the resuspended solids. Even though these particles would contain higher concentrations of PCBs, it may not be as nutritionally beneficial to the minnow because of its non-organic quality (insignificant correlation between %fines and TOC; Table 9).

Ankley *et al.*, (1992) observed that in laboratory exposures using PCB-contaminated sediments, fathead minnow tissue concentrations were not necessarily representative of chemical exposure attained by benthic invertebrate species nor would they accurately reflect levels obtained in the field given the controlled state of the laboratory test. Also, one would anticipate a higher availability to sediment dwelling organisms given the same test design. Although we did not find fathead minnows to be a responsive organism for PCB uptake, given due consideration to the type of sediment and sediment chemical concentrations, another laboratory study did prove that fathead minnows were quite capable of bioaccumulating levels of PCBs higher than those measured in the sediment (Bedard and Petro, 1992). Fathead minnow PCB whole organism concentrations ranged from 12.6 to 16.6 $\mu\text{g/g}$ (dry weight) for the

most contaminated sites (PCB sediment concentrations: 3.08 to 6.04 µg/g). After correcting for differences in sediment TOC and biota percent lipid, the biota-sediment accumulation factors averaged 12.8. Jaagumagi and Petro (1991) exposed fathead minnows and mayfly nymphs to sediment containing PCB levels ranging from non-detectable (<0.02µg/g) to 6.14 µg/g, and found no differences in PCB tissue levels between pelagic and benthic species.

4.0 CONCLUSIONS

The surficial sediments collected from Muddy Creek and Wheatley Harbour and used for laboratory sediment toxicity testing were found to be generally not lethal or sublethal to the three test organisms. There was a lack of consistent, significant biological response among sites (Table 10). The few exceptions included a moderate toxic response to the fathead minnow, *Pimephales promelas* at Station #4, located in Muddy Creek downstream of the Omstead Foods Ltd. discharge, and was attributed to ammonia build-up during the test. Similarly, only one of the test sediments elicited a significant sublethal effect to one of the benthic organisms. A minor reduction in *Hexagenia limbata* growth was found in Station #6 sediment collected in the main navigational channel of the harbour. Overall, the toxicological information does not provide overwhelming evidence to suggest the potential for severe sediment degradation under laboratory conditions.

Sediment bulk chemistry did not reveal any exceedences above the Provincial Sediment Quality Guidelines Severe Effect Level concentrations for the inorganic and organic chemicals selected for analysis and were either measured at concentrations at or slightly above their respective Lowest Effect Level concentrations or were comparable to background levels for the general area.

Sediment-bound organic contaminants were not readily available to the fathead minnow and failed to accumulate sufficient quantities of those chemicals found at measurable levels in the sediment (e.g. polychlorinated biphenyls, hexachlorobenzene, pp-DDE, pp-DDD and α -chlordane). A number of abiotic and biotic factors need to be taken into account in order to understand the fate and movement of organic contaminants and several explanations were provided to account for the lack of chemical uptake.

In summary, the findings from this laboratory study have shown a low degree of biological effects and organic chemical bioavailability from sediments collected in the Wheatley Harbour AOC and supports the previous work of others, both in the laboratory and the field for this area (Wheatley Harbour RAP, 1992; OMOE, 1987).

TABLE 10. Association between sediment toxicity and sediment total PCBs concentrations.

Station	Total PCBs (µg/g dry wt)	Mayfly Mortality	Mayfly Ave. wet wt.	Midge Mortality	Midge Ave. wet wt	Minnow Mortality
Control	ND	N	N	N	N	N
Upstream Reference Station #1	<u>0.18 <T</u>	N	N	N	N	N
Muddy Creek Station #2	<u>0.20 <T</u>	N	N	N	N	N
Muddy Creek Station #3	<u>0.32</u>	N	N	N	N	N
Muddy Creek Station #4	<u>0.32</u>	N	N	N	N	T
Wheatley Harbour Station #5	<u>0.33</u>	N	N	N	N	N
Wheatley Harbour Station #6	<u>0.32</u>	N	T	N	N	N

N — Not Toxic; T — Toxic.

Underlining indicate sediment chemical concentrations that exceed PSOG—LELs.

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APPENDICES

TABLE A1. Analytical detection limits for nutrients, inorganic and organic contaminants in sediment and biota samples.

Matrix: Sediment (units reported on a dry weight basis)			Matrix: Biota (units reported on a wet weight basis)		
Class	Parameter	Detection Limit	Class	Parameter	Detection Limit
Nutrient	Loss on Ignition	1.0 mg/g	Organic	Total PCBs	20 ng/g
	Total Organic Carbon	0.2 mg/g		Aldrin	1 ng/g
	Total Kjeldahl Nitrogen	0.025 mg/g		alpha-BHC	1 ng/g
	Total Phosphorus	-		beta- BHC	1 ng/g
Trace Metal	Arsenic	-		gamma-BHC	1 ng/g
	Cadmium	0.05 µg/g		alpha-Chlordane	2 ng/g
	Chromium	1.0 µg/g		gamma-Chlordane	2 ng/g
	Copper	0.5 µg/g		pp-DDD	5 ng/g
	Iron	200 µg/g		pp-DDE	1 ng/g
	Lead	1.25 µg/g		op-DDT	5 ng/g
	Mercury	0.01 µg/g		pp-DDT	5 ng/g
	Nickel	0.2 µg/g		Heptachlor	1 ng/g
Organic	Zinc	2.0 µg/g		Hexachlorobenzene	1 ng/g
	Total PCBs	0.020 µg/g		Mirex	5 ng/g
	Aldrin	0.001 µg/g		Octachlorostyrene	1 ng/g
	alpha-BHC	0.001 µg/g		Toxaphene	200 ng/g
	beta-BHC	0.001 µg/g			
	gamma-BHC	0.001 µg/g		Percent lipid	-
	alpha-Chlordane	0.002 µg/g			
	gamma-Chlordane	0.002 µg/g			
	Oxychlordane	0.002 µg/g			
	pp-DDD	0.005 µg/g			
	pp-DDE	0.001 µg/g			
	op-DDT	0.005 µg/g			
	pp-DDT	0.005 µg/g			
	Dieldrin	0.002 µg/g			
	Endosulfan I	0.002 µg/g			
	Endosulfan II	0.004 µg/g			
	Endosulfan Sulphate	0.004 µg/g			
	Endrin	0.004 µg/g			
	Heptachlor	0.001 µg/g			
	Heptachlor epoxide	0.001 µg/g			
	Hexachlorobenzene	0.001 µg/g			
	Methoxychlor	0.005 µg/g			
	Mirex	0.005 µg/g			
	Octachlorostyrene	0.001 µg/g			

TABLE A2. Summary of concentrations for nutrients and trace metals in surficial sediments collected at various locations in the Wheatley Harbour area in 1987 (OMOE, 1987).

MOEE 1992 Survey Station	OMOE 1987 Survey Station	TOC mg/g	TKN mg/g	Al µg/g	As µg/g	Cd µg/g	Cr µg/g	Cu µg/g	Fe µg/g	Hg µg/g	Mn µg/g	Ni µg/g	Pb µg/g
Upstream Reference Stn #1	Upstream Reference Stn #19	<u>40</u>	3.68	25000	5.0	<u>1.10</u>	<u>34</u>	<u>32</u>	<u>25000</u>	0.07	230	<u>28</u>	23
Muddy Creek Stn #2	Muddy Creek Stn #3	<u>63</u>	<u>6.64</u>	21000	<u>6.6</u>	<u>1.20</u>	<u>33</u>	<u>40</u>	<u>24000</u>	0.11	230	<u>28</u>	27
Stn #3	Stn #5	<u>78</u>	<u>5.80</u>	14000	5.4	<u>0.98</u>	25	<u>34</u>	<u>17000</u>	0.08	220	<u>24</u>	29
-	Stn #21	<u>70</u>	<u>5.56</u>	20000	<u>6.0</u>	<u>1.20</u>	<u>31</u>	<u>40</u>	<u>22000</u>	0.11	220	<u>28</u>	<u>39</u>
Wheatley Harbour Stn #5	Wheatley Harbour Stn #7	<u>47</u>	4.32	24000	<u>6.6</u>	<u>1.20</u>	<u>37</u>	<u>40</u>	<u>26000</u>	0.09	280	<u>31</u>	29
Stn #6	Stn #9	<u>28</u>	2.42	8500	4.3	0.55	23	<u>22</u>	<u>15000</u>	0.05<T	410	<u>18</u>	<u>32</u>
-	Stn #10	<u>40</u>	3.24	19000	<u>7.4</u>	<u>1.20</u>	<u>32</u>	<u>38</u>	<u>24000</u>	0.07	360	<u>29</u>	<u>34</u>
-	Lake Erie Stn #12	<u>5</u>	0.16	3700	<u>7.1</u>	0.39	11	10	<u>9300</u>	ND	370	9	7
PSQG LEL Conc. (µg/g dry weight)		1	4.80	NA	6.0	0.6	26	16	20000	0.2	460	16	31

< T - Trace amount measured; ND Not Detected; NA - Not Available
Underlining Indicates sediment trace metal concentrations that exceed PSOG-LELs.

TABLE A3. Summary of concentrations for selected organochlorine pesticides and total polychlorinated biphenyls in surficial sediments collected at various locations in the Wheatley Harbour area in 1987 (OMOE, 1987).

MOEE 1992 Survey Station	OMOE 1987 Survey Station	Total PCBs µg/g	Hexachlorbenzene µg/g	Octachlorostyrene µg/g	pp-DDE µg/g
Upstream Reference Stn #1	Upstream Reference Stn #19	ND	ND	ND	<u>.009</u>
Muddy Creek Stn #2	Muddy Creek Stn #3	<u>0.130 <T</u>	0.002 <T	ND	<u>0.019</u>
Stn #3	Stn #5	<u>0.455</u>	0.002 <T	0.004	<u>0.047</u>
-	Stn #21	<u>0.390</u>	ND	0.003	<u>0.033</u>
Wheatley Harbour Stn #5	Wheatley Harbour Stn #7	<u>0.090 <T</u>	ND	ND	<u>0.013</u>
Stn #6	Stn #9	<u>0.115 <T</u>	ND	ND	<u>0.012</u>
-	Stn #10	<u>0.100 <T</u>	ND	ND	<u>0.008</u>
-	Lake Erie Stn #12	ND	ND	ND	ND
PSQG LEL Conc. (µg/g dry weight)		0.070	0.020	NA	0.005

< T - Trace amount measured; ND - Not Detected; NA - Not Available.

Underlining indicates sediment trace metal concentrations that exceed PSOG-LELs.

TABLE A4. Summary of biological results on mayfly and minnow sediment bioassays for control(s) and Wheatley Harbour sediments collected in 1987 (OMOE, 1987).

Test Organism	<i>Hexagenia limbata</i> (Mayfly)	<i>Pimephales promelas</i> (Fathead Minnow)
Station	% Mortality (N=3 / 5 animals per rep)	% Mortality (N=3 / 5 animals per rep)
Control	A 0 (0)	A 0 (0)
Upstream Reference	A	A
Station #19	0 (0)	6.6 (12)
Muddy Creek	A	B*
Station #3	0 (0)	100 (0)
Muddy Creek	A	A
Station #5	0 (0)	0 (0)
Muddy Creek	A	A
Station #21	0 (0)	0 (0)
Wheatley Harbour	A	A
Station #7	0 (0)	0 (0)
Wheatley Harbour	A	A
Station #9	6.6 (12)	0 (0)
Wheatley Harbour	A	A
Station #10	6.6 (12)	0 (0)
Wheatley Harbour	A	A
Station #11	6.6 (12)	0 (0)
Lake Erie	B*	A
Station #12	60.0 (40)	0 (0)

Mean values ± standard deviation.

* % Mortality value is significantly different than the control and reference sediment (Dunnett's t-test; $p < 0.05$).

A Means sharing a common letter within a column are not significantly different; Tukey's HSD test ($p < 0.05$).

TABLE A5. Raw biological data for 1992 Wheatley Harbour laboratory sediment bioassays.

Test Organism: Mayfly Nymph				Test Organism: Mayfly Nymph			
Station	Test	#Survived Replicate (Max. 10)	Individual Body Weight Replicate Mean (s.d.) (mg, wet wt)	Station	Test	#Survived Replicate (Max. 10)	Individual Body Weight Replicate Mean (s.d.) (mg, wet wt)
Control Honey H.	Rep A	10	16.92 (5.69)	Muddy Cr Stn #4	Rep A	10	42.74 (11.05)
	Rep B	10	14.79 (11.35)		Rep B	10	42.18 (16.95)
	Rep C	9	18.31 (11.90)		Rep C	10	44.20 (17.28)
	Rep D	10	12.89 (7.70)		Rep D	10	42.05 (21.26)
Reference Stn #1	Rep A	10	27.16 (10.72)	Wheatley H. Stn #5	Rep A	10	35.75 (17.46)
	Rep B	10	33.71 (11.67)		Rep B	10	32.55 (11.39)
	Rep C	10	31.12 (12.68)		Rep C	10	35.34 (14.99)
	Rep D	10	27.36 (10.32)		Rep D	10	32.88 (11.76)
Muddy Cr. Stn #2	Rep A	10	40.47 (17.52)	Wheatley H. Stn #6	Rep A	10	19.32 (7.74)
	Rep B	10	44.33 (10.21)		Rep B	10	19.27 (10.02)
	Rep C	10	50.19 (13.61)		Rep C	10	26.71 (12.84)
	Rep D	10	41.12 (19.16)		Rep D	10	21.89 (12.17)
Muddy Cr. Stn #3	Rep A	9	42.16 (13.17)				
	Rep B	10	34.58 (10.53)				
	Rep C	10	38.02 (10.90)				
	Rep D	10	41.16 (18.73)				

TABLE A5. Continued.

Test Organism: Midge Larvae				Test Organism: Midge Larvae			
Station	Test	#Survived (Max. 15)	Individual Body Weight Replicate Mean (s.d.) (mg, wet wt)	Station	Test	#Survived (Max. 15)	Individual Body Weight Replicate Mean (s.d.) (mg, wet wt)
Control Honey H.	Rep A	13	4.32 (1.74)	Muddy Cr. Stn #4	Rep A	15	6.61 (2.38)
	Rep B	14	2.86 (0.92)		Rep B	14	5.57 (2.65)
	Rep C	15	3.15 (1.73)		Rep C	15	4.03 (2.21)
	Rep D	15	4.86 (1.87)		Rep D	11	7.08 (4.08)
Reference Stn #1	Rep A	8	5.06 (2.38)	Wheatley H. Stn #5	Rep A	15	3.68 (2.17)
	Rep B	6	6.71 (3.07)		Rep B	15	4.34 (1.72)
	Rep C	15	3.80 (1.61)		Rep C	15	2.95 (1.62)
	Rep D	15	4.63 (3.14)		Rep D	11	4.49 (2.14)
Muddy Cr. Stn #2	Rep A	15	5.57 (2.44)	Wheatley H. Stn #6	Rep A	14	3.01 (1.05)
	Rep B	14	4.33 (2.27)		Rep B	14	2.70 (1.32)
	Rep C	15	4.96 (2.89)		Rep C	11	1.84 (0.55)
	Rep D	14	4.55 (1.99)		Rep D	15	3.72 (2.39)
Muddy Cr. Stn #3	Rep A	15	4.29 (1.72)				
	Rep B	14	4.27 (1.43)				
	Rep C	12	3.79 (2.44)				
	Rep D	14	3.43 (1.45)				

TABLE A5. Continued.

Test Organism: Fathead Minnow			Test Organism: Fathead Minnow		
Station	Test Replicate	#Survived (Max. 10)	Station	Test Replicate	#Survived (Max. 10)
Control Honey H.	Rep A	10	Muddy Cr. Stn #4	Rep A	7
	Rep B	10		Rep B	9
	Rep C	10		Rep C	3
Reference Stn #1	Rep A	10	Wheatley H. Stn #5	Rep A	10
	Rep B	10		Rep B	10
	Rep C	10		Rep C	10
Muddy Cr. Stn #2	Rep A	10	Muddy Cr. Stn #2	Rep A	10
	Rep B	10		Rep B	10
	Rep C	9		Rep C	10
Muddy Cr. Stn #3	Rep A	10			
	Rep B	10			
	Rep C	10			