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BIOLOGICAL ASSESSMENT OF SEDIMENT QUALITY IN THUNDER BAY NORTH HARBOUR, 2005

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BIOLOGICAL ASSESSMENT OF SEDIMENT QUALITY IN THUNDER BAY NORTH HARBOUR, 2005

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SUMMARY

In 2002, Environment Canada sampled 19 sites in Thunder Bay North Harbour where elevated levels of sediment mercury had been observed. The impact of this elevated sediment mercury on benthic invertebrate communities, sediment toxicity, and the bioavailability of this mercury to resident benthic invertebrates and its potential for effects on fish and wildlife through biomagnification was assessed. Results showed that sediment mercury concentrations were elevated above sediment quality guidelines and above reference conditions at most sites and mercury concentrations in the resident benthos were also elevated compared to reference conditions. Sediments were found to be toxic at several sites and benthic communities were generally different than those at Great Lakes reference sites. There were several sites where potentially adverse effects due to mercury biomagnification were observed.

The objective of this study was to fill existing data gaps in the areas of Thunder Bay North Harbour that showed toxicity and/or the potential for mercury biomagnification from the 2002 study. In 2005, 15 sites were sampled focusing on two areas: (a) in the vicinity of the paper mill where toxicity and potential for biomagnification were observed, and (b) in the vicinity of the Current River mouth, where potential for biomagnification was observed. Sediment, overlying water, and benthic invertebrates were collected for: (a) chemical and physical analysis (sediment and overlying water), (b) measurement of tissue concentrations of total and methyl mercury (resident benthic invertebrates), and (c) laboratory sediment toxicity tests. A decision-making framework for sediment contamination, developed under the Canada-Ontario Agreement respecting the Great Lakes Basin Ecosystem, was applied to data from both studies (total of 34 sites) to determine whether contaminated sediments were degraded.

Sediment mercury concentrations range from 0.04 to 7.6 µg/g and 4 of the 15 test sites are above the provincial Severe Effect Level. (With both studies combined, total mercury ranges from 0.03 to 39.7 µg/g.) Total mercury tissue concentrations in one or both of the resident invertebrate taxa collected at all Thunder Bay sites except one are elevated above those at reference sites; methyl mercury tissue concentrations in one taxon (chironomids) are elevated above those at reference at 10 Thunder Bay sites. Three sites are either toxic or severely toxic and these sites are in

similar locations to where toxicity was observed in 2002. Toxicity is related to metal contaminants; however, there may be unmeasured stressors involved as well as substrate related factors with respect to paper material found at some sites. In 2002, toxicity to the mayfly *Hexagenia* could be partially explained by sediment methyl mercury; however, there are no effects on *Hexagenia* in the current study.

According to the decision-making framework, required management actions are indicated for 3 sites due to elevated mercury, sediment toxicity, benthos alteration, and in 2 of the 3 cases, the potential for mercury biomagnification. Benthic community structure was not assessed in the current study. However, if it is assumed that benthic communities for the 2005 sites are likely different from reference conditions (as observed for 17 of the 19 sites from the 2002 study), then the assessment outcome will change for a number of sites. Primarily, management actions would be indicated for 7 sites as opposed to 3. With respect to the "potential for biomagnification" line of evidence, significant biomagnification can be determined when there is site specific evidence, such as from fish advisories or previous research in the area. Currently, there are fish consumption advisories in the Thunder Bay Area of Concern due to mercury; however, the last fish survey that took place in the sampling area adjacent to the paper mill was in 1998. Information from a new fish survey would reduce uncertainty in the assessment of the risk of mercury biomagnification.

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Abstract

Elevated levels of mercury in sediments of Thunder Bay North Harbour, Lake Superior, have led to several investigations by Environment Canada. The impact on invertebrate communities, sediment toxicity, and the bioavailability of this mercury and its potential for effects on fish and wildlife through biomagnification was assessed in 2002. The purpose of this study was to fill data gaps in two critical areas of the harbour that were determined from the 2002 study: (a) in the vicinity adjacent to the paper mill where toxicity and potential for biomagnification were observed, and (b) in the vicinity of the Current River mouth, where potential for biomagnification was observed. In June 2005, overlying water and surficial sediment samples were collected at 15 sites for: (a) chemical and physical analysis of surficial sediment and overlying water; (b) measurement of tissue concentrations of total and methyl mercury in resident benthic invertebrates; and (c) laboratory sediment toxicity tests. A decision-making framework for sediment contamination, developed under the Canada-Ontario Agreement respecting the Great Lakes Basin Ecosystem, was applied to data from both studies (total of 34 sites) to determine whether contaminated sediments were degraded.

This study involved (a) comparisons of benthic toxicological response in laboratory toxicity tests to those established for Great Lakes reference sites using multivariate techniques (ordination) (b) comparisons of total and methyl mercury concentration in sediment and benthic invertebrates (oligochaetes and chironomids) from Thunder Bay to those from Lake Superior reference sites, (c) analyses of the relationships of total and methyl mercury concentrations in invertebrates to those in sediment (regression analysis), and (d) predictions of concentrations of methyl mercury in representative consumers of benthic invertebrates and their predators (White Sucker, Yellow Perch, Walleye, Great Blue Heron, Mink) using screening-level trophic transfer models with biomagnification factors obtained from a review of pre-existing studies.

Sediment mercury levels are high in some cases (range 0.04 to 7.6 µg/g), exceeding the provincial Severe Effect Level at 4 of the 15 sites. Methyl mercury tissue concentrations in resident chironomids are elevated above those at reference at 10 of the 15 Thunder Bay sites. Toxicity is observed at 3 sites; these sites are in similar locations to where toxicity was observed in 2002. Toxicity is related to metal contaminants; however, there may be unmeasured stressors involved as well as substrate related factors with respect to paper material found at some sites. According to the decision-making framework, management actions are indicated for 3 sites due to elevated mercury, sediment toxicity, benthos alteration, and in 2 of the 3 cases, the potential for mercury biomagnification. Benthic community structure was not assessed in the current study. However, if it is assumed that benthic communities at the 2005 sites are likely different from reference conditions (as observed for 17 of the 19 sites from the 2002 study), then management actions would be indicated at 7 sites as opposed to 3. Information from a new fish survey in the sampling area would reduce uncertainty in the assessment of the risk of mercury biomagnification.

Résumé

Les concentrations élevées de mercure dans les sédiments du havre nord de Thunder Bay, au lac Supérieur, ont donné lieu à plusieurs études d'Environnement Canada. Ainsi, les impacts sur les communautés d'invertébrés, la toxicité des sédiments, ainsi que la biodisponibilité du mercure et les effets possibles de sa bioamplification sur le poisson et la faune ont été évaluées en 2002. Notre étude visait à combler les lacunes dans les données sur deux secteurs du havre jugés préoccupants dans l'étude de 2002 : a) les environs de l'usine de papier où l'on a observé de la toxicité et un potentiel de bioamplification et b) les environs de l'embouchure de la rivière Current où l'on a observé un potentiel de bioamplification. En juin 2005, nous avons recueilli des échantillons de sédiments superficiels et d'eau sus-jacente à 15 sites pour effectuer : a) l'analyse de leurs caractéristiques physicochimiques, b) le dosage du mercure total et du méthylmercure dans les invertébrés benthiques et c) des essais de toxicité des sédiments en laboratoire. Nous avons appliqué aux données des deux études (34 sites au total) le cadre décisionnel en matière de contamination des sédiments, élaboré en vertu de l'entente Canada-Ontario sur l'écosystème du bassin des Grands Lacs, afin de déterminer si les sédiments contaminés sont dégradés.

Notre étude a consisté à : a) comparer, au moyen de méthodes d'analyse multivariée (ordination), les résultats d'essais de toxicité effectués en laboratoire sur des invertébrés benthiques à ceux obtenus pour des sites de référence dans les Grands Lacs, b) comparer les concentrations de mercure total et de méthylmercure dans les sédiments et des invertébrés benthiques (oligochètes et chironomides) des sites de Thunder Bay et des sites de référence dans le lac Supérieur, c) analyser par régression les relations entre les concentrations de mercure total et de méthylmercure dans les invertebrés et celles dans les sédiments, et d) prédire les concentrations de méthylmercure dans des consommateurs représentatifs d'invertébrés benthiques et dans leurs prédateurs (meunier noir, perchaude, doré, grand héron et vison) à l'aide de modèles de transferts trophiques d'évaluation préalable et des facteurs de bioamplification établis dans le cadre d'études antérieures.

Les concentrations de mercure dans les sédiments sont élevées dans certains cas (elles varient de 0,04 à 7,6 µg/g), dépassant à 4 des 15 sites le seuil d'effet grave établi par la province. À 10 des 15 sites de Thunder Bay, les concentrations de méthulmercure dans les chironomides sont supérieures à celles mesurées pour les sites de référence. De la toxicité a été observée à trois sites situés à peu près aux mêmes endroits où elle avait été constatée dans l'étude de 2002. La toxicité est associée à des polluants métalliques, mais d'autres facteurs de stress non mesurés pourraient également y jouer un rôle, tout comme des facteurs liés au substrat dans certains sites où l'on a trouvé du papier. Selon le cadre décisionnel en matière de contamination des sédiments, des mesures de gestion s'imposent pour trois sites en raison des concentrations élevées de mercure, de la toxicité des sédiments et de l'altération du benthos, et dans deux des trois cas, en raison du potentiel de bioamplification du mercure. La structure de la communauté benthique n'a pas été évaluée dans notre étude. Toutefois, si l'on présume que les communautés benthiques des sites de 2005 sont différentes de celles des sites de référence (comme il a été observé à 17 des 19 sites de l'étude 2002), alors des mesures de gestion devraient être appliquées à sept sites plutôt que trois. Les données qui seront obtenues dans un nouveau relevé des poissons dans l'aire d'étude réduiront sans doute l'incertitude de l'évaluation du risque de bioamplification du mercure.

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ABBREVIATIONS, ACRONYMS AND SYMBOLS

adj	adjusted
AOC	Area of Concern
BMF	biomagnification factor
BSAF	biota-sediment accumulation factor
dw	dry weight
EC	Environment Canada
FCM	food chain multiplier
GL	Great Lakes
GLWQA	Great Lakes Water Quality Agreement
Hg	mercury; used where form (MeHg or THg) is unspecified
HMDS	hybrid multidimensional scaling
IJC	International Joint Commission
inv	invertebrate
LEL	lowest effect level
max	maximum
MeHg	methyl mercury
min	minimum
MOE	Ministry of the Environment
NWRI	National Water Research Institute
PCA	principal components analysis
QA/QC	quality assurance/quality control
RAP	Remedial Action Plan
rec	receptor
ref	reference
RPD	relative percent difference
sed	sediment
SEL	severe effect level
THg	total mercury
TKN	total Kjeldahl nitrogen
TN	total nitrogen
TOC	total organic carbon
TP	total phosphorus
TRG	tissue residue guideline
wt	weight
ww	wet weight
$[x]_i$	concentration of substance x in matrix i

1 INTRODUCTION

1.1 Background

In 2002, Environment Canada assessed the impacts of elevated sediment mercury (Hg) in Thunder Bay North Harbour on benthic invertebrate communities, sediment toxicity, and the bioavailability of this mercury and its potential for effects on fish and wildlife through biomagnification (Milani and Grapentine 2005). Nineteen sites in Thunder Bay North Harbour and 20 reference sites (north shore of Lake Superior) were sampled.

This study involved:

(a) Comparisons of benthic invertebrate communities to those established for Great Lakes (GL) reference sites,

(b) Comparisons of laboratory toxicological response to those established for GL reference sites,

(c) Comparisons of total and methyl Hg concentration in sediment and benthic invertebrate tissue from Thunder Bay North Harbour to those from Lake Superior reference sites,

(d) Analyses of the relationships of total and methyl Hg concentrations in invertebrate tissue to those in sediment, and

(e) Predictions of concentrations of methyl Hg in representative consumers of benthic invertebrates and their predators using screening-level trophic transfer models.

Results showed elevated levels of Hg in the sediment and benthos compared to reference conditions at most sites, with the highest Hg concentrations found along the northern shore of the study area, and at the sites that contained white paper fibres. The spatial pattern of these results showed strong evidence for a local (as opposed to regional) source of Hg to the area. There was acute toxicity at five sites, most of which were located along the northern shore of study area. Increased sediment methyl Hg concentration was found to partially explain toxicity to the mayfly; however, there may have been unmeasured stressors involved as well as substrate related factors with respect to paper fibres found at some sites.

Benthic invertebrates were shown to accumulate Hg; whole body [Hg]s at test sites for chironomids were elevated above those at reference sites. Additionally, sediment [Hg] was found to affect invertebrate [Hg]. Under "average" conditions, five sites were predicted to have receptor (fish) methyl mercury concentrations above tissue residue guidelines for the protection of fish-consuming wildlife and above predicted maximum reference site concentrations. Generally, benthic invertebrate communities were very different from reference conditions, mainly characterized by the absence of a predominant reference site amphipod species (haustoriid) and the enrichment of other taxa such as tubificids, chironomids and sphaeriids. Enrichment was found to be associated with increased total organic carbon in some cases.

Risk management evaluation was recommended for 9 sites. For 8 of the 9 sites, this was due to the potential for Hg biomagnification and for one site it was due to elevated sediment mercury with co-occurrence of sediment toxicity and benthic alteration. Sampling efforts in 2002 provided adequate coverage to distinguish two critical areas (where toxicity and potential for Hg biomagnification occurred). Refinement of these areas would aid in further delineating the extent of the problems.

1.2 Study Objective

The objective of the current study is to fill existing data gaps in the areas of Thunder Bay North Harbour that showed toxicity and/or potential for biomagnification from the 2002 study. There were two focus areas: (a) in the vicinity adjacent to the paper mill, where toxicity and potential for biomagnification were seen, and (b) the vicinity of the Current River mouth, where potential for biomagnification was observed. Fifteen test sites were sampled to further delineate these areas. Five Lake Superior reference sites were also sampled (4 of which were sampled in 2002). Because benthic communities at sites sampled in 2002 were generally different from reference, sampling efforts in 2005 did not include the benthic community as it was deemed that this line of evidence would provide little additional valuable information for delineating the extent of sediment contamination within the two focus areas.

2 METHODS

2.1 Sample Collection

Fifteen stations were sampled June 10 – 15, 2005 in Thunder Bay North Harbour (Figure 1) and 5 reference sites were sampled in Lake Superior (outside Thunder Bay). Station positions, site depths, and a visual description of the sediments are provided in Table 1. Site positions were obtained using a differentially corrected global positioning receiver (MX300), with the exception of two sites (S05-05 and S05-13). Corrections were received from a shore reference station in the area (Bare Point).

At each site, overlying water and surficial (top 10 cm) sediment samples were collected for: (a) chemical and physical analysis of surficial sediment and overlying water; (b) resident benthic invertebrates for measuring tissue concentrations of total and methyl mercury; and (c) laboratory sediment toxicity tests. A visual description of the sediment was noted. Two separate taxa (chironomids and oligochaetes) were collected at all test and reference sites. Details on sampling techniques and methods for sample collection are provided in Milani and Grapentine (2005).

2.2 Physico-Chemical Analyses

Analytes measured in each compartment (water, sediment and biota) are listed in Table 2. Analytical procedures are provided in Milani and Grapentine (2005). Sediments were analyzed for (a) total and methyl mercury, (b) particle size (percents sand, silt, clay and gravel), (c) trace metals and major oxides, and (d) nutrients (total phosphorus, total Kjeldahl nitrogen, total organic carbon). Overlying water (0.5 m from the bottom) was analyzed for alkalinity, total phosphorus, total Kjeldahl nitrogen, nitrates +nitrites and ammonia. Temperature, conductivity, pH, and dissolved oxygen in the overlying water were measured on-site using HYDROLAB water quality instruments. Resident benthic invertebrates were analyzed for total and methyl mercury.

2.3 Sediment Toxicity Tests

Four sediment toxicity tests were performed: *Chironomus riparius* 10-day survival and growth test, *Hyalella azteca* 28-day survival and growth test, *Hexagenia* spp. 21-day survival and growth test, and *Tubifex tubifex* 28-day survival and reproduction test. Sediment handling procedures and toxicity test methods are provided in Milani and Grapentine (2005).

2.4 Data Analysis

2.4.1 BEAST analysis – toxicity

Test sites were assessed using BEAST methodology, a predictive approach for assessing sediment quality in the Great Lakes using the ordination technique Hybrid Multidimensional Scaling (HMDS) (Reynoldson et al. 1995, 2000; Reynoldson and Day 1998). Toxicological responses from Thunder Bay sites were compared to those established for reference sites (sampled from uncontaminated areas in the Great Lakes over a three year period). A complete description of BEAST data analyses is provided in Milani and Grapentine (2005).

2.4.2 Sediment toxicity and contaminant concentrations

Relationships between sediment toxicity and sediment contamination for the Thunder Bay sites were assessed graphically (site data were ordinated again by HMDS, as a single group and without the reference site data) and by regression analysis. Data analyses are described in Milani and Grapentine (2005).

2.4.3 Potential for mercury biomagnification

Sites in which concentrations of Hg in invertebrates ($[Hg]_{inv}$) were significantly elevated above background levels for the study area were identified by comparing $[Hg]_{inv}$ for the test sites to the upper 99th % percentile for the Lake Superior reference sites. Relationships between concentrations of Hg in sediment and invertebrates were determined using regression analysis, separately for each invertebrate taxon. Concentrations of MeHg in the tissues of receptors ($[MeHg]_{rec}$) were predicted by multiplying measured body concentrations in the resident invertebrates ($[MeHg]_{inv}$) by a relevant food chain multiplier (FCM):

$$[MeHg]_{rec} = FCM \times [MeHg]_{inv}$$

The FCM represents the cumulative biomagnification of a substance from one trophic level to a higher trophic level (USEPA 1997). Whereas a BMF applies to only one trophic level transfer, a FCM refers to one or more, and may be a multiple of more than one BMF. Thus, $FCM = BMF_1 \times BMF_2 \times BMF_3 \times \ldots \times BMF_n$, where 1, 2, 3,..., n are transfers of one trophic level. Biomagnification factors were obtained previously from a literature review (see Milani and Grapentine 2005 for details on literature review). For each site, minimum, intermediate and maximum concentrations of MeHg for each receptor were predicted by using corresponding low, medium and high $[MeHg]_{inv}$ and FCMs. Complete methodology is provided in Milani and Grapentine (2005).

2.5 Quality Assurance/Quality Control

2.5.1 Field replication

Triplicate overlying water and sediment samples were collected at one test site (S05-05) and one reference site (5103) for the determination of within-site and among-sample variability. Variability in a measured analyte was expressed as the coefficient of variation (CV = standard deviation / mean × 100).

2.5.2 Laboratory

Quality control procedures for Caduceon Environmental Laboratory involved control charting of influences, standards, and blanks. Reference material was used in each analytical run. Calibration standards were run before and after each run. Run blanks and reference standards were run 1 in 20 samples, while duplicates were run 1 in 10 samples. Sample duplicate measurements of sediment metals, major oxides and nutrients were expressed as the Relative Percent Difference (RPD):

$RPD = (x_1 - x_2) / ((x_1 + x_2)/2) \times 100$

Flett Research Ltd. conducted determinations of total and methyl mercury in sediment and benthic invertebrates. Quality control evaluation for these procedures included analyses of sample duplicates and repeats, and recovery of matrix spikes, quality control samples, and certified reference standards. For sediment samples, sample duplicates or repeat aliquots were analyzed at least once every 10 samples, and matrix spikes were performed on every tenth sediment sample to determine mercury recoveries. The quality control samples "MESS-2", "OPR", "IAEA 405" and "Alfa" were concurrently digested and analysed total or methyl mercury. For biota samples, sample duplicates were analyzed at least once every 20 samples and "DORM-2", "OPR" and "Alfa" reference materials were concurrently digested for total or methyl mercury. The mercury reference standards (Hg STD 1 to 5) were analyzed for total mercury in each digestion.

3 RESULTS

3.1 Sediment and Water Physico-Chemical Properties

3.1.1 Overlying water

Conditions of overlying water 0.5 m above the sediment are generally similar across Thunder Bay sites (Table 3). The ranges of dissolved oxygen, pH, conductivity, alkalinity, temperature, NH_3, NO_3/NO_2, total Kjeldahl nitrogen, and total phosphorus across Thunder Bay sites are: 2.2mg/L, 1.4 pH units, 26µS/cm, 8.1 mg/L, 10.6°C, 0.09mg/L, 0.09mg/L, 0.19mg/L, and 0.02mg/L, respectively. Sites S05-08 and S05-07 (located in most southern part of sampling area near the break wall opening, Figure 1) are cooler in temperature due to greater depths (see Table 1); site S05-08 also has the lowest conductivity, total Kjeldahl nitrogen and total phosphorus. Dissolved oxygen is ≥9.6 mg/L at all sites and pH ranges from 7.5 to 8.9.

3.1.2 Particle size

Percents sand, silt, clay, and gravel for Thunder Bay sediment are shown in Table 4. Thunder Bay sediments consist mainly of silt (ranging from 6.6 to 70.7%, median 52%), and clay (ranging from 4.0 to 49.5%, median 25%), or of sand (ranging from 0.6 to 83.6%, median 14%), and silt/clay. Site S05-10, located at the mouth of Current River, is mostly sand (84%), and S05-10, S05-14 and S05-15b, have the coarsest sediment, with gravel ranging from 0.3 to 9.7%. There is no gravel present at any other site.

A visual inspection of the sediment at the time of sampling noted the presence of paper fibres at sites S05-01 and S05-11 (located in close proximity to each other at northeastern end of sampling area, Figure 1); a sulphur odour was also detected at these two sites (Table 1). An oily residue became apparent at sites S05-04 and S05-14 when the sediment was stirred up from sampling.

3.1.3 Sediment mercury

Total mercury

Total mercury was analyzed by two laboratories (Caduceon and Flett laboratories). Results are provided in Table 5 and Figure 2. Thunder Bay [THg]s range from 0.04 to 7.6 µg/g. The Severe Effect Level (2.0µg/g - Persaud et al. 1992) is exceeded at 4 sites (results from both labs considered): S05-01, S05-03, S05-04, and S05-11. These sites are located in the northern part of the sampling area. On a dry weight basis, lower [THg]s are found in the Lake Superior reference sediments (range 0.009 to 0.022 µg/g); The Lowest Effect Level (LEL) for THg (0.2µg/g) is not exceeded at reference site, while it is exceeded at all Thunder Bay sites except S05-08, S05-10, and S05-14. Higher sediment [THg]s were observed at 2002 Thunder Bay sites (range: 0.03 to 39.7 µg/g - Milani and Grapentine 2005), but the area of highest [Hg] for both studies is along the northern shore of the sampling area (sites closest to paper mill).

Four of the five reference sites sampled in 2005 were re-sampled in 2002: 5101, 5103, 5108 and 5109. Mercury concentrations from both studies for these reference sites are provided in Appendix A; Figure A1. Total [Hg] are generally similar to what was found at these sites in 2002 (range: 0.005 to 0.083 µg/g) with the exception of 5103. Total Hg was ~5× higher at 5103 in 2002 than in 2005. This could be due to difference in sampling location (<10 m), and likely reflects small scale natural heterogeneity.

Comparison of sediment mercury at test sites to reference sites

All test sites are above the 99th percentile of the Lake Superior reference sites (Figure 2). Most Thunder Bay sites are 1 to 2 orders of magnitude higher in [THg] than the maximum [THg] of the reference sites. The median [THg] of the Thunder Bay sites ~35× the median of the reference sites. In the 2002, Thunder Bay sites were 1 to 4 orders of magnitude higher than the reference sites and the median [THg] of the Thunder Bay sites was ~37× the median of the reference sites (Milani and Grapentine 2005).

Methyl mercury

Thunder Bay [MeHg]s range from 0.8 to 27 ng/g (Figure 3, Table 5). The highest concentrations occur at S05-03 and S05-04, the same as those observed for THg. These concentrations are slightly lower than those seen in 2002, where [MeHg] ranged from 1.5 to 49.8 ng/g (Milani and Grapentine 2005). Methyl mercury concentrations are lower at Lake Superior reference sites, ranging from 0.03 to 0.2 ng/g. Comparisons of methyl [Hg] at reference sites sampled in both 2002 and 2005 are shown in Appendix A; Figure A1. As with total Hg, methyl [Hg] is generally similar (range: 0.08 to 0.36 ng/g) with the exception of 5103, which was 12× higher in 2002.

Comparison of sediment methyl mercury at test sites to reference sites

All test sites exceed the upper 99th percentile of the reference sites by 1 to 2 orders of magnitude (Figure 3). The median [MeHg] of the Thunder Bay sites is ~83× the median of the reference sites. In 2002, all test sites exceeded the upper 99th percentile of the reference sites by 1 to 4 orders of magnitude and the median [MeHg] of the Thunder Bay sites was 110× the median of the reference sites (Milani and Grapentine 2005).

Methyl mercury-total mercury relationship

The relationship between methyl mercury and total mercury in the sediment (log-transformed) is shown in Figure 4. A significant strong positive correlation ($P<0.001$, $r^2 = 0.91$, slope = 0.957) exists between the methyl and total mercury concentrations in the sediment. This is similar to the relationship in the 2002 study ($P<0.001$, $r^2 = 0.84$, slope = 1.066) (Milani and Grapentine 2005).

3.1.4 Sediment trace metals and nutrients

Sediment trace metal, nutrient and major oxide concentrations for Thunder Bay sites are provided in Table 6; data for Lake Superior reference sites are provided in Appendix A; Table A1. Excluding mercury, metal concentrations are below the SEL for the Thunder Bay sites (Table 6). There are, however, exceedences of LELs at up to 15 sites for up to 9 metals, including arsenic (As - 1 site), cadmium (Cd - 9 sites), chromium (Cr - 15 sites), copper (Cu - 15 sites), iron (Fe - 11 sites), manganese (Mn - 1 site), nickel (Ni - 14 sites), lead (Pb - 4 sites) and zinc (Zn - 9 sites) (Table 6). The LEL is exceeded for 7 metals at sites S05-01, S05-03, and S05-04 (As and Mn do not exceed the LEL at these 3 sites). Metal concentrations at reference sites are low, either below LELs or just above for most metals (Appendix A; Table A1).

Total organic carbon (TOC) at Thunder Bay sites is high generally, ranging from 0.7 to 13.6% (median 4.1%), exceeding the SEL at S05-01 (Table 6). Total nitrogen ranges from 383 to 4550 µg/g (median 1530µg/g) and total phosphorus ranges from 486 to 1130 µg/g (median 694µg/g). High TOC (up to 25.7%) was observed in 2002 as well, especially at sites which contained the paper fibres (along the northern part of the sampling area), similar to the current study. There is a significant positive relationship between total organic carbon and total mercury in sediment (logged, Figure 5; $R^2 = 0.898$, $p< 0.001$), which was also found in a 1998 study performed in the same area (Stantec 2003). The highest TOC is consistently found along the northern part of the study area. Reference site sediment nutrient concentrations are lower than those at Thunder Bay sites. Mean TOC, total Kjeldahl nitrogen and total phosphorus concentrations are 0.65% 564μg/g, and 550μg/g, respectively (Appendix A; Table A1).

3.2 Sediment Toxicity Tests

Mean survival, growth and reproduction are shown in Table 7. The established numerical criteria from Reynoldson and Day (1998) for each category (non-toxic, potentially toxic and toxic) for each species are included.

There is acute toxicity to *Hyalella* (survival: 27 and 48%) at two sites (S05-01 and S05-16) and acute toxicity to *Chironomus* (survival: 13 and 49%) at two sites (S05-01 and S05-11) (Table 7). There is potential toxicity to *Hyalella* (63% survival) at S05-13. In 2002, acute toxicity to *Hyalella* and/or *Chironomus* was observed at 4 sites, including one site near Site S05-01 and one site near Site S05-16. There is no toxicity to *Hexagenia* or *Tubifex*; however, *Tubifex* young production is lower at sites S05-11 to S05-16 (no. young per adult: 17.7 to 21.6) compared to remaining sites (no. young: 24.3 to 39.4) and the reference control mean (29.0). Comparing current toxicity results to those from the 2002 study, a major difference is the lack of acute response in the mayfly *Hexagenia* in the current study. In the 2002 study, there was acute toxicity to *Hexagenia* at 3 of the 19 sites (sites with paper fibres present; survival: 12 to 44%) (Milani and Grapentine 2005), while in the current study mayfly survival is high (≥96%; Table 7), even at sites that contain some paper fibres. The amount of fibrous material present at sites in the 2002 study was generally greater though. (Sediments at 2 of the 3 sites that showed acute toxicity to *Hexagenia* consisted almost entirely of paper fibres and as a result particle size analysis was not possible.)

BEAST assessment of toxicity

Results of the BEAST toxicity evaluation are summarized in Table 7. Ordinations are shown in Appendix B; Figures B1 and B2 (stress < 0.10). Each figure represents a separate ordination of a subset of 7 and 8 Thunder Bay 2005 sites. Six of the 10 endpoints are significant ($p \leq 0.05$) in each ordination; *Hyalella* survival and *Chironomus* survival are the most significant endpoints in the ordinations ($r^2 \geq 0.82$). The relationship between toxicological response and habitat variables was examined by correlation of the ordination of the toxicity data and the habitat information. There are no high correlations ($r^2 \leq 0.24$).

Thunder Bay sites fall into the following bands (Table 7):

Band 1 (non-toxic):	11 sites
Band 2 (potentially toxic):	1 site (S05-13)
Band 3 (toxic):	1 site (S05-16)
Band 4 (severely toxic):	2 sites (S05-01 and S05-11)

The majority of Thunder Bay sites are non-toxic. The 3 sites in Bands 3 and 4 are associated with decreased *Hyalella* survival (sites are located along the same vector line as *Hyalella* in the opposite direction – Figure A1), or with decreased *Hyalella* or *Chironomus* survival (Figure A2). Sites S05-01 and S05-11 are most toxic, located farthest from the reference centroid in ordination space; S05-01 is associated with decreased *Hyalella* survival and S05-11 is associated with decreased *Chironomus* survival. Site S05-16 is associated with decreased *Hyalella* survival and site S05-13, which is potentially toxic, has slight decreased *Hyalella* survival. The cause of toxicity may differ depending on the site. For example, there is no toxicity to *Hyalella* at S05-11 but S05-01 is acutely toxic to *Hyalella*. These sites, however, are within close proximity to each other and both have paper fibres present. Sites that fall in Band 3 and 4, as well as sites showing toxicity from 2002 study, are shown in Figure 6.

Elevated sediment mercury and total organic carbon (TOC) concentrations are associated with sites S05-01 (Figure B1) and S05-11 (Figure B2), although correlations are not high (Hg r^2= 0.093, 0.24; TOC r^2= 0.095, 0.11). Sites S05-01 and S05-11 have the highest total organic carbon (13.6 and 8.5%, respectively) and high sediment [Hg] (7.6 and 4.2 µg/g, respectively; Table 6). These two sites have paper fibres present and there was a sulphurous odour to the sediment (Table 1); sites are located in close proximity to each other in the northeastern part of the sampling area (see Figure 1).

3.3 Toxicity-Contaminant Relationships

HMDS and principal axis correlation

The ordination of the multiple measurements of sediment toxicity by HMDS for the Thunder Bay sites produced two descriptors of sediment toxicity (Appendix C; Figure C1). The resultant axes represent the original 10-dimensional among-site resemblances well (stress = 0.06). Principal axis correlation produces a vector for each toxicity endpoint along which the projections of sites in ordination space are maximally correlated. *Hyalella* survival is negatively correlated with Axes 1 and 2; therefore, the greater the toxicity to amphipod survival, the higher its score for Axes 1 and 2. *Chironomus* survival is positively correlated with Axis 1 and negatively correlated with Axis 2; therefore, the greater the toxicity to midge survival, the lower its score for Axis 1 and the higher its score for Axis 2.

Principal components analysis (PCA)

The concentrations of 8 metals (Cr, Cu, Fe, Hg, Mn, Ni, Pb, and Zn) were ordinated by PCA. The first and second principal components (PC1 and PC2) account for 71.5% and 22.4% of the variation and the remaining components each account for ≤2.8%; therefore, most of the structure in the data is captured in two components or dimensions. Copper, mercury, lead and zinc are negatively loaded for PC1; sites elevated in these metals and nutrients score low for PC1 (similar to that seen in 2002 study). Overall, the magnitude of the negative loadings are similar, ranging from −0.212 (Pb) to −0.340 (Hg). These principle components – denoted as "metPC1" and "metPC2"– are considered fair descriptors of general metal contamination.

Toxicity-contaminant relationships

The integrated descriptors of sediment toxicity (Axis 1 and 2 scores from the HMDS) and the most significant individual toxicity endpoints (survival of *Hyalella* and *Chironomus*) were plotted against the metal contaminant descriptors "metPC1" and "metPC2" (Appendix C; Figure C2). Relationships among individual measurement variables were also evaluated by plotting the integrated toxicity descriptors (HMDS Axes 1 and 2) as well as the two toxicity endpoints against individual concentrations of metals and nutrients and grain size distribution (Appendix C; Figures C3 to C5).

General contaminant descriptor relationships

For the integrated toxicity descriptor HMDS Axis 2 ('ToxAxis 2'), the contaminant descriptor ("metPC1") accounts for ~44% of the variability (P =0.004 for the regression) (similar to that found in 2002 study). There are no significant relationships for the HMDS Axis 1 toxicity descriptor ("ToxAxis 1").

ToxAxis2 = - 0.000 - 0.226 metPC1

A stronger relationship is found between the individual toxicity endpoints and the integrated metal contaminant descriptors (Figure C2). For *Chironomus* survival, the regression is significant for metPC1 at P< 0.001; the model accounts for ~75% of the variability. There is no significant relationship for *Hyalella* survival.

Chironomus survival = 0.826 + 0.0594 metPC1 - 0.105 metPC2

Individual contaminant relationships

Best subset regressions of HMDS Axes 1 and 2 and the individual toxicity endpoints and the individual measurement contaminant, nutrient, and grain size variables show some significant relationships ($P \leq 0.001$) (Figures C3 to C5). The following model explains ~83% of the variance in the Axis 1 toxicity descriptor:

ToxAxis 1 = - 34.8 - 5.39 log Cr + 6.25 log Mn + 10.9 log Zn - 2.02 Silt + 24.1 NO_3/NO_2
(R^2_{adj}= 82.8%, P< 0.001)
Predictors are significant at $P \leq 0.017$; predictors with positive regression coefficients are potentially toxic to *Hyalella* survival.

For the Axis 2 toxicity descriptor, the following model explains 75% of the variance:

ToxAxis 2 = 23.8 - 6.56 log Ni - 5.24 log TP + 1.16 log TOC
Predictors are significant at $P \leq 0.033$; predictors with positive regression coefficients are potentially toxic to *Hyalella* and *Chironomus* survival.

For individual endpoints, a significant relationship for *Hyalella* includes sediment nutrient variables (total phosphorus, total organic carbon). Similar results were found in the 2002 study, where most of the variation in *Hyalella* survival (36%) was explained by sediment nutrients (total Kjeldahl nitrogen and total phosphorus) (Milani and Grapentine 2005). A significant relationship was also found with total mercury (which is significantly correlated to TOC); however, the relationship is weaker than that seen with TOC. For the *Chironomus* model, the predictors (methyl Hg and Ni) have positive regression coefficients, which indicate a decrease in toxicity with increase in contaminant concentration. This does not suggest a causal relationship.

Hyalella survival = - 4.83 + 2.05 log total phosphorus - 0.598 log total organic carbon
(R^2_{adj} = 60.5%, P = 0.002)

Hyalella survival = - 3.46 + 1.47 log total phosphorus - 0.298 log total Hg
(R^2_{adj} = 31.0%, P = 0.043)

Chironomus survival = - 3.06 + 0.188 log methyl Hg + 2.64 log Ni
(R^2_{adj} = 83.2%, $P < 0.001$)

Overall, toxicity is best explained by the measured metal contaminant (not Hg) and/or nutrient variables. Individual contaminant descriptors explain more of the variability than the integrated contaminant descriptor. The weakest relationship between toxicity and sediment contaminant concentration for individual endpoints is for *Hyalella* survival (R^2_{adj} = 61%). Regression of *Chironomus* survival and individual metals produce a stronger relationship (R^2_{adj} = 83%). However, predictors with coefficients indicating decrease in toxicity with increase in contaminant concentration do not suggest causal relationships. These include the positive coefficients for the survival variables. After excluding predictors not indicative of toxicity relationships, it is not clear what is causing toxicity to *Chironomus* or *Hyalella*, as neither is associated with any metal contaminant. (*Hyalella* toxicity is most strongly associated with decreased total organic carbon.) There may be unmeasured stressors involved as well as substrate related issues with respect to the paper fibre at some sites, similar to what was concluded from the 2002 study. Increased methyl mercury may have partially explained toxicity to the mayfly *Hexagenia* in the 2002 study; however, in the current study, there are no effects on *Hexagenia*.

3.4 Biomagnification Potential

3.4.1 Invertebrate mercury levels

Total mercury

On a whole-body, uncleared-gut, dry weight basis, total Hg accumulation in chironomids and oligochaetes is similar across sites (chironomids: 209 to 545 ng/g, median 302 ng/g; oligochaetes: 142 to 691 ng/g, median 389 ng/g; Table 8). However, oligochaetes have slightly higher [THg]s than chironomids at 11 of 15 test sites and at 4 of 5 reference sites. (Similar results were found in 2002.) Logged concentrations of THg in chironomids and oligochaetes (test and reference sites) are significantly correlated ($r=0.73$, $P=0.0002$).

Comparison of total Hg in benthic invertebrates at test sites to reference sites

Figure 7 shows the concentrations of total mercury in chironomids and oligochaetes at test sites compared to the Lake Superior reference sites.

Chironomids 9 test sites are above the 99th percentile of the reference site concentrations (S05-01 is just slightly above) (Figure 7). Excluding reference, the lowest total Hg accumulation in chironomids is at S05-10, S05-11 and S05-15, which show very similar concentrations (209 to 218 ng/g). The greatest accumulation occurs at Sites S05-03 (545 ng/g) and S05-12 (507 ng/g).

Oligochaetes 14 of 15 test sites are above the 99th percentile of the reference site concentrations (Figure 7). Excluding reference, the lowest total mercury accumulation in oligochaetes is at S05-10 (142 ng/g), and the greatest accumulation is at S05-13 (691 ng/g) and S05-05 (615 ng/g), which are similar in concentration.

Methyl mercury

Oligochaetes show a greater range of methyl Hg accumulation across Thunder Bay sites (0.2 to 51 ng/g; median 2.2 ng/g) compared to the chironomids (5.3 to 78 ng/g; median 32; Table 9). However, chironomids have higher [MeHg]s than oligochaetes at all sites except one (~1 order of magnitude higher in some cases), different than that seen for THg (oligochaete [THg] is higher at most sites). Logged concentrations of MeHg in chironomids and oligochaetes are significant correlated (R=0.57, P=0.0086).

Comparison of methyl Hg in benthic invertebrates at test sites to reference sites

Figure 8 shows the concentrations of methyl mercury in chironomids and oligochaetes at test sites compared to the Lake Superior reference sites.

Chironomids 10 test sites are above the 99th percentile of the reference site concentrations (Figure 8). Excluding reference, the lowest methyl mercury accumulation in chironomids is at S05-01 (5.3 ng/g) and S05-02 (7.0 ng/g), and the greatest accumulation occurs at S05-07 and S05-08, which show similar concentrations (78 and 76 ng/g, respectively).

Oligochaetes 3 test sites are above the 99th percentile for the reference site concentrations (Figure 8). Among test sites, the lowest methyl mercury accumulation is at S05-02 (0.2 ng/g) and the greatest accumulation is seen in oligochaetes at S05-14 (51 ng/g).

3.4.2 Biota-sediment accumulation factors

Biota-sediment accumulation factors (BSAFs) for total and methyl mercury are shown by area (reference and Thunder Bay) for each taxon in Figure 9. For THg, BSAFs for Thunder Bay sites (based on whole-body, uncleared-gut concentrations) are similar for both taxa, ranging from 0.08 to 4.9 (median 0.5) and from 0.09 to 4.27 (median 0.9) for chironomids and oligochaetes, respectively. Total Hg BSAFs are >1 for 6 of the 15 sites. For MeHg, BSAFs are higher than those for THg. Chironomid BSAFs, ranging from 0.6 to 70.9 (median 6.5) at test sites, are higher than those for oligochaetes, which range from 0.04 to 56.7 (median 0.4). Higher methyl Hg BSAFs were also observed for chironomids in the 2002 study, which had a similar BSAF range (1.2 to 51.1; Milani and Grapentine 2005). Methyl Hg BSAFS are >1 for 14 of the 15 test sites. Sites S05-07, S05-08, S05-10 and S05-14 have the highest BSAFs for total and methyl Hg. Reference site BSAFs for THg and MeHg are all >1 and much higher than those for the test sites, especially for MeHg. For THg, BSAFs range overall from 2.7 to 20.2, and for MeHg range overall from 37 to 680 for reference sites. Tissue concentrations do not increase as much as sediment concentrations at highly contaminated sites; therefore, higher BSAFs observed for the reference sites are not unusual, and was also observed in the 2002 study. Gut contents are included in the mercury analyses of the biota, which could obscure true BSAFs. As the amount of sediment in the gut increases, the measured BSAF will converge to 1. A true BSAF < 1 will be overestimated because the concentration in the sediment is greater than the tissue concentration, whereas a true BSAF >1 will be underestimated because sediment concentrations are lower than that found in the tissue (Bechtel Jacobs 1998).

3.4.3 Relationships between mercury concentrations in benthic invertebrates and sediment

Total mercury

Concentrations of THg in each invertebrate taxon vs. THg in sediment are plotted in Figure 10, with fitted regression lines using sediment [THg] alone as the predictor (Model A). The slopes are similar for the two taxa (Table 10) and the relationships are significant (chironomid: $P < 0.001$, $R^2 = 0.524$; oligochaete: $P < 0.001$, $R^2 = 0.525$) (Figure 10, Table 10). Predictions of $[THg]_{inv}$ are improved for the chironomids with manganese (Mn), clay and dissolved oxygen (O_2) in the model and for the oligochaetes with iron (Fe) as an additional predictor (Model B) (Table 10); R^2_{adj} values are increased to 0.816 and 0.759 for the chironomids and oligochaetes, respectively. For both taxa, $[THg]_{sed}$ is the strongest predictor ($P \leq 0.001$) in the Model B scenarios; coefficients for Mn and Fe are positive and clay and dissolved oxygen are negative.

Methyl mercury

The relationships between MeHg in benthic invertebrates and MeHg in sediment are not significant for either taxon, and the slope is negative for the oligochaete model (Figure 11, Table 10). With $[MeHg]_{sed}$ alone as the predictor (Model A), the R^2_{adj} are 0.072 and 0.051 ($P= 0.134$ and 0.173) for chironomids and oligochaetes, respectively. For the chironomids, the regression accounts for more variability in $[MeHg]_{chir}$ with total organic carbon, total Kjeldahl nitrogen (water), alkalinity, conductivity and dissolved O_2 in the model ($P< 0.0001$, $R^2_{adj} = 0.772$) (Model B). All additional coefficients are negative except dissolved O_2, and MeHg is the strongest predictor ($P \leq 0.0001$) in the model. For the oligochaetes, the regression becomes significant with sediment total nitrogen in the model ($P< 0.0001$, $R^2_{adj} = 0.550$). (This is the same as seen in the 2002 study.) The coefficient for total nitrogen is negative. For both taxa, low nutrient conditions are important in the uptake of MeHg; low nitrogen conditions are important for oligochaetes and low TOC and nitrogen conditions are important for chironomids.

3.4.4 Predictions of methyl mercury concentrations in receptors

Presentation of model outcomes

Predicted concentrations of methyl mercury in each receptor species at each sampling site, calculated by multiplying observed methyl mercury concentrations in invertebrates (wet weight values – from Table 9) by the appropriate FCMs are provided in Table 11. Receptor MeHg concentrations are presented for minimum (min), intermediate (med) and maximum (max) levels of mercury exposure and uptake scenarios. In Table 11, three criteria are marked: (1) sites that are above the tissue residue guideline (TRG) for the fishes, (2) sites that are above the 99th percentile of the predicted $[\text{MeHg}]_{\text{rec}}$ for the reference, and (3) sites that are above both. The TRG applies only to the fish receptors and it refers to the concentration of MeHg in the diets of wildlife that consume aquatic biota. The TRG used for MeHg is the lowest of the reference concentrations derived by Environment Canada (2002) for the protection of wildlife receptors in the AOC that consume aquatic biota: 92 ng/g ww. This pertains to the American mink (Table 12 of Environment Canada 2002). The recommended TRG for the protection of *all* wildlife species – 33 ng/g ww – was not considered appropriate because it is based on the reference concentration for the Wilson's Storm Petrel, which is not native to the Thunder Bay area.

Exceedences of criteria

Methyl Hg – minimum

The low predictions of $[\text{MeHg}]_{\text{rec}}$ result in 3 Thunder Bay sites exceeding the 99th percentile for the reference sites (Table 11a). Of these, 1 site (S05-14) exceeds the TRG for perch. There are no exceedences of the TRG predicted for any receptor for the reference sites.

Methyl Hg – intermediate

The medium predictions of $[\text{MeHg}]_{\text{rec}}$ result in 4 Thunder Bay sites exceeding the 99th percentile for the reference sites (Table 11a). Of these, 3 sites (S05-07, S05-08, and S05-14) exceed the TRG for perch and 4 sites (S05-07, S05-08, S05-11, and S05-14) exceed the TRG for Walleye. There are no exceedences of the TRG predicted for any receptor for the reference sites. Figure 6 shows the 2005 and 2002 sites where potential for adverse effects due to mercury biomagnification occurs (based on the intermediate exposure and uptake scenario or "average" conditions).

Methyl Hg – maximum

The maximum predictions of $[\text{MeHg}]_{\text{rec}}$ result in 6 sites exceeding the 99th percentile for the reference sites (Table 11a). Of these, 5 sites (S05-04, S05-07, S05-08, S05-11, and S05-14) exceed the TRG for perch and all 6 sites (S05-03, S05-04, S05-07, S05-08, S05-11, and S05-14) exceed the TRG for Walleye. However, all reference and Thunder Bay sites exceed the TRG for walleye. In comparison, there are no reference site exceedences of the TRG predicted for the sucker or perch under the maximum exposure and uptake scenario.

3.5 Quality Assurance/Quality Control

Three replicate van Dorn and mini-ponar samples were collected at test site S05-05 and reference site 5103. Variability among site replicates in a measured analyte has three sources: natural within-site heterogeneity in the distribution of the analyte in sediment or water, differences in handling among samples, and laboratory measurement error. Among-triplicate variability indicates the overall "error" associated with quantifying conditions at a site based on a single sample.

Variability among field-replicated sites, expressed as the coefficient of variation (CV), is shown in Appendix D; Table D1. The CVs range from 0.3 to 128 % (median 6.1%), not uncommon for field-replicated samples (samples were taken from three separate box core drops); most CVs (85%) are below 20%. Differences in variability are seen among sites and among parameters for the same site. The highest variability is noted for total Kjeldahl nitrogen (overlying water) and total organic carbon for site 5103.

Laboratory duplicate measurements for sediment and overlying water variables and corresponding analyses of reference materials for Caduceon Laboratory are provided in Appendix D; Tables D2 and D3. There is good agreement between sample duplicates. The overall mean relative percent difference (RPD) for sample duplicates measurements is low

(3.2%, range: 0 to 22.2%; Table D2). Overall mean recovery for three reference materials (LKSD-3, GS89-2 and WH89-1), is 88%, ranging from 30 (molybdenum in LKSD-3) to 107% (Table D3). Recovery in reference materials is good for most parameters measured (>80%).

For Flett Laboratory, sample duplicates and repeats for sediment and biota samples and are shown in Tables 5 and 8, respectively. Percent mercury recoveries for sample spikes, quality control samples, and reference standards are provided in Appendix D; Tables D4 and D5. For sediment samples, there is good agreement between sample duplicates and repeats, with the RPD ranging from 1.8 to 25.0 (mean = 10.4). The percent Hg recovery for sample spikes ranges from 80.8 to 105.0% (mean = 96.0%); the mean RPD for sample spikes is low, ranging from 0.1 to 4.0 (Table D4). Percent recoveries for quality control samples (Mess-2, OPR, IAEA 405, Alfa) range from 66.6 to 116.6% (overall mean = 96.5%), and percent recovery for mercury reference standards (Hg STD 1 to 5) range from 98 to 102 (overall mean = 100%). For biota samples, there is very good agreement between sample duplicates, with the RPD ranging from 0.2 to 1.3. The percent Hg recovery for sample spikes ranges from 90.3 to 108.8% (mean = 99.2%); the mean RPD for sample spikes ranges from 1.0 to 1.8 (Table D5). Percent recoveries for quality control samples (Dorm-2, OPR, Alfa) range from 90.9 to 117.7% (overall mean = 98.5%), and percent recovery for mercury reference standards (Hg STD 1 to 5) range from 96 to 102 (overall mean = 100%). Overall, recoveries are good, indicating confidence in the Hg values reported for sediment and biota samples.

3.6 Decision–Making Framework for Sediment Contamination: 2002 and 2005 data

A risk-based, decision-making framework for the management of sediment contamination was recently developed by the Canada-Ontario Agreement Sediment Task Group using four lines of evidence (sediment chemistry, toxicity, community structure and potential for biomagnification). This decision framework was developed from the Sediment Triad and BEAST frameworks, and is described in Chapman and Anderson (2005). The overall assessment of a test site is achieved by integrating the information obtained both within and among the four lines of evidence. This framework was applied to the Thunder Bay North Harbour studies (2002 and 2005 data). The community structure component of the framework was not conducted in the 2005 study; therefore, the assessment for 2005 sites is based on three lines of evidence.

The decision matrix for the weight of evidence categorization of Thunder Bay sites is shown in Table 12. Substances exceeding SELs are indicated. For the sediment chemistry column, sites with exceedences of a sediment quality guideline (SQG) – low are indicated by "◉"; sites with SQG-high exceedences by "●". For the toxicity and benthos alteration columns, sites determined from BEAST analyses as toxic/severely toxic or different/very different from reference, respectively, are indicated by "●"; and sites determined as potentially toxic or possibly different from reference by "◉". Sites with no SQG exceedences, no sediment toxicity, or benthic communities equivalent to reference conditions are indicated by "○".

Management actions

Management actions are indicated for 3 sites: P1, P6 and P12 (2002 sites). This is due to elevated Hg concentrations, altered benthic communities and sediment toxicity at all 3 sites and also due to the potential risk of mercury biomagnification at P6 and P12. Whereas assessments of 2002 sites are based on four lines of evidence, 2005 assessments are based on three lines of evidence (benthic community structure was not assessed). In 2002, 17 of the 19 sites had different benthic communities from reference. If the assumption is made that benthic communities would most likely be different from reference for the 2005 sites, the assessment outcome would change for a number of sites. In addition to Sites P1, P6 and P12, management actions would also be indicated for S05-01, S05-11, S05-13, and S05-16, due to elevated sediment mercury, sediment toxicity and benthos alteration. (Site S05-11 also has potential risk of mercury biomagnification.)

Determine reason for benthos alteration

This is required for 14 of the 19 sites sampled in 2002. Sediment [Hg] is above the LEL or SEL at all sites except P22 and P23. Sites P22 and P23 also require the need to fully assess risk of biomagnification (see below). If the assumption is made that benthic communities are different from reference for the 2005 sites, then determining the reasons for benthos alteration would also be indicated for S05-02 to S05-08, S05-12 and S05-15.

Determine reason for sediment toxicity

This is required for 6 sites: P3, P7, S05-01, S05-11, S05-13 and S05-16. Sediment [Hg] is above the LEL (2 sites) or SEL (4 sites) and there is no benthos alteration at 2002 (P3, P7) sites. Communities may have acclimated/adapted or there is insufficient stress to cause population-level responses. There is, however, the potential for adverse effects at these sites and thus the benthic community should be monitored for change in status. Toxicity may be related to paper matter visually observed at 4 of the 6 sites. Sites P7 and S05-11 also require full risk assessment for biomagnification (see below).

Fully assess risk of biomagnification

This is required for 7 sites: P7, P22, P23, S05-07, S05-08, S05-11 and S05-14. Sediment [Hg] is below the LEL at 4 of these sites (P22, P23, S05-08 and S05-14). Significant biomagnification can be determined when there is site specific evidence, such as significant evidence from fish advisories or previous research in the area. If sufficient evidence exists, significant biomagnification is indicated by a "●" (replacing "○"), and management actions would be required.

The Sport Fish Contaminant Monitoring Program includes regular collections of Walleye, Lake trout and White sucker (as well as other fish species) from the Thunder Bay AOC (Inner and Outer Harbours). Sport fish consumption restrictions for total mercury begin at levels above 610 ng/g and total restriction is advised for levels above 1840 ng/g for the general population (OMOE 2005). For the sensitive population, restrictions begin at levels above 260 ng/g, and there is complete restriction for levels above 520 ng/g (OMOE 2005). The most recent survey of sport fish contaminant levels included collections from the Inner Harbour in 2002 (OMOE 2003).

Currently, there are consumption restrictions (4 meals per month) due to Hg for Walleye >60 cm long (general population) and complete restriction for Walleye >55 cm (sensitive population) (OMOE 2005). For the White sucker, consumption restrictions (4 meals per month) due to Hg start at fish >40 cm long and complete restriction at fish >50 cm (sensitive population) (OMOE 2005). There are also consumption restrictions due to Hg for Northern pike, Round whitefish (sensitive population) and Ling (sensitive population) for the Inner Harbour (OMOE 2005). These observations of [Hg] in receptor species residing in the Thunder Bay AOC suggest that mercury is accumulating in tissues of higher trophic level members of aquatic food webs. However, fish collections occurred around the mouths of the Kaministiquia and Mission Rivers, which are approximately 6-8 km south of the sampling area.

In 1998, young-of-the-year White sucker and adult Walleye were collected from the study area adjacent to the paper mill (Stantec 2003). Total Hg levels were reported as ranging from 11 to 86 ng/g ww for the young-of-year sucker and from 170 to 850 ng/g ww for the adult Walleye (length 40 – 56 cm). All Walleye values were above the TRG for the protection of fish consuming wildlife (92 ng/g), and in some cases, values were above consumption restriction levels (>610 ng/g). For the sensitive population, some Walleye values were above the complete restriction level (>520 ng/g). A more recent fish survey in the study area may be warranted to compare Hg levels in fish to the 1998 levels and to confirm that biomagnification is occurring in the sampling area. This could have implications on assessment outcomes for sites that show potential for biomagnification. The information from a new fish survey would reduce uncertainty in the assessment of the risk of mercury biomagnification.

4 CONCLUSIONS

4.1 Mercury Levels

Sediment (2002)

Sediment total and methyl Hg levels were elevated above reference at all 19 Thunder Bay sites. Total mercury ranged from 0.03 to 39.7 μg/g and exceeded the provincial Severe Effect Level at 7 sites. The highest mercury concentrations were found along the northern shore of the study area, and at the sites that contained the white fibrous paper material. The spatial pattern of these results was strong evidence for a local (as opposed to regional) source of mercury to the area.

Sediment (2005)

Thunder Bay total mercury concentrations range from 0.04 to 7.6 μg/g, exceeding the provincial Severe Effect Level at 4 of the 15 sites. Visual inspections at the time of sampling noted the presence of paper fibres at two sites (S05-01, S05-11), and total organic carbon is significantly related to total mercury concentrations. Sediment total and methyl Hg levels are elevated above reference conditions at all Thunder Bay sites. The highest Hg concentrations are found along the northern shore of the study area, and at the sites that contain paper fibres.

Benthic Invertebrates (2002)

Total and methyl mercury concentrations in 1 of the 2 resident invertebrate taxa assessed (chironomids) at the majority of Thunder Bay sites were elevated above those at reference sites. This indicated that benthic invertebrates accumulated Hg. Methyl mercury in sediment was significantly predictive of methyl mercury in chironomids indicating that sediment [MeHg] was affecting invertebrate [MeHg].

Benthic Invertebrates (2005)

Total mercury concentrations in the tissues of 1 or 2 of the resident invertebrate taxa assessed at all sites except one are elevated above those at reference sites. Methyl mercury concentrations in the chironomids are elevated above those at reference sites at 10 of the 15 sites.

There is a significant relationship between total mercury in the sediment and total mercury in the tissues of benthic invertebrates. For methyl mercury, with sediment [MeHg] alone as a predictor, there is no significant relationship for either taxon. For the chironomid model, the addition of sediment total organic carbon, and overlying water total Kjeldahl nitrogen, conductivity, alkalinity, and dissolved oxygen results in a significant strong relationship ($R^2_{adj} = 0.772$). For the oligochaete model, the addition of sediment total nitrogen results in a significant relationship ($R^2_{adj} = 0.550$). This indicates that other variables (both water and sediment variables for the chironomids and a sediment variable for the oligochaetes) are important in the uptake of MeHg in invertebrates.

4.2 Benthic Community Structure

In 2002, most Thunder Bay sites (17 of 19) had different communities than reference sites, generally due to either: (a) increased diversity and the absence of the pollution-sensitive haustoriid amphipods and enrichment of more tolerant organisms such as tubificids and chironomids, or (b) to decreased taxon diversity and increased abundance of more tolerant organisms such as tubificids and chironomids. Enrichment is likely associated with increased total organic carbon in some cases. Benthic community structure was not assessment in 2005.

4.3 Toxicity

2002

There was acute sediment toxicity at 5 of the 19 sites. Four of the five sites are located along the northern shore of the sampling area. Increased methyl mercury may partially explain toxicity to the mayfly; however, there may be unmeasured stressors and/or substrate related factors involved.

2005

Three sites are toxic/severely toxic; sites are located in the northern and in the central part of the sampling area (Figure 6). Acute toxicity is evident to *Hyalella* and /or *Chironomus*. There is no toxicity to *Hexagenia*, in contrast to that observed in the 2002 study, where acute toxicity to *Hexagenia* was evident at three sites along the northern part of the sampling area. Metal contamination (as either the integrated metal descriptor "metPC1" or individual metal concentrations) appears to be most strongly related to toxicity; significant individual endpoint – individual contaminant relationships do not indicate mercury as a potential cause of toxicity. Unmeasured stressors or substrate related factors may be responsible for toxicity.

4.4 Potential for Biomagnification

2002

Under the intermediate mercury-exposure and uptake assumptions, 5 sites have predicted [MeHg]s in receptors higher than the TRG and the 99th percentile of the reference site $[\text{MeHg}]_{\text{rec}}$. This indicated that mercury could bioaccumulate in Yellow perch and Walleye to levels that were not protective of adverse effects at 5 sites. (See Figure 6 for the location of these sites.)

2005

Under the intermediate exposure and uptake scenario ("average" conditions), 4 sites have predicted [MeHg] in receptors higher than the TRG and the 99th percentile of the reference site $[\text{MeHg}]_{\text{rec}}$.. This indicates that mercury could bioaccumulate in Yellow perch and Walleye to levels that are not protective of adverse effects at 4 sites. (See Figure 6 for the location of these sites.)

4.5 Decision-Making Framework for Sediment Contamination

2002 and 2005

The decision-making framework described in Chapman an Anderson (2005) was applied to data from 2002 and 2005 Thunder Bay studies. Management actions are indicated for 3 sites. Eight sites require no further action. For remaining sites, determine reasons for benthos alteration or sediment toxicity, and/or fully assess risk of mercury biomagnification are indicated. If benthic communities are assumed to be different from reference for the 2005 sites, then management actions would be indicated for an additional 4 sites for a total of 7.

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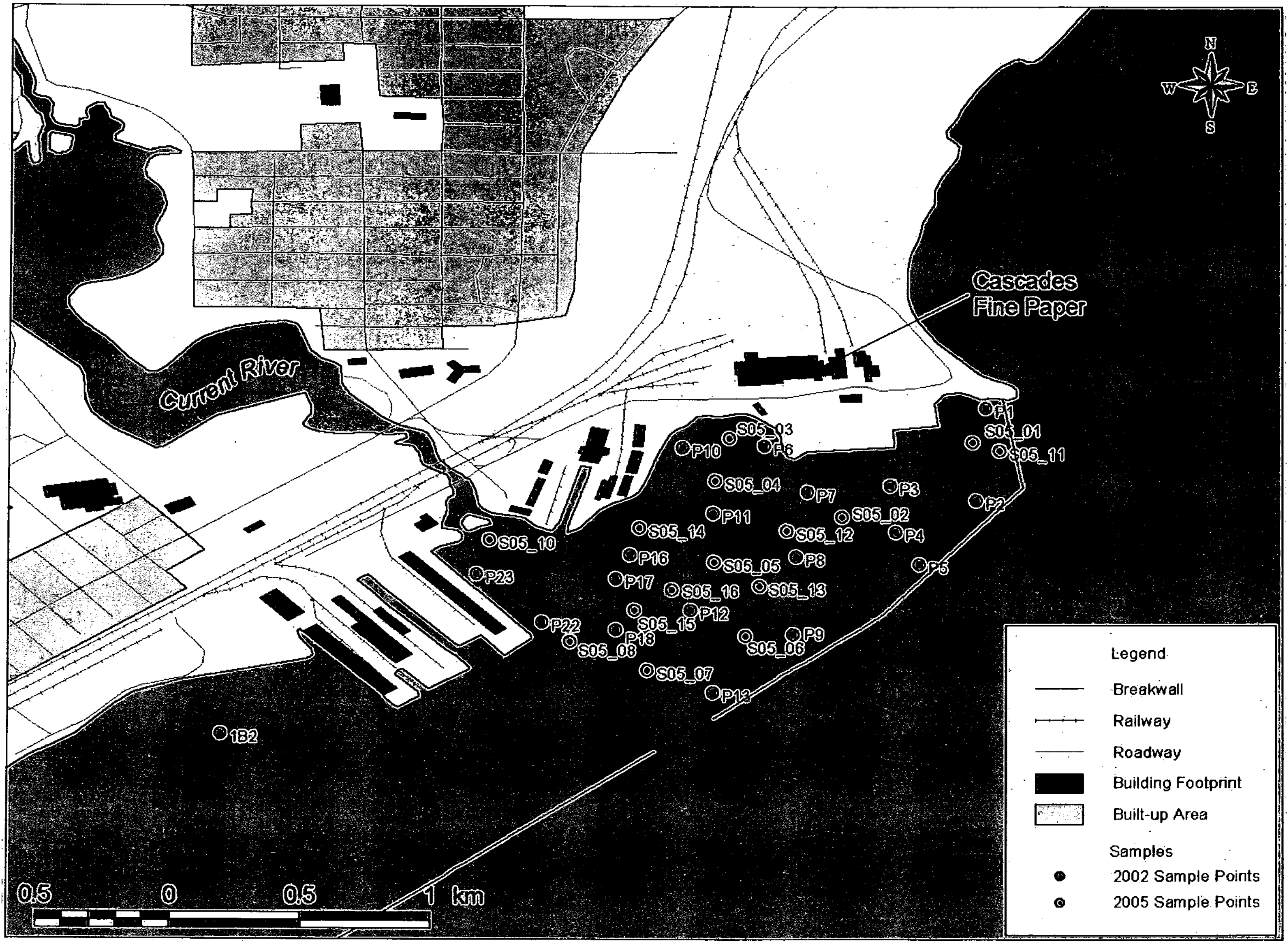


Figure 1. Location of test sites in Thunder Bay North Harbour in 2002 (green) and 2005 (red).

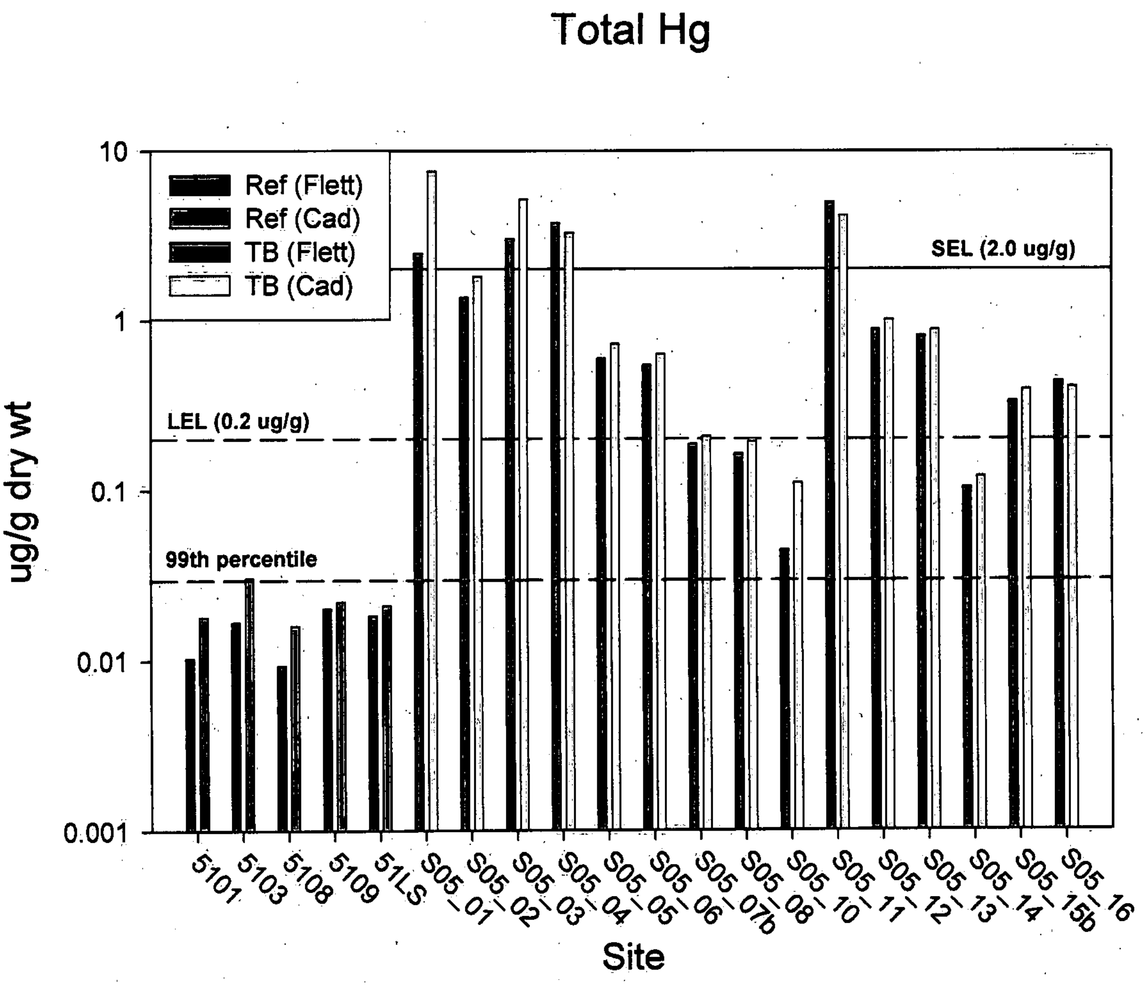


Figure 2. Sediment total mercury concentration (µg/g dw) in Thunder Bay (TB) sites (black/grey) and reference (Ref) sites (green) in 2005. Flett and Caduceon (Cad) laboratory results are shown.

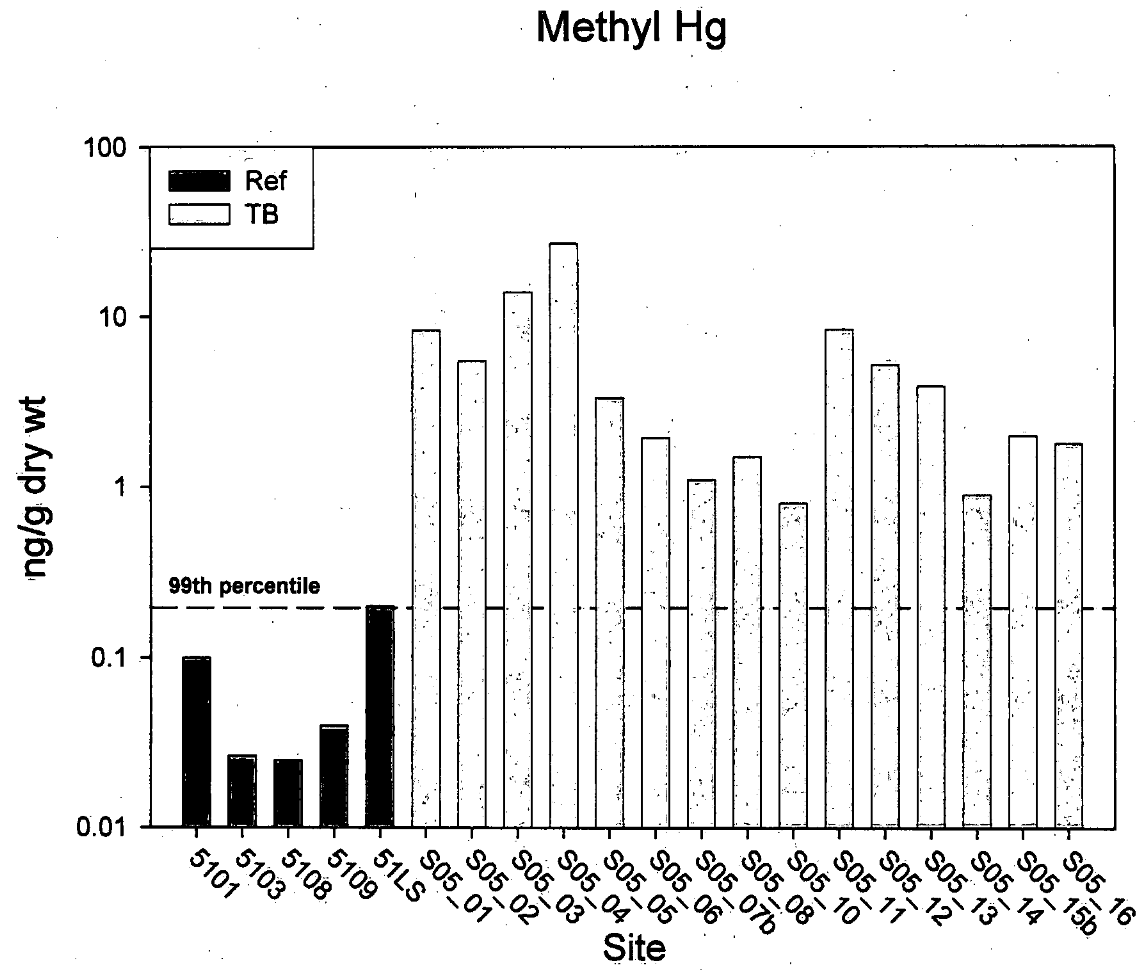


Figure 3. Sediment methyl mercury concentration (ng/g dw) in Thunder Bay (TB) sites (grey) and reference (Ref) sites (green) in 2005.

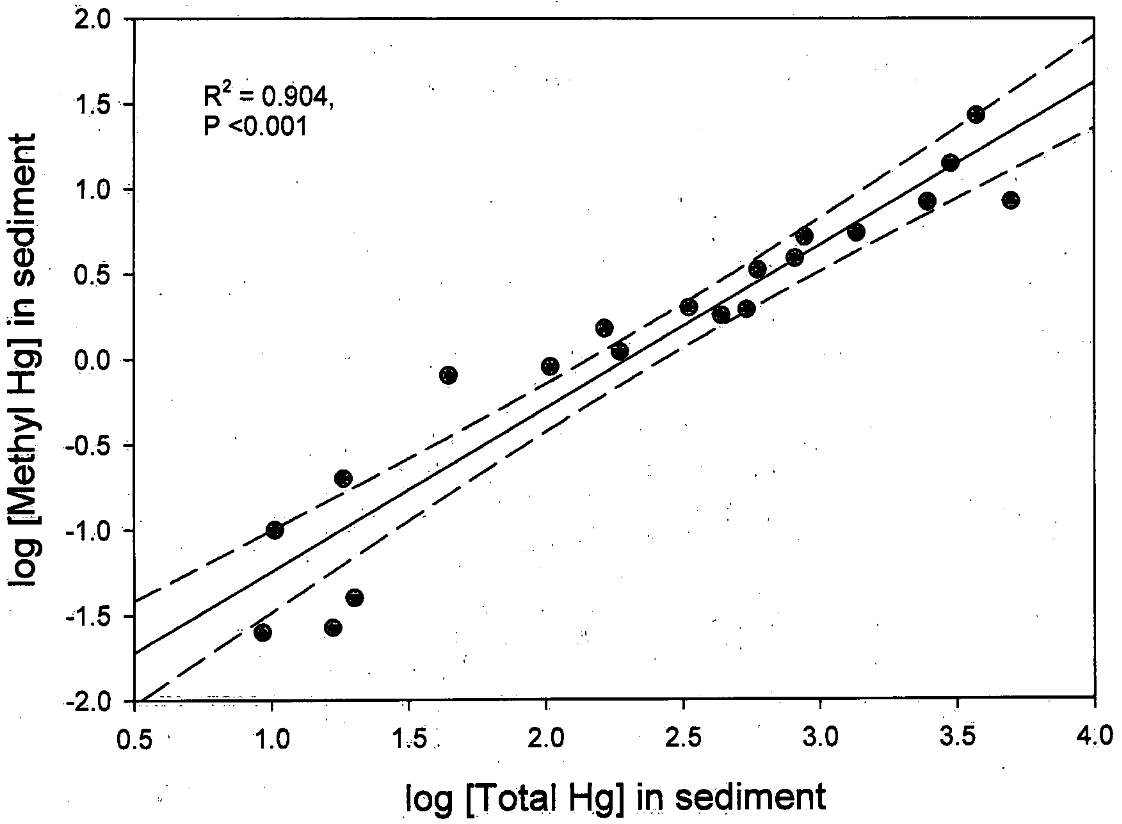


Figure 4. Log scatter plot of methyl mercury versus total mercury in sediment in 2005. The 95% confidence interval for the regression equation is shown by the dashed lines.

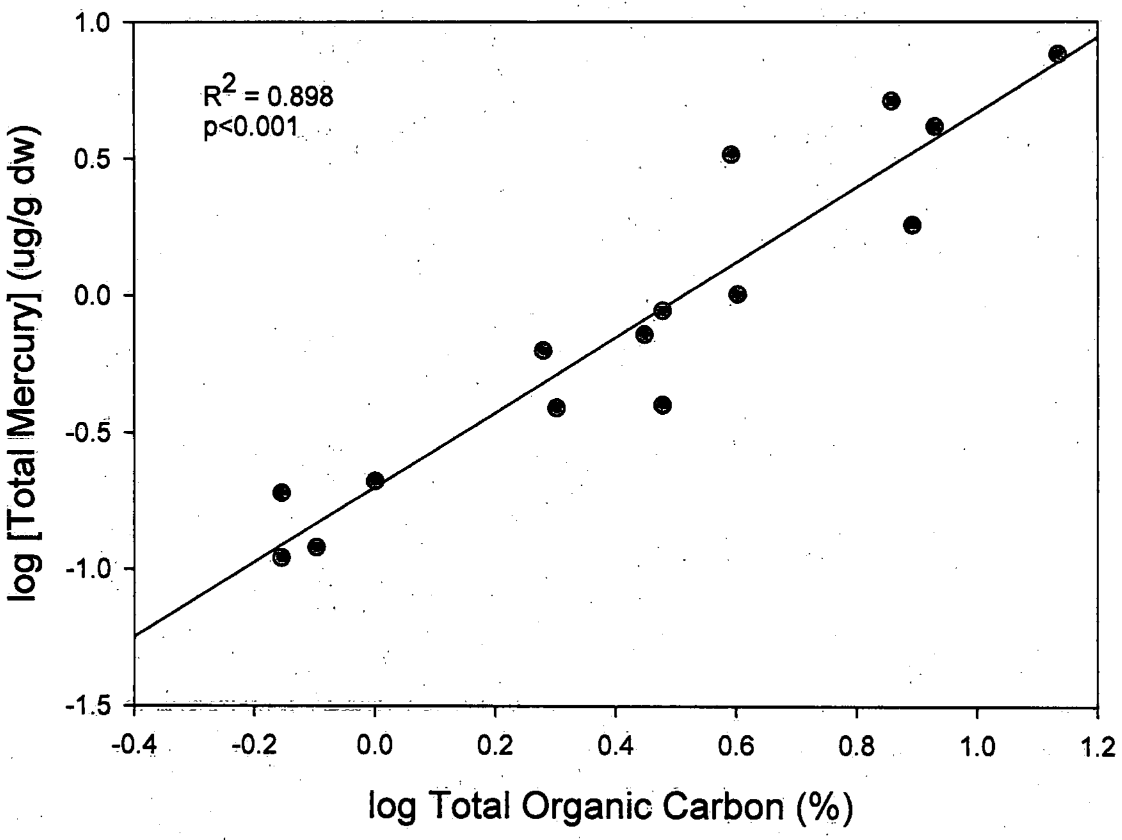


Figure 5. Relationship between total mercury and total organic carbon in sediment in 2005.

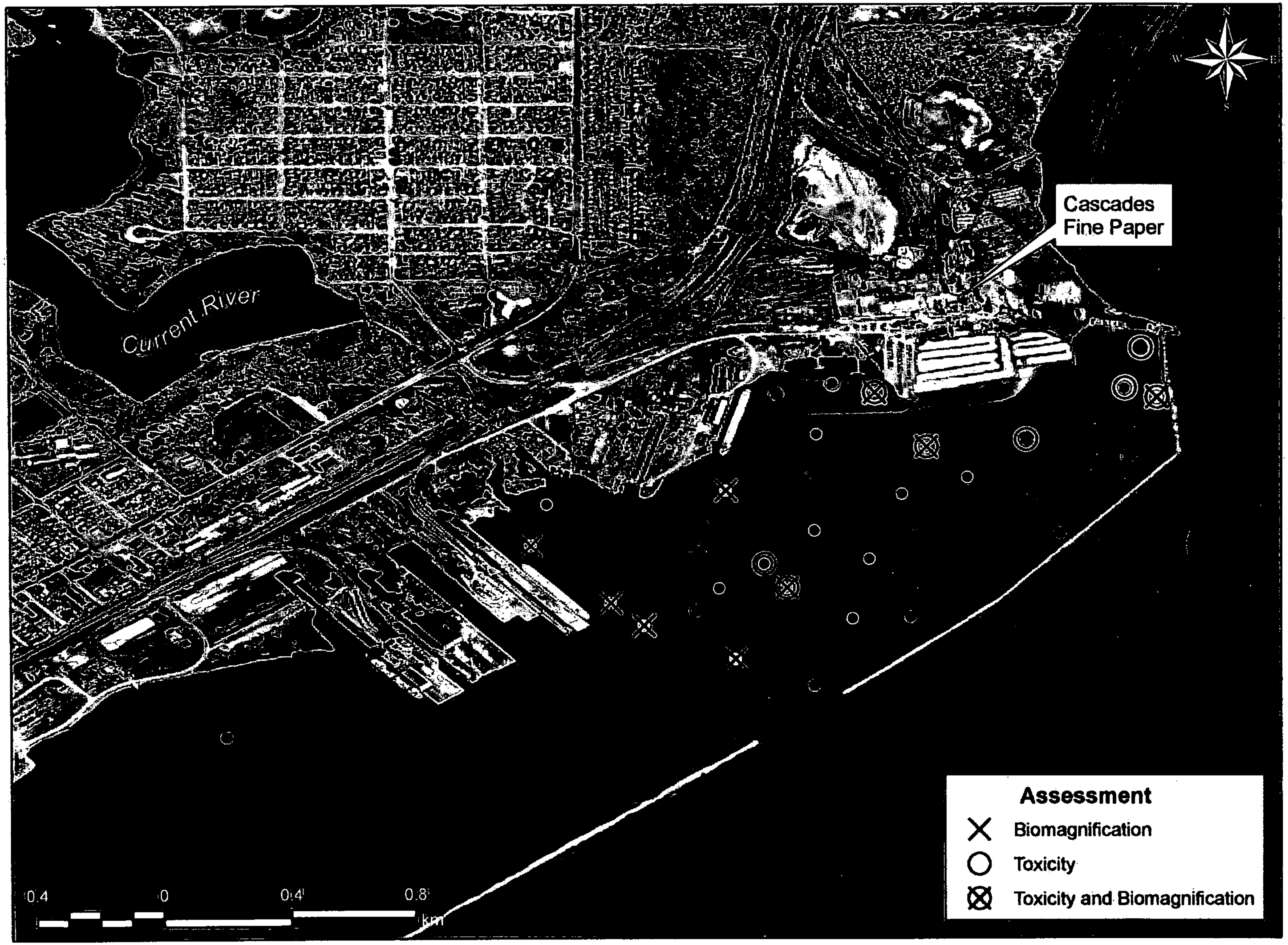


Figure 6. Thunder Bay 2002 (green) and 2005 (red) sites that show toxicity (Band 3 or 4 from BEAST assessment) and potentially adverse effects due to mercury biomagnification (based on intermediate or "average" conditions).

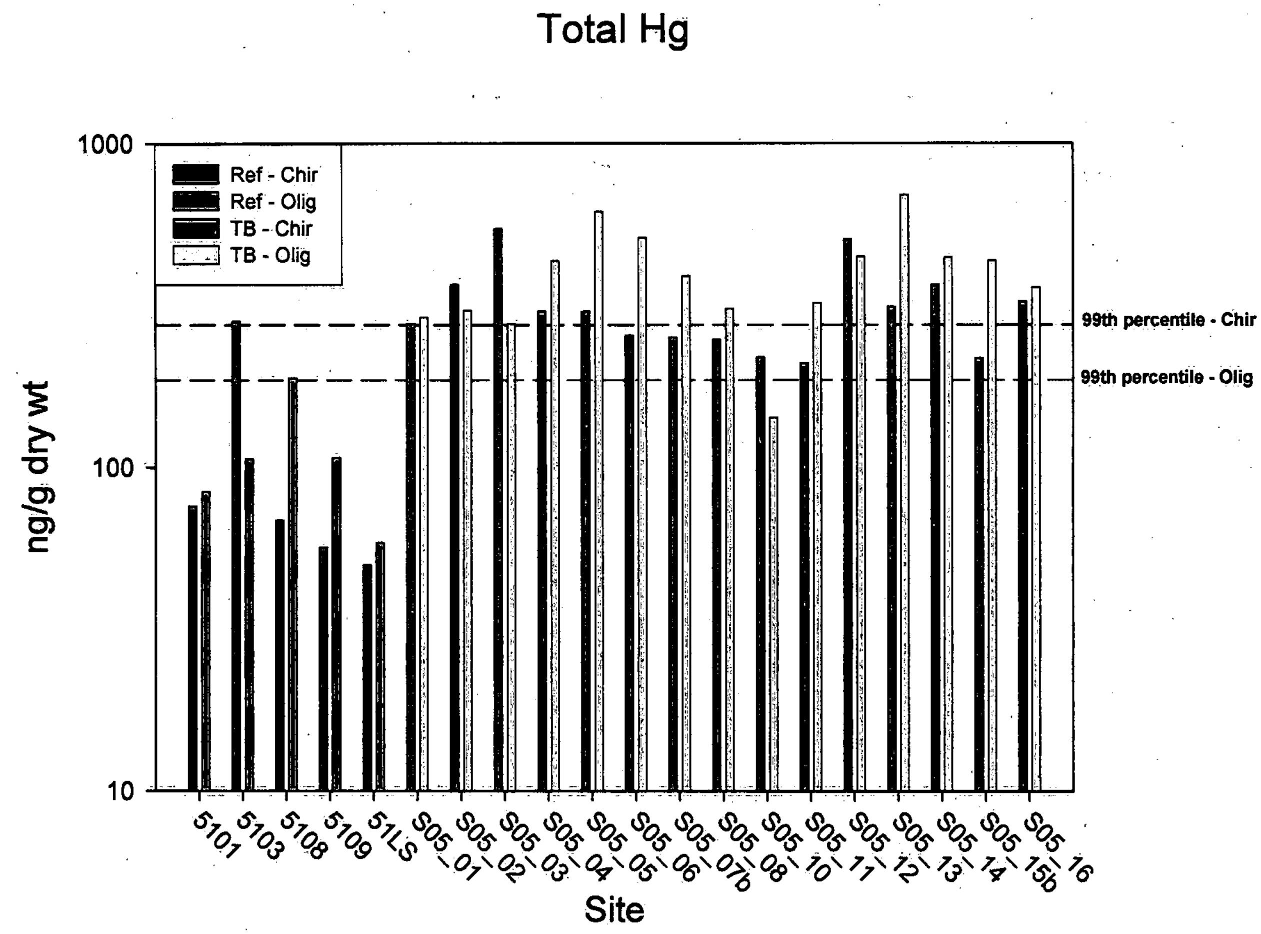


Figure 7. Benthic invertebrate total mercury concentration (ng/g dw) in Thunder Bay (TB) sites (grey) and reference (Ref) sites (green) in 2005.

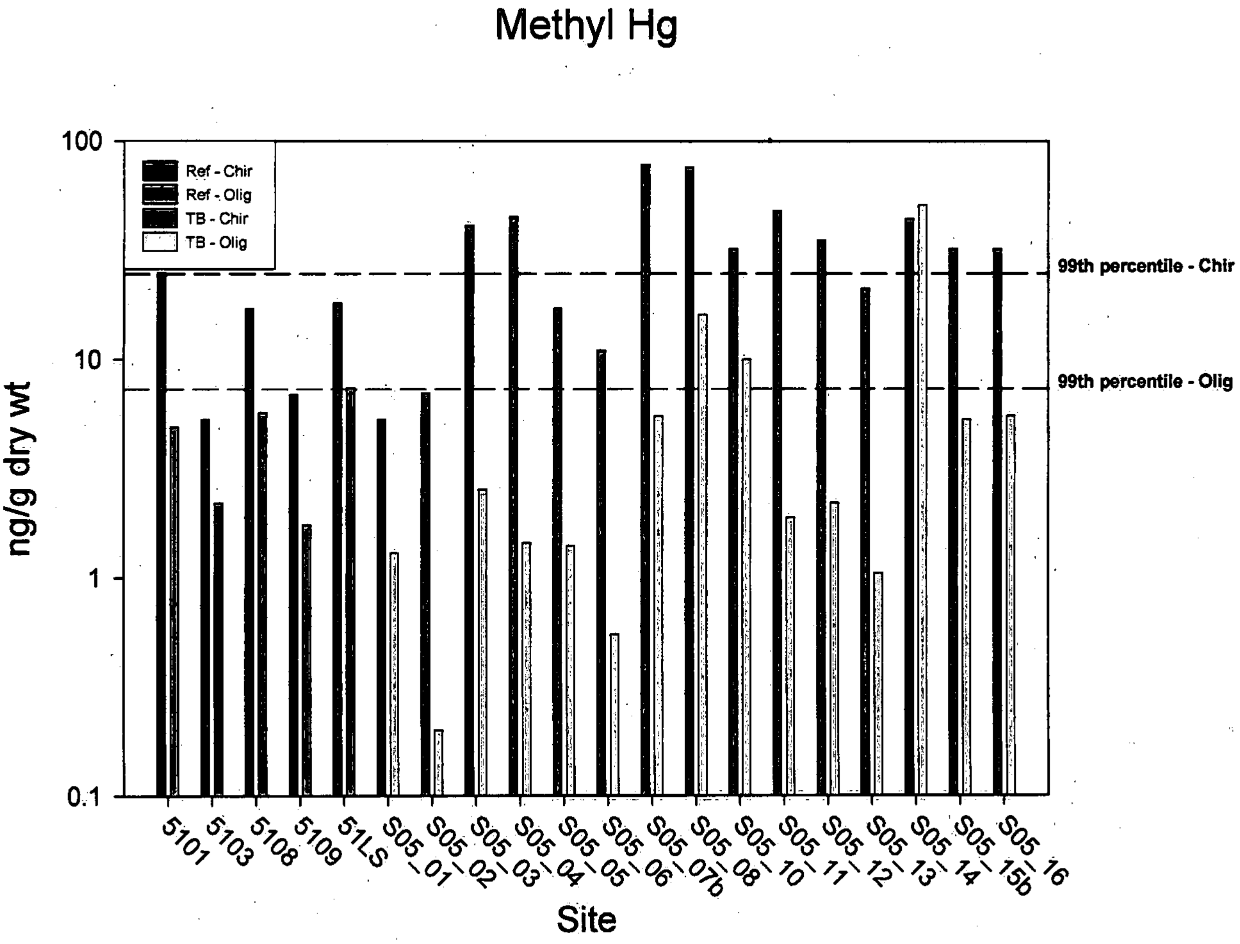


Figure 8. Benthic invertebrate methyl mercury concentration (ng/g dw) in Thunder Bay (TB) sites (grey) and reference (Ref) sites (green) in 2005.

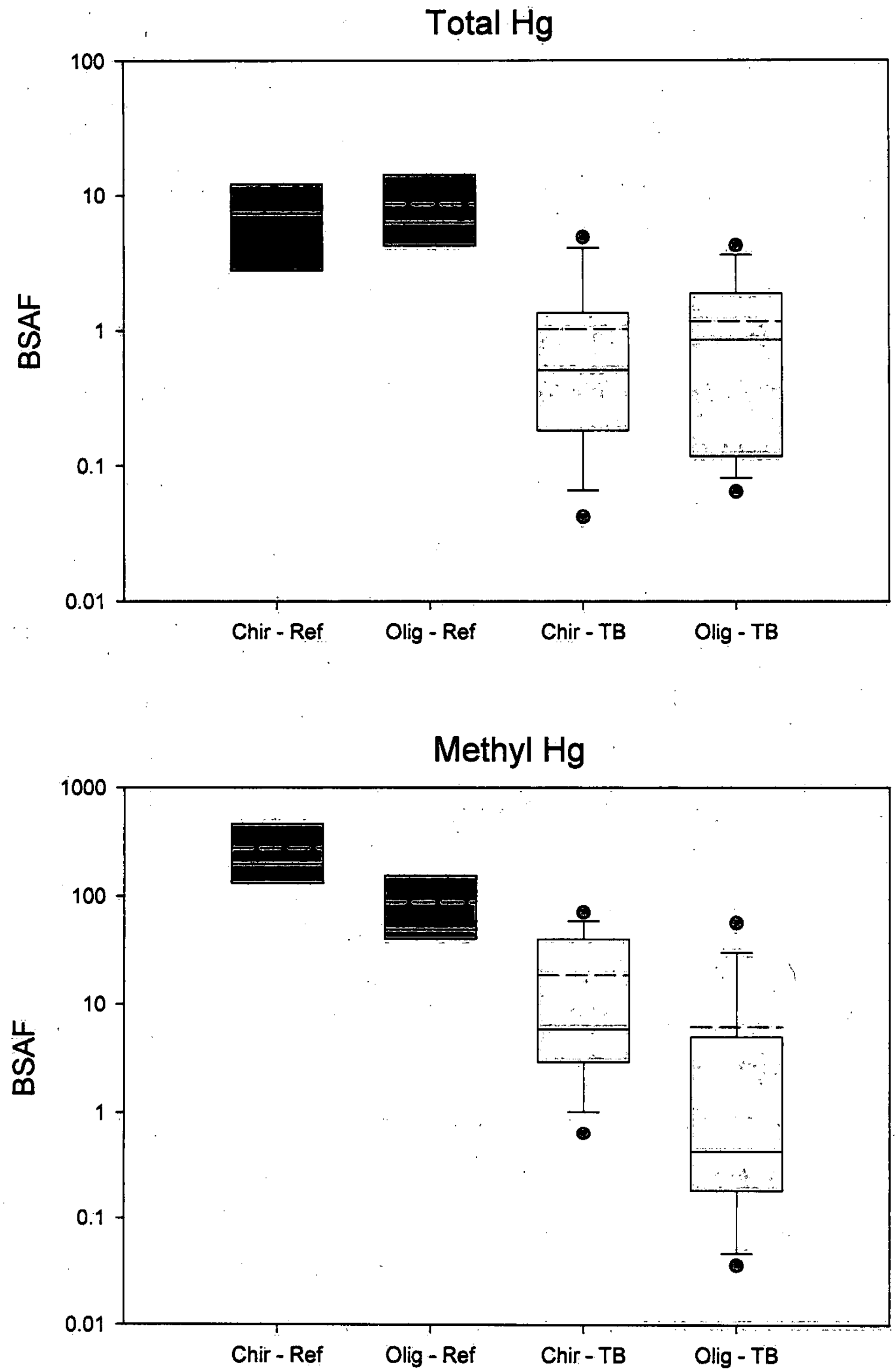


Figure 9. Biota-sediment accumulation factors (BSAF) for Thunder Bay (TB -grey) and reference sites (Ref - green) in 2005. Boxplots of BSAFs (= $[Hg]_{inv}$ / $[Hg]_{sed}$) for each taxon for each area show 90th and 10th percentile (whiskers above and below boxes), inter-quartile ranges (box boundaries closest and farthest from zero), median (horizontal line within boxes) and mean (dotted line). Outliers (solid circles) are shown for Thunder Bay sites only.

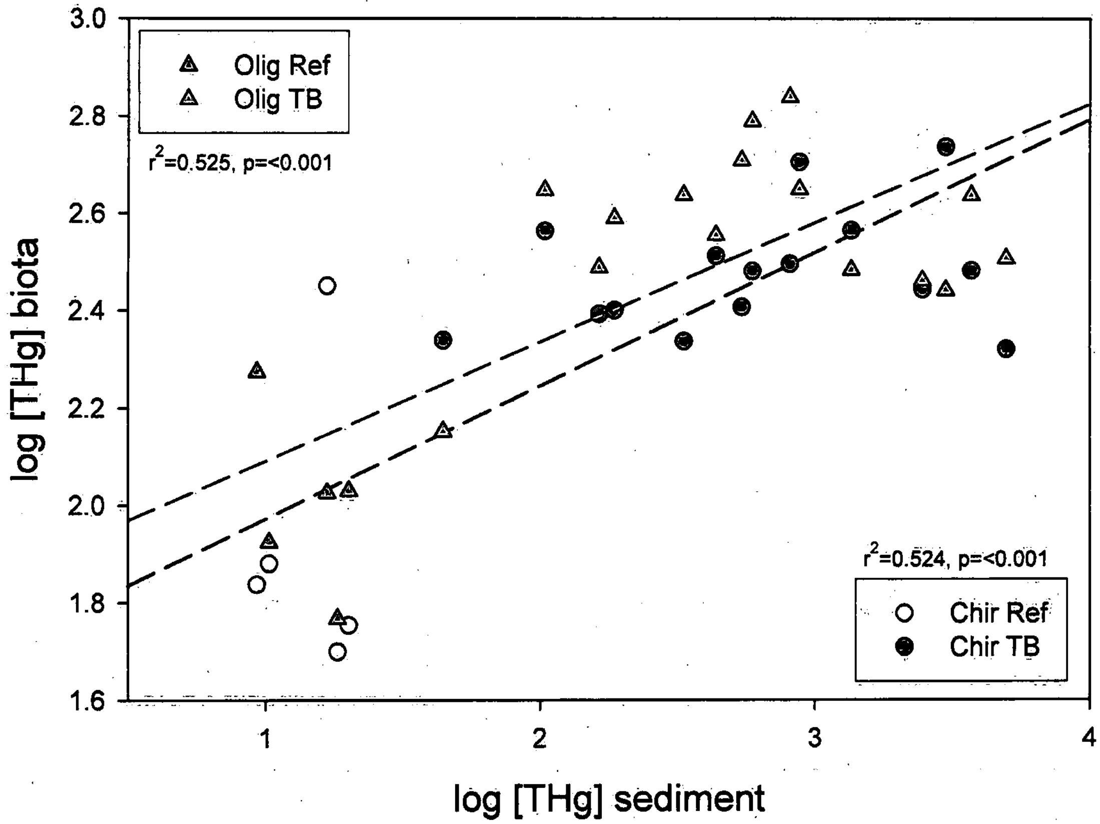


Figure 10. Relationships between total mercury in benthic invertebrates and total mercury in sediment in 2005. Separate regression lines are shown for each taxon.

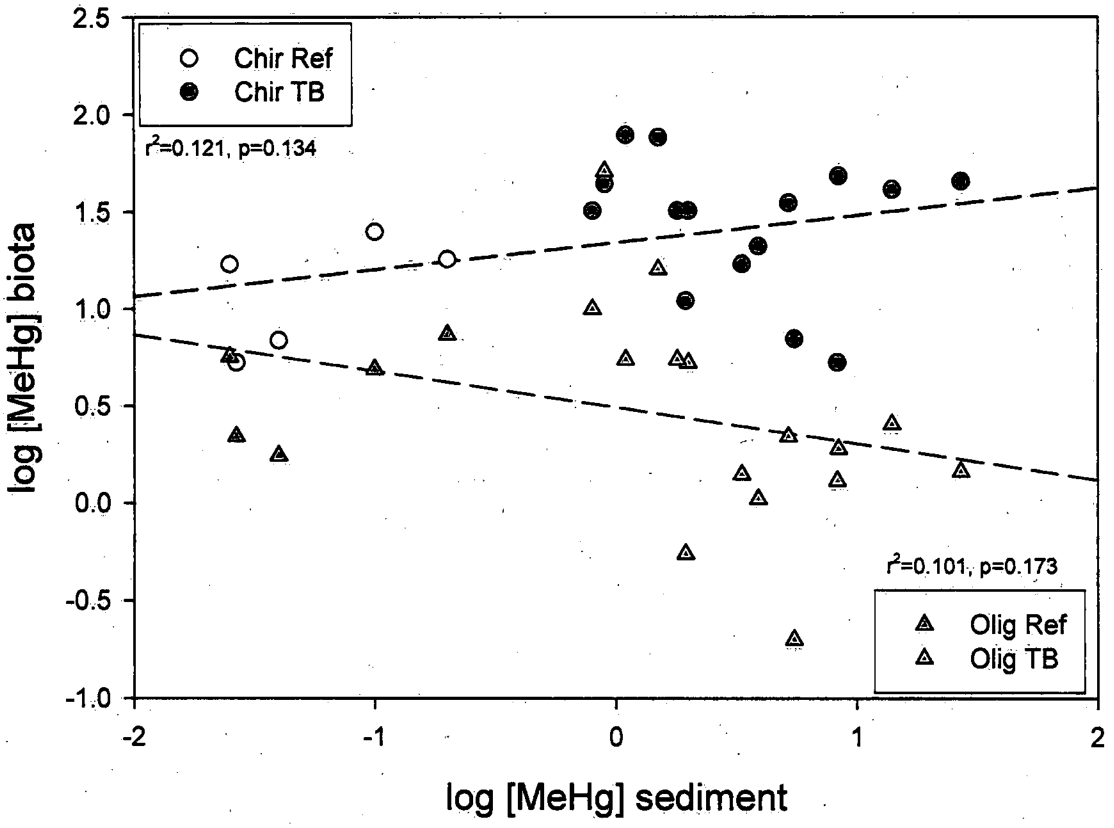


Figure 11. Relationships between methyl mercury in benthic invertebrates and methyl mercury in sediment in 2005. Separate regression lines are shown for each taxon.

Table 1. Thunder Bay 2005 site positions (NAD83), site depth, and sediment description.

Site	Site Depth (m)	Northing	Easting	Visual Description (on site)
S05-01	2.3	5369070.6	339800.8	silty mud with paper fibres/organics present/wood chips; sulphur odour
S05-02	4.2	5368794.6	339463.2	brown silt over grey mud; wood debris
S05-03	3.1	5369103.5	339187.5	soft brown mud; plants
S05-04	4.3	5368941.9	339149.1	light brown over grey mud (clay); oil came up with ponars
S05-05[a]	4.2	5368633	339136	light brown mud over grey; some plant material
S05-06	5.7	5368348.1	339208.3	brown silt over grey mud; plants
S05-07	7.4	5368225.5	338957.8	brown mud over grey clay
S05-08	8.5	5368338.6	338769.3	light brown mud over grey clay/fine sand
S05-10	2.0	5368733.6	338576.5	sand/ gravel + small amounts of mud
S05-11	3.1	5369039.8	339867.4	white paper fibres with some brown mud; wood chips and wood debris; sulphur odour
S05-12	4.5	5368746.3	339323.6	light brown mud over grey with plant material
S05-13[a]	5.2	5368537.5	339249.1	brown silt over grey mud
S05-14	5.5	5368766.8	338953.4	sand, gravel and grey clay; oil came up with ponars
S05-15	4.3	5368453.6	338931.1	fine silt over grey mud
S05-16	4.8	5368529.3	339027.7	find brown silt over grey mud
5101[a]	1.2	5407909	445305	brown silt over clay and sand
5103[a]	17.0	5405899	444996	brown silt over grey clay
5108	23.0	5361458	382222	brown silt over clay and sand
5109	8.0	5369584	382123	light brown silt over grey mud and fine sand
51LS[a]	3.5	5406938	444499	brown silt over grey clay/sand; plant material

[a] no corrections received (Broadcast GPS); therefore, positions are in Datum WGS84

Table 2. Environmental variables measured at each site.

Field	Overlying Water	Sediment (top 10 cm)	Resident Biota
Northing	Alkalinity	Total and Methyl Mercury	Total Mercury
Easting	Conductivity	Trace Metals / Major Oxides	Methyl Mercury
Site Depth	Dissolved Oxygen	Total Phosphorus	
	pH	Total Kjeldahl Nitrogen	
	Temperature	Total Organic Carbon	
	Total Kjeldahl Nitrogen	Loss on Ignition	
	Nitrates/Nitrites	% Clay, Silt, Sand and Gravel	
	Ammonia		
	Total Phosphorus		

Table 3. Characteristics of Thunder Bay and Lake Superior reference site overlying water (2005). Values in mg/L unless otherwise noted.

Site	Alkalinity	Conductivity µS/cm	Dissolved O_2	NH_3	NO_3/NO_2	pH	Temp °C	Total Kjeldahl N	Total P µg/L
S05-01	42.2	100.0	9.6	0.037	0.280	8.5	18.6	0.299	0.021
S05-02	42.7	104.1	10.2	0.038	0.275	7.6	11.6	0.221	0.018
S05-03	43.0	90.0	10.3	0.064	0.288	8.3	16.6	0.240	0.029
S05-04	43.0	90.0	10.7	0.063	0.286	8.9	16.1	0.285	0.023
S05-05	41.6	106.3	10.7	0.035	0.303	7.7	11.1	0.303	0.013
S05-06	38.2	105.9	10.2	0.027	0.250	7.9	11.0	0.309	0.015
S05-07	41.6	99.4	11.8	0.010	0.331	7.9	8.9	0.141	0.009
S05-08	43.1	80.0	11.0	0.012	0.341	7.5	8.0	0.137	0.007
S05-10	35.0	90.0	10.2	0.045	0.319	8.5	18.5	0.335	0.018
S05-11	41.4	90.0	10.1	0.021	0.264	8.6	18.1	0.188	0.013
S05-12	42.6	104.7	11.1	0.036	0.302	7.7	11.2	0.169	0.015
S05-13	41.5	105.2	10.5	0.019	0.303	7.6	10.2	0.186	0.009
S05-14	42.6	90.0	10.7	0.095	0.299	8.5	14.6	0.238	0.015
S05-15	42.7	90.0	11.5	0.025	0.327	8.5	16.8	0.154	0.007
S05-16	43.1	90.0	10.7	0.039	0.321	8.6	15.5	0.201	0.020
5101	47.4	90.0	11.6	0.011	0.300	8.6	15.0	0.121	0.005
5103	43.0	80.0	11.3	0.010	0.385	8.5	8.7	0.509	0.005
5108	42.4	108.9	12.6	0.007	0.385	7.3	5.5	0.113	0.004
5109	44.1	109.0	11.7	0.012	0.343	6.9	9.4	0.116	0.005
51LS	50.8	100.0	11.8	0.023	0.276	8.5	13.6	0.197	0.013

Table 4. Physical characteristics of Thunder Bay and Lake Superior reference 2005 sediment (top 10 cm).

Site	% Sand	% Silt	% Clay	% Gravel
S05-01	0.6	49.9	49.5	0.0
S05-02	2.3	66.7	31.0	0.0
S05-03	14.1	62.4	23.5	0.0
S05-04	23.4	52.4	24.2	0.0
S05-05	11.5	65.9	22.6	0.0
S05-06	15.4	36.8	47.8	0.0
S05-07	20.2	41.5	37.7	0.6
S05-08	2.1	53.5	44.4	0.0
S05-10	83.6	6.6	4.0	5.9
S05-11	6.5	64.4	29.2	0.0
S05-12	5.3	70.7	24.0	0.0
S05-13	7.8	66.4	25.9	0.0
S05-14	52.6	13.7	24.1	9.7
S05-15	46.7	35.0	18.0	0.3
S05-16	25.9	49.6	24.6	0.0
5101	70.7	10.6	16.2	2.6
5103	18.5	17.6	59.0	4.9
5108	58.6	30.5	11.0	0.0
5109	12.2	41.5	46.3	0.0
51LS	98.0	2.0	0.0	0.0

Table 5. Total and methyl mercury concentrations in Thunder Bay and Lake Superior reference sediment in 2005 (µg/g dry weight). Values > the Severe Effect Level (2.0 µg/g) are highlighted.

Area	Site	Total Hg (Flett) (µg/g)	Total Hg (Caduceon) (µg/g)	Methyl Hg (Flett) (ng/g)
Thunder Bay	S05-01	2.47	7.6	8.3
	S05-02	1.36	1.81	5.5
	S05-03	3.00	5.15	14.0
	S05-04	3.72	3.27	27.0
	S05-05	0.59 (0.58)[a] (0.61)[a]	0.67 (0.73)[a] (0.77)[a]	3.6 (3.0)[a] (3.4)[a]
	S05-06	0.54	0.63	1.7 (2.2)[b]
	S05-07	0.18 (0.19)[b]	0.21	1.1
	S05-08	0.16	0.19	1.6 (1.4)[c]
	S05-10	0.04	0.11	0.8
	S05-11	4.98	4.15	8.2 (8.6)[b]
	S05-12	0.88	1.01	5.2
	S05-13	0.81	0.88	3.9
	S05-14	0.10	0.12	0.9
	S05-15	0.33	0.39	2.0
	S05-16	0.44	0.40	1.9 (1.7)[b]
Reference	5101	0.010	0.018	0.1
	5103	0.017 (0.020)[a] (0.019)[b] (0.014)[a]	0.031 (0.029)[a] (0.031)[a]	0.03 (0.03)[a] (0.02)[a,d]
	5108	0.009	0.016	0.02[d] (0.03)[b]
	5109	0.020	0.022	0.04
	51LS	0.018	0.021	0.2

[a] field replicate; [b] duplicate; [c] repeat aliquot; [d] below official detection limit for this analyte in this matrix

Table 6. Sediment trace metal, nutrient and major oxide concentrations in Thunder Bay 2005 sediment (Caduceon laboratory results). Values > the Severe Effect Level (SEL) are highlighted.

Parameter	Units	LEL	SEL	S05-01	S05-02	S05-03	S05-04	S05-05-1	S05-05-2	S05-05-3	S05-06	S05-07b	S05-08	S05-10	S05-11	S05-12	S05-13	S05-14	S05-15b	S05-16
Aluminum	%			1.07	1.11	0.977	0.934	1.04	1.05	1.03	1.12	1.2	1.13	0.879	0.808	1.12	1.12	1.21	0.999	1.03
Antimony	µg/g			< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
Arsenic	µg/g	6	33	< 5	< 5	< 5	< 5	< 5	< 5	< 5	6	< 5	< 5	< 5	< 5	< 5	< 5	7	< 5	< 5
Barium	µg/g			39.8	70.8	62.9	52	71.2	71.7	70.2	80.8	102	92.6	56.8	37.1	74.4	75.7	89.8	68	71.3
Beryllium	µg/g			0.2	0.3	0.2	0.3	0.3	0.3	0.3	0.4	0.5	0.4	0.3	< 0.2	0.3	0.3	0.5	0.3	0.3
Bismuth	µg/g			< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
Boron	µg/g			3.8	8.6	7	7.3	9.9	10.1	9.6	10.5	13.6	11.5	10.6	3.8	10.1	10.6	15.2	10.3	10.4
Cadmium	µg/g	0.6	10	1.2	1.1	1.1	0.8	0.6	0.6	0.7	0.7	< 0.5	< 0.5	< 0.5	1	0.8	0.8	< 0.5	< 0.5	< 0.5
Calcium	%			3.55	6.73	5.25	5.88	7.89	8.1	8.11	9.54	15	20.9	4.22	3.09	7.64	9.39	12.9	7.33	7.55
Chromium	µg/g	26	110	39.9	47.6	56.9	40.6	52.9	46.9	46.9	50.2	64.6	47.6	49.3	29.1	50	46.8	49.2	45.5	45.1
Cobalt	µg/g			5	11	12	10	12	12	12	12	15	13	13	4	12	12	15	12	13
Copper	µg/g	16	110	87.1	62.6	87	56.5	48.1	50.9	51	55.1	41.6	43.7	33.9	53.5	58.7	54.9	39.1	45.7	47.3
Iron	%	2	4	0.962	2.02	1.7	1.86	2.39	2.41	2.35	2.45	2.81	2.51	2.78	0.918	2.34	2.45	3.44	2.39	2.43
Lead	µg/g	31	250	37.0	30	89	46	29	33	29	26	25	14	19	22	30	33	15	28	29
Magnesium	%			3.92	6.77	5	6.2	8.04	8.28	8.24	9.33	13.8	16.5	6.33	3.21	7.81	9.08	13.6	8.08	7.84
Manganese	µg/g	460	1100	104	215	189	203	298	295	290	294	442	373	326	82.9	274	321	531	295	297
Molybdenum	µg/g			2	< 1	1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	1	< 1	< 1	< 1	< 1	< 1
Nickel	µg/g	16	75	19	26	31	24	28	28	28	29	33	31	31	15	28	28	33	28	29
Phosphorus	µg/g			800	890	820	610	650	670	650	780	570	560	470	660	790	740	510	530	590
Potassium	%			1.03	1.51	1.18	1.15	1.52	1.56	1.53	1.73	2.98	2.34	1.08	0.85	1.6	1.64	2.39	1.56	1.51
Silicon	µg/g			903	1220	1160	1000	1430	1440	1360	1350	1730	1900	1360	970	1260	1340	1890	1350	1340
Silver	µg/g			< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Sodium	%			0.88	1.22	1.23	1.47	1.22	1.42	1.23	1.49	1.18	1.4	0.96	0.8	1.29	1.52	1.07	1.24	1.31
Strontium	µg/g			11.5	19.1	18.7	16.3	19.3	19.6	19.1	20.9	24.3	25.3	14.1	9.8	20.2	20.2	21.1	18.1	19.5
Tin	µg/g			< 10	< 10	20	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Titanium	µg/g			391	690	634	841	872	905	870	837	1200	1010	814	365	866	897	926	975	971
Vanadium	µg/g			36	63.9	59.6	77.6	82.3	85	82.2	80.2	80.5	74.5	76.9	28.3	79.1	81.3	64.5	83.2	85.6
Zinc	µg/g	120	820	241	178	167	153	124	133	132	143	95.5	89.2	90	163	149	140	77.5	98.5	115
Zirconium	µg/g			1	2	2	3	3	3	3	3	13	10	4	< 1	2	3	13	5	4
Aluminum (Al_2O_3)	%			17.6	13.4	13.5	13.7	12.3	11	10.7	11.2	10.8	12.5	11.3	14.3	11	10.7	12.2	10	12.1
Barium (BaO)	%			0.023	0.037	0.039	0.039	0.041	0.036	0.034	0.037	0.040	0.046	0.043	0.021	0.034	0.035	0.049	0.036	0.042
Calcium (CaO)	%			1.68	3.34	3.77	4.1	4.25	3.73	3.62	3.6	3.93	5.64	3.75	1.51	3.24	3.57	3.97	3.5	4.33
Chromium (Cr_2O_3)	%			0.01	0.01	0.02	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.02	0.01	0.01	0.01	0.02	0.01	0.02
Iron (Fe_2O_3)	%			2.73	5.25	5.55	5.9	6.85	5.91	5.71	5.57	6.01	6.56	7.78	2.78	5.22	5.38	8.22	5.9	6.93
Magnesium (MgO)	%			1.75	2.72	2.6	3.13	3.28	2.86	2.79	2.78	3.24	4.41	3.15	1.74	2.5	2.7	3.87	2.83	3.32
Manganese (MnO)	%			0.03	0.06	0.07	0.08	0.09	1.05	1.02	0.95	1.16	0.09	0.1	0.38	0.89	0.99	0.11	1.09	0.09
Phosphorus (P_2O_5)	%			0.21	0.22	0.2	0.16	0.16	1.98	1.9	2.18	1.69	0.14	0.1	3	2.32	2.07	0.13	1.55	0.14
Potasium (K_20)	%			1.01	1.57	1.55	1.67	1.77	3.24	3.13	3.28	3.74	2.11	1.94	1.91	3.05	3.13	2.3	3.28	1.8
Silica (SiO_2)	%			38.1	50.9	52.9	58.8	58.3	53.7	52.4	52.2	50.9	56.7	65.9	36.7	50.2	50.4	60.2	53.1	60
Sodium (Na_2O)	%			1.07	1.52	1.96	2.04	2.13	5.4	5.18	5.25	5.02	2.09	2.24	2.44	4.95	5.05	2.03	5.22	2.15
Titanium (TiO_2)	%			1.4	0.98	1.05	0.97	0.85	2.12	2.07	2.04	1.73	0.73	0.71	3.31	2.11	2	0.55	2	0.81
Whole Rock Total	%			99.9	98.5	100	101	98.5	99.9	97.4	99.5	97.4	99	101	98.3	96.9	96.3	99.6	96.6	99.3
Loss on Ignition	%			34.3	18.4	17.1	9.98	8.51	8.8	8.75	10.4	9.08	7.98	4.11	30.2	11.4	10.3	5.92	8.03	7.58
Total Phosphorus	µg/g	600	2000	713	1010	871	694	624	657	665	770	607	564	503	1130	945	768	486	655	629
Total Nitrogen	µg/g	550	4800	4550	2780	2880	1570	1430	1560	1470	1920	930	649	591	4320	2320	1810	383	1290	1130
Total Organic Carbon	% by wt	1	10	13.6	7.8	7.2	3.9	2.9	2.7	2.9	1.9	1	0.7	0.7	8.5	4	3	0.8	2	3

Table 7. Mean percent survival, growth (mg dry wt) and reproduction in Thunder Bay 2005 sediments and BEAST difference-from-reference band. Toxicity (based on numerical guidelines) is highlighted red and potential toxicity is highlighted yellow.

Site	*C. riparius* %Survival	*C. riparius* Growth	*H. azteca* %Survival	*H. azteca* Growth	*Hexagenia* %Survival	*Hexagenia* Growth	*T. tubifex* %Survival	*T. tubifex* No. Cocoons/adult	*T. tubifex* %Hatch	*T. tubifex* No. Young/adult	BEAST BAND
GL reference mean	87.1	0.35	85.6	0.50	96.2	3.03	97.9	9.9	57.0	29.0	-
S05-01	49.33	0.419	26.67	0.141	100	5.76	100	10.7	60.9	39.4	**Severely toxic**
S05-02	98.67	0.377	88.33	0.296	98	4.30	100	10.5	60.1	30.9	Non-toxic
S05-03	96.00	0.339	88.00	0.338	96	5.18	100	9.7	60.3	31.3	Non-toxic
S05-04	94.67	0.371	70.67	0.418	100	3.24	100	10.1	62.9	32.1	Non-toxic
S05-05	91.99	0.309	81.67	0.369	100	3.18	100	10.2	60.5	27.8	Non-toxic
S05-06	92.00	0.397	90.00	0.384	100	4.32	100	10.7	62.9	32.6	Non-toxic
S05-07	89.33	0.335	76.00	0.492	98	3.72	100	10.1	67.8	27.2	Non-toxic
S05-08	92.00	0.321	93.33	0.437	100	2.94	100	9.2	67.4	24.3	Non-toxic
S05-10	78.67	0.490	91.11	0.542	98	5.48	100	11.8	62.1	25.9	Non-toxic
S05-11	13.33	0.424	90.67	0.294	98	2.87	100	9.1	53.6	21.6	**Severely toxic**
S05-12	78.33	0.428	86.67	0.537	100	4.60	100	10.5	57.8	20.9	Non-toxic
S05-13	86.67	0.529	***62.67***	0.473	100	4.78	100	10.6	58.1	18.7	***Potentially toxic***
S05-14	96.00	0.434	80.00	0.426	100	4.18	100	8.9	67.2	17.4	Non-toxic
S05-15	89.33	0.426	85.00	0.304	100	4.62	100	10.1	62.8	19.4	Non-toxic
S05-16	93.33	0.409	48.00	0.278	100	4.10	100	9.0	65.6	17.7	**Toxic**
Non-toxic	≥67.7	0.49 – 0.21	≥67.0	0.75 – 0.23	≥85.5	5.0 – 0.9	≥88.9	12.4 – 7.2	78.1 – 38.1	46.3 – 9.9	-
Pot. toxic	67.6 – 58.8	0.20 – 0.14	66.9 – 57.1	0.22 – 0.10	85.4 – 80.3	0.8 – 0	88.8 – 84.2	7.1 – 5.9	38.0 – 28.1	9.8 – 0.8	-
Toxic	**< 58.8**	**< 0.14**	**< 57.1**	**< 0.10**	**< 80.3**	**negative**	**< 84.2**	**< 5.9**	**< 28.1**	**< 0.8**	-

Note: The upper limit for non-toxic category is set using 2 × SD of the mean and indicates excessive growth or reproduction.

Table 8. Total mercury concentrations in resident benthic invertebrates in 2005 (ng/g dry weight – recovery corrected) (Flett laboratory results). Wet weight values were converted from dry weight values (see text).

		Chironomid		**Oligochaete**	
Area	**Site**	**ng/g dry wt**	**ng/g wet wt**	**ng/g dry wt**	**ng/g wet wt**
Thunder Bay	S05-01	277	58.80	289	39.26
	S05-02	367 (366)[a]	48.90	304	42.31
	S05-03	545	70.91	276	26.85
	S05-04	303	39.26	433	66.38
	S05-05	302	47.17	615	95.83
	S05-06	255	42.54	511	88.22
	S05-07	251	39.96	389	61.27
	S05-08	247	33.12	308	51.36
	S05-10	218	29.31	142	23.24
	S05-11	209	32.78	321	48.93
	S05-12	507	71.94	446	72.19
	S05-13	313	48.65	691	100.41
	S05-14	366	64.82	444	66.14
	S05-15	217	34.70	434	68.18
	S05-16	325	51.01	362 (355)[a]	56.86
Reference	5101	75.9	16.08	84.0	15.76
	5103	282	52.26	106	15.64
	5108	68.8	9.36	188	32.71
	5109	56.7	9.98	107	15.24
	51LS	50.0	8.46	58.5	9.49

[a] duplicate

Table 9. Methyl mercury concentrations in resident benthic invertebrates in 2005 (ng/g dry weight – recovery corrected) (Flett laboratory results). Wet weights were converted from dry weights (see text).

		Chironomid		**Oligochaete**	
Area	**Site**	**ng/g dry wt**	**ng/g wet wt**	**ng/g dry wt**	**ng/g wet wt**
Thunder Bay	S05-01	5.3	1.13	1.3	0.18
	S05-02	7.0	0.93	0.2	0.03
	S05-03	41	5.33	2.8 (2.3)[b]	0.25
	S05-04	45	5.83	1.2 (1.7)[b]	0.22
	S05-05	17	2.66	1.4	0.22
	S05-06	11	1.84	0.5 (0.6)[b]	0.09
	S05-07	78	12.42	5.4 (5.6)[b]	0.87
	S05-08	76	10.19	16	2.67
	S05-10	32	4.30	10	1.64
	S05-11	48	7.53	1.9	0.29
	S05-12	38 (32)[a]	4.97	2.2	0.36
	S05-13	21	3.26	1.0 (1.1)[b]	0.15
	S05-14	44	7.79	51	7.60
	S05-15	35 (29)[a]	5.12	5.3	0.83
	S05-16	32	5.02	5.5	0.87
Reference	5101	25	5.30	4.9	0.92
	5103	5.3	0.98	2.2	0.32
	5108	17	2.31	5.7	0.99
	5109	6.9	1.22	2.0 (1.5)[b]	0.25
	51LS	16 (20)[a]	3.05	7.4	1.20

[a] duplicate [b] repeat aliquot

Table 10. Prediction of whole body concentrations of total and methyl mercury in two invertebrate taxa based on sediment mercury concentration alone ("A" models), and sediment mercury concentration + other physico-chemical variables ("B" models) from 2005. The groups of multiple predictors listed are from the models that best predicted $[\mathrm{Hg}]_{\mathrm{inv}}$ using sediment and water variables. $[\mathrm{Hg}]_{\mathrm{sed}}$ was retained in all models. All variables in the models shown were transformed: arcsine-square root (x) for the particle size variables; log(x) for the others.

Response ($[\mathrm{Hg}]_{\mathrm{inv}}$)	**Model**	**Predictor ([X])**	**Coefficient**	**P (predictor)**	**R^2**	**R^2_{adj}**	**P (regression)**
Total Hg Chironomid	A	Total Hg	0.2445	< 0.001	0.524	0.498	< 0.001
	B	Total Hg	0.3461	< 0.001	0.855	0.816	< 0.001
		Manganese	0.7426	< 0.001			
		Dissolved O_2	-0.1463	0.025			
		Clay	-0.4914	0.021			
Total Hg Oligochaete	A	Total Hg	0.2443	< 0.001	0.525	0.499	< 0.001
	B	Total Hg	0.3367	< 0.001	0.784	0.759	< 0.001
		Iron	1.1826	< 0.001			
Methyl Hg Chironomid	A	Methyl Hg	0.1393	0.136	0.121	0.072	0.134
	B	Methyl Hg	0.4883	< 0.001	0.844	0.772	< 0.001
		Total Organic C	-0.4517	0.013			
		Total Kjeldahl N	-0.8717	0.015			
		Conductivity	-3.5010	0.007			
		Alkalinity	-3.6010	0.032			
		Dissolved O_2	0.2599	0.033			
Methyl Hg Oligochaete	A	Methyl Hg	-0.1881	0.173	0.101	0.051	0.173
	B	Methyl Hg	0.3208	< 0.001	0.597	0.550	< 0.001
		Total Nitrogen	-1.7648	0.039			

Table 11a. Predicted methyl mercury concentrations (ng/g wet weight) in fish receptor species for Thunder Bay sites and Lake Superior reference sites for 2005. Bolded values exceed the 99th percentile for the reference sites. Yellow highlighted values exceed Environment Canada (2002) tissue residue guideline (92 ng/g ww) applicable for fishes. Bolded and highlighted values exceed both the TRG and the 99th percentile for the reference sites.

Receptor		White sucker			Yellow perch			Walleye		
Reference 99th percentile		4.09	10.53	17.87	20.44	52.66	89.36	4.58	80.05	578.83
Area	Site	min	med	max	min	med	max	min	med	max
Reference	5101	3.16	10.67	18.18	15.78	53.34	90.90	3.53	81.08	588.83
	5103	1.10	2.23	3.36	5.49	11.15	16.81	1.23	16.95	108.88
	5108	3.40	5.66	7.92	16.98	28.30	39.62	3.80	43.02	256.64
	5109	0.86	2.52	4.18	4.29	12.61	20.92	0.96	19.16	135.54
	51LS	4.12	7.29	10.46	20.58	36.44	52.31	4.61	55.40	338.86
Thunder Bay	S05-01	0.62	2.25	3.88	3.09	11.23	19.38	0.69	17.08	125.54
	S05-02	0.10	1.65	3.19	0.51	8.23	15.95	0.12	12.51	103.32
	S05-03	0.86	9.57	**18.28**	4.29	47.85	**91.41**	0.96	72.74	**592.16**
	S05-04	0.75	10.38	**20.00**	3.77	51.88	**99.98**	0.84	78.86	**647.71**
	S05-05	0.75	4.94	9.12	3.77	24.70	45.62	0.84	37.54	295.53
	S05-06	0.31	3.31	6.31	1.54	16.55	31.56	0.35	25.16	204.42
	S05-07	2.98	**22.79**	**42.60**	14.92	**113.96**	**213.00**	3.34	**173.24**	**1379.86**
	S05-08	**9.16**	**22.05**	**34.95**	**45.79**	**110.27**	**174.76**	**10.25**	**167.63**	**1132.11**
	S05-10	**5.63**	10.19	14.75	**28.13**	50.94	73.75	**6.30**	77.43	477.73
	S05-11	0.99	**13.41**	**25.83**	4.97	**67.06**	**129.14**	1.11	**101.93**	**836.58**
	S05-12	1.23	9.14	17.05	6.17	45.70	85.24	1.38	69.48	552.17
	S05-13	0.51	5.85	11.18	2.57	29.24	55.91	0.58	44.45	362.19
	S05-14	**26.07**	**26.39**	**26.72**	**130.34**	**131.97**	**133.60**	**29.18**	**200.61**	**865.47**
	S05-15	2.85	10.20	17.56	14.23	51.02	87.81	3.19	77.56	568.83
	S05-16	2.98	10.10	17.22	14.92	50.51	86.09	3.34	76.78	557.72

Table 11b. Predicted methyl mercury concentrations (ng/g wet weight) in bird and mammal receptor species for Thunder Bay and Lake Superior reference sites for 2005. Bolded values exceed the 99th percentile for the reference sites.

Receptor		Great Blue heron			Mink		
Reference 99th percentile		**17.38**	**98.23**	**607.49**	**6.95**	**151.17**	**893.52**
Area	Site	min	med	max	min	med	max
Reference	5101	13.41	99.49	617.98	5.36	153.11	908.95
	5103	4.67	20.79	114.27	1.87	32.00	168.07
	5108	14.43	52.78	269.35	5.77	81.23	396.17
	5109	3.65	23.51	142.25	1.46	36.18	209.23
	51LS	17.50	67.98	355.63	7.00	104.61	523.08
Thunder Bay	S05-01	2.62	20.95	131.76	1.05	32.25	193.80
	S05-02	0.44	15.36	108.44	0.17	23.63	159.50
	S05-03	3.65	89.25	**621.48**	1.46	137.35	**914.10**
	S05-04	3.21	96.77	**679.78**	1.28	148.92	**999.85**
	S05-05	3.21	46.07	310.16	1.28	70.89	456.19
	S05-06	1.31	30.87	214.54	0.52	47.51	315.56
	S05-07	12.68	**212.57**	**1448.17**	5.07	**327.13**	**2130.03**
	S05-08	**38.93**	**205.70**	**1188.15**	**15.57**	**316.55**	**1747.59**
	S05-10	**23.91**	95.01	501.38	**9.56**	146.21	737.45
	S05-11	4.23	**125.08**	**878.00**	1.69	**192.49**	**1291.40**
	S05-12	5.25	85.25	579.50	2.10	131.20	852.36
	S05-13	2.19	54.54	380.12	0.87	83.94	559.09
	S05-14	**110.81**	**246.16**	**908.31**	**44.31**	**378.82**	**1335.99**
	S05-15	12.10	95.17	596.99	4.84	146.46	878.08
	S05-16	12.68	94.21	585.33	5.07	144.98	860.93

Table 12. Decision matrix for weight of evidence categorization of Thunder Bay sites (2002 and 2005 data). The assessment for 2005 sites is based on 3 lines of evidence. For the sediment chemistry column, sites with exceedences of the Severe Effect Level (SEL) are indicated by "●", and sites with exceedences of the Lowest Effect Level (LEL) by "◉". For the toxicity and benthos alteration columns, sites determined from BEAST analyses as toxic/severely toxic or different/very different from reference, respectively, are indicated by "●"; and sites determined as potentially toxic or possibly different from reference by "◉". Sites with no SQG exceedences, no sediment toxicity, or benthic communities equivalent to reference conditions are indicated by "○". Substances exceeding SELs are listed.

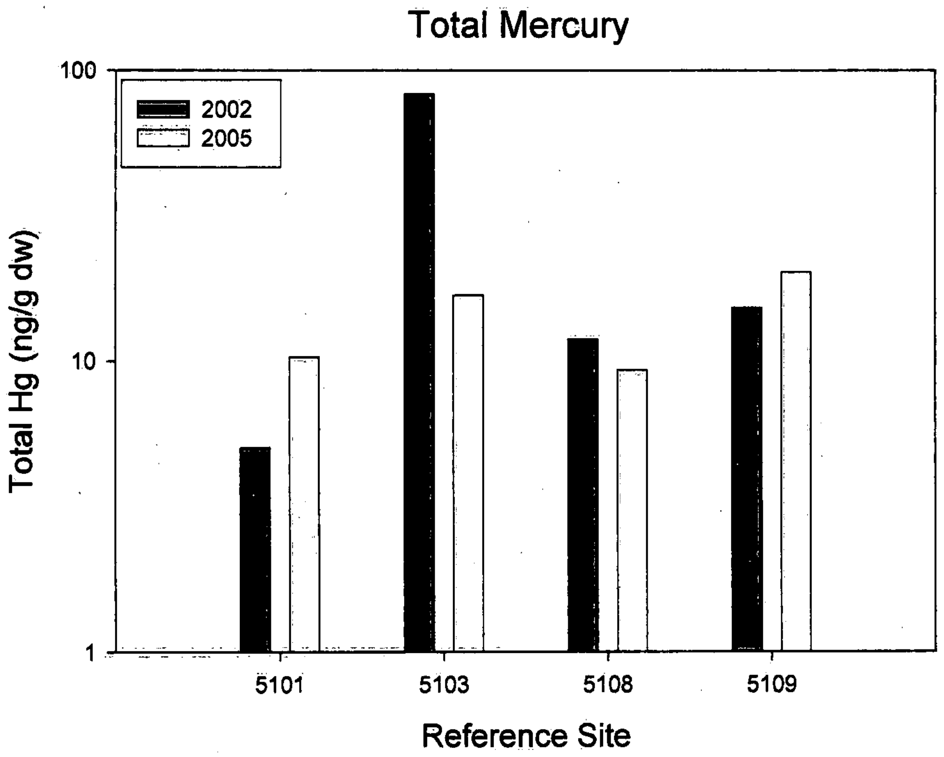
Site	Year	Sediment Chemistry	Toxicity	Benthos Alteration[a]	Biomagnification potential[b,c]	> SEL	Assessment
P1	2002	●	●	●	○	Hg, TKN, TOC, TP	Management actions required
P2	2002	●	○	●	○	Hg, TKN	Determine reasons for benthos alteration[d]
P3	2002	●	●	○	○	Hg, TKN, TOC	Determine reasons for sediment toxicity
P4	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P5	2002	●	○	●	○	Hg	Determine reasons for benthos alteration[d]
P6	2002	●	●	●	◉	Hg, TOC	Management actions required
P7	2002	●	●	○	◉	Hg, Cu, TOC	Determine reasons for sediment toxicity and fully assess risk of biomagnification
P8	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P9	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P10	2002	●	○	●	○	Hg	Determine reasons for benthos alteration[d]
P11	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P12	2002	◉	●	●	◉		Management actions required
P13	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P16	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P17	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P18	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
P22	2002	○	○	◉	◉		Determine reasons for benthos alteration[d] and fully assess risk of biomagnification
P23	2002	○	○	●	◉		Determine reasons for benthos alteration and fully assess risk of biomagnification
IB2	2002	◉	○	●	○		Determine reasons for benthos alteration[d]
S05-01	2005	●	●	-	○	Hg, TOC	Determine reasons for sediment toxicity
S05-02	2005	◉	○	-	○		No further actions needed
S05-03	2005	●	○	-	○	Hg	No further actions needed
S05-04	2005	●	○	-	○	Hg	No further actions needed
S05-05	2005	◉	○	-	○		No further actions needed
S05-06	2005	◉	○	-	○		No further actions needed
S05-07	2005	◉	○	-	◉		Fully assess risk of biomagnification
S05-08	2005	○	○	-	◉		Fully assess risk of biomagnification
S05-10	2005	○	○	-	○		No further actions needed
S05-11	2005	●	●	-	◉	Hg	Determine reasons for sediment toxicity and fully assess risk of biomagnification
S05-12	2005	◉	○	-	○		No further actions needed
S05-13	2005	◉	◉	-	○		Determine reasons for sediment toxicity
S05-14	2005	○	○	-	◉		Fully assess risk of biomagnification
S05-15	2005	◉	○	-	○		No further actions needed
S05-16	2005	◉	●	-	○		Determine reasons for sediment toxicity

[a] benthic community structure assessment performed in 2002 only; [b] based on step 3 in Chapman and Anderson (2005); [c] based on the intermediate exposure and uptake scenario

[d] benthos alteration may be due to other factors, either natural (e.g., competition/predation, habitat differences) or human-related (e.g., water column contamination) (Chapman and Anderson 2005)

TKN=total Kjeldahl nitrogen, TOC=total organic carbon, TP=total phosphorus

APPENDIX A: Comparison of 2002 and 2005 Reference Site Mercury Levels



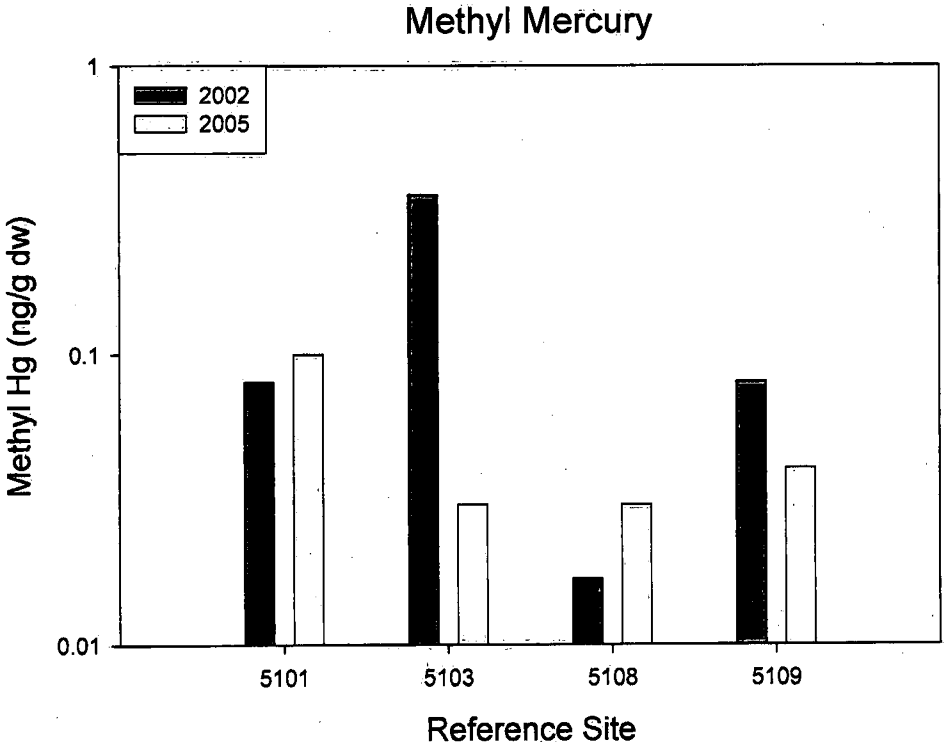


Figure A1. Comparison of total and methyl Hg concentrations from reference sites collected in 2002 and 2005.

Table A1. Sediment trace metal, nutrient and major oxide concentrations in Lake Superior reference sediment, 2005 (Caduceon laboratory results).

Parameter	**Units**	**LEL**	**SEL**	**5101**	**5103-1**	**5103-2**	**5103-3**	**5108**	**5109**	**51LS**
Aluminum	%			0.772	1.05	1.01	0.998	0.876	0.977	1.03
Antimony	µg/g			< 5	< 5	< 5	< 5	< 5	< 5	< 5
Arsenic	µg/g	6	33	< 5	11	12	8	< 5	< 5	< 5
Barium	µg/g			49.7	423	425	317	62.4	58.4	86.5
Beryllium	µg/g			0.2	0.5	0.5	0.4	0.3	0.3	0.5
Bismuth	µg/g			< 5	< 5	< 5	< 5	< 5	< 5	< 5
Boron	µg/g			7.3	15.9	15.6	13.8	11.4	9.6	12.8
Cadmium	µg/g	0.6	10	< 0.5	0.5	0.6	< 0.5	< 0.5	< 0.5	< 0.5
Calcium	%			18.9	78.7	75.5	75.1	13.8	15.7	69.8
Chromium	µg/g	26	110	38.7	42.3	39.3	40.6	53	32.4	43.6
Cobalt	µg/g			8	18	18	15	16	10	10
Copper	µg/g	16	110	20.1	29.7	26.8	24.7	32.4	27.3	22.9
Iron	%	2	4	1.56	2.98	3.06	2.67	2.84	2.01	2.03
Lead	µg/g	31	250	5	14	12	10	11	8	8
Magnesium	%			9.52	19	18.6	18	10.5	14.1	17.9
Manganese	µg/g	460	1100	252	4030	3900	2850	711	394	429
Mercury	µg/g	0.2	2	0.018	0.031	0.029	0.031	0.016	0.022	0.021
Molybdenum	µg/g			< 1	< 1	< 1	< 1	< 1	< 1	< 1
Nickel	µg/g	16	75	18	31	29	26	50	24	23
Phosphorus	µg/g			320	570	580	530	630	580	400
Potassium	%			1.52	3.06	2.74	2.69	1.15	1.57	3.28
Silicon	µg/g			1790	1670	1490	1330	1190	1080	1380
Silver	µg/g			< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Sodium	%			1.04	0.84	0.9	0.72	1.15	1.33	0.79
Strontium	µg/g			21.4	60.3	56.2	54.9	22.5	21.7	50
Tin	µg/g			< 10	< 10	< 10	< 10	< 10	< 10	< 10
Titanium	µg/g			1330	1510	1300	1270	2800	1310	1240
Vanadium	µg/g			36.8	52.7	50.5	46.7	97.4	70.6	41.5
Zinc	µg/g	120	820	33.1	62.2	58.7	56.6	56.3	45.8	51.3
Zirconium	µg/g			14	16	15	15	20	10	22
Aluminum (Al_2O_3)	%			8.45	8.73	8.46	8.62	9.23	10.5	10.7
Barium (BaO)	%			0.036	0.069	0.071	0.061	0.031	0.035	0.046
Calcium (CaO)	%			4.16	9.57	9.57	9.71	5.47	4.96	10.9
Chromium (Cr_2O_3)	%			0.02	0.01	< 0.01	< 0.01	0.05	0.01	0.01
Iron (Fe_2O_3)	%			3.86	5.18	5.39	4.83	7.21	5.02	4.51
Magnesium (MgO)	%			2.59	3.03	3.02	2.98	3.9	3.7	3.58
Manganese (MnO)	%			0.05	7.01	6.89	5.2	2.01	1.18	0.07
Phosphorus (P_2O_5)	%			0.08	1.64	1.66	1.42	1.72	1.61	0.1
Potasium (K_20)	%			1.8	3.76	3.63	3.82	2.59	3.09	2.25
Silica (SiO_2)	%			71	42.6	40.5	42.2	51.4	53.3	53.5
Sodium (Na_2O)	%			1.77	4.21	4.2	4.11	6.06	6.14	1.79
Titanium (TiO_2)	%			0.48	1.28	1.19	1.19	3.27	1.89	0.46
Whole Rock Total	%			99.1	101	99.2	98.5	96.8	96.9	101
Loss on Ignition	%			4.79	14.1	14.6	14.4	3.93	5.43	12.9
Total Phosphorus	µg/g	600	2000	297	568	647	590	688	620	443
Total Nitrogen	µg/g	550	4800	419	524	519	540	456	871	618
Total Organic	% by	1	10	0.4	< 0.1	0.4	0.8	1.1	0.8	0.4

APPENDIX B: Toxicity Ordinations

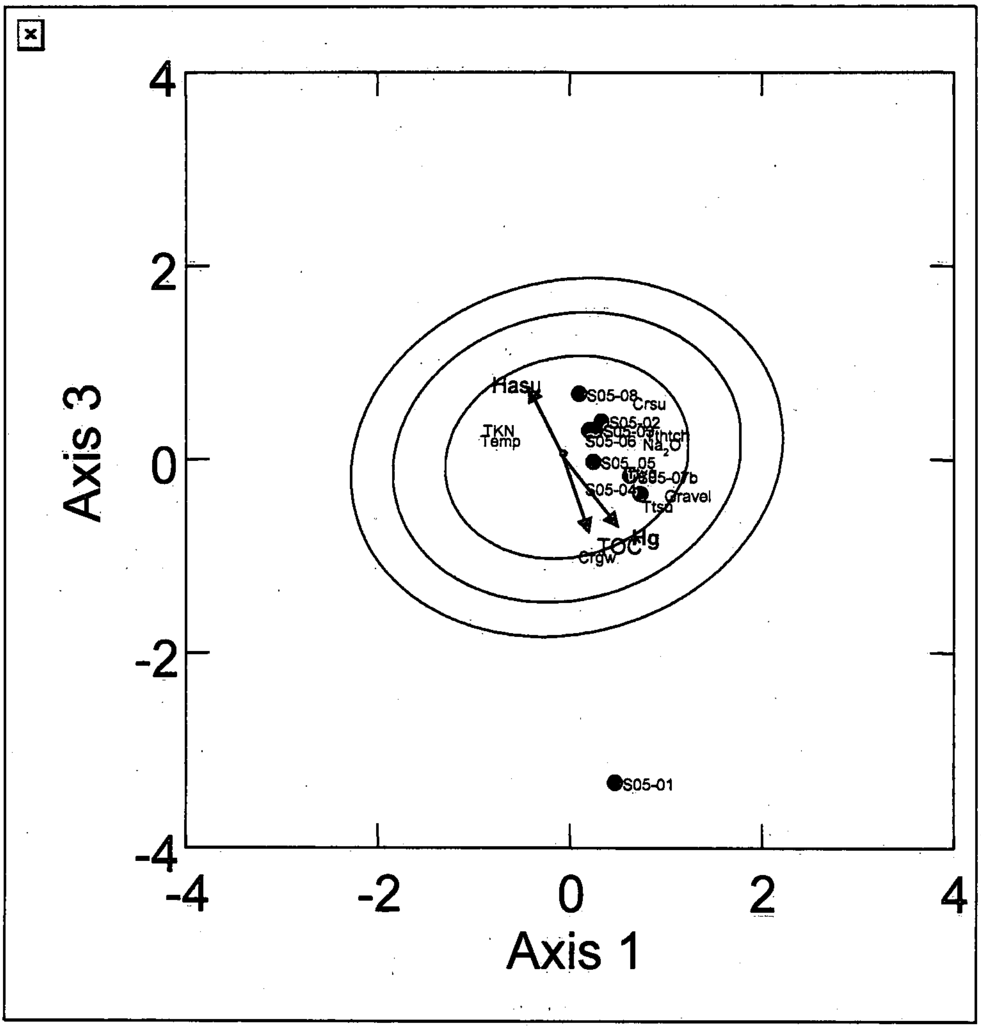


Figure B1. Assessment of a subset of test sites using 10 toxicity test endpoints, summarized on Axes 1 and 3, showing 90%, 99%, and 99.9% probability ellipses around reference sites. (Reference site scores are not shown.) Endpoints contributing significantly to the site scores, and environmental variables most correlated to the scores, are shown. Arrows indicate the most important relationships. Hasu = *Hyalella* survival; Crsu = *Chironomus* survival; Crgw = *Chironomus* growth; Ttsu = *Tubifex* survival; Ttht = *Tubifex* hatch; Ttyg = *Tubifex* young. Stress level = 0.098.

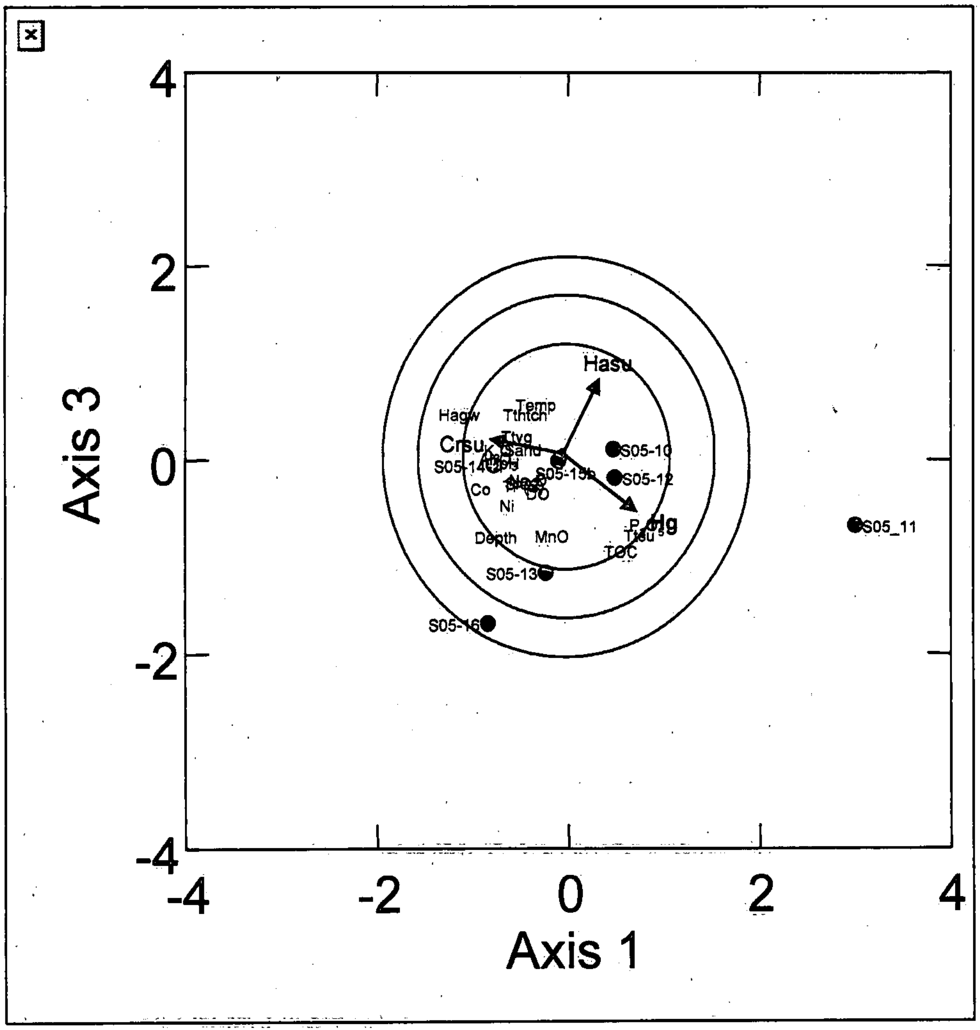


Figure B2. Assessment of a subset of test sites using 10 toxicity test endpoints, summarized on Axes 1 and 3, showing 90%, 99%, and 99.9% probability ellipses around reference sites. (Reference site scores are not shown.) Endpoints contributing significantly to the site scores, and environmental variables most correlated to the scores, are shown. Arrows indicate the most important relationships. Hasu = *Hyalella* survival; Crsu = *Chironomus* survival; Crgw = *Chironomus* growth; Ttsu = *Tubifex* survival; Ttht = *Tubifex* hatch; Ttyg = *Tubifex* young. Stress level = 0.10.

APPENDIX C: Toxicity – Contaminant Relationships

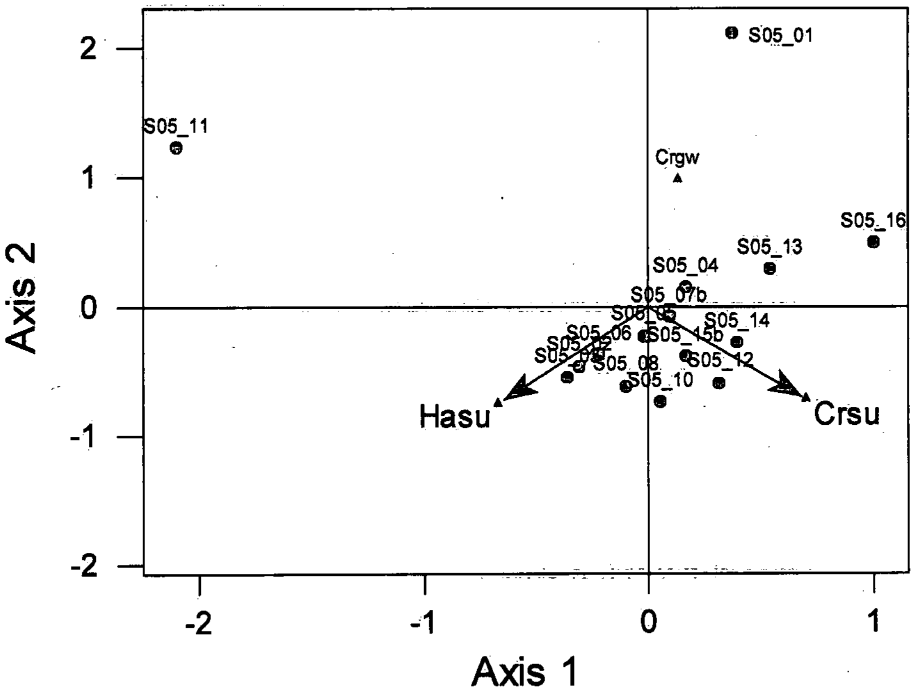


Figure C1. Toxicological response of Thunder Bay 2005 sites represented by 2-dimensional HMDS (stress = 0.06). The direction of maximum correlation of *Hyalella* and *Chironomus* survival endpoints (Hasu, Crsu) with sites are shown as vectors. High values for Axes 1 and 2 correspond to sites with high relative toxicity to *Hyalella* survival and low values for Axis 1 and high values for Axis 2 corresponds to sites with high relative toxicity to *Chironomus* survival.

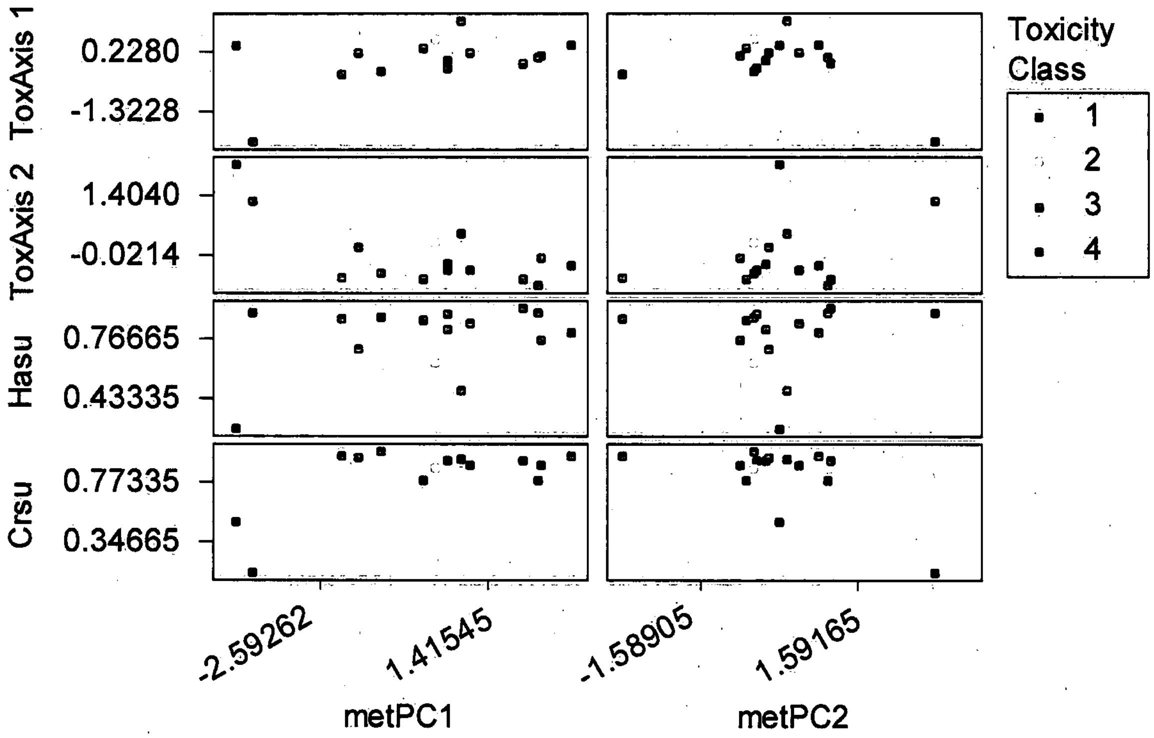


Figure C2. Thunder Bay sediment toxicity relationships to contaminant concentrations based on integrated toxicity descriptors (HMDS axes 1 and 2) and *Hyalella* and *Chironomus* survival endpoints (Hasu, Crsu) and the integrated metal descriptor (see text for derivation of variables). Sites are colour-coded by toxicity class as determined by BEAST assessment with reference sites. High values for Axes 1 and 2 correspond to sites with high relative toxicity to *Hyalella* survival and low values for Axis 1 and high values for Axis 2 corresponds to sites with high relative toxicity to *Chironomus* survival.

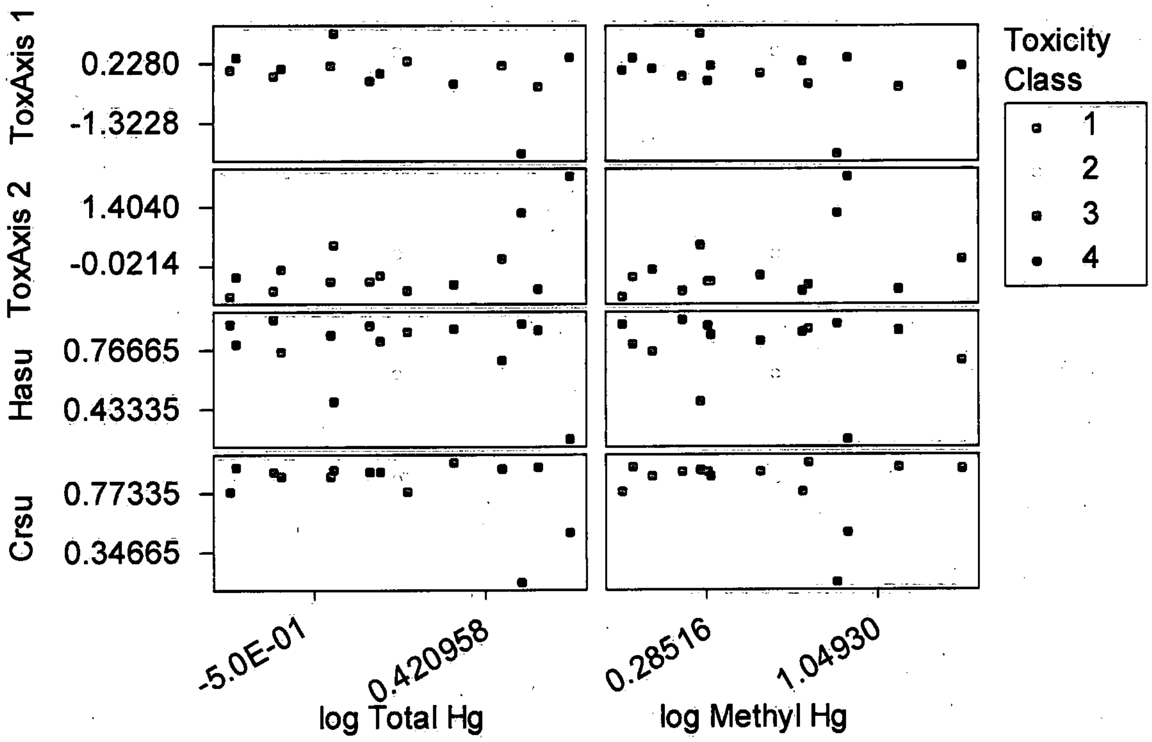


Figure C3. Thunder Bay sediment toxicity relationships to contaminant concentrations based on integrated toxicity descriptors (HMDS axes 1 and 2) and *Hyalella* and *Chironomus* survival endpoints (Hasu, Crsu) and total and methyl mercury (see text for derivation of variables). Sites are colour-coded by toxicity class as determined by BEAST assessment with reference sites.

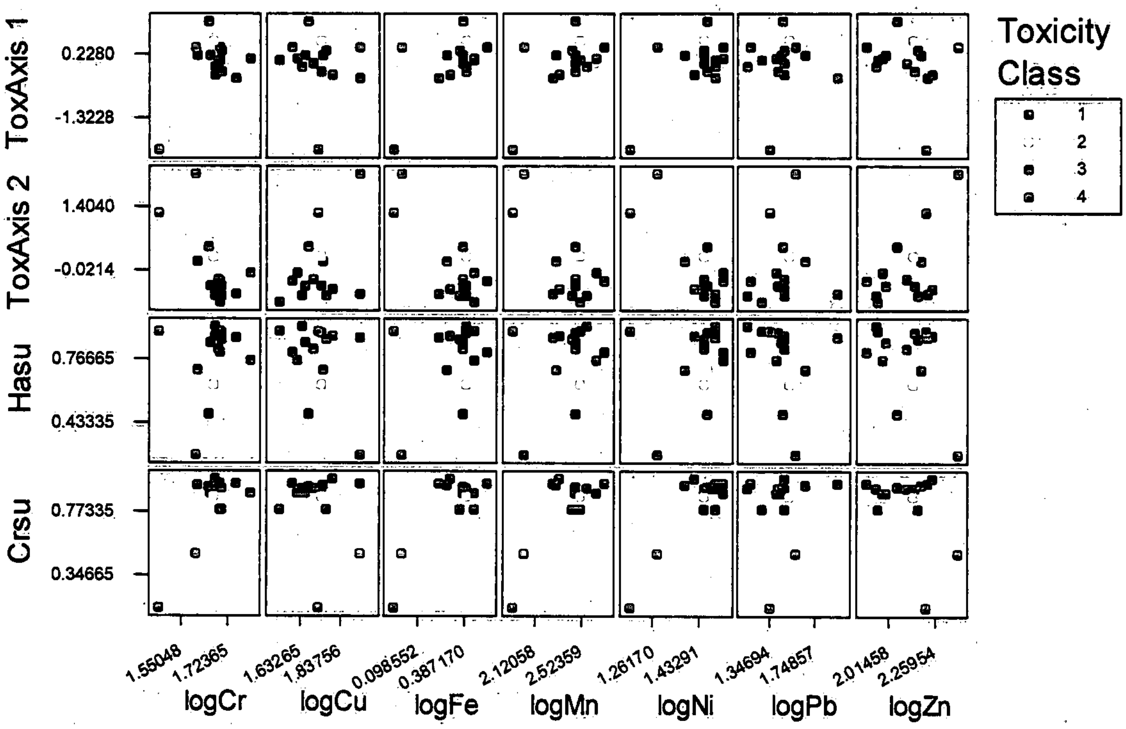


Figure C4. Thunder Bay sediment toxicity relationships to contaminant concentrations based on integrated toxicity descriptors (HMDS axes 1 and 2) and *Hyalella* and *Chironomus* survival endpoints (Hasu, Crsu) and individual metal concentrations. Sites are colour-coded by toxicity class as determined by BEAST assessment with reference sites.

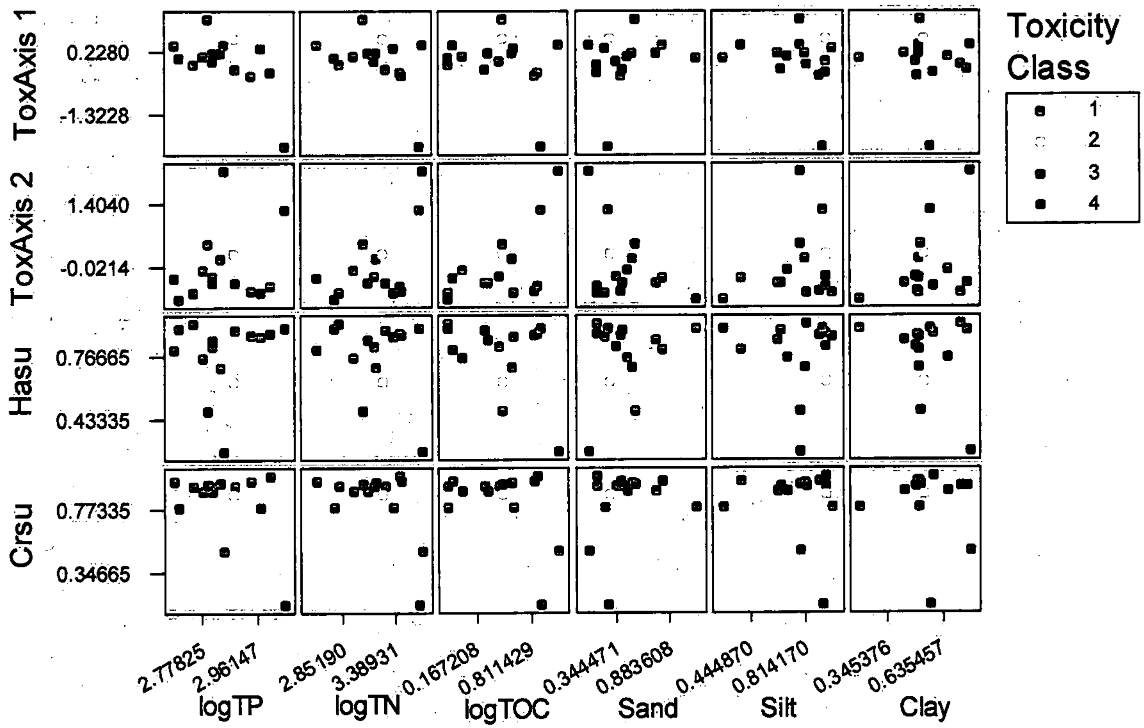


Figure C5. Thunder Bay sediment toxicity relationships to contaminant concentrations based on integrated toxicity descriptors (HMDS axes 1 and 2) and *Hyalella* and *Chironomus* survival endpoints (Hasu, Crsu) and sediment nutrient concentration and grain size. Sites are colour-coded by toxicity class as determined by BEAST assessment with reference sites.

APPENDIX D: Quality Assurance/Quality Control

Table D1. Coefficients of variation for field-replicated samples.

Parameter	5103	S05_05
Al (%)	2.7	1.0
Al2O3 (%)	1.6	7.5
Alkalinity (mg/L)	1.0	0.5
As (ppm)	20.1	-
B (ppm)	7.5	2.6
Ba (ppm)	15.9	1.1
BaO (%)	7.9	9.7
Be (ppm)	12.4	-
Ca (%)	2.6	1.5
CaO (%)	0.8	8.7
Cd (ppm)	40.1	9.1
Clay (%)	-	10.1
Co (ppm)	10.2	-
Cr (ppm)	3.7	7.1
Cr2O3 (%)	43.3	43.3
Cu (ppm)	9.3	3.3
Fe (%)	7.1	1.3
Fe2O3 (%)	5.5	9.9
Gravel (%)	-	-
Hg (ppm)	3.8	7.0
K (%)	7.1	1.4
K2O (%)	2.6	30.2
LOI (%)	1.8	1.8
Mg (%)	2.7	1.6
MgO (%)	0.9	8.9
Mn (ppm)	18.0	1.4
MnO (%)	15.9	75.8
NH3 (mg/L)	15.8	74.3
Na (%)	11.2	8.7
Na2O (%)	1.3	43.1
Ni (ppm)	8.8	-
No3No2 (mg/L)	0.3	1.0
P2O5 (%)	8.5	76.4
PSize mean (Microns)	-	17.8
PSize 25% (Microns)	-	14.9
PSize 75% (Microns)	-	16.5
Pb (ppm)	16.7	7.6
Sand (%)	-	29.8
Si (ppm)	11.4	3.1
SiO2 (%)	2.7	5.7
Silt (%)	-	4.6
Sr (ppm)	4.9	1.3
TKN (mg/L)	128.1	38.9
TN (ppm)	2.1	4.5
TOC (%)	90.1	4.1
TP(Sed) (ppm)	6.8	3.4
TP(Wat) (mg/L)	3.6	4.1
Ti (ppm)	9.6	2.2
TiO2 (%)	4.3	42.8
V (ppm)	6.1	1.9
Whole Rock (%)	1.3	1.3
Zn (ppm)	4.8	3.8
Zr (ppm)	3.8	-

Table D2. Relative percent difference (RPD) for sample duplicates (Caduceon).

Parameter	Units	M.D.L.	Reference Method	S05_01	S05_01Dup	R.P.D.	S05_07b	S05_07bDup	R.P.D.	S05_08	S05_08Dup	R.P.D.	S05_10	S05_10Dup	R.P.D.	510301	510301	R.P.D.	510302	510302Dup	R.P.D.
Aluminum	µg/g	10	EPA 6010	10700	10900	1.9	12000			11300			8790	8810	0.2	10100			9980	10200	2.2
Antimony	µg/g	5	EPA 6010	< 5	<5	0	< 5			< 5			< 5	<5	0	< 5			< 5	<5	0
Arsenic	µg/g	5	EPA 6010	< 5	<5	0	< 5			< 5			< 5	<5	0	12			8	10	22.2
Barium	µg/g	0.1	EPA 6010	39.8	40.1	0.8	102			92.6			56.8	56.5	0.5	425			317	320	0.9
Beryllium	µg/g	0.2	EPA 6010	0.2	0.2	0	0.5			0.4			0.3	0.3	0	0.5			0.4	0.5	22.2
Bismuth	µg/g	5	EPA 6010	< 5	<5	0	< 5			< 5			< 5	<5	0	< 5			< 5	<5	0
Boron	µg/g	0.5	EPA 6010	3.8	4.0	5.1	13.6			11.5			10.6	10.6	0	15.6			13.8	13.9	0.7
Cadmium	µg/g	0.5	EPA 6010	1.2	1.2	0	< 0.5			< 0.5			< 0.5	<0.5	0	0.6			< 0.5	<0.5	0
Calcium	µg/g	10	EPA 6010	3550	3600	1.4	15000			20900			4220	4250	0.7	75500			75100	76100	1.3
Chromium	µg/g	0.2	EPA 6010	39.9	40.7	2.0	64.6			47.6			49.3	49	0.6	39.3			40.6	42.1	3.6
Cobalt	µg/g	1	EPA 6010	5	5	0	15			13			13	13	0	18			15	15	0
Copper	µg/g	0.2	EPA 6010	87.1	86.3	0.9	41.6			43.7			33.9	33.1	2.4	26.8			24.7	25.5	3.2
Iron	µg/g	10	EPA 6010	9620	9700	0.8	28100			25100			27800	27500	1.1	30600			26700	27100	1.5
Lead	µg/g	2	EPA 6010	37.0	36.7	0.8	25			14			19	23	19.0	12			10	11	9.5
Magnesium	µg/g	10	EPA 6010	3920	3940	0.5	13800			16500			6330	6270	1.0	18600			18000	18300	1.7
Manganese	µg/g	0.1	EPA 6010	104	104	0	442			373			326	326	0	3900			2850	2870	0.7
Mercury	µg/g	0.005	EPA 7471A	7.6	6.1	21.9	0.207			0.193			0.11	0.10	9.5	0.029			0.031	0.026	17.5
Molybdenum	µg/g	1	EPA 6010	2	2	0	< 1			< 1			< 1	<1	0	< 1			< 1	<1	0
Nickel	µg/g	1	EPA 6010	19	19	0	33			31			31	30	3	29			26	27	3.8
Phosphorus	µg/g	10	EPA 6010	800	796	0.5	570			560			470	467	0.6	580			530	535	0.9
Potassium	µg/g	30	EPA 6010	1030	1060	2.9	2980			2340			1080	1060	1.9	2740			2690	2840	5.4
Silicon	µg/g	1	EPA 6010	903	985	8.7	1730			1900			1360	1300	4.5	1490			1330	1310	1.5
Silver	µg/g	0.5	EPA 6010	< 0.5	<0.5	0	< 0.5			< 0.5			< 0.5	<0.5	0	< 0.5			< 0.5	<0.5	0
Sodium	µg/g	20	EPA 6010	880	927	5.2	1180			1400			960	1070	10.8	900			720	895	21.7
Strontium	µg/g	0.1	EPA 6010	11.5	11.6	0.9	24.3			25.3			14.1	13.7	2.9	56.2			54.9	56.4	2.7
Tin	µg/g	10	EPA 6010	< 10	<10	0	< 10			< 10			< 10	<10	0	< 10			< 10	<10	0
Titanium	µg/g	0.5	EPA 6010	391	412	5.2	1200			1010			814	782	4.0	1300			1270	1317	3.6
Vanadium	µg/g	0.5	EPA 6010	36	36	0	80.5			74.5			76.9	76.6	0	50.5			46.7	47.7	2.1
Zinc	µg/g	0.5	EPA 6010	241	240	0.4	95.5			89.2			90	93	3.3	58.7			56.6	57.8	2.1
Zirconium	µg/g	1	EPA 6010	1	2	66.7	13			10			4	4	0	15			15	14	6.9
Aluminum (Al2O3)	%	0.01	IN-HOUSE	17.6			10.8	10.7	0.9	12.5			11.3			8.46	8.57	1.3	8.62		
Barium (BaO)	%	0.001	IN-HOUSE	0.023			0.040	0.039	2.5	0.046			0.043			0.071	0.072	1.4	0.061		
Calcium (CaO)	%	0.01	IN-HOUSE	1.68			3.93	3.85	2.1	5.64			3.75			9.57	9.65	0.8	9.71		
Chromium (Cr2O3)	%	0.01	IN-HOUSE	0.01			0.01	0.01	0.0	0.01			0.02			< 0.01	0.01	0.0	< 0.01		
Iron (Fe2O3)	%	0.05	IN-HOUSE	2.73			6.01	5.76	4.2	6.56			7.78			5.39	5.44	0.9	4.83		
Magnesium (MgO)	%	0.01	IN-HOUSE	1.75			3.24	3.16	2.5	4.41			3.15			3.02	3.05	1.0	2.98		
Manganese (MnO)	%	0.01	IN-HOUSE	0.03			1.16	1.13	2.6	0.09			0.1			6.89	6.89	0.0	5.2		
Phosphorus (P2O5)	%	0.03	IN-HOUSE	0.21			1.69	1.52	10.6	0.14			0.1			1.66	1.62	2.4	1.42		
Potasium (K20)	%	0.01	IN-HOUSE	1.01			3.74	3.67	1.9	2.11			1.94			3.63	3.69	1.6	3.82		
Silica (SiO2)	%	0.01	IN-HOUSE	38.1			50.9	50.2	1.4	56.7			65.9			40.5	41	1.2	42.2		
Sodium (Na2O)	%	0.01	IN-HOUSE	1.07			5.02	4.97	1.0	2.09			2.24			4.2	4.24	0.9	4.11		
Titanium (TiO2)	%	0.01	IN-HOUSE	1.4			1.73	1.68	2.9	0.73			0.71			1.19	1.21	1.7	1.19		
Whole Rock Total	%		IN-HOUSE	99.9			97.4	95.8	1.7	99			101			99.2	99.9	0.7	98.5		
Loss on Ignition	%	0.05	IN-HOUSE	34.3			9.08			7.98	8.05	0.9	4.11			14.6	14.64	0.3	14.4		
Phosphorus-Total	µg/g	0.01	EPA 365.4	713			607			564			503			647			590		
Total Kjeldahl Nitrogen	µg/g	0.05	EPA 351.2	4550			930			649			591			519			540		
Total Organic Carbon	% by wt	0.1	LECO	13.6			1			0.7			0.7			0.4			0.8		

Table D3. Percent recovery for reference materials (Caduceon).

	Raw Data (µg/g)			QC Sample Recovery	
LKSD-3	**QC Result**	**Reference Value**	**Lab Mean**	**% Recovery**	**Control Limits**
Silver	2.1	2.4	2.3	**88**	75 - 117
Arsenic	21.9	23	23.4	**95**	60 - 140
Barium	158	N/A	173	**91**	79 - 120
Beryllium	0.5	N/A	0.5	**100**	40 - 180
Cadmium	0.4	0.6	0.6	**67**	17 - 200
Cobalt	26.6	30	29.5	**89**	78 - 119
Chromium	46	51	48.1	**90**	77 - 111
Copper	30.5	34	34.7	**90**	78 - 126
Iron	27043	35000	30340	**77**	66 - 108
Manganese	1125	1220	1282	**92**	88 - 127
Molybdenum	0.6	2	1.2	**30**	0 - 150
Nickel	39.9	44	43.6	**91**	78 - 120
Lead	27.8	26	25.2	**107**	70 - 124
Strontium	24.2	N/A	26.5	**91**	52 - 148
Titanium	870	N/A	1007	**86**	55 - 145
Vanadium	45	55	49.8	**82**	65 - 116
Zinc	123	139	137.9	**88**	57 - 142
GS89-2					
Mercury	1.99	2.08		**96**	57 - 142
WH89-1					
Aluminum (Al2O3)	11.0	12.1		**91**	75 - 125
Barium (BaO)	0.27	0.29		**92**	75 - 125
Calcium (CaO)	5.29	5.9		**90**	75 - 125
Chromium (Cr2O3)	0.03	0.03		**100**	75 - 125
Iron (Fe2O3)	6.18	6.9		**90**	75 - 125
Magnesium (MgO)	3.16	3.5		**90**	75 - 125
Manganese (MnO)	1.16	1.38		**84**	75 - 125
Phosphorus (P2O5)	2.38	2.48		**96**	75 - 125
Potasium (K20)	4.01	4.51		**89**	75 - 125
Silica (SiO2)	59.7	60.5		**99**	75 - 125
Sodium (Na2O)	3.63	4.0		**91**	75 - 125
Titanium (TiO2)	2.1	2.57		**82**	75 - 125

Table D4. Percent recoveries for sample spikes, quality control samples and reference standards for sediment samples (Flett Research).

TOTAL MERCURY - SEDIMENT

Sample Spike Recovery

Sample No.	Recovery (%)
S05_0500	101.2
S05_0500	102.4
RPD	**3.3**
S05_10	103.0
S05_10	99.0
RPD	**3.7**
5108	97.5
5108	97.2
RPD	**0.1**
PH15	98.7
PH15	103.6
RPD	**4.0**

Quality Control Samples

Mess-2 (92 ng/g)		
Sample No.	**Net THg (ng/g dw)**	**Recovery (%)**
1	95	103.7
2	95	103.2
3	91.3	99.2
	Mean	**102.0**
OPR (solids) (1 ng/mL)		
Sample No.	**Net THg (ng/mL)**	**Recovery (%)**
1	0.99	99.4
2	1.01	101.0
3	1.0	95.9
	Mean	**98.8**

Standards

Standard	Recovery (%)
Hg STD 1	98
Hg STD 2	101
Hg STD 3	98
Hg STD 4	101
Hg STD 5	102
Mean	**100**
Hg STD 1	100
Hg STD 2	98
Hg STD 3	100
Hg STD 4	101
Hg STD 5	101
Mean	**100**

METHYL MERCURY - SEDIMENT

Sample Spike Recovery

Sample No.	Recovery (%)
S05_01	89.4
S05_01	90.3
S05_07b	80.8
S05_07b	92.3
51LS	105.0
51LS	83.4
Mean	**90.2**

Quality Control Samples

IAEA 405 (5.49 ± 0.53 ng/g)		
Sample No.	**Net CH3Hg (ng/g dw)**	**Recovery (%)**
1	4.11	74.9
2	4.58	83.4
3	4.23	77.0
4	4.21	76.7
5	4.96	90.3
6	3.66	66.6
	Mean	**78.15**
Alfa (200 ng/L)		
Sample No.	**Net CH3Hg (ng/L)**	**Recovery (%)**
1	210	105.1
2	233	116.6
3	198	98.9
	Mean	**106.9**

Table D5. Percent recoveries for sample spikes, quality control samples and reference standards for biota samples (Flett Research).

TOTAL MERCURY - BIOTA

Sample Spike Recovery

Sample No.	Recovery (%)
4521	96.2
4521	95.8
RPD	**1.6**
4532	95.6
4532	94.5
RPD	**1.6**
4514	96.2
4514	94.9
RPD	**1.0**
4502	100.0
4502	99.3
RPD	**1.8**

Quality Control Samples

DORM-2 (4640 ng/g)

Sample No.	Net THg (ng/g dw)	Recovery (%)
1	4312	92.9
2	4454	96.0
3	4607	99.3
4	4540	97.9
	Mean	**96.5**

OPR (solids) (1 ng/mL)

Sample No.	Net THg (ng/mL)	Recovery (%)
1	0.98	98.4
2	0.96	95.5
3	0.97	96.9
4	0.97	97.4
	Mean	**97.1**

Standards

Standard	Recovery (%)
Hg STD 1	100
Hg STD 2	101
Hg STD 3	101
Hg STD 4	100
Hg STD 5	98
Mean	**100**
Hg STD 1	102
Hg STD 2	101
Hg STD 3	101
Hg STD 4	100
Hg STD 5	96
Mean	**100**

METHYL MERCURY - BIOTA

Sample Spike Recovery

Sample No.	Recovery (%)
4500	108.8
4500	105.5
4510	91.7
4510	90.3
4520	103.4
4520	108.6
4530	102.1
4530	103.6
Mean	**101.8**

Quality Control Samples

Dorm-2 (4.47±0.32 mg/kg)

Sample No.	Net CH3Hg (ng/g dw)	Recovery (%)
1	4327	96.8
2	4701	105.2
3	4063	90.9
4	4145	92.7
5	4801	107.4
6	4583	102.5
7	4257	95.2
8	4284	95.8
9	4446	99.5
10	4409	98.6
11	4576	102.4
	Mean	**98.8**

Alfa (200 ng/L)

Sample No.	Net CH3Hg (ng/L)	Recovery (%)
1	234	117.0
2	183	91.4
3	197	98.3
4	188	94.2
5	184	91.9
6	235	117.7
	Mean	**101.8**

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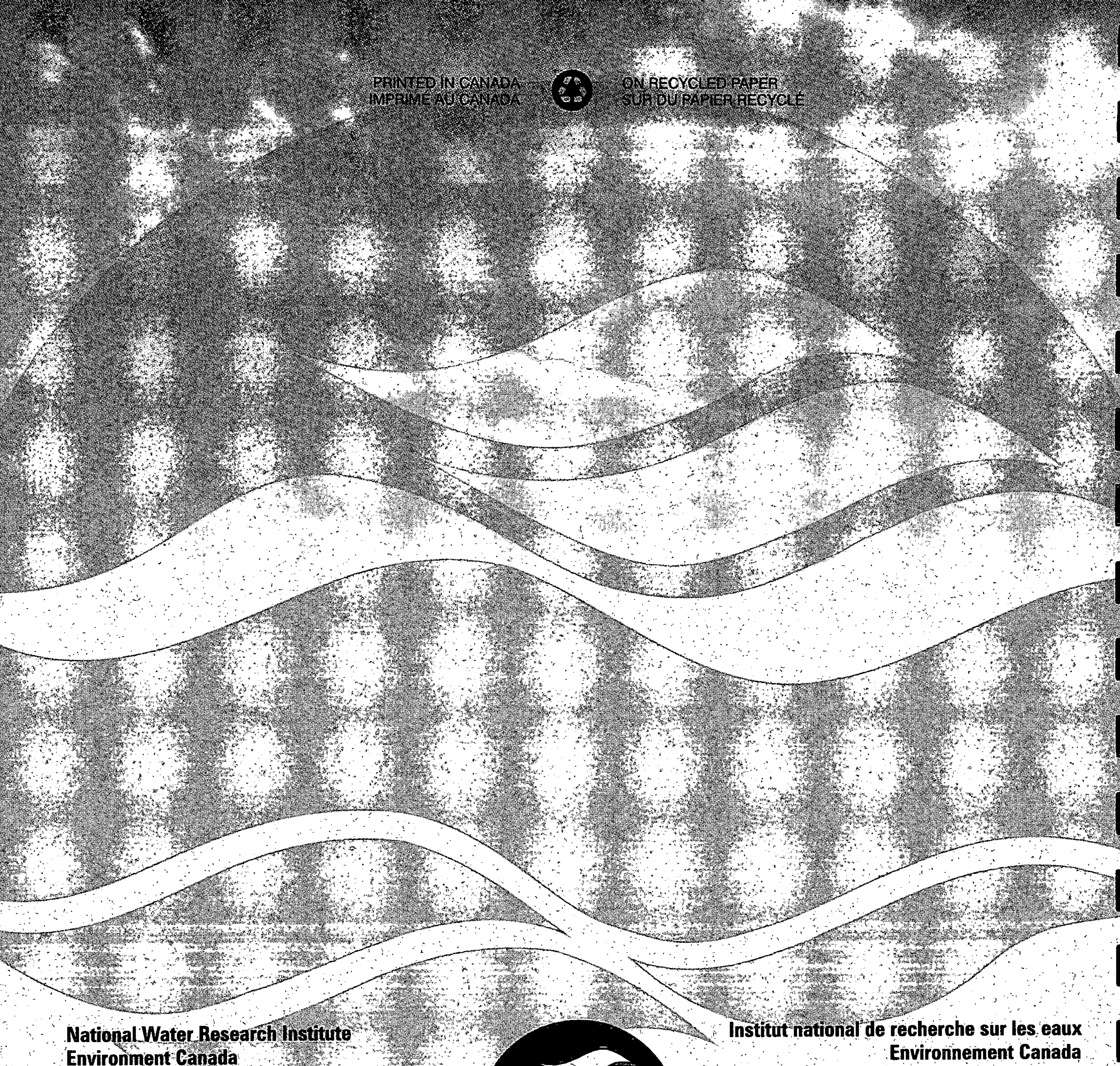

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