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Toxicity of Sediments near an Aluminum Production Plant on the St. Lawrence River to Freshwater Organisms, with Emphasis on Fluoride Part II: Phase II TIE, Effects of Simulated Dredging Conditions on Survival, Growth and MFO Induction in Fish, and Assessment of the Toxicity of Fluoride to Freshwater Benthic Organisms

Metcalfe-Smith, J.L., K.E. Holtze, P.V. Hodson, L.J. Novak, J.J. Reid and S.R. de Solla

NWRI Contribution No. 01-320

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Part II: Phase II TIE, effects of simulated dredging conditions on survival, growth and MFO induction in fish, and assessment of the toxicity of fluoride to freshwater benthic organisms

J.L. Metcalfe-Smith[1], K.E. Holtze[2], P.V. Hodson[3], L.J. Novak[2], J.J. Reid[2] and S.R. de Solla[4]

[1]Aquatic Ecosystem Impacts Research Branch
National Water Research Institute
P.O. Box 5050, 867 Lakeshore Road
Burlington, Ontario L7R 4A6

[2]ESG International
Nicholas Beaver Park
R.R. 3, Guelph, Ontario, N1H 6H9

[3]Queen's University
School of Environmental Studies, Department of Biology
Kingston, Ontario K7L 3N6

[4]158 Herkimer Street
Hamilton, Ontario L8P 2H4

NWRI Contribution No. 01-320

Management Perspective

The Reynolds Metals Company (RMC) aluminum production plant at Massena, NY, is located within the St. Lawrence River Area of Concern (AOC). This study represents Part II of a two-part study to determine the toxicity of sediments in the vicinity of the plant to benthic organisms, and to assess the risk to the Canadian environment of dredging activities proposed for this site under a Superfund administrative order. Approximately 51,500 cu yds of sediment contaminated with PCBs, PAHs, cyanide, fluoride, aluminum and dibenzofurans, among other substances, are scheduled to be dredged from this site in the summer of 2001. The impetus behind our study was two-fold: (i) Inorganic fluorides were declared toxic under the Canadian Environmental Protection Act (CEPA) in 1993; however, the Priority Substances List Assessment Report on inorganic fluorides recommended the acquisition and evaluation of additional data on "the relationship between the levels of fluoride in sediment and toxicity to benthic organisms (in areas of Canada where high levels of inorganic fluorides in sediments are known or expected to occur)." (ii) The Canadian Review Panel for Massena Superfund Sites, of which the senior author is a member, is responsible for assessing the risk of Superfund-ordered remediation activities to the Canadian environment. As the plant is located across the river from Cornwall, Ontario, and only a few kilometres upstream of the U.S.A./Quebec border, there is potential for such activities to impact aquatic communities in Canadian waters.

In Part I of this study, we determined that nearly 40% of the sediment to be dredged from the RMC Study Area was either acutely or chronically toxic to aquatic test organisms, and concluded that removal of this sediment would contribute to the restoration of a healthy ecosystem in the AOC. However, sediment elutriate tests also showed that substances responsible for toxicity readily desorb from disturbed sediments, and are therefore likely to enter the water column during dredging. Further testing in Part II implicated ammonia, fluoride, and an as-yet-unidentified compound as the likely sources of toxic effects, including growth, survival and MFO induction, on a variety of aquatic test organisms. Exposure to disturbed sediments was acutely toxic to fathead minnows and rainbow trout, whereas exposure to undisturbed sediments caused no mortality but did affect growth. It is apparent from these results that disturbing the sediment during dredging will greatly increase the bioavailability of contaminants and may cause harmful effects *in situ*. Results of bioassays to determine the toxicity of fluoride showed that the amount of fluoride likely to enter the water column during dredging was high enough to cause mortality of sensitive test organisms (e.g., the amphipod *Hyallella azteca*), and that the highest concentration measured in sediment may affect the survival and/or growth of several aquatic organisms.

Abstract

The Reynolds Metals Company aluminum production plant at Massena, NY, discharges into the international section of the St. Lawrence River across from Cornwall, Ontario. Under a U.S. EPA Superfund administrative order, approximately 15,500 cu yds of sediment contaminated with PCBs, PAHs, cyanide, fluoride, aluminum and dibenzofurans are scheduled to be dredged from the river in the vicinity of the plant in the summer of 2001. This study represents Part II of a two-part study to determine the toxicity of sediments from this site to benthic organisms and to assess the potential impact of dredging activities on aquatic communities in Canadian waters. In Part I of this study, sediment was collected from 7 sites at varying distances from the plant outfall, analyzed for contaminants, and bioassayed. Results showed that nearly 40% of the sediment to be dredged was either acutely or chronically toxic to freshwater test organisms, and that the toxic substance(s) are likely to enter the water column during dredging. Based on preliminary tests with fathead minnows, *Pimephales promelas,* fluoride did not appear to contribute to the observed toxicity. Objectives of Part II were to (i) conduct expanded TIE testing to identify the toxic agent(s) in the sediments; (ii) simulate dredging conditions in the laboratory and determine the effects on survival, growth and induction of mixed function oxygenases or "MFOs" in fish; and (iii) conduct definitive bioassays to determine the toxicity of fluoride to several species of benthic organisms.

Sediment was collected from sites identified as "contaminated" (B-2) and "field control" (C-9) in Part I. Concentrations of major contaminants in these sediments were (µg/g dry weight; data for site B-2 given first): PCBs - 75 vs. 2.3; PAHs - 2376 vs. 17.4; fluoride - 1155 vs. 5.5; cyanide - 27.3 vs. 1.0; Al - 125,500 vs. 59,700. Exposure of rainbow trout (*Oncorhynchus mykiss)* to both disturbed (i.e., simulated dredging) and undisturbed sediment caused a 50-fold induction of EROD activity relative to the reference sediment at dilutions as low as 0.25% sediment in water, showing that inducing substances are highly mobile. The 20% treatment of C-9 sediment caused a marginally significant increase in activity (3.0 pmole/mg/min vs. 1.0 pmole/mg/min). Elutriate from site B-2 caused significant induction at the highest concentration tested only (20%), suggesting that some of the activity was due to substances attached to particles <1µ in size. Sediment from site B-2 also had a carcinogenic potency of over 400 µg/g BaP equivalents. Exposure to a 20% sediment: water dilution of disturbed sediment for 21 d caused 80% mortality of rainbow trout (within 4 d) and complete mortality of fathead minnows, whereas exposure to undisturbed sediment caused no mortality but did affect growth. Concentrations of ammonia were 2 to 4x higher at the start of tests with B-2 sediments than control sediments, and there is evidence that ammonia reached toxic levels in some tests. However, treatment with zeolite reduced, but did not eliminate, toxicity of B-2 elutriate to fathead minnows, suggesting that ammonia was not the sole toxic agent. Results of Phase II TIE tests on B-2 elutriate with *Daphnia magna* confirmed findings from the previous year, i.e., that toxicity was reduced by filtration and C18 adsorption, and that these treatments were more effective under acidic than neutral or alkaline conditions. Activated carbon, XAD resin, and cation exchange

treatments had no effect on toxicity; however, anion exchange removed all toxicity, suggesting that the toxic agent could be an anion, a metallic ionic complex or a weakly ionized acid, but there are other possibilities.

Fathead minnows and D. *magna* were more tolerant of fluoride in standard 96 h or 48 h aqueous exposures (LC_{50}s = 262.4 - 282.8 mg/L) than *Chironomus tentans* (LC_{50} = 124.1 mg/L), *Hexagenia limbata* (LC_{50} = 32.3 mg/L) and *H. azteca* (LC_{50} = 14.6 mg/L). The concentration of fluoride in B-2 elutriate was high enough to cause mortality of *H. azteca,* while concentrations in overlying water during sediment tests reached levels high enough to cause mortality of both *H. azleca* and *H. limbata.* Based on results of spiked sediment tests, survival and growth of fathead minnows and survival of chironomids would be unaffected by even the highest concentration known to occur in the Canadian environment (3460 µg/g). However, the highest concentration reported for sediment from the Reynolds Metals Study Area (1680 µg/g) could possibly affect survival and growth of *H. limbata* (LC_{50} = 1650 µg/g; IC_{25} = 1220 µg/g) and survival of *H. azteca* (LC_{50} = 1115 µg/g), and would likely affect growth of *H. azteca* (IC_{25} = 290 µg/g) and C *tentans* (IC_{25} = 660 µg/g). The removal of toxic sediment from the RMC Study Area will contribute to the restoration of a healthy aquatic ecosystem in the St. Lawrence River Area of Concern. Substances responsible for toxicity are unknown, but may include ammonia and fluoride. Toxic agents will readily enter the water column during dredging and may have harmful effects *in situ.*

Sommaire à l'intention de la direction

L'usine de production d'aluminium de la société Reynolds Metals Company (RMC), à Massena (New York), est située dans le secteur préoccupant (SP) du fleuve Saint-Laurent. Cet article est la deuxième et dernière partie d'une étude visant à déterminer la toxicité des sédiments pour les organismes benthiques dans le voisinage de l'usine, ainsi qu'à évaluer les risques pour l'environnement canadien des activités de dragage proposées pour cet emplacement, en application d'une ordonnance administrative dans le cadre du Superfund. Au cours de l'été 2001, on doit draguer de ce site environ 51 500 verges cubes de sédiments contaminés, notamment par les PCB, les HAP, le cyanure, le fluorure, l'aluminium et les dibenzofuranes. Cette étude vise un double but : i) en 1993, on a déclaré toxiques les fluorures inorganiques, en application de la *Loi canadienne sur la protection de l'environnement* (LCPE); toutefois, le Rapport d'évaluation des fluorures inorganiques (Programme de la liste des substances d'intérêt prioritaire) recommandait l'acquisition et l'évaluation de données supplémentaires sur les rapports entre les teneurs en fluorure des sédiments et leur toxicité pour les organismes benthiques (dans des régions du Canada où l'on observe ou prévoit des concentrations élevées de fluorures inorganiques dans les sédiments); ii) le comité canadien d'examen pour les sites visés par le Superfund à Massena, dont l'auteur principal de cet article est membre, est responsable de l'évaluation des risques pour l'environnement canadien des activités d'assainissement entreprises en application d'une ordonnance du Superfund. Comme cette usine est située de l'autre côté du fleuve, en face de Cornwall (Ontario), et à seulement quelques kilomètres en

amont de la frontière Québec - États-Unis, ces activités pourraient avoir un impact sur les communautés aquatiques des eaux canadiennes.

Dans la partie I de cette étude, on a déterminé que presque 40 % des sédiments qu'on doit draguer dans le secteur à l'étude de RMC présentaient une toxicité aiguë ou chronique pour les organismes expérimentaux aquatiques, et on a conclu que l'élimination de ces sédiments devrait contribuer au rétablissement d'écosystèmes sains dans le SP. Toutefois, des essais d'élutriation des sédiments ont également montré que les substances responsables de la toxicité se désorbaient rapidement des sédiments perturbés, et qu'il était probable qu'elles pénétreraient dans la colonne d'eau pendant le dragage. Au cours de la partie II de l'étude, d'autres essais ont permis de déterminer que l'ammoniac, le fluorure et un composé encore non identifié étaient les sources probables d'effets toxiques, notamment sur la croissance, la survie et l'induction de perturbateurs du système endocrinien (PSE) chez divers organismes aquatiques expérimentaux. L'exposition à des sédiments perturbés causait des réactions de toxique aiguë chez les têtes-de-boules et les truites arc-en-ciel, alors que l'exposition à des sédiments non perturbés réduisait la croissance sans causer de mortalité. Ces résultats montrent que la perturbation des sédiments pendant le dragage augmentera fortement la biodisponibilité des contaminants et pourrait avoir des effets *in situ* nocifs pour l'homme. Les résultats d'épreuves biologiques visant à déterminer la toxicité du fluorure ont indiqué que la quantité de fluorure devant pénétrer dans la colonne d'eau pendant le dragage était suffisamment élevée pour causer la mortalité d'organismes expérimentaux sensibles (p. ex. l'amphipode *Hyallella azteca*), et que la concentration la plus forte mesurée dans les sédiments pourrait avoir des effets sur la survie et/ou la croissance de plusieurs organismes aquatiques.

Résumé

L'usine de production d'aluminium de la société Reynolds Metals Company (RMC), à Massena (New York), déverse ses effluents dans la section internationale du Saint-Laurent, en face de Cornwall (Ontario). Par suite d'une ordonnance administrative du Superfund de l'EPA, on doit draguer, au cours de l'été 2001, environ 15 500 verges cubes de sédiments contaminés par les PCB, les HAP, le cyanure, le fluorure, l'aluminium et les dibenzofuranes dans le voisinage de l'usine. Cet article est la deuxième et dernière partie d'une étude visant à déterminer la toxicité des sédiments pour les organismes benthiques dans le voisinage de l'usine, ainsi qu'à évaluer l'impact possible des activités de dragage sur les communautés aquatiques dans les eaux canadiennes. Dans la partie I de cette étude, on a recueilli des sédiments à 7 sites situés à diverses distances de l'exutoire de l'usine, qui ont servi à l'analyse des contaminants et à des épreuves biologiques. Les résultats ont montré que presque 40 % des sédiments qu'on doit draguer causaient des symptômes de toxicité aiguë ou chronique pour les organismes expérimentaux d'eau douce, et que des substances toxiques devraient vraisemblablement pénétrer dans la colonne d'eau pendant le dragage. Selon des essais préliminaires effectués avec des têtes-de-boules (*Pimephales promelas*), le fluorure ne semblait pas contribuer à la toxicité observée.

Les objectifs de la partie II étaient : i) d'effectuer des essais élargis d'évaluation de l'indice de toxicité afin d'identifier les agents toxiques dans les sédiments; ii) de simuler en laboratoire les conditions de dragage et de déterminer les effets sur la survie, la croissance et l'induction des oxygénases de la fonction mixte (ou sur les perturbateurs du système endocriniens – PSE) chez les poissons, et iii) d'effectuer des épreuves biologiques définitives afin de déterminer la toxicité du fluorure pour plusieurs espèces d'organismes benthiques.

Au cours de la partie I de l'étude, on a recueilli des sédiments de lieux désignés comme « sites contaminés » (B-2) et « sites témoins » (C-9). Les concentrations des principaux contaminants dans ces sédiments étaient les suivantes (µg/g de poids sec; les valeurs de B-2 sont en premier) : PCB : 75 / 2,3; HAP : 2 376 / 17,4; fluorure : 1 155 / 5,5; cyanure : 27,3 / 1,0; Al : 125 500 / 59 700. L'exposition des truites arc-en-ciel (*Oncorhynchus mykiss)* à des sédiments perturbés (par dragage simulé) et non perturbés à des dilutions aussi faibles que 0,25 % de sédiments dans l'eau a causé une induction de l'activité de l'EROD 50 fois supérieure à celle observée avec des sédiments de référence, ce qui indique que les substances responsables de l'induction sont très mobiles. Le traitement à 20 % des sédiments C-9 a causé une augmentation à peine significative de l'activité (3,0 pmole/mg/min / 1,0 pmole/mg/min). L'élutriat du site B-2 ne causait une induction significative qu'à la plus forte concentration testée (20 %), ce qui semble indiquer qu'une certaine partie de l'activité était due à des substances liées à des particules d'une taille supérieure à 1 µ. De plus, les sédiments du site B-2 avaient un potentiel cancérogène supérieur à 400 µg/g d'équivalent de BaP. L'exposition à une dilution 20 % de sédiments perturbés dans l'eau pendant 21 jours causait une mortalité de 80 % des truites arc-en-ciel (en 4 jours ou moins) et de 100 % des têtes-de-boules, alors que l'exposition aux sédiments non perturbés ne causait pas de mortalité, mais ralentissait la croissance. Les concentrations d'ammoniac des sédiments B-2 étaient de 2 à 4 fois supérieures à celles des sédiments témoins au début des essais, et l'ammoniac atteignait des concentrations toxiques au cours de certains essais. Toutefois, un traitement par la zéolite réduisait, sans l'éliminer, la toxicité de l'élutriat B-2 pour les têtes-de-boules, ce qui semble indiquer que l'ammoniac n'était pas le seul agent toxique. Les résultats des essais d'évaluation de l'indice de toxicité de la phase II avec *Daphnia magna* pour l'élutriat B-2 ont confirmé les constatations de l'année précédente, c.-à-d. que la toxicité était réduite par filtration et adsorption des composés en C18, et que ces traitements étaient plus efficaces en conditions acides qu'en conditions neutres ou alcalines. Des traitements au charbon activé, à la résine XAD et par échange cationique n'avaient pas d'effets sur la toxicité; toutefois, l'échange anionique éliminait toute la toxicité, ce qui semble indiquer que l'agent toxique pourrait être un anion, un complexe ionique métallique ou un acide faiblement ionisé, mais il y a aussi d'autres possibilités.

Les têtes-de-boules et *D. magna* toléraient mieux le fluorure lors d'expositions normalisées en milieu aqueux de 96 ou de 48 h (CL_{50}: 262,4 – 282,8 mg/L) que *Chironomus tentans* (CL_{50} : 124,1 mg/L), *Hexagenia limbata* (CL_{50}: 32,3 mg/L) et *H. azteca* (CL_{50} : 14,6 mg/L). La concentration de fluorure dans l'élutriat B-2 était assez

élevée pour causer des mortalités chez *H. azteca,* alors que les valeurs dans l'eau sus-jacente pendant les essais de sédiments atteignaient des concentrations assez élevées pour causer des mortalités chez *H. azteca* et *H. limbata.* D'après les résultats d'essais effectués avec des sédiments enrichis, les taux de survie et de croissance des têtes-de-boules et les taux de survie des chironomidés ne devraient pas être touchés, même par les plus fortes concentrations observées dans l'environnement canadien (3 460 µg/g). Toutefois, la plus forte concentration déclarée pour les sédiments de la zone d'étude de l'usine de Reynolds Metals (1 680 µg/g) pourrait peut-être avoir des effets sur la survie et la croissance de *H. limbata* (CL_{50} : 1 650 µg/g; Ci_{25} : 1 220 µg/g) et sur la survie de *H. azteca* (CL_{50} : 1 115 µg/g), et elle devrait vraisemblablement avoir des effets sur la croissance de *H. azteca* (CI_{25} : 290 µg/g) et de *C tentans* (CI_{25} : 660 µg/g). L'élimination des sédiments toxiques de la zone d'étude de RMC contribuera au rétablissement d'un écosystème aquatique sain dans le secteur préoccupant du Saint-Laurent. Les substances responsables de la toxicité sont inconnues, mais il pourrait s'agir, entre autres, de l'ammoniac et du fluorure. Ces agents toxiques passeront facilement dans la colonne d'eau pendant le dragage et pourraient avoir des effets nocifs *in situ.*

INTRODUCTION

The Reynolds Metals Company (RMC) aluminum production plant at Massena, New York, discharges into the international section of the St. Lawrence River directly opposite Cornwall, Ontario, and several kilometres upstream of the U.S.A./Quebec border. River sediments in the vicinity of the plant are contaminated with substances such as PCBs, PAHs, aluminum, cyanide, fluoride and dibenzofurans, which are characteristic of smelting operations (WCC 1991). The RMC plant is a "Superfund" site; there are two other Superfund sites at Massena: the General Motors Corporation Central Foundry Division and the Aluminum Company of America (ALCOA). RMC merged with ALCOA in May, 1999, but will be referred to throughout this report as RMC. The Record of Decision for the RMC plant specifies the removal of sediments containing more than 1 ppm PCBs, 10 ppm PAHs and 1 ppb TDBFs (U.S. EPA 1993a). Approximately 51,500 cu yd of sediment were scheduled to be dredged from the Reynolds Metals Study Area in 1996 (Janette Anderson, Environment Canada, Ontario Region, personal communication, August 1995; Fig. 1). However, dredging was postponed and is now scheduled for the summer of 2001. The Reynolds Metals Study Area is of interest to Environment Canada for two reasons: (i) inorganic fluorides were recently declared toxic under the Canadian Environmental Protection Act (CEPA), however, information on the toxicity of sediment-associated fluoride to benthic organisms was considered incomplete (Environment Canada and Health Canada 1993b); and (ii) the Canadian Review Panel for Massena Superfund sites is responsible for assessing the risk of Superfund activities to the Canadian environment.

In Part I of this study, Metcalfe-Smith *et. al.* (1996) estimated that nearly 40% of the sediment to be dredged from the Reynolds Metals Study Area was either acutely or chronically toxic to fathead minnows (*Pimephales promelas*) and mayflies (*Hexagenia limbata*). The removal of this sediment should therefore contribute to the restoration of a healthy ecosystem in the St. Lawrence River Area of Concern. An elutriate prepared with sediment from the most contaminated site was acutely toxic to fathead minnows and the cladoceran, *Ceriodaphnia dubia*, showing that some toxicants can readily enter the water column when sediments are disturbed.

This finding suggested that dredging operations may themselves pose a threat to the aquatic environment. Results of a Phase I TIE suggested that the main toxic agent was probably an organic contaminant. Metcalfe-Smith *et al.* (1996) also determined that sediment-associated fluoride was highly mobile but did not appear to contribute to the toxicity of either sediments or elutriates from the Study Area. As higher levels of fluoride have been found in sediments from other industrial sites in Canada (Environment Canada and Health Canada 1994a), these results do not conclusively show that sediment-associated fluoride is harmless to benthic organisms under all conditions likely to be encountered in the Canadian environment.

The objectives of Part II of this study were to (i) conduct expanded TIE testing to more specifically identify the toxic agent(s) in sediments from the Reynolds Metals Study Area; (ii) simulate dredging conditions in the laboratory and determine the effects on survival, growth and induction of mixed function oxygenases or "MFOs" in fish; and (iii) conduct definitive bioassays to determine the toxicity of sediment-associated fluoride to four species of benthic organisms.

MATERIALS AND METHODS

Field Methods

Two of the 1993 study sites were revisited on 16 August 1994 for the collection of water and sediment samples. Based on results of the previous year's work, site B-2 was chosen as the "contaminated" site and site C-9 was selected as the "field control" (Fig. 2). The procedure at both sites was as follows: the Hydrolab was lowered to obtain *in situ* water quality measurements (temperature, pH, conductivity, % saturation and dissolved oxygen) at three depths (surface, bottom and 0.5 m from bottom), and the data were recorded directly to a computer. A Van Dorn bottle was then cast to collect a water sample at a depth of 0.5 m from the bottom, and a 250 mL subsample was taken for determining hardness (acidified by adding 1 mL concentrated nitric acid at the time of collection). Sediment was collected using a hand cast mini-ponar dredge. Sediment

was first placed in a 12 L plastic bucket, where large pieces of debris were removed and excess water decanted. The contents of the bucket were then transferred to a 20 L bucket lined with a plastic bioassay bag. When the 20 L bucket was full, air was expelled from the bioassay bag, the bag was closed off with a cable tie, and the bucket was sealed with a lid. A total of seven 20 L buckets of sediment were collected from site B-2 and five from site C-9. Sediment from both sites had the consistency of "black ooze". Water and sediment samples were transported to NWRI the same day and stored overnight at 4°C. They were transferred to the ESG International (formerly B.A.R. Environmental Inc.) laboratory in Guelph, Ontario on 17 August 1994, where they were stored in the dark at 4°C.

Laboratory Methods

Preparation and toxicity testing of sediments

On 27 August 1994, the 100 L of C-9 sediment were composited in a rectangular 120 L plastic tank using a large wooden paddle. In accordance with procedures recommended by Environment Canada (1994), the sediment was mixed thoroughly for about 30 minutes then returned to the original buckets and stored in the dark at 4°C until required for testing. On 29 August 1994, the 140 L of B-2 sediment were divided into two batches that were composited separately in the 120 L tank, then mixed with each other. After mixing, the sediment was returned to the original buckets and stored in the dark at 4°C until required for testing. Half of the material was labelled B-2A and the other half B-2B, and the former was used up first. Subsamples were taken from both batches for analysis of PCBs, PAHs, fluoride, cyanide, Al, 11 other elements, % TOC, and % moisture at the time of homogenization. Results from each batch were compared to ensure that the sediment had been thoroughly homogenized. Results from both batches were also compared with the results for sediments collected from the same sites in 1993. Additional subsamples of B-2 sediment were taken after 3 to 4 months of storage, as TIE tests were still being conducted at that time. Sediment collected from Long Point, Lake Erie on 13

September 1993 was used as a clean or "negative" control sediment in this study. This sediment is routinely used as a control sediment by Environment Canada (e.g., Day *et al.* 1995). Long Point sediment had been characterized in 1993, and was not reanalyzed.

For continuity with the previous year's work, sediment toxicity tests for 21 d growth and survival of fathead minnows (*P. promelas*) and mayflies (*H. limbata*) were repeated using the same Ontario Ministry of the Environment and Energy (OMOEE) test protocols (Bedard *et al.* 1992). As in 1993, significant changes to the protocols were as follows: to approximate conditions in the St. Lawrence River, pH and hardness of the dilution water were adjusted to 7.1 ± 0.1 and 140-150 mg/L as $CaCO_3$, respectively, prior to testing; mayflies were fed; growth of both organisms was measured as the difference between the average final weight and average initial weight of all animals in each replicate. The test conditions for fathead minnows and mayflies are summarized in Tables 1 and 2, respectively. The only difference in methods between years was that the mayflies were larger in 1994 (40-80 mg wet weight) than in 1993 (10-25 mg). Tests with fathead minnows and mayflies began on 30 August 1994 and 01 September 1994, respectively. As fish may contribute significant amounts of ammonia to the overlying water during static tests, ammonia concentrations were determined on days 1, 10, 18 and 21 in one test container of each treatment (B-2, C-9 and Control) during the fathead minnow test. Concentrations of un-ionized ammonia were calculated from concentration of total ammonia adjusted for test temperature and pH using the formula of Thurston *et al.* (1981). On day 21, samples of overlying water were removed from two replicates of each treatment of the fathead minnow test and one replicate of each treatment of the mayfly test for analysis of fluoride. All series of tests included a negative control.

Effects of simulated dredging conditions on fish: MFO induction

Mixed function oxygenases or "MFOs" are a group of membrane-bound enzymes known to metabolize a wide variety of substrates. Exposure of fish to certain contaminants, including

PAHs, co-planar PCBs, dioxins and furans causes an increase in activity or "induction" of the enzymes, and this activity can be measured. The toxicological consequences of induction vary depending on whether the contaminants are persistent (such as dioxins) or readily metabolized (such as PAHs). In mammals, MFO induction has been linked with toxic effects such as thymic atrophy, reduced growth and tumour development (Hodson 1996).

The mixed function oxygenase (MFO) experimental protocol involved the exposure of five juvenile rainbow trout (*Oncorhynchus mykiss*) in 10 L of test solution for 4 d under static renewal conditions, and duplicating each treatment. At the end of the exposure period, the livers were removed, weighed, homogenized with a grinding buffer and centrifuged, and the supernatant and microsomal layers were frozen in liquid nitrogen until assayed. The assay measured the enzymatic conversion of ethoxyresorufin to resorufin, and the "EROD" activity was expressed as pmole/mg of protein/minute. A typical response curve consisted of a dose-response relationship up to a plateau, followed by a decrease in activity due to toxic effects (Hodson *et al* 1991).

To determine if sediments from the Study Area had the potential to cause MFO induction in rainbow trout, we first conducted a preliminary test using freeze-dried sediment containing 2500 µg/L PAHs that had been collected from site B-2 in 1993. A 4:1 (v:v) methanol extract containing 625 µg/L PAHs was prepared as a stock solution. Five dilutions ranging from nominal concentrations of 10 to 1000 µg/L PAHs were then tested. This test represented a "worst case" scenario, as methanol would extract inducing substances from the sediment more efficiently than water. Based on results from the methanol extract tests, we proceeded with further experiments using fresh sediment collected in 1994.

The experimental protocol for tests conducted on 1994 sediment samples was as described above, except that renewal of the test solutions was not possible. Three treatments of B2-A sediment were tested in September 1994: undisturbed sediment, disturbed sediment (e.g., with sediment in suspension to simulate dredging conditions), and elutriate. Effects on survival and

MFO induction were assessed over a 4 d exposure period using juvenile rainbow trout. Five fish were added to each test vessel. Tests were replicated such that a total of 10 fish were exposed in each test and the control. Test conditions for 4 d rainbow trout MFO exposures are summarized in Table 3.

As in 1993, the 1994 elutriates were prepared by mixing water and sediment in a 4:1 (v:v) ratio. The mixture was vigorously stirred for 30 seconds every 10 minutes over a 1 hour period, then tightly covered and stored at 4°C for 48 hours to settle. The elutriate was then decanted and filtered through 1 μ glass fibre filter. Due to problems with "break-through" when using cellulose acetate filters in 1993, glass fibre filters were used in combination with a positive-pressure filtration system in 1994. Positive pressure was used rather than vacuum filtration to avoid degassing and loss of volatiles. The raw (unfiltered) elutriate was coffee-coloured and opaque; filtration with this system produced the clear, colourless elutriate that was used in the MFO tests. B2-A sediment and raw (unfiltered) elutriate were analyzed for PCBs and PAHs. The filters were also analyzed in order to determine the amount of PCBs and PAHs retained by the filters.

A series of 5 dilutions were run for each treatment, beginning with the usual 4:1 (v:v) water:sediment (or 20% sediment:water) dilution. The other dilutions were 2.5%, 0.25%, 0.025% and 0.0025% (v:v) sediment:water. A full series of dilutions was run for the disturbed treatment of the field control sediment (C-9), but only the 20% dilution was run for the undisturbed and elutriate treatments of C-9 sediment and all three treatments of the Long Point, Lake Erie, reference sediment. An outline of the overall test approach is provided in Table 4. All tests involved aeration of the overlying water. In the tests on undisturbed sediment, water was aerated near the surface to ensure minimal disturbance of the sediment. Water was also aerated near the surface in the elutriate tests. In the tests on disturbed sediments, aeration was provided at the sediment/water interface in order to keep a portion of the sediment in suspension at all times.

Effects of simulated dredging conditions on fish: Growth and survival

Twenty-one day growth and survival tests were conducted on 20% sediment:water dilutions of undisturbed and disturbed B-2 sediment using fathead minnows and rainbow trout. The standard Ontario Ministry of the Environment and Energy (OMOEE) fathead minnow sediment bioassay protocol (Bedard *et al.* 1992) calls for aeration of the overlying water. Under control conditions, the overlying water normally remains cloudy throughout the entire exposure period due to activity of the fish. However, tests conducted previously on B-2 sediment in 1993 showed that the level of fish activity was reduced relative to the controls (i.e., the fish were avoiding the sediment), such that the overlying water remained relatively clear. In order to assess the potential effects of dredging, which included the presence of suspended solids in the water column, the standard 21 d growth and survival test with fathead minnows was repeated under conditions whereby the B-2A sediments were manually disturbed. Aeration was used to keep the sediment in suspension. The normal rate of aeration was provided, except that instead of providing aeration immediately below the water surface, it was provided immediately above the sediment layer. The standard bioassay was conducted concurrently for comparison. Testing was initiated on 20 October 1994 using fathead minnows with an average wet weight of 0.355 g at the start of testing. General test conditions for 21 d fathead minnow exposures are summarized in Table 1.

The toxicity of undisturbed B-2B sediment to rainbow trout was also assessed using a 21 d growth and survival bioassay. Tests were conducted in quadruplicate with ten fish per test vessel (clean 20 L polyethylene pails). Since all trout died within 4 d in the disturbed treatment of the MFO test, this treatment was excluded from the 21 d growth and survival test. To prevent the build up of ammonia in the overlying test water, fish were transferred to fresh solutions of sediment and water every 7 d. These solutions were prepared in the same manner and at the same time as the initial treatments, but fish were not added to these solutions until the appropriate time. The renewal solutions remained in sealed containers and were stored at 15 °C. To facilitate the

transfer of fish with minimal stress, the test fish were placed into a transfer vessel that fit inside the test container. The transfer vessel consisted of a clean polyethylene pail with a screened bottom. This transfer vessel, when situated inside the test vessel, was positioned immediately above the sediment-water interface. As this system did not permit direct contact of the fish with the sediment, the transfer vessel was raised and lowered three times daily to permit some circulation and exchange between the overlying water and the sediment. Testing was initiated on 20 October 1994 using trout with an average wet weight of 0.318 g at the start of testing. General test conditions for 21 day rainbow trout exposures are summarized in Table 5.

To determine the potential for various contaminants to enter the water column during dredging, raw elutriate (i.e., settled but unfiltered) was analyzed for PCBs, PAHs, fluoride, cyanide and metals, and compared with the data for sediment. One litre samples of elutriate were taken for analysis of PCBs and PAHs, and 250 mL samples were taken for analysis of the other contaminants. At termination of the exposures, overlying water from 2 replicates each of the Control and B-2B undisturbed treatments (rainbow trout) and 2 replicates each of the Control, B2-A undisturbed and disturbed treatments (fathead minnows) were analyzed for fluoride.

To check for build-up of ammonia in the test solutions, samples were taken during the tests on Control and B2-B undisturbed treatments with rainbow trout and on Control, B-2A undisturbed and disturbed sediment with fathead minnows. Just prior to test completion, total suspended solids (TSS) was measured in 2 replicates in each of the Control, B-2A undisturbed and disturbed treatments with fathead minnows in order to measure the amount of material in suspension and to determine if the fathead minnows were foraging in the sediment.

Expanded Toxicity Identification Evaluation (TIE) testing

As a step toward determining the probable cause(s) of toxicity, a Phase I Toxicity Identification Evaluation (TIE) was conducted in 1993. The general methodology followed

protocols developed by the U.S. EPA (1989, 1991, 1993b), but included recommendations by Ankley *et al.* (1992) for conducting TIEs to determine toxicity associated with the disposal or resuspension of dredged materials. The TIE process is divided into three phases. The purpose of Phase I is to characterize the physical and chemical properties of the toxicant(s) by means of a standard series of chemical and physical manipulations and toxicity tests (U.S. EPA 1991). The procedure involves determining the toxicity of untreated elutriate, then subjecting it to various treatments to remove specific types of toxicants, and finally testing the treated elutriate to see if toxicity is enhanced, reduced or unaltered. Phase II involves identification of the suspected toxicant(s), and relies on results from Phase I (U.S. EPA 1989). Confirmation of the suspected toxicants occurs in Phase III (U.S. EPA 1993b). In 1993, a Phase I TIE had been conducted on B-2 elutriate using fathead minnows. The results pointed towards an organic contaminant being the probable cause of toxicity, with ammonia possibly being a contributing factor. Based on these results, a Phase II TIE was conducted in 1995 in an attempt to more specifically identify the properties of the toxic agent(s).

As previously mentioned, the glass fibre filters and pressure filtration used to prepare elutriates in 1994 was found to be more efficient at removing particulate material from the raw elutriate than the cellulose acetate filters and vacuum filtration used in 1993. In fact, toxicity of filtered elutriate from site B-2 was found to be highly variable and in many cases non-toxic in 1994. Expanded TIE testing (Phase II) was therefore conducted on raw, *unfiltered* elutriate (i.e., settled for 48 hours and decanted). Prior to initiating the TIE, preliminary tests were conducted to determine the relative contribution of ammonia to toxicity, since ammonia had been identified as a possible source of toxic effects in 1993. Fathead minnows and *Daphnia magna* were exposed to 100%, 50%, 25%, 13%, 6% and 0% solutions of raw elutriate before and after treatment with zeolite. Zeolites are naturally occurring crystalline aluminosilicates that preferentially remove ammonia from aqueous solutions (Sherman 1978). Raw, unfiltered elutriate was passed through the zeolite column and tested on 15 February 1995. Exposure periods for fathead minnows and *Daphnia magna* were 96 and 48 hours, respectively.

Following the preliminary ammonia treatments, Phase II TIE testing on raw B-2 elutriate was initiated on 03 March 1995. Results from the baseline tests indicated that *Daphnia magna* were more sensitive than fathead minnows to the raw elutriate, i.e., daphnid immobility and mortality occurred. An immobile daphnid was defined as an organism that did not display movement during 10 seconds of observation and yet still had a visible "heartbeat" under microscopic examination. We therefore decided to use *Daphnia magna* immobility as the TIE endpoint. General test conditions for TIE exposures with *Daphnia magna* are summarized in Table 6. Samples of raw elutriate were passed through a series of column treatments (anion and cation exchange, activated carbon, and XAD resin) known to remove different types of substances. For example, XAD-4 resin removes a broad range of low molecular weight organic contaminants. Cation and anion exchange columns remove ionic species from aqueous solutions and may also remove organic ions of high molecular weight and metallic anionic complexes. For continuity with the previous year's work, filtration under acidic and neutral conditions and the C18 solid phase extraction treatment to remove non-polar organics were repeated.

Toxicity of water-borne and sediment-associated fluoride to benthic organisms

I) Water-borne fluoride

In 1993, the toxicity of water-borne fluoride was determined using the 7 d growth and survival test with larval fathead minnows. In the current study, a series of spiked water bioassays was conducted to determine the aqueous toxicity of fluoride to one fish and four species of invertebrates. The test organisms were: an amphipod (*Hyalella azteca*), a mayfly (*Hexagenia limbata*), a midge (*Chironomus tentans*), a crustacean (*Daphnia magna*), and the fathead minnow (*Pimephales promelas*). Appropriate amounts of analytical reagent grade sodium fluoride (NaF) were added to laboratory dilution water (hardness = 140-150 mg/L as $CaCO_3$; pH = 7.8 ± 0.2) to obtain the desired concentration(s) of fluoride (F^-). Sodium fluoride is the compound most

frequently used to determine the toxicity of fluoride to aquatic organisms (Environment Canada and Health Canada 1994a). Exposure concentrations were dependant on the test species and ranged from 6 mg/L to 5,600 mg/L.

The 48 h static non-renewal daphnid bioassay followed the Environment Canada test protocol (Environment Canada 1990). The 48 h static non-renewal amphipod bioassay followed a method recently developed by Environment Canada. The 96 h static-renewal fathead minnow bioassay methodology was adapted from the U.S. EPA (1993c) acute toxicity testing protocol. The 96 h static non-renewal tests with *C. tentans* and *H. limbata* followed methods used by the OMOEE (D. Bedard, OMOEE, personal communication). The LC_{50} for survival was calculated for each bioassay. General test conditions for aqueous fluoride exposures are summarized in Tables 7 to 11.

II) Sediment-associated fluoride

The test organisms included *H. azteca*, *H. limbata*, *C. tentans* and *P. promelas*. Clean sediment from Long Point, Lake Erie was spiked with sodium fluoride (NaF) for testing. Prior to spiking, the appropriate amount of sediment was measured and added to the exposure vessel, and any excess surface water was decanted. Appropriate amounts of analytical reagent grade NaF were weighed, dissolved in 10 mL of dilution water, and thoroughly blended with the previously dispensed de-watered (wet) control sediment. The specific exposure concentrations were dependant on the test species and ranged from 175 mg/kg to 5,600 mg/kg. The test container was covered to prevent evaporation, and was left to equilibrate overnight prior to testing.

Tests with fish, mayflies and chironomids (21 d exposures) were conducted following standardized test procedures developed by the Ontario Ministry of the Environment (Bedard *et al.* 1992). The 28 d amphipod bioassay followed the method used by Environment Canada. All bioassays were conducted in glass jars containing a 4:1 water to test sediment ratio under static

test conditions. Test conditions included appropriate temperature control and gentle aeration of the overlying water throughout the test. Samples of overlying water were taken from the mayfly bioassay on days 0, 12 and 21 for fluoride analysis. Two endpoints were used, namely, the LC_{50} for survival and the IC_{25} (25% inhibition concentration) for growth. General test conditions for the fluoride-spiked sediment exposures are summarized in Tables 1, 2, 12 and 13.

Analytical methods

Hardness was measured using the EDTA titrimetric method (APHA-AWWA-WPCF, 1975). Ammonia was measured using the Nessler Method and HACH® 2000. Samples of overlying water from the sediment toxicity tests were analyzed for total kjeldahl nitrogen and NH_3-N by the Wastewater Technology Centre, Burlington, Ontario (WTC), using Industrial Method Nos. 786-86T and 780-86T, respectively, as described in the Technicon Traacs 800™ Operation Manual (Technicon Industrial Systems Corporation, 1987). Samples of raw and filtered elutriate were analyzed for fluoride by the WTC using Method 4500-F C. Ion-selective electrode method (Eaton *et al.* 1995). All samples except those from the fluoride spiking tests were also analyzed by the WTC for cyanide using Method 4500-CN E. Colorimetric method (Eaton *et al.* 1995) and for Al, Cu, Ni and Zn using Method 3030 F. Nitric acid-hydrochloric acid digestion (Eaton *et al.* 1995).

Sediment samples were freeze-dried prior to analysis using a LABCONCO Lyph-Lock 6® freeze-dryer fitted with a Model 77560 Lyph-Lock Stoppering Tray Dryer® for precise temperature control. After drying, the samples were homogenized using a mortar and pestle. Samples were analyzed by the WTC for PAHs (16 compounds) using Method MSS-1 described in their Analytical Methods Manual (WTC 1994), for PCBs (sum of 69 congeners and co-eluting congeners) using U.S. EPA Method 608 - Organochlorine Pesticides and PCBs (U.S. EPA 1988), for cyanide using Method 4500-CN C. Total cyanide after distillation (Eaton *et al.* 1995), and for fluoride using Method 4500-F C. Ion-selective electrode method (Eaton *et al.* 1995). Samples

were analyzed for total and extractable Al, Cr, Cu, Pb and Zn, extractable Cd, Fe, Mn and Ni, and total Hg, As and Se by the NLET using standard procedures described in their Manual of Analytical Methods (NLET 1994). Particle size distribution and organic content were determined by the Sedimentology Laboratory, NWRI, using standard procedures (Dalton 1994).

RESULTS AND DISCUSSION

Water and Sediment Chemistry, and Sediment Toxicity

Water quality data for samples collected at the surface, bottom (2.4 m) and 0.5 m from the bottom are presented for sampling sites C-9 and B-2 in Table 14a. With the exception of hardness measurements, which were conducted in the laboratory, all water quality measurements were taken *in situ* using a HydroLab. Water quality was generally comparable to data gathered at similar locations in 1993 (Table 14b). Water temperature was higher in 1994 than 1993 due to the time of year when sampling was conducted (August vs. October).

Results of chemical analyses on the sediment samples are presented in Table 15. Results are provided for each batch at the time of homogenization and for selected periods during storage. Data for congener-specific PCBs and individual PAH compounds are shown in Appendix I. Comparisons between sediment batches B-2A and B-2B (29 August, 1994 samples) indicate that the sediment was thoroughly homogenized. Consequently, tests conducted with either batch were considered to be comparable. Additional subsamples of B-2 sediment were analyzed after approximately 2 and 4 months of storage, because tests were still being conducted at that time. As in 1993 (Metcalfe-Smith *et al.* 1996), storage did not result in the significant loss of any contaminant, including cyanide, which was the substance most likely to degrade over time.

Comparisons between the 1993 and 1994 sediment chemistry data indicated that there was virtually no difference between years for site C-9 (Fig. 3), but that concentrations in sediment from site B-2 were slightly lower for all contaminants except PAHs, in 1994 (Fig. 4). Comparisons were based on the average concentration for B-2 and B-2E in 1993 and the average concentration for B-2A and B2-B for August 1994. There was only one value for C-9 in both years. Concentrations for site C-9 in 1993 vs. 1994 were (µg/g dry weight) as follows: 2.0 vs. 2.3 PCBs, 16.8 vs. 17.4 PAHs, 6.8 vs. 5.5 fluoride, 1.3 vs. 1.0 cyanide and 57,300 vs. 59,700 aluminum. Concentrations for site B-2 in 1993 vs. 1994 were as follows: 108 vs. 75 PCBs, 2148 vs. 2376 PAHs, 1435 vs. 1155 fluoride, 31.4 vs. 27.3 cyanide and 154,000 vs. 125,500 aluminum.

Data for the fathead minnow and mayfly bioassays conducted using sediment collected from sites C-9 and B-2 in 1994 are presented in Appendix II. Fathead minnows lost weight in all treatments, including the controls. This phenomenon was also observed in the 1993 bioassays and appears to be a feature of the OMOEE protocol. In comparison, mayflies gained weight in all treatments, including the controls. Thus, the results are referred to as "weight change" rather than growth. Fathead minnows exposed to the B-2A sediment had a significantly lower survival compared to the Long Point control sediment ($t=3.5$; $p=0.05$). Similarly, a significant growth effect was observed in those organisms exposed to the B-2A sediment when compared to the control ($t=6.96$; $p=0.05$). Exposure to the B-2A sediment also had a significant effect on *H. limbata* survival ($t=11$; $p=0.05$), but not on growth ($t=2.2$; $p=0.05$), i.e., those mayflies that survived in the B-2A sediment grew as much as the control organisms.

Comparisons between the 1993 and 1994 bioassay results indicate that the effects on survival and growth of fathead minnows were the same at both sites for both years (Table 16). Fathead minnows exposed to sediment collected from site B-2 had significantly lower survival and growth when compared to the controls in both 1993 and 1994. However, fish exposed to B-2 sediment lost slightly more weight in 1993 compared to 1994. Fathead minnow mortality was not observed in the control sediments in either year. In both years, the fathead minnows noticeably

avoided the B-2 sediment, did not engage in any typical foraging behaviour at the sediment-water interface, and remained near the water surface. They also appeared to be much less energetic and were visibly thinner upon test completion.

Survival of mayflies in sediment from site B-2 was greater in 1994 (42.5 %) than 1993 (0 %; Table 17). It was concluded that sediment obtained from site B-2 in 1994 was possibly slightly less contaminated and toxic than sediment collected in 1993. Such differences were not unexpected, due to the dynamic movement of sediment deposits and the difficulty in revisiting a sampling location with complete accuracy. As indicated earlier, the only difference in methods between years was that the mayflies were larger in 1994 (40-80 mg wet weight) than in 1993 (10-25 mg). Consequently, another possible explanation for differences in survival is that the older, larger mayflies were slightly more tolerant to the sediment contaminants than the smaller and presumably younger organisms used in 1993. In both years, the mayflies attempted to avoid contact with the B-2 sediment, refused to burrow and subsequently died on the sediment surface. Sediment from site C-9 was non-toxic to both test organisms in both years.

As in 1993, overlying water from the fathead minnow bioassays was analyzed for ammonia to determine if the build-up of ammonia from waste products could have contributed to the observed toxicity (Table 18). Total ammonia levels appeared to gradually increase in all tests at the start of testing to about day 10, followed by a gradual decrease from day 10 to 21. The largest increase was observed in the B-2A overlying water, where total ammonia concentrations reached 18.6 mg/L on day 18. Note that pH was not measured on day 18 and therefore the un-ionized ammonia concentration could not be calculated. However, the un-ionized ammonia level on day 10 (0.131 mg/L) was well below the toxic threshold of 0.653 to 0.888 mg/L as determined by Thurston *et al* (1981) for fathead minnows at 15°C and a pH of 7.82 to 7.83. These results differ from the 1993 fathead minnow bioassays where the concentrations of un-ionized ammonia in B-2 overlying water increased from 0.369 mg/L at the start of testing to 0.718 mg/L at test completion. These results indicate that ammonia was not a likely source of toxicity in the 1994

tests, but may have contributed to fish mortality in 1993.

Results from fluoride measurements taken from the overlying water on day 21 of the fathead minnow and mayfly tests are provided in Tables 19a and 19b. Results were similar for both tests, with the highest concentration of fluoride occurring in overlying water from the B-2A sediments (39.2 - 41.5 mg/L). Concentrations in the negative control (Long Point) and field control (C-9) ranged from 0.177 to 0.241 mg/L. These results will be discussed in the section on fluoride toxicity.

Effects of simulated dredging conditions on fish: MFO induction

Results of the preliminary test on methanol-extracted B-2 sediment from 1993 showed that there was significant induction of MFOs in rainbow trout at all five dilutions tested (Fig. 5). A dose-response relationship was not observed, because enzyme activity had already reached a plateau at the lowest concentration tested. The highest level of activity observed was 380 pmole/mg/min in the 100 µg/L treatment (a 100-200 fold induction increase), and there was evidence of toxicity in the 1000 µg/L treatment. For comparison, Martel *et al.* (1996) observed up to a 15.5-fold induction increase in fish exposed to pulp mill effluent. Results from this test showed that inducing substances were present in the sediment and were easily extracted. Based on these findings, we proceeded with further testing on fresh sediment collected in 1994.

As noted earlier, 3 treatments of B-2A sediment were tested: undisturbed sediment, disturbed sediment (i.e., sediment in suspension to simulate dredging conditions), and elutriate. All raw MFO data is provided in Appendix III. The only mortalities of trout occurred in the 20% dilution of the disturbed treatment of B-2A sediment (80% mortality) and the 20% dilution of the B-2A elutriate treatment (10% mortality; Appendix IV). In terms of MFO induction, a similar dose-response relationship was observed in both the disturbed and undisturbed treatments of B-2A sediment (Fig. 6); EROD activity plateaued at about 50 pmole/mg/min in both treatments,

suggesting that the contaminants responsible for induction were very mobile, i.e., they readily entered the water column - even from undisturbed sediment. The B-2A elutriate caused significant induction at only the highest concentration tested (20%; Fig. 6). It is not surprising that the elutriate would contain lower concentrations of contaminants than the other two treatments, as particles greater than 1μ had been removed by filtration. In fact, results from the chemical analysis of B2-A sediment, raw elutriate and filters showed that although virtually no PCBs passed through the filters, approximately 110 μg/L PAHs would have been present in the 20% dilution of elutriate (Table 20, Appendix Va). This was the nominal concentration of PAHs that was associated with very high induction (380 pmole/mg/min) in the methanol extract test. One possible explanation for the difference in induction between the methanol extracted vs. fresh sediment tests is that the methanol may have extracted PCBs and/or other inducing substances from the sediment. A marginally significant increase in EROD activity occurred in the 20% dilution of the disturbed treatment of the field control (C-9) sediment (i.e., 3.0 pmole/mg/min vs. approximately 1.0 pmole/mg/min in all reference sediment treatments; Fig. 6), suggesting that inducing compounds are present at low concentrations in sediment outside the area to be dredged.

BaP equivalents for the 16 PAHs analyzed in the test sediments were calculated from Toxic Equivalency Factors (TEFs) given in Nisbet and LaGoy (1992). TEFs provide a numerical ranking of the carcinogenic potency of various PAHs relative to the potency of benzo-a-pyrene (BaP; TEF=1). BaP equivalents were calculated by multiplying the TEF for each compound by its concentration in the test sediment (Appendix Vb). Total concentrations of PAHs were 2320.34-3447.72 μg/g in sediments from site B-2 and 17.39 μg/g in sediment from site C-9, while BaP equivalents were 409.95 - 643.28 μg/g (17-20%) at site B-2 and 4.11 μg/g (24%) at site C-9 (Table 21). Although BaP equivalents are not a direct measure of toxicity, they do provide an indication of the environmental hazard of these sediments relative to sediments from other contaminated sites. For comparison, Balch *et al.* (1995) observed a concentration of 137 μg/g dry weight of total PAHs in sediment from Hamilton Harbour, and a carcinogenic potency of 12.6 μg/g BaP equivalents (9% of total).

Dredging of contaminated sediment was conducted at a nearby Federal Superfund site (General Motors Central Foundry Division) in 1995. It is our understanding an "action" level of 25 mg/L total suspended solids (TSS) was used at this site, whereby dredging would be halted due to possible harmful effects on the environment if TSS outside the silt curtain exceeded 25 mg/L. In context of the present experiment, this translates into a 0.0125% sediment:water ratio (detailed calculations are presented in Appendix VI). This action level appears to be protective of MFO induction for the RMC Study Area, but it may not be for two reasons: i) suspended solids occurring outside the silt curtain would likely contain a higher proportion of fine particles than the disturbed B-2 treatment, and finer particles tend to be more contaminated, and ii) we know that not all of the material in our treatments was always in suspension (see next section). Thus, it is possible that significant MFO induction could occur in wild fish or in fish caged in the vicinity of the dredging operations at Reynolds Metals if an action level of 25 mg/L TSS is used at this site. Hodson *et al.* (1996) concluded from these studies that dredging contaminated sediments from the Reynolds Metals Study Area may expose fish to highly toxic chemicals.

Also of interest is the relationship between a TSS "action" level of 25 mg/L and the possible occurrence of phototoxicity during dredging. Davenport and Spacie (1991) observed increased toxicity to *Daphnia magna* exposed to PAH-contaminated elutriates in the presence of sunlight and near-UV light, while elutriates were non toxic in the absence of light. Their results suggested that even though suspended sediments were not directly toxic, those known to be contaminated with PAHs could result in photoxicity during dredging.

Effects of simulated dredging conditions on fish: growth and survival

Raw data from the 21 d growth and survival tests conducted on 20% sediment:water dilutions of undisturbed and disturbed sediment using fathead minnows (B-2A) and rainbow trout (B-2B) are provided in Appendix VII. As anticipated, fathead minnows avoided the sediment in

the undisturbed treatment. There was no significant mortality of either fathead minnows or rainbow trout in the undisturbed treatment; however, there was a significant effect on growth of both species (Table 22). Fathead minnows lost weight in B-2A undisturbed sediment relative to the Control ($t=3.1$; $p=0.05$). Rainbow trout actually gained weight in B-2B undisturbed sediment; however, the amount of weight gained was significantly less than that gained by the Control fish ($t=3.3$; $p= 0.05$). In the disturbed treatment, complete mortality of fathead minnows occurred between days 13 and 19. As mentioned earlier, trout were not tested because 80% had died within 4 d in the disturbed treatment of the MFO induction experiment. These results demonstrate that disturbing the sediment during dredging will greatly increase the bioavailability of contaminants and may cause harmful effects *in situ*.

Concentrations of un-ionized ammonia in the undisturbed B-2B sediment treatment were well below the toxic threshold of 0.374 to 0.588 mg/L as determined by Thurston *et al.* (1981) for rainbow trout at approximately 13°C and a pH of 7.3 to 7.87 (Table 23). Low ammonia concentrations in the trout assay were likely due to the test methodology; i.e., fish were transferred to fresh sediment/water solutions every 7 days, thereby preventing the build up of ammonia in the overlying water. In comparison, un-ionized ammonia levels in both the disturbed and undisturbed fathead minnow bioassays reached levels that exceeded the toxic threshold of 0.653 to 0.888 mg/L for this species (Thurston *et al.* 1981; Table 24). Complete mortality occurred in the disturbed test as compared with only 32% mortality in the undisturbed test, even though concentrations of un-ionized ammonia were similar throughout the 21 day testing period in both treatments (Fig. 7). These results suggest that ammonia was not the sole toxic agent.

Results for fluoride measurements taken from the overlying water on day 21 of the fathead minnow and rainbow trout tests are shown in Tables 25a and 25b. Disturbing the sediment had no observable effect on fluoride concentrations in the water column in the fathead minnow tests. However, fluoride concentrations in the undisturbed trout assay were approximately one half those in the fathead minnow assay. It is suspected that the trout test system greatly reduced

sediment disturbance, thereby reducing the amount of fluoride released into the overlying water. Unlike the fathead minnow test, a screened transfer container (used to transfer fish to fresh sediment/water solutions) physically separated trout from the test sediment. Furthermore, the fathead minnows were exposed to the same sediment/water solution for 21 days, while trout were provided with a new (pre-prepared) solution every 7 days.

Results of the TSS determinations on samples from the control, undisturbed and disturbed treatments with fathead minnows indicate that very little material was actually in suspension in the disturbed treatment at termination of the test (Table 26). Laboratory notes indicated that, over time, sediment in the disturbed treatment formed compacted "clumps" that settled at the bottom of the test container and did not get mixed back into the water column. Higher TSS values in overlying water from the control sediment indicate that fathead minnows spent more time foraging in the control sediment and than the B-2 sediment. This avoidance behaviour was supported by visual observations made throughout the 21 d exposure. Control fish were observed to actively swim about the test container, causing the overlying water to become turbid. In contrast, fish in the undisturbed and disturbed treatments were observed mostly at the water surface. The reason why TSS levels were actually higher in the undisturbed than the disturbed treatment is probably because of the difference in mortality levels and hence the number of fish moving about in the containers at the end of the test. It is apparent that the test system needs to be modified to adequately simulate dredging conditions in the laboratory.

Disturbed sediment was obviously more toxic than undisturbed sediment to both trout and fathead minnows. It follows that disturbing the sediment during dredging could result in harmful effects on biota. Both sediment and raw (settled, but unfiltered) elutriate from site B-2 were analyzed to determine the amounts of contaminants that would theoretically be available to enter the water column in the disturbed and undisturbed tests. Preparation of 1L of elutriate requires 1L of water and 250 mL of wet sediment. Sediments were analyzed on a dry weight basis, assuming that 250 mL of wet sediment weighed 49.3 g dry (see Appendix VI). Thus, multiplying

concentrations of contaminants in sediment (μg/g dry weight) by 49.3 g yields the total amount (μg) of each contaminant that is theoretically available to enter the water column. These amounts are presented in Table 27, where they are compared with the actual concentrations of selected contaminants measured in raw elutriate. Results showed that 41% of the fluoride, 2% of the cyanide, 0.7% of the PCBs, 0.6% of the PAHs, and 0.2% of the Al actually entered the water column (data for remaining parameters are shown in Appendices Va and VIII). The organic contaminants may be bound to very fine particles that were excluded from the elutriate, but would be present in the water column during dredging. It should be noted here that the contaminant(s) responsible for MFO induction are not necessarily the same contaminants that cause direct toxic effects, i.e., reductions in survival and growth.

Expanded TIE testing

Ammonia was identified as a possible source of toxic effects in 1993. Because substances such as ammonia and hydrogen sulphide commonly occur in contaminated sediments, Ankley *et al.* (1992) recommended that testing to determine their contribution to overall toxicity should precede detailed TIE investigations. Therefore, fathead minnows and *Daphnia magna* were exposed to 100%, 50%, 25%, 13%, 6% and 0% solutions of raw (unfiltered) elutriate before and after treatment with zeolite, which removes ammonia. Treatment with zeolite reduced concentrations of un-ionized ammonia (the toxic form) in all solutions to levels as low as those in the 13% solution of raw elutriate (Tables 28a and b, Appendix IX). Treatment with zeolite did not change the pattern of mortality in the *D. magna* tests - complete mortality occurred in all tests except the controls. However, mortality of fathead minnows was somewhat reduced in the 50% and 100% treatments. We concluded that ammonia may contribute to the toxicity of B-2 elutriate, but is not the main toxic agent. Results from the graduated pH test conducted in 1993 indicated that hydrogen sulphide was not a likely source of toxicity (Metcalfe-Smith *et al.* 1996).

Following the initial zeolite treatments, further Phase II testing was conducted using the more sensitive test organism, *D. magna*. The TIE results confirmed that toxicity was reduced by filtration and C18 adsorption, and that these treatments were more effective under acidic than neutral or alkaline conditions (Table 29). Ineffectiveness of the carbon and XAD treatments tended to rule out phenolics, PCBs and PAHs. Ineffectiveness of the cation exchange resin confirmed that the toxic agent was not a heavy metal. The anion exchange resin removed all toxicity, suggesting that the toxic agent could be a metallic anionic complex (e.g., aluminum with cyanide or fluoride), a weakly ionized acid, or an anion other than fluoride (which was ruled out in 1993), but there are many other possibilities.

Wallis *et al.* (1996) suspected that low levels of fluoride may cause the mobilization of Al in St. Lawrence River sediments. Fluoride is generally thought to reduce the toxicity of Al to fish, largely through the formation of less toxic aluminum fluoride complexes (Driscoll *et al.* 1980; Parkhurst *et al.* 1990; Wilkinson *et al.* 1990), but the relationship between Al and fluoride with respect to their toxicity to aquatic organisms is complex. For example, Hamilton and Haines (1995) showed that low concentrations of fluoride inhibited Al toxicity to Atlantic salmon (*Salmo salar*) at neutral pH, but high concentrations of fluoride had no effect; however, the reverse was true at low pH, i.e., low concentrations of fluoride did not influence Al toxicity, but high concentrations of fluoride increased the toxicity of Al. Aluminum fluoride complexes may also increase toxicity to *S. salar* if the presence of inorganic aluminum (Al^{3+}, $AL(OH)_n$) is maintained (Wilkinson *et al.* 1990).

Anionic arsenic is another potential cause of the observed toxicity. Arsenic is rapidly removed from the dissolved state, and is deposited in sediment as organic and inorganic particulates (Nriagu 1983). In anaerobic conditions, As tends to be in the form H_3AsO_3, but this form is unstable and generally oxidizes to $HAsO_4^{-2}$ and $H_2AsO_4^{-}$ (Environment Canada and Health Canada 1993a). According to Ontario's Provincial Sediment Quality Guidelines for the protection of aquatic biological resources (Persaud *et al.* 1992), the LOEL (lowest effect level; i.e., the level

that can be tolerated by the majority of benthic organisms) and SEL (severe effect level) for As in sediment are 6 µg/g and 33 µg/g dry weight, respectively. The maximum concentration of As measured in sediment from site B-2 was 19.8 µg/g, which exceeds the LOEL but not the SEL (Table 15).

Toxicity of water-borne and sediment-associated fluoride to benthic organisms

I) Water-borne fluoride

Results of tests to determine the toxicity of aqueous fluoride (as NaF) to five species of benthic organisms are presented in Table 30. *Hyalella azteca* was the most sensitive species (LC_{50} = 14.6 mg/L), followed by *H. limbata* (LC_{50} = 32.3 mg/L) and *C. tentans* (LC_{50} = 124.1 mg/L). Fathead minnows and *D. magna* were less sensitive to fluoride, with LC_{50}s ranging from 262.4 to 282.8 mg/L. Tests were conducted at 20° C and a hardness of 140-150 mg/L $CaCO_3$ for 48 hr (*D. magna* and *H. azteca*) or 96 h (all others). Other studies have reported similar results for fathead minnows and cladocerans. Smith *et al.* (1985) reported 96-hour LC_{50}s of 205 and 180 mg F^-/L for fathead minnows at 20° C and hardnesses of 256 and 92 mg/L $CaCO_3$, respectively, and Fieser (1986) reported 96-hour LC50s of 124 to 194 mg F^-/L for fatheads at 15° C and hardnesses ranging from 69 to 292 CaCO. Fieser *et al.* (1986) estimated the 48-hour LC_{50} for *D. magna* to be 247 mg F^-/L at 20 °C. Hickey (1989) found that four species of cladocerans (*D. magna, Daphnia carinata, Simocephalus vetulus, Ceriodaphnia dubia*) were relatively tolerant of NaF, with 24 hour LC_{50}s' ranging from 353.6 mg F^-/L (*D. magna*) to 157.9 mg F^-/L (*C. dubia*) at 20° C. The LC_{50} for *C.* cf. *pulchella* was only 83.2 mg F^-/L, but the author noted that this species had poor survival in laboratory settings.

Concentrations of fluoride in river water collected from 11 sites in the study area in 1990 ranged from 0.10 to 0.21 mg/L, except that a concentration of 1.40 mg/L was reported immediately adjacent to the main plant outfall (WCC 1991). These concentrations are well below

levels that would be toxic to the benthic organisms tested in this study. However, we have presented evidence that fluoride associated with sediments readily enters the water column when these sediments are disturbed. For example, elutriate prepared with sediment from site B-2 contained 23.3 mg F^-/L (Table 27), which is high enough to cause mortality of *H. azteca* in 48-hour tests. Concentrations of fluoride in overlying water at the end of 21-day exposures of fathead minnows and mayflies to disturbed and/or undisturbed sediment from site B2 ranged from 38.6 to 43.3 mg/L (Tables 19a,b and 25a). These concentrations are high enough to cause mortality of both *H. azteca and H. limbata.*

II) Sediment-associated fluoride

Results of tests to determine the toxicity of sediment-associated fluoride (as NaF) to four species of benthic organisms are presented in Table 31 and compared with environmental concentrations in Fig. 7. Raw data are tabulated in Appendix X. The highest concentration of fluoride observed in sediment from the RMC Study Area was 1680 µg/g at site B-2 in October 1993, and the highest concentration reported to date in the Canadian environment is 3460 µg/g (Environment Canada and Health Canada 1993b). Results of sediment toxicity tests suggest that growth and survival of fathead minnows and survival of chironomids would be unaffected by even the highest concentrations likely to be encountered in the Canadian environment. However, the highest concentration measured in sediment from the Reynolds Metals Study Area could possibly affect growth and survival of *H. limbata* and survival of *H. azteca*, and would be likely to affect growth of *H. azteca* and *C. tentans*. Since sediments from the RMC Study Area that contained only 40-60 µg/g fluoride significantly affected the growth of fathead minnows and mayflies in tests conducted in 1993 (Metcalfe-Smith *et al.* 1996), it is clear that the main toxic agent in these sediments is not fluoride.

Relative sensitivity of the test organisms to fluoride was similar in both the spiked sediment and spiked water tests. In the aqueous tests, fathead minnows were the most tolerant,

followed by chironomids, then mayflies, then amphipods. Survival followed the same pattern in the spiked sediment tests except that fathead minnows and chironomids were equally tolerant, while chironomids were more sensitive than mayflies for the growth endpoint.

Samples of overlying water were collected on days 0, 12 and 21 of the spiked sediment tests with *H. limbata*. Concentrations in water appeared to reach a plateau by day 12 in all treatments (Table 32). Furthermore, aqueous concentrations in the 1400, 2800 and 5600 ppm treatments were almost exactly $1/100^{th}$ of the original spiked concentrations. This relationship may prove useful for predicting the amounts of fluoride that could enter the water column under various dredging scenarios.

Fluoride may have significant sublethal effects on freshwater organisms. The first symptoms of acute fluoride toxicity in carp (*Cyprinus carpio*) and rainbow trout are apathetic behaviour and anorexia (Neuhold and Sigler 1960; Sigler and Neuhold 1972), and at sublethal concentrations, toxicity is characterized by a reduced respiratory rate (Camargo and Tarazona 1991). Chitra *et al.* (1983) argued that the toxic action of fluoride on fish may be due to the inhibition of enzymatic activity. Similarly, after an initial increase, oxygen consumption and acetylcholinesterase activity was reduced in the freshwater field crab (*Barytelphusa guerini*) after 15 days of exposure to fluoride (Reddy and Venugopal 1990). Fluoride may also cause avoidance behaviour in exposed animals. In this study, *P. promelas* and *H. limbata* avoided sediment that was highly contaminated with fluoride. Damkaer and Dey (1989) found that the migration of Pacific salmon (*Oncorhynchus* sp.) was inhibited by fluoride levels occurring downstream of an aluminum plant. Using flume routes, they demonstrated that salmon selectively avoided routes with fluoride levels as low as 0.5 mg F^-/L. After the aluminum plant reduced its emissions of fluoride, fish passage times improved and losses of fish were reduced (Damkaer and Dey 1989).

Results of this study agree with other studies that suggest certain freshwater invertebrates are more sensitive to fluoride than freshwater fish. For example, Camargo *et al.* (1992) found

that net-spinning caddisfly larvae (F. Hydropsychidae) were more sensitive than either brown trout or rainbow trout. The fingernail clam, *Musculium transversum* is believed to be the freshwater species most sensitive to fluoride (Environment Canada and Health Canada 1993b).

SUMMARY AND CONCLUSIONS

In Part I of this study (Metcalfe-Smith *et al.* 1996), we demonstrated that nearly 40% of the sediment to be dredged from the St. Lawrence River in the vicinity of the Reynolds Metals Company aluminum production plant was either acutely or chronically toxic to freshwater benthic organisms (i.e., fathead minnows and the mayfly, *Hexagenia limbata*). As such, removal of this sediment should make a significant contribution to the restoration of a healthy aquatic ecosystem in the St. Lawrence River Area of Concern. However, results also showed that the dredging operations may themselves pose a threat to aquatic biota. Elutriate prepared with sediment from the most contaminated site was acutely toxic to fathead minnows and cladocerans, indicating that toxic chemicals can readily desorb from disturbed sediment and enter the water column either in dissolved form or bound to very fine particles (<1μ). Sediment-associated fluoride was found to be highly mobile, but results of preliminary toxicity tests indicated that concentrations likely to be released during dredging would be below toxic levels.

In Part II of this study, the RMC Study Area was revisited and sediment was collected from the most contaminated site (B-2) and a field control site (C-9) for further testing. For continuity purposes, chemical analysis of the sediment and 21 d growth and survival tests with fathead minnows and mayflies were repeated. Results showed that sediment from site B-2 in 1994 was slightly less contaminated and toxic than sediment taken from this site in 1993. Both organisms avoided the sediment in both years, i.e., fathead minnows did not forage and mayfly nymphs did not burrow.

Dredging of contaminated sediment has the potential to pose a toxicological risk to aquatic ecosystems, primarily by way of the resuspension of toxic compounds (Fichet *et al.* 1998), and can reduce survivorship and growth of exposed aquatic organisms (Fichet *et al.* 1998; Bonnet *et al.* 2000). Dredging conditions were simulated in the laboratory to determine the effects on survival, growth and MFO induction in fish. To test for MFO induction, rainbow trout were exposed to undisturbed sediment, disturbed sediment (i.e., sediment in suspension to simulate conditions during dredging) and unfiltered elutriate from sites B-2 and C-9, as well as a control sediment from Long Point, Lake Erie. Data for the disturbed and undisturbed B-2 sediment treatments exhibited typical response curves, i.e., a dose-response relationship up to a plateau. EROD activity approached an asymptote at about 50 pmole/mg/min in both treatments, indicating that the contaminant(s) responsible for induction can readily enter the water column - even from undisturbed sediment. A decrease in EROD activity was observed at the highest concentration of the B-2 disturbed treatment, indicating toxic effects. Mortality of 80% of the fish in this test confirmed that it was toxic. Elutriate from site B-2 caused significant induction only at the highest concentration tested, suggesting that the contaminant(s) responsible for toxicity may be mainly associated with fine suspended particles. An action level of 25 mg/L TSS may be applied to dredging operations in the RMC Study Area. There is insufficient information at present to determine if this action level would prevent MFO induction in exposed fish.

The effect of dredging on survival and growth of aquatic organisms has the potential to be severe. Mortality of fathead minnows and rainbow trout exposed to undisturbed sediment from site B-2 for 21 d did not differ from mortality in the controls, although fathead minnows avoided the sediment and there was a significant effect on the growth of both species. However, exposure to disturbed sediment from the same site caused 80% mortality of rainbow trout within 4 d, and mortality of all fathead minnows between days 13 and 19. It is apparent that disturbing the sediment during dredging will greatly increase the bioavailability of contaminants and may cause harmful effects *in situ*.

Ammonia was identified as a possible cause of toxicity in sediments tested in 1993. Ammonia is often present in sediments from industrial sites (e.g., Ankley *et al.* 1992), but it can also build up in test containers during long-term static exposures due to the accumulation of waste products from the test fish. Ammonia was measured during 21 d sediment toxicity tests with fathead minnows and simulated dredging tests with fathead minnows and rainbow trout. Concentrations of total ammonia at the start of these tests (day 5 for the fathead minnow dredging tests), were 2 to 4 × higher in the B-2 treatments than the controls. However, in all cases, un-ionized ammonia concentrations (the toxic form) were below the toxic thresholds for these species. Concentrations of un-ionized ammonia consistently exceeded the toxic threshold for fathead minnows after day 7 in the B-2 disturbed treatment and on most occasions after day 7 in the B-2 undisturbed treatment. Corresponding mortality rates were 100% and 32%, respectively. No mortality occurred in the control, where ammonia concentrations remained well below toxic levels throughout the tests. These results suggest that sediments were the source of the ammonia, which may have contributed to mortality of fathead minnows. Treatment with zeolite to remove ammonia reduced, but did not eliminate, toxicity of B-2 elutriate to this species. We therefore conclude that the amount of ammonia that would be released from the sediment during dredging may be harmful to aquatic organisms *in situ*.

Results of the Phase II TIE tests with *D. magna* confirmed findings from the previous year that toxicity of elutriate from site B-2 was reduced by filtration and C18 adsorption, and that these treatments were more effective under acidic than neutral or alkaline conditions. Activated carbon, XAD resin, and cation exchange treatments had no effect on toxicity; however, anion exchange removed all toxicity, suggesting that the toxic agent could be an anion, a metallic ionic complex or a weakly ionized acid.

Toxicity of fluoride was specifically investigated, using several aquatic test species. Results showed that the amount of fluoride likely to enter the water column during dredging was high enough to cause mortality of the amphipod, *H. azteca*, and possibly the mayfly, *H. limbata*,

in 48 h laboratory exposures (assuming a 4:1 v:v ratio of water to sediment). Results of sediment toxicity tests suggest that growth and survival of fathead minnows and survival of chironomids would be unaffected by the highest concentrations of sediment-associated fluoride likely to be encountered in the Canadian environment. However, the highest concentration measured in sediment from the Reynolds Metals Study Area could possibly affect growth and survival of *H. limbata* and survival of *H. azteca*, and would be likely to affect growth of *H. azteca* and *C. tentans*. Since sediments containing non-toxic levels of fluoride significantly affected the growth of fathead minnows and mayflies in tests conducted in 1993, fluoride is not the only toxic agent in these sediments.

In conclusion, the removal of toxic sediment from the RMC Study Area will make a significant contribution to the restoration of a healthy aquatic ecosystem in the St. Lawrence River Area of Concern. The substance or substances responsible for toxicity are unknown, but may include ammonia and fluoride. Further TIE testing is recommended to specifically identify the sources of toxicity. It *is* known that the toxic agents are highly bioavailable and will readily enter the water column, either in dissolved form or adsorbed to very fine particles, during dredging. We therefore recommend that RMC choose a dredging methodology for this project that will cause the least disturbance of the sediment during its removal from the river. On-site toxicity testing should be conducted concurrently with the dredging operations to ensure that harmful effects to the aquatic ecosystem are minimized.

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Table 1. Summary of test conditions for the fathead minnow 21 d survival and growth test (Bedard *et al.* 1992, with modifications).

1.	Test type:	Static
2.	Test duration:	21 days
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	1 % of aver. body wt./d based on initial wt.
8.	Test chamber size:	1.8 L wide mouth glass jar
9.	Sediment volume:	325 mL (approx. 2 cm depth)
10.	Water volume:	1,300 mL
11.	Source:	Laboratory culture
12.	Age of test organisms:	3 - 4 mo. (80 - 110 mg wet weight for spiked sediment exposures; 200-400 mg for B2/C9 exposures)
13.	No. animals per test chamber:	10
14.	No. of rep. test chambers/conc'n:	4
15.	No. of animals per test level:	40
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness and pH adjusted to that of St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1 (v:v)
19.	Measured water chemistry parameters:	pH, dissolved oxygen, water temperature at day 0, 10 and 21.
20.	Measured sediment chem. parameters:	pH (at start of test)
21.	Measured end points:	Survival (%) and aver. growth - measured as the difference between final and initial wet wt).

Preparation of Bioassay Chamber:

- 1.8 L glass widemouth jars were acid washed, hexane and distilled-water rinsed, and filled to a depth of 2 cm with sediment.
- Resuspended sediment was allowed to settle overnight (no aeration).
- Aeration (oil-free compressed air) of the overlying water was provided 1 hr prior to adding the test organisms and continued throughout the 21 d exposure.

Table 2. Summary of test conditions for the *Hexagenia limbata* 21 d survival and growth test (Bedard *et al.* 1992, with modifications).

1.	Test type:	Static
2.	Test duration:	21 days
3.	Temperature:	20 $\pm$1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	1 mL of Hexagenia diet weekly per jar
8.	Test chamber size	1.8 L wide mouth glass jar
9.	Sediment volume:	325 mL (approx. 2 cm depth)
10.	Water volume:	1,300 mL
11.	Source:	Laboratory culture
12.	Age of test organisms:	3 - 4 mo (5-25 mg wet weight for spiked sediment exposures; 40-80 mg for B2 exposures)
13.	No. animals per test chamber:	10
14.	No. of rep. test chambers/conc'n:	4
15.	No. of animals per test level:	40
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness adjusted to St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1 (v:v)
19.	Measured water chemistry parameters:	pH, conductivity, dissolved oxygen, water temperature at day 0, 10 and 21
20.	Measured sediment chem. parameters:	pH (at start of the test)
21.	Measured end points:	Survival (%) and aver. growth - measured as mg wet wt (growth determined as difference between final and initial wts.)

Preparation of Bioassay Chamber:

- 1.8 L glass widemouth jars were acid washed, hexane and distilled-water rinsed, and filled to a depth of 2 cm with sediment.
- Resuspended sediment was allowed to settle overnight (no aeration).
- Aeration (oil-free compressed air) of the overlying water was provided 1 hr prior to adding the test organisms and continued throughout the 21 d exposure.

Table 3. Summary of test conditions for the rainbow trout 4 d survival and MFO tests (Hodson *et al* 1991).

1.	Test type:	Static
2.	Test duration:	96 hours
3.	Temperature:	15 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	None
8.	Test chamber size	22.5 L plastic pail
9.	Sediment volume:	2.5 L
10.	Water volume:	10 L
11.	Source:	Rainbow Springs Hatchery
12.	Age of test organisms:	juveniles (< 1g)
13.	No. animals per test chamber:	5
14.	No. of rep. test chambers/conc'n:	2
15.	No. of animals per test level:	10
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness adjusted to that of St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1, 4:0.1, 4:0.01, 4:0.001 and 4:0.0001 (v:v)
19.	Measured water chemistry parameters:	pH, dissolved oxygen, water temperature at day 0 and 4.
20.	Measured sediment chem. parameters:	pH (at start of test)
21.	Measured end points:	Survival (%) and abnormal behaviour.

Table 4. Summary of MFO exposure methods and experimental design.

Conditions:

- 4 day static, 10L tank, no renewal.
- <1 g rainbow trout, 2 reps, 5 fish/rep.

Treatments:

- Undisturbed; Disturbed; Elutriate

Sediment/water (%)	B-2			C-9			Long Point		
	U	D	E	U	D	E	U	D	E
20.0% (4:1)	•	•*	•	•	•	•	•	•	•
2.5%	•	•	•		•				
0.25%	•	•	•		•				
0.025%	•	•	•		•				
0.0025%	•	•	•		•				

*80% mortality

Table 5. Summary of test conditions for the rainbow trout 21 d survival and growth test.

1.	Test type:	Static
2.	Test duration:	21 days
3.	Temperature:	15 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	1% body weight/day based on initial weights
8.	Test chamber size:	22.5 L plastic pail
9.	Sediment volume:	2.5 L
10.	Water volume:	10 L (renewal of test solution every 7 days)
11.	Source:	Rainbow Springs Hatchery
12.	Age of test organisms:	juveniles (< 1g)
13.	No. animals per test chamber:	10
14.	No. of rep. test chambers/conc'n:	4
15.	No. of animals per test level:	40
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness adjusted to that of St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1 (v:v)
19.	Measured water chemistry parameters:	pH, dissolved oxygen, water temperature at day 0, 10 and 21.
20.	Measured sediment chem. parameters:	pH (at start of test)
21.	Measured end points:	Survival (%) and aver. growth - measured as the difference between final and initial wet wt).

Table 6. Summary of test conditions for toxicity identification evaluations using *Daphnia magna* (U.S. EPA 1991).

1.	Test type:	Static
2.	Test duration:	48 hours
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	None
8.	Test chamber size	100 mL plastic container
9.	Water volume:	80 mL
10.	Source:	Laboratory culture
11.	Age of test organisms:	First instar (<24 hr old)
12.	No. animals per test chamber:	5
13.	No. of test chambers/conc'n:	1 (min)
14.	No. of animals per test level:	5 (Minimum)
15.	Aeration:	Only in aeration treatment
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured water chemistry parameters:	pH, dissolved oxygen, alkalinity, water hardness
18.	Measured end points:	Mortality, immobility, stressed behaviour

Table 7. Summary of test conditions for the *Daphnia magna* acute lethality test (Environment Canada 1990).

1.	Test type:	Static non-renewal
2.	Test duration:	48 hours
3.	Temperature:	20 $\pm$1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	None
8.	Test chamber size	55 mL glass test tube
9.	Water volume:	50 mL (15 mL/animal)
10.	Source:	Laboratory culture
11.	Age of test organisms:	first instar (<24 hr old)
12.	No. animals per test chamber:	3
13.	No. of rep. test chambers/conc'n:	4
14.	No. of animals per test level:	12
15.	Aeration:	None
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured water chemistry parameters:	pH, dissolved oxygen, water hardness
18.	Measured end points:	Mortality, stressed behaviour

Table 8. Summary of test conditions for *Hyalella azteca* acute lethality test (Environment Canada, standard operating procedures: culture and sediment bioassay methods for: *Chironomus riparius, Hexagenia* spp., *Hyalella azteca* and *Tubifex tubifex*).

1.	Test type:	Static non-renewal
2.	Test duration:	48 hours
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	50 to 100 ft candles
6.	Photoperiod:	16 h light, 8 h darkness
7.	Feeding regime:	None
8.	Test chamber size	250 mL beaker
9.	Test solution volume:	200 mL
10.	Age of test organisms:	1-7 days old
11.	No. animals per test chamber:	5
12.	No. of rep. test chambers/conc'n:	2 (5 conc. + 1 control)
13.	No. of animals per test level:	10
14.	Aeration:	None
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured end points:	Mortality, stressed behaviour

Table 9. Summary of test conditions for the fathead minnow acute lethality test (U.S. EPA 1993c).

1.	Test type:	Static renewal
2.	Test duration:	96 hours
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	50 to 100 ft candles
6.	Photoperiod:	16 h light, 8 h darkness
7.	Feeding regime:	0.2 mL brine shrimp following 48 hour renewal
8.	Test chamber size	1 L plastic container
9.	Test solution volume:	250 mL
10.	Renewal of test solution:	48 hours
11.	Age of test organisms:	juveniles or 10 day old
12.	No. animals per test chamber:	10
13.	No. of rep. test chambers/conc'n:	2 (5 conc. + 1 control)
14.	No. of animals per test level:	20
15.	Aeration:	N/A
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured end points:	Mortality, stressed behaviour

Table 10. Summary of test conditions for *Hexagenia limbata* acute lethality test (D. Bedard, OMOEE, pers. comm.).

1.	Test type:	Static non-renewal
2.	Test duration:	96 hours
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	50 to 100 ft candles
6.	Photoperiod:	16 h light, 8 h darkness
7.	Feeding regime:	0.2 mL *Hexagenia* diet
8.	Test chamber size	250 mL beaker
9.	Test solution volume:	200 mL
10.	Age of test organisms:	3 - 4 mo
12.	No. animals per test chamber:	5
13.	No. of rep. test chambers/conc'n:	2 (5 conc. + 1 control)
14.	No. of animals per test level:	10
15.	Aeration:	None
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured end points:	Mortality, stressed behaviour

Table 11. Summary of test conditions for *Chironomus tentans* acute lethality test (D. Bedard, OMOEE, pers. comm.).

1.	Test type:	Static non-renewal
2.	Test duration:	96 hours
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	50 to 100 ft candles
6.	Photoperiod:	16 h light, 8 h darkness
7.	Feeding regime:	2 g silica sand and fed 0.005g of tetra on the first day of the test
8.	Test chamber size	250 mL beaker
9.	Test solution volume:	200 mL
10.	Renewal of test solution:	None
11.	Age of test organisms:	10 days old
12.	No. animals per test chamber:	10
13.	No. of rep. test chambers/conc'n:	2 (5 conc. + 1 control)
14.	No. of animals per test level:	20
15.	Aeration:	N/A
16.	Dilution water:	Well water (hardness adjusted to St. Lawrence R. water)
17.	Measured end points:	Mortality, stressed behaviour

Table 12. Summary of test conditions for the *Chironomus tentans* 10 d survival and growth test (Bedard *et al.* 1992, with modifications).

1.	Test type:	Static
2.	Test duration:	10 days
3.	Temperature:	20 ± 1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	1 mL of Chironomid diet per test chamber
8.	Test chamber size:	1.8 L wide mouth glass jar
9.	Sediment volume:	325 mL (approx. 2 cm depth)
10.	Water volume:	1,300 mL
11.	Source:	Laboratory culture
12.	Age of test organisms:	10-12 days
13.	No. animals per test chamber:	15
14.	No. of rep. test chambers/conc'n:	3
15.	No. of animals per test level:	45
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness and pH adjusted to that of St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1 (v:v)
19.	Measured water chemistry parameters:	pH, dissolved oxygen, water temperature at day 0 and 10.
20.	Measured sediment chem. parameters:	pH (at start of test)
21.	Measured end points:	Survival (%) and aver. dry weight of each replicate.

Preparation of Bioassay Chamber:

- 1.8 L glass widemouth jars were acid washed, hexane and distilled-water rinsed, and filled to a depth of 2 cm with sediment.
- Resuspended sediment was allowed to settle overnight (no aeration).
- Aeration (oil-free compressed air) of the overlying water was provided 1 hr prior to adding the test organisms and continued throughout the 21 d exposure.

Table 13. Summary of test conditions for the *Hyalella azteca* 28 d survival and growth test (Environment Canada, standard operating procedures: culture and sediment bioassay methods for: *Chironomus riparius, Hexagenia* spp., *Hyalella azteca* and *Tubifex tubifex* (with modifications).

1.	Test type:	Static
2.	Test duration:	28 days
3.	Temperature:	20 ±1°C
4.	Light quality:	Ambient laboratory illumination
5.	Light intensity:	40 to 50 ft candles
6.	Photoperiod:	16 h light, 8 h dark
7.	Feeding regime:	weekly per jar
8.	Test chamber size	250 mL wide mouth glass jar
9.	Sediment volume:	50 mL
10.	Water volume:	200 mL
11.	Source:	Aquatic Research Organisms (Hampton,NH)
12.	Age of test organisms:	1-7 days old
13.	No. animals per test chamber:	10
14.	No. of rep. test chambers/conc'n:	3
15.	No. of animals per test level:	30
16.	Aeration:	Yes
17.	Dilution water	Well water (hardness adjusted to St. Lawrence R. water)
18.	Water:Sediment Ratio	4:1 (v:v)
19.	Measured water chemistry parameters:	pH, conductivity, dissolved oxygen, water temperature at day 0, 10 and 21
20.	Measured sediment chem. parameters:	pH (at start of the test)
21.	Measured end points:	Survival (%) and aver. dry weight of each replicate

Preparation of Bioassay Chamber:

- 1.8 L glass widemouth jars were acid washed, hexane and distilled-water rinsed, and filled to a depth of 2 cm with sediment.
- Resuspended sediment was allowed to settle overnight (no aeration).
- Aeration (oil-free compressed air) of the overlying water was provided 1 hr prior to adding the test organisms and continued throughout the 21 d exposure.

Table 14a. Water quality data for samples collected from sites B-2 and C-9 on 16 August 1994. All measurements except hardness were determined *in situ*.

Site	Depth	Temp. (°C)	pH	Conductivity (µS)	saturation	Percent DO (mg/L)	Hardness (mg/L as $CaCO_3$)
B-2	Surface	20.52	8.16	262	108.60	9.72	-
	0.5 m from bottom	20.50	8.22	265	108.20	9.70	133
	Bottom (2.4 m)	20.47	8.18	265	108.50	9.73	-
C-9	Surface	20.22	7.91	261	100.00	9.01	-
	0.5 m from bottom	20.25	7.94	264	100.40	9.04	140
	Bottom (2.4 m)	20.25	7.95	265	100.20	9.03	-

Table 14b. Selected water quality data for samples collected from site B-2 on 6 October 1993 and site C-9 on 5 October 1993. All measurements except hardness were determined *in situ*.

Site	Depth	Temp.(°C)	pH[a]	Conductivity (µS)	Percent saturation	DO (mg/L)	Hardness (as $CaCO_3$)
B-2	0.5 m from bottom	14.1	8.2	305	92.6	9.47	130
C-9	0.5 m from bottom	14.3	8.2	299	92.0	9.37	140

[a] Hydrolab pH readings were off calibration and were not used. Thus, pH was determined in the laboratory using samples originally collected for analysis of fluoride and Al.

Table 15. Chemical and physical characterization of sediments collected from the Reynolds Metals Study Area on 16 August 1994. Samples were homogenized 27-29 August, and used as needed in toxicity tests; sediment from site B-2 was stored in two batches (A and B). Concentrations reported as µg/g dry weight except where otherwise indicated.

Parameter and Date	B-2A 29 August 1994	B-2A 11 November 1994	B-2B 29 August 1994	B-2B 20 December 1994	C-9 30 August 1994
Total PCBs	77.6	83.0	72.4	75.1	2.27
Total PAHs	2320.34	3447.72	2430.94	2346.17	17.39
Fluoride	1160	891	1150	1130	5.5
Cyanide	27.8	28.4	26.8	26.6	1.00
Al - T[b]	126000	129000	125000	103000	59700
Al - E[c]	30400	29600	29600	30600	3900
Cr - T	44.0	33.3	35.6	40.5	46.3
Cr - E	12.9	12.9	13.4	13.9	9.52
Cu - T	214	207	217	229	43.7
Cu - E	81.8	80.6	84.2	76.1	32.3
Pb - T	55.1	54.5	57.1	57.9	35.1
Pb - E	49.1	46.8	49.6	50.2	30.1
Zn - T	1020	1010	1040	1150	261
Zn - E	857	852	875	916	260
Hg - T	0.12	0.117	0.119	0.149	0.135
As - T	18.8	18.3	18.7	19.8	4.52
Se - T	4.61	4.48	4.43	4.81	1.01
Cd - E	2.24	2.22	2.33	2.32	1.14
Fe - E	8870	9410	9210	9820	5790
Mn - E	204	201	210	225	197
Ni - E	49.2	49.6	50.4	41.5	13.5
TOC (%)	11.2	11.6	11.1	11.0	3.49
Moisture (%)	73.11	-	-	-	64.17

[a] data from Metcalfe-Smith *et al.* (1996) for sediment collected 13 September 1993 (Control sediment not analyzed in 1994).
[b] total;[c] extractable.

Table 16. Comparison of 1993 and 1994 fathead minnow 21 d bioassay results.

Year	Sample	Overall Survival (%)	Average weight change (mg)
1994	B-2A	75	- 42.6
	C-9	100	- 1.7
	Long Point Control	100	- 8.2
1993	B-2[a]	87.5	- 80.1
	B-2E[b]	72.5	- 64.2
	C-9	100	- 4.2
	Long Point Control	100	- 5.0

[a] sample from site B-2 collected on 6 October 1993.
[b] sample from site B-2 collected on 17 November1993.
Note: average survival and weight change for site B-2 in 1993 was 80% and -72.2 mg, respectively.

Table 17. Comparison of 1993 and 1994 *Hexagenia limbata* 21 d bioassays.

Year	Sample	Overall Survival (%)	Average weight change (mg)
1994	B-2A	42.5	23
	C-9	100	27.4
	Long Point Control	97.5	29.1
1993	B-2[a]	0	n/a [c]
	B-2E[b]	0	n/a
	C-9	90	23.5
	Long Point Control	100	17.9

[a] sample from site B-2 collected on 6 October 1993.
[b] sample from site B-2 collected on 17 November 1993.
[c] not applicable.

Table 18. Selected ammonia measurements taken during fathead minnow 21 d survival and growth tests conducted on test sediments (08/30/94).

Sample	Day	Replicate	Total Ammonia (mg/L)	pH	Un-ionized Ammonia (mg/L)
Control	1	a	1.1	7.2	0.007
	10	b	6.4	7.9	0.195
	18	c	0.5	-[a]	-
	21	d	0.14	7.8	0.003
C-9	1	a	1.6	7.4	0.016
	10	b	2.9	7.8	0.071
	18	c	0.5	-	-
	21	d	0.18	7.7	0.004
B-2A	1	a	4.3	7.4	0.042
	10	b	16	8.0	0.131
	18	c	18.6	-	-
	21	d	1.7	7.4	0.017

[a] pH was not measured, therefore un-ionized ammonia concentration could not be calculated. Note: all ammonia measurements taken using Nessler Method and HACH 2000.

Table 19a. Selected fluoride measurements taken on day 20 of the fathead minnow 21-day survival and growth tests conducted on test sediments (08/30/94).

Sample	Replicate	Fluoride (mg/L)
Control	a	0.177
	c	0.227
C-9	a	0.241
	c	0.208
B-2A	a	39.4
	c	39.0

Table 19b. Selected fluoride measurements taken on day 21 of the *Hexagenia limbata* 21-day survival and growth tests conducted on test sediments (08/30/94).

Sample	Replicate	Fluoride (mg/L)
Control	a	0.180
C-9	a	0.183
B-2A	a	41.5

Table 20. Concentrations of PCBs and PAHs in sediments, unfiltered elutriate and filtered elutriate samples tested for MFO induction.

Contaminant	B-2A Sediment (µg/g)	C-9 Sediment (µg/g)	B-2A Unfiltered Elutriate (µg/L)	B-2A Filtered (µg/L)	B-2A Filtered Elutriate[a] (µg/L)
Total PAHs	2320	17	697.9	588	109.9
Total PCBs	77.6	2.3	26.5	28.6	- 2.1

[a] difference between concentration measured in unfiltered elutriate and concentration retained on filter paper.

Table 21. Total PAH concentrations and BaP equivalents for B-2 sediment and filtered B-2A elutriate. All values are µg/g dry weight except where otherwise noted.

Sample	B-2A	B-2A	B-2B	B-2B	C-9	B-2A Filt.Elt.[a]
Date Analyzed	11/11/94	29/08/94	29/08/94	20/12/94	30/08/94	19/09/94
Total PAHs	3447.72	2320.34	2430.94	2346.17	17.39	588.39
BaP Equivalents	643.28	430.34	409.95	458.92	4.11	144.74

[a] filtered elutriate (µg/L)

Table 22. Summary of rainbow trout and fathead minnow 21 d bioassays using disturbed and undisturbed B-2 sediment samples.

Test Organism	Sample	Overall Survival (%)	Average weight change (mg)
Fathead minnows	B-2A Undisturbed	68	- 105.5
	B-2A Disturbed	0	n/a
	Long Point Control	98	23.7
Rainbow trout	B-2B Undisturbed	73	88.3
	B-2A Disturbed	n/t[a]	n/t
	Long Point Control	95	154

[a] not tested (see text).

Table 23. Selected ammonia measurements taken during the rainbow trout 21 d survival and growth tests conducted on undisturbed test sediments (10/20/94).

Sample	Day	Replicate	New/Old[a]	Total Ammonia (mg/L)	pH	Un-ionized Ammonia (mg/L)
B-2B Undisturbed	1	a	N	0.25	7.8	0.005
	14	b	N	0.39	7.0	0.001
			O	6.41	7.8	0.113
	21	c	O	4.37	7.7	0.064
Long Point	1	a	N	0	7.7	0
	14	b	N	0.1	7.0	0.0003
			O	1.3	7.9	0.029
	21	c	O	0.21	7.9	0.005

[a] new or old solution.
Note: all ammonia measurements taken using Nessler Method and HACH 2000.

Table 24. Selected ammonia measurements taken during the fathead minnow 21 d survival and growth tests conducted on disturbed and undisturbed test sediments (10/20/94).

Sample	Day	Replicate	Total Ammonia (mg/L)	pH	Un-ionized Ammonia (mg/L)
B2-A Disturbed	5	b	13.7	-	-[a]
	7	c	21.8	7.9	0.67
	9	c	24	8.1	1.14
	10	d	21.8	7.9	0.67
	15	d	29.3	8.1	1.39
	18	b	29.5	8.2	1.74
		c	30	8.2	1.77
	20	n/a[b]	-	-	-
B2-A Undisturbed	5	a	14.5	-	-
	7	c	22	7.8	0.54
	9	d	22	8.1	1.04
	10	d	22	7.8	0.54
	15	d	27.3	8.1	1.29
	18	c	28	8.2	1.65
		d	28.8	8.2	1.70
	20	a	29.3	7.9	0.89
Control	5	a	7.5	-	-
	7	c	11.3	7.6	0.18
	9	c	10.8	7.6	0.17
	10	d	11.3	7.9	0.34
	15	d	11.8	7.6	0.18
	18	d	7.7	7.6	0.12
	20	a	9.25	7.4	0.09

[a] pH was not measured, therefore un-ionized ammonia concentration could not be calculated.
[b] complete mortality.
Note: all ammonia measurements taken using Nessler Method and HACH 2000.

Table 25a. Selected fluoride measurements taken on day 20 of the fathead minnow 21 d survival and growth tests conducted on disturbed and undisturbed test sediments (10/20/94).

Sample	Replicate	Fluoride (mg/L)
Long Point Control	b	0.194
	d	0.208
B-2A Undisturbed	b	38.6
	d	43.3
B-2A Disturbed	b	41.1
	d	41.1

Table 25b. Selected fluoride measurements taken on day 22 of the rainbow trout 21 d survival and growth tests conducted on disturbed and undisturbed test sediments (10/20/94).

Sample	Replicate	Fluoride (mg/L)
Long Point Control	a	0.147
	b	0.155
B-2B Undisturbed	a	20.1
	b	21.2

Table 26. TSS measurements taken on day 21 of the fathead minnow 21 d survival and growth tests conducted on disturbed and undisturbed test sediments (10/20/94).

Treatment	Replicate	TSS (mg/L)
Control	B	257
	D	273
B2-A Undisturbed	B	38
	D	18
B2-A Disturbed	B	7
	D	3

Table 27. Estimation of the percentage of available substance observed in unfiltered elutriate.

Substance	Measured Sediment Concentration (µg/g)	Theoretical Amount Available to Enter (µg)[a]	Unfiltered Elutriate Concentration (µg/L)	% Available Substance in Unfiltered Elutriate[b]
PCBs	77.6	3825.7	26.5	0.7%
PAHs	2320.3	114,392	697.9	0.6%
fluoride	1160	57,188	23,300	41%
cyanide	27.8	1370.5	29	2%
aluminum	126,000	6,211,800	15,000	0.2%

[a] measured sediment concentration x 49.3 g dry sediment (see text).
[b] theoretical amount available ÷ measured elutriate concentration.
[c] extractable.

Table 28a. Influence of ammonia on elutriate toxicity to *Daphnia magna*. Ranges of un-ionized ammonia are based on pH measured at the start and end of testing and total ammonia levels of 10.8 mg/L and 1.6 mg/L in the raw and zeolite-treated samples, respectively (see Appendix IX for raw data).

	Raw Elutriate		Zeolite - Treated Elutriate	
Elutriate Concentration	Mortality (%)	Un-ionized ammonia (mg/L)	Mortality (%)	Un-ionized ammonia (mg/L)
100	100 *	0.13 - 0.98	100	0.04 - 0.14
50	100	0.08 - 0.49	100 *	0.02 - 0.07
25	100	0.05 - 0.24	100	0.01 - 0.04
13	100	0.02 - 0.06	100	< 0.02
6	100	- [a]	100	-
0	0	< 0.02	0	< 0.02

* 0 mortality, but 100% immobility.
[a] pH not measured.

Table 28b. Influence of ammonia on elutriate toxicity to fathead minnows. Ranges of un-ionized ammonia are based on pH measured at the start and end of testing and total ammonia levels of 10.8 mg/L and 1.6 mg/L in the raw and zeolite-treated samples, respectively (see Appendix IX for raw data).

	Raw Elutriate		Zeolite - Treated Elutriate	
Elutriate Concentration	Mortality (%)	Un-ionized ammonia (mg/L)	Mortality (%)	Un-ionized ammonia (mg/L)
100	100	0.13 - 0.98	40	0.04 - 0.14
50	60	0.08 - 0.49	20	0.02 - 0.07
25	0	0.05 - 0.24	0	0.01 - 0.04
13	0	0.03 - 0.12	0	< 0.02
6	0	0.02 - 0.06	0	< 0.02
0	0	< 0.02	0	< 0.02

Table 29. *Daphnia magna* LC_{50} and EC_{50} values (%) for B-2 elutriate samples subjected to Toxicity Identification Evaluation treatments.

Test	48 hour LC_{50} (%) (95% confidence limits)	48 hour EC_{50} (%) (95 % confidence limits)
Baseline pH 7.8	nd	<6%
Filtered pH 3	0 *	40 **
Filtered pH 8	0 *	100 **
C-18 pH 3	74.9 (43 - 100)	27.1 (14.4 - 51.9)
C-18 pH 8	54.9 (26 - 100)	8.8 (6 - 13)
C-18 pH 11	24.4 (14.2 - 41.8)	13.8 (5.9, 24.6)
Anion	non-lethal	non-lethal
Cation	nd	10.2 (6 - 13)
Zeolite	nd	4.9 (0 - 16.3)
Carbon	24.4 (14.2 - 41.8)	10.2 (6 - 13)
XAD	28.4 (14.7 - 57.1)	10.2 (6 - 13)

nd = no dose response.
* % dead in 100% exposure concentration.
** % immobile in 100 % exposure concentration.

Table 30. Summary of fluoride (as NaF) aqueous exposures using fathead minnows, *Daphnia magna, Hyalella azteca, Hexagenia limbata,* and *Chironomus tentans.*

Species Tested	Test Duration (hours)	LC_{50} (mg/L) (95 % confidence limits)
Fathead minnow (juveniles)	96	282.8 (200-400)
Fathead minnow (10 day old)	96	262.4 (200-400)
Daphnia magna	48	282.8 (200-400)
Hyalella azteca	48	14.6 (12.5-25)
Hexagenia limbata	96	32.3 (10.3-51.6)
Chironomus tentans	96	124.1 (91.6-152.9)

Table 31. Summary of fluoride spiked sediment toxicity tests.

Test Organism	Test Type	LC_{50} (µg/g)	IC_{25} (µg/g)	95 % confidence limits (µg/g)
Hexagenia limbata	survival	1652.2	-	1338.6 - 2059.7
	growth	-	1221.3	565.4 - 1856.2
Chironomus tentans	survival	>5600	-	-
	growth	-	661.4	471.2 - 1644.1
Hyalella azteca	survival	1114.6	-	700 - 1400
	growth	-	290.2	153.2 - 416.1
Fathead minnows	survival	>5600	-	-
	growth	-	>5600	-

Table 32. Concentrations of fluoride (as NaF) in overlying water taken during *Hexagenia limbata* spiked sediment tests at 0 and 12 days.

Concentration spiked into sediment (µg/g or ppm)	Concentration measured in overlying water (mg/L or ppm)			Conversion Factor[a] (Water to Sediment)
Day	0	12	21	
Control	0.169	0.247	<0.03	19 X
700	3.10	11.3	10.4	62 X
1400	3.26	13.7	14.0	102 X
2800	7.89	24.9	26.8	112 X
5600	17.7	56.8	60.9	98 X

[a] based on day 12 concentrations

FIGURE CAPTIONS

Figure 1. Location of the Reynolds Metals Company aluminum production plant on the St. Lawrence River at Massena, New York, showing the area of contaminated river sediment to be dredged. Locations of the two other Superfund sites, i.e., the General Motors Foundry (GM) and the ALCOA aluminum smelter, are also shown.

Figure 2. Locations of the test sites relative to concentrations of total PCBs (μg/g dry weight) in surface sediment. Site B-2 is the "contaminated" site and site C-9 is the "field control." Locations of other sites that were sampled in 1993 are also shown.

Figure 3. Comparison of concentrations of major contaminants in sediment collected from site C-9 in 1993 and 1994.

Figure. 4. Comparison of concentrations of major contaminants in sediment collected from site B-2 in 1993 and 1994. Values are averages for sites B-2 and B2-E in 1993, and sites B2-A and B2-B in 1994.

Figure 5. MFO induction in rainbow trout exposed to methanol extracts of sediment collected from site B-2 in 1993. Five dilutions representing nominal concentrations of 10 to 1000 µg/L PAHs were tested.

Figure 6. MFO induction in rainbow trout exposed to disturbed sediment, undisturbed sediment and elutriate from site B-2 and disturbed sediment from site C-9 (field control).

Figure 7. Toxicity of sediment-associated fluoride to four species of freshwater benthic organisms relative to environmentally realistic exposure levels, using two endpoints: LC_{50} for survival and IC_{25} for growth.

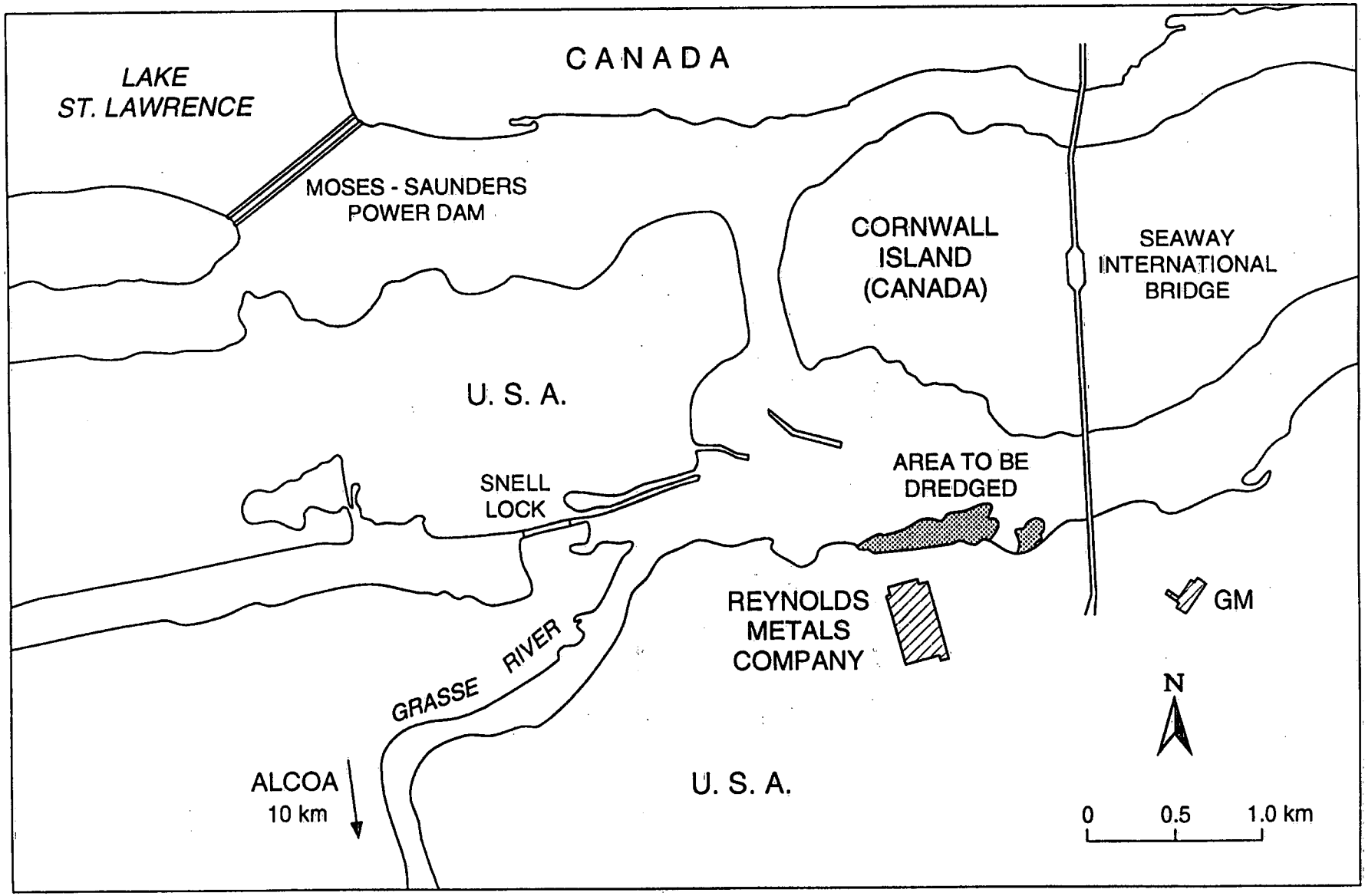
LAKE
ST. LAWRENCE
CANADA
MOSES - SAUNDERS
POWER DAM
CORNWALL
ISLAND
(CANADA)
SEAWAY
INTERNATIONAL
BRIDGE
U. S. A.
SNELL
LOCK
AREA TO BE
DREDGED
REYNOLDS
METALS
COMPANY
GM
GRASSE RIVER
N
ALCOA
10 km
U. S. A.
0
0.5
1.0 km

Figure 1.

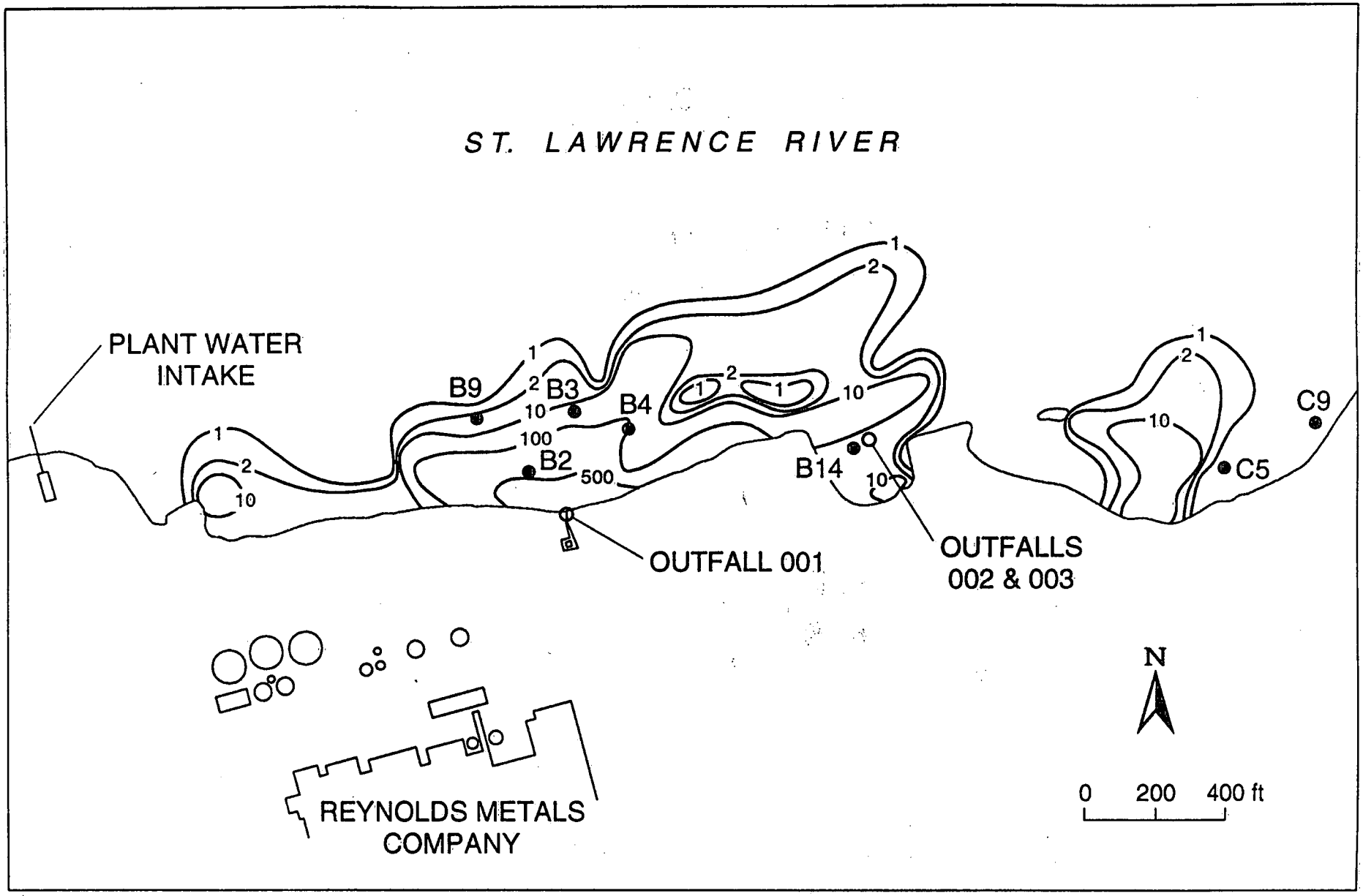
ST. LAWRENCE RIVER
PLANT WATER INTAKE
B9
B3
B4
B2
B14
C9
C5
1
2
10
100
500
OUTFALL 001
OUTFALLS 002 & 003
REYNOLDS METALS COMPANY
N
0 200 400 ft

Figure 2.

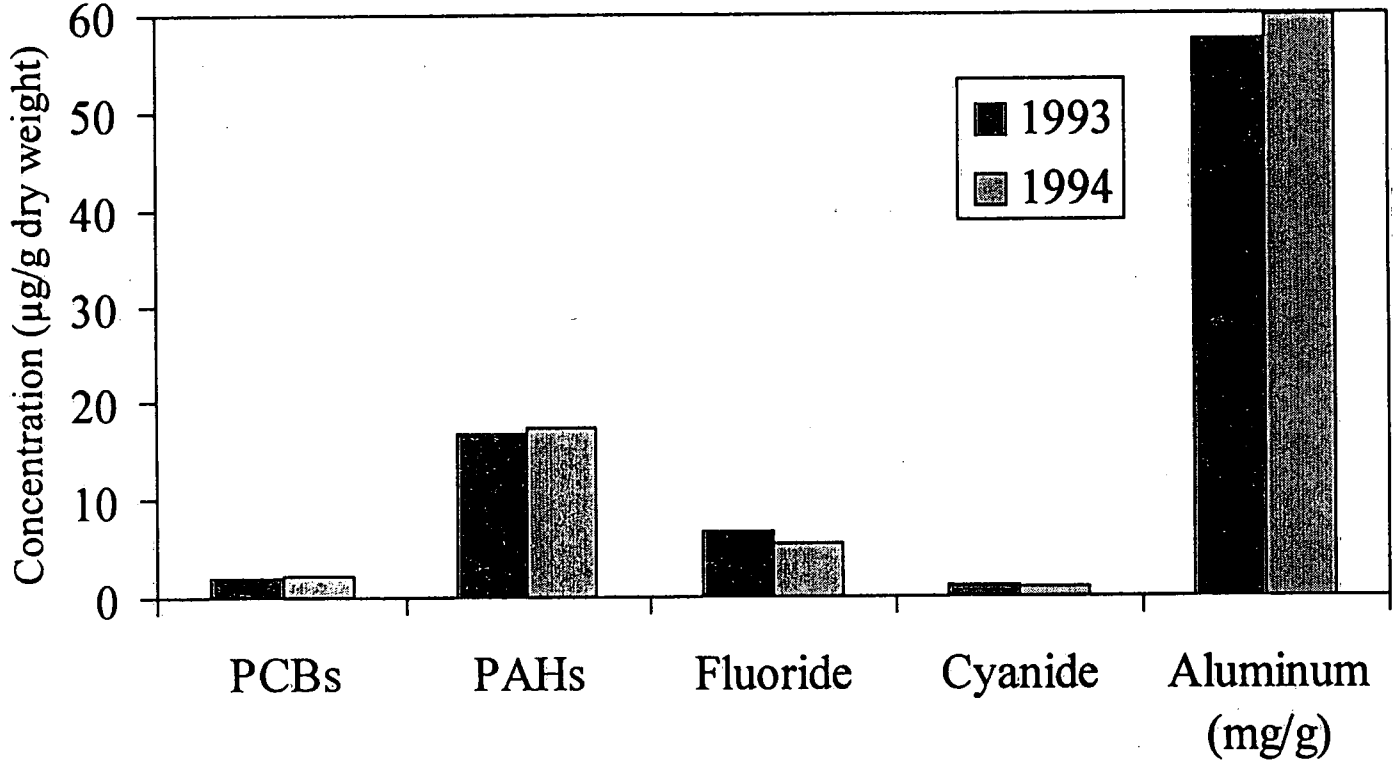
60
50
40
30
20
10
0
Concentration (µg/g dry weight)
1993
1994
PCBs
PAHs
Fluoride
Cyanide
Aluminum (mg/g)

Figure 3.

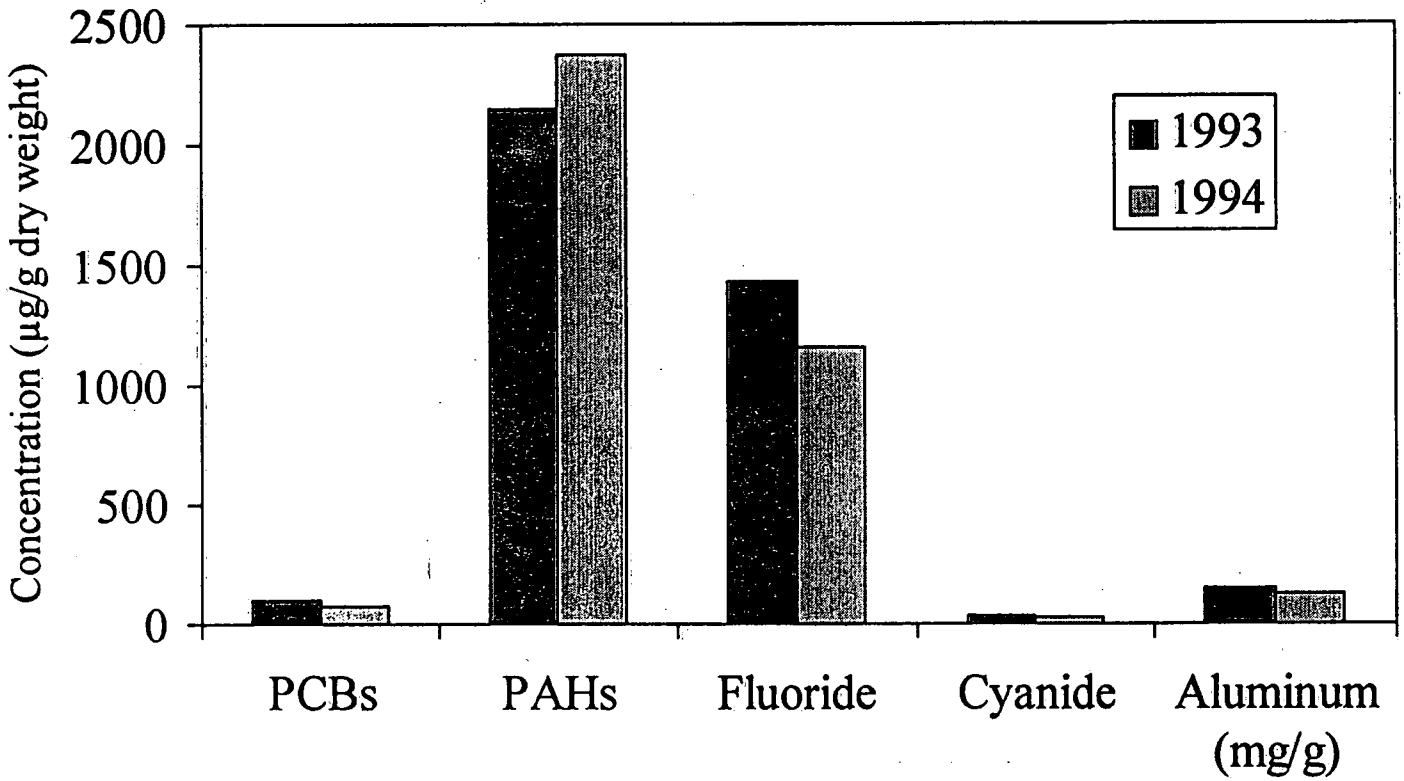
2500
2000
1500
1000
500
0
Concentration (µg/g dry weight)
1993
1994
PCBs
PAHs
Fluoride
Cyanide
Aluminum (mg/g)

Figure 4.

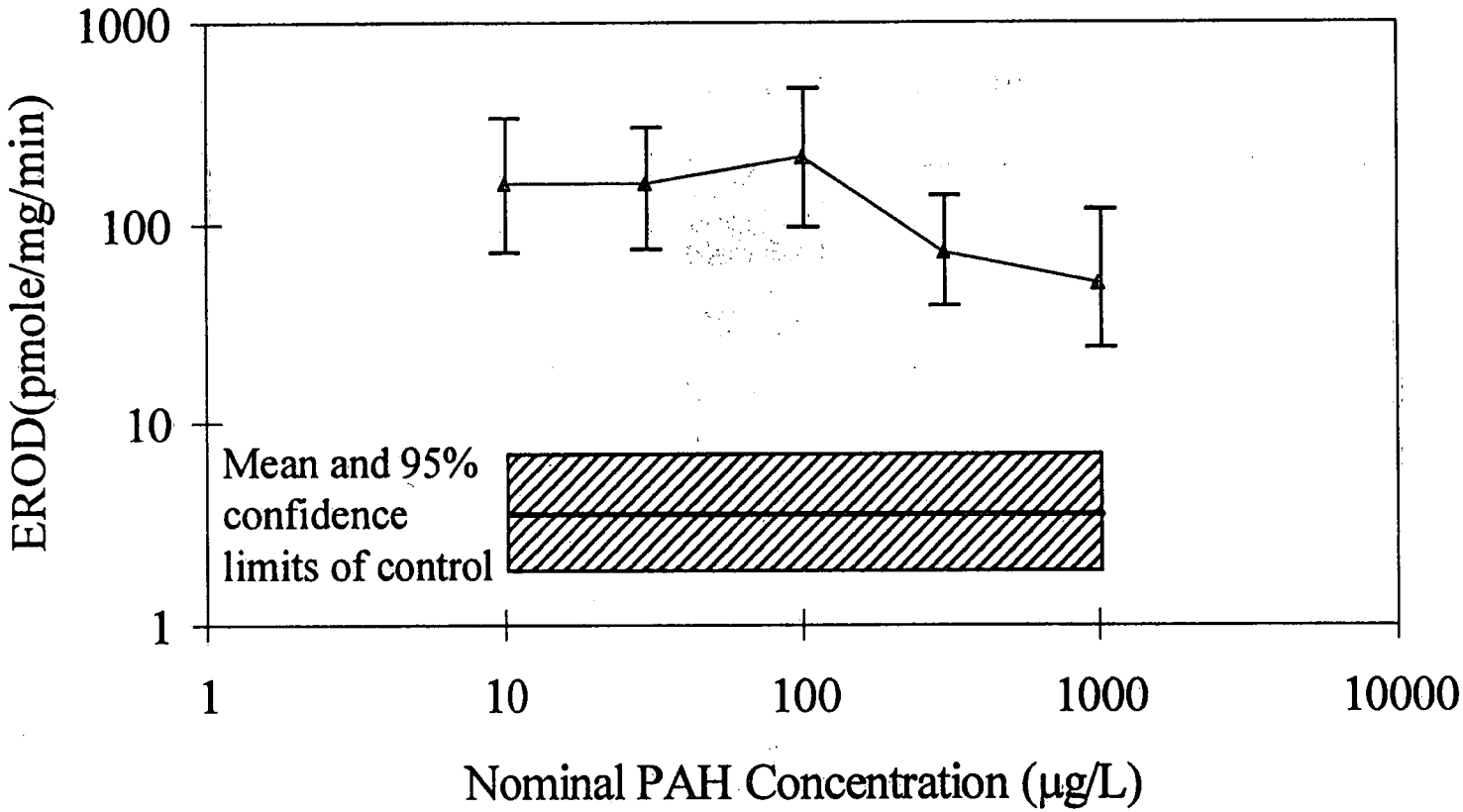
1000
100
10
1
EROD(pmole/mg/min)
Mean and 95%
confidence
limits of control
1
10
100
1000
10000
Nominal PAH Concentration (µg/L)

Figure 5.

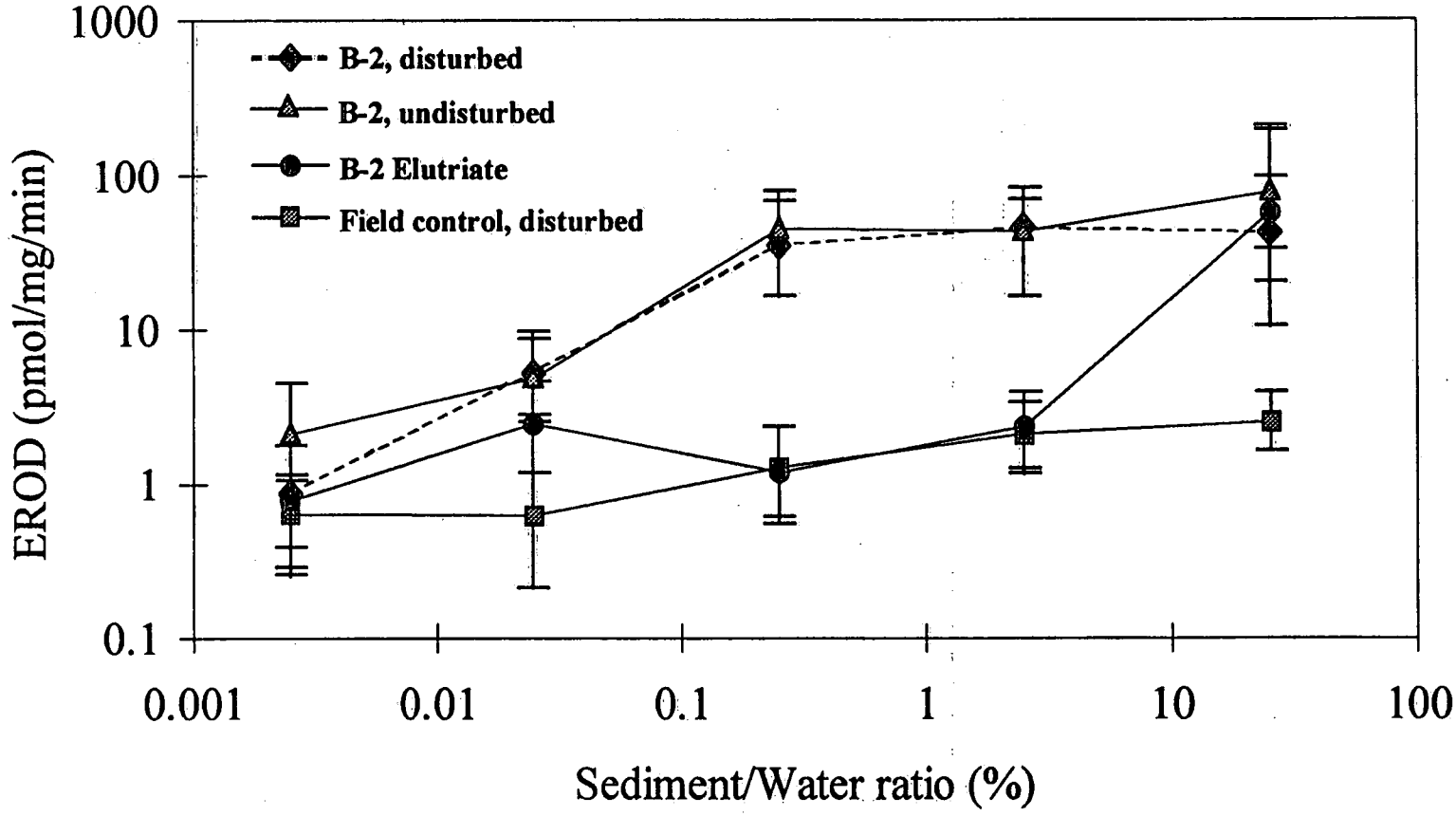
1000
100
10
1
0.1
EROD (pmol/mg/min)
B-2, disturbed
B-2, undisturbed
B-2 Elutriate
Field control, disturbed
0.001
0.01
0.1
1
10
100
Sediment/Water ratio (%)

Figure 6.

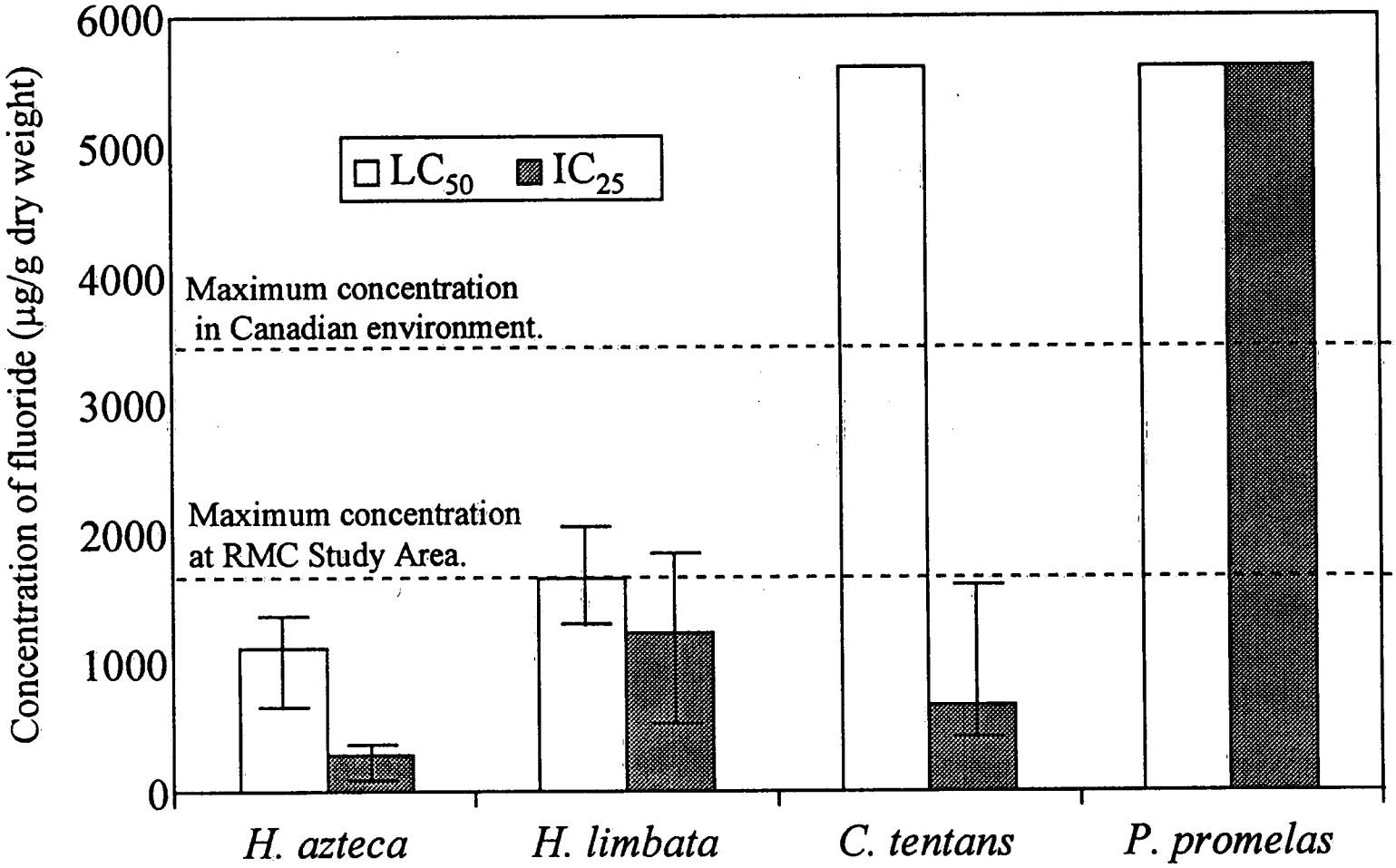
6000
5000
4000
3000
2000
1000
0
Concentration of fluoride (µg/g dry weight)
LC50
IC25
Maximum concentration in Canadian environment.
Maximum concentration at RMC Study Area.
H. azteca
H. limbata
C. tentans
P. promelas

Figure 7.

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867 Lakeshore Rd., Burlington, Ontario L7R 4L7

Appendix I. PAHs and PCBs in sediment and filter papers.

WTC LABORATORY
REPORT OF ANALYSIS

NWRI
Attention: Janice Smith
CCIW

WTC Group #: 3802
Reported: 03/21/95

P.O.#: 677650 & 678132

WTC Sample #: / Client Identification: / Date Received: / Parameter	Units	95-00077 B2A 01 4-11-94 01/05/95 Result	MDL	95-00078 B2A 02 29-08-94 01/05/95 Result	MDL	95-00079 B2B 01 29-08-94 01/05/95 Result	MDL	95-00080 B2B 02 20-12-94 01/05/95 Result	MDL
Naphthalene	ug/g	1.22	0.3	0.82	0.3	0.68	0.3	0.85	0.3
Acenaphthylene	ug/g	0.26t	0.3	0.17t	0.3	0.16t	0.3	0.19t	0.3
Acenaphthene	ug/g	2.17	0.3	2.03	0.3	1.62	0.3	1.54	0.3
Fluorene	ug/g	1.47	0.3	1.32	0.3	1.08	0.3	1.19	0.3
Phenanthrene	ug/g	43.2	0.3	37.1	0.3	32.8	0.3	34.0	0.3
Anthracene	ug/g	20.0	0.3	15.3	0.3	11.2	0.3	11.2	0.3
oranthene	ug/g	323	0.2	241	0.2	265	0.2	244	0.2
rene	ug/g	260	0.3	197	0.3	219	0.3	195	0.3
Benzo(a)anthracene	ug/g	281	0.3	184	0.3	191	0.3	187	0.3
Chrysene	ug/g	814	0.2	521	0.2	562	0.2	535	0.2
Benzo(b)fluoranthene	ug/g	912	0.3	607	0.3	586	0.3	607	0.3
Benzo(k)fluoranthene	ug/g	155	0.2	109	0.2	129	0.2	104	0.2
Benzo(a)pyrene	ug/g	204	0.2	135	0.2	132	0.2	135	0.2
Indeno(1,2,3-c,d)pyrene	ug/g	164	0.3	102	0.3	116	0.3	108	0.3
Dibenzo(a,h)anthracene	ug/g	55.4	0.2	37.6	0.2	33.4	0.2	43.2	0.2
Benzo(g,h,i)perylene	ug/g	211	0.3	130	0.3	150	0.3	139	0.3
Surrogates:									
Naphthalene-d8	% Rec	79		69		72		82	
Acenaphthene-d10	% Rec	84		75		81		84	
Fluorene-d10	% Rec	76		76		82		86	
Phenanthrene-d10	% Rec	97		94		103		110	
Pyrene-d10	% Rec	73		80		72		77	
Chrysene-d12	% Rec	68		65		50		75	

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: Natureia Dellotte
Validated by: Natureia Dellotte
Brian MacGillivray
Head, Mass Spectrometry

Format: WTC4

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 #: 3802
Reported: 03/21/95

P.O.#: 677650 & 678132

Parameter	Units	WTC Sample #: 95-00081 Client Identification: C90 30.08.94 Date Received: 01/05/95 Result	MDL	95-00082 B2A 19.09.94 01/05/95 FILTERS Result	MDL
Naphthalene	ug/g	0.14t	0.3	0.79t	3.0
Acenaphthylene	ug/g	0.04t	0.3	0.20t	1.8
Acenaphthene	ug/g	0.02t	0.3	0.32t	2.2
Fluorene	ug/g	0.03t	0.3	0.88t	2
Phenanthrene	ug/g	0.39	0.3	16.5	2.2
Anthracene	ug/g	w	0.3	5.58	1.8
·ıoranthene	ug/g	0.90	0.2	233	1.5
,rene	ug/g	0.78	0.3	210	2.2
Benzo(a)anthracene	ug/g	1.21	0.3	217	2.2
Chrysene	ug/g	2.64	0.2	435	1
Benzo(b)fluoranthene	ug/g	5.07	0.3	946	2.2
Benzo(k)fluoranthene	ug/g	1.70	0.2	143	1
Benzo(a)pyrene	ug/g	0.99	0.2	218	1.5
Indeno(1,2,3-c,d)pyrene	ug/g	1.33	0.3	192	1.8
Dibenzo(a,h)anthracene	ug/g	0.43	0.2	69.7	1.5
Benzo(g,h,i)perylene	ug/g	1.72	0.3	254	1.8
Surrogates:					
Naphthalene-d8	% Rec	69		65	
Acenaphthene-d10	% Rec	75		69	
Fluorene-d10	% Rec	76		73	
Phenanthrene-d10	% Rec	94		79	
Pyrene-d10	% Rec	80		83	
Chrysene-d12	% Rec	65		88	

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: Patricia Villette
Validated by: Patricia Villette
Brian MacGillivray
Head, Mass Spectrometry

Format: WTC4

Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.

WTC LABORATORY
QA/QC DATA REPORT

NWRI

WTC Group #: 3802

QC Type	Parameter	Matrix	Actual	Found	Percent
PLICATE	Acenaphthene	SOLID	0.02t	0.02t	
	Acenaphthylene	SOLID	0.04t	0.04t	
	Anthracene	SOLID	w	0.09t	
	Benzo(a)pyrene	SOLID	0.99	1.52	42.2
	Benzo(a)anthracene	SOLID	1.21	1.47	19.4
	Benzo(b)fluoranthene	SOLID	5.07	6.00	16.8
	Benzo(g,h,i)perylene	SOLID	1.72	2.43	34.2
	Benzo(k)fluoranthene	SOLID	1.70	1.89	10.6
	Chrysene	SOLID	2.64	3.21	19.5
	Dibenzo(a,h)anthracene	SOLID	0.43	0.67	43.6
	Fluoranthene	SOLID	0.90	0.95	5.4
	Fluorene	SOLID	0.03t	0.03t	
	Indeno(1,2,3-c,d)pyrene	SOLID	1.33	1.91	35.8
	Naphthalene	SOLID	0.13t	0.14t	
	Phenanthrene	SOLID	0.39t	0.47	
	Pyrene	SOLID	0.78	0.85	8.6
	Total PCB's	SOLID	75100	72100	4.1
METH-BLANK	Acenaphthene	SOLID	0.0	w	
	Acenaphthylene	SOLID	0.0	w	
	Anthracene	SOLID	0.0	w	
	Benzo(a)pyrene	SOLID	0.0	0.02t	
	Benzo(a)anthracene	SOLID	0.0	0.02t	
	Benzo(b)fluoranthene	SOLID	0.0	0.07t	
	Benzo(g,h,i)perylene	SOLID	0.0	0.06t	
	Benzo(k)fluoranthene	SOLID	0.0	0.05t	
	Chrysene	SOLID	0.0	0.04t	
	Dibenzo(a,h)anthracene	SOLID	0.0	0.03t	
	Fluoranthene	SOLID	0.0	0.03t	
	Fluorene	SOLID	0.0	0.02t	
	Indeno(1,2,3-c,d)pyrene	SOLID	0.0	0.05t	
	Naphthalene	SOLID	0.0	0.08t	
	Phenanthrene	SOLID	0.0	0.06t	
	Pyrene	SOLID	0.0	0.01t	
	Total PCB's	SOLID	0.0	w	
SPIKED-BLK	Total PCB's	SOLID	625	562	89.9
SURR-BLANK	Acenaphthene-d10	SOLID	100	79	79.0
	Fluorene-d10	SOLID	100	105	105.0
	Phenanthrene-d10	SOLID	100	94	94.0
	Pyrene-d10	SOLID	100	86	86.0
	Chrysene-d12	SOLID	100	63	63.0
	Naphthalene-d8	SOLID	100	77	77.0
SURR-DUP	Acenaphthene-d10	SOLID	100	82	82.0
	Fluorene-d10	SOLID	100	86	86.0
	Phenanthrene-d10	SOLID	100	97	97.0
	Pyrene-d10	SOLID	100	85	85.0
	Chrysene-d12	SOLID	100	77	77.0
	Naphthalene-d8	SOLID	100	80	80.0
SURR-REF	Acenaphthene-d10	SOLID	100	87	87.0
	Fluorene-d10	SOLID	100	86	86.0
	Phenanthrene-d10	SOLID	100	90	90.0
	Pyrene-d10	SOLID	100	89	89.0
	Chrysene-d12	SOLID	100	83	83.0
	Naphthalene-d8	SOLID	100	81	81.0

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.
IKE: A sample with a known addition of analyte.
IKE-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.

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Sample ID	95-00077	95-00078	95-00079	95-00080	95-00080 Dup	95-00081	95-00082
PCB Congener nber							
004+010	0	0	0	0	0	54	0
007	0	0	0	0	0	2	0
006	0	0	0	0	0	20	0
008+005	256	312	291	275	268	91	0
019	106	128	119	115	115	16	0
018	654	830	596	584	569	66	590
017	121	160	145	140	140	36	241
024+027	44	52	48	45	46	10	0
016/032	461	505	482	455	453	41	594
026	276	264	250	247	234	43	288
025	0	0	0	0	0	1	0
031+028	2309	2380	2278	2209	2143	175	2496
021/033/053	528	560	529	509	498	37	455
022	88	109	104	103	94	5	131
045	42	53	53	57	56	6	130
046	73	81	74	71	71	6	290
052/043	1939	1889	1798	1821	1728	132	3284
049	864	836	800	812	758	73	1559
047+048	699	709	673	687	644	71	1272
044	1438	1416	1370	1365	1270	75	2478
042	265	263	254	254	235	21	447
041+071/041	887	897	860	865	809	64	1571
1040	134	148	141	140	131	12	221
063	0	0	0	0	0	0	0
074	192	68	69	53	51	14	167
070+076	0	0	0	0	0	0	0
066/095	7058	6613	6458	6432	6084	293	13146
091	247	304	341	227	277	31	636
056+060	590	610	602	577	555	33	788
089	0	0	0	0	0	0	0
084	366	129	117	113	124	54	710
101	2374	2046	1991	1966	1939	48	4656
099	307	245	241	242	231	17	512
083	32	48	47	59	44	6	0
097	193	209	202	205	195	14	327

Wastewater Technology Centre
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Sample ID	95-00077	95-00078	95-00079	95-00080	95-00080 Dup	95-00081	95-00082
PCB Congener nber							
081/087	359	364	363	356	352	19	744
085	44	49	49	52	48	9	160
136	1185	1112	1067	1058	1068	15	2907
077/110	1659	1589	1523	1538	1470	86	3379
082	134	151	168	164	147	15	0
151	484	713	643	642	637	16	677
144+135	2863	2898	2723	2772	2715	21	5414
107	60	199	123	134	148	1	0
149/118	5111	4598	4337	4421	4281	81	10438
134+114	447	1287	452	586	596	21	971
146	2840	2825	2315	2373	2344	27	5189
153+132	7227	6279	6010	6090	5891	74	13301
141	1285	1228	1168	1176	1163	7	2367
137	91	0	0	0	0	0	0
176/130	867	789	764	933	767	8	1577
138	3511	3196	3082	3229	3070	30	6328
158	0	0	0	0	0	0	0
178/129	836	792	747	798	783	13	1665
175	0	0	0	0	0	0	0
187+182	3046	2658	2533	2598	2601	32	5650
183	1994	2035	1890	1954	1992	16	3891
128 185/ :/167	217 176	277 440	192 358	228 387	287 439	7 4	627 602
174	2973	2694	2530	2802	2609	22	5748
177	877	804	746	770	778	10	1596
171/156	639	571	423	787	545	7	1105
173	58	36	275	0	0	0	0
172+197	840	665	531	645	685	5	1558
180	7812	7079	6688	7011	6838	55	12649
199	132	163	94	176	187	2	329
170+190	3810	3391	3235	3375	3273	33	5375
201	1796	1681	1565	2176	1641	19	2654
203+196	1595	1519	1451	1552	1482	15	2334
195	1549	1785	1679	1672	1747	14	3821
194	1797	1369	1289	1463	1313	15	2719
206	2154	468	463	506	454	8	643
TOTAL PCB	83011	77568	72409	75052	72113	2274	143407

Analyzed by: L. Laird

Validated by: B Hong-Yu

Appendix II. Raw data from 21 d fathead minnow and *Hexagenia limbata* bioassays on B-2A sediment collected in August 1994.

Results of fathead minnow 21 d survival and growth tests conducted on test sediments (08/30/94).

Sample	Replicate	Number of Survivors	Initial Weight (mg)		Final Weight (mg)		Weight Gain (mg)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
B2-A	A	6	242.8	229.7	190.5	187.1	- 52.3	- 42.6
	B	7	230.7		201.5		- 29.2	
	C	10	218.6		172.2		- 46.4	
	D	7	226.6		184.1		- 42.5	
C9	A	10	245.4	224.4	242.8	222.7	- 2.6	- 1.7
	B	10	209.8		213.4		3.6	
	C	10	217.4		209.8		- 7.6	
	D	10	224.9		224.9		0.0	
Control	A	10	256.1	230.6	243.6	222.5	- 12.5	- 8.2
	B	10	228.3		224.7		- 3.6	
	C	10	209.4		206.0		- 3.4	
	D	10	228.7		215.6		- 13.1	

Appendix II. (Continued)

Results of *Hexagenia limbata* 2 d survival and growth tests conducted on test sediments (09/01/94).

Sample	Replicate	Number of Survivors	Initial Weight (mg)		Final Weight (mg)		Weight Gain (mg)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
B2-A	A	4	59.2	56.9	91.2	79.9	32.0	23.0
	B	5	64.8		82.0		17.2	
	C	5	44.5		79.1		34.6	
	D	3	59.2		67.4		8.2	
C9	A	10	59.0	59.3	85.6	86.7	26.6	27.4
	B	10	64.4		86.6		22.2	
	C	10	57.5		91.0		33.5	
	D	10	56.3		83.5		27.2	
Control	A	10	70.6	56.8	98.6	86.0	28.0	29.1
	B	9	52.3		98.3		46.0	
	C	10	55.5		63.4		7.9	
	D	10	48.9		83.5		34.6	

Appendix III. MFO data.

BAR Sediments sep 23 -27

Concentration	SITE	TYPE	Rep	EROD	EROD+0.2	logEROD
0.0001	B	D	1	1.202	1.40219604	0.1468087
0.0001	B	D	1	2.270	2.47036718	0.3927615
0.0001	B	D	1	1.355	1.55534592	0.191827
0.0001	B	D	1	0.796	0.996316202	-0.001603
0.0001	B	D	1	1.299	1.4990734	0.1758229
0.0001	B	D	2	0.576	0.776020404	-0.110127
0.0001	B	D	2	0.355	0.55481973	-0.255848
0.0001	B	D	2	0.477	0.677429828	-0.169136
0.0001	B	D	2	0.949	1.14913244	0.0603701
0.0001	B	E	1	1.734	1.93374012	0.2863981
0.0001	B	E	1	0.540	0.739612763	-0.130996
0.0001	B	E	1	1.339	1.53937715	0.187345
0.0001	B	E	1	1.687	1.88716823	0.2758106
0.0001	B	E	1	1.264	1.46363136	0.1654317
0.0001	B	E	2	0.317	0.516995837	-0.286513
0.0001	B	E	2	0.185	0.385359748	-0.414134
0.0001	B	U	1	2.813	3.0134308	0.4790612
0.0001	B	U	1	2.682	2.88155277	0.4596266
0.0001	B	U	1	8.782	8.98159344	0.9533534
0.0001	B	U	1	19.341	19.5411835	1.2909509
0.0001	B	U	1	2.760	2.95983252	0.4712671
0.0001	B	U	2	0.766	0.966187481	-0.014939
0.0001	B	U	2	0.638	0.837677626	-0.076923
0.0001	B	U	2	1.083	1.28326801	0.1083174
0.0001	B	U	2	0.702	0.901808371	-0.044886
0.0001	B	U	2	1.089	1.2890425	0.1102672
0.0001	C	D	1	0.517	0.716945896	-0.144514
0.0001	C	D	1	0.537	0.736578254	-0.132781
0.0001	C	D	1	1.428	1.62807044	0.2116732
0.0001	C	D	1	0.874	1.07434178	0.0311425
0.0001	C	D	1	1.193	1.39312312	0.1439895
0.0001	C	D	2	0.410	0.609579483	-0.21497
0.0001	C	D	2	0.526	0.725868018	-0.139142
0.0001	C	D	2	0.254	0.4544706	-0.342494
0.001	B	D	1	6.268	6.46836197	0.8107943
0.001	B	D	1	28.148	28.3478341	1.4525199
0.001	B	D	1	9.887	10.086978	1.0037611
0.001	B	D	1	2.834	3.03418297	0.4820418
0.001	B	D	1	8.850	9.04951094	0.9566251
0.001	B	D	2	11.423	11.623022	1.0653191
0.001	B	D	2	2.012	2.21155505	0.3446978
0.001	B	D	2	2.760	2.95959885	0.4712328

Concentration	SITE	TYPE	Rep	EROD	EROD+0.2	logEROD
0.001	B	D	2	1.501	1.70059967	0.2306021
0.001	B	E	1	4.248	4.4483777	0.6482017
0.001	B	E	1	0.987	1.18728131	0.0745536
0.001	B	E	1	5.596	5.79627725	0.7631492
0.001	B	E	1	17.744	17.9443108	1.2539268
0.001	B	E	1	1.286	1.48563835	0.1719131
0.001	B	E	2	9.492	9.69242037	0.9864322
0.001	B	E	2	0.751	0.950975474	-0.021831
0.001	B	E	2	0.192	0.391842546	-0.406888
0.001	B	U	1	5.108	5.30755983	0.7248949
0.001	B	U	1	7.400	7.6000314	0.8808154
0.001	B	U	1	2.803	3.00305967	0.477564
0.001	B	U	1	3.327	3.52659327	0.5473554
0.001	B	U	1	6.759	6.95852324	0.8425171
0.001	B	U	2	4.375	4.57489686	0.6603813
0.001	B	U	2	2.293	2.49349858	0.3968091
0.001	B	U	2	23.631	23.8305052	1.3771332
0.001	B	U	2	2.225	2.42508666	0.3847273
0.001	C	D	1	0.389	0.589028172	-0.229864
0.001	C	D	1	0.453	0.653235693	-0.18493
0.001	C	D	1	0.229	0.42862811	-0.367919
0.001	C	D	1	0.541	0.741394086	-0.129951
0.001	C	D	1	10.192	10.3917682	1.0166895
0.001	C	D	2	0.241	0.440718323	-0.355839
0.001	C	D	2	0.340	0.539580151	-0.267944
0.01	B	D	1	40.579	40.785	1.6105056
0.01	B	D	1	16.192	16.3921401	1.2146357
0.01	B	D	1	48.515	48.7152278	1.6876647
0.01	B	D	1	70.178	70.3778866	1.8474362
0.01	B	D	1	20.388	20.588	1.3136056
0.01	B	D	2	15.198	15.3978943	1.1874613
0.01	B	D	2	31.101	31.3006132	1.4955528
0.01	B	D	2	186.618	186.817634	2.2714179
0.01	B	D	2	41.317	41.5172273	1.6182283
0.01	B	D	2	49.529	49.7289602	1.6966094
0.01	B	E	1	2.431	2.63145087	0.4201953
0.01	B	E	1	1.863	2.06299574	0.3144983
0.01	B	E	1	3.768	3.96817675	0.598591
0.01	B	E	1	1.643	1.84318382	0.2655686
0.01	B	E	2	0.476	0.675836016	-0.170159
0.01	B	E	2	0.454	0.65435152	-0.184189
0.01	B	E	2	2.090	2.28974539	0.3597872
0.01	B	E	2	0.687	0.887413073	-0.051874
0.01	B	E	2	0.545	0.745489399	-0.127559

Concentration	SITE	TYPE	Rep	EROD	EROD+0.2	logEROD
0.01	B	U	1	122.258	122.4577329	2.0879862
0.01	B	U	1	70.526	70.72580039	1.8495779
0.01	B	U	1	70.182	70.38207021	1.847462
0.01	B	U	1	73.181	73.38060338	1.8655813
0.01	B	U	1	118.599	118.79903	2.0748129
0.01	B	U	2	37.734	37.9340331	1.579029
0.01	B	U	2	74.536	74.7360571	1.8735302
0.01	B	U	2	21.168	21.3680663	1.3297652
0.01	B	U	2	71.105	71.3051866	1.8531211
0.01	B	U	2	16.749	16.9494341	1.2291552
0.01	C	D	1	1.114	1.31395984	0.1185821
0.01	C	D	1	2.505	2.70505428	0.432176
0.01	C	D	1	1.928	2.12783113	0.3279372
0.01	C	D	1	4.121	4.32074244	0.6355584
0.01	C	D	1	1.328	1.52820534	0.1841817
0.01	C	D	2	1.065	1.26485568	0.102041
0.01	C	D	2	1.326	1.52567313	0.1834615
0.01	C	D	2	0.565	0.764806709	-0.116448
0.1	B	D	1	51.559	51.7586695	1.7139831
0.1	B	D	1	36.764	36.96439766	1.5677836
0.1	B	D	1	47.585	47.78480855	1.6792899
0.1	B	D	1	46.709	46.90867483	1.6712532
0.1	B	D	1	45.568	45.76801619	1.6605621
0.1	B	D	2	80.146	80.3455734	1.904962
0.1	B	D	2	60.104	60.3035499	1.7803429
0.1	B	D	2	68.669	68.8685435	1.8380209
0.1	B	D	2	10.191	10.390859	1.0166515
0.1	B	D	2	40.605	40.8053859	1.6107175
0.1	B	E	1	2.465	2.66491033	0.4256826
0.1	B	E	1	50.254	50.4541389	1.7028968
0.1	B	E	1	7.851	8.05115098	0.905858
0.1	B	E	1	5.223	5.422995616	0.7342393
0.1	B	E	1	1.146	1.34616328	0.1290977
0.1	B	E	2	1.172	1.37173466	0.1372701
0.1	B	E	2	0.777	0.977176184	-0.010027
0.1	B	E	2	1.104	1.30420733	0.1153466
0.1	B	E	2	1.152	1.35189809	0.130944
0.1	B	E	2	0.616	0.816381992	-0.088107
0.1	B	U	1	25.423	25.6229746	1.4086295
0.1	B	U	1	101.044	101.244259	2.0053704
0.1	B	U	1	55.656	55.856	1.7470669
0.1	B	U	1	32.623	32.823	1.5161818
0.1	B	U	1	8.333	8.53293156	0.9310983

Concentration	SITE	TYPE	Rep	EROD	EROD+0.2	logEROD
0.1	B	U	2	56.681	56.880907	1.7549665
0.1	B	U	2	52.968	53.1675877	1.725647
0.1	B	U	2	101.814	102.014068	2.0086601
0.1	B	U	2	60.034	60.2337917	1.7798402
0.1	B	U	2	15.490	15.6900412	1.1956241
0.1	C	D	1	3.176	3.3757859	0.5283749
0.1	C	D	1	3.072	3.27164801	0.5147666
0.1	C	D	1	5.673	5.87341854	0.7688909
0.1	C	D	1	3.265	3.46492086	0.5396933
0.1	C	D	1	1.018	1.21760392	0.085506
0.1	C	D	2	2.611	2.81149647	0.4489375
0.1	C	D	2	0.687	0.887446432	-0.051858
0.1	C	D	2	0.867	1.06672684	0.0280532
0.1	C	D	2	0.494	0.694100969	-0.158577
0.1	C	D	2	7.954	8.15417758	0.9113802
1	B	D	1	40.321	40.5212571	1.6076829
1	B	D	1	61.589	61.7886327	1.7909086
1	B	E	1	42.397888	42.59788822	1.6293881
1	B	E	1	92.996	93.1957793	1.9693962
1	B	E	1	40.359207	40.55920702	1.6080895
1	B	E	1	55.040	55.2404958	1.7422576
1	B	U	2	56.506	56.7063962	1.753632
1	B	U	2	53.297	53.4969962	1.7283294
1	B	U	2	35.057	35.2570464	1.5472459
1	B	U	2	181.350	181.550065	2.2589964
1	B	U	2	83.866	84.0659451	1.9246201
1	C	D	1	2.747	2.9473725	0.469435
1	C	D	1	3.251	3.45140066	0.5379954
1	C	D	1	4.663	4.86339478	0.6869395
1	C	D	1	4.527	4.72677755	0.6745652
1	C	D	1	9.153	9.35283879	0.9709434
1	C	D	2	1.293	1.49343623	0.1741867
1	C	D	2	1.296	1.49569662	0.1748435
1	C	D	2	1.265	1.46509683	0.1658663
1	C	D	2	5.920	6.11989689	0.7867441
1	C	D	2	2.755	2.95512695	0.4705761
1	C	E	1	3.359	3.55925016	0.5513585
1	C	E	1	0.612	0.811800123	-0.090551
1	C	E	1	1.572	1.77249939	0.2485861
1	C	E	1	1.778	1.97835774	0.2963048
1	C	E	1	1.524	1.72422889	0.2365949
1	C	E	2	0.246	0.44578946	-0.35087
1	C	E	2	0.179	0.378926756	-0.421445
1	C	E	2	0.121	0.321252345	-0.493154
1	C	E	2	0.331	0.530728745	-0.275127

Concentration	SITE	TYPE	Rep	EROD	EROD+0.2	logEROD
1	C	U	1	2.188	2.38794589	0.3780245
1	C	U	1	7.773	7.97342029	0.9016447
1	C	U	1	3.466	3.66585111	0.5641748
1	C	U	1	1.359	1.55876803	0.1927815
1	C	U	1	10.342	10.5424386	1.0229411
1	C	U	2	2.343	2.54324778	0.4053887
1	C	U	2	2.168	2.36849524	0.3744725
1	C	U	2	4.273	4.47335849	0.6506337
1	C	U	2	0.766	0.966386664	-0.014849
1	L	D	1	3.181	3.38093439	0.5290367
1	L	D	1	0.475	0.674846213	-0.170795
1	L	D	1	0.546	0.74583571	-0.127357
1	L	D	1	0.803	1.00339316	0.0014711
1	L	D	1	1.691	1.89089395	0.2766672
1	L	D	2	1.043	1.24250098	0.0942967
1	L	D	2	0.722	0.921950366	-0.035292
1	L	E	1	0.445	0.645295324	-0.190241
1	L	E	1	0.740	0.940141503	-0.026807
1	L	E	1	0.445	0.645295324	-0.190241
1	L	E	1	0.390	0.589601256	-0.229442
1	L	E	1	0.363	0.563043204	-0.249458
1	L	E	2	0.396	0.596467995	-0.224413
1	L	E	2	0.420	0.620416722	-0.207317
1	L	E	2	0.172	0.372259976	-0.429154
1	L	U	1	0.517	0.716945896	-0.144514
1	L	U	1	0.254	0.4544706	-0.342494
1	L	U	1	1.428	1.62807044	0.2116732
1	L	U	2	0.598	0.797720219	-0.098149
1	L	U	2	2.579	2.77873336	0.4438469

Appendix IV. Survival data from 4 d MFO induction tests with rainbow trout. Tests conducted on B-2A and C-9 sediments (n=10; 2 replicates; 5 organisms per replicate).

Sample Type	Water : Sediment Ratio	Mortalities	
		Replicate 1 September 23, 1994	Replicate 2 September 30, 1994
B-2A Elutriate	4:1	0	1
	4:0.1	0	0
	4:0.01	0	0
	4:0.001	0	0
	4:0.0001	0	0
C-9 Elutriate	4:1	0	0
Long PointElutriate	4:1	0	0
Undisturbed B-2A	4:1	0	0
	4:0.1	0	0
	4:0.01	0	0
	4:0.001	0	0
	4:0.0001	0	0
Undisturbed C-9	4:1	0	0
Undisturbed Long Point	4:1	0	0
Disturbed B-2A	4:1	5	3
	4:0.1	0	0
	4:0.01	0	0
	4:0.001	0	0
	4:0.0001	0	0
Disturbed Long Point	4:1	0	0
Disturbed C-9	4:1	0	0
	4:0.1	0	0
	4:0.01	0	0
	4:0.001	0	0

Appendix Va. PAHs and PCBs in B-2 elutriate.

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 #: 4114
Reported: 04/17/95

P.O.#: 687818 Item #4

Parameter	Units	WTC Sample #: 95-02347 Client Identification: B2 elutriate UNFILTERED Date Received: 03/06/95 Result	MDL
Naphthalene	ug/L	0.27t	1.2
Acenaphthylene	ug/L	0.14t	1
Acenaphthene	ug/L	0.39t	0.9
Fluorene	ug/L	0.32t	0.9
Phenanthrene	ug/L	1.59	0.8
Anthracene	ug/L	w	0.9
uoranthene	ug/L	78.9	1
rene	ug/L	61.8	1
Benzo(a)anthracene	ug/L	50.2	1.2
Chrysene	ug/L	84.1	1
Benzo(b)fluoranthene	ug/L	209	1.2
Benzo(k)fluoranthene	ug/L	42.6	0.8
Benzo(a)pyrene	ug/L	48.2	1.2
Indeno(1,2,3-c,d)pyrene	ug/L	45.2	1.2
Dibenzo(a,h)anthracene	ug/L	14.7	1.4
Benzo(g,h,i)perylene	ug/L	60.5	1.4
		697.91	
Surrogates:			
Naphthalene-d8	% Rec	79	
Acenaphthene-d10	% Rec	70	
Fluorene-d10	% Rec	66	
Phenanthrene-d10	% Rec	79	
Pyrene-d10	% Rec	75	
Chrysene-d12	% Rec	96	

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: Patricia Dallotte

Validated by: Brian MacGillivray

Brian MacGillivray
Head, Mass Spectrometry

Format: WTC4

Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.

WTC LABORATORY
QA/QC DATA REPORT

NWRI

WTC Group #: 4114

Type	Parameter	Matrix	Actual	Found	Percent
METH-BLANK	Acenaphthene	WATER	0.0	w	
	Acenaphthylene	WATER	0.0	w	
	Anthracene	WATER	0.0	w	
	Benzo(a)pyrene	WATER	0.0	w	
	Benzo(a)anthracene	WATER	0.0	w	
	Benzo(b)fluoranthene	WATER	0.0	w	
	Benzo(g,h,i)perylene	WATER	0.0	w	
	Benzo(k)fluoranthene	WATER	0.0	w	
	Chrysene	WATER	0.0	w	
	Dibenzo(a,h)anthracene	WATER	0.0	w	
	Fluoranthene	WATER	0.0	0.19t	
	Fluorene	WATER	0.0	w	
	Indeno(1,2,3-c,d)pyrene	WATER	0.0	w	
	Naphthalene	WATER	0.0	w	
	Phenanthrene	WATER	0.0	w	
	Pyrene	WATER	0.0	w	
SURR-BLANK	Acenaphthene-d10	WATER	100	85	85.0
	Fluorene-d10	WATER	100	79	79.0
	Phenanthrene-d10	WATER	100	106	106.0
	Pyrene-d10	WATER	100	86	86.0
	Chrysene-d12	WATER	100	109	109.0
	Naphthalene-d8	WATER	100	51	51.0

TH-BLANK: A blank processed as a sample.
..PLICATE: 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.

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 #: 4113
Reported: 04/18/95

P.O.#: 687818 Item #4

WTC Sample#	Client Identification	Received	Parameter: Total PCB's Units: ug/L MDL: 0.2		
95-02346	B2 elutriate UNFILTERED	03/06/95	26.5		

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: M. Zumbrock

Validated by: R Hong-You

Robert Hong-You
Head, Chromatography Section

Format: WTC1

Wastewater Technology Centre
Operated by Rockcliffe Management Inc.

PCB Congener number	95-02346 ug/L
004+010	0.000
007	0.000
006	0.000
008+005	0.127
9	0.047
3	0.185
017	0.086
024+027	0.019
016/032	0.110
026	0.096
025	0.000
031+028	0.818
021/033/053	0.175
022	0.030
045	0.027
046	0.024
052/043	0.695
049	0.320
047+048	0.269
044	0.504
042	0.000
041+071/061	0.305
040	0.050
063	0.000
074	0.028
070+76+66/95	1.624
091	0.000
056+060	0.123
089	0.000
084	0.118
101	0.768
099	0.093
083	0.012
097	0.083
1/087	0.154
5	0.026
136	0.499
077/110	0.606
082	0.094
151	1.048
144+135	0.810
107	0.021
149/118	1.633
134+114	0.065
146	0.862
153+132	2.205
141	0.413
137	0.095
176/130	0.120
138	2.107
158	0.158
178/129	0.258
175	0.028
187+182	0.963
183	0.714
128	0.104
185/167	0.159
174	0.974
177	0.243
171/156	0.193
173	0.000
172+197	0.209
180	2.136
199	0.044
9+190	1.095
1	0.461
203+196	0.402
195	0.477
194	0.363
206	0.058
TOTAL PCB	26.530

Analyzed by : [signature] April 18/95.

Validated by : [signature]

Wastewater Technology Centre
Operated by Rockcliffe Research Management Inc.

WTC LABORATORY
QA/QC DATA REPORT

NWRI

WTC Group #: 4113

QC Type	Parameter	Matrix	Actual	Found	Percent
METH-BLANK	Total PCB's	WATER	w	w	
SPIKED-BLK	Total PCB's	WATER	615	408	66.3

METH-BLANK: A blank processed as a sample.
?LICATE: 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 Vb. **Concentrations of PAHs (µg/g dry weight) and BaP equivalents in sediment collected from site B-2 in August, 1994.**

PAH	TEF	11/11/1994 B2A-01	BAP EQUIV.	29/08/1994 B2A-02	BAP EQUIV.	29/08/1994 B2B-01	BAP EQUIV.
N	0.001	1.22	0.00122	0.82	0.00082	0.68	0.00068
AY	0.001	0.26	0.00026	0.17	0.00017	0.16	0.00016
AE	0.001	2.17	0.00217	2.03	0.00203	1.62	0.00162
FL	0.001	1.47	0.00147	1.32	0.00132	1.08	0.00108
PH	0.001	43.2	0.0432	37.1	0.0371	32.8	0.0328
AN	0.01	20	0.2	15.3	0.153	11.2	0.112
F	0.001	323	0.323	241	0.241	265	0.265
PY	0.001	260	0.26	197	0.197	219	0.219
BaA	0.1	281	28.1	184	18.4	191	19.1
CH	0.01	814	8.14	521	5.21	562	5.62
BbF	0.1	912	91.2	607	60.7	586	58.6
BkF	0.1	155	15.5	109	10.9	129	12.9
BaP	1	204	204	135	135	132	132
IP	0.1	164	16.4	102	10.2	116	11.6
DA	5	55.4	277	37.6	188	33.4	167
BP	0.01	211	2.11	130	1.3	150	1.5
		3447.72	643.281	2320.34	430.342	2430.94	408.952
			19%		19%		17%

Appendix Vb. (Continued)

PAH	TEF	20/12/1994 B2B-02	BAP EQUIV.	30/08/1994 C9	BAP EQUIV.	19/09/1994 B2A-filt.	BAP EQUIV.
N	0.001	0.85	0.00085	0.14	0.00014	0.79	0.00079
AY	0.001	0.19	0.00019	0.04	0.00004	0.2	0.0002
AE	0.001	1.54	0.00154	0.02	0.00002	0.32	0.00032
FL	0.001	1.19	0.00119	0.03	0.00003	0.88	0.00088
PH	0.001	34	0.034	0.39	0.00039	16.5	0.0165
AN	0.01	11.2	0.112		0	5.58	0.0558
F	0.001	244	0.244	0.9	0.0009	233	0.233
PY	0.001	195	0.195	0.78	0.00078	210	0.21
BaA	0.1	187	18.7	1.21	0.121	217	21.7
CH	0.01	535	5.35	2.64	0.0264	435	4.35
BbF	0.1	607	60.7	5.07	0.507	946	94.6
BkF	0.1	104	10.4	1.7	0.17	143	14.3
BaP	1	135	135	0.99	0.99	218	218
IP	0.1	108	10.8	1.33	0.133	192	19.2
DA	5	43.2	216	0.43	2.15	69.7	348.5
BP	0.01	139	1.39	1.72	0.0172	254	2.54
		2346.17	458.929	17.39	4.1169	2941.97	723.707
			20%		24%		25%

Appendix VI. Calculation of total suspended solids (TSS) in disturbed sediment exposures.

Ten litres of water were mixed with 2.5L sediment for a 4:1 exposure. The moisture content of B-2A sediment was 73% by weight, and 100 mL dry sediment weighs 42 g.

The 42 g dry sediment would have been 27% of the total weight of wet sediment, thus the wet sediment would have weighed 42/0.27 = 155g. 155 - 42 = 133 g water, which would have a volume of 113 mL. Thus a total volume of 100 mL dry sediment + 113 mL water = 213 mL wet sediment. this gives us the volume of wet sediment (213 mL) that would contain 42 g fry sediment. Since we used 2.5 L wet sediment in our exposure, this is 493 g dry sediment. The approx. 500g was mixed with 10 L water for a total volume of 12.5 L, thus the concentration of TSS (assuming all in suspension) was about 40g/L, or 40 000 mg/L. Similar calculations can be made for the other treatments:

4:0.1 treatment - ratio of 4 L water per 0.1 L sediment for a total of 10 L water and 0.25 L sediment. 250 mL wet sediment = 250/213 X 42 = 49 g dry sediment. TSS = 49/10.25 L = 4.8 g/L or approx. 5000 mg/L.

4:0.01 treatment - ratio of 4 L water per 0.01 L sediment for a total of 10 L water and 0.025 L sediment. 25 mL wet sediment = 25/213 X 42 = 5 g dry sediment. TSS = 5/10.025 L = 0.5 g/L or 500 mg/L.

Thus, the nominal TSS in each treatment, assuming that all material is in suspension, would be:

4:1 = 50,000 mg/L
4:0.1 = 5000 mg/L
4:0.01 = 500 mg/L
4:0.001 = 50 mg/L
4:0.0001 = 5 mg/L

The "action level" for TSS outside of the silt curtain is 25 mg/L (for the GM Superfund site). Thus, the action level is mid-way between the two lowest treatments (4:0.0005 water to sediment or a sediment to water ratio of 0.0125%).

Appendix VII. **Raw data from the 21 d fathead minnow and rainbow trout bioassays on disturbed and disturbed sediment collected in August 1994.**

Results of Fathead Minnow 21 d survival and growth tests conducted on disturbed and undisturbed test sediments (10/20/94).

Sample	Replicate	Number of Survivors	Initial Weight (mg)		Final Weight (mg)		Weight Gain (mg)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
B2-A (Disturbed)	A	0	357.3	357.0	-	-	-	-
	B	0	339.6		-		-	
	C	0	358.3		-		-	
	D	0	372.6		-		-	
B2-A (Undisturbed)	A	8	368.0	351.1	259.3	245.6	-108.7	-105.5
	B	10	344.8		249.3		- 95.5	
	C	7	332.4		242.1		- 90.3	
	D	2*	359.2		231.6		-128.0	
Control	A	10	366.4	356.3	393.9	380.0	27.5	23.7
	B	9	341.4		374.6		33.2	
	C	10	337.8		352.2		14.4	
	D	10	379.7		399.3		19.6	

* mortalities occurred on day 17 (4) and day 18 (4).

Appendix VII. (Continued)

Results of Rainbow Trout 21 d survival and growth tests conducted on test sediments (10/20/94).

Sample	Replicate	Number of Survivors	Initial Weight (mg)		Final Weight (mg)		Weight Gain (mg)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
B2-B (Undisturbed)	A	9	302.4	315.0	387.3	403.4	84.9	88.3
	B	7	347.9		470.5		122.6	
	C	9	301.5		408.0		106.5	
	D	4*	308.3		347.6		39.3	
Control	A	9	325.4	321.4	499.1	475.4	173.7	154.0
	B	10	310.6		446.3		135.7	
	C	9	334.9		496.1		161.2	
	D	10	314.7		460.2		145.5	

* Only 4 live fish were recovered; several fish escaped through the bottom of the screen that separated the fish from the sediment, and were found dead at the end of the test. It is not clear if mortality is due to the sediment or if the fish were injured when escaping through the screen.

Appendix VIII. Concentrations of metals in unfiltered and filtered B-2 elutriate.

Parameter (mg/L)	Unfiltered B2 Eutriate (03/03/95)		Filtered B2 Elutriate (06/03/95)	
	Total Metals (mg/L)	Extractable Metals (mg/L)	Total Metals (mg/L)	Extractable Metals (mg/L)
aluminum	15.0	13.0	0.841	0.83
arsenic	0.0095	-	0.0014	-
barium	0.0750	0.0713	0.0417	0.0382
berylium	3.03	0.0035	0.99	0.0011
cadmium	0.0014	0.001	0.0008	<0.001
cobalt	0.0030	0.002	0.0004	<0.001
chromium	0.0085	0.007	0.0006	<0.001
copper	0.0459	0.027	0.0126	0.009
iron	5.17	4.06	0.0231	0.022
lithium	0.595	0.594	0.451	0.447
manganese	0.689	0.680	0.579	0.572
molybdenum	0.0052	0.001	0.0050	0.005
nickel	0.0439	0.019	0.0093	0.009
lead	0.0192	0.019	0.0002	<0.005
selenium	0.0025	-	0.0005	-
strontium	0.163	0.157	0.133	0.126
vanadium	0.249	0.214	0.0444	0.043
zinc	0.460	0.457	0.0228	0.021

Appendix IX. Total ammonia measured at the start of testing, and pH measured at the start (pH_i) and end (pH_f) of testing, for the raw and zeolite-treated elutriate samples. Total ammonia concentrations are calculated from the initial concentrations of 10.8 mg/L in the 100% raw elutriate and 1.6 mg/L in the 100% zeolite-treated elutriate.

Daphnia magna

	Raw Elutriate		Zeolite-treated Elutriate	
Dilution of elutriate	Total ammonia (mg/L)	pH_i - pH_f	Total ammonia (mg/L)	pH_i - pH_f
100	10.8	7.5 - 8.4	1.6	7.8 - 8.4
50	5.4	7.6 - 8.4	0.8	7.9 - 8.4
25	2.7	7.7 - 8.3	0.4	7.9 - 8.4
13	1.35	7.8 - 8.1	0.2	7.9 - 8.4
6	0.68	not measured	0.1	not measured
0	0	7.8 - 8.4	0	7.8 - 8.4

Pimephales promelas

	Raw Elutriate		Zeolite-treated Elutriate	
Dilution of elutriate	Total ammonia (mg/L)	pH_i - pH_f	Total ammonia (mg/L)	pH_i - pH_f
100	10.8	7.5 - 8.4	1.6	7.8 - 8.4
50	5.4	7.6 - 8.4	0.8	7.9 - 8.4
25	2.7	7.7 - 8.3	0.4	7.9 - 8.4
13	1.35	7.8 - 8.4	0.2	7.9 - 8.4
6	0.68	7.8 - 8.4	0.1	7.9 - 8.4
0	0	7.8 - 8.3	0	7.8 - 8.5

Appendix X. Raw data from bioassays on fluoride-spiked sediment.

Results of 21 d fluoride-spiked sediment tests with *Hexagenia limbata.*

Sample	Replicate	No. of Survivors	Initial Weight (g)		Final Weight (g)		Weight Gain (g)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
5600 mg/kg spikef	A	0	0.0198	0.0197	0	0	0	0
	B	0	0.0194		0		0	
	C	0	0.0200		0		0	
2800 mg/kg spike	A	1	0.0158	0.02	0.0330	0.0321	0.0172	0.0121
	B	3	0.0166		0.0247		0.0081	
	C	2	0.0276		0.0385		0.0109	
1400 mg/kg spike	A	0	0.0166	0.0189	0	0.0394*	0	0.0193
	B	8	0.0196		0.0429		0.0233	
	C	7	0.0206		0.0359		0.0153	
700 mg/kg spike	A	9	0.0199	0.0187	0.0376	0.0412	0.0177	0.0225
	B	9	0.0160		0.0415		0.0255	
	C	8	0.0203		0.0444		0.0241	
CONTROL	A	10	0.0199	0.0191	0.0453	0.0459	0.0254	0.0268
	B	10	0.0166		0.0422		0.0256	
	C	10	0.0208		0.0502		0.0294	

* group average includes replicates B and C; organisms in replicate A were not added to exposure chamber.

** all concentrations started with 10 organisms per replicate

Appendix X. (Continued)

Results of 10 d fluoride-spiked sediment tests with *Chironomus tentans*.

Sample	Replicate	No. of Survivors	Final Weight (g) x 10^{-3}	
			mean/ animal	group average
5600 mg/kg spike	A	14	0.7786	0.6945
	B	15	0.5627	
	C	14	0.7436	
2800 mg/kg spike	A	15	0.9107	0.8159
	B	13	0.7885	
	C	14	0.7486	
1400 mg/kg spike	A	15	1.0033	0.8620
	B	15	0.7380	
	C	13	0.8446	
700 mg/kg spike	A	14	0.9079	0.9540
	B	14	0.7692	
	C	14	1.185	
CONTROL	A	15	1.3247	1.2973
	B	15	1.3533	
	C	16	1.2138	

* all concentrations started with 15 organisms per replicate: extra organism added to control replicate C.

Appendix X. (Continued)

Results of 28 d fluoride-spiked sediment tests with *Hyalella azteca*.

Sample	Replicate	No. of Survivors	Final Weight (g)	
			mean/animal	group average
1400 mg/kg spike	A	4	0.083	0.098
	B	7	0.135*	
	C	2	0.075	
700 mg/kg spike	A	7	0.124	0.118
	B	7	0.109	
	C	7	0.121	
350 mg/kg spike	A	10	0.186	0.150
	B	8	0.126*	
	C	10	0.138	
175 mg/kg spike	A	9	0.244	0.195
	B	8	0.211	
	C	10	0.131	
CONTROL	A	5	0.198	0.221
	B	7	0.272	
	C	9	0.192	

* one organism was lost before weighing, thus, growth results are based on 1 less organism. (all concentrations started with 10 organisms per replicate)

Appendix X. (Continued)

Results of 21 d fluoride-spiked sediment tests with fathead minnows.

Sample	Replicate	Number of Survivors	Initial Weight (g)		Final Weight (g)		Weight Gain (g)	
			mean/ animal	group average	mean/ animal	group average	mean/ animal	group average
5600 mg/kg spike	A	10	0.0978	0.0953	0.0981	0.0934	0.0003	-0.0019
	B	10	0.0876		0.0857		-0.0019	
	C	10	0.1004		0.0963		-0.0041	
2800 mg/kg spike	A	10	0.0912	0.0962	0.0922	0.1015	0.001	0.0053
	B	7	0.1045		0.1192		0.0147	
	C	10	0.0929		0.0930		0.0001	
1400 mg/kg spike	A	10	0.0924	0.0928	0.0882	0.0911	-0.0042	-0.0017
	B	10	0.0956		0.0962		0.0006	
	C	10	0.0905		0.0890		-0.0015	
700 mg/kg spike	A	10	0.1045	0.0959	0.1008	0.0952	-0.0037	-0.0007
	B	10	0.0840		0.0851		0.0011	
	C	10	0.0991		0.0998		0.0007	
CONTROL	A	10	0.0910	0.0953	0.0903	0.0946	-0.0007	-0.0007
	B	10	0.1005		0.0990		-0.0015	
	C	9	0.0945		0.0946		0.0001	

* all concentrations started with 10 organisms per replicate.

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National Water Research Institute
Environment Canada
Canada Centre for Inland Waters
P.O. Box 5050
867 Lakeshore Road
Burlington, Ontario
L7R 4A6 Canada

National Hydrology Research Centre
11 Innovation Boulevard
Saskatoon, Saskatchewan
S7N 3H5 Canada

NATIONAL WATER RESEARCH INSTITUTE
INSTITUT NATIONAL DE RECHERCHE SUR LES EAUX

Institut national de recherche sur les eaux
Environnement Canada
Centre canadien des eaux intérieures
Case postale 5050
867, chemin Lakeshore
Burlington, Ontario
L7R 4A6 Canada

Centre national de recherche en hydrologie
11, boul. Innovation
Saskatoon, Saskatchewan
S7N 3H5 Canada

Environment Canada Environnement Canada

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