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NWRI Contribution Number 00-066

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MANAGEMENT PERSPECTIVE

This is part of Environment Canada's NATURE business line, Result: Human impacts on the health of ecosystems are understood and reduced, Sub-result: Contribute to the actions to reduce the negative impacts on the health of ecosystems, NWRI project: Sediment Assessment and Restoration. This study is part of the Metals In The Environment Research Network (MITERN) collaborative research program between government (Environment Canada, Fisheries and Oceans, Natural Resources), universities and industry (Mining Association of Canada (MAC) and Ontario Power Technologies-formerly Ontario Hydro) co-ordinated through the Canadian Network of Toxicology Centres (CNTC). It constitutes collaborative work between the University of Waterloo (D.G. Dixon) and Environment Canada (U. Borgmann, NWRI) conducted under the Impacts Domain in the first year of research prior to MITERN receiving funding from the Natural Sciences and Engineering Research Council of Canada (NSERC). This project was funded by EC and by a grant from MAC to D.G. Dixon.

This study addresses the issue of the impact of metal emissions from smelters on aquatic ecosystems. The work was conducted on sediments collected by the Geological Survey of Canada (NRCan) and EC in the Rouyn-Noranda area, a location selected as a major site of collaborative research by MITERN. In particular, the study was designed to determine if sediments from lakes in this region were toxic to the amphipod *Hyalella azteca*, and to use recently derived bioaccumulation-toxicity relationships to determine if toxicity could be attributed to any specific metal. Concentrations of Cd were found to be accumulated in *Hyalella* in amounts sufficient to cause toxicity upon exposure to sediments from the only non-headwater lake sampled.

Results from this study will provide a basis for the design of the future field component of collaborative research between the University of Waterloo and NWRI under the impacts domain of the NSERC funded MITERN program, and will contribute to the overall assessment of metal impacts at Rouyn-Noranda by MITERN researchers.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Cette étude s'inscrit dans le cadre du Secteur d'activité NATURE d'Environnement Canada, résultat : Compréhension et réduction de l'impact des activités humaines sur la santé des écosystèmes, sous-résultat : Contribuer aux mesures visant à réduire les impacts négatifs sur la santé des écosystèmes, Projet de l'INRE : Évaluation de l'état et assainissement des sédiments. Elle fait partie du programme de recherche du MITERN (réseau de recherche Les métaux dans l'environnement), une collaboration entre le gouvernement (Environnement Canada, Pêches et Océans, Ressources naturelles), des universités et l'industrie (L'Association minière du Canada (AMC) et Ontario Power Technologies - autrefois Ontario Hydro) coordonnée via le Réseau canadien des centres de toxicologie (RCCT). L'étude est une activité associative entre l'Université de Waterloo (D.G. Dixon) et Environnement Canada (U. Borgmann, INRE) menée dans le cadre du domaine Impacts dans la première année, avant que le MITERN ne reçoive un financement du Conseil de recherches en sciences naturelles et en génie (CRSNG). Le projet a été financé par EC et par une subvention de l'AMC à D.G. Dixon.

Cette étude concerne l'impact des émissions métalliques des fonderies sur les écosystèmes aquatiques. Le travail a été effectué sur des sédiments recueillis par la Commission géologique du Canada (RNCan) et EC dans la région de Rouyn-Noranda, choisie comme important site de recherche associative par le MITERN. En particulier, elle a été conçue pour déterminer si les sédiments des lacs de la région étaient toxiques pour l'amphipode *Hyalella azteca,* et pour utiliser les relations bioaccumulation-toxicité récemment établies pour déterminer si la toxicité pouvait être attribuable à un métal donné. On a observé que Cd se concentrait dans *Hyalella* en quantités suffisantes pour être toxique sur exposition dans le seul lac échantillonné qui ne soit pas un lac d'amont.

Les résultats de l'étude constitueront une base pour la conception du futur volet de terrain des recherches associatives entre l'Université de Waterloo et l'INRE dans le domaine Impacts du programme MITERN financé par le CRSNG, et contribueront à l'évaluation globale des impacts des métaux à Rouyn-Noranda par les chercheurs du MITERN.

Abstract

Survival of *Hyalella azteca* exposed to sediments from selected (mainly headwater) lakes in the Rouyn-Noranda area was minimally to moderately reduced in most sediments relative to Hamilton Harbour and Lake Erie reference sediments; high (>60%) mortality occurred in only three test containers. Reduced survival in Lac Dufault sediments, the only non-headwater lake sampled and the only sediment which also caused reduced growth, were attributed to Cd induced toxicity. Bioaccumulation of Cd exceeded the critical body concentration know to cause toxicity (LBC25). Cadmium bioavailability was also high in sediments from "Gravel Pit Lake" with bioaccumulation reaching 52% of the 4-week LBC25. Concentrations of Cu in *Hyalella* were not useful in quantifying possible Cu induced chronic toxic effects due to regulation of body copper by this amphipod. Copper bioavailability might be relatively high in Lac Dufault sediment, based on Cu leached into the overlying water, but Cd appeared to be the primary cause of toxicity based on comparison of metal concentrations in overlying water with 4-week LC25s. High mortality (67-80%) in *Hyalella* exposed to sediments from Captain Lake and Lac de la Ligne a l'Eau could not be attributed to any of the ten metals measured in *Hyalella* and overlying test water. These results might either be spurious or due to an unknown agent. Metal bioavailability in sediments from headwater lakes near Rouyn-Noranda was generally below that required to cause 25% mortality in *Hyalella* after four weeks of exposure, but sediments from more deep-water lakes should be examined, with special attention given to Cd bioavailability.

Résumé

La survie de *Hyalella azteca* exposés aux sédiments de certains lacs (surtout des lacs d'amont) de la région de Rouyn-Noranda était minimalement à modérément réduite dans la plupart des sédiments, par rapport à des sédiments de référence du port de Hamilton et du lac Érié; on n'a observé de mortalité élevée (>60 %) que dans trois bassins. La réduction de la survie dans les sédiments du lac Dufault, seul lac échantillonné qui ne soit pas un lac d'amont et seuls sédiments qui aient aussi causé une réduction de la croissance, a été attribuée à une toxicité induite par Cd. La bioaccumulation de Cd dépassait la concentration corporelle critique reconnue pour être toxique (CCL25). La biodisponibilité du cadmium était également élevée dans les sédiments du lac « Gravel Pit », avec des bioaccumulations atteignant 52 % de la CCL25 sur 4 semaines. Les concentrations de Cu dans *Hyalella* n'ont pas permis de quantifier d'éventuels effets toxiques chroniques induits par Cu, en raison de la régulation du cuivre corporel chez cet amphipode. La biodisponibilité du cuivre pourrait être relativement élevée dans les sédiments du lac Dufault, si l'on en juge par le Cu lixivié dans les eaux sus-jacentes, mais Cd semble être la cause première de la toxicité, à la lumière des comparaisons des concentrations de métal dans l'eau sus-jacente avec les CCL25 sur 4 semaines. La mortalité élevée (67 à 80 %) chez les *Hyalella* exposés aux sédiments du lac Captain et du lac de la Ligne à l'Eau n'a pu être attribuée à aucun des 10 métaux mesurés chez *Hyalella* et dans l'eau d'essai sus-jacente. Il pourrait s'agir soit de résultats parasites, soit de l'effet d'un agent inconnu. La biodisponibilité des métaux dans les sédiments de lacs d'amont près de Rouyn-Noranda était généralement inférieure à celle nécessaire pour causer une mortalité de 25 % chez *Hyalella* après quatre semaines d'exposition, mais les sédiments de lacs plus profonds devraient être étudiés, en portant une attention spéciale à la biodisponibilité de Cd.

1. INTRODUCTION

The Rouyn-Noranda area, which has a long history of mining and smelting activity, was chosen as a major site for collaborative research on metal effects in the environment by the Metals In The Environment Research Network (MITERN), a consortium of university, industry and government scientists. Prior to conducting more detailed research at Environment Canada or the University of Waterloo, a preliminary analysis of metal bioavailability and sediment toxicity was performed on selected sediments from lakes in this area to determine the potential for toxic effects to *Hyalella azteca* and to identify which metals are of greatest environmental concern. Surface (2-10 cm depth) and deeper historical (20-30 cm) sediment samples were collected by the Geological Survey of Canada (Department of Natural Resources) from eleven lakes in 1998, and ponar grab samples from three additional lakes were collected by the National Water Research Institute (Environment Canada) as part of other studies in this area. The present study was designed as a preliminary assessment of potential effects, rather than a definitive environmental assessment, so replicate samples were not taken. Furthermore, the potential use of inductively-coupled mass spectrometry (ICP-MS) for measuring multiple metal concentrations in *Hyalella* was tested using procedures newly developed in collaboration with the National Laboratory for Environmental Testing (NLET). Presented here are the results of chronic (4-week) toxicity tests with *Hyalella*, together with measurements of metals in sediment and in *Hyalella* and overlying test water at the end of toxicity test. Metal concentrations accumulated by *Hyalella* were compared to critical body concentrations know to cause toxic effects for those metals where such data were available.

2. MATERIALS AND METHODS

Sediment samples were collected with a sediment corer from headwater lakes in the Rouyn-Noranda area by the Geological Survey of Canada in July 1998. Sediment core sections from duplicate samples from eleven of these lakes were sent to NWRI for toxicity testing and metal bioavailability determination. Core sections were obtained from near the surface of the core (2-

10 cm interval) representing recent deposition and from deeper in the core (20-30 cm) to obtain sediments presumably deposited before European colonization. Additional sediment samples were collected by mini-ponar grab sampler from two headwater lakes ("Gravel Pit Lake" near Rouyn and the more distant "Green Lake") and from the larger Lac Dufault near Rouyn in October 1998 by the Technical Operations Branch of NWRI (Table 1). Some surface water chemistry parameters for these lakes are summarized in Table 1. Two reference sediments (Hamilton Harbour site 1 and Lake Erie site 303) were tested at the same time for comparison with the test sites. Survival and growth of *Hyalella* have always been high in these reference sediments in previous experiments.

Total metal concentrations in sediments were determined on samples which were freeze dried and ground with a mortar and pestle. A 0.5 g subsample was digested with concentrated nitric (5 mL) and hydrofluoric (3 mL) acid in Teflon beakers on a hotplate at 95° C and evaporated to dryness. Residues were redissolved in hydrogen peroxide (30%, 1 mL) and nitric acid (0.4 M, 5 mL) and gently heated for 1 h. Samples were then cooled, diluted to 50 mL with 0.4 M nitric acid and centrifuged at 5000 rpm for 30 minutes. Metals were analyzed on a Jy 74 inductively coupled argon plasma optical emission system (ICAP-OES). Recovery of metals from certified reference material (NIST-2704 Buffalo River sediment) was within 10% of the certified values. The detection limit for Cd by ICAP-OES was rather high (3.4 µg/g dry weight), so the sediment digests were re-analyzed by graphite furnace atomic absorption spectrophotometry (GFAAS) using a Varian SpectraAA 400 graphite furnace atomic absorption spectrophotometer with Zeeman background correction using a partition tube. Cadmium concentrations measured by both methods are listed in Table 2.

Four-week chronic sediment toxicity tests with *Hyalella azteca* were conducted according to Borgmann and Norwood 1999. Tests were conducted in polycarbonate Imhoff settling cones, plugged with a #4 silicone rubber stopper. Fifteen mL of sediment and 1 L of overlying water was added to each cone. Tests with Imhoff settling cones instead of beakers allow the use of large water to sediment ratios, improving overlying water quality during long term tests and negating the need for periodic replacement of the water. All metals released by the sediment into the overlying water are retained in the test chamber and can be measured at the end of the test (Borgmann and Norwood 1999a). Experiments were conducted at 23° C with a 16 h light:8 h dark photo-period and with dechlorinated Burlington city tap water (originating from Lake Ontario, hardness 130 mg/L, alkalinity 90 mg/L, DOC 2.3 mg/L, pH 7.8-8.6) overlying the test sediments. The test containers received gentle aeration through a glass tube capped with the end of a 200 µL, 25 mm capillary polypropylene pipette tip to provide a small diameter opening. Test chambers were allowed to equilibrate under aeration for 14 days before addition of fifteen 0-1 week old *Hyalella*. Food consisted of ground Tetra-Min® fish food flakes added at the rate of 2.5, 5.0, 7.5 and 10 mg/wk in weeks 1, 2, 3 and 4 respectively.

The overlying water was carefully decanted and then filtered through acid rinsed 0.4 micron polycarbonate filters at the end of the test for metal analysis by ICP-mass spectrometry by the National Laboratory for Environmental Testing (NLET), Environment Canada, Burlington, Ontario. A subsample of the water was also analyzed for dissolved organic (DOC) and inorganic (alkalinity) carbon by UV digestion and infrared detection by NLET. The pH in the test chambers ranged from 7.88 to 8.29. Ammonia was non-detectable in all water samples at the time of addition of animals and at the end of the experiment, except for a trace (non-toxic) amount (approx. 0.06 mM) in test containers with surface sediments from Lac Vose at the start of the test.

Metal concentrations in *Hyalella* were measured in animals which had been placed in clean water with 50 µmol/L EDTA, cotton gauze as a substrate, and 5 mg Tetra-Min fish food flakes for 24 hours to clear their guts before drying at 60° C in an oven. Groups of six dried amphipods (2.0-3.6 mg total dry mass) were digested together, unless fewer than six animals survived, in which case all surviving animals were digested together. Each vial received 150 µL of 70% nitric acid and was allowed to digest at room temperature for 1 week. Then 120 µL of 30% hydrogen peroxide was added and digestion allowed to continue for another 24 h. Each sample was made up to 6.0 mL with Milli-Q de-ionized water. Metal analysis were then conducted by ICP-mass spectrometry by NLET. A 10 second integration time was used on the ICP-MS instead of the usual 20 seconds, in order to reduce the amount of sample required for analysis and increase the number of metals which could be analyzed.

3. RESULTS AND DISCUSSION

3.1 Sediment toxicity and chemical analysis

An initial test was performed to compare chemical analysis results for Cd measured by ICP-MS with those obtained by GFAAS. This was done on overlying water samples collected from previous toxicity tests with sediments from lakes in the Sudbury area. The ICP-MS and GFAAS programs used were identical to those used in subsequent *Hyalella* tissue analyses. Agreement between the methods was good for most samples above 100 ng/L (n=59, Fig. 1). Two samples which had minimal Cd (1 and 4 ng/L) measured by ICP-MS gave higher measurements by GFAAS (90 and 180 ng/L), possibly because samples were contaminated before GFAAS analysis, and one sample had a slightly lower GFAAS estimate (290 ng/L) compared to the ICP-MS analysis (440 ng/L). All other samples were in reasonable agreement. On average, ICP-MS samples were greater by 11 ng/L (SD 32 ng/L, n=56). The detection limit for Cd by ICP-MS (based on the 95% CI for blanks) estimated during subsequent analysis of overlying water from toxicity tests with the Rouyn-Noranda sediments was 13 ng/L (Table 3). By comparison, the median detection limit for Cd in tissue digests was 8 ng/L, equivalent to a median body concentration of 16 ng/g. This was deemed adequate for body concentration analysis, since the critical body concentration resulting in 25% mortality in four weeks (LBC25) was 30 000 ng/g (Borgmann et al. 1998).

Survival of *Hyalella* after 4-week exposure to sediments from the Rouyn-Noranda area was fair to good in most of the sediments. Only 4 of the test containers had less than 50% survival (20% in Lac de la Ligne a l'Eau surface sediment, 27% in Lac Dufault sediment, 33% in Captain Lake deep sediment, and 47% in Lac Bousseur sediment, Table 2). Growth was similarly good in most sediments (1.25-2.59 mg final wet weight/individual), but was low in *Hyalella* exposed to Lac Dufault sediment (0.30 mg). A clear and strong relationship between sediment chemistry and toxicity was not observed, except for Lac Dufault sediment which had the second lowest survival, lowest growth rate, and highest Cd, Cu, Pb and Zn concentrations (Table 2). The high sediment concentrations for Cd, Cu and Zn metals coincided with high concentrations in the overlying water (Table 3), and high concentrations of Cd, but not Cu or Zn, in *Hyalella* at the end of the toxicity test (Table 4). Selenium and Tl in overlying water, and Cr in *Hyalella*, were also highest in the test container with Lac Dufault sediment.

The metal concentration in the overlying water correlated most closely with that in sediment for Cd ($R^2 = 0.75$), but was also high for Cu ($R^2 = 0.67$, Table 5, Fig. 2). Although statistically significant, correlations between metals in water and sediment were poor ($R^2 < 0.5$) for Co and Zn. Concentrations of Cr, Mn, Ni, and Pb in overlying water were generally low and not correlated well with concentrations in the sediment.

Metal bioavailability, as determined by concentration in *Hyalella*, was very strongly correlated to metal in water and sediment for Cd ($R^2 > 0.78$). Thallium in *Hyalella* also correlated very well with Tl in water (Tl in sediment not measured). Concentrations of Cu and Zn in *Hyalella* were weakly to moderately correlated to Cu and Zn in water and sediment ($R^2 = 0.20$-0.54), although the regressions were statistically significant. Since body concentrations of Cu and Zn are regulated by *Hyalella* (Borgmann and Norwood 1995a), a poorer correlation between body and environmental concentrations than that obtained for Cd is expected. Body concentrations of As, Cr, and Ni in *Hyalella* were low and not significantly correlated to water or sediment concentrations. Modest, but statistically significant ($R^2 = 0.24$-0.48) correlations were also observed between Mn and Pb in *Hyalella* and sediment and between Co and Mn in *Hyalella* and water (Table 5).

In all cases where sediments were collected from both the top and bottom of a sediment core, Cd, Cu and Zn concentrations were higher in surface sediments than in deep sediments (Table 2). Similarly, Cd and Cu measured in *Hyalella* were consistently higher in animals exposed to surface sediments (Table 4). Dissolved Cu and Cd in overlying water were also higher in test containers with surface sediments, although Cd concentrations were below the detection limit in many samples (Table 3). The trends for other metals were not consistent. Surface enrichment of both total and bioavailable metals was, therefore, clearly demonstrated for Cd and Cu. The enrichment factor was greater for Cd than for Cu in all lakes except Lake Wilson (Table 2). The surface enrichment seen for Zn was not, however, clearly reflected in Zn in *Hyalella*, which was relatively constant (Table 4).

3.2 Comparison to critical body and water concentrations

Data on critical water and body concentrations of metals resulting in chronic toxicity are available for Cd, Cu, Ni, Pb, Tl and Zn, allowing estimation of the contribution of these metals to sediment toxicity. In general, body concentrations are better indicators of toxic effects to *Hyalella* than are concentrations in water or sediment for Cd, Ni, Pb and Tl (Borgmann et al. 1991, Borgmann et al. 1998, Borgmann and Norwood 1999b, unpublished data). Regulation of tissue metal concentrations makes if more difficult to predict toxic effects of Zn, and especially of Cu, from concentrations in *Hyalella* (Borgmann and Norwood 1997). Since bioavailability and toxicity of Cd, Cu, Ni and Pb to *Hyalella* appear to be controlled primarily by dissolved metal in the water rather than total metal in the sediment (Deaver and Rodgers 1996, Warren et al. 1998, Borgmann and Norwood 1999b, unpublished data) metal concentrations in overlying water can serve as a secondary estimate of metal bioavailability, especially for Cu and Zn. However, caution must be used when interpreting metal concentrations in overlying water because organic matter released from sediments can significantly reduce the toxicity of metals in water (Borgmann and Norwood 1999b). Overlying water concentrations of metals which exceed concentrations previously shown to be toxic are, therefore, indicative of potential toxicity, but are not definitive.

Concentrations of metals in *Hyalella* and in overlying water are compared to lethal body (LBC25) or water (LC25) concentrations resulting in 25% mortality after 4 weeks in Tables 6 and 7 for data from some of the more heavily contaminated sediments. The only case where any metal concentration in *Hyalella* exceeded the critical body concentration was for Cd in animals exposed to Lac Dufault sediments. Body concentrations were 2.5 times above the 4-week LBC25 (Table 6). Cadmium induced toxicity would be expected for *Hyalella* exposed to this sediment, and high mortality and low growth were, indeed, observed. Cadmium concentration in

Hyalella exposed to sediments from Gravel Pit Lake were also high (0.5 times the LBC25), and Cd in this sediment might be contributing to the slightly reduced survival (60%), but the effect was small. Copper concentrations in *Hyalella* were high in all test chambers because Cu is essential and regulated by *Hyalella*, and body concentrations of Cu do not help to define cause and effect. Copper in overlying water in the test chamber with Lac Dufault sediment was almost at the LC25 (Table 7) and might be contributing somewhat to metal toxicity, although most of the toxicity was clearly being caused by Cd, which exceeded the LC25 by a factor of 4.7.

Metal concentrations in *Hyalella* and overlying water were low and do not help identify the cause of mortality in animals exposed to bottom sediments from Captain Lake (33% survival) or surface sediments from Lac de la Ligne à l'Eau (20%, Tables 3 and 4). Ammonia, both at the time animals were added and at the end of the experiment was non-detectable and not responsible for mortality. This suggests that the observed toxicity in these tests was either spurious or caused by some unknown factor.

Data of critical body concentrations for As, Co, Cr, and Se in *Hyalella* are not yet available and definitive conclusions about the possible role of these metals in toxicity cannot be made. However, maximum body concentrations were quite low (Table 4), and well below the critical body concentration of any of the metals for which LC25s are available. It is unlikely, therefore, that any of these metals were contributing to toxicity. Of possible concern, however, is the high concentration of Mn in *Hyalella* and overlying water in the container with deep sediments from Lac Vose. Survival was relatively low in this container (47%). The critical body concentration of this metal in *Hyalella* still needs to be determined.

4. CONCLUSIONS

Cadmium was the only metal accumulated by *Hyalella* in excess of the 4-week LBC25, and this only occurred in *Hyalella* exposed to sediment from Lac Dufault, the only non-headwater lake sampled in this study. As expected, this sediment was toxic. Copper bioavailability in this sediment might also be high, as judged from Cu in overlying water, but this only equals the 4-week LC25 for metal in water, whereas Cd in water exceeds the LC25 by a factor of 4.7. Unlike Cd, concentrations of Cu in *Hyalella* were not useful in quantifying possible Cu induced chronic toxic effects due to regulation of body copper by this amphipod. Metal bioavailability in sediments from headwater lakes near Rouyn-Noranda was generally below that required to cause 25% mortality in *Hyalella* after four weeks of exposure, but sediments from more deep-water lakes should be examined, with special attention given to Cd bioavailability.

5. ACKNOWLEDGMENTS

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Table 1. Sampling stations in the Rouyn-Noranda area and surface water chemistry.

Station	Lake name	Latitude	Longitude	Conductivity (µS/cm)	Alkalinity (mg/L)	pH	Temperature (°C)	Dissolved oxygen (mg/L)
RN98-005	Lake Wilson	48.1333	79.9125	183	71	7.77	23.1	9.9
RN98-010	Captain Lake	48.2939	79.6633	42	13	6.42	23.0	10.9
RN98-011	Lake Mulvin	48.2206	79.6389	55	15	7.12	23.4	11.0
RN98-012	Lac de la Frontiere	48.0842	79.5144	36	5	6.27	22.4	9.0
RN98-013	Lac Delaas	48.1900	79.5031	50	14	7.16	23.2	10.5
RN98-015	Lac Bousseur	48.1403	79.4289	54	8	6.96	23.4	10.9
RN98-016	Lac Fabie	48.4056	79.3975	71	28	7.51	22.2	10.1
RN98-017	Lac Derry	48.4239	79.1283	21	1	5.64	23.4	9.8
RN98-021	Lac Martin	47.8639	79.1161	30	6	7.08	23.0	9.4
RN98-025	Lac Vose	48.4728	79.8306	27	5	6.10	20.7	8.7
RN98-030	Lac de la Ligne a l'Eau	48.3339	79.2186	25	9	6.23	19.7	9.1
RN97-SE-15	Gravel Pit Lake	47.8215	78.2652	16	1	5.49	20.2	8.9
RN97-NE-01	Green Lake	48.3047	78.9182	31	3	4.40	20.0	8.2
-	Lac Dufault	48.2879	79.0213	-	-	-	-	-

Stations labelled RN98 were sampled by the Geological Survey of Canada in July 1998 using a sediment corer.
Chemistry data for stations labelled RN97 were collected by the Geological Survey of Canada in 1997.
Green, Gravel Pit, and Dufault Lakes were sampled by Environment Canada in October 1998 using a ponar grab.
All lakes except Lac Dufault are headwater lakes.

Table 2. *Hyalella* survival, *Hyalella* final dry weight, metal concentration in sediment measured by ICAP-OES, and cadmium concentration measured by graphite furnace atomic absorption spectrophotometry (Cd GFAAS) (µg/g dry weight).

Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	Ca	Cd	Cd GFAAS	Co	Cr	Cu	Fe	Mg	Mn	Ni	Pb	Zn
Lake Wilson	Top	73	2.06	17730	ND	1.3	13.9	81.5	463.7	44500	8132	486	22.7	362.5	205
Lake Wilson	Bottom	80	2.40	14710	ND	0.9	8.9	42.3	69.8	17420	3178	274	21.0	17.2	93
Captain Lake	Top	80	2.05	12390	ND	2.3	6.6	23.9	35.5	9034	2102	101	27.9	51.0	134
Captain Lake	Bottom	33	1.83	12240	ND	0.8	6.7	27.8	24.5	7986	2443	109	23.1	ND	99
Lake Mulvin	Top	67	1.80	22410	ND	1.2	16.7	86.8	24.3	39560	11340	547	43.9	ND	100
Lac de la Frontiere	Top	87	1.87	15270	ND	2.8	16.3	54.5	43.0	27020	6774	232	37.7	13.0	178
Lac de la Frontiere	Bottom	67	2.09	14450	ND	0.8	11.3	54.3	29.9	22640	7014	223	29.9	ND	82
Lac Delaas	Top	87	1.63	19280	ND	2.6	11.5	60.6	37.6	34460	5667	299	34.0	28.8	162
Lac Delaas	Bottom	73	2.03	17840	ND	0.9	11.4	61.0	23.1	27870	5803	308	30.4	ND	96
Lac Bousseur	Top	47	2.03	13380	ND	1.2	30.6	98.0	34.3	36290	13120	478	68.1	ND	201
Lac Fabie	Top	53	2.36	25030	ND	1.3	28.1	122.1	22.9	64060	20375	738	59.0	ND	140
Lac Fabie	Bottom	73	2.08	24360	ND	0.5	26.9	122.3	15.1	61520	27630	691	59.2	ND	114
Lac Derry	Top	93	1.28	13080	14.9	14.4	5.7	33.8	311.1	10540	2853	89	36.9	280.1	331
Lac Derry	Bottom	87	1.78	12510	4.3	4.7	6.2	31.3	290.9	10180	2632	110	32.5	108.7	213
Lac Martin	Top	53	2.59	17050	ND	2.8	23.5	102.3	62.0	45050	12000	543	65.1	53.8	192
Lac Martin	Bottom	93	1.44	17300	ND	1.0	24.0	111.5	31.2	47820	14000	597	63.5	ND	137
Lac Vose	Top	53	1.52	14590	8.5	13.9	41.4	37.3	243.4	39150	2187	494	36.0	266.5	383
Lac Vose	Bottom	47	1.53	15510	ND	1.4	21.6	40.1	40.7	18560	2256	482	20.8	ND	85
Lac de la Ligne a l'Eau	Top	20	1.55	13690	ND	1.3	17.4	76.8	28.1	41950	9773	373	48.5	ND	128
Lac de la Ligne a l'Eau	Bottom	67	1.55	18820	ND	0.6	21.3	102.1	21.6	53190	13370	462	59.1	ND	114
Gravel Pit Lake	Top	60	1.42	14460	13.6	15.5	17.5	72.9	615.5	39730	9366	253	44.2	409.4	450
Green Lake	Top	77	1.25	10840	3.1	4.9	9.6	79.9	67.1	22890	5425	209	41.2	116.3	195
Lac Dufault	Top	27	0.30	18620	21.0	26.6	32.2	77.7	1739.0	51500	12700	518	42.7	434.8	1643
HH1	Top	87	1.40	-	-	-	11.5	119.2	101.1	45400	0	1722	46.9	113.4	1235
LE303	Top	93	1.76	-	-	-	2.7	34.3	24.5	21900	0	682	19.4	ND	69
maximum		93	2.59	25030	21.0	26.6	41.4	122.3	1739.0	64060	27630	1722	68.1	434.8	1643
detection limit		-	-	0.5	3.4	0.08	0.9	0.9	1.0	1.2	0.3	0.2	2.0	2.5	0.9

HH1 and LE303 are Hamilton Harbour and Lake Erie reference sediments known to give good survival and growth of *Hyalella*.

Table 2. *Hyalella* survival, *Hyalella* final dry weight, metal concentration in sediment measured by ICAP-OES, and cadmium concentration measured by graphite furnace atomic absorption spectrophotometry (Cd GFAAS) (µg/g dry weight).

Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	Ca	Cd	Cd GFAAS	Co	Cr	Cu	Fe	Mg	Mn	Ni	Pb	Zn
Lake Wilson	Top	73	2.06	17730	ND	1.3	13.9	81.5	463.7	44500	8132	486	22.7	362.5	205
Lake Wilson	Bottom	80	2.40	14710	ND	0.9	8.9	42.3	69.8	17420	3178	274	21.0	17.2	93
Captain Lake	Top	80	2.05	12390	ND	2.3	6.6	23.9	35.5	9034	2102	101	27.9	51.0	134
Captain Lake	Bottom	33	1.83	12240	ND	0.8	6.7	27.8	24.5	7986	2443	109	23.1	ND	99
Lake Mulvin	Top	67	1.80	22410	ND	1.2	16.7	86.8	24.3	39560	11340	547	43.9	ND	100
Lac de la Frontiere	Top	87	1.87	15270	ND	2.8	16.3	54.5	43.0	27020	6774	232	37.7	13.0	178
Lac de la Frontiere	Bottom	67	2.09	14450	ND	0.8	11.3	54.3	29.9	22640	7014	223	29.9	ND	82
Lac Delaas	Top	87	1.63	19280	ND	2.6	11.5	60.6	37.6	34460	5667	299	34.0	28.8	162
Lac Delaas	Bottom	73	2.03	17840	ND	0.9	11.4	61.0	23.1	27870	5803	308	30.4	ND	96
Lac Bousseur	Top	47	2.03	13380	ND	1.2	30.6	98.0	34.3	36290	13120	478	68.1	ND	201
Lac Fabie	Top	53	2.36	25030	ND	1.3	28.1	122.1	22.9	64060	20375	738	59.0	ND	140
Lac Fabie	Bottom	73	2.08	24360	ND	0.5	26.9	122.3	15.1	61520	27630	691	59.2	ND	114
Lac Derry	Top	93	1.28	13080	14.9	14.4	5.7	33.8	311.1	10540	2853	89	36.9	280.1	331
Lac Derry	Bottom	87	1.78	12510	4.3	4.7	6.2	31.3	290.9	10180	2632	110	32.5	108.7	213
Lac Martin	Top	53	2.59	17050	ND	2.8	23.5	102.3	62.0	45050	12000	543	65.1	53.8	192
Lac Martin	Bottom	93	1.44	17300	ND	1.0	24.0	111.5	31.2	47820	14000	597	63.5	ND	137
Lac Vose	Top	53	1.52	14590	8.5	13.9	41.4	37.3	243.4	39150	2187	494	36.0	266.5	383
Lac Vose	Bottom	47	1.53	15510	ND	1.4	21.6	40.1	40.7	18560	2256	482	20.8	ND	85
Lac de la Ligne a l'Eau	Top	20	1.55	13690	ND	1.3	17.4	76.8	28.1	41950	9773	373	48.5	ND	128
Lac de la Ligne a l'Eau	Bottom	67	1.55	18820	ND	0.6	21.3	102.1	21.6	53190	13370	462	59.1	ND	114
Gravel Pit Lake	Top	60	1.42	14460	13.6	15.5	17.5	72.9	615.5	39730	9366	253	44.2	409.4	450
Green Lake	Top	77	1.25	10840	3.1	4.9	9.6	79.9	67.1	22890	5425	209	41.2	116.3	195
Lac Dufault	Top	27	0.30	18620	21.0	26.6	32.2	77.7	1739.0	51500	12700	518	42.7	434.8	1643
HH1	Top	87	1.40	-	-	-	11.5	119.2	101.1	45400	0	1722	46.9	113.4	1235
LE303	Top	93	1.76	-	-	-	2.7	34.3	24.5	21900	0	682	19.4	ND	69
maximum		93	2.59	25030	21.0	26.6	41.4	122.3	1739.0	64060	27630	1722	68.1	434.8	1643
detection limit		-	-	0.5	3.4	0.08	0.9	0.9	1.0	1.2	0.3	0.2	2.0	2.5	0.9

HH1 and LE303 are Hamilton Harbour and Lake Erie reference sediments known to give good survival and growth of *Hyalella*.

Table 3. *Hyalella* survival, *Hyalella* final dry weight, and overlying water chemistry (dissolved organic carbon (DOC, mg/L), alkalinity (mg/L) and metal concentration measured by ICP-MS (µg/L)) at the end of sediment toxicity tests.

Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	DOC	Alkalinity	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Se	Tl	Zn
Lake Wilson	Top	73	2.06	3.3	68.9	0.53	ND	0.10	0.60	7.40	1.47	0.86	0.31	0.51	0.05	2.28
Lake Wilson	Bottom	80	2.40	3.8	64.8	0.47	ND	0.04	0.69	1.04	2.21	ND	0.27	0.29	0.05	1.55
Captain Lake	Top	80	2.05	2.5	68.3	0.71	0.03	0.04	0.86	0.88	0.09	0.37	0.25	ND	0.07	3.35
Captain Lake	Bottom	33	1.83	4.5	74.5	0.52	ND	0.03	0.59	0.88	0.23	0.21	0.24	ND	0.05	2.94
Lake Mulvin	Top	67	1.80	2.7	60.3	0.77	0.03	0.04	0.38	1.02	0.15	0.25	0.44	ND	0.07	2.74
Lac de la Frontiere	Top	87	1.87	3.6	57.5	0.85	0.04	0.12	0.52	1.10	0.14	0.25	0.26	0.35	0.06	2.01
Lac de la Frontiere	Bottom	67	2.09	4.4	61.6	0.65	0.01	0.05	0.56	0.69	ND	0.18	0.33	0.54	0.04	0.46
Lac Delaas	Top	87	1.63	2.1	66.7	0.79	0.03	0.04	0.60	1.18	0.08	0.35	0.32	ND	0.06	1.52
Lac Delaas	Bottom	73	2.03	2.4	65.1	0.46	ND	0.03	0.68	0.42	0.09	0.11	0.28	0.34	0.06	ND
Lac Bousseur	Top	47	2.03	3.1	63.4	1.00	0.03	0.12	0.50	1.53	ND	0.21	0.24	0.34	0.06	0.80
Lac Fabie	Top	53	2.36	2.3	66.6	1.24	0.02	0.02	0.60	2.11	ND	0.37	0.26	ND	0.04	ND
Lac Fabie	Bottom	73	2.08	2.8	68.3	0.59	0.02	0.03	0.59	1.03	0.09	0.39	0.32	0.40	0.05	1.33
Lac Derry	Top	93	1.28	3.9	55.7	4.86	0.11	0.05	0.46	2.64	0.08	ND	0.68	0.28	0.05	2.77
Lac Derry	Bottom	87	1.78	3.8	56.5	10.53	0.03	0.05	0.54	2.02	0.49	ND	0.25	ND	0.06	12.07
Lac Martin	Top	53	2.59	3.4	59.1	0.99	0.05	0.04	0.57	1.25	ND	0.53	0.16	0.92	0.07	8.18
Lac Martin	Bottom	93	1.44	3.1	60.6	0.45	ND	0.05	0.69	0.72	0.11	0.27	0.18	0.36	0.06	10.07
Lac Vose	Top	53	1.52	3.8	39.8	1.60	0.13	0.86	0.49	3.24	8.19	1.33	0.18	0.73	0.09	12.57
Lac Vose	Bottom	47	1.53	4.6	62.7	0.46	ND	0.47	0.72	0.69	47.72	0.32	0.16	0.38	0.05	10.47
Lac de la Ligne a l'Eau	Top	20	1.55	3.9	59.8	0.78	0.02	0.03	0.64	1.42	0.15	ND	0.17	0.53	0.08	8.46
Lac de la Ligne a l'Eau	Bottom	67	1.55	4.1	60.8	0.53	ND	0.04	0.87	0.97	0.13	ND	0.16	0.36	0.05	6.02
Gravel Pit Lake	Top	60	1.42	4.1	53.7	3.25	0.31	0.04	0.57	6.02	0.19	0.49	0.27	0.55	0.09	11.57
Green Lake	Top	77	1.25	4.3	61.5	4.31	0.03	0.06	0.61	1.29	0.14	0.52	0.27	0.54	0.06	13.47
Lac Dufault	Top	27	0.30	2.3	52.0	1.84	1.85	0.04	0.33	19.95	0.14	ND	0.40	1.90	0.12	39.17
HH1	Top	87	1.40	3.1	88.7	2.19	0.13	0.05	0.69	4.78	0.15	1.59	0.90	0.69	0.10	10.07
LE303	Top	93	1.76	3.3	89.8	1.13	0.02	0.01	0.78	3.39	ND	ND	0.20	0.24	0.06	0.27
maximum		93	2.59	4.6	89.8	10.53	1.85	0.86	0.87	19.95	47.72	1.59	0.90	1.90	0.12	39.17
detection limit		-	-	-	-	0.18	0.013	0.010	0.05	0.32	0.08	0.10	0.09	0.22	0.003	0.23

Table 4. *Hyalella* survival, *Hyalella* final dry weight, and metal concentration in *Hyalella* measured by ICP-MS (µg/g dry weight).

Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	As	Cd	Co	Cr	Cu	Mn	Ni	Pb	Se	Tl	Zn
Lake Wilson	Top	73	2.06	1.27	1.24	0.39	1.38	114	13.94	0.75	1.07	3.62	0.07	68
Lake Wilson	Bottom	80	2.40	1.42	0.50	0.27	1.62	108	6.91	0.55	0.12	2.94	0.08	65
Captain Lake	Top	80	2.05	0.53	1.56	0.22	1.44	101	5.26	0.63	0.26	2.70	0.24	66
Captain Lake	Bottom	33	1.83	ND	0.56	0.16	1.36	68	6.34	0.43	0.03	1.56	0.08	52
Lake Mulvin	Top	67	1.80	0.44	0.98	0.23	1.60	84	7.23	0.63	0.13	2.31	0.16	60
Lac de la Frontiere	Top	87	1.87	2.22	1.12	0.36	1.48	97	11.21	0.82	0.78	2.37	0.15	65
Lac de la Frontiere	Bottom	67	2.09	ND	0.54	0.17	1.43	78	6.57	0.29	0.07	2.18	0.10	63
Lac Delaas	Top	87	1.63	1.29	2.32	0.16	1.50	112	4.90	0.41	0.10	2.11	0.21	68
Lac Delaas	Bottom	73	2.03	0.85	0.49	0.21	1.67	71	5.74	0.96	0.10	2.44	0.09	66
Lac Bousseur	Top	47	2.03	ND	1.45	0.24	1.41	80	6.39	0.56	0.11	1.85	0.21	66
Lac Fabie	Top	53	2.36	0.38	2.17	0.30	2.03	105	10.35	0.49	0.10	2.15	0.11	66
Lac Fabie	Bottom	73	2.08	ND	0.75	0.46	2.64	96	14.47	1.22	0.34	2.59	0.10	68
Lac Derry	Top	93	1.28	2.22	8.89	1.59	2.78	136	6.76	1.13	1.59	2.15	0.13	69
Lac Derry	Bottom	87	1.78	1.50	1.91	0.17	1.29	114	7.71	0.54	0.65	1.84	0.20	62
Lac Martin	Top	53	2.59	0.64	3.00	0.22	1.48	114	6.62	0.55	0.12	2.32	0.32	64
Lac Martin	Bottom	93	1.44	ND	0.79	0.30	1.78	97	7.46	0.82	0.14	2.48	0.24	63
Lac Vose	Top	53	1.52	1.68	12.26	1.54	1.63	140	19.50	0.61	0.72	1.29	0.39	72
Lac Vose	Bottom	47	1.53	0.87	0.73	0.90	1.79	104	42.89	0.66	0.20	1.87	0.07	69
Lac de la Ligne a l'Eau	Top	20	1.55	1.67	3.52	0.23	2.53	103	6.11	ND	ND	ND	0.26	62
Lac de la Ligne a l'Eau	Bottom	67	1.55	1.43	0.44	0.22	1.84	87	9.62	0.92	0.14	1.11	0.20	69
Gravel Pit Lake	Top	60	1.42	0.71	15.92	0.37	1.61	105	8.79	0.53	1.33	1.66	0.41	67
Green Lake	Top	77	1.25	1.15	0.89	0.33	2.03	110	11.83	0.95	1.27	2.05	0.22	67
Lac Dufault	Top	27	0.30	ND	74.55	1.19	5.01	134	12.77	ND	1.13	ND	0.44	91
HH1	Top	87	1.40	0.94	5.19	0.32	1.98	121	18.92	1.39	1.21	3.17	0.47	75
LE303	Top	93	1.76	0.73	1.69	0.49	1.56	87	16.99	0.85	0.22	2.97	0.25	62
maximum		93	2.59	2.22	74.55	1.59	5.01	140	42.89	1.39	1.59	3.62	0.47	91
median detection limit		-	-	0.40	0.02	0.02	0.10	0.14	0.05	0.30	0.02	0.36	0.005	0.58

Detection limit depends on total dry weight of *Hyalella* digested. Median detection limit is based on 6 *Hyalella* (2.843 mg) in 6 mL digest volume (total weight per sample: median = 2.843 mg, minimum = 0.307, maximum=3.666).

Table 5. Intercept, slope, R^2 (in bold font if >0.5) and n for log-log regressions of metal concentration in water or *Hyalella* (Y) against sediment or water (X) for each metal.

Metal	Y	X	intercept	slope	R^2	n	P
Cd	water	sediment	-1.757	0.929	**0.752**	16	***
Co	water	sediment	-1.923	0.556	0.161	25	*
Cr	water	sediment	-0.142	-0.047	0.012	25	NS
Cu	water	sediment	-0.820	0.574	**0.667**	25	***
Mn	water	sediment	-1.380	0.336	0.021	20	NS
Ni	water	sediment	-0.673	0.155	0.007	18	NS
Pb	water	sediment	-0.702	0.093	0.056	13	NS
Zn	water	sediment	-1.479	0.913	0.363	23	**
Cd	*Hyalella*	sediment	-0.103	1.025	**0.789**	23	***
Co	*Hyalella*	sediment	-0.735	0.229	0.050	25	NS
Cr	*Hyalella*	sediment	-0.055	0.170	0.083	25	NS
Cu	*Hyalella*	sediment	1.815	0.105	0.486	25	***
Mn	*Hyalella*	sediment	0.061	0.360	0.251	25	*
Ni	*Hyalella*	sediment	-0.460	0.185	0.038	23	NS
Pb	*Hyalella*	sediment	-1.424	0.584	0.482	13	**
Zn	*Hyalella*	sediment	1.616	0.091	**0.545**	25	***
As	*Hyalella*	water	0.003	0.106	0.035	19	NS
Cd	*Hyalella*	water	1.661	0.940	**0.834**	18	***
Co	*Hyalella*	water	-0.002	0.366	0.243	25	*
Cr	*Hyalella*	water	0.132	-0.520	0.147	25	NS
Cu	*Hyalella*	water	1.976	0.132	0.378	25	**
Mn	*Hyalella*	water	1.099	0.212	0.455	20	**
Ni	*Hyalella*	water	-0.101	0.192	0.113	18	NS
Pb	*Hyalella*	water	0.057	1.091	0.161	24	NS
Se	*Hyalella*	water	0.303	-0.109	0.018	17	NS
Tl	*Hyalella*	water	1.549	1.894	**0.707**	25	***
Zn	*Hyalella*	water	1.799	0.037	0.198	23	*

*, P<0.05; **, P<0.01; ***, P<0.001; NS, not significant
Units are µg/L or µg/g.

Table 6. *Hyalella* survival, final dry weight, critical body concentration (4-wk LBC25) and ratio of metal concentration in *Hyalella* to the LBC25 (in bold font if >0.5) for selected lakes.

				Cd	Cu	Ni	Pb	Tl	Zn
4 week LBC25-μg/g	-	-	-	30	151	11	26	59	289
Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	Body concentration / LBC25					
Captain Lake	Bottom	**33**	1.83	0.02	0.45	0.04	0.001	0.001	0.18
Lake Wilson	Top	73	2.06	0.04	**0.75**	ND	ND	0.001	0.23
Lac de la Ligne a l'Eau	Top	**20**	1.55	0.12	**0.68**	ND	ND	0.004	0.21
HH1	Top	87	1.40	0.17	**0.80**	0.12	0.046	0.008	0.26
Lac Derry	Top	93	1.28	0.29	**0.90**	0.10	0.061	0.002	0.24
Lac Vose	Top	53	1.52	0.40	**0.92**	0.05	0.027	0.007	0.25
Gravel Pit Lake	Top	60	1.42	0.52	**0.70**	0.05	0.051	0.007	0.23
Lac Dufault	Top	**27**	**0.30**	**2.46**	**0.89**	0.00	0.043	0.008	0.32

LBC25 and LC25 values from Borgmann et al. 1998, Borgmann and Norwood 1999, Borgmann and Norwood 2000.

Table 7. *Hyalella* survival, final dry weight, 4-week LC25 for metal in water and ratio of metal concentration in overlying water to the LC25 (in bold font if >0.5) for selected lakes

				Cd	Cu	Ni	Pb	Tl	Zn
4 week LC25-μg/L	-	-	-	0.39	21	26	3.7	9.8	111
Sediment source	Core section	*Hyalella* survival (%)	*Hyalella* weight (mg)	Water concentration / LC25					
Captain Lake	Bottom	**33**	1.83	ND	0.04	0.01	0.06	0.005	0.03
Lake Wilson	Top	73	2.06	ND	0.35	ND	0.08	0.005	0.02
Lac de la Ligne a l'Eau	Top	**20**	1.55	0.06	0.07	ND	0.04	0.008	0.08
HH1	Top	87	1.40	0.32	0.23	0.06	0.24	0.010	0.09
Lac Derry	Top	93	1.28	0.28	0.13	ND	0.18	0.005	0.02
Lac Vose	Top	53	1.52	0.34	0.15	0.05	0.05	0.009	0.11
Gravel Pit Lake	Top	60	1.42	**0.78**	0.29	0.02	0.07	0.009	0.10
Lac Dufault	Top	**27**	**0.30**	**4.70**	**0.95**	ND	0.11	0.012	0.35

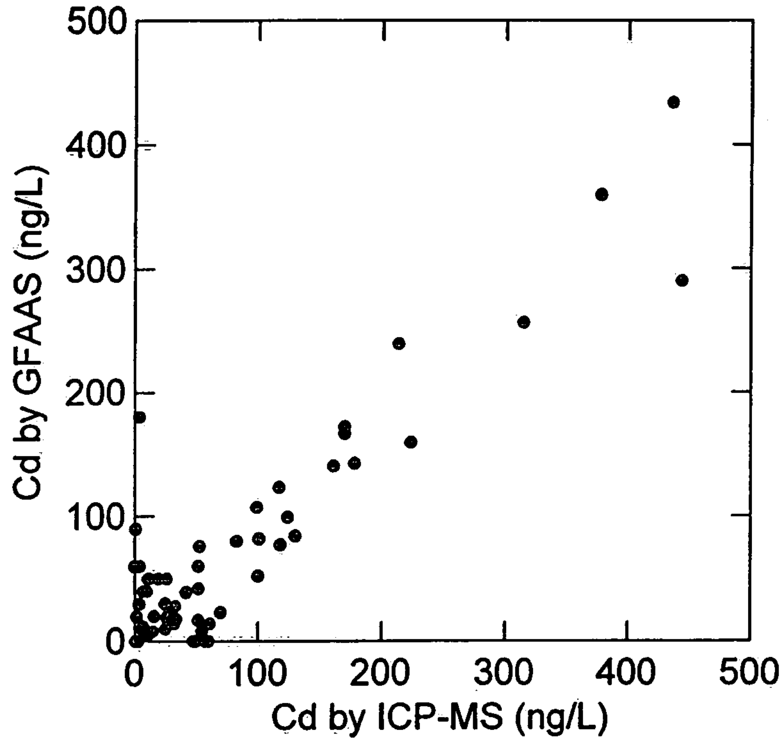

Figure 1. Comparison of cadmium concentrations in water samples measured by graphite furnace atomic absorption spectrophotometry (GFAAS) and inductively coupled plasma mass spectrometry (ICP-MS).

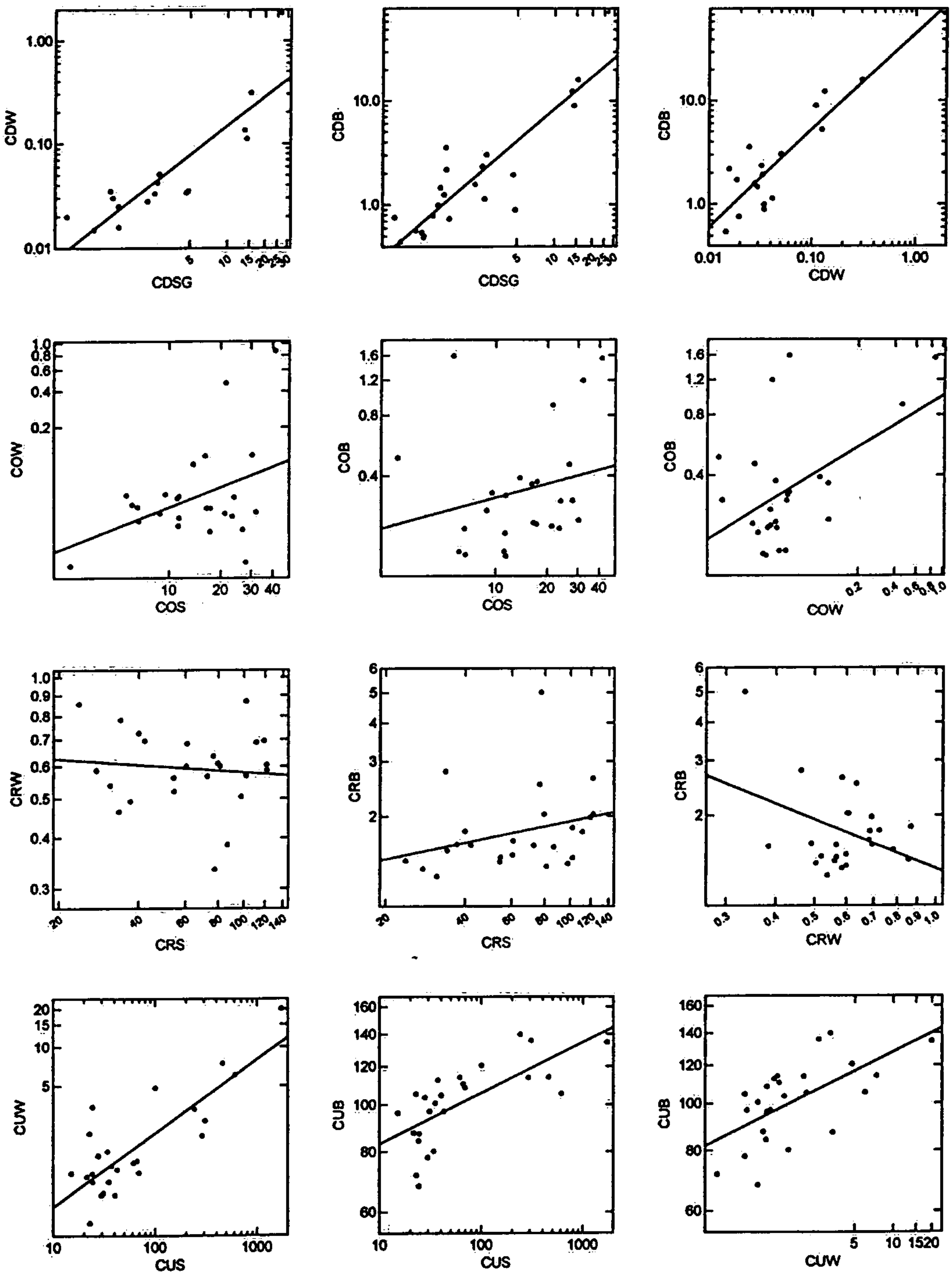

Figure 2. Concentrations of cadmium (CD), cobalt (CO), chromium (CR) and copper (CU) measured in overlying water (W) or *Hyalella* (B) plotted against concentrations in sediment (S) or overlying water in toxicity tests with different sediment samples.

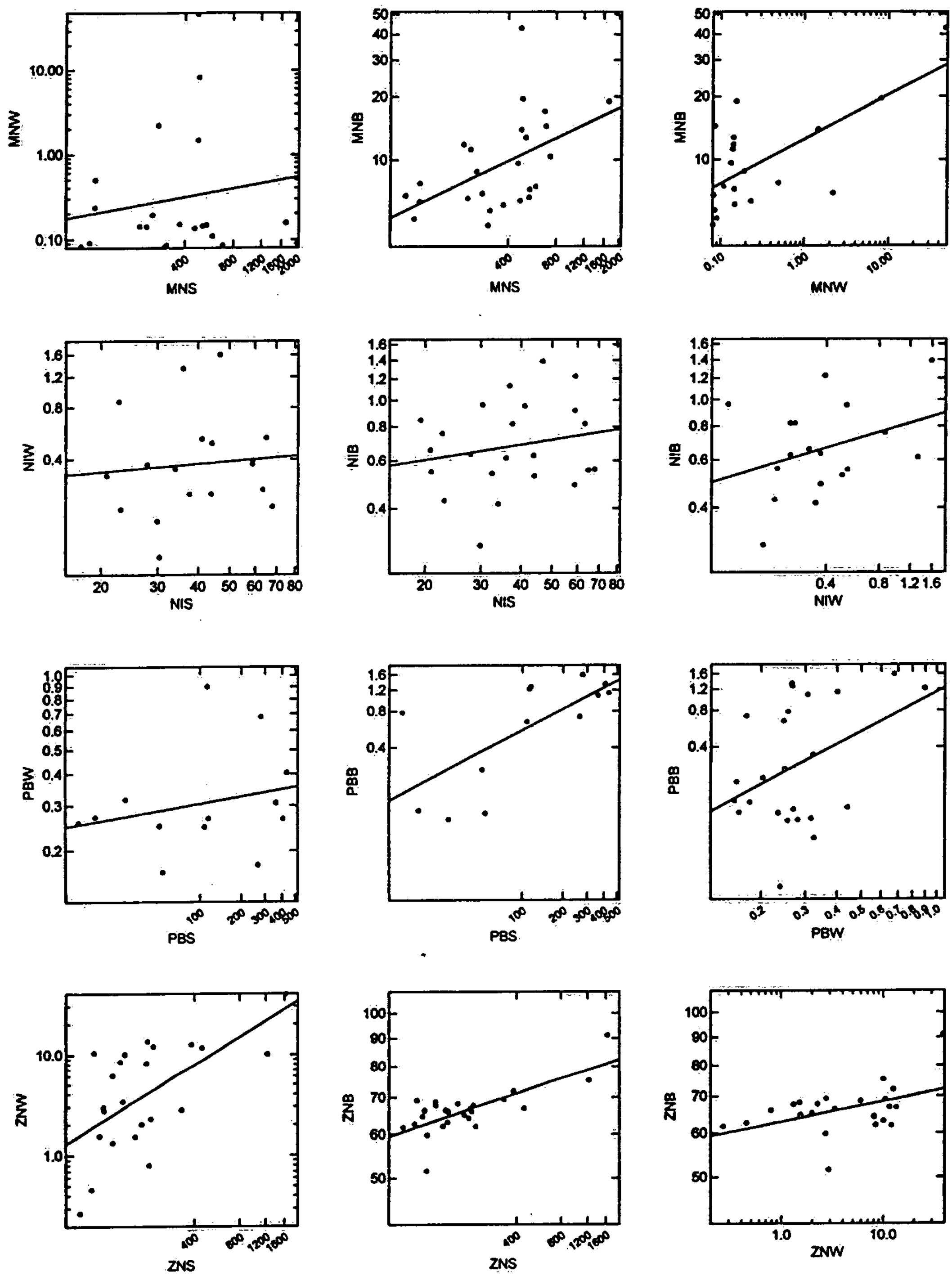

Figure 3. Concentrations of manganese (MN), nickel (NI), lead (PB) and zinc (ZN) measured in overlying water (W) or *Hyalella* (B) plotted against concentrations in sediment (S) or overlying water in toxicity tests with different sediment samples.

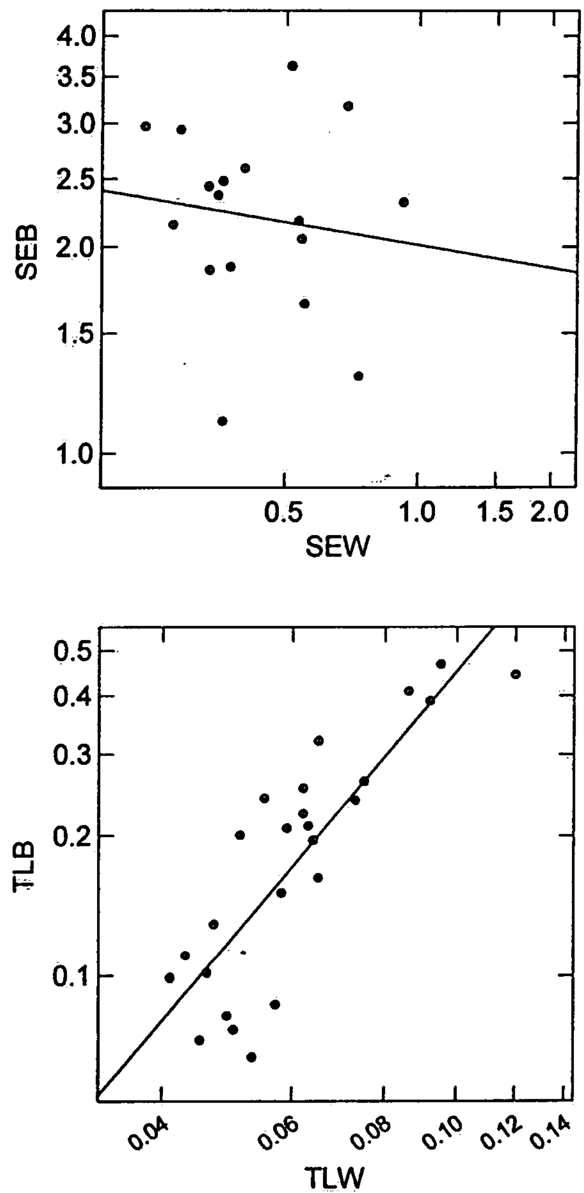

Figure 4. Concentrations of selenium (SE) and thallium (TL) measured in *Hyalella* (B) plotted against concentrations in overlying water in toxicity tests with different sediment samples.

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