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**BIOMONITORING POTENTIAL OF VARIOUS
AQUATIC ORGANISMS AND ANALYTICAL
CONSIDERATIONS FOR THE ONTARIO
MINISTRY OF ENVIRONMENT AND
ENERGY'S GREAT LAKES LONG-TERM
SENSING SITES PROJECT**

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SITES PROJECT**

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MANAGEMENT PERSPECTIVE

The Ontario Ministry of Environment and Energy's (MOEE) Great Lakes Long-Term Sensing Sites Project (LTSSP) was initiated in 1988 for the purpose of monitoring temporal trends in environmental quality in the nearshore waters of the Great Lakes. Caged mussels were used to monitor the bioavailability of contaminants at sites indicative of ambient (background) conditions, but results to date have been disappointing. Even after fairly lengthy exposure periods, residues accumulated by mussels were either non-detectable or extremely variable. In 1993, the National Water Research Institute (NWRI) was approached by the Project leader for assistance with (i) identifying a more suitable biomonitoring organism for the LTSSP and (ii) determining the analytical sensitivity that would be required to measure contaminant trends at these relatively uncontaminated sites. Samples of biota and sediment were collected from a representative site on the Niagara River and analyzed by NWRI, with assistance from the National Laboratory for Environmental Testing (NLET) and the Wastewater Technology Centre (WTC), for a number of organic and inorganic contaminants of interest. Some samples were also analyzed by MOEE using their routine analytical methods.

Zebra mussels and caged mussels appeared to be the most suitable organisms for monitoring metals and organic contaminants, respectively, but further studies must be conducted at other sites before firm recommendations can be made. An unexpected finding of the study was that caged mussels accumulated moderately high and relatively consistent concentrations of many contaminants, suggesting that they may be suitable biomonitors after all. The reason for this discrepancy with results from previous years was that NWRI, NLET and WTC used analytical methods with lower limits of quantitation for many of the contaminants than MOEE's routine methods. The results of this study provided MOEE with useful information on suitable biomonitoring organisms for the Great Lakes Long-Term Sensing Site Project, and on the level of analytical sensitivity that would be required to monitor temporal trends in contamination at these sites.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Le Projet des stations de surveillance à long terme des Grands Lacs (Great Lakes Long-Term Sensing Sites Project (LTSSP), du ministère de l'Environnement et de l'Énergie de l'Ontario (MEEO), a été lancé en 1988. L'objectif était de déterminer l'évolution chronologique de la qualité de l'environnement aquatique dans les secteurs côtiers des Grands Lacs. On employait des moules placées dans des cages pour surveiller l'assimilabilité des contaminants à des stations représentatives des conditions ambiantes (de fond); cependant, les résultats obtenus ont été décevants jusqu'à maintenant. Même au bout d'assez longues périodes d'exposition, soit que les résidus accumulés dans les tissus des moules n'étaient pas détectables, soit que leur concentration était très variable. En 1993, le directeur du projet a demandé l'aide de l'Institut national de recherche sur les eaux (INRE) pour (i) trouver un organisme plus indiqué pour le projet, et (ii) déterminer à quelle échelle de mesure il faudrait fonctionner pour observer l'évolution de la contamination à ces stations relativement peu polluées. Avec le concours du Laboratoire national des essais environnementaux (LNEE) et du Centre technique des eaux usées (CTEU), l'INRE a prélevé des échantillons d'organismes et de sédiments à un site représentatif de la rivière Niagara, et a dosé des contaminants organiques et inorganiques d'intérêt. Le MEEO a également analysé certains échantillons en utilisant ses méthodes courantes d'analyse.

Les moules zébrées et les moules en cage paraissent être les organismes qui se prêtent le mieux à la surveillance de la concentration des métaux et des contaminants organiques, respectivement, mais il faut poursuivre la recherche à d'autres stations avant de pouvoir formuler des recommandations certaines. L'étude a eu un résultat inattendu : on a constaté que les moules en cage accumulaient un grand nombre de contaminants en concentration modérément élevée et assez constante; cela nous fait dire qu'elles peuvent constituer de bons indicateurs biologiques malgré tout. L'explication de l'écart entre les résultats obtenus ici et ceux des années antérieures tient à ce que l'INRE, le LNEE et le CTEU ont appliqué des méthodes d'analyse ayant un seuil de détection de nombreux contaminants inférieur à celui des méthodes courantes employées par le MEEO. L'étude a fourni à ce ministère des renseignements utiles sur les organismes susceptibles de constituer de bons

indicateurs biologiques dans le cadre du Projet des stations de surveillance à long terme des Grands Lacs ainsi que sur le degré de sensibilité requis, lors des dosages, pour suivre l'évolution de la contamination dans le temps à ces stations.

ABSTRACT

In 1993, NWRI and MOEE conducted a collaborative study to identify suitable biomonitoring organisms for MOEE's Great Lakes Long-Term Sensing Sites Project, and to determine the analytical sensitivity required to measure contaminant trends at the relatively uncontaminated sites monitored in this project. Samples of zebra and quagga mussels, amphipods, snails, crayfish, spottail shiners, caged mussels (*Elliptio complanata*) and sediment from a representative site on the Niagara River were analyzed for PCBs, PAHs, tri- and tetra-chlorobenzenes, hexachlorobutadiene, hexachloroethane, organochlorine pesticides (sediment only) and selected metals (sediment, mussels and spottail shiners only) by NWRI, NLET or WTC. Some samples were also analyzed by MOEE using their routine analytical methods.

Concentrations of metals were higher in all types of mussels than shiners. As variability among replicates was lowest for zebra mussels, they were tentatively recommended as biomonitors for metals. Concentrations of total PCBs were highest in spottail shiners and large quagga mussels (≈ 400 ng/g dry weight), moderate in amphipods and caged mussels (≈ 250 ng/g), and lowest in small zebra mussels that were analyzed with their shells on (34 ng/g). As variability among replicates was lowest for caged mussels, they were tentatively recommended as biomonitors for PCBs. PCB congener-class profiles differed greatly among organisms, suggesting different routes of exposure and/or metabolic capabilities. All biota contained higher proportions of the more highly chlorinated PCBs than sediment. Only traces of PAHs, CBs, HCB and HCE were detected in a few biota samples, thus no suitable biomonitor could be recommended for these compounds.

Levels of some target contaminants in samples from the Niagara River were too low to be detected using MOEE's routine analytical methods, but could be quantified using the methods of other laboratories. Specifically, NLET reported values for Ni, Pb and Hg in mussels, and Cd and Pb in sediment, NWRI reported values for Σ PCBs in biota and sediment, and PECB and HCB in sediment, and WTC reported values for PAHs in sediment that were all below MOEE's limits of quantitation. In order to monitor contaminant trends at the long-term sensing sites, it is recommended that MOEE adopt more sensitive analytical methods for some of the contaminants of interest.

RÉSUMÉ

En 1993, l'INRE et le MEEQ ont procédé à une étude conjointe dont l'objectif était d'identifier des organismes susceptibles de constituer de bons indicateurs biologiques pour le Projet des stations de surveillance à long terme des Grands Lacs du MEEQ. On voulait aussi déterminer quel degré de sensibilité doivent avoir les méthodes d'analyse pour mesurer l'évolution dans le temps de la contamination aux stations assez peu contaminées qui ont été choisies pour la tenue de ce projet. L'INRE, le LNEE ou le CTEU ont dosé les BPC, les HAP, les tri- et les tétrachlorobenzènes, l'hexachlorobutadiène, l'hexachloroéthane, les pesticides organochlorés (dans les sédiments uniquement) et certains métaux (dans les sédiments, les moules et la queue à tache noire uniquement) dans les tissus de moules zébrées et quagga, d'amphipodes, d'escargots, d'écrevisses, de queues à tache noire et de moules en cage (*Elliptio complanata*) ainsi que dans les sédiments prélevés à une station représentative de la rivière Niagara. Le MEEQ a également analysé certains échantillons en utilisant ses méthodes courantes d'analyse.

La concentration des métaux était plus élevée dans tous les types de moules que dans les tissus de la queue à tache noire. Puisque la variabilité entre spécimens était la plus réduite chez la moule zébrée, on a provisoirement recommandé cet organisme comme indicateur biologique de la pollution par les métaux. La concentration du total des BPC était la plus élevée chez la queue à tache noire et les moules quagga de grande taille ($\approx 400 \text{ ng.g}^{-1}$ en masse sèche), modérée chez les amphipodes et les moules en cage ($\approx 250 \text{ ng.g}^{-1}$) et la plus faible chez les moules zébrées de petite taille soumises au dosage avec leur coquille ($\approx 34 \text{ ng.g}^{-1}$). Puisque la variabilité entre spécimens était la plus réduite chez les moules placées en cage, on a provisoirement recommandé cet organisme comme indicateur biologique de la pollution par les BPC. Les profils obtenus selon les classes toxiques de congénères de BPC différaient considérablement entre les organismes; cela paraît indiquer l'existence de différentes voies d'exposition ou encore de différents taux de métabolisme de ces substances. L'ensemble du biote contenait une proportion des BPC les plus chlorés supérieure à celle mesurée dans les sédiments. On n'a détecté que des traces de HAP, de CB, de HCBd et de HCE dans quelques spécimens biologiques. Il était donc impossible de recommander un indicateur biologique utile pour ces substances.

La concentration de certains des contaminants dosés dans des échantillons provenant de la rivière Niagara ne pouvait pas être détectée au moyen des méthodes courantes d'analyse du MEEQ, mais pouvait l'être par celles des autres laboratoires. De manière précise, le LNEE a dosé le Ni, le Pb et le Hg dans les tissus des moules, ainsi que le Cd et le Pb dans les sédiments; l'INRE a dosé l'ensemble des BPC dans les spécimens biologiques et les sédiments, ainsi que le penta-et l'hexachlorobenzène dans les sédiments, et le CTEU a dosé les HAP dans les sédiments, tous à des concentrations inférieures aux seuils de quantification des méthodes utilisées par le MEEQ. Pour qu'il soit en mesure de relever les tendances de la contamination à ces stations de surveillance à long terme, il est recommandé que le MEEQ adopte des méthodes d'analyse plus sensibles pour certains contaminants d'intérêt.

INTRODUCTION

In 1993, a collaborative study was conducted by J.L. Metcalfe-Smith, Aquatic Ecosystem Protection Branch, National Water Research Institute (NWRI), and E.T. Howell, Environmental Monitoring and Reporting Branch, Ontario Ministry of Environment and Energy (MOEE), to identify potential biomonitors for the Great Lakes Long-Term Sensing Sites Project (LTSSP). The objective of the LTSSP is to identify changes in environmental conditions in the nearshore areas of the Great Lakes by monitoring water and sediment quality and biological indicators over time at index stations. In the past, caged mussels (*Elliptio complanata*) were used to monitor the bioavailability of contaminants at sites throughout the Great Lakes. Sampling was conducted at sites considered to represent exposure to contaminants under ambient (background) conditions, and at sites on the periphery of impacted locations where chronic low-level exposure to contaminants was expected. Results to date have been disappointing. Even after fairly lengthy exposure periods, concentrations of metals in the soft tissues of mussels typically did not change from pre-exposure levels, and concentrations of organic contaminants were frequently non-detectable or extremely variable (e.g., Pope 1993). The main goal of the present study was to explore alternative biomonitors that may be more suitable candidates for the LTSSP. The ideal biomonitor would: (i) be readily available at a majority of sites over a range of habitat conditions, (ii) accumulate high concentrations of the contaminants of interest over a relatively short period of time, and (iii) exhibit minimal variation in residue levels among individuals or replicate samples collected from the same site at the same time.

In previous years, caged mussels from the LTSSP were analyzed for organic contaminants using methods specifically designed for MOEE's Sport Fish Contaminant Monitoring Program. There was some concern that these methods might not be sensitive enough to support the analytical requirements of the LTSSP, which focuses on relatively uncontaminated sites. Therefore, a second goal of the study was to identify the level of detection that would be needed to provide quantitative results for target compounds in biota obtained from the long-term sensing sites. This was accomplished using a combination of research-level analysis and analytical services available to NWRI.

MATERIALS AND METHODS

Study Site

A site on the lower Niagara River, namely, station 129 near the outflow of the river (Fig. 1), was selected for study. Pollution levels at this location are typical of the exposure regimes that characterize many of the long-term sensing sites. There is a long record of environmental data available from various agencies that indicates chronic low-level exposure to a variety of contaminants in the lower river near the outflow (e.g., Suns *et al.* 1991; The Niagara River Data Interpretation Group "C" 1994). Further information on habitat and environmental conditions at station 129 is provided by Howell (in preparation).

Sampling Methods

To maximize the probability of locating a suitable biomonitor, a range of organisms in terms of both taxonomy and food habits was sought. In an attempt to obtain large numbers of a variety of organisms, several types of artificial substrate samplers were deployed at station 129 in early May 1993, and allowed to colonize until the end of July. On 27 July 1993, the samplers were retrieved and the organisms removed. The collection was supplemented by grab sampling and trapping in the vicinity of the station. Samples of zebra mussels (*Dreissena polymorpha*), quagga mussels (*Dreissena bugensis*), oligochaetes, chironomids, amphipods (*Gammarus* sp.), snails (*Physella gyrina* and *Helisoma anceps*) and crayfish (*Orconectes propinquus*) were obtained. Samples of surface sediment were collected from the same site on 28 July 1993 using a Shipek grab. On 13 September 1993, young-of-the-year spottail shiners (*Notropis hudsonius*) were collected using a beach seine. Due to the absence of habitat for spottail shiners at station 129, the collections were made at a more suitable location approximately 1650 m downstream. Composite samples of each type of organism were wrapped in hexane-rinsed aluminum foil, placed in Whirl-pac® bags and frozen on dry ice at the time of collection. Three samples of spottail shiners were not wrapped in foil, so that they would be suitable for the analysis of metal residues.

Sample Preparation

As organic contaminants were the main focus of the study, samples of all types of organisms were prepared for analysis of organic compounds. Where sufficient material was available (zebra mussels, quagga mussels and spottail shiners), samples were also prepared for analysis of metals. As previously mentioned, the main reason for conducting the study was to identify one or more biomonitors that would accumulate higher and/or less variable concentrations of contaminants than caged mussels. However, caged mussels were not deployed in 1993. In order to compare caged mussels with the alternative biomonitors, archived mussels that had been exposed at station 129 for two 10-week periods in 1992 (May to July and July to October) were also analyzed for organic contaminants. The mussels were obtained from Balsam Lake, Ontario, and deployed in wire cages placed on the river bottom. Further information on the incubation procedures for caged mussels is given in Howell (1993). A list and description of all samples prepared for organic and inorganic analysis is presented in Table 1.

Preparation of samples for analysis of organic contaminants

Large zebra mussels, large quagga mussels and caged mussels were shucked, and only the soft tissues were analyzed for contaminant residues. All other organisms were analyzed whole. Zebra and quagga mussels > 1.5 cm in length were considered to be "large", and those < 1.5 cm in length were considered to be "small". Spottail shiner samples NR-SP-4, NR-SP-5 and NR-SP-6 each consisted of 10 specimens averaging 5.0, 5.3 and 5.7 cm in total length, respectively. The sample of large crayfish consisted of 8 females and 5 males measuring 16.6 to 33.7 cm in length; the small crayfish measured 6.5 to 10.1 cm in length, and were probably young of the year (Dr. Premek Hamr, NWRI, pers. comm.). Samples were placed in pre-cleaned glass jars and the wet weights were recorded. The samples were then freeze-dried using a LABCONCO Lyph-Lock 6® freeze-dryer fitted with a Model 77560 Lyph-Lock Stoppering Tray Dryer®, and homogenized in a stainless steel blender. Sediment samples were also freeze-dried and were homogenized using a mortar and pestle. Bourbonniere *et al.* (1986) found that concentrations of HCB, PCBs, DDT derivatives and Mirex did

not differ significantly among subsamples of Lake Ontario sediment analyzed wet vs. freeze-dried. Furthermore, results for freeze-dried samples were less variable, suggesting that freeze-dried sediment can be more thoroughly homogenized than wet sediment. Similarly, Metcalfe-Smith *et al.* (unpublished data) found that recoveries of organochlorine pesticides and PCBs from wet and freeze-dried mussel tissues were similar, and that variability among replicates was slightly lower for freeze-dried samples. Where less than 5 g of dry biological material were available, the entire sample was extracted and analyzed. Where more than 5 g were available (small zebra mussels and large crayfish), a subsample of approximately 5 g was taken. For sediments, a subsample of approximately 10 g was extracted.

Preparation of samples for analysis of metals

Biota and sediment samples were prepared as described above, then submitted to Environment Canada's National Laboratory for Environmental Testing (NLET), Burlington, Ontario, for inorganic analysis. Soft tissues and shells of the large zebra mussels were analyzed separately for metal residues. Spottail shiner samples NR-SP-7, NR-SP-8 and NR-SP-9 each consisted of 10 specimens averaging 5.7, 5.1 and 4.9 cm in total length, respectively.

Analytical Methods

Organic contaminants

Extraction and cleanup - biota samples: Each sample to be extracted was weighed, mixed thoroughly with an equal weight of sodium sulphate, and placed into a glass fibre thimble. The sodium sulphate was Soxhlet-extracted for 24 hrs with dichloromethane (DCM) and baked overnight at 110°C to remove moisture and contaminants prior to use. The thimble was inserted into the Soxhlet extractor, and the sample was extracted for 6 hrs at 12 cycles per hour with 300 mL DCM. After cooling to room temperature, the sample was passed through a 3 to 5 cm bed of sodium sulphate via vacuum filtration and collected in a 500 mL round bottom flask. The Soxhlet flask was

rinsed with 2 x 25 mL volumes of hexane, which were passed through the sodium sulphate and combined with the sample. The sample was then evaporated to 5 mL using a rotary evaporator, and further evaporated to 2 mL using a nitrogen evaporator.

Lipids were removed by gel permeation chromatography (GPC) with 2.1 cm i.d. x 50 cm columns of SX-3 Bio-Beads® (200-400 mesh), using 1:1 (v:v) DCM:hexane as the eluent. Lipids eluted in the first 130 mL fraction, and were evaporated to dryness and weighed to determine the lipid content of the sample. The second 130 mL fraction contained the contaminants of interest. This fraction was evaporated to 5 mL using a rotary evaporator, solvent exchanged into hexane, and concentrated to approximately 1 mL using a nitrogen evaporator.

The hexane extracts were cleaned up by elution from a silica gel column into two fractions. The silica gel was activated at 350°C overnight and cooled prior to use. Columns (1.0 cm i.d. x 20 cm) were packed with 0.5 cm anhydrous sodium sulphate above 20 cm silica gel, and rinsed with hexane. The extracts were applied to the column, and they eluted in two fractions: 50 mL hexane (Fraction A) and 50 mL 1:1 (v:v) DCM:hexane (Fraction B). One mL of toluene was added to each fraction, and both fractions were evaporated to approximately 5 mL using a rotary evaporator, then concentrated to 1 mL using a nitrogen evaporator. The two fractions, now in toluene, were transferred to GC autosampler vials, capped with pre-fired aluminum foil and crimp-capped. One method blank was run with these samples.

Extraction and cleanup - sediment samples: Each sample to be extracted was weighed into a flask, 50 mL DCM were added, and the mixture was homogenized for 3 minutes in a Polytron® homogenizer. The solids were allowed to settle, then the supernatant was transferred by pipet to an Allihn funnel containing 2 to 3 cm of pre-rinsed sodium sulphate. The sample was passed through the sodium sulphate via vacuum filtration and collected in a 500 mL round bottom flask. Homogenization and filtration was repeated twice, then the solids were washed with 50 mL hexane and passed through the sodium sulphate as well. The extract was evaporated to 5 mL using a rotary evaporator, transferred to a centrifuge tube, and allowed to air-evaporate to approximately 1 mL.

The hexane extracts were cleaned up as previously described for biota samples, except that the columns were packed with silica gel only. Also, after evaporating Fractions A and B to 1 mL in toluene, 0.5 mL of triple distilled mercury was added to remove organic sulphur. The samples were agitated on a vortex stirrer for about 5 minutes, then allowed to settle overnight. The extracts were then transferred to GC autosampler vials. One method blank was run with these samples.

Analysis: Samples of biota and sediment were analyzed for 19 organochlorine pesticides (OCs), pentachlorobenzene (PECB), hexachlorobenzene (HCB), octachlorostyrene (OCS) and 94 individual PCB congeners or co-eluting congeners by NWRI. Samples were also analyzed for 16 PAHs, 5 chlorobenzenes (CBs), hexachlorobutadiene (HCBd) and hexachloroethane (HCE) by the Wastewater Technology Centre (WTC), Burlington. These compounds were chosen because they are of concern in the Niagara River (Analytical Protocol Group 1992). A list of all compounds sought is presented in Table 2.

Of the 22 pesticides and industrial organic compounds analyzed by NWRI, 5 (PECB, HCB, aldrin, OCS and mirex) eluted in Fraction A only, 2 (heptachlor and p,p'-DDE) eluted in both fractions (30-40% in Fraction A and 70-80% in Fraction B), and the remaining 15 compounds eluted in Fraction B only. Most of the 94 PCB congeners eluted in Fraction A; however, 32 could elute in either fraction. Both fractions were analyzed for the compounds that could occur in either fraction. As PAHs eluted in Fraction B only, a portion of Fraction B was submitted to WTC for analysis of PAHs. As hexachlorobutadiene (HCBd), hexachloroethane (HCE) and the tri- and tetrachlorobenzenes (CBs) eluted in Fraction A only, a portion of Fraction A was also submitted to WTC for analysis of these compounds. Analytical procedures used by WTC were EPA method 3540 for sample extraction, EPA method 8270 for analysis of PAHs, and EPA method 8080 for analysis of CBs, HCBd and HCE (U.S. EPA, 1990). Analytical procedures used by NWRI for the remaining compounds are described below.

Sample extracts were analyzed for contaminant residues using a Hewlett-Packard (HP) 5890 capillary column gas chromatograph equipped with dual electron-capture detectors. Two 30 m high

performance fused silica capillary columns, one coated with SPB-1 (100% dimethylpolysiloxane) and the other with SPB-5 (5% diphenyl-/95% dimethylsiloxane) were used. Sample injections of 2 μL were made by means of a HP 7673A autosampler, with a split/splitless valve and the following chromatographic conditions: initial column temperature of 80°C for 2 minutes, then temperature-programmed to 280°C at 3°/minute. The carrier gas was hydrogen with a head pressure of 60 kPa. Individual PCB congeners were identified and quantified as described by Swackhamer (1988). Congeners were quantified using a standard prepared by M.E. Comba, NWRI, from purchased Aroclor solutions (Supelco® 4-8701, 4-8705, 4-8707, 4-4810). Aroclors 1016, 1221, 1242, 1254 and 1262 were reconstituted in hexane in ratios of 1:1:1:1:1 to a final concentration of 2 $\mu\text{g mL}^{-1}$ and calibrated using the Green Bay PCB standard prepared by Mullin (1985). Congener assignments were confirmed using congeners obtained from the National Research Council of Canada. Organochlorines were quantified using standards prepared from purchased solutions.

Limits of Detection (LODs) and Limits of Quantitation (LOQs) for OCs and PCBs were determined using a representative biota sample (NRSZO-2) and a representative sediment sample (93SED-35). Calculations were based on a 2 μL injection of a 1 mL extract, using standards of 100 $\text{pg}/\mu\text{L}$ for the OCs and 2972 $\text{pg}/\mu\text{L}$ ΣPCBs . For each sample, the baseline of the chromatogram was attenuated such that background noise could be measured. Approximately 60 noise peaks were measured, and the mean and SD calculated. LODs were defined as 3SD above noise and LOQs as 10SD above noise (Analytical Protocol Group 1992). LODs and LOQs were derived for each signal and fraction, and were applied as follows: sample responses falling below the LOD for a particular compound were rejected, and those falling between the LOD and LOQ were flagged. Sample responses were then compared with the blank responses. Sample responses that were $\leq 2\times$ the blank response were rejected, and those that were $> 2\times$ the blank response were blank-corrected. All blank-corrected values falling below the LOD for that compound were rejected, and those values falling between the LOD and LOQ were reported and labelled "bql" (below quantitation limit). Nominal LODs and LOQs for PCBs in biota, PCBs in sediment and OCs in sediment based on signal 1 (the most sensitive detector) of Fraction A are presented in Appendix A. It should be noted that all biota and sediment samples contained significantly higher concentrations of target compounds than

the blanks. Prior to blank-correcting, concentrations of total PCBs in the *least contaminated* biota sample (NRSZO-1) and sediment sample (93SED-33) were 3× and 6× the concentrations in the blanks, respectively. The concentration of total OCs in the sediment sample least contaminated with these compounds (93SED-35) was 8× the concentration in the blank.

Contaminants were quantified by relating their detector responses to the standard response factors. Where blank-corrected responses on the two columns differed by less than 40% (calculated as $[|S_1 - S_2| / \{(S_1 + S_2) / 2\}] \times 100\%$, where S_1 = signal 1 response and S_2 = signal 2 response), the values were averaged; where responses differed by more than 40% but less than 80%, the lower value was reported; where responses differed by more than 80%, no value was reported. These criteria were derived from the variation in dual column responses observed for OCs and PCBs in method standards, that is, standards that had been subjected to the same extraction, cleanup and fractionation procedures as samples. In this laboratory, we found that the mean variation between responses for 21 to 23 OCs subjected to both Soxhlet and Polytron® procedures was 51%, with a SD of 18% ($n = 66$). For PCBs, the mean variation between responses for 97 congeners subjected to both procedures was 43%, with a SD of 27% ($n = 194$). There were no differences in variation between procedures. Based on mean variations of 51% and 43% for OCs and PCBs, respectively, we chose 40% as the upper limit for averaging dual column responses in this study. Given that an uncertainty of ± 2 SD is generally accepted as the control limit for method performance (Analytical Protocol Group 1992; MOEE 1994d), differences in dual column responses of up to 85% for OCs and 97% for PCBs would be considered acceptable. In this study, we used 80% as the upper limit for confirming the presence of a compound. For PCB congeners that co-eluted with other congeners, quantification was more complicated. The calculations are explained in Appendix B.

Spike-recovery tests were conducted to assess method performance. Three spikes for organochlorines (OCs) and three for PCBs were run through the Soxhlet extraction method, and one spike for OCs and one for PCBs were run through the Polytron® extraction method. Results for each method are presented in Appendices C and D, respectively. Recoveries of 80 to 120% are considered acceptable. Recovery of Σ PCB by Soxhlet extraction was 99% (mean of means for all

congeners; CV = 23%). Only 6 congeners, namely 54, 46, 70/76, 119, 198 and 206, had recoveries outside the acceptable range (75-130%). Recovery of Σ PCB by Polytron® extraction was 86% (CV = 14%). Recoveries for individual congeners were generally lower than by Soxhlet extraction, with 20 congeners having recoveries outside the acceptable range. Of these, all had recoveries greater than 70% except congeners 54 (51%), 60/56 (59%), 89 (52%) and 82 (44%). Recoveries of OCs by Polytron® extraction were within the acceptable range except for p,p'-DDT (130%) and β -endosulfan (3%). Low recovery of β -endosulfan is a routine occurrence in this and other NWRI laboratories. It appears that the compound does not elute from the silica gel column during extraction. As a result, we were unable to determine concentrations of β -endosulfan in these samples. Recoveries of OCs by Soxhlet extraction are not reported, for reasons to be discussed later. Sample concentrations were not corrected for recovery efficiencies.

Metals

Biota samples were analyzed for total residues of 10 metals by NLET, using standard procedures described in their Manual of Analytical Methods (NLET 1994a). Briefly, the analytical methods and associated detection limits (DLs) on a $\mu\text{g/g}$ dry weight basis for the tested elements were: Hg - cold vapour atomic absorption (AA) spectroscopy, DL = 0.03; Al, Cr, Cu, Fe, Mn, Ni and Zn - direct aspiration AA spectroscopy, DLs = 0.50 (Ni), 2.0 (Cr, Cu, Zn), 10.0 (Fe, Mn) and 50.0 (Al); Cd and Pb - graphite furnace AA spectroscopy, DLs = 0.01 (Cd) and 0.20 (Pb). Samples were analyzed in accordance with NLET's routine quality assurance (QA) procedures, which include duplicate analyses to determine sample homogeneity, analysis of three reference materials to determine accuracy, spike-recovery tests to assess interference, and analysis of blanks to determine contamination due to laboratory procedures. Samples that do not meet the QA objectives are reanalyzed, and those that still do not meet the standards are rejected. No samples from this study were rejected.

Sediment samples were analyzed for total and extractable Al, Cr, Cu, Pb and Zn, total As, Hg and Se, and extractable Cd, Fe, Mn and Ni by NLET, using standard procedures described in their

Manual of Analytical Methods (NLET 1994b). The analytical methods and associated detection limits (DLs) on a $\mu\text{g/g}$ dry weight basis for the tested elements were: total Al, Cr, Cu, Pb and Zn - AA spectroscopy, DLs = 10.0 (Al), 5.0 (Pb), 1.0 (Cr, Cu, Zn); total Hg - cold vapour AA spectroscopy, DL = 0.01; total As and Se - atomic emission spectroscopy using an inductively coupled argon plasma (ICAP) system, DL = 0.2 for both elements; extractable Al, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn - AA spectroscopy (after extraction in a 5% hydrochloric acid solution), DLs - 2.0 (Al), 1.0 (Fe, Pb), 0.6 (Ni), 0.2 (Cd, Cr, Cu, Mn, Zn). Samples were analyzed in accordance with routine QA procedures similar to those described above for biota. All samples met the QA objectives.

RESULTS AND DISCUSSION

Metal Residues in Biota and Sediment Samples

Biota

Concentrations of 10 metals in the nine biota samples are shown in Table 3. Soft tissues of large zebra mussels contained the highest concentrations of all metals with the following exceptions: small zebra and quagga mussels contained higher levels of Cr (23.5 to 31.7 vs. 8.9 $\mu\text{g/g}$), even with their shells on, and spottail shiners contained similar levels of Zn (151 to 168 vs. 149 $\mu\text{g/g}$). With the exception of Zn, concentrations of metals in the soft tissues of zebra mussels were 2 to 52 \times higher than average concentrations in shiners.

In order to compare large and small zebra mussels directly, whole body residues in large zebra mussels were back-calculated from separately-determined values for soft tissues and shells using the dry weights given in Table 1d. The comparison is shown in Table 4. It is worth noting here that the shells of large zebra mussels contained a substantial proportion of the whole body residue of most metals: Cd - 17%; Cr - 25%; Zn - 35%; Cu - 45%; Ni - 49%; Fe - 53%; Al - 56%; Pb - 68%; Hg - 71%; Mn - 89%. Concentrations of Al, Cd, Mn, Zn and Hg were similar in large and small zebra mussels, but concentrations of Fe, Cu, Ni and Cr were 1.5 \times , 2 \times , 4 \times and 40 \times higher, respectively, in

small zebra mussels. Only Pb was higher (2×) in large mussels. Small quagga mussels had levels of metals similar to those in small zebra mussels. Spottail shiners and small zebra mussels can be compared in terms of both average metal concentrations and variability among replicate composite samples (Table 4). Concentrations of Zn were an order of magnitude higher in shiners and Hg was always detectable, suggesting that spottail shiners would be more suitable biomonitors for these two elements *unless* the mussels were shucked prior to analysis. Chromium and Pb were frequently non-detectable in shiners. Concentrations of the remaining six metals were higher in zebra mussels, and for Al, Cd and Cu, variability was considerably lower. It therefore appears that zebra mussels have some advantages over spottail shiners as biomonitors for metals at this site.

Caged mussels from 1992 could not be analyzed for metals by NLET, because no additional archived specimens were available. However, samples had previously been analyzed by MOEE's Rexdale Laboratory for 7 of the 10 metals using analytical methods described in MOEE (1993) and MOEE (1994g). The raw data are presented in Appendix E, and results are compared with those for large zebra mussels in Table 5. Data on pre-exposure Balsam Lake mussels were also available for 1992 (analyzed by MOEE) and 1994 (analyzed by NLET in connection with another study). As the Rexdale Lab analyzes biological samples wet, concentrations were converted to $\mu\text{g/g}$ dry weight using a soft tissue moisture content of 90%. Metcalfe-Smith *et al.* (1992) reported an average moisture content of 91% for 47 *E. complanata* collected from the Ottawa River in the summers of 1985 and 1986, and J.L. Metcalfe-Smith (unpublished data) observed an average moisture content of 89% in composite samples of *E. complanata* collected from 12 sites on the St. Lawrence River in June of 1989. These values are for fresh mussel tissues. Moisture contents of the 1992 caged mussels were lower (Table 1c), because the samples had become desiccated while stored in the freezer for 18 months prior to analysis.

Comparable results were obtained for concentrations of metals in Balsam Lake mussels from 1992 (analyzed by MOEE) and 1994 (analyzed by NLET), although the Rexdale Lab was unable to quantify Ni or Pb in these samples and reported only trace levels of Hg. Concentrations of Ni, Pb and Hg reported by NLET were about 55%, 60% and 35% lower, respectively, than MOEE's routine

quantification limits for these elements in this type of sample. Large zebra mussels contained higher levels of Cu (3×) and Ni (>10×) than caged mussels, whereas caged mussels contained higher concentrations of Mn (40×), Zn (4×) and Cd (1.5 - 4×). Concentrations of Hg and Pb appeared to be similar in both zebra mussels and caged mussels. It is possible that Ni, but not Pb or Hg, would have been quantifiable in large zebra mussels using MOEE's routine methods. In a 1990 survey of contaminant levels in zebra mussels vs. native *E. complanata* from the St. Lawrence River, Metcalfe-Smith (unpublished data) also found that zebra mussels contained higher concentrations of Ni than native mussels (2×), and that native mussels contained much higher levels of Mn (50×) and Zn (2.5×) and slightly higher levels of Cd (1.3×). In contrast, however, concentrations of Cu did not differ between zebra mussels and native mussels, and Hg and Pb were higher in native mussels (3× and 2×, respectively). Caged mussels and small zebra mussels can be compared in terms of the variability in concentrations of Cd, Cu, Mn and Zn among replicate samples. Coefficients of variation (CV) for the latter are shown in Table 4. In all cases, variability was lower for zebra mussels. Overall, our findings suggest that zebra mussels may have a slight advantage over caged mussels as biomonitors for metals.

According to Table 5, concentrations of Cu and Zn in caged mussels increased 2-fold and 2- to 3-fold, respectively, over pre-exposure levels, after 10 weeks' incubation in the Niagara River. Cadmium and Mn also increased 3-fold in July, but neither metal was elevated over pre-exposure levels in October. With the exception of Mn, all increases were statistically significant ($p < 0.05$).

Sediment

Three sediment samples were analyzed by NLET for total and extractable Al, Cr, Cu, Pb and Zn, total As, Hg and Se, and extractable Cd, Fe, Mn and Ni. These samples were also analyzed by the Rexdale Lab for total concentrations of the same 12 metals (E.T. Howell, unpublished data), using analytical methods described in MOEE (1994a and 1994f). Thus, a comparison between labs was possible for total Al, As, Cr, Cu, Hg, Pb, Se and Zn in sediment. All data are presented in Table 6. Results from both labs were generally comparable, except that NLET reported concentrations of

Cr and Al that were 2× and 10× higher, respectively, than those reported by the Rexdale Lab. The reason for this discrepancy is unknown. Neither lab could detect Se in these samples. Although values for Pb were similar between labs, two of MOEE's three values were qualified as trace levels only. When compared with the Provincial Sediment Quality Guidelines for the protection of aquatic biological resources (Persaud *et al.*, 1992), concentrations of all metals were below the Lowest Effect Levels except for Cr (as determined by NLET) and Hg, which was borderline.

No attempt was made to compare concentrations of metals in biota with those in sediment because of the limited amount of data available.

Organic Contaminant Residues in Biota and Sediment Samples

Biota

A total of 24 biota and 3 sediment samples were analyzed by NWRI for 22 organochlorine pesticides and industrial organic compounds, and 94 PCB congeners. Normally, biota samples of less than 0.50 g are not analyzed, because the results would be unreliable. Although samples of chironomids (0.13 g) and oligochaetes (0.45 g) were analyzed, the results will not be discussed for this reason. Unfortunately, all biota samples were inadvertently contaminated with OC standards during preparation. As a result, no data are available for these compounds. PCBs, however, were unaffected. Congener-specific PCB data for all biota samples are presented in Appendix F, and Σ PCB residues are shown in Table 7. The various biota ranked as follows in order of decreasing concentrations of Σ PCBs (ng/g dry weight basis for all; where replicates were analyzed, mean values are presented): soft tissues of large quagga mussels (423) > spottail shiners (395) > amphipods (236) > soft tissues of caged mussels (230) > crayfish (118) > soft tissues of large zebra mussels (92) > snails (83) > small zebra mussels (34). As expected, snails and small zebra mussels, which were analyzed with their shells on, contained the lowest concentrations of Σ PCBs. Shells of large zebra mussels were not analyzed for organic contaminants, therefore PCB residues in large and small zebra mussels could not be directly compared. Russell Kreis (U.S. EPA, Large Lakes Research Station,

Grosse Ile, MI, pers. comm.) found that the shells of zebra mussels contain a negligible portion (approximately 1/400) of the total body burden of organic contaminants, and in the present study we determined that shells constituted 94.6% of the dry whole weight. Thus, concentrations of Σ PCBs in the soft tissues of small zebra mussels would have been about 630 ng/g. This value is similar to the 423 ng/g observed in the soft tissues of large quagga mussels, but higher than the 92 ng/g found in the soft tissues of large zebra mussels.

There was no apparent relationship between the lipid content of an organism and its body burden of Σ PCBs. For example, large zebra mussels had lipid contents similar to shiners, but much lower Σ PCB concentrations. Also, caged mussels had lower lipid contents than shiners, but similar concentrations of Σ PCBs. Data for small zebra mussels and spottail shiners were more variable than those for caged mussels: coefficients of variation (CV) in Σ PCB residues were 39% for small zebra mussels, 35% for shiners, 24% for caged mussels from July/92 and 13% for caged mussels from October/92. These results suggest that caged mussels, by virtue of their relatively high body burdens and relatively low individual variability, may be the most suitable biomonitors for PCBs at this site.

Caged mussels exposed at Niagara River station 129 for 71-73 d in May to July 1992 ($n = 3$) had also been analyzed by MOEE's Rexdale Laboratory for Σ PCB residues (Appendix E) using analytical methods described in MOEE (1994b). All samples were analyzed on a wet weight basis, using a detection limit of 20 ng/g. It should be noted that MOEE's packed column/multi-peak Aroclor quantitation procedure is much less sensitive than NWRI's dual capillary column/congener-specific method for the determination of PCBs. Of the 3 individual mussels analyzed, none contained detectable levels of Σ PCBs. In contrast, NWRI was able to detect PCBs in the six specimens they analyzed from the May to July and July to October exposures, at concentrations ranging from 169 to 304 ng/g dry weight. Assuming a moisture content of approximately 90% for these samples (discussed above), concentrations would have been about 16.9 to 30.4 ng/g on a wet weight basis. These concentrations are near or below MOEE's detection limit for Σ PCBs, which explains why the Rexdale Lab was unable to provide quantitative data for these samples. An additional factor may be

that MOEE analyzed 20% as much material as NWRI, i.e., one-fifth of a whole-mussel extract vs. the entire mussel.

Portions of 20 of the biota samples were submitted to the Wastewater Technology Centre (WTC) for analysis of PAHs, CBs, HCBd and HCE. Results, including raw data, detection limits and quality control reports, are presented in Appendices G (PAHs) and H (other compounds). The chironomids and two replicates of small zebra mussels were not analyzed. Although the oligochaetes were analyzed, the results will not be discussed as the sample was so small. Measurable concentrations of several PAHs were found in only a few of the biota samples. Specifically, phenanthrene (140 ng/g) and benzo(b)fluoranthene (100 ng/g) were found in the soft tissues of large quagga mussels, fluorene (120 ng/g) and phenanthrene (170 ng/g) were found in amphipods, and phenanthrene (310 ng/g) was found in one caged mussel from July 1992. Trace levels of most other PAHs were found in the soft tissues of large zebra and quagga mussels, and in amphipods. There were fewer detections, at the trace level only, in snails (6 to 10 compounds), crayfish (7), caged mussels (6 to 8), small zebra mussels (6 to 7) and spottail shiners (2 to 4). These results suggest that it may have been possible to obtain quantitative data on PAHs for at least some of the biota samples if the extracts had been concentrated further. As 500 μ L are required for this analysis, and the extracts were already at 1 mL, it would only have been possible to concentrate the samples by a factor of 2.

No caged mussels from the 1992 exposures were analyzed for PAHs by the Rexdale Lab. However, data are available for mussels incubated in previous years (E.T. Howell, unpublished data) and analyzed according to the methods described in MOEE (1994c). Caged mussels exposed in November 1988, contained trace levels of all eight of the PAHs identified by WTC as occurring in 1992 mussels, plus three others (acenaphthene, fluorene and benzo(a)pyrene). Caged mussels exposed in 1989 contained trace or measurable amounts of 10 PAHs, including seven of the eight found at trace levels in 1992 mussels (naphthalene, phenanthrene, fluoranthene, pyrene, benzo(a)anthracene, chrysene and benzo(b)fluoranthene) and three others not found (acenaphthene, acenaphthylene and fluorene). Caged mussels exposed in May to July and September to October of

1990 did not contain measurable amounts of PAHs. However, the two individuals exposed from July to September each contained 70 ng/g chrysene, and one also contained 110 ng/g fluoranthene and 90 ng/g pyrene.

Chlorobenzenes, HCBd and HCE were not detected in Niagara River biota, even in trace amounts, with the following exceptions: traces of 1,2,3,4-TTCB were found in amphipods, large crayfish and one composite sample of spottail shiners. Of the 6 caged mussels incubated in 1992 and previously analyzed by MOEE's Rexdale Laboratory, none were found to contain chlorobenzenes, HCBd or HCE (Appendix E). Because of the low frequency of detection for PAHs, chlorobenzenes, HCBd and HCE in biota, no recommendation could be made as to the most appropriate biomonitor for these compounds.

Sediment

Three sediment samples were analyzed by NWRI for 22 organochlorine pesticides and industrial organic compounds and 94 PCB congeners, and by WTC for 16 PAHs and seven additional industrial organic compounds. The same samples were analyzed for most of the same compounds by MOEE's Rexdale Laboratory (E.T. Howell, unpublished data), using analytical methods described in MOEE (1994d and 1994e). MOEE extracts 5 g wet sediment, whereas NWRI extracts 10 g dry sediment. Based on the moisture content of the sediment (Table 1b), this means that MOEE extracted about 40% as much material as NWRI for these analyses. Again, MOEE used the less sensitive packed column/multi-peak Aroclor quantitation procedure for the determination of PCBs in sediment. Summaries of both sets of data are presented in Table 8 for comparison. Data on congener-specific PCBs and the 22 other compounds analyzed by NWRI are presented for each sample in Appendices I1 and I2, respectively, and data on PAHs and the additional 7 compounds are presented in Appendices G and H, respectively.

The Rexdale Lab reported traces of 12 of the 16 PAHs in sediment from station 129, whereas WTC reported quantitative data for 10 PAHs in all three sediment samples and traces of 2 additional

compounds. Acenaphthylene, acenaphthene, anthracene and dibenzo(a,h)anthracene were not detected in any sample by either lab. Only fluorene, phenanthrene and benzo(b)fluoranthene had been found above trace levels in a few biota samples. WTC reported traces of the two tetrachlorobenzenes in all sediment samples, at levels below the detection limits of MOEE's routine method. Traces of 1,2,3,4-TTCB had also been detected in a few biota samples. NWRI detected PCBs in all three sediment samples, and reported an average concentration of 42 g/g Σ PCBs. All biota samples also contained PCBs. PCBs were not detected in sediment (< 20 ng/g) by MOEE, using their less sensitive method. NWRI reported quantitative data for PCB and HCB in all samples, and for α -BHC, γ -BHC, p,p'-DDE, endrin, p,p'-DDD, p,p'-DDT and mirex in one or more samples. In all cases, concentrations were below the detection limits of MOEE's routine method. Due to the inadvertent contamination of the biota samples with OC standards, no data on residues of these compounds in biota are available for comparison with sediment. Results of the sediment analyses showed that concentrations of PAHs, CBs, HCB, HCE, PCBs and OCs in sediment from Niagara River station 129 were too low to be detected by MOEE's routine analytical methods. Concentrations of HCB, HCE, the lower chlorinated CBs and many of the OCs were also too low to be detected by NWRI and WTC's methods.

Concentrations of Σ PCBs, Σ PAHs and Σ CBs (except PCB and HCB, which were not determined in biota) in sediment vs. biota are compared in Table 9. Trace values were used in calculating total concentrations of PAHs and CBs. All biota, with the exception of small zebra mussels, contained higher concentrations of Σ PCBs than sediment. In contrast, most biota contained lower concentrations of Σ PAHs than sediment; exceptions were the soft tissues of large quagga mussels and caged mussels. Chlorobenzenes were detected in only three types of organisms (amphipods, large crayfish and spottail shiners), at concentrations similar to those in sediment.

PCB Congener-Class Distributions in Biota and Sediment

PCB congener-class distributions, i.e., the percentage of Σ PCBs attributed to each congener class, were determined for each sample. Results for all sediment and biota samples are presented in

Appendix J. In all cases where replicate samples were analyzed (small zebra mussels, spottail shiners, caged mussels in July 1992, caged mussels in October 1992 and sediment), profiles in replicates were very similar; thus, average proportions were computed for each type of sample for illustrative purposes. Profiles for the soft tissues of large zebra and large quagga mussels were also very similar, so they were also combined. Profiles differed somewhat between large and small crayfish and between the two species of snails, therefore the data for these samples were not combined. Congener class profiles are presented in Figs. 2 to 4. Data for chironomids and oligochaetes are included for the purpose of this comparison.

Sediment (Fig. 2a) contained predominantly lower chlorinated PCBs, i.e., 64% mono- to tetrachlorobiphenyls vs. 36% penta- to octachlorobiphenyls. In contrast, all biota contained higher proportions of the more highly chlorinated PCBs, i.e., 14-46% mono- to tetrachlorobiphenyls vs. 54-85% penta- to octachlorobiphenyls. The PCB profile in chironomids (Fig. 2c) was most similar to the profile in sediment, especially with respect to the low proportions of hexa- to octachlorobiphenyls (22% in chironomids and 18% in sediment). Oligochaetes (Fig. 2b), amphipods (Fig. 2d) and both species of snail (Fig. 4a,b) were next in similarity to sediment, as they also contained relatively low proportions of hexa- to octachlorobiphenyls (approximately 30%); however, proportions of pentachlorobiphenyls were much higher in these organisms than in sediment (25 to 36% vs. 18%). These four types of organisms would probably have the most direct contact with sediment. Proportions of hexa- to octachlorobiphenyls were higher again (40-50%) in the crayfish (Fig. 3a,b) and caged mussels (Fig. 3c,d), and crayfish contained very low proportions of mono- to trichlorobiphenyls relative to sediment (3-5% vs. 19%). Profiles in spottail shiners (Fig. 4c) were most dissimilar to those in sediment, as they consisted mainly of hexa- to octachlorobiphenyls (72%) and virtually no mono- to trichlorobiphenyls (1%). This suggests that fish may selectively accumulate the more highly chlorinated congeners, or are able to metabolize the lower congeners. Alternatively, they may reflect patterns in the water or in their food. Profiles in the soft tissues of large zebra and quagga mussels (Fig. 4e) were similar to those in spottail shiners. Small zebra mussels (Fig. 4d) were the only organisms that contained higher proportions of mono- to trichlorobiphenyls (24%) than sediment (19%). These organisms displayed the most even distribution of PCBs among the congener

classes, i.e., 24% mono- to trichlorobiphenyls, 22% tetrachlorobiphenyls, 21% pentachlorobiphenyls and 33% hexa- to octachlorobiphenyls.

SUMMARY AND CONCLUSIONS

A collaborative study between NWRI and MOEE was initiated in 1993 to identify candidate organisms to replace caged mussels as biomonitors for MOEE's Long-Term Sensing Sites Project (LTSSP). The main goal of the study was to identify a naturally-occurring organism or organisms that would accumulate higher and less variable concentrations of the organic and inorganic contaminants of interest than caged mussels. A second goal was to determine if the routine methods currently used by MOEE to analyze biota and sediment samples from the long-term sensing sites were sensitive enough to support the data requirements of the Project. Niagara River station 129 was selected for study because it was considered representative of the type of station targeted by the LTSSP, that is, stations in areas subject to chronic low-level exposure to contaminants.

Between July and September 1993, samples of zebra mussels, quagga mussels, oligochaetes, chironomids, amphipods, snails, crayfish, young-of-the-year spottail shiners and sediment were collected from the study site. Archived samples of caged mussels exposed for ten-week periods in May to July and July to October 1992, were also obtained for comparison. Samples were analyzed by the National Water Research Institute (NWRI) for residues of 19 organochlorine pesticides and 3 industrial organic compounds (sediment only) and for 94 PCB congeners, by the Wastewater Technology Centre (WTC) for 16 priority PAHs and 7 other industrial organic compounds, and by the National Laboratory for Environmental Testing (NLET) for 10 metals in biota (zebra and quagga mussels, caged mussels and spottail shiners only) and 12 metals in sediment. Portions of the sediment samples were also analyzed by MOEE's Rexdale Laboratory for the same 12 metals, 16 PAHs and 10 industrial organic compounds, as well as for Σ PCBs and 15 of the 19 pesticides. MOEE also analyzed caged mussels from both of the 1992 exposure periods for 7 of the 10 metals, Σ PCBs and 7 industrial organic compounds, but not Σ PAHs. We were therefore able to compare the detection and quantitation of certain compounds in these samples using MOEE's routine analytical methods vs.

NLET, NWRI and WTC's techniques. Replicate samples were analyzed for some types biota, thus permitting a comparison of within-site variability in contaminant concentrations among these organisms.

Concentrations of metals in the soft tissues of large zebra mussels were 2 to 52× higher than those in spottail shiners. An exception was Zn, which was similar in both organisms. Small zebra and quagga mussels contained higher concentration of most metals than spottail shiners, even with their shells on. Large zebra mussels accumulated higher levels of Cu and Ni than caged mussels (soft tissues for both), but caged mussels accumulated higher levels of Cd, Mn and Zn. Contrary to findings in previous years (i.e., Pope 1993), mussels caged at Niagara River station 129 for 10 weeks in 1992 accumulated residues of several metals that were significantly above pre-exposure levels. Small zebra mussels exhibited less variability in metal concentrations among replicate samples than either spottail shiners or caged mussels, suggesting that zebra mussels may be the most suitable biomonitors for metals at this index station.

The soft tissues and shells of large zebra mussels were analyzed separately for metal residues. Shells were found to contain significant proportions of the whole body residues of most metals, ranging from 17% for Cd to 89% for Mn. Residues in shells may not reflect recent exposure to metals, and may be altered by factors such as erosion or dissolution. If so, trends based on animals analyzed with their shells on may differ from trends based on soft tissue analyses. As it is not feasible to shuck small (< 1.5 cm shell length) zebra mussels, and large specimens may not be available at all index stations, this problem requires further investigation before zebra mussels could be recommended for monitoring trends in metal bioavailability at the long-term sensing sites.

Biota samples ranked as follows in order of decreasing concentrations of Σ PCBs (ng/g dry weight): soft tissues of large quagga mussels > spottail shiners > amphipods > soft tissues of caged mussels > crayfish > soft tissues of large zebra mussels > snails > small zebra mussels. With the exception of small zebra mussels, all biota contained higher concentrations of Σ PCBs than sediment. Spottail shiners and small zebra mussels were more variable in terms of PCB residues than caged

mussels. These results suggest that caged mussels, by virtue of their moderately high body burdens and relatively low individual variability, may be the most suitable biomonitors for PCBs at this site.

Sediment contained predominantly lower chlorinated PCBs (64% mono- to tetrachlorobiphenyls), whereas all biota contained higher proportions of the more highly chlorinated PCBs (54-85% penta- to octachlorobiphenyls vs. 14-46% mono- to tetrachlorobiphenyls). PCB congener-class profiles in chironomids, followed by those in oligochaetes, amphipods and snails, were most similar to those in sediment, probably reflecting the close contact these organisms would have with sediment in the environment. Crayfish and caged mussels had profiles similar to each other, but contained higher proportions of hexa- to octachlorobiphenyls than the sediment-dwelling organisms (40-50% vs. 20-30%). Congener-class patterns in spottail shiners were the most dissimilar to those in sediment, consisting of 72% hexa- to octachlorobiphenyls vs. only 18% in sediment. This suggests that fish may have different routes of exposure and/or metabolic capabilities than invertebrates. These results indicate that different organisms will provide complementary information on PCB exposure at index stations, and that some knowledge of the life histories, feeding behaviours, detoxification processes, etc., of the various organisms will be necessary for proper interpretation of residue data.

Measurable concentrations of only a few PAHs were found in large quagga mussels (soft tissues), amphipods and one caged mussel, although traces of most other PAHs were detected in these samples and in the soft tissues of large zebra mussels. There were fewer detections (traces only) in other biota, and the fewest in spottail shiners. In general, biota contained lower concentrations of PAHs than sediment. Except for traces of 1,2,3,4-TTCB in amphipods, large crayfish and one sample of spottail shiners, and traces of both 1,2,3,4- and 1,2,4,5-TTCB in sediment, CBs, HCBd and HCE were not detected in samples from Niagara River station 129.

The limits of quantitation of MOEE's routine analytical methods for some target compounds in biota and/or sediment were too high to permit the assessment of contaminant concentrations at Niagara River station 129 and, by extrapolation, to support the data requirements of the Long-Term Sensing Sites Project. Other laboratories, i.e., NLET, NWRI and WTC, were able to provide

quantitative data for some, but not all, of these compounds. For example, NLET reported concentrations of Ni, Pb and Hg in mussels and Cd and Pb in sediment that were below MOEE's limits of quantitation. NLET also reported concentrations of Cr and Al in sediment that were 2× and 10× higher, respectively, than values reported by MOEE. These discrepancies should be investigated. Levels of Se in sediment were below the LOQs of both labs. Concentrations of ΣPCBs in caged mussels and sediment were below MOEE's LOQs, but were measurable in all mussel and sediment samples analyzed by NWRI. NWRI also quantified PCB and HCB in all three sediment samples; however, nearly two-thirds of the organochlorine pesticides were non-detectable and the remainder were found in only one or two of the samples. MOEE did not detect any of these compounds in sediment. WTC provided quantitative results for 10 of 16 PAHs in all three sediment samples, whereas MOEE reported only traces of most of the same compounds. PAHs in biota, and CBs, HCB and HCE in biota and sediment, were infrequently detected by both MOEE and the WTC.

RECOMMENDATIONS

- (1) The results of this study showed that caged mussels accumulated moderately high and relatively consistent concentrations of many contaminants. Thus, it may not be necessary to replace them with other organisms as biomonitors for the Long-Term Sensing Sites Project.
- (2) MOEE's analytical methods for some target elements and compounds should be modified such that concentrations typically occurring in sediment and biota from the long-term sensing sites can be measured and contaminant trends assessed. This may not be possible for all contaminants of interest, due to the very low levels encountered at these ambient sites.

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Figure Captions:

- Figure 1.** Location of the study site, Niagara River station 129.
- Figure 2.** PCB congener-class distributions in sediment (average for three samples), oligochaetes, chironomids, and amphipods from Niagara River station 129.
- Figure 3.** PCB congener-class distributions in small crayfish, large crayfish, caged mussels in July (average for three specimens), and caged mussels in October (average for three specimens) from Niagara River station 129.
- Figure 4.** PCB congener-class distributions in snails (*Physella gyrina* and *Helisoma anceps*), spottail shiners (average for three composite samples), small zebra mussels (average for six composite samples), and large zebra and quagga mussels from Niagara River station 129.

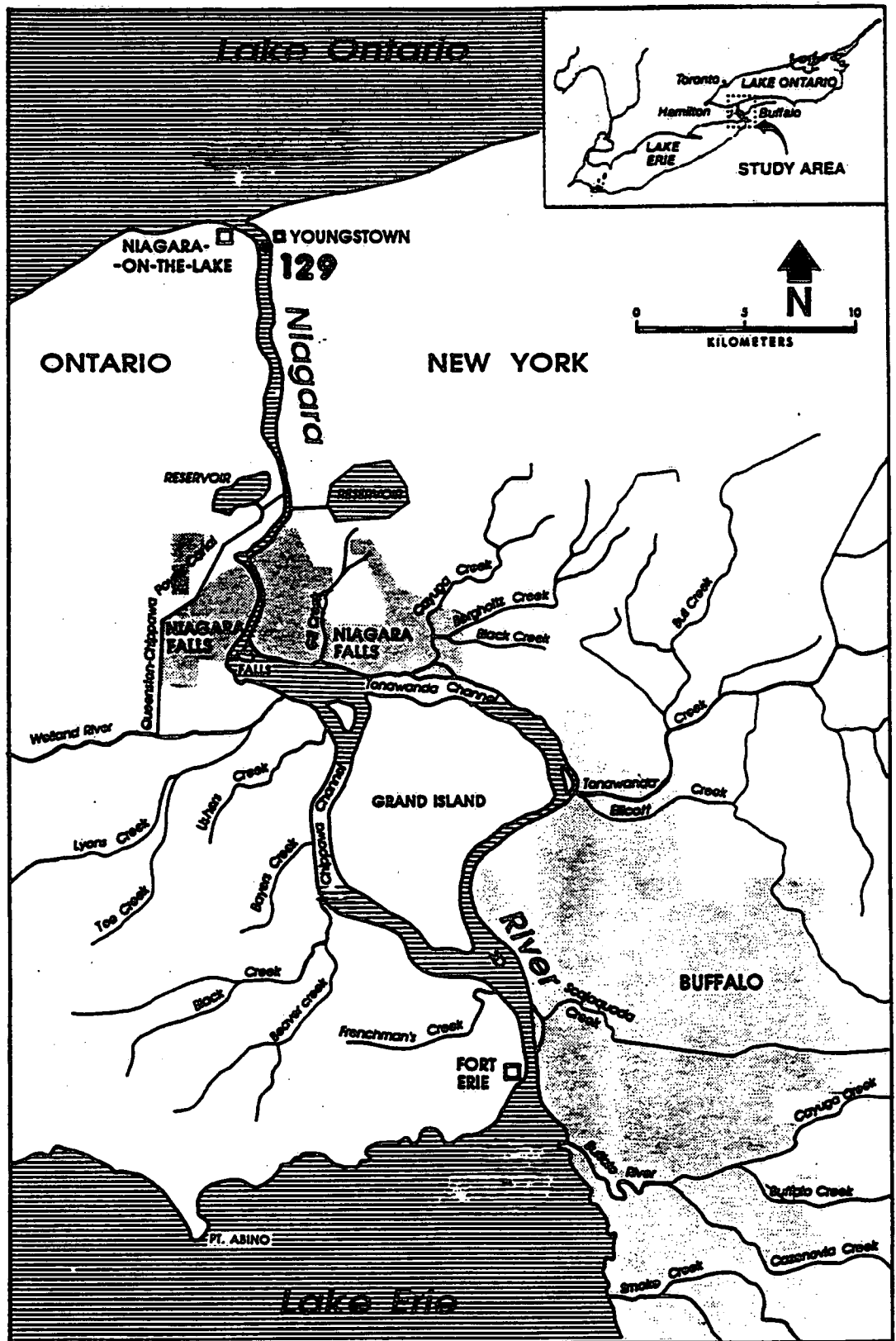


Figure 1

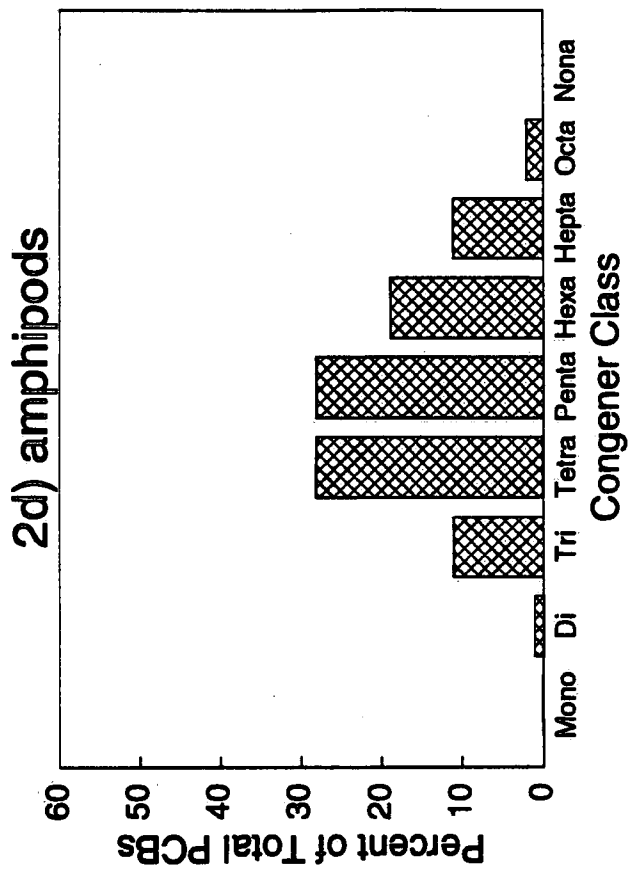
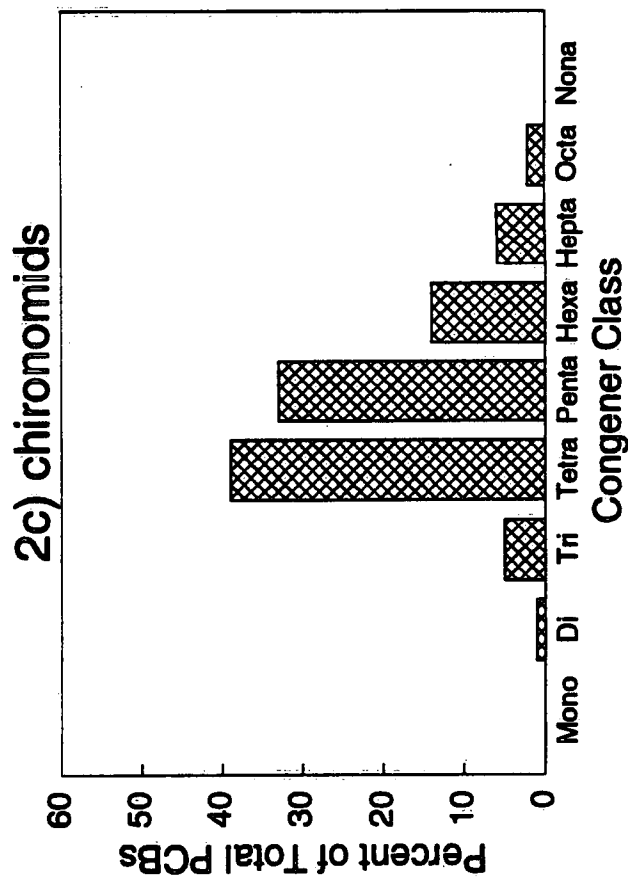
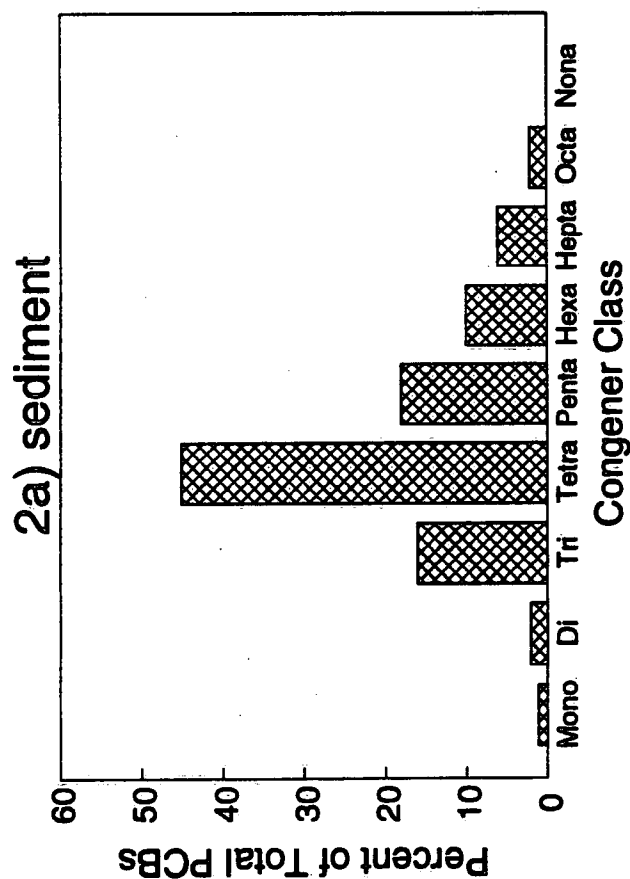
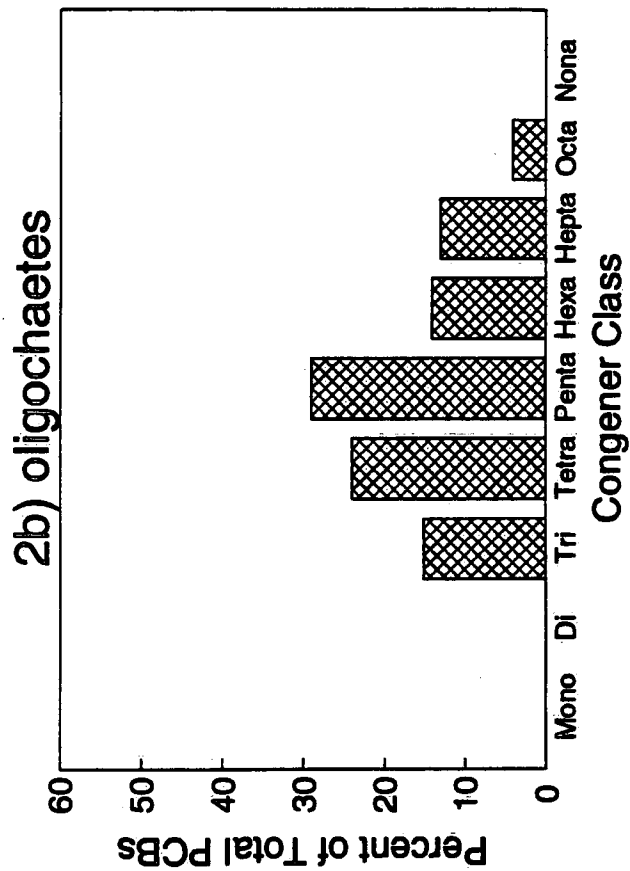


Figure 2. PCB congener-class distributions in sediment (average for three samples), oligochaetes, chironomids, and amphipods from Niagara River station 129.

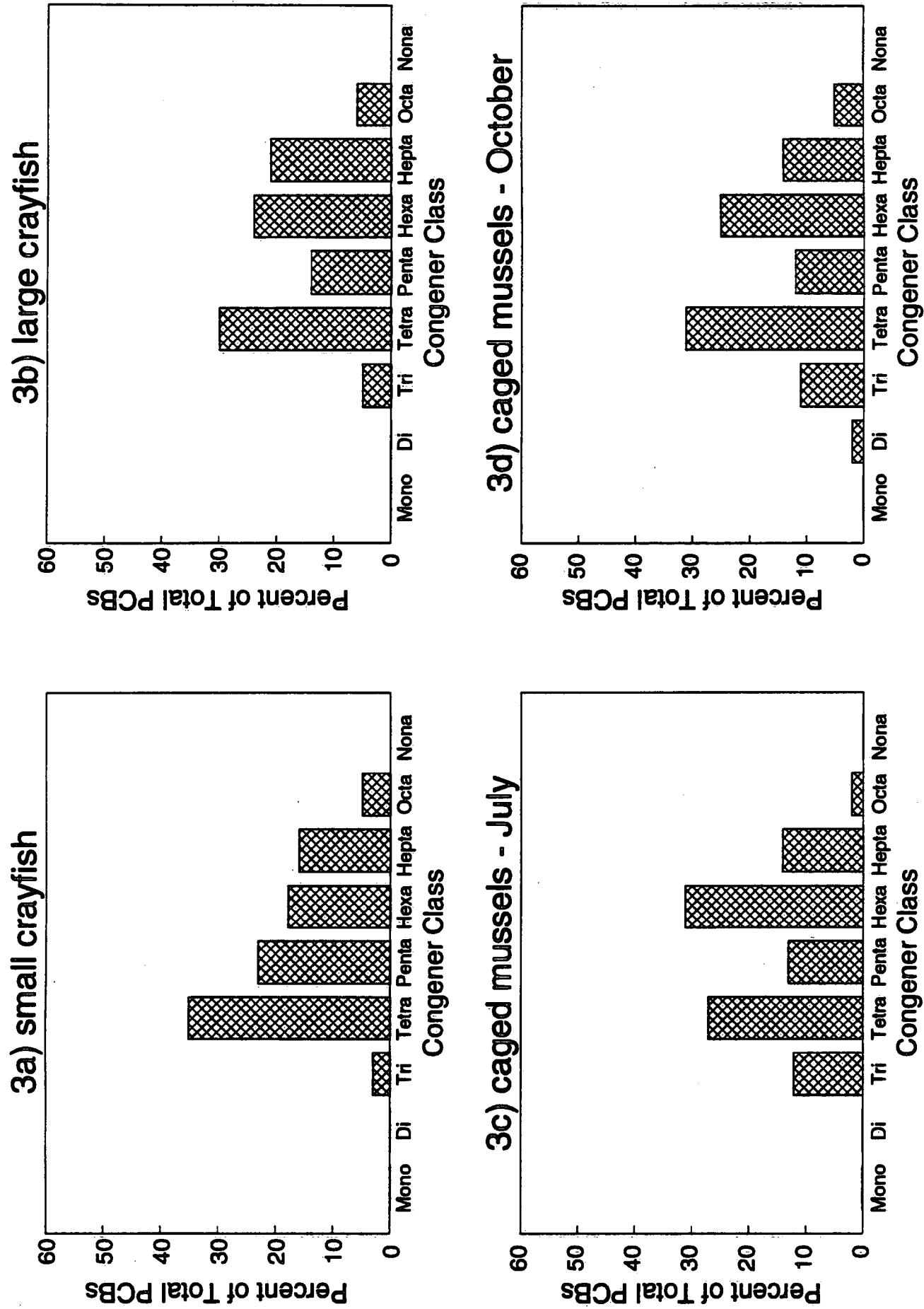


Figure 3. PCB congener-class distributions in small crayfish, large crayfish, caged mussels in July (average for three specimens), and caged mussels in October (average for three specimens) from Niagara River station 129.

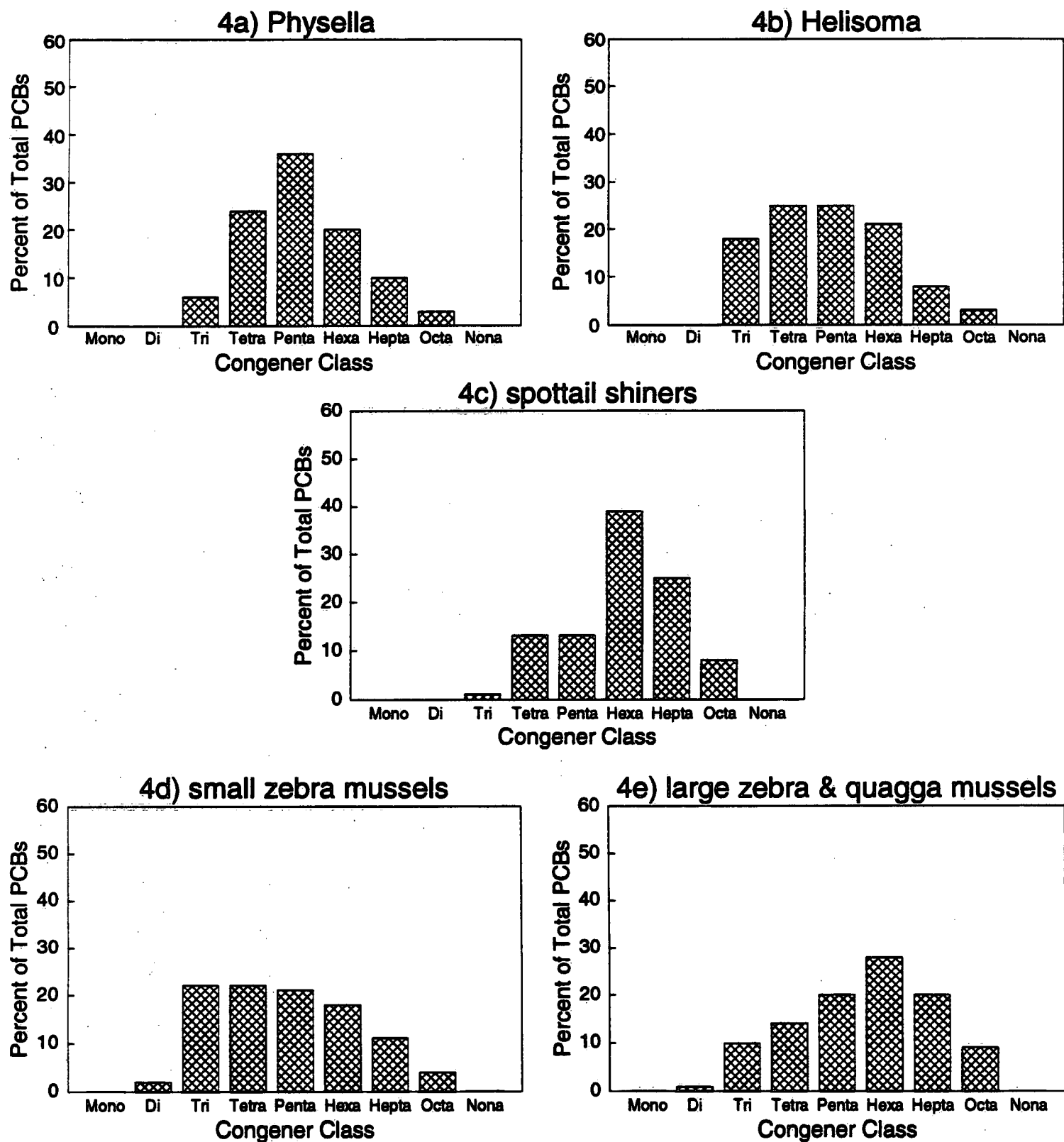


Figure 4. PCB congener-class distributions in snails (*Physella gyrina* and *Helisoma anceps*), spottail shiners (average for three composite samples), small zebra mussels (average for six composite samples), and large zebra and quagga mussels from Niagara River station 129.

Table 1. Biota and sediment samples from Niagara River station 129 analyzed for organic contaminants and metals. All organisms were analyzed whole except where otherwise indicated.

Table 1a. 1993 biota samples analyzed for organic contaminants.

DATE COLLECTED	SAMPLE ID	SAMPLE DESCRIPTION	% MOISTURE	DRY WT (g)
July 27/93	NRLZO	100 large zebra mussels (soft tissues)	95.15	1.77
July 27/93	NRLQUAG	55 large quagga mussels (soft tissues)	95.32	1.55
July 27/93	NROLIG	Oligochaetes	80.89	0.45
July 27/93	NRMIDGES	Chironomids	90.30	0.13
July 27/93	NRAMPH	Amphipods	86.54	4.16
July 27/93	NRSNA-1	Snails (Physella gyrina)	52.11	3.13
July 27/93	NRSNA-2	Snails (Helisoma anceps)	73.34	2.81
July 27/93	NRSZO-1	199 small zebra mussels	58.18	36.82
July 27/93	NRSZO-2	188 small zebra mussels	57.58	37.98
July 27/93	NRSZO-3	183 small zebra mussels	57.61	38.28
July 27/93	NRSZO-4	185 small zebra mussels	57.57	40.05
July 27/93	NRSZO-5	195 small zebra mussels	57.49	39.31
July 27/93	NRSZO-6	199 small zebra mussels	57.53	40.39
July 27/93	NRCRAY-L	13 large crayfish	72.24	13.41
July 27/93	NRCRAY-S	46 small crayfish	79.13	2.13
Sept 13/93	NR-SP-4	10 spottail shiners	77.37	2.28
Sept 13/93	NR-SP-5	10 spottail shiners	77.19	2.20
Sept 13/93	NR-SP-6	10 spottail shiners	74.38	2.85

Table 1b. 1993 sediment samples analyzed for organic contaminants.

DATE COLLECTED	SAMPLE ID	SAMPLE DESCRIPTION	% MOISTURE	DRY WT (g)
July 28/93	93SED-33	Sediment	25.13	295.91
July 28/93	93SED-34	Sediment	27.78	275.93
July 28/93	93SED-35	Sediment	22.17	269.49

Table 1c. 1992 caged mussels analyzed for organic contaminants.

DATE RETRIEVED	SAMPLE ID	SAMPLE DESCRIPTION	% MOISTURE	DRY WT (g)
July/92	92JUL-1	Individual mussel (soft tissues)	87.08	0.73
July/92	92JUL-2	Individual mussel (soft tissues)	86.27	0.50
July/92	92JUL-3	Individual mussel (soft tissues)	83.70	0.61
Oct/92	92OCT-1	Individual mussel (soft tissues)	83.60	0.86
Oct/92	92OCT-2	Individual mussel (soft tissues)	85.26	0.71
Oct/92	92OCT-3	Individual mussel (soft tissues)	83.77	0.64

Table 1d. 1993 biota and sediment samples analyzed for metals.

DATE COLLECTED	SAMPLE ID	SAMPLE DESCRIPTION	% MOISTURE	DRY WT (g)
July 27/93	NRSZM-1	68 small zebra mussels	58.03	14.18
July 27/93	NRSZM-2	69 small zebra mussels	57.46	14.48
July 27/93	NRSZM-3	70 small zebra mussels	58.01	14.31
July 27/93	NRLZM	37 large zebra mussels (soft tissues)	95.41	0.65
July 27/93	NRSQM-1	70 small quagga mussels	59.38	16.58
July 27/93	NRLZSH	37 large zebra mussel shells	-	11.44
Sept 13/93	NR-SP-7M	10 spottail shiners	80.48	2.86
Sept 13/93	NR-SP-8M	10 spottail shiners	81.70	1.77
Sept 13/93	NR-SP-9M	10 spottail shiners	81.46	2.03
July 28/93	93SED-33	Sediment	25.13	295.91
July 28/93	93SED-34	Sediment	27.78	275.93
July 28/93	93SED-35	Sediment	22.17	269.49

Table 2. Organic contaminants sought in the Niagara River biota and sediment samples.

Analysis performed by NWRI

**Organochlorine pesticides
and
Industrial organic compounds:**

PECB	o,p'-DDE
α-BHC	α-Chlordane
β-BHC	Dieldrin
γ-BHC	p,p'-DDE
HCB	o,p'-DDD
Aldrin	Endrin
OCS	p,p'-DDD
γ-Chlordane	o,p'-DDT
α-Endosulfan	p,p'-DDT
Mirex	Heptachlor
β-Endosulfan	
Heptachor Epoxide	

PCBs:

-94 individual congeners
or co-eluting groups
of congeners.

Analysis performed by WTC

Industrial organic compounds:

1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,3,5-Trichlorobenzene
1,2,3,4-Tetrachlorobenzene
1,2,4,5-Tetrachlorobezene
Hexachlorobutadiene
Hexachloroethane

PAHs:

Naphthalene
Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indo(1,2,3-c,d)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

Table 3. Concentrations of metals in biota samples from Niagara River station 129 (ug/g dry weight basis).

Sample ID	NRSZM-1	NRSZM-2	NRSZM-3	NRLZM
Sample Description	68 small zebra mussels	69 small zebra mussels	70 small zebra mussels	37 large zebra mussels (soft tissue)
Al	507	411	473	3890
Cd	0.28	0.21	0.19	3.6
Cr	28.1	23.5	31.7	8.9
Cu	10.5	8.5	8	43.3
Fe	550	445	458	2830
Mn	56	52.8	59.5	132
Ni	17.7	14.8	19.9	40.8
Pb	0.51	0.39	0.43	4.86
Zn	12.1	11.4	11.5	149
Hg	<0.05	<0.05	<0.05	0.363

Sample ID	NRSQM-1	NRLZSH	NR-SP-7M	NR-SP-8M	NR-SP-9M
Sample Description	70 small quagga mussels	37 large zebra mussel shells	10 spottail shiners	10 spottail shiners	10 spottail shiners
Al	334	281	131	93.9	149
Cd	0.33	0.043	0.146	0.01	0.1
Cr	24.6	<2	<2	<2	<2
Cu	8.1	<2	4.9	10.1	6.6
Fe	379	184	125	125	145
Mn	50.4	58	15.7	14.9	15.5
Ni	15.5	2.25	0.84	0.88	0.66
Pb	0.61	0.595	<2	<2	0.26
Zn	8.5	4.5	151	164	168
Hg	<0.05	<0.05	0.158	0.199	0.172

Table 4. Comparison of whole body concentrations of metals (ug/g dry weight) in large vs. small zebra mussels (concentrations in the former calculated from separately-determined values for soft tissues and shells), and in small zebra mussels vs. spottail shiners. Variability among replicate composite samples (C V) also compared for the latter.

Metals	Large zebra mussels	Small zebra mussels		Spottailshiners	
		Mean (n=3)	C V	Mean (n=3)	C V
Al	475	464	11%	125	23%
Cd	0.23	0.23	20%	0.09	82%
Cr	0.7	27.8	15%	<0.2- <2	-
Cu	4.2	9	15%	7.2	37%
Fe	326	484	12%	132	9%
Mn	62	56	6%	15.4	3%
Ni	4.3	17.5	15%	0.8	15%
Pb	0.82	0.44	14%	<0.2- 0.26	-
Zn	12.3	11.7	3%	161	6%
Hg	0.07	<0.05	-	0.176	12%

Table 5. Comparison of metal concentrations (ug/g dry weight) in the soft tissues of large zebra mussels vs. caged mussels exposed at Niagara River Station 129 in 1992 and pre-exposure Balsam Lake mussels. Values determined on a wet weight basis were converted to dry weight basis using a moisture content of 90 % (see text).

Metals	Balsam Lake mussels (pre-exposure)				Caged mussels 71 days		73 days		Zebra mussels July 1993 ** (n=1) ***
	May 1992 * Mean (n=3)	C V	July 1992 * Mean (n=3)	C V	May to July 1992 * Mean (n=3)	C V	July to Oct 1992 * Mean (n=3)	C V	
Cd	4.4	52%	6.2	34%	15.9	36%	5.1	62%	3.6
Cu	9.0	19%	8.1	7%	17.0	21%	12.7	12 %	43.3
Mn	6200	73%	6330	65%	12667	76%	4933	22%	132.0
Ni	< 4		< 3.7		< 4.0		< 3.5		40.8
Pb	< 6.3		< 6.0		< 6.3		< 5.3		4.9
Zn	240	29%	237	22%	710	21%	400	24 %	149.0
Hg	0.27 (t)		0.2 (t)		0.3 (t)		0.27 (t)		0.4

* analyzed by MOEE (wet wgt basis); ** analyzed by NLET (dry wgt basis).

*** composite sample of 37 individuals.

NA - not analyzed.

t - measurable trace amount ; interpret with caution.

Table 6. Comparison of concentrations of metals in sediment from Niagara River station 129 as determined by the Rexdale Laboratory (MOEE) vs. the National Laboratory for Environmental Testing (NLET). All concentrations expressed as ug/g dry weight.

Metal	93SE D-33		93SE D-34		93SE D-35	
	MOEE	NLET	MOEE	NLET	MOEE	NLET
Al-Total	4900	40600	4600	37300	4800	38800
Al-Ext.*	-	701	-	610	-	738
As-Total	1.7	2.08	1.7	1.99	1.6	2.04
Cd-Total	.50 (t)	-	.31 (t)	-	.62 (t)	-
Cd-Ext.	-	0.205	-	<.2	-	0.254
Cr-Total	13	33.4	14	25.6	15	29.4
Cr-Ext.	-	3.92	-	3.58	-	4.16
Cu-Total	10	12.5	8.5	7.92	9.5	11.3
Cu-Ext.	-	7.05	-	6.68	-	7.77
Fe-Total	15000	-	14000	-	15000	-
Fe-Ext.	-	2760	-	2440	-	2840
Hg-Total	0.15	0.181	0.16	0.199	0.2	0.205
Mn-Total	320	-	310	-	330	-
Mn-Ext.	-	255	-	227	-	260
Ni-Total	9.9	-	9.7	-	11	-
Ni-Ext.	-	3.12	-	3.03	-	3.65
Pb-Total	9.3 (t)	9.08	8.1 (t)	5.9	11	8.01
Pb-Ext.	-	6.62	-	6.51	-	7
Se-Total	<.20	<.2	<.20	<.2	<.20	<.2
Zn-Total	47	69.8	46	59.6	59	62.8
Zn-Ext.	-	25.3	-	21.8	-	28.1

*Ext. - extractable

t - measurable trace amount; interpret with caution.

Table 7. Total PCBs (sum of all congeners) and lipid contents in biota samples from the Niagara River.

SAMPLE ID	SAMPLE DESCRIPTION	Total PCBs (ng/g dry wgt)	Lipid content (%dry wgt)
NRLZO	large zebra mussels	91.7	5.28%
NRLQUAG	large quagga mussels	423.4	7.59%
NRAMPH	amphipods	235.6	2.57%
NRSNA-1	Physella gyrina	115.1	0.61%
NRSNA-2	Helisoma anceps	50.4	0.45%
NRSZO-1	small zebra mussels	15.8	0.12%
NRSZO-2	small zebra mussels	33.7	0.21%
NRSZO-3	small zebra mussels	33.7	0.19%
NRSZO-4	small zebra mussels	36.3	0.26%
NRSZO-5	small zebra mussels	57.1	0.22%
NRSZO-6	small zebra mussels	29.0	0.43%
NRCRAY-L	large crayfish	109.6	4.00%
NRCRAY-S	small crayfish	126.6	1.41%
NR-SP-4	spottail shiners	411.5	5.42%
NR-SP-5	spottail shiners	524.5	6.81%
NR-SP-6	spottail shiners	249.4	2.98%
92JUL-1	caged mussel	303.7	2.62%
92JUL-2	caged mussel	293.0	2.62%
92JUL-3	caged mussel	189.4	2.47%
92OCT-1	caged mussel	169.0	2.22%
92OCT-2	caged mussel	212.1	2.12%
92OCT-3	caged mussel	213.3	1.89%

Table 8. Average concentrations (ng/g dry wgt.) of total PCBs, organochlorine pesticides, industrial organic compounds and PAHs in sediment samples 33, 34, and 35 from Niagara River station 129 in July, 1993, as determined by NWRI and WTC versus MOEE.

Compound	NWRI and WTC Mean or Range (C V)	MOEE Range
Naphthalene	<= 10 t	< 20 - 24 t
Acenaphthylene	< 10	< 20
Acenaphthene	< 10	< 20
Fluorene	<= 10 t	< 20 - 43 t
Phenanthrene	40 (35%)	< 20 - 26 t
Anthracene	< 10	< 20
Fluoranthene	60 (29%)	42 - 83 t
Pyrene	50 (35%)	34 - 83 t
Benzo(a)anthracene	60 (44%)	25 - 51 t
Chrysene	40 (48%)	31 - 76 t
Benzo(b)fluoranthene	130 (20%)	33 - 73 t
Benzo(k)fluoranthene	50 (12%)	32 - 61 t
Benzo(a)pyrene	60 (58%)	30 - 50 t
Indeno(1,2,3-c,d)pyrene	60 (44%)	< 40 - 58 t
Dibenzo(a,h)anthracene	< 10	< 40
Benzo(g,h,i)perylene	70 (38%)	< 40 - 49 t
1,2,3 - TCB	< 2	< 2
1,2,4 - TCB	< 2	< 2
1,3,5 - TCB	< 2	< 2
1,2,3,4 - TTCB	0.37 (41%) t	< 1
1,2,4,5 - TTCB	0.34 (3%) t	< 1
HCBD	< 1.5	< 1
HCE	< 1.2	< 1
Total PCBs	41.97 (36%)	< 20
a-BHC	ND - 0.10	< 1
b-BHC	ND	< 1
g-BHC	ND - 0.09	< 1
Heptachlor	ND	< 1
Heptachlor epoxide	ND	< 1
g-Chlordane	ND	< 2
a-Endosulfan	ND	NA
a-Chlordane	ND	< 2
Dieldrin	ND	< 2
p,p'-DDE	ND - 0.49	< 1
o,p'-DDD	ND	NA
Endrin	ND - 0.47	< 4
b-Endosulfan	no data *	NA
p,p'-DDD	ND - 1.1	< 5
o,p'-DDT	ND	< 5
p,p'-DDT	ND - 2.6	< 5
PECB	0.84 (37%)	< 1
HCB	1.9 (23%)	< 1 - 2 t
Aldrin	ND	< 1
o,p'-DDE	ND	NA
Mirex	ND - 1.5	< 5
OCS	ND	< 1

t - measurable trace amount ; interpret with caution.

NA - not analyzed.

ND - not detected.

* compound did not elute ; see text.

Table 9. Concentrations of total PCBs, total PAHs and total CBs in sediment and biota from Niagara River station 129 (ng/g dry weight for all samples). Trace values were used in calculating total PAHs and total CBs.

Sample	Total PCBs	Total PAHs	Total CBs*
Sediment	42 **	630 **	0.70**
Large zebra mussels	92	450	ND
Large quagga mussels	423	640	ND
Amphipods	236	460	1.09
Snails (P. gyrina)	115	70	ND
Snails (H. anceps)	50	180	ND
Small zebra mussels	34 ***	73 **	ND **
Large crayfish	110	180	0.54
Small crayfish	127	90	ND
Spottail shiners	395**	27 **	0.44****
Caged mussels (July)	262**	507 **	ND **
Caged mussels (October)	198**	677 **	ND **

* total CBs refers to the tri- and tetrachlorobenzenes analyzed by the WTC.

** average of three samples.

*** average of six samples.

**** in one sample only.

ND - not detected.

APPENDICES

Appendix A1. Limits of Detection (LOD) for PCB congeners - in biota samples.
Based on 1 millilitre sample extract and 2 uL injection.

PCB congener	Limit of Detection LOD (ng)
PCB-1	0.61
PCB-3	0.94
PCB-4-10	0.28
PCB-7	0.03
PCB-6	0.09
PCB-8-5	0.09
PCB-19	0.09
PCB-12-13	0.06
PCB-18**	0.09
PCB-17**	0.05
PCB-15*	u
PCB-24-27	0.10
PCB-16**	0.04
PCB-32**	0.33
PCB-54 (ocr)	0.10
PCB-29 (ocr)	0.06
PCB-26	0.05
PCB-25	0.04
PCB-31-28	0.06
PCB-33-53	0.06
PCB-22-51	0.04
PCB-45	0.04
PCB-46	0.05
PCB-52	0.05
PCB-49	0.04
PCB-48**	0.05
PCB-47**	0.03
PCB-44	0.04
PCB-42-37	0.04
PCB-64-41-71	0.04
PCB-40	0.04

PCB congener	Limit of Detection LOD (ng)
PCB-100	0.02
PCB-63	0.03
PCB-74	0.03
PCB-70-76	0.04
PCB-66**	0.03
PCB-95**	0.03
PCB-91-121	0.07
PCB-60-56	0.03
PCB-89	0.00
PCB-84-92	0.05
PCB-101	0.03
PCB-99	0.03
PCB-119 (ocr)	0.04
PCB-83	0.08
PCB-97	0.03
PCB-87-81	0.03
PCB-95-86	0.03
PCB-136-77**	0.03
PCB-110**	0.03
PCB-52	0.03
PCB-151	0.02
PCB-135-144	0.05
PCB-107-123 (ocr)	0.05
PCB-149-118	0.46
PCB-134-114	0.02
PCB-146-105**	0.05
PCB-132**	0.02
PCB-153**	0.03
PCB-141	0.02
PCB-137-176	0.03
PCB-138-163	0.03

PCB congener	Limit of Detection LOD (ng)
PCB-178-126	0.02
PCB-158 (ocr)	0.02
PCB-129 (ocr)	0.04
PCB-175	0.02
PCB-187-182**	0.03
PCB-183	0.02
PCB-167-185	0.02
PCB-174	0.02
PCB-177	0.02
PCB-171-156	0.04
PCB-157-173-202***	0.03
PCB-200***	0.03
PCB-172**	0.02
PCB-197*	0.03
PCB-180**	0.02
PCB-193	0.02
PCB-191	0.03
PCB-199	0.02
PCB-170**	0.02
PCB-190**	0.02
PCB-198	0.02
PCB-201	0.02
PCB-203-196	0.02
PCB-189 (ocr)	0.02
PCB-195-208	0.03
PCB-207	0.01
PCB-194	0.02
PCB-205	0.02
PCB-206	0.02

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.
- u unknown

Appendix A2. Limits of Quantitation (LOQ) for PCB congeners - in biota samples.

Based on 1 millilitre sample extract and 2 uL injection.

To determine the LOQs in ng/g for a given sample, divide these values by the weight of the sample (g).

PCB congener	Limit of Quantitation LOQ (ng)
PCB-1	2.0
PCB-3	3.1
PCB-4-10	0.94
PCB-7	0.11
PCB-6	0.31
PCB-8-5	0.29
PCB-19	0.30
PCB-12-13	0.20
PCB-18**	0.29
PCB-17**	0.16
PCB-15*	u
PCB-24-27	0.32
PCB-16**	0.15
PCB-32**	1.1
PCB-54 (ocr)	0.33
PCB-29 (ocr)	0.21
PCB-28	0.16
PCB-25	0.14
PCB-31-28	0.22
PCB-33-53	0.21
PCB-22-51	0.15
PCB-45	0.15
PCB-46	0.16
PCB-52	0.15
PCB-48	0.15
PCB-48**	0.16
PCB-47**	0.11
PCB-44	0.13
PCB-42-37	0.13
PCB-64-41-71	0.14
PCB-40	0.12

PCB congener	Limit of Quantitation LOQ (ng)
PCB-100	0.08
PCB-63	0.11
PCB-74	0.11
PCB-70-76	0.12
PCB-68**	0.11
PCB-95**	0.11
PCB-91-121	0.23
PCB-60-56	0.09
PCB-89	0.00
PCB-84-92	0.17
PCB-101	0.11
PCB-99	0.10
PCB-119 (ocr)	0.13
PCB-83	0.26
PCB-97	0.11
PCB-87-81	0.10
PCB-85-86	0.09
PCB-136-77**	0.10
PCB-110**	0.09
PCB-82	0.09
PCB-151	0.08
PCB-135-144	0.16
PCB-107-123 (ocr)	0.15
PCB-149-118	1.5
PCB-134-114	0.07
PCB-146-105**	0.17
PCB-132**	0.05
PCB-153**	0.09
PCB-141	0.06
PCB-137-176	0.11
PCB-138-163	0.10

PCB congener	Limit of Quantitation LOQ (ng)
PCB-178-126	0.07
PCB-158 (ocr)	0.08
PCB-129 (ocr)	0.14
PCB-175	0.08
PCB-187-182**	0.11
PCB-183	0.07
PCB-167-185	0.08
PCB-174	0.07
PCB-177	0.07
PCB-171-156	0.13
PCB-157-173-202***	0.08
PCB-200***	0.09
PCB-172**	0.07
PCB-197*	0.09
PCB-180**	0.07
PCB-193	0.07
PCB-191	0.09
PCB-199	0.08
PCB-170**	0.06
PCB-190**	0.08
PCB-198	0.08
PCB-201	0.07
PCB-203-196	0.08
PCB-189 (ocr)	0.07
PCB-195-208	0.09
PCB-207	0.03
PCB-194	0.06
PCB-205	0.08
PCB-206	0.07

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.
- u unknown

Appendix A3. Limits of Detection (LOD) for PCB congeners- in sediment samples.
Based on 1 millilitre sample extract and 2 uL injection.

PCB congener	Limit of Detection LOD (ng)
PCB-1	0.81
PCB-3	1.2
PCB-4-10	0.37
PCB-7	0.05
PCB-6	0.13
PCB-8-5	0.12
PCB-19	0.12
PCB-12-13	0.08
PCB-18**	0.12
PCB-17**	0.07
PCB-15*	u
PCB-24-27	0.13
PCB-16**	0.06
PCB-32**	0.44
PCB-34 (ocr)	0.13
PCB-28 (ocr)	0.08
PCB-26	0.07
PCB-25	0.05
PCB-31-28	0.09
PCB-33-53	0.08
PCB-22-51	0.06
PCB-45	0.06
PCB-46	0.07
PCB-52	0.06
PCB-49	0.06
PCB-48**	0.06
PCB-47**	0.04
PCB-44	0.05
PCB-42-37	0.05
PCB-84-41-71	0.05
PCB-40	0.05

PCB congener	Limit of Detection LOD (ng)
PCB-100	0.03
PCB-63	0.04
PCB-74	0.05
PCB-70-76	0.05
PCB-66**	0.04
PCB-95**	0.04
PCB-91-121	0.09
PCB-60-36	0.04
PCB-89	0.00
PCB-84-92	0.07
PCB-101	0.05
PCB-99	0.04
PCB-119 (ocr)	0.05
PCB-83	0.10
PCB-97	0.04
PCB-87-81	0.04
PCB-85-96	0.04
PCB-136-77**	0.04
PCB-110**	0.03
PCB-92	0.03
PCB-151	0.03
PCB-135-144	0.07
PCB-149-118	0.62
PCB-107-123 (ocr)	0.08
PCB-134-114	0.03
PCB-146-105**	0.07
PCB-132**	0.02
PCB-153**	0.03
PCB-141	0.02
PCB-137-176	0.04
PCB-138-163	0.04

PCB congener	Limit of Detection LOD (ng)
PCB-178-126	0.03
PCB-158 (ocr)	0.03
PCB-129 (ocr)	0.05
PCB-175	0.03
PCB-187-182**	0.04
PCB-183	0.03
PCB-167-185	0.03
PCB-174	0.03
PCB-177	0.03
PCB-171-156	0.05
PCB-157-173-202***	0.03
PCB-200***	0.04
PCB-172**	0.03
PCB-187*	0.04
PCB-180**	0.03
PCB-193	0.03
PCB-191	0.04
PCB-199	0.03
PCB-170**	0.02
PCB-190**	0.03
PCB-198	0.03
PCB-201	0.03
PCB-203-196	0.03
PCB-189 (ocr)	0.03
PCB-195-208	0.04
PCB-207	0.01
PCB-194	0.02
PCB-205	0.03
PCB-206	0.03

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.
- u unknown

Appendix A4. Limits of Quantitation (LOQ) for PCB congeners- In sediment samples.

Based on 1 millilitre sample extract and 2 uL Injection.

To determine the LOQs in ng/g for a given sample, divide these values by the weight of the sample (g).

PCB congener	Limit of Quantitation LOQ (ng)
PCB-1	2.7
PCB-3	4.2
PCB-4-10	1.2
PCB-7	0.15
PCB-6	0.42
PCB-8-5	0.39
PCB-19	0.40
PCB-12-13	0.26
PCB-18**	0.39
PCB-17**	0.22
PCB-15*	u
PCB-24-27	0.43
PCB-16**	0.20
PCB-32**	1.5
PCB-54 (ocr)	0.43
PCB-29 (ocr)	0.28
PCB-28	0.22
PCB-25	0.18
PCB-31-28	0.29
PCB-33-53	0.28
PCB-22-51	0.19
PCB-45	0.20
PCB-46	0.22
PCB-52	0.20
PCB-49	0.19
PCB-48**	0.22
PCB-47**	0.14
PCB-44	0.18
PCB-42-37	0.17
PCB-64-41-71	0.18
PCB-40	0.16

PCB congener	Limit of Quantitation LOQ (ng)
PCB-100	0.11
PCB-63	0.15
PCB-74	0.15
PCB-70-76	0.16
PCB-66**	0.15
PCB-95**	0.15
PCB-81-121	0.31
PCB-60-56	0.12
PCB-89	0.01
PCB-84-92	0.23
PCB-101	0.15
PCB-99	0.14
PCB-119 (ocr)	0.17
PCB-83	0.35
PCB-97	0.15
PCB-87-81	0.13
PCB-85-86	0.13
PCB-136-77**	0.14
PCB-110**	0.12
PCB-82	0.11
PCB-151	0.11
PCB-135-144	0.22
PCB-149-118	2.1
PCB-107-123 (ocr)	0.25
PCB-134-114	0.10
PCB-146-105**	0.23
PCB-132**	0.07
PCB-153**	0.12
PCB-141	0.08
PCB-137-176	0.14
PCB-138-163	0.14

PCB congener	Limit of Quantitation LOQ (ng)
PCB-178-126	0.09
PCB-158 (ocr)	0.10
PCB-129 (ocr)	0.18
PCB-175	0.10
PCB-187-182**	0.14
PCB-183	0.10
PCB-167-185	0.10
PCB-174	0.10
PCB-177	0.09
PCB-171-156	0.18
PCB-157-173-202**	0.11
PCB-200**	0.12
PCB-172**	0.09
PCB-197*	0.13
PCB-180**	0.09
PCB-193	0.09
PCB-191	0.12
PCB-199	0.10
PCB-170**	0.07
PCB-190**	0.10
PCB-198	0.11
PCB-201	0.10
PCB-203-196	0.11
PCB-189 (ocr)	0.09
PCB-195-208	0.12
PCB-207	0.04
PCB-194	0.07
PCB-205	0.11
PCB-206	0.10

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.
- u unknown

Appendix A5. Limits of Detection (LOD) and Limits of Quantitation (LOQ) for Organochlorines- In sediment samples.
Based on 1 millilitre sample extract and 2uL Injection.

To determine the LOQs in ng/g for a given sample, divide these values by the weight of the sample (g).

Organochlorines	Limit of Detection (LOD) (ng)
a-BHC	0.17
b-BHC	0.42
g-BHC	0.22
Heptachlor	0.36
Heptachlor epoxide	0.24
g-Chlordane	0.19
a-Endosulfan	0.24
a-Chlordane	0.19
Dieldrin	0.31
p,p-DDE	0.25
o,p-DDD	0.41
Endrin	0.75
b-Endosulfan	0.28
p,p-DDD	0.39
o,p-DDT	0.82
p,p-DDT	1.1
PCB	0.03
PCB	0.02
Aldrin	0.03
o,p-DDE	0.35
Mirex	0.04
OCS	0.03

Organochlorines	Limit of Quantitation (LOQ) (ng)
a-BHC	0.58
b-BHC	1.4
g-BHC	0.72
Heptachlor	1.2
Heptachlor epoxide	0.80
g-Chlordane	0.65
a-Endosulfan	0.79
a-Chlordane	0.65
Dieldrin	1.0
p,p-DDE	0.83
o,p-DDD	1.4
Endrin	2.5
b-Endosulfan	0.94
p,p-DDD	1.3
o,p-DDT	2.7
p,p-DDT	3.8
PCB	0.09
PCB	0.08
Aldrin	0.11
o,p-DDE	1.2
Mirex	0.13
OCS	0.09

Appendix B. Interpretation of GC responses for coeluting PCB congeners.

The following are the labels placed on the specific PCB congeners when they are listed in the final data tables.
(S1 signal from SPB-1 column & S2 is signal from SPB-5 column).

- determined by subtraction.
- single column result plus subtracted value.
- single column result confirmed by second column.
- ocr one column result, not identified by other column.

(1) Congeners requiring calculations.

(i) PCB-15, PCB-17, PCB-18

S1 PCB-18(15)	S2 PCB-18
S1 PCB-17	S2 PCB-17(15)

When the co-eluting concentration is > the concentration of the congener eluting on its own, then PCB-15 is reported. It must be confirmed by both columns.

When $[PCB-18(15)] > [PCB-18]$ and $[PCB-17(15)] > [PCB-17]$ the presence of PCB-15 can be confirmed.

$$\begin{aligned}[PCB-18(15)] - [PCB-18] &= [PCB-15] \text{ from S1} \\ [PCB-17(15)] - [PCB-17] &= [PCB-15] \text{ from S2}\end{aligned}$$

The concentrations can be reported as follows:

PCB-18 ••
PCB-17 ••
PCB-15 •

(ii) PCB-136, PCB-110, PCB-77

S1 PCB-136(77)	S2 PCB-136
S1 PCB-110	S2 PCB-110(77)

When the co-eluting concentration is > the concentration of the congener eluting on its own, then PCB-77 is reported. It must be confirmed by both columns.

When $[PCB-136(77)] > [PCB-136]$ and $[PCB-110(77)] > [PCB-110]$ the presence of PCB-77 can be confirmed.

$$\begin{aligned}[PCB-136(77)] - [PCB-136] &= [PCB-77] \text{ from S1} \\ [PCB-110(77)] - [PCB-110] &= [PCB-77] \text{ from S2}\end{aligned}$$

The concentrations can be reported as follows:

PCB-136 ••
PCB-110 ••
PCB-77 •

(iii) PCB-146, PCB-105

S1 PCB-146(105)	S2 PCB-146
S1 PCB-132	
S1 PCB-153	S2 PCB-132(153)(105)

When the co-eluting concentration is > the concentration of the congener eluting on its own, then PCB-105 is reported. It must be confirmed by both columns.

When $[PCB-146(105)] > [PCB-146]$ and $[PCB-132(153)(105)] > ([PCB-132] + [PCB-153])$, then PCB-105 is confirmed by both columns.

If there is a response for PCB-132(153)(105) then PCB-132 and PCB-153 are confirmed: it doesn't have to equal $[PCB-132] + [PCB-153]$.

The concentrations can be reported as follows:

PCB-146 ••
PCB-105 •
PCB-132 ••
PCB-153 ••

(iv) PCB-158-171, PCB-157-173-202, PCB-200

S1 PCB-158(171)	S2 PCB-158(171)202
S1 PCB-157-173(202)	
S1 PCB-200	S2 PCB-200(157)

Response for PCB-158(171)202 confirms PCB-158(171).

$[PCB-158(171)202] - [PCB-158(171)] = [PCB-202]$

-this value for [PCB-202] usually ends up being 0, so then the two responses are averaged for PCB-158(171).

Then PCB-157(202) can be reported as a coeluting set which is confirmed if response for PCB-157(200) > PCB-200.

The concentrations can be reported as follows:

PCB-158(171) •••
PCB-157-173(202) •••
PCB-200 ••

(v) PCB-187(182), PCB-128

S1 PCB-187-182(128)	S2 PCB-187-182
	S2 PCB-128

When the co-eluting concentration is > the concentration of the congener eluting on its own, then PCB-128 is confirmed by both columns.

That is $[PCB-187-182(128)] > [PCB-187-182]$ confirms the response given by signal 2 for PCB-128.

The concentrations can be reported as follows:

PCB-187-182 ••
PCB-128 •

(vi) PCB-172, PCB-180, PCB-197

S1 PCB-172	S2 PCB-172-(197)
S1 PCB-180(197)	S2 PCB-180

When the co-eluting concentration is > the concentration of the congener eluting on its own, then PCB-197 is reported. It must be confirmed by both columns.

If $[PCB-172(197)] > [PCB-172]$ and $[PCB-180(197)] > [PCB-180]$, then PCB-197 is confirmed by both columns.

The concentrations can be reported as follows:

PCB-172 ••
PCB-197 •
PCB-180 ••

(2) Congeners that coelute on one column but are separated on the other.

If they are both present on one column and there is a response for the coeluting pair on the other column then they can be reported as: PCB-123 ••

The relevant PCBs in our set are listed below:

Set #	SPB-1 elution set	SPB-5 elution set
(1)	PCB-16 PCB-32	PCB-16(32)
(2)	PCB-47 PCB-48	PCB-47(48)
(3)	PCB-66 PCB-65	PCB-66(65)
(4)	PCB-170 PCB-190	PCB-170(190)

(3) Congeners that are identified only on one column.

Congeners flagged "ocr" are generally found in trace amounts. At the dates of analysis there was no analytical confirmation for these compounds on one of the columns using the GB PCB standard. Relevant PCBs are listed below:

PCBs confirmed only on SPB-1

PCB-54
PCB-29
PCB-119
PCB-158
PCB-129
PCB-189

PCBs confirmed only on SPB-5

PCB-107-123

Appendix C. Recoveries of PCBs by Soxhlet extraction.

PCB congeners	PCB meth spk-1	PCB meth spk-2	PCB meth spk-3	average % recovery
PCB-1	85.26	98.44	90.14	91.28
PCB-3	87.42	99.77	147.71	111.63
PCB-4-10	99.18	109.60	100.32	103.03
PCB-7	77.75	97.59	82.60	85.98
PCB-6	86.11	116.38	118.52	107.00
PCB-8-5	79.12	87.02	99.22	88.45
PCB-19	91.54	119.13	124.78	111.82
PCB-12-13	111.78	110.11	85.56	102.48
PCB-18-15	87.78	101.93	112.57	100.76
PCB-17	83.74	100.44	115.70	99.96
PCB-24-27	99.23	98.31	98.50	98.68
PCB-16	106.14	109.84	107.88	107.95
PCB-32	104.42	109.88	107.69	107.33
PCB-54	37.14	120.00	74.00	77.05
PCB-29	30.00	122.50	89.50	80.67
PCB-26	104.03	93.14	101.18	99.45
PCB-25	113.57	102.73	106.82	107.70
PCB-31-28	89.31	91.49	103.83	94.88
PCB-33-53	96.75	105.00	90.57	97.44
PCB-22-51	87.74	89.95	124.21	100.63
PCB-45	100.19	92.28	93.56	95.34
PCB-46	122.68	128.32	113.87	121.62
PCB-52	83.74	101.73	94.23	93.23
PCB-49	99.85	105.38	117.20	107.48
PCB-48	118.48	117.14	102.00	112.54
PCB-47	93.55	82.70	107.90	94.72
PCB-44	111.08	121.14	106.85	113.02
PCB-42-37	97.49	112.45	102.21	104.05
PCB-64-41-71	101.46	117.63	103.53	107.54
PCB-40	100.96	93.42	91.23	95.21
PCB-100	90.77	92.73	96.73	93.41
PCB-63	134.38	78.66	111.25	108.09
PCB-74	93.63	109.74	113.94	105.77
PCB-70-76	88.97	49.61	87.85	75.47
PCB-66	99.40	119.86	100.66	106.64
PCB-95	99.37	103.97	100.66	101.33
PCB-91-121	94.84	105.08	110.65	103.52
PCB-60-56	69.33	100.40	116.64	95.46
PCB-92-84	84.82	100.33	103.30	96.15
PCB-89	58.36	98.14	122.43	92.98
PCB-101	98.08	119.18	92.67	103.31
PCB-99	98.47	118.66	116.12	111.09
PCB-119	145.50	148.90	90.00	128.13
PCB-83	103.38	117.34	113.75	111.49

Appendix C. (cont'd)

PCB congeners	PCB meth spk-1	PCB meth spk-2	PCB meth spk-3	average % recovery
PCB-97	88.51	126.99	108.29	107.93
PCB-87-81	99.19	94.47	105.76	99.81
PCB-85	116.30	83.26	119.57	106.38
PCB-136-77	106.19	106.79	113.55	108.84
PCB-110	93.75	103.00	92.13	96.29
PCB-82	51.84	94.38	96.21	80.81
PCB-151	100.52	111.19	110.68	107.47
PCB-135-144	95.65	104.92	114.36	104.97
PCB-107-123	57.14	118.57	105.71	93.81
PCB-149-118	100.58	105.35	114.94	106.96
PCB-134-114	107.45	122.16	118.79	116.13
PCB-146-105	112.16	118.94	113.71	114.94
PCB-132	98.91	114.88	111.25	108.35
PCB-153	106.56	110.05	110.25	108.95
PCB-141	82.99	105.95	128.91	105.95
PCB-137-176	113.58	109.34	114.19	112.37
PCB-138-163	114.10	98.54	94.50	102.38
PCB-158	122.69	119.23	92.31	111.41
PCB-129	118.00	96.70	106.00	106.90
PCB-178-126	98.86	108.51	110.48	105.95
PCB-175	106.25	112.50	108.33	109.03
PCB-187-182-128	95.49	110.48	106.33	104.10
PCB-183	103.18	113.21	94.24	103.54
PCB-128	91.82	100.00	118.18	103.33
PCB-185-167	105.72	108.53	110.20	108.15
PCB-174	104.98	109.40	106.25	106.88
PCB-177	105.72	109.12	106.92	107.25
PCB-156-171	93.40	111.88	107.84	104.37
PCB-157-173-202	55.73	103.64	108.91	89.43
PCB-200	99.68	104.13	103.70	102.50
PCB-172	106.93	109.06	109.38	108.45
PCB-180-197	99.03	108.87	99.74	102.55
PCB-193	105.32	94.58	122.58	107.49
PCB-191	98.20	98.98	105.00	100.73
PCB-199	96.59	99.89	98.64	98.37
PCB-170	107.30	99.47	105.17	103.98
PCB-190	117.63	109.88	112.50	113.33
PCB-198	132.93	149.30	108.67	130.30
PCB-201	106.65	110.18	106.58	107.80
PCB-203-196	105.72	107.25	107.29	106.75
PCB-189	69.89	66.93	125.00	87.27
PCB-195-208	104.58	107.81	105.39	105.93
PCB-207	97.77	98.14	115.45	103.79
PCB-194	109.19	109.97	107.81	108.99
PCB-205	129.72	101.61	111.11	114.15
PCB-206	122.83	126.96	119.57	123.12

Appendix D1. Recoveries of PCBs by Polytron extraction.

PCB congeners	% recovery
PCB-1	99.00
PCB-3	104.00
PCB-4-10	94.00
PCB-7	118.00
PCB-6	94.87
PCB-8-6	98.59
PCB-19	85.94
PCB-12-13	99.33
PCB-18	89.66
PCB-15	76.29
PCB-17	85.49
PCB-24-27	95.55
PCB-16	71.10
PCB-32	86.76
PCB-64	51.43
PCB-29	75.00
PCB-26	79.67
PCB-25	94.53
PCB-31-28	83.37
PCB-33-63	98.42
PCB-22-51	99.56
PCB-45	104.15
PCB-46	106.34
PCB-52	103.43
PCB-49	95.69
PCB-48	84.12
PCB-47	100.34
PCB-44	104.63
PCB-42-37	104.92
PCB-64-41-71	106.44
PCB-40	102.88

PCB congeners	% recovery
PCB-100	81.86
PCB-63	107.00
PCB-74	91.81
PCB-70-76	88.46
PCB-66	85.62
PCB-95	72.97
PCB-91-121-55	79.71
PCB-60-56	58.86
PCB-92-84	79.54
PCB-89	51.71
PCB-101	93.62
PCB-99	95.20
PCB-119	78.00
PCB-83	80.31
PCB-97	80.91
PCB-87-81	78.87
PCB-85	87.83
PCB-136-77	74.45
PCB-110	76.29
PCB-82	43.55
PCB-151	89.12
PCB-135-144	88.25
PCB-107-123	102.86
PCB-149-118	88.87
PCB-134-114	88.29
PCB-146-106	91.86
PCB-132	70.72
PCB-153	89.31
PCB-141	88.00
PCB-137-176	82.37

PCB congeners	% recovery
PCB-138-163	90.39
PCB-158	86.12
PCB-129	82.80
PCB-178-126	81.59
PCB-175	77.83
PCB-187-182-128	85.40
PCB-183	86.31
PCB-128	78.18
PCB-185-167	82.90
PCB-174	83.71
PCB-177	85.13
PCB-166-171	86.27
PCB-157-173-202	73.70
PCB-200	80.65
PCB-172	77.38
PCB-197	86.73
PCB-180	87.56
PCB-193	85.94
PCB-191	84.50
PCB-199	79.57
PCB-170	87.30
PCB-190	77.55
PCB-198	104.00
PCB-201	83.64
PCB-203-196	84.28
PCB-189	77.73
PCB-195-208	83.79
PCB-207	80.23
PCB-194	86.08
PCB-205	81.22
PCB-206	72.26

Appendix D2. Recoveries of OCs by Polytron extraction.

OCs	%recovery
a-BHC	97.75
b-BHC	109.32
g-BHC	103.53
Heptachlor	112.37
Heptachlor epoxide	109.61
g-Chlordane	106.87
a-Endosulfan	107.75
a-Chlordane	105.96
Dieldrin	94.78
p,p-DDE	106.84
o,p-DDD	106.86
Endrin	80.16
b-Endosulfan *	2.84
p,p-DDD	107.71
o,p-DDT	115.64
p,p-DDT	129.71
PECB	90.87
HCB	98.88
Aldrin	97.49
o,p-DDE	107.75
Mirex	120.13
OCS	58.95

* compound not eluted ; see text.

Appendix E. Data listing for the MOEE analysis of caged mussels (Elliptio complanata) collected from Balsam Lake and incubated at station 129 in 1992.

Variable	<W (ng/g)	Balsam Lake- May replicate samples	Balsam Lake- July replicate samples	Station 129 (May to July) replicate samples	Station 129 (July to October) replicate samples
Hexachlorobutadiene	(1)	<W	<W	<W	<W
Hexachlorobenzene	(1)	<W	<W	<W	<W
Hexachloroethane	(1)	<W	<W	<W	<W
Pentachlorobenzene	(1)	<W	<W	<W	<W
2,3,6-Trichlorotoluene	(1)	<W	<W	<W	<W
2,4,6-Trichlorotoluene	(1)	<W	<W	<W	<W
1,2,3-Trichlorobenzene	(2)	<W	<W	<W	<W
1,2,3,4-Tetrachlorobenzene	(1)	<W	<W	<W	<W
1,2,4-Trichlorobenzene	(2)	<W	<W	<W	<W
1,2,4,5-Tetrachlorobenzene	(1)	<W	<W	<W	<W
1,3,6-Trichlorobenzene	(2)	<W	<W	<W	<W
Aldrin	(1)	<W	NA	<W	NA
a-BHC	(1)	<W	NA	<W	NA
b-BHC	(1)	<W	NA	<W	NA
g-BHC	(1)	<W	NA	<W	NA
a-Chlordane	(2)	<W	NA	<W	NA
g-Chlordane	(2)	<W	NA	<W	NA
Heptachlor	(1)	<W	NA	<W	NA
Mirex	(5)	<W	NA	<W	NA
Octachlorostyrene	(1)	<W	NA	<W	NA
o,p'-DDT	(5)	<W	NA	<W	NA
Total PCBs	(20)	<W	NA	<W	NA
p,p'-DDD	(5)	<W	NA	<W	NA
p,p'-DDE	(1)	<W	NA	<W	NA
p,p'-DDT	(5)	<W	NA	<W	NA
Toxaphene	(200)	<W	NA	<W	NA
Arsenic	(-)	0.69	0.25	0.65	0.37
Cadmium	(-)	0.32	0.71	2.10	0.64
Copper	(-)	1.0	1.0	2.1	1.3
Mercury	(0.01)	0.02<T	0.04<T	0.03<T	0.03<T
Manganese	(-)	560	1100	2300	620
Nickel	(-)	<0.6<	<0.3<	<0.4<	<0.4<
Lead	(-)	<0.9<	<0.5<	<0.6<	<0.7<
Selenium	(-)	0.58	0.33	0.48	0.56
Zinc	(-)	24	31	80	45

Note:

NA no analysis.

< actual result is less than reported value.

< T a measurable trace amount (interpret with caution).

< W no measurable response (zero) [less than reported value].

Appendix F. Congener-specific PCB data for Niagara River biota samples.
Total PCBs calculated without bql values.

Sample ID	NRLZO	NROUAG	NROLIG	NRMIDGES	NRAMPH	NRSNA-1	NRSNA-2
Sample wgt extracted (g)	1.77	1.55	0.45	0.13	4.16	3.13	2.81
Lipid wgt (g)	0.093	0.018	0.032	0.001	0.107	0.019	0.013
% lipid / dry wgt	5.28	7.59	7.19	1.06	2.57	0.61	0.45
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
PCB-1	13.4	bql	nd	nd	8.7	bql	8.7
PCB-3	nd	nd	nd	nd	nd	nd	nd
PCB-4-10	nd	nd	nd	nd	nd	nd	nd
PCB-7	nd	nd	nd	1.2	bql	nd	nd
PCB-8	nd	nd	nd	nd	nd	0.46	bql
PCB-8-8	0.88	1.8	2.8	19.2	1.8	nd	nd
PCB-19	nd	nd	nd	nd	nd	nd	nd
PCB-12-13	nd	nd	nd	nd	nd	nd	nd
PCB-18**	2.31	bql	8.8	12.2	nd	3.4	nd
PCB-16*	nd	nd	nd	nd	nd	nd	nd
PCB-17**	0.82	2.3	5.2	17.4	1.2	0.43	0.32
PCB-24-27	nd	nd	nd	nd	nd	nd	nd
PCB-18**	nd	0.74	9.6	nd	nd	nd	nd
PCB-32**	0.89	2.6	25.7	bql	nd	1.7	4.7
PCB-84	(ocr)	0.39	0.24	2.0	nd	0.32	1.0
PCB-29	(ocr)	2.58	0.14	4.2	bql	1.3	0.29
PCB-26	1.0	2.0	6.6	16.6	nd	0.80	bql
PCB-26	nd	1.3	3.7	10.6	0.71	nd	0.27
PCB-31-28	4.4	8.6	32.4	nd	12.8	3.6	3.1
PCB-33-63	1.9	6.7	22.8	nd	3.6	0.98	0.65
PCB-22-61	nd	2.8	8.1	31.4	1.7	nd	nd
PCB-46	1.7	1.8	2.4	16.7	0.77	0.26	nd
PCB-46	nd	0.83	1.7	4.4	0.68	0.60	bql
PCB-62	int	int	int	int	int	12.7	7.6
PCB-49	8.0	17.2	60.4	163.7	12.1	6.4	2.8
PCB-48**	nd	nd	nd	52.1	10.1	nd	nd
PCB-47**	nd	nd	nd	60.4	nd	nd	nd
PCB-44	1.8	7.1	22.3	162.9	16.7	2.3	2.1
PCB-42-37	nd	1.7	6.2	nd	3.4	0.49	bql
PCB-64-61-71	4.6	8.1	10.3	nd	12.0	6.4	0.77
PCB-40	0.68	1.0	5.6	23.5	0.91	0.41	0.45
PCB-100	nd	nd	nd	nd	nd	nd	nd
PCB-63	nd	nd	nd	nd	nd	nd	nd
PCB-74	nd	6.9	22.1	nd	8.0	nd	nd
PCB-70-76	nd	nd	nd	nd	nd	nd	nd
PCB-66**	nd	nd	nd	nd	nd	nd	nd
PCB-66**	nd	nd	nd	nd	nd	nd	nd
PCB-91-121	1.6	8.8	26.1	61.1	8.0	3.6	1.4
PCB-60-66	nd	5.0	23.2	61.5	4.7	1.8	1.3
PCB-82-84	3.2	11.9	41.5	103.0	14.5	8.4	3.0
PCB-89	nd	0.44	0.48	nd	nd	nd	nd
PCB-101	int	int	int	int	int	int	int
PCB-99	2.7	14.9	34.4	84.3	11.7	6.5	2.6
PCB-119	(ocr)	0.27	0.95	2.5	4.8	0.91	0.57
PCB-83	nd	6.9	9.1	18.7	6.3	3.2	nd
PCB-97	2.0	9.8	24.4	67.4	6.4	6.5	2.1
PCB-87-81	2.9	13.8	32.8	86.4	11.8	8.7	2.8
PCB-85	nd	nd	nd	nd	nd	nd	nd
PCB-136**	nd	nd	nd	nd	nd	nd	nd
PCB-77*	nd	nd	nd	nd	nd	nd	nd
PCB-110**	int	int	int	int	int	int	int
PCB-82	0.82	2.6	6.4	17.1	1.9	0.84	0.59
PCB-181	2.1	8.7	18.4	28.1	4.2	1.9	0.95
PCB-136-144	2.9	11.9	18.6	38.1	6.4	nd	nd
PCB-107-123	(ocr)	0.83	2.5	6.3	1.6	0.60	0.20
PCB-149-118	8.3	39.1	38.5	63.4	10.1	8.32	nd
PCB-134-114	0.45	1.7	3.3	5.0	1.2	0.76	0.22
PCB-146**	3.16	18.7	21.2	28.6	7.4	4.0	1.8
PCB-106*	nd	nd	nd	nd	nd	nd	nd
PCB-132**	nd	nd	nd	nd	nd	2.6	1.1
PCB-183**	nd	nd	nd	nd	nd	6.7	3.0
PCB-141	0.61	nd	3.8	6.4	nd	nd	nd
PCB-137-176	nd	nd	nd	nd	nd	nd	nd
PCB-138-163	8.1	44.4	nd	63.6	18.6	nd	3.0
PCB-168	(ocr)	0.79	3.1	4.2	5.7	1.6	0.94
PCB-129	(ocr)	0.72	2.9	3.9	4.3	1.1	0.86
PCB-178-126	nd	3.9	3.6	4.2	1.3	0.62	0.21
PCB-176	nd	nd	nd	nd	nd	nd	nd
PCB-187-182**	8.9	28.6	38.1	28.0	7.4	3.6	1.4
PCB-183	nd	10.6	8.9	10.6	3.6	1.6	nd
PCB-128*	nd	nd	nd	nd	nd	nd	nd
PCB-185-167	0.39	2.4	2.2	2.6	0.71	0.24	0.14
PCB-174	2.8	11.9	8.7	8.8	2.9	1.2	0.69
PCB-177	1.2	8.3	6.1	6.0	2.3	0.86	0.28
PCB-166-171***	2.0	8.8	8.2	8.9	2.4	1.0	0.51
PCB-167-173-202***	0.63	3.3	nd	nd	nd	0.33	nd
PCB-200**	0.37	1.9	nd	1.7	0.43	0.18	nd
PCB-172**	0.36	2.5	2.0	1.6	0.62	0.25	0.17
PCB-197*	nd	nd	nd	nd	nd	nd	nd
PCB-180**	3.7	26.3	17.7	16.3	6.0	1.9	1.0
PCB-193	0.68	nd	2.0	nd	0.46	0.16	nd
PCB-191	nd	nd	nd	nd	nd	nd	nd
PCB-199	nd	1.7	1.1	nd	0.22	nd	nd
PCB-179**	nd	nd	nd	nd	nd	nd	nd
PCB-190**	nd	nd	nd	nd	nd	nd	nd
PCB-198	nd	nd	nd	nd	nd	nd	nd
PCB-201	3.1	10.6	8.5	9.7	1.9	1.3	0.51
PCB-203-196	2.1	11.9	8.3	7.3	2.0	1.3	0.47
PCB-189	(ocr)	0.07	0.58	0.41	0.31	0.09	0.03
PCB-196-206	nd	4.7	3.2	2.6	0.68	0.39	0.22
PCB-207	nd	0.76	0.38	nd	nd	0.84	nd
PCB-194	1.2	8.2	4.7	4.6	0.71	0.49	0.33
PCB-205	nd	1.0	nd	nd	nd	0.66	nd
PCB-206	nd	nd	nd	nd	nd	nd	nd
Total PCBs	91.7	423.4	672.4	1404.5	236.6	115.1	50.4

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.

- bolded values $V \leq 40\%$
- unbolded values $40\% < V \leq 80\%$
- nd not detected
- int interference on both signal responses
- bql below Limit of Quantitation

Sample ID	NRS201	NRS202	NRS203	NRS204	NRS205	NRS206	NRCRAY-L	NRCRAY-S
Sample wgt extracted (g)	4.05	5.01	5.17	5.81	5.80	5.72	5.37	2.13
Lipid wgt (g)	0.005	0.010	0.010	0.015	0.013	0.024	0.215	0.030
% lipid / dry wgt (g)	0.12	0.21	0.19	0.26	0.22	0.43	4.00	1.41
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
PCB-1	6.0	6.7	6.2	4.4	nd	6.0	6.7	10.4
PCB-3	nd	nd	nd	nd	nd	nd	nd	nd
PCB-4-10	nd	nd	nd	nd	nd	nd	1.8	nd
PCB-7	nd	0.07	0.10	0.04	0.04	0.10	nd	nd
PCB-6	nd	nd	nd	nd	nd	nd	nd	nd
PCB-8-5	0.18	0.67	0.91	0.40	0.60	0.67	nd	3.1
PCB-19	nd	nd	nd	nd	nd	nd	nd	1.4
PCB-12-13	nd	nd	nd	nd	nd	nd	nd	nd
PCB-18**	0.55	1.9	3.41	0.82	1.4	3.3	nd	nd
PCB-15*	nd	nd	nd	nd	nd	nd	nd	nd
PCB-17**	0.30	0.66	0.70	0.36	0.51	nd	0.19	nd
PCB-24-27	nd	nd	nd	nd	nd	nd	nd	nd
PCB-16**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-32**	0.64	1.1	nd	nd	nd	nd	nd	nd
PCB-64	nd	nd	nd	nd	nd	nd	nd	0.30
PCB-29 (ocr)	0.66	0.13	0.49	0.05	0.10	0.47	0.53	1.6
PCB-26 (ocr)	0.13	0.36	0.39	0.37	0.49	0.32	nd	nd
PCB-25	0.22	nd	nd	0.18	0.27	nd	nd	nd
PCB-31-28	1.4	4.0	4.6	4.0	4.9	2.9	4.8	4.1
PCB-33-63	0.62	0.28	nd	nd	2.3	nd	0.66	1.2
PCB-22-51	nd	0.45	nd	nd	0.83	nd	nd	nd
PCB-45	nd	0.66	nd	0.44	0.40	nd	0.36	1.2
PCB-46	nd	0.22	0.36	0.16	0.30	0.35	nd	nd
PCB-62	nd	int	int	int	int	int	9.6	18.6
PCB-49	nd	3.1	3.0	3.3	4.0	2.4	6.2	7.7
PCB-48**	nd	nd	0.43	nd	1.2	nd	6.9	5.1
PCB-47**	nd	nd	0.45	nd	1.5	nd	nd	nd
PCB-44	nd	0.90	0.86	1.9	5.4	0.63	3.0	4.8
PCB-42-37	nd	nd	nd	nd	nd	nd	nd	nd
PCB-64-41-71	nd	0.64	0.57	3.0	3.8	2.7	1.7	3.0
PCB-40	nd	nd	nd	nd	0.41	nd	0.09	nd
PCB-100	nd	nd	nd	nd	nd	nd	nd	nd
PCB-63	nd	nd	nd	nd	nd	nd	nd	nd
PCB-74	nd	nd	nd	nd	1.4	nd	3.2	3.5
PCB-70-76	nd	nd	nd	nd	4.3	nd	nd	nd
PCB-66**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-68**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-91-121	nd	1.2	1.3	1.3	1.6	1.0	1.3	3.6
PCB-60-66	0.47	1.1	0.74	0.90	1.2	nd	1.8	1.4
PCB-92-84	0.73	1.5	nd	1.4	2.8	nd	nd	4.4
PCB-89	nd	nd	nd	nd	nd	nd	nd	nd
PCB-101	int	int	int	int	int	int	int	int
PCB-99	nd	1.6	1.1	1.4	2.0	nd	6.0	7.9
PCB-119 (ocr)	0.07	0.08	0.07	0.12	0.14	0.06	0.41	0.47
PCB-83	nd	nd	nd	nd	nd	nd	nd	nd
PCB-97	0.62	1.1	1.1	1.4	1.6	1.0	2.3	4.1
PCB-97-81	0.91	1.6	1.4	1.8	2.2	1.3	3.6	6.2
PCB-86	nd	nd	nd	nd	nd	nd	nd	nd
PCB-136**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-77*	nd	nd	nd	nd	nd	nd	nd	nd
PCB-110**	int	int	int	int	int	int	int	int
PCB-82	0.31	0.41	nd	0.31	0.43	nd	0.32	0.68
PCB-151	0.46	0.67	0.83	0.83	0.77	0.71	2.0	1.8
PCB-136-144	nd	nd	nd	nd	nd	nd	2.7	nd
PCB-107-123 (ocr)	0.05	0.11	0.15	0.15	0.15	0.28	0.85	0.61
PCB-149-118	0.93	2.8	3.1	2.6	3.4	2.8	nd	5.10
PCB-134-114	0.08	0.10	nd	0.15	0.14	nd	0.46	0.48
PCB-146**	0.71	0.77	nd	0.70	0.77	nd	5.6	4.8
PCB-105*	nd	nd	nd	nd	nd	nd	nd	nd
PCB-132**	0.35	nd	nd	nd	nd	nd	nd	nd
PCB-153**	1.2	nd	nd	nd	nd	nd	nd	nd
PCB-141	nd	0.29	0.27	0.26	nd	0.24	nd	nd
PCB-137-176	nd	nd	nd	nd	nd	nd	nd	nd
PCB-136-163	1.3	1.8	1.6	2.0	1.9	1.8	12.6	9.9
PCB-168 (ocr)	0.14	0.16	0.17	0.17	0.16	0.18	0.99	0.81
PCB-129 (ocr)	0.11	0.11	0.10	0.11	0.14	0.11	0.88	0.69
PCB-178-126	0.11	nd	nd	nd	nd	nd	nd	1.1
PCB-178	nd	nd	nd	nd	nd	nd	nd	nd
PCB-187-182**	0.70	0.94	1.3	1.2	1.9	1.2	6.8	6.9
PCB-183	nd	0.40	0.76	0.47	0.36	0.47	2.8	1.8
PCB-125*	nd	nd	nd	nd	nd	nd	nd	nd
PCB-186-167	0.07	0.09	0.13	0.11	0.09	0.11	0.42	0.36
PCB-174	0.32	0.41	0.61	0.63	0.41	0.56	2.0	1.6
PCB-177	0.14	0.19	0.31	0.26	0.20	0.28	1.9	1.6
PCB-166-171***	0.25	0.30	0.41	0.37	0.29	0.42	2.7	1.6
PCB-187-173-202***	0.12	0.15	0.21	0.17	0.12	0.23	0.68	0.46
PCB-206**	0.07	0.11	0.23	0.13	0.05	0.22	0.32	0.25
PCB-172**	0.06	0.11	0.19	0.11	0.06	0.18	0.76	0.68
PCB-197*	nd	nd	nd	nd	nd	nd	nd	nd
PCB-180**	0.81	0.67	1.1	0.86	0.67	1.1	6.6	6.1
PCB-193	0.04	nd	nd	0.07	0.06	nd	nd	0.40
PCB-191	nd	nd	nd	nd	nd	nd	0.21	0.19
PCB-199	nd	0.06	0.09	0.06	nd	0.11	0.33	0.12
PCB-170**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-190**	nd	nd	nd	nd	nd	nd	nd	nd
PCB-198	nd	nd	nd	nd	nd	nd	nd	nd
PCB-201	0.36	0.42	nd	0.54	0.33	nd	2.3	2.3
PCB-203-196	0.27	0.32	0.63	0.47	0.29	0.50	1.7	1.4
PCB-189 (ocr)	nd	0.01	0.02	0.01	0.01	0.01	0.16	0.10
PCB-195-208	0.09	0.19	0.21	0.12	0.13	0.17	0.67	0.63
PCB-207	0.02	0.02	0.02	0.02	0.02	0.03	0.12	0.08
PCB-184	0.13	0.18	0.26	0.21	0.16	0.26	1.1	1.1
PCB-205	nd	0.02	0.03	nd	0.04	0.04	0.11	nd
PCB-206	nd	nd	nd	nd	nd	nd	nd	nd
Total PCBs	16.8	33.7	33.7	34.3	67.1	29.0	109.6	126.6

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.

- bolded value $V \leq 40\%$.
- unbolded value $40\% < V \leq 60\%$.
- nd not detected.
- int interference on both signal responses.
- bql below Limit of Quantitation.

Sample ID	NRSP4	NRSP6	NRSP8
Sample wgt extracted (g)	2.28	2.20	2.85
Lipid wgt (g)	0.123	0.150	0.085
% lipid / dry wgt (g)	5.42	6.81	2.98
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
PCB-1	nd	nd	nd
PCB-3	nd	nd	nd
PCB-4-10	nd	nd	nd
PCB-7	nd	nd	nd
PCB-6	nd	nd	nd
PCB-8-5	nd	nd	nd
PCB-19	nd	nd	nd
PCB-12-13	nd	nd	nd
PCB-16**	nd	nd	nd
PCB-16*	nd	nd	nd
PCB-17**	0.15	0.29	0.18
PCB-24-27	nd	nd	nd
PCB-16**	nd	nd	nd
PCB-32**	nd	nd	nd
PCB-64 (ocr)	nd	nd	nd
PCB-29 (ocr)	nd	nd	nd
PCB-26	nd	nd	nd
PCB-25	nd	nd	0.70 bql
PCB-31-38	6.0	6.7	2.3
PCB-33-63	nd	nd	nd
PCB-22-61	nd	nd	nd
PCB-46	0.13 bql	0.12 bql	0.39
PCB-46	nd	nd	nd
PCB-62	8.8	13.6	7.9
PCB-49	7.6	10.4	6.0
PCB-48**	5.4	7.3	3.8
PCB-47**	nd	nd	nd
PCB-44	6.6	6.7	1.4 bql
PCB-42-37	2.4	3.0	nd
PCB-64-61-71	6.9	7.4	nd
PCB-40	0.31	0.33	0.71
PCB-100	nd	nd	nd
PCB-63	nd	nd	nd
PCB-74	6.3	8.6	2.9
PCB-70-76	9.1	11.0	4.9
PCB-66**	nd	nd	nd
PCB-66**	nd	nd	nd
PCB-91-121	4.3	6.1	3.1
PCB-60-66	4.0	4.8	2.2
PCB-62-64	6.7	nd	4.7
PCB-89	nd	nd	nd
PCB-101	int	int	int
PCB-99	10.7	16.4	6.6
PCB-119 (ocr)	0.62	0.68	0.31
PCB-83	nd	7.0	3.9
PCB-97	6.0	9.6	4.2
PCB-97-61	9.7	12.7	6.8
PCB-66	nd	nd	nd
PCB-136**	nd	nd	nd
PCB-77*	nd	nd	nd
PCB-110**	int	int	int
PCB-82	2.0	2.6	1.1
PCB-161	6.8	7.0	3.6
PCB-136-144	nd	nd	nd
PCB-107-123 (ocr)	2.5	3.4	1.3
PCB-149-118	nd	20.9	11.1
PCB-134-114	1.4	1.7	0.8
PCB-146**	24.6	30.3	14.0
PCB-106*	nd	nd	nd
PCB-132**	7.7	8.8	4.1
PCB-153**	46.4	62.6	25.6
PCB-141	nd	nd	nd
PCB-137-176	nd	nd	nd
PCB-138-163	60.2	73.6	34.6
PCB-168 (ocr)	4.5	5.1	2.5
PCB-129 (ocr)	3.3	3.4	1.7
PCB-178-126	4.5	5.1	3.1
PCB-176	1.6	nd	nd
PCB-167-162**	27.6	36.6	16.0
PCB-163	16.6	19.7	8.6
PCB-128*	nd	nd	nd
PCB-166-167	2.0	2.6	1.1
PCB-174	7.9	9.8	6.1
PCB-177	7.8	9.4	6.0
PCB-166-171***	9.8	12.1	5.8
PCB-167-173-202**	2.9	3.2	1.6
PCB-200***	1.8	1.8	0.9
PCB-172**	3.9	4.6	2.1
PCB-197*	nd	nd	nd
PCB-180**	int	int	int
PCB-193	2.2	nd	nd
PCB-191	nd	nd	nd
PCB-199	1.0	1.2	0.41
PCB-170**	28.7	36.9	19.0
PCB-190**	3.8	4.9	2.6
PCB-196	nd	nd	nd
PCB-201	9.1	10.9	6.2
PCB-203-196	10.6	13.2	6.1
PCB-189 (ocr)	0.78	0.83	0.40
PCB-196-208	3.6	4.6	2.2
PCB-207	0.62	0.61	0.28
PCB-194	6.4	6.9	3.4
PCB-205	0.86	0.96	0.67
PCB-206	nd	nd	nd
Total PCBs	411.5	524.5	249.4

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column result confirmed by second column.
- ocr one column result, not identified by other column.

- bolded values $V \leq 40\%$.
- unbolded values $40\% < V \leq 80\%$.
- nd not detected.
- int interference on both signal responses.
- bql below Limit of Quantitation.

Sample ID	92JUL-1	92JUL-2	92JUL-3	92OCT-1	92OCT-2	92OCT-3
Sample wgt extracted (g)	0.73	0.50	0.61	0.86	0.71	0.64
Lipid wgt (g)	0.019	0.013	0.015	0.190	0.015	0.012
% lipid / dry wgt (g)	2.62	2.62	2.47	2.22	2.12	1.89
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
PCB-1	27.1 bql	nd	nd	30.0 bql	36.9 bql	nd
PCB-3	nd	nd	nd	nd	nd	nd
PCB-4-10	10.6 bql	nd	nd	10.8 bql	10.9	nd
PCB-7	nd	nd	nd	nd	nd	nd
PCB-6	nd	nd	nd	nd	nd	nd
PCB-8-5	1.5	nd	nd	nd	nd	nd
PCB-19	nd	nd	nd	nd	nd	nd
PCB-12-13	nd	nd	nd	nd	nd	nd
PCB-18 [•]	3.6	9.5 bql	12.2	nd	6.9	nd
PCB-18 ^{••}	nd	nd	nd	nd	nd	nd
PCB-17 ^{•••}	1.8	0.76 bql	5.3 bql	1.7 bql	nd	nd
PCB-24-27	nd	nd	nd	nd	nd	nd
PCB-16 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-32 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-54 (ocr)	18.2	20.6	17.1	13.0	17.7	nd
PCB-29 (ocr)	10.4	18.9	12.2	8.0	8.5	nd
PCB-26	0.98	2.2	nd	nd	nd	0.66
PCB-25	nd	1.2	nd	nd	nd	nd
PCB-31-38	15.6	13.1	nd	11.2	9.4	11.6
PCB-33-53	1.9	9.6 bql	8.4 bql	3.5	nd	4.2
PCB-22-51	nd	nd	nd	nd	nd	nd
PCB-45	1.1	1.9	7.1 bql	1.1	3.9 bql	nd
PCB-46	1.2	nd	nd	nd	nd	nd
PCB-52	24.2	22.6	8.2 bql	16.6	9.7	18.2
PCB-49	11.0	10.7	5.6 bql	7.3	5.9	7.6 bql
PCB-48 ^{•••}	nd	nd	nd	nd	nd	4.7 bql
PCB-47 ^{•••}	nd	nd	nd	nd	nd	3.6
PCB-44	nd	nd	12.7	nd	nd	21.0
PCB-42-37	4.7	nd	nd	nd	8.8	6.3 bql
PCB-64-41-71	14.6	12.7	13.2	8.1	20.3	7.6 bql
PCB-40	1.4	nd	nd	nd	nd	nd
PCB-100	nd	nd	nd	nd	nd	nd
PCB-63	nd	nd	nd	nd	nd	nd
PCB-74	9.8	9.0	nd	5.4	5.8	5.4
PCB-76-76	nd	nd	nd	nd	7.4	6.7
PCB-66 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-95 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-61-121	6.4	5.5	nd	nd	nd	nd
PCB-60-56	5.9	5.3	nd	2.3	nd	nd
PCB-92-84	10.3	7.2	nd	4.5	nd	6.6
PCB-89	0.78	0.26	nd	0.12	0.04	nd
PCB-101	int	int	int	int	int	int
PCB-99	9.5	9.7	7.1	5.1	5.4	6.2
PCB-119 (ocr)	0.94	1.0	0.81	0.88	0.54	0.89
PCB-83	nd	nd	nd	nd	nd	nd
PCB-67	6.5	6.7	3.9	4.0	nd	4.2
PCB-57-51	9.1	9.7	5.4	6.6	4.2	6.1
PCB-85	nd	nd	nd	nd	nd	nd
PCB-136 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-77 ^{••}	nd	nd	nd	nd	nd	nd
PCB-110 ^{••}	int	int	int	int	int	int
PCB-82	1.9	1.9	nd	nd	nd	nd
PCB-181	4.2	5.7	2.6	nd	nd	nd
PCB-135-144	6.6	5.2	8.2	2.9	3.1	3.9
PCB-167-123 (ocr)	0.16 bql	nd	nd	nd	nd	0.31
PCB-149-118	nd	nd	nd	8.9	7.8	19.2
PCB-134-114	1.1	1.1	nd	0.90	0.89	1.45
PCB-146 ^{••}	7.8	9.8	6.2	6.1	6.1	6.7
PCB-105 [•]	nd	nd	nd	nd	nd	nd
PCB-132 ^{••}	6.4	6.4	6.0	nd	1.9	4.0
PCB-153 ^{••}	27.2	20.7	22.6	nd	13.3	15.6
PCB-141	nd	nd	0.63	1.2	nd	nd
PCB-137-176	nd	nd	nd	nd	nd	nd
PCB-138-163	26.1	28.0	22.0	16.2	16.3	20.6
PCB-158 (ocr)	1.9	1.6	2.2	1.0	1.0	1.7
PCB-129 (ocr)	2.7	3.4	2.5	1.3	1.5	1.9
PCB-175-126	2.6	2.1	2.4	0.94	nd	nd
PCB-175	nd	nd	nd	nd	nd	nd
PCB-167-162 ^{••}	11.2	11.5	9.0	7.2	9.0	8.2
PCB-163	3.7	3.3	4.2	1.4	1.6	2.3
PCB-128 [•]	nd	nd	nd	nd	nd	nd
PCB-185-167	0.93	0.90	0.60	nd	nd	nd
PCB-174	6.4	5.6	3.4	4.1	3.8	4.4
PCB-177	2.7	3.5	2.1	2.3	2.2	2.8
PCB-166-171 ^{•••}	3.5	3.4	2.5	2.3	2.4	2.6
PCB-167-173-202 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-200 ^{•••}	nd	nd	nd	nd	nd	nd
PCB-172 ^{••}	1.3	1.2	0.58	0.67	0.67	1.6
PCB-197 [•]	nd	nd	nd	nd	nd	nd
PCB-160 ^{••}	7.94	10.6	6.5	6.7	8.6	8.3
PCB-183	0.87	0.88	0.49	0.46	0.63	0.70
PCB-191	nd	nd	nd	nd	nd	nd
PCB-199	nd	nd	nd	0.56	nd	nd
PCB-170 ^{••}	nd	nd	nd	nd	nd	nd
PCB-190 ^{••}	nd	nd	nd	nd	nd	nd
PCB-188	nd	nd	nd	nd	nd	nd
PCB-201	nd	3.1	nd	2.7	3.3	2.5
PCB-203-196	2.5	3.2	2.6	1.9	2.7	2.6
PCB-189 (ocr)	0.25	nd	0.23	0.37	nd	0.30
PCB-195-208	1.6	1.6	1.3	1.3	1.6	1.4
PCB-207	nd	0.00	nd	nd	nd	nd
PCB-194	1.5	2.3	nd	1.6	2.2	1.5
PCB-205	nd	nd	nd	nd	nd	nd
PCB-206	nd	nd	nd	nd	nd	nd
Total PCBs	303.7	283.0	189.4	169.0	212.1	213.3

Data qualifiers:

- determined by subtraction.
- single column result plus subtracted value.
- single column result confirmed by second column.
- ocr one column result, not identified by other column.

- boided values $V \leq 40\%$.
- unbolded values $40\% < V \leq 80\%$.
- nd not detected
- int interference on both signal responses.
- bql below Limit of Quantitation.

Appendix G. PAH data for Niagara River biota and sediment samples.

Appendix G. PAH data for Niagara River biota and sediment samples.



Wastewater Technology Centre
 Operated by Rockcliffe Research Management Inc.
 867 Lakeshore Rd., Burlington, Ontario L7R 4L7

WTC LABORATORY
 REPORT OF ANALYSIS

NWRI
 Attention: Janice Smith
 CCIW

WTC Group #: 4036
 Reported: 03/03/95

P.O.#:

WTC Sample #:		95-01836		95-01837		95-01838		95-01839	
Client Identification:		1 B (NR129L30)		2 B (NR129L30)		3 B (NR129L30)		5 B (NR129AMPB)	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
Naphthalene	ug/g	0.03t	0.1	0.03t	0.1	0.11	0.1	0.02t	0.1
Acenaphthylene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Acenaphthene	ug/g	w	0.1	0.01t	0.1	0.01t	0.1	0.03t	0.1
Fluorene	ug/g	0.02t	0.1	0.04t	0.1	0.04t	0.1	0.12	0.1
Phenanthrene	ug/g	0.08t	0.1	0.14	0.1	0.26	0.1	0.17	0.1
Anthracene	ug/g	0.02t	0.1	w	0.1	w	0.1	w	0.1
Fluoranthene	ug/g	0.04t	0.1	0.06t	0.1	0.23	0.1	0.02t	0.1
Pyrene	ug/g	0.05t	0.1	0.06t	0.1	0.25	0.1	0.03t	0.1
Benzo(a)anthracene	ug/g	0.03t	0.1	0.05t	0.1	0.20	0.1	0.02t	0.1
Chrysene	ug/g	0.02t	0.1	0.06t	0.1	0.21	0.1	0.02t	0.1
Benzo(b)fluoranthene	ug/g	0.08t	0.1	0.10	0.1	0.31	0.1	0.02t	0.1
Benzo(k)fluoranthene	ug/g	0.02t	0.1	0.03t	0.1	0.08t	0.1	0.01t	0.1
Benzo(a)pyrene	ug/g	0.02t	0.1	0.02t	0.1	w	0.1	w	0.1
Indeno(1,2,3-c,d)pyrene	ug/g	0.02t	0.1	0.02t	0.1	w	0.1	w	0.1
Dibenzo(a,h)anthracene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Benzo(g,h,i)perylene	ug/g	0.02t	0.1	0.02t	0.1	w	0.1	w	0.1

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
 Head, Mass Spectrometry



WTC LABORATORY
REPORT OF ANALYSIS

Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.
867 Lakeshore Rd., Burlington, Ontario L7R 4L7

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4036
Reported: 03/03/95

P.O.#:

WTC Sample #:		95-01840		95-01841		95-01842		95-01843	
Client Identification:		6 B (NR1295NA-1)		7 B (NR1295NA-2)		8 B (NR1295201)		9 B (NR1295202)	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
Naphthalene	ug/g	0.01t	0.1	0.02t	0.1	0.01t	0.1	0.01t	0.1
Acenaphthylene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Acenaphthene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Fluorene	ug/g	w	0.1	0.01t	0.1	0.01t	0.1	0.01t	0.1
Phenanthrene	ug/g	0.02t	0.1	0.04t	0.1	0.01t	0.1	0.02t	0.1
Anthracene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Fluoranthene	ug/g	0.01t	0.1	0.02t	0.1	0.01t	0.1	0.01t	0.1
Pyrene	ug/g	0.01t	0.1	0.02t	0.1	0.01t	0.1	0.01t	0.1
Benzo(a)anthracene	ug/g	w	0.1	0.01t	0.1	w	0.1	w	0.1
Chrysene	ug/g	0.01t	0.1	0.02t	0.1	w	0.1	0.01t	0.1
Benzo(b)fluoranthene	ug/g	0.01t	0.1	0.02t	0.1	0.01t	0.1	0.01t	0.1
Benzo(k)fluoranthene	ug/g	w	0.1	0.01t	0.1	w	0.1	w	0.1
Benzo(a)pyrene	ug/g	w	0.1	0.01t	0.1	w	0.1	w	0.1
Indeno(1,2,3-c,d)pyrene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Dibenzo(a,h)anthracene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1
Benzo(g,h,i)perylene	ug/g	w	0.1	w	0.1	w	0.1	w	0.1

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
Head, Mass Spectrometry



Wastewater Technology Centre
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867 Lakeshore Rd., Burlington, Ontario L7R 4L7

WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4036
Reported: 03/03/95

P.O.#:

WTC Sample #:		95-01844	95-01845		95-01846	95-01847	
Client Identification:		108 (NR1295203)	148 (NR129CRAY-L)		158 (NR129CRAY-L)	168 (NR-SP-4)	
Date Received:		02/16/95	02/16/95		02/16/95	02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL
Naphthalene	ug/g	0.01t	0.1	0.01t	0.1	0.03t	0.1
Acenaphthylene	ug/g	w	0.1	w	0.1	w	0.1
Acenaphthene	ug/g	0.01t	0.1	w	0.1	w	0.1
Fluorene	ug/g	w	0.1	0.01t	0.1	0.04t	0.1
Phenanthrene	ug/g	0.02t	0.1	0.03t	0.1	0.06t	0.1
Anthracene	ug/g	w	0.1	w	0.1	w	0.1
Fluoranthene	ug/g	0.01t	0.1	0.01t	0.1	0.01t	0.1
Pyrene	ug/g	0.01t	0.1	0.01t	0.1	0.02t	0.1
Benzo(a)anthracene	ug/g	0.01t	0.1	0.01t	0.1	0.01t	0.1
Chrysene	ug/g	w	0.1	w	0.1	w	0.1
Benzo(b)fluoranthene	ug/g	0.01t	0.1	0.01t	0.1	0.01t	0.1
Benzo(k)fluoranthene	ug/g	w	0.1	w	0.1	w	0.1
Benzo(a)pyrene	ug/g	w	0.1	w	0.1	w	0.1
Indeno(1,2,3-c,d)pyrene	ug/g	w	0.1	w	0.1	w	0.1
Dibenzo(a,h)anthracene	ug/g	w	0.1	w	0.1	w	0.1
Benzo(g,h,i)perylene	ug/g	w	0.1	w	0.1	w	0.1

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
Head, Mass Spectrometry



Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.
867 Lakeshore Rd., Burlington, Ontario L7R 4L7

WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4036
Reported: 03/03/95

P.O.#:

WTC Sample #:		95-01848		95-01849		95-01850		95-01851	
Client Identification:		17B (NR-SP-5)		18B (NR-SP-4)		92JUL-18		92JUL-28	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
Naphthalene	ug/g	0.01t	0.1	0.01t	0.1	0.03t	0.3	0.06t	0.3
Acenaphthylene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3
Acenaphthene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3
Fluorene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3
Phenanthrene	ug/g	0.01t	0.1	0.01t	0.1	0.03t	0.3	0.31	0.3
Anthracene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3
Fluoranthene	ug/g	0.01t	0.1	w	0.1	0.01t	0.2	0.05t	0.2
Pyrene	ug/g	0.01t	0.1	w	0.1	0.01t	0.3	0.10t	0.3
Benzo(a)anthracene	ug/g	w	0.1	w	0.1	0.02t	0.3	0.14t	0.3
Chrysene	ug/g	w	0.1	w	0.1	0.01t	0.1	0.06t	0.1
Benzo(b)fluoranthene	ug/g	w	0.1	w	0.1	w	0.3	0.03t	0.3
Benzo(k)fluoranthene	ug/g	w	0.1	w	0.1	w	0.1	0.01t	0.1
Benzo(a)pyrene	ug/g	w	0.1	w	0.1	w	0.2	w	0.2
Indeno(1,2,3-c,d)pyrene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3
Dibenzo(a,h)anthracene	ug/g	w	0.1	w	0.1	w	0.2	w	0.2
Benzo(g,h,i)perylene	ug/g	w	0.1	w	0.1	w	0.3	w	0.3

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
Head, Mass Spectrometry



Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.
867 Lakeshore Rd., Burlington, Ontario L7R 4L7

WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4036
Reported: 03/03/95

P.O.#:

Parameter	WTC Sample #:		95-01852		95-01853		95-01854		95-01855	
	Client Identification:		92JUL-38		92OCT-18		92OCT-28		92 OCT-38	
	Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
	Units		Result	MDL	Result	MDL	Result	MDL	Result	MDL
Naphthalene	ug/g		0.06t	0.3	0.04t	0.3	0.04t	0.3	0.06t	0.3
Acenaphthylene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3
Acenaphthene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3
Fluorene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3
Phenanthrene	ug/g		0.26t	0.3	0.20t	0.3	0.26t	0.3	0.29t	0.3
Anthracene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3
Fluoranthene	ug/g		0.05t	0.2	0.04t	0.2	0.07t	0.2	0.06t	0.2
Pyrene	ug/g		0.09t	0.3	0.08t	0.3	0.15t	0.3	0.14t	0.3
Benzo(a)anthracene	ug/g		0.09t	0.3	0.07t	0.3	0.10t	0.3	0.09t	0.3
Chrysene	ug/g		0.05t	0.1	0.03t	0.1	0.07t	0.1	0.05t	0.1
Benzo(b)fluoranthene	ug/g		0.04t	0.3	0.04t	0.3	0.06t	0.3	0.03t	0.3
Benzo(k)fluoranthene	ug/g		0.01t	0.1	0.01t	0.1	0.03t	0.1	0.02t	0.1
Benzo(a)pyrene	ug/g	w		0.2	w	0.2	w	0.2	w	0.2
Indeno(1,2,3-c,d)pyrene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3
Dibenzo(a,h)anthracene	ug/g	w		0.2	w	0.2	w	0.2	w	0.2
Benzo(g,h,i)perylene	ug/g	w		0.3	w	0.3	w	0.3	w	0.3

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
Head, Mass Spectrometry



Wastewater Technology Centre
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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4036
Reported: 03/03/95

P.O.#:

WTC Sample #:		95-01856		95-01857		95-01858		
Client Identification:		92SED-338		92SED-348		92SED-358		
Date Received:		02/16/95		02/16/95		02/16/95		
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	
Naphthalene	ug/g	0.01t	0.01	0.01	0.01	0.01	0.01	
Acenaphthylene	ug/g	w	0.01	w	0.01	w	0.01	
Acenaphthene	ug/g	w	0.01	w	0.01	w	0.01	
Fluorene	ug/g	w	0.01	w	0.01	0.01	0.01	
Phenanthrene	ug/g	0.04	0.01	0.03	0.01	0.06	0.01	
Anthracene	ug/g	w	0.01	w	0.01	w	0.01	
Fluoranthene	ug/g	0.07	0.01	0.04	0.01	0.07	0.01	
Pyrene	ug/g	0.06	0.01	0.03	0.01	0.06	0.01	
Benzo(a)anthracene	ug/g	0.08	0.01	0.03	0.01	0.07	0.01	
Chrysene	ug/g	0.06	0.01	0.02	0.01	0.05	0.01	
Benzo(b)fluoranthene	ug/g	0.15	0.01	0.10	0.01	0.13	0.01	
Benzo(k)fluoranthene	ug/g	0.04	0.01	0.05	0.01	0.05	0.01	
Benzo(a)pyrene	ug/g	0.08	0.01	0.02	0.01	0.08	0.01	
Indeno(1,2,3-c,d)pyrene	ug/g	0.06	0.01	0.03	0.01	0.08	0.01	
Dibenzo(a,h)anthracene	ug/g	w	0.01	w	0.01	w	0.01	
Benzo(g,h,i)perylene	ug/g	0.08	0.01	0.04	0.01	0.09	0.01	

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Brian MacGillivray
Head, Mass Spectrometry

NWRI

WTC Group #: 4036

QC Type	Parameter	Matrix	Actual	Found	Percent
DUPLICATE	Acenaphthene	SOLID	0.01t	w	
	Acenaphthene	SOLID	w	w	
	Acenaphthene	SOLID	w	w	
	Acenaphthene	SOLID	w	w	
	Acenaphthene	SOLID	w	w	
	Acenaphthylene	SOLID	w	w	
	Acenaphthylene	SOLID	w	w	
	Acenaphthylene	SOLID	w	w	
	Acenaphthylene	SOLID	w	w	
	Acenaphthylene	SOLID	w	w	
	Anthracene	SOLID	w	w	
	Anthracene	SOLID	w	w	
	Anthracene	SOLID	w	w	
	Anthracene	SOLID	w	w	
	Anthracene	SOLID	w	w	
	Benzo (a) pyrene	SOLID	0.02	0.02	0.0
	Benzo (a) pyrene	SOLID	w	w	
	Benzo (a) pyrene	SOLID	w	w	
	Benzo (a) pyrene	SOLID	w	w	
	Benzo (a) pyrene	SOLID	w	w	
	Benzo (a) anthracene	SOLID	0.03	0.04	28.6
	Benzo (a) anthracene	SOLID	0.09t	0.09t	
	Benzo (a) anthracene	SOLID	0.09t	0.11t	
	Benzo (a) anthracene	SOLID	0.20	0.20	0.0
	Benzo (a) anthracene	SOLID	w	w	
	Benzo (b) fluoranthene	SOLID	0.03t	0.05t	
	Benzo (b) fluoranthene	SOLID	0.04t	0.04t	
	Benzo (b) fluoranthene	SOLID	0.10	0.06	50.0
	Benzo (b) fluoranthene	SOLID	0.31	0.29	6.7
	Benzo (b) fluoranthene	SOLID	w	w	
	Benzo (g,h,i) perylene	SOLID	0.04	0.04	0.0
	Benzo (g,h,i) perylene	SOLID	w	w	
	Benzo (g,h,i) perylene	SOLID	w	w	
	Benzo (g,h,i) perylene	SOLID	w	w	
	Benzo (g,h,i) perylene	SOLID	w	w	
	Benzo (k) fluoranthene	SOLID	0.01t	0.02t	
	Benzo (k) fluoranthene	SOLID	0.02t	0.03t	
	Benzo (k) fluoranthene	SOLID	0.05	0.03	50.0
	Benzo (k) fluoranthene	SOLID	0.08	0.08	0.0
	Benzo (k) fluoranthene	SOLID	w	w	
	Chrysene	SOLID	0.02	0.02	0.0
	Chrysene	SOLID	0.05t	0.05t	
	Chrysene	SOLID	0.05t	0.07t	
	Chrysene	SOLID	0.21	0.20	4.9
	Chrysene	SOLID	w	w	
	Dibenzo (a,h) anthracene	SOLID	w	w	
	Dibenzo (a,h) anthracene	SOLID	w	w	
	Dibenzo (a,h) anthracene	SOLID	w	w	
	Dibenzo (a,h) anthracene	SOLID	w	w	
	Dibenzo (a,h) anthracene	SOLID	w	w	
	Fluoranthene	SOLID	0.04	0.03	28.6
	Fluoranthene	SOLID	0.05t	0.05t	
	Fluoranthene	SOLID	0.06t	0.06t	
	Fluoranthene	SOLID	0.23	0.22	4.4
	Fluoranthene	SOLID	w	w	
	Fluorene	SOLID	0.04t	0.04t	
	Fluorene	SOLID	w	w	
	Fluorene	SOLID	w	w	
	Fluorene	SOLID	w	w	
	Fluorene	SOLID	w	w	
	Indeno (1,2,3-c,d) pyrene	SOLID	0.03	0.03	0.0
	Indeno (1,2,3-c,d) pyrene	SOLID	w	w	
	Indeno (1,2,3-c,d) pyrene	SOLID	w	w	
	Indeno (1,2,3-c,d) pyrene	SOLID	w	w	
	Indeno (1,2,3-c,d) pyrene	SOLID	w	w	
	Naphthalene	SOLID	0.01	0.01	0.0
	Naphthalene	SOLID	0.01t	0.01t	
	Naphthalene	SOLID	0.06t	0.06t	
	Naphthalene	SOLID	0.06t	0.07t	
	Naphthalene	SOLID	0.11	0.12	8.7
	Phenanthrene	SOLID	0.01t	0.01t	
	Phenanthrene	SOLID	0.03	0.03	0.0
	Phenanthrene	SOLID	0.26	0.25	3.9
	Phenanthrene	SOLID	0.26	0.26	0.0
	Phenanthrene	SOLID	0.29	0.34	15.9
	Pyrene	SOLID	0.03	0.03	0.0
	Pyrene	SOLID	0.09t	0.09t	
	Pyrene	SOLID	0.14t	0.14t	
	Pyrene	SOLID	0.25	0.25	0.0
	Pyrene	SOLID	w	w	

METH-BLANK: A blank processed as a sample.

DUPLICATE: Two identical portions of sample processed separately.

REFERENCE: A sample with a known concentration of analyte.

SPIKE: A sample with a known addition of analyte.

SPIKE-BLK: A blank with a known addition of analyte.

Spikes and references are expressed as % recovery of target values.

Appendix H. Data on di- and tri-chlorobenzenes, hexachlorobutadiene and hexachloroethane in Niagara River biota and sediment samples.

Appendix H. Data on di- and tri-chlorobenzenes, hexachlorobutadiene and hexachloroethane in Niagara River biota and sediment samples.



Wastewater Technology Centre
 Operated by Rockcliffe Research Management Inc.
 867 Lakeshore Rd., Burlington, Ontario L7R 4L7

WTC LABORATORY
 REPORT OF ANALYSIS

NWRI
 Attention: Janice Smith
 CCIW

WTC Group #: 4037
 Reported: 03/14/95

P.O.#:

WTC Sample #:		95-01859		95-01860		95-01861		95-01862	
Client Identification:		1 A (NR129 LZO)		2 A (NR129 LQUH)		3 A (NR129 OUF)		5 A (NR129 AMPH)	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,2,4-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,3,5-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,2,3,4-tetrachlorobenzene	ng/g	W	2	W	2	W	2	1.09t	2
1,2,4,5-tetrachlorobenzene	ng/g	W	2	W	2	W	2	W	2
Hexachlorobutadiene	ng/g	W	1.5	W	1.5	W	1.5	W	1.5
Hexachloroethane	ug/g	W	1.2	W	1.2	W	1.2	W	1.2

t: Constituent detected but at less than the MDL.
 w: Constituent not detected.
 n/a: Not available.
 MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You
 Head, Chromatography Section



Wastewater Technology Centre
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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4037
Reported: 03/14/95

P.O.#:

WTC Sample #:		95-01863	95-01864	95-01865	95-01866
Client Identification:		6 A (NR1295NA-1)	7 A (NR1295NA-2)	8 A (NR1295Z01)	9 A (NR1295Z02)
Date Received:		02/16/95	02/16/95	02/16/95	02/16/95
Parameter	Units	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	W	2	W	2
1,2,4-trichlorobenzene	ng/g	W	2	W	2
1,3,5-trichlorobenzene	ng/g	W	2	W	2
1,2,3,4-tetrachlorobenzene	ng/g	W	2	W	2
1,2,4,5-tetrachlorobenzene	ng/g	W	2	W	2
Hexachlorobutadiene	ng/g	W	1.5	W	1.5
Hexachloroethane	ug/g	W	1.2	W	1.2

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You
Head, Chromatography Section



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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4037
Reported: 03/14/95

P.O.#:

WTC Sample #:		95-01867		95-01868		95-01869		95-01870	
Client Identification:		10A (NR1295202)		14A (NR129000V-S)		15A (NR129000V-L)		16A (NR-SP-4)	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,2,4-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,3,5-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,2,3,4-tetrachlorobenzene	ng/g	w	2	w	2	0.54t	2	w	2
1,2,4,5-tetrachlorobenzene	ng/g	w	2	w	2	w	2	w	2
Hexachlorobutadiene	ng/g	w	1.5	w	1.5	w	1.5	w	1.5
Hexachloroethane	ug/g	w	1.2	w	1.2	w	1.2	w	1.2

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You

Head, Chromatography Section



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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4037
Reported: 03/14/95

P.O.#:

WTC Sample #:		95-01871		95-01872		95-01873		95-01874	
Client Identification:		17A (NR-SP-5)		18A (NR-SP-6)		92 JUL-1A		92 JUL-2A	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,2,4-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,3,5-trichlorobenzene	ng/g	W	2	W	2	W	2	W	2
1,2,3,4-tetrachlorobenzene	ng/g	0.44t	2	W	2	W	2	W	2
1,2,4,5-tetrachlorobenzene	ng/g	W	2	W	2	W	2	W	2
Hexachlorobutadiene	ng/g	W	1.5	W	1.5	W	1.5	W	1.5
Hexachloroethane	ug/g	W	1.2	W	1.2	W	1.2	W	1.2

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You
Head, Chromatography Section



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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4037
Reported: 03/14/95

P.O.#:

WTC Sample #:		95-01875		95-01876		95-01877		95-01878	
Client Identification:		92 JUL-3A		92-OCT-1A		92-OCT-2A		92-OCT-3A	
Date Received:		02/16/95		02/16/95		02/16/95		02/16/95	
Parameter	Units	Result	MDL	Result	MDL	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,2,4-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,3,5-trichlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,2,3,4-tetrachlorobenzene	ng/g	w	2	w	2	w	2	w	2
1,2,4,5-tetrachlorobenzene	ng/g	w	2	w	2	w	2	w	2
Hexachlorobutadiene	ng/g	w	1.5	w	1.5	w	1.5	w	1.5
Hexachloroethane	ug/g	w	1.2	w	1.2	w	1.2	w	1.2

t: Constituent detected but at less than the MDL.

w: Constituent not detected.

n/a: Not available.

MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You
Head, Chromatography Section



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WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 4037
Reported: 03/14/95

P.O.#:

WTC Sample #: 95-01879		95-01880		95-01881			
Client Identification: SED33-A		SED34-A		SED35-A			
Data Received: 02/16/95		02/16/95		02/16/95			
Parameter	Units	Result	MDL	Result	MDL	Result	MDL
1,2,3-trichlorobenzene	ng/g	W	2	W	2	W	2
1,2,4-trichlorobenzene	ng/g	W	2	W	2	W	2
1,3,5-trichlorobenzene	ng/g	W	2	W	2	W	2
1,2,3,4-tetrachlorobenzene	ng/g	0.54t	2	0.28t	2	0.28t	2
1,2,4,5-tetrachlorobenzene	ng/g	0.33t	2	0.33t	2	0.35t	2
Hexachlorobutadiene	ng/g	W	1.5	W	1.5	W	1.5
Hexachloroethane	ug/g	W	1.2	W	1.2	W	1.2

t: Constituent detected but at less than the MDL.

W: Constituent not detected.

n/a: Not available.

MDL: Method Detection Limit as defined for the Ontario MISA Program.

Analyzed by:

Validated by:

Robert Hong-You
Head, Chromatography Section

NWRI

WTC Group #: 4037

QC Type	Parameter	Matrix	Actual	Found	Percent
DUPLICATE	1,2,3-trichlorobenzene	SOLID	W	W	
	1,2,4-trichlorobenzene	SOLID	W	W	
	1,3,5-trichlorobenzene	SOLID	W	W	
	1,2,3,4-tetrachlorobenzene	SOLID	W	W	
	1,2,4,5-tetrachlorobenzene	SOLID	W	W	
	Hexachlorobutadiene	SOLID	W	W	
	Hexachloroethane	SOLID	W	W	
METH-BLANK	1,2,3-trichlorobenzene	SOLID	W	W	
	1,2,4-trichlorobenzene	SOLID	W	W	
	1,3,5-trichlorobenzene	SOLID	W	W	
	1,2,3,4-tetrachlorobenzene	SOLID	W	W	
	1,2,4,5-tetrachlorobenzene	SOLID	W	W	
	Hexachlorobutadiene	SOLID	W	W	
	Hexachloroethane	SOLID	W	W	

METH-BLANK: A blank processed as a sample.

DUPLICATE: Two identical portions of sample processed separately.

REFERENCE: A sample with a known concentration of analyte.

SPIKE: A sample with a known addition of analyte.

SPIKE-BLK: A blank with a known addition of analyte.

Spikes and references are expressed as % recovery of target values.

Duplicates are expressed in % difference between duplicate values as a ratio to their average.

Appendix H. Congener-specific PCB data for Niagara River sediment samples.
Total PCBs calculated without bql values.

Sample ID	93SED-33	93SED-34	93SED-35
Sample wgt extracted (g)	11.48	11.56	10.94
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
PCB-1	1.9 bql	nd	2.4
PCB-3	nd	nd	nd
PCB-4-10	nd	nd	1.2
PCB-7	nd	nd	0.13
PCB-8	nd	nd	0.29
PCB-8-5	0.34	0.27	0.35
PCB-19	nd	nd	nd
PCB-12-13	0.14	0.16 bql	0.08
PCB-18**	0.56	0.77	1.3
PCB-15*	nd	nd	nd
PCB-17**	0.30	0.28	0.48
PCB-24-27	nd	nd	nd
PCB-18**	nd	nd	nd
PCB-32**	0.78	0.70	1.0
PCB-54 (ocr)	0.14	0.12	0.13
PCB-29 (ocr)	0.40	0.37	0.85
PCB-26	nd	nd	nd
PCB-25	nd	nd	nd
PCB-31-28	1.3	2.7	5.0
PCB-33-53	0.39	1.1	1.6
PCB-22-51	0.31	0.25	0.50
PCB-45	0.13	0.09	0.21
PCB-46	nd	0.60	0.14
PCB-52	2.3	4.1	6.0
PCB-49	1.6	2.2	3.2
PCB-48**	0.94	1.3	1.8
PCB-47**	0.43	0.55	0.79
PCB-44	1.3	1.6	2.9
PCB-42-37	0.76	0.75	1.2
PCB-64-41-71	1.9	2.1	3.4
PCB-40	nd	nd	0.75
PCB-100	nd	0.06	0.28
PCB-63	nd	nd	nd
PCB-74	1.4	1.4	1.9
PCB-70-76	nd	2.4	3.2
PCB-66**	nd	nd	nd
PCB-65**	nd	nd	nd
PCB-61-121	1.0	1.0	1.6
PCB-60-56	1.1	0.88	1.4
PCB-62-84	0.67	1.4	1.8
PCB-59	nd	nd	nd
PCB-101	int	int	int
PCB-99	1.1	1.2	1.6
PCB-119 (ocr)	0.13	0.13	0.17
PCB-83	0.84	0.63	1.0
PCB-97	0.78	0.76	1.2
PCB-87-81	0.88	1.0	1.4
PCB-85	nd	nd	nd
PCB-136**	int	int	int
PCB-77*	int	int	int
PCB-110**	int	int	int
PCB-82	nd	nd	nd
PCB-151	nd	nd	nd
PCB-135-144	0.24	0.45	0.42
PCB-107-123 (ocr)	0.17	0.09	0.12
PCB-149-118	nd	nd	nd
PCB-134-114	0.18	0.17	0.19
PCB-146**	0.33	0.60	0.60
PCB-105*	nd	nd	nd
PCB-132**	nd	0.46	nd
PCB-153**	nd	1.6	1.3
PCB-141	nd	0.16	0.07
PCB-137-176	nd	nd	nd
PCB-138-163	1.1	1.9	0.88
PCB-158	0.23	0.28	0.19
PCB-129 (ocr)	0.41	0.25	0.29
PCB-178-126	nd	0.19	0.10
PCB-175	nd	nd	nd
PCB-157-182**	0.67	0.90	0.31
PCB-183	nd	0.36	0.11
PCB-128*	nd	nd	nd
PCB-185-167	nd	nd	nd
PCB-174	nd	0.66	0.16
PCB-177	0.19	0.39	nd
PCB-156-171***	0.22	0.44	0.17
PCB-157-173-202***	nd	nd	nd
PCB-200***	nd	nd	nd
PCB-172**	nd	0.20	0.07
PCB-197*	nd	nd	nd
PCB-169**	0.37	1.2	0.27
PCB-183	nd	0.11	0.06
PCB-191	nd	nd	nd
PCB-199	nd	nd	nd
PCB-170**	nd	nd	nd
PCB-190**	nd	nd	nd
PCB-198	nd	nd	nd
PCB-201	0.14	0.42	0.15
PCB-203-196	0.29	0.49	nd
PCB-189 (ocr)	0.02	0.11	0.08
PCB-195-208	0.09	0.23	0.23
PCB-207	nd	nd	0.02
PCB-194	0.09	0.25	0.13
PCB-205	0.02 bql	0.04	nd
PCB-206	nd	nd	nd
Total PCBs	26.6	42.5	56.6

Data qualifiers:

- * determined by subtraction.
- ** single column result plus subtracted value.
- *** single column response confirmed by second column.
- ocr one column result, not identified by other column.

- bolded values $V \leq 40\%$.
- unbolded values $40\% < V \leq 80\%$.
- nd not detected.
- int interference on both signal responses.
- bql below Limit of Quantitation.

Appendix I2. Organochlorine pesticides and industrial organic compounds data for Niagara River sediment samples.

Sample ID	93SED-33	93SED-34	93SED-35
Sample wgt extracted (g)	11.48	11.56	10.94
Concentration	ng/g dry wgt	ng/g dry wgt	ng/g dry wgt
a-BHC	nd	0.1	nd
b-BHC	nd	nd	nd
g-BHC	0.07	nd	0.09
Heptachlor	nd	nd	nd
Heptachlor epoxide	nd	nd	nd
g-Chlordane	nd	nd	nd
a-Endosulfan	nd	nd	nd
a-Chlordane	nd	nd	nd
Dieldrin	nd	nd	nd
p,p-DDE	nd	0.49	0.48
o,p-DDD	nd	nd	nd
Endrin	nd	0.39	0.47
b-Endosulfan	no data*	no data*	no data*
p,p-DDD	1.1	0.58	nd
o,p-DDT	nd	nd	nd
p,p-DDT	2.6	1.7	nd
PECB	1.2	0.64	0.69
HCB	2.2	1.4	2.1
Aldrin	nd	nd	nd
o,p-DDE	nd	nd	nd
Mirex	nd	nd	1.5
OCS	nd	nd	nd

Data qualifiers:

- * compound did not elute from silica gel cleanup; see text.
- nd not detected
- bolded values $V \leq 40\%$.
- unbolded values $40\% < V \leq 80\%$.

Appendix J. PCB Congener - Class Distributions in Niagara River Samples.
(as percent of total PCBs- bql values not included)

Congener Class	NRLZO	NRQUAG	NROLIG	NRMIDGES	NRAMPH	NRSNA-1	NRSNA-2
Mono	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Di	1.00	0.00	0.00	1.00	1.00	0.00	0.00
Tri	13.00	7.00	15.00	5.00	11.00	6.00	18.00
Tetra	16.00	11.00	24.00	39.00	28.00	25.00	25.00
Penta	19.00	21.00	29.00	33.00	28.00	36.00	25.00
Hexa	27.00	28.00	14.00	14.00	19.00	20.00	21.00
Hepta	17.00	23.00	13.00	6.00	11.00	10.00	8.00
Octa	8.00	9.00	4.00	2.00	2.00	3.00	3.00
Nona	0.00	0.00	0.00	0.00	0.00	0.00	0.00

Congener Class	NRSZ01	NRSZ02	NRSZ03	NRSZ04	NRSZ05	NRSZ06
Mono	0.00	0.00	0.00	0.00	0.00	0.00
Di	1.00	2.00	3.00	1.00	1.00	3.00
Tri	24.00	24.00	27.00	16.00	17.00	22.00
Tetra	4.00	18.00	18.00	27.00	41.00	22.00
Penta	19.00	25.00	19.00	24.00	21.00	16.00
Hexa	32.00	17.00	16.00	16.00	11.00	18.00
Hepta	14.00	9.00	14.00	10.00	5.00	14.00
Octa	6.00	4.00	4.00	4.00	2.00	4.00
Nona	0.00	0.00	0.00	0.00	0.00	0.00

Congener Class	NRCRAY-L	NRCRAY-S	NRSP4	NRSP5	NRSP6
Mono	0.00	0.00	0.00	0.00	0.00
Di	0.00	0.00	0.00	0.00	0.00
Tri	5.00	3.00	1.00	1.00	1.00
Tetra	30.00	35.00	14.00	14.00	12.00
Penta	14.00	23.00	11.00	13.00	14.00
Hexa	24.00	18.00	39.00	39.00	39.00
Hepta	21.00	16.00	26.00	25.00	25.00
Octa	6.00	5.00	9.00	8.00	8.00
Nona	0.00	0.00	0.00	0.00	1.00

Congener Class	92JUL-1	92JUL-2	92JUL-3	92OCT-1	92OCT-2	92OCT-3
Mono	0.00	0.00	0.00	0.00	0.00	0.00
Di	0.00	0.00	0.00	0.00	5.00	0.00
Tri	11.00	12.00	13.00	13.00	12.00	8.00
Tetra	30.00	29.00	23.00	32.00	35.00	27.00
Penta	15.00	14.00	9.00	14.00	6.00	15.00
Hexa	28.00	28.00	37.00	20.00	23.00	33.00
Hepta	13.00	14.00	16.00	15.00	13.00	14.00
Octa	2.00	3.00	2.00	5.00	5.00	4.00
Nona	0.00	0.00	0.00	0.00	0.00	0.00

Congener Class	93SED-33	93SED-34	93SED-35
Mono	0.00	0.00	4.00
Di	2.00	1.00	4.00
Tri	15.00	14.00	19.00
Tetra	45.00	43.00	48.00
Penta	22.00	15.00	16.00
Hexa	9.00	14.00	7.00
Hepta	5.00	10.00	2.00
Octa	2.00	3.00	1.00
Nona	0.00	0.00	0.00

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