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**Methods and Preliminary Results for  
Metals, Polycyclic Aromatic  
Hydrocarbons, Polychlorinated  
Biphenyls and Chlorinated  
Pesticides in Surface Sediments of  
the Mactaquac Headpond.**

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**DISCLAIMER**

Intended use and technical limitations of the report, “Revised 2014 Preliminary Methods and Sampling of Metals, Polycyclic Aromatic Hydrocarbons and Chlorinated Pesticides in Surface Sediments of the Mactaquac Headpond”. This report is a revision of the original 2015 report (2015-018) describing the shallow surface sediment chemistry and is intended to inform the ongoing development of the MAES sediment survey programme. The CRI doesn’t assume liability for any use of the included data or analyses outside the stated scope.

## Introduction

**The Report:** The following report focuses on the contaminants and nutrients found in surface sediments from the headpond above the Mactaquac Dam on the Saint John River, New Brunswick. The report is linked to the CRI Deliverables 1.3.1 - Assess Current Sediment Accumulation and Chemical Composition (a database), 1.3.1.1 – A sediment survey, and 1.3.1.2 A map of sediment accumulation and chemical composition. This report describes the surface sediment chemistry methods and preliminary analyses; data are available in the MAES Data Management System.

The main contaminants of interest are a suite of metals, chlorinated pesticides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), and nutrients (nitrogen (N) and phosphorous (P)) that have natural (metals, PAHs, nutrients) and human (metals, chlorinated pesticides, PAHs, PCBs, nutrients) sources in the watershed. Many of these contaminants have Canadian Council of Ministers of the Environment (CCME) sediment quality guidelines that can be used to assess whether any are above thresholds of concern; these are levels that may have effects on sediment-dwelling organisms. The report describes the results from preliminary sampling (November 2014) and analyses of surface sediments from 13 sites (12 in the headpond and 1 downstream of dam) for these contaminants. The goals of this sampling were two-fold: 1) to preliminarily assess whether there were any sites with higher contaminants than others (potential sites of concern/hotspots), and 2) to understand whether any contaminants exceeded the CCME sediment quality guideline values.

**Background:** Pesticides are produced and used to control pest insects, weeds, fungi, etc. in agriculture, forestry, and urban areas. Although current-use pesticides tend to be short-lived in the environment, some of the pesticides used in the 1950s through 1970s (i.e. chlorinated pesticides like DDT) in Canada are not easily degraded in the environment and thus will persist for years in soils at sites of application. These chlorinated pesticides enter rivers via overland runoff and erosion of soils and are deposited and accumulated in aquatic sediments. They also have the properties (high affinities for fats) to concentrate through aquatic food webs (biomagnification) to potentially toxic levels in upper-trophic-level species (fish, fish-eating birds). Although these pesticides have been banned from use in Canada for several decades, they are still found in the environment. They tend to be widely distributed because of their high historical use and ability to be transported long distances by wind and water currents from areas where they were used or are still used outside of Canada (i.e. DDT is still used for malaria control). Some of these chlorinated pesticides were used in New Brunswick. For example, the insecticide DDT was used from 1952-1966 to control spruce budworm; however, during the latter few years its use was restricted to areas away from streams because of concerns over its effects on fishes (Keachie and Côté, 1973).

PAHs are a complex group of aromatic contaminants that are naturally present in fossil fuels (oil, coal, gas). They are also created when fossil fuels, wood, and garbage are burned, and they are released by industries (e.g., coking ovens), incinerators, processing of asphalts, and households (i.e., wood stoves, barbecues). As a result, they have both natural and human sources to surface waters. These compounds are of concern as they are persistent in the environment, accumulating and persisting in soils and sediments, and some forms are believed to be carcinogenic (e.g. benzo[a]pyrene) and immunotoxic.

PCBs were once widely used in North America as coolants and lubricants for transformers and capacitors, and in hydraulic fluids, carbonless copy paper, plastics, caulking, paints, and other products because of their resistance to heat and chemical stability. Some local sources in the Saint John River valley include their historical use in paints at the Mactaquac hatchery and as evaporation retardants in pesticides (Keachie and Côté, 1973). There are 209 different PCBs that have between 1 and 10 chlorines. Their import, use, release and storage in Canada have been regulated since the late 1970s. Although they are all very persistent in the environment, PCBs with more chlorines tend to be more persistent in the environment and will concentrate more readily through food webs. There are concerns that these chemicals have accumulated to harmful levels in top predators of some aquatic food webs and affected their ability to reproduce.

Metals are present naturally in soils and rocks and they leach into aquatic environments through geological weathering and surface runoff. Therefore the natural composition of the land will influence what is found in nearby surface and ground waters. In New Brunswick there are natural sources of several metals (e.g., lead, aluminum, iron, nickel, copper, zinc, arsenic, mercury and cadmium) that influence what is found in waters and their sediments (Travers, 1976; Keachie and Côté, 1973; NB Department of Environment 2008; Kidd et al. 2011). However, aquatic environments can also become contaminated with metals through human activities such as mining (e.g., Lake George antimony mine; Travers, 1976), smelters (e.g., Belldune, NB; Bonham-Carter et al., 2005), other industrial discharges, agriculture (fertilizers, pesticides, composts, sludge and manures; Wuana and Okieimen, 2011), and municipal wastewater effluents. Agricultural runoff or direct discharges from industries and municipalities can result in the contamination of surface waters with metals. A review of water quality data from the Saint John River found similar concentrations of aluminum, iron, copper, zinc and lead across sites (2000-08), suggesting that the recent inputs of these metals are from natural sources (Kidd et al. 2011). Some historical water data (1970s-90s) were higher at a few sites in the river, suggesting some historical inputs of metals from human activities (Kidd et al. 2011). Many metals bind to particulates and therefore will accumulate in soils and sediments; they do not degrade and therefore will remain and cycle in the aquatic environment.

Mercury is a metal of particular concern because it is transformed in the aquatic environment to a form – methyl mercury – that will biomagnify through food webs and reach concentrations that may be toxic to the fish and fish-eating wildlife and humans. Anthropogenic sources of mercury in the Saint John River may include wastes from the following industries: coal mining (existed in Saint John River basin until 2009; Stone, 2010), other mining (Travers, 1976), agriculture, pulp and paper mills and food processing. Other sources include household waste and domestic sewage effluents (Health Canada, 2009). Mercury is also found naturally in volcanic rock deposits and deposition from forest fires (Health Canada, 2009). Until the early 1970s mercury was in fungicides and other agricultural products used in the Saint John River valley. Even though these pesticides are not used today, they may still be contaminating the river system through overland runoff (Travers, 1976).

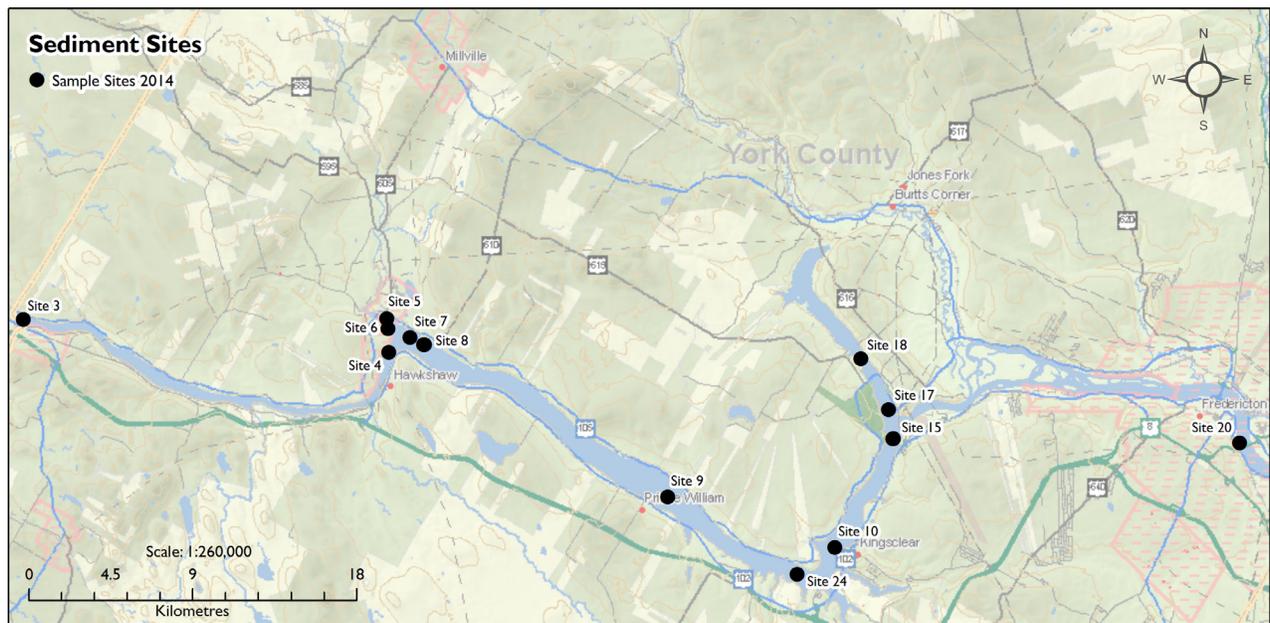
As for metals, nitrogen (N) and phosphorous (P) have both natural and human sources to surface waters. Municipal wastewater discharges and overland runoff of fertilizers from domestic and agricultural use are the main sources of these nutrients to freshwaters, and can lead to eutrophication (algal blooms) if excessive concentrations are released. A review of the historical

and recent concentrations of N and P in the river is reported in Kidd et al. (2011). This review indicated elevated aqueous concentrations of these nutrients in the river from the 1960s to 1980s, and more recent reductions in concentrations with the improved treatment of municipal wastewaters and industrial effluents (Kidd et al. 2011). Despite these improvements, water concentrations of these nutrients were still above “ideal” levels in reaches upstream from the Mactaquac dam in the early 2000s (Kidd et al. 2011).

## Methods

### Chemical Analysis of Sediments

Sediments were collected in the fall of 2014 from 12 sites in the headpond (Figure 1) and from one site downstream of the Fredericton wastewater treatment plant discharge. Sediments were difficult to collect from other sites downstream of the dam because of the nature of the substrate. We will continue to search for sites suitable for collections of downstream surface sediments in 2015. A dredge was used to collect sediments, and from that a clean core tube was used to obtain a subsample of the top 5 cm. This sample was placed in pre-cleaned glass jars, kept on ice in the field, and then frozen upon return to the lab until used for analysis. Sampling details are provided in *MAES Report Series 2015-003, METHODS PAPER: Reservoir Sediment Sampling*.



**Figure 1:** Locations of sites for sediment sampling in fall 2014.

Frozen sediment samples were thawed prior to analyses and macroinvertebrates or debris (e.g., pieces of wood or plastic) were manually removed. All moisture, metal, PAH, PCB, and pesticide analyses were done in the Environmental Chemistry Lab at the Canadian Rivers Institute (CRI), University of New Brunswick Saint John. Analyses of sediments for % organic carbon, total

Kjeldahl (TKN) and total phosphorous (TP) were done at RPC in Fredericton. When available, data were compared against CCME freshwater sediment quality guidelines (CCME, 1998-2001) or NOAA Screening Quick Reference Tables (SQuiRT) for freshwater sediment (Buchman, 2008) when a CCME guideline does not exist.

**Moisture Content**

An empty container was weighed (Sartorius CP323S balance) and then reweighed with the wet sample. The container was covered with a Kimwipe and then placed in the freeze dryer (Labconco FreezeZone12) for a minimum of three days. The container was re-weighed and the percent moistures were calculated as follows:

$$\% \text{ Moisture} = \left( 1 - \left[ \frac{(\text{weight of container} + \text{dry sample}) - \text{weight of container}}{(\text{weight of container} + \text{wet sample}) - \text{weight of container}} \right] \right) \times 100\%$$

After freeze drying, the sample was homogenized using a dried, acid washed glass mortar and pestle. Aliquots of the dried sample were removed for determination of metals/elements, total mercury (Hg), PAHs, chlorinated pesticides and PCBs. Sample requirements and methods for the different physicochemical analyses are summarized in Table 1 and described in detail in subsequent sections.

**Table 1:** Summary of physiochemical analysis of sediment samples by CRI, UNB Saint John.

Test	Mass of sample	Unit Reported	Method Used
Moisture	100 g wet	%	Gravimetric
Elements (Metals)	0.5 g dry	mg/kg dw	Digestion and ICP-OES quantification based on US EPA 3051, 200.7, and 6010C methods
Total Hg	0.03 g dry	µg/kg dw	DMA-80 based on US EPA 7473 method
PAHs		mg/kg dw	ASE extraction, GPC cleanup and GC/MS quantification based on US EPA 3545, 3640A and 8270C methods
PCBs/Chlorinated pesticides	≥20 g dry	µg/kg dw	ASE extraction, GPC and Florisil cleanup and GC/ECD quantification based on US EPA 3545, 3640A, 3620C, and 8082 methods

**Metals and Other Elements**

Sample digestion and analysis of metals followed a test method based on US Environmental Protection Agency (US EPA) standard testing protocols 3051A (US EPA, 2007a), 200.7 (US EPA, 1994a), and 6010C (US EPA, 1998a). A 0.5 g aliquot of homogenized, dried sample was digested using a microwave digestion (CEM Mars 5) and 10 mL of metal grade nitric acid (Fisher Scientific, Canada). After the digestion process, 40 mL of Milli-Q water was added along with a known amount of Yttrium (Y) (SCP Science, QC) as an internal standard. Samples were filtered using Millex syringe filters (0.45 µm) and disposable syringes with polyethylene barrels and

polypropylene plungers (Fisher Scientific) into polypropylene test tubes (Fisher Scientific) for analysis. The following 22 elements were quantified using an inductively coupled plasma-optical emissions spectrophotometer (ICP-OES, iCAP 6500 Duo, Thermo Fisher Scientific) using an internal standard calibration method. Limit of quantification (LOQ; see below for explanation) and wavelengths used for quantification are listed in Table 2.

**Table 2:** Summary of LOQs and wavelengths for individual metals and other elements.

Element	Symbol	LOQ (mg/kg dw)	Wavelength ( $\lambda$ )
aluminum	Al	<2.7	396.1
arsenic	As	<1.6	189.0
cadmium	Cd	<0.08	214.4
chromium	Cr	<0.12	267.7
cobalt	Co	<0.15	228.6
copper	Cu	<0.19	324.7
iron	Fe	<0.64	259.9
lanthanum	La	<1.0	333.7
lead	Pb	<0.77	220.3
magnesium	Mg	<4.3	279.0
manganese	Mn	<0.03	257.6
nickel	Ni	<0.13	221.6
phosphorus	P	<0.67	178.2
rubidium	Rb	<0.86	780.0
selenium	Se	<1.1	196.0
silver	Ag	<0.22	328.0
strontium	Sr	<0.004	407.7
sulphur	S	<1.1	180.7
thallium	Tl	<0.55	190.8
uranium	U	<6.4	409.0
vanadium	V	<0.15	309.3
zinc	Zn	<0.04	202.5, 206.2, 213.8

Quality assurance/quality control (QA/QC) procedures included the following: each batch of 11 samples included a method blank (MB), certified reference material (CRM) [National Institute of Standards & Technology (NIST) Standard Reference Material (SRM) 2702 Inorganics in Marine Sediment], and sample duplicate. The MB consisted of Ottawa sand (Fisher Scientific, Ottawa, ON) which was run through the entire testing process. The target MB value was equal to or less than the LOQ. For instances where the MB was greater than the LOQ, the LOQ was increased to the level found in the blank. CRM and calibration check results were reported as percent recovery based on the certificate's certified and calculated target values. The duplicate samples were reported as relative percent differences. Instrument blanks and calibration checks were routinely done throughout the analysis. All standards (SCP Science, QC), calibration checks (SCP Science, QC), and reference materials were certified with a certificate of analysis. Instrument detection limits (IDL) were determined by running 20 repeats of a blank ( $IDL = \text{average}_{\text{blanks}} + 3 \times$

$SD_{\text{blanks}}$ ; based on US EPA 200.7). The LOQs were calculated as 5 times the IDL (Montaser and Golightly, 1992; see Table 2 for the LOQs).

### **Total Mercury**

Sample preparation and analysis of THg followed a test method based on US EPA standard testing protocol 7473 (US EPA, 1998b). A 0.03 g aliquot of homogenized, dried sample was run on a direct mercury analyzer (Milestone DMA-80). Quality assurance/quality control procedures included the following. Each batch of 10 samples included an instrument blank, MB, CRM (NIST SRM 2702 Inorganics in Marine Sediment), calibration standard checks, and sample duplicate. The QA/QC procedures for the MB, CRMs, standard checks and sample duplicates followed those used for the analysis of other metals. All standards (Ultra Scientific, N. Kingstown, RI, USA), calibration checks, and reference materials were certified with a certificate of analysis. The limit of detection (LOD) was determined by averaging all the method blanks run in the batch and adding it to 3 times the SD of the method blanks for that batch (LOD 0.83  $\mu\text{g}/\text{kg dw THg}$ ).

### **Sample Preparation for PAHs, PCBs and Chlorinated Pesticides**

Sample extraction and clean up followed a test method based on US EPA standard testing protocols 3545A (US EPA, 2007b), 3640A (US EPA, 1994b) and 3660B (US EPA, 1996d). A minimum of 20 g of homogenized, dried sample was spiked with a surrogate solution containing: nitrobenzene-d5, 2-fluorobiphenyl and p-terphenyl (certified standards SPEX Certiprep, Metuchen, NJ, USA) for the PAH method. It was then spiked with a second surrogate solution containing: 4-chloro-3nitrobenzotrifluoride, m-tetrachloro-m-xylene, PCB 30, PCB 103, PCB 198 and PCB 204 for the PCBs and chlorinated pesticides method (Accustandard, New Haven, CT, USA) and was then extracted using an Accelerated Solvent Extractor (Dionex ASE 300) with distilled in glass (DIG) grade 50:50 dichloromethane (DCM):acetone (Optima grade, Fisher Scientific, Ottawa, ON). Extracted samples were concentrated to 6 mL of 50:50 DCM:hexane using a Büchi rotavapor (R-200) and nitrogen evaporator (N-EVAP™112, Organomation Associates Inc.). Samples were run through a gel permeation column (J2 Scientific Automated Gel Permeation System) using 50:50 DCM:hexane to remove heavier contaminants that may interfere with the quantification of PAHs and chlorinated pesticides. The sample was then concentrated into 2.0 mL of hexane (Optima grade, Fisher Scientific) using the same techniques mentioned above. The sample was split: 1 mL was run for PAHs and 1 mL was run for PCBs and chlorinated pesticides.

### **Polycyclic Aromatic Hydrocarbons**

Analysis of PAHs followed a test method based on US EPA standard testing protocol 8270C (US EPA, 1996c). The fraction was solvent transferred to isooctane using a Büchi rotavapor (R-200) and nitrogen evaporator (N-EVAP™112, Organomation Associates Inc.) to a final volume of 1 mL. A known standardized amount of internal standard solution (naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12 and perylene-d12) was added to each sample prior to quantification. The concentrated extracts were run on a gas chromatograph-mass spectrometer (Agilent 6890/5975B GC-MS) and quantified using an internal standard

calibration and single ion monitoring mode. Analyses included quantification of 16 PAHs using the quantification ions listed in Table 3.

Quality assurance/quality control procedures included the following. Each batch of 8 samples included a MB, MS and sample duplicate. With every 21 samples a CRM (NIST SRM 1941b Organics in Marine Sediments) was included. Individual sample recoveries were determined by adding a known amount of three surrogates (nitrobenzene-d5, 2-fluorobiphenyl, p-terphenyl; certified standards SPEX Certiprep, Metuchen, NJ, USA) to each sample prior to extraction, and analyzing MBs, MSs, CRMs, and sample duplicates. The surrogates were reported as percent recovery based on the calculated target concentration. In addition, instrument performance was verified by running tune check standards and calibration check standards for both the target and surrogate compounds. All calibration standards, calibration check standards, tune standards, surrogates standards, and internal standards were certified (SPEX Certiprep, Metuchen, NJ, USA). Method detection limits (MDLs) for individual PAHs were determined by running 8 low level spike samples (5x higher than the expected MDL) through the entire process. The t value (n = 8, 95%) was multiplied by the standard deviation of the 8 runs to determine the MDL for each PAH (<0.01 mg/kg dw). The MDL for total PAHs was determined by taking the square root of the sum of squares of the individual PAH MDLs (MDL <0.04 mg/kg). For the purposes of this report, data are reported as individual PAHs and individual PAHs were also summed and reported as total PAHs.

**Table 3:** Quantification ion used for quantifying the individual PAHs.

PAH	Quantification Ion
acenaphthene	153
acenaphthylene	152
anthracene	178
benzo[a]anthracene	228
benzo[a]pyrene	252
benzo[b]fluoranthene	252
benzo[k]fluoranthene	252
benzo[g,h,i]perylene	276
dibenzo[a,h]anthracene	278
chrysene	228
fluoranthene	202
fluorine	166
indeno[1,2,3-cd]pyrene	276
naphthalene	128
phenanthrene	178
pyrene	202

### **Polychlorinated Biphenyls and Chlorinated Pesticides**

Analysis of PCBs and chlorinated pesticides followed a test method based on US EPA standard testing protocol 8082 (US EPA, 1996b). The 1 mL of hexane extract from above was eluted through a manually packed 1.2% deactivated (using HPLC grade water; Fisher Scientific) Florisil column (Fisher Scientific). Three fractions were collected from the Florisil column (US EPA, 1996a) using hexane to elute fraction A, 15:85 DCM:hexane to elute fraction B and 50:50 DCM:hexane to elute fraction C. Fractions A, B, and C were each concentrated into 1.0 mL of isoctane. Each fraction was spiked with a known amount of internal standard containing pentachloronitrobenzene and PCB 209 (AccuStandard, New Haven, CT, USA). The three fractions were run on a gas chromatograph-electron capture detector (Agilent 6890 GC-ECD) and quantified using an internal standard calibration on a DB-5, 60m, 0.25 mm, 0.25  $\mu$ m Agilent J&W column. PCBs were verified using a GC-ECD and DB-XLB, 60m 0.25mm, 0.25 $\mu$ m Agilent J&W column.

Quality assurance/quality control procedures included the following. Each batch of 8 samples included a MB, MS and sample duplicate. Every 21 samples included a CRM (NIST SRM 1941b Organics in Marine Sediments). The QA/QC procedures for the MBs, CRMs, standard checks and sample duplicates followed those listed for PAH analysis. MDLs were determined by running 8 low level spike samples (5x higher than the expected MDL) through the entire process. The t value ( $n = 8$ , 95%) was multiplied by the standard deviation of the 8 runs to determine the MDL for each PCB congener and chlorinated pesticide. The reporting limit (RL) for PCBs and chlorinated pesticides was based on the amount of the lowest calibration standard and determined to be 0.12  $\mu$ g/kg dw for each pesticide. When two or three congeners co-eluted, the RL was 0.24 or 0.36  $\mu$ g/kg dw, respectively. The MDLs were equal to or less than the RL. All calibration standards, calibration check standards, surrogates standards, and internal standards were certified (AccuStandard, New Haven, CT, USA). Tune standards were also certified (SPEX Certiprep, Metuchen, NJ, USA).

For this study, 62 individual PCB congeners were examined and those that were above detection limits were summed and reported as total PCBs ( $\mu$ g/kg dw; see list in Table 4 for IUPAC congener numbers). Analyses included quantification of the following 26 chlorinated pesticides (see Table 4). Chlorinated pesticides were reported as individual pesticides (hexachlorobenzene, aldrin, dieldrin and methoxychlor) and  $\Sigma$ DDTs [sum of p,p'-dichlorodiphenyltrichloroethane (p,p'-DDT), o,p'-dichlorodiphenyltrichloroethane (o,p'-DDT), p,p'-dichlorodiphenyldichloroethylene (p,p'-DDE), o,p'-dichlorodiphenyldichloroethylene (o,p'-DDT), p,p'-dichlorodiphenyldichloroethane (p,p'-DDD) and o,p'-dichlorodiphenyl dichloroethane (o,p'-DDE)],  $\Sigma$ chlordane (sum of  $\alpha$ -chlordane and  $\gamma$ -chlordane),  $\Sigma$ endrin (sum of endrin, endrin aldehyde and endrin ketone),  $\Sigma$ HCH (sum of  $\alpha$ -hexachlorocyclohexane,  $\beta$ -hexachlorocyclohexane,  $\delta$ -hexachlorocyclohexane and  $\gamma$ -hexachlorocyclohexane),  $\Sigma$ heptachlor (sum of heptachlor epoxide (isomer B) and heptachlor),  $\Sigma$ nonachlor (sum of cis-nonachlor and trans-nonachlor) and  $\Sigma$ endosulfan (sum of endosulfan I, endosulfan II and endosulfan sulfate). Individual chlorinated pesticides (dieldrin, heptachlor epoxide and endrin) and  $\Sigma$ DDD (sum of p,p'-DDD and o,p'-DDD),  $\Sigma$ DDE (sum of p,p'-DDE and o,p'-DDE),  $\Sigma$ DDT (sum of p,p'-DDT and o,p'-DDT) and  $\Sigma$ chlordane (sum of  $\alpha$ -chlordane and  $\gamma$ -chlordane) were reported for comparison to the CCME or SQUIRT (US NOAA) sediment quality guidelines (see Table 8).  $\Sigma$ TDDTs is reported as the total sum of  $\Sigma$ DDD,  $\Sigma$ DDE and  $\Sigma$ DDT.

**Table 4:** Individual PCBs and chlorinated pesticides that were examined in surface sediments.

PCB 8/5	PCB 83	$\alpha$ -HCH
PCB 8	PCB 97	hexachlorobenzene
PCB17/15	PCB 77/110	$\beta$ -HCH
PCB 16/32	PCB 85	$\gamma$ -HCH
PCB 28	PCB 151	$\delta$ -HCH
PCB 31	PCB 135/144	heptachlor
PCB 33	PCB 118	aldrin
PCB 53	PCB 123/149	heptachlor epoxide (Isomer B)
PCB 22	PCB 187	$\gamma$ -chlordane
PCB 52	PCB 132/105	o,p'-DDE
PCB 49	PCB 153	endosulfan I
PCB 47	PCB 146PCB 163/138	$\alpha$ -chlordane
PCB 48	PCB 126/178	trans-Nonachlor
PCB 44	PCB 174	p,p'-DDE
PCB 71/64/41	PCB 128/167	dieldrin
PCB 74	PCB 180	o,p'-DDD
PCB 66/95	PCB 199	cis-Nonachlor
PCB 70	PCB 170	endrin
PCB 76	PCB 201	endosulfan II
PCB 91	PCB 194	p,p'-DDD
PCB 81/87	PCB 206	o,p'-DDT
PCB 56/60/92		endrin aldehyde
PCB 101		p,p'-DDT
PCB 99		endosulfan sulfate
		endrin ketone
		methoxychlor

**Table 5:** Metals and other elements in sediment samples collected in November, 2014. Bolded numbers are above the CCME ISQG or SQuiRT TEL. *Bolded and italicized numbers are above the CCME or SQuiRT PEL.* Results highlighted in red are above the CCME Soil Quality Guidelines for agricultural use.

Site #	n	THg	Al	As	Cd	Co	Cr	Cu	Fe	La	Mg
Units	-	µg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw
ISQG or TEL	-	170 <sup>a</sup>	-	5.90 <sup>a</sup>	0.60 <sup>a</sup>	-	37.3 <sup>a</sup>	35.7 <sup>a</sup>	-	-	-
PEL	-	486 <sup>a</sup>	-	17.0 <sup>a</sup>	3.50 <sup>a</sup>	-	90.0 <sup>a</sup>	197 <sup>a</sup>	-	-	-
SQG	-	6600 <sup>a</sup>	-	12 <sup>a</sup>	1.4 <sup>a</sup>	40 <sup>a</sup>	64 <sup>a</sup>	63 <sup>a</sup>	-	-	-
Site 17	1	114	35700	<b>17.3</b>	0.23	18.3	<b>61.1</b>	24.0	42600	30.5	9900
Site 18	1	120	39100	<b>22.4</b>	0.20	17.9	<b>56.8</b>	21.9	45200	29.2	8310
Site 15	5	97.7 ± 3.8	33700 ± 827	<b>13.4 ± 1.8</b>	0.18 ± 0.06	17.1 ± 0.6	<b>57.3 ± 1.3</b>	22.5 ± 0.9	37600 ± 1750	30.1 ± 1.0	9420 ± 323
Site 9	1	90.1	32100	<b>14.1</b>	0.17	16.4	<b>54.3</b>	23.0	34600	28.8	8920
Site 24	1	90.3	32400	<b>14.7</b>	0.19	16.3	<b>55.7</b>	21.7	36300	29.8	9140
Site 10	1	116	35200	<b>13.9</b>	0.24	18.6	<b>60.0</b>	25.2	37000	31.5	9420
Site 20	1	15.6	16500	5.64	0.10	9.31	<b>29.7</b>	6.78	21600	14.7	6200
Site 4	1	58.8	25100	<b>7.98</b>	0.12	12.9	<b>45.9</b>	16.4	28300	27.7	7610
Site 8	5	73.1 ± 7.9	26800 ± 2660	<b>9.53 ± 0.91</b>	0.10 ± 0.07	13.4 ± 0.8	<b>47.7 ± 3.7</b>	19.6 ± 1.3	27800 ± 1819	27.1 ± 1.7	7570 ± 492
Site 7	1	52.5	24800	<b>6.87</b>	0.14	13.0	<b>47.5</b>	15.7	25400	24.3	7410
Site 6	1	94.6	36900	<b>14.6</b>	0.11	17.6	<b>60.0</b>	23.1	34700	31.5	8880
Site 5	1	81.7	30900	<b>10.3</b>	0.14	15.9	<b>53.4</b>	21.9	31400	28.6	8260
Site 3	1	30.6	20800	5.75	0.08	11.5	<b>40.4</b>	11.7	24000	22.4	7320

CCME ISQG is Interim Sediment Quality Guideline (ISQG). PEL is Probable Effects Level (CCME or NOAA SQuiRT). TEL is Threshold Effects Level (NOAA SQuiRT). SQG is the Soil Quality Guideline for agricultural use. Samples and guidelines are in mg/kg-dry weight except for THg which is ug/kg-dry weight. Average ± Standard Deviation is reported for sites with n=5. When only 1 or 2 sample were > DLs, data are reported as a range. <sup>a</sup>CCME Guideline (CCME, 1998-2001; CCME, 1991-2009). <sup>b</sup>SQuiRT Guideline (Buchman, 2008).

**Table 5 (continued):** Metals and other elements in sediment samples collected in November, 2014. Bolded numbers are above the CCME ISQG or SQuiRT TEL. *Bolded and italicized numbers are above the CCME or SQuiRT PEL values.* Results highlighted in red are above the CCME Soil Quality Guidelines for agricultural use.

Site #	n	Mn	Ni	P	Pb	Rb	S	Sr	Tl	V	Zn
Units	-	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw	mg/kg dw
ISQG or TEL	-	-	18.0 <sup>b</sup>	-	35.0 <sup>a</sup>	-	-	-	-	-	123 <sup>b</sup>
PEL	-	-	36.0 <sup>b</sup>	-	91.3 <sup>a</sup>	-	-	-	-	-	315 <sup>b</sup>
SQG	-	-	50 <sup>a</sup>	-	70 <sup>a</sup>	-	-	-	1 <sup>a</sup>	130 <sup>a</sup>	200 <sup>a</sup>
Site 17	1	2050	<b>51.6</b>	1560	20.1	48.2	748	39.3	<b>1.34</b>	83.4	114
Site 18	1	1400	<b>43.3</b>	1140	19.4	63.7	616	33.6	<0.55	89.9	108
Site 15	5	2180 ± 372	<b>50.3 ± 1.8</b>	1320 ± 139	18.0 ± 0.7	45.0 ± 2.6	634 ± 98	38.0 ± 0.9	<b>1.30 ± 0.52</b>	78.3 ± 2.3	106 ± 4
Site 9	1	2020	<b>47.5</b>	1160	16.8	44.4	712	39.2	<0.66	75.6	103
Site 24	1	2630	<b>48.0</b>	1400	16.7	42.6	535	39.2	<b>1.84</b>	75.0	101
Site 10	1	4210	<b>52.5</b>	1160	20.4	48.4	575	45.7	<b>2.11</b>	82.4	115
Site 20	1	480	<b>27.8</b>	445	8.38	20.1	73.8	16.8	<0.55	46.4	58.2
Site 4	1	840	<b>39.3</b>	947	12.8	32.1	441	38.5	<0.55	60.2	79.8
Site 8	5	1180 ± 118	<b>40.1 ± 2.4</b>	999 ± 115	14.2 ± 0.9	38.2 ± 5.6	570 ± 26	42.6 ± 2.2	<0.66	64.0 ± 5.9	84.2 ± 5.5
Site 7	1	600	<b>38.8</b>	809	11.7	34.2	730	30.7	<0.66	60.9	83.7
Site 6	1	1340	<b>46.2</b>	1270	19.0	55.3	752	42.4	<0.66	86.5	116
Site 5	1	780	<b>43.1</b>	1140	18.1	42.7	908	39.2	<0.66	74.1	108
Site 3	1	520	<b>38.0</b>	523	10.2	27.0	139	24.7	<0.66	55.1	68.5

CCME ISQG is Interim Sediment Quality Guideline (ISQG). PEL is Probable Effects Level (CCME or NOAA SQuiRT). TEL is Threshold Effects Level (NOAA SQuiRT). SQG is the Soil Quality Guideline for agricultural use. Samples and guidelines are in mg/kg-dry weight except for THg which is ug/kg-dry weight. Average ± Standard Deviation is reported for sites with n=5. When only 1 or 2 sample were > DLs, data are reported as a range. <sup>a</sup>CCME Guideline (CCME, 1998-2001; CCME, 1991-2009). <sup>b</sup>SQuiRT Guideline (Buchman, 2008).

**Table 6:** Polycyclic aromatic hydrocarbons (mg/kg-dry weight) in sediment samples collected in November, 2014. Bolded numbers are above the CCME ISQG or NOAA SQuiRT TEL. *Bolded and italicized numbers are above the CCME or NOAA SQuiRT PEL.*

Site # or Guideline Value	n	Acenaphthylene	Fluorene	Phenanthrene	Anthracene	Fluoranthene	Pyrene	Benz(a)anthracene	Chrysene
ISQG or TEL	-	0.006 <sup>a</sup>	0.021 <sup>a</sup>	0.042 <sup>a</sup>	0.047 <sup>a</sup>	0.11 <sup>a</sup>	0.053 <sup>a</sup>	0.032 <sup>a</sup>	0.057 <sup>a</sup>
PEL	-	0.13 <sup>a</sup>	0.14 <sup>a</sup>	0.52 <sup>a</sup>	0.25 <sup>a</sup>	2.36 <sup>a</sup>	0.88 <sup>a</sup>	0.39 <sup>a</sup>	0.86 <sup>a</sup>
Site 17	1	<0.010	0.018	0.041	<0.01	0.081	<b>0.079</b>	<0.010	<b>0.067</b>
Site 18	1	<0.010	<0.010	0.025	0.025	0.041	0.041	<0.010	0.040
Site 15	5	<0.010	0.012 ± 0.004	<b>0.044 ± 0.017</b>	0.019 ± 0.010	0.069 ± 0.011	<b>0.067 ± 0.010</b>	<0.010	0.040 ± 0.010
Site 9	1	<0.010	0.012	<b>0.047</b>	0.023	0.097	<b>0.087</b>	<0.010	0.048
Site 24	1	<0.010	0.011	0.040	0.019	0.075	<b>0.069</b>	0.030	0.042
Site 10	1	<0.010	0.014	0.042	0.018	0.066	<b>0.061</b>	0.026	0.038
Site 20	1	<0.010	0.013	<b>0.091</b>	0.043	<b>0.174</b>	<b>0.141</b>	<b>0.094</b>	<b>0.086</b>
Site 4	1	<0.010	0.011	<b>0.044</b>	0.017	0.063	<b>0.060</b>	0.027	0.034
Site 8	5	<b>&lt;0.010-0.017</b>	0.011 ± 0.001	0.041 ± 0.015	0.020 ± 0.005	0.089 ± 0.016	<b>0.082 ± 0.017</b>	<b>0.037 ± 0.005</b>	0.050 ± 0.003
Site 7	1	<0.010	<0.010	0.035	0.012	0.069	<b>0.060</b>	0.027	0.038
Site 6	1	<0.010	<0.010	0.037	0.021	0.108	<b>0.100</b>	<b>0.041</b>	<b>0.064</b>
Site 5	1	<b>0.028</b>	<0.010	<b>0.043</b>	0.028	<b>0.141</b>	<b>0.137</b>	<b>0.062</b>	<b>0.090</b>
Site 3	1	<0.010	<0.010	0.032	0.012	0.053	0.047	0.022	0.030

CCME ISQG is Interim Sediment Quality Guideline. PEL is Probable Effects Level (CCME or SQuiRT Tables). TEL is Threshold Effects Level (SQuiRT Tables). Samples and guidelines are in mg/kg-dry weight. Average ± Standard Deviation is reported for sites with n=5. When only 1 or 2 sample were > DLs data are reported as a range. <sup>a</sup>CCME Guideline (CCME, 1998-2001; CCME, 1991-2009). <sup>b</sup>SQuiRT Guideline (Buchman, 2008).

**Table 6 (continued):** Polycyclic aromatic hydrocarbons (mg/kg-dry weight) in sediment collected in November, 2014. Bolded numbers are above the CCME ISQG or SQuiRT TEL. *Bolded and italicized numbers are above the CCME or SQuiRT PEL.*

Site #	n	Benzo(b)fluoranthene	Benzo(k) fluoranthene	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene	Benzo(g,h,i)perylene	TPAHs
ISQG or TEL	-			0.032 <sup>a</sup>			1.61 <sup>b</sup>
PEL	-			0.78 <sup>a</sup>			
Site 17	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.286
Site 18	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.173
Site 15	5	<0.010	<0.010	<0.010	<0.010	<0.010	0.250 ± 0.058
Site 9	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.313
Site 24	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.287
Site 10	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.260
Site 20	1	0.057	0.087	<b>0.083</b>	0.056	0.045	0.978
Site 4	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.256
Site 8	5	<0.010 - 0.073	<0.010	<0.010	<0.010 - 0.058	<0.010 - 0.053	0.367 ± 0.050
Site 7	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.240
Site 6	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.369
Site 5	1	0.107	0.069	<b>0.083</b>	0.088	<0.010	0.874
Site 3	1	<0.010	<0.010	<0.010	<0.010	<0.010	0.196

CCME ISQG is Interim Sediment Quality Guideline. PEL is Probable Effects Level (CCME or SQuiRT Tables). TEL is Threshold Effects Level (SQuiRT Tables). Samples and guidelines are in mg/kg-dry weight. Average ± Standard Deviation is reported for sites with n=5. When only 1 or 2 sample were > DLs data are reported as a range. <sup>a</sup>CCME Guideline (CCME, 1998-2001; CCME, 1991-2009). <sup>b</sup>SQuiRT Guideline (Buchman, 2008).

**Table 7:** Chlorinated pesticides ( $\mu\text{g}/\text{kg}$  dry weight) in sediment samples collected in November, 2014 (see Table 8 for guideline exceedances).

Site #	n	Hexachlorobenzene	Aldrin	Dieldrin	Methoxychlor	$\Sigma$ HCHs	$\Sigma$ Heptachlor	$\Sigma$ Chlordane	$\Sigma$ Nonachlor	$\Sigma$ TDDTs	$\Sigma$ Endosulfan	$\Sigma$ Endrin
Site 17	1	<DL	<DL	0.31	1.05	<DL	<DL	0.34	<DL	49.3	2.35	0.42
Site 18	1	<DL	<DL	0.18	1.62	<DL	<DL	<DL	<DL	21.2	0.30	<DL
Site 15	5	0.16 $\pm$ 0.07	<DL-0.13	0.30 $\pm$ 0.10	1.61 $\pm$ 0.33	0.11 $\pm$ 0.05	<DL	0.19 $\pm$ 0.15	<DL-0.13	43.9 $\pm$ 13.3	2.46 $\pm$ 0.95	0.80 $\pm$ 0.49
Site 9	1	0.24	0.17	0.39	1.94	0.16	<DL	0.42	0.20	77.6	4.73	1.71
Site 24	1	<DL	<DL	0.25	5.94	0.13	<DL	0.38	0.13	55.4	3.54	1.00
Site 10	1	<DL	<DL	0.38	6.00	<DL	<DL	0.59	0.22	65.5	4.02	1.65
Site 20	1	<DL	<DL	<DL	3.05	0.19	<DL	<DL	<DL	1.49	0.12	<DL
Site 4	1	<DL	0.17	0.19	3.80	<DL	<DL	0.25	0.14	24.7	1.20	0.39
Site 8	5	0.16 $\pm$ 0.10	0.21 $\pm$ 0.09	0.23 $\pm$ 0.06	3.32 $\pm$ 0.61	0.22 $\pm$ 0.13	<DL	0.58 $\pm$ 0.33	0.28 $\pm$ 0.13	48.4 $\pm$ 9.8	2.23 $\pm$ 0.59	0.68 $\pm$ 0.28
Site 7	1	0.20	0.29	0.19	2.83	0.34	0.31	0.68	0.36	42.5	2.16	0.70
Site 6	1	0.20	0.24	0.40	2.03	0.14	<DL	0.53	0.14	51.6	2.04	0.67
Site 5	1	<DL	<DL	0.40	1.73	0.14	<DL	0.17	<DL	53.6	1.64	0.68
Site 3	1	<DL	<DL	<DL	1.36	<DL	<DL	<DL	<DL	9.57	0.40	<DL

Average  $\pm$  Standard Deviation is reported for sites with n=5. When only 1 or 2 sample were > DLs, data are reported as a range.

## RESULTS

### Metals and Other Elements

All of the metals and other elements analyzed in sediments are shown in Table 5. Of the elements consistently measured across sites, the ones at the highest and lowest (above DLs) concentrations were iron (21,600 to 42,600 mg/kg dw) and total mercury (15.6 to 120 µg/kg dw), respectively. Arsenic, cadmium, cobalt, chromium, copper, iron, lanthanum, magnesium, manganese, nickel, phosphorous, lead, rubidium, sulphur, strontium, vanadium and zinc were also measured at all sites. Concentrations of these elements in the headpond varied up to 8 fold among sites and tended to be highest at sites 6, 10, 17, and 18 and lowest at sites 3, 4, and 7 (using average ranks of all metals). Thallium and selenium were above detection limits at 4 and 1 sites (1.31 mg/kg dw, n=1, site 10), respectively. The last two elements, silver and uranium were < DLs for all sites and are not listed in Table 5.

Of those elements with sediment quality guidelines, nickel was consistently above the PEL (NOAA SQiRT guideline) at all sites except site 20 and above the soil quality guideline (CCME SQG for agricultural use) at 3 of the 13 sites (Table 5). Arsenic was above the PEL for 2 sites, the ISQG for 9 sites, and the SQG for 7 sites. Similarly, chromium was above the ISQG for all sites.

Few data exist on sediment metal concentrations from previous studies. Natural background concentrations of mercury were suggested to be in the 50-200 ppb (µg/kg-dw) range for freshwater sediment in the Saint John River with no industrial influences (Travers, I, 1976). Travers (1976) suggests that from Edmundston to Woodstock, NB, the background Hg should be from 50-60 ppb based on the composition of the bedrock. Fredericton to Gagetown was predicted to have a higher background (100-200 ppb) of Hg present due to the differences in geological composition in this area. These concentrations are similar to those measured in this study.

### Polycyclic Aromatic Hydrocarbons (PAHs)

Of the 16 individual PAHs measured in sediments, naphthalene, acenaphthene and dibenz(a)anthracene were below detection limits for all sites and are not listed in Table 6. The remaining 13 were found in sediments from at least two sites. More specifically, 6 PAHs (acenaphthylene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, and benzo(g,h,i)perylene) were found at 2 or 3 sites (sites 5, 8 and 20). Fluorene and benz(a)anthracene were detectable at 8 and 9 sites, respectively. Phenanthrene, fluoranthene, anthracene (except site 17), pyrene and chrysene were found at all sites, and often 2X higher at site 20 than the other locations. The PAHs with the highest individual concentrations were fluoranthene (0.041 to 0.174 mg/kg dw) and pyrene (means of 0.041 to 0.141 mg/kg dw; Table 6). Concentrations of total PAHs in the headpond ranged from 0.173 mg/kg dw at site 18 to 0.874 mg/kg dw at site 5. No data for other sites in the Saint John River basin have been found to date. As such, it is not known whether these concentrations are typical for the region or indicative of localized contamination.

Of the PAHs with sediment quality guidelines, exceedances were found for benzo(a)pyrene, fluoranthene and acenaphthylene at two sites, benz(a)anthracene and chrysene at 4 sites (3 that were similar), and phenanthrene at 5 sites (above ISQGs). Finally, pyrene exceeded ISQGs at all

sites but two. Total PAHs and 6 other individual PAHs did not exceed TEL or ISQGs at any of the sites.

### **Polychlorinated Biphenyls**

Concentrations of individual PCBs in the Saint John River sediments were very low, and most were below detection limits. Of the 62 PCBs examined, only 1 to 5 were found in sediment samples from 7 of 13 sites at concentrations above the detection limits. These PCBs were 52, 151, 153, 180 and 206. Total PCBs across sites ranged from not detected to 0.32 µg/kg-dry weight and all concentrations were well below the CCME ISQG (Table 8).

### **Chlorinated Pesticides**

Of the individual chlorinated pesticides or breakdown products analyzed in this study, several were found below the detection limits and include endrin aldehyde, heptachlor epoxide and all isomers of HCH except for  $\gamma$ -HCH (lindane). Of the others,  $\Sigma$ DDT (0.38 to 34.8 µg/kg dw; Table 8) were found at the highest concentrations across all sites. Dieldrin (0.18 to 0.40 µg/kg dw; Table 8) and  $\gamma$ -HCH at the lowest concentrations (0.13 to 0.26 µg/kg dw; Table 8). Although chlorinated pesticides were commonly found across sites, results for some locations (sites 3, 18 and 20) were below detection limits for endrin,  $\Sigma$ chlordane, and  $\gamma$ -HCH (except site 20). Also, hexachlorobenzene was found at 5 sites, aldrin at 6 sites,  $\Sigma$ nonachlor at 8 sites, and  $\Sigma$ heptachlor at 1 site (Table 7). When results for all of the chlorinated pesticides were ranked for the headpond, sites 7, 9 and 10 had the highest concentrations whereas sites 3, 4, 17 and 18 had the lowest concentrations.

Table 8 shows the pesticides that were found above the ISQG or PEL levels.  $\Sigma$ DDD was above ISQG and PEL values for 6 and 7 sites, respectively, whereas  $\Sigma$ DDT and  $\Sigma$ DDE were above PELs for all but 2 sites. Several of the chlorinated pesticides did not exceed sediment quality guidelines ( $\gamma$ -HCH, heptachlor epoxide, dieldrin, endrin and  $\Sigma$ chlordane).

In 1972 sediment from the headponds of the Saint John River were found to have concentrations of p,p'-DDE and p,p'-DDT ranging from 36-90 ppb\* (\*dry or wet weight not specified; dry weight was assumed given report's reference to DDT concentrations in Dimond et al. 1971) in locations ranging from Grand Falls to Mactaquac, NB (Keachie and Côté, 1973). These concentrations are similar to or higher than those measured in the current study. Other pesticides were measured by Keachie and Côté (1973) but found to be less than the 1972 detection limit of 5 ppb. These were:  $\alpha$ -HCH, lindane, heptachlor, heptachlor epoxide, dieldrin, aldrin, endosulfan isomers,  $\alpha$ -chlordane,  $\gamma$ -chlordane, methoxychlor, p,p'-DDD and o,p'-DDT. In the current study, pp'-DDD and methoxychlor are the only pesticides that are above this historical detection limit of 5 ppb; all others are below this value or below the current detection limits.

### **Organic Carbon, Nitrogen and Phosphorous**

Percent organic carbon in the surface sediments from the Mactaquac headpond ranged from 0.8 to 3.9% (Table 9). Below the dam at the Fredericton sewage outfall, the sediments contained 0.2% organic carbon. Total P concentrations varied 3 fold among sites in the headpond, ranging from 530 to 1490 mg/kg dw. Similarly, TKN also varied by up to 3.5 fold among sites (930 to

3760 mg/kg dw; Table 9). Sites that were high in P tended to be high in N, suggesting similar sources of these nutrients to the sediments.

## Summary

Metals, PAHs, PCBs, and chlorinated pesticides were found in surface sediments from the Mactaquac headpond. Of these, some exceeded the CCME or NOAA sediment quality guidelines as follows:

- Nickel was consistently above the PEL (USA NOAA) across all sites, except at site 20 (which was above the ISQG)
- Arsenic was above the PEL (CCME) for 2 sites and the ISQG for 9 sites
- Chromium was above the ISQG (CCME) for all sites
- Benzo(a)pyrene, fluoranthene and acenaphthylene exceeded ISQGs (CCME) at two sites
- Benz(a)anthracene and chrysene exceeded the ISQGs (CCME) at 4 sites
- Phenanthrene exceeded the ISQG (CCME) at 5 sites
- Pyrene exceeded the ISQG (CCME) at all sites but two
- ΣDDD was above ISQG and PEL (CCME) values for 6 and 7 sites, respectively
- ΣDDT and ΣDDE were above PELs (CCME) for all but 2 sites
- ΣPCB did not exceed any sediment quality guidelines

The results suggest that sediment contaminant concentrations were similar at most sites in the headpond, with the lowest concentrations found at the site furthest upstream (site 3). Some of the spatial variability in contaminants was likely due to the differences in sediment composition as site 3 tended to have lower organic carbon than other sites in the headpond. Concentrations of N and P in sediments in the headpond also varied among sites, with site 3 having the lowest nutrient concentrations.

There are no clear indications of any sediment chemistry “hotspots” related to human activities in the headpond based on the interim sampling. We will be expanding the spatial coverage to verify these observations, i.e., to better understand both the human and the natural geological influences on sediment chemistry in the river and in the accumulated sediments of the headpond.

**Next Steps:** Sampling in 2015 and 2016 will be designed based on these findings. There are three objectives: 1) increase the spatial coverage; 2) include additional reference sites outside the influence of the headpond; and 3) collect sediment cores to understand the historical profile of metals, PAHs, PCBs and chlorinated pesticides. Once complete, these results will provide a comprehensive assessment of the current and historical contamination of the headpond sediments, of the influence of human versus natural activities on the levels of metals and PAHs, and of any sites of concern above the dam.

**Table 8:** Chlorinated pesticides and total polychlorinated biphenyls ( $\mu\text{g}/\text{kg}$ -dry weight) with CCME Guidelines in sediment samples collected in November, 2014. Bolded numbers are above the CCME ISQG. *Bolded and italicized numbers are above the CCME PEL values.*

Site #	n	$\gamma$ -HCH (Lindane)	Heptachlor epoxide (Isomer B)	Dieldrin	Endrin	$\Sigma$ chlordane ( $\alpha + \gamma$ )	$\Sigma$ DDE	$\Sigma$ DDD	$\Sigma$ DDT	Total PCBs
ISQG	-	0.94	0.6	2.85	2.67	4.5	1.42	3.54	1.19	34.1
PEL	-	1.38	2.74	6.67	62.4	8.87	6.75	8.51	4.77	277
Site 17	1	<DL	<DL	0.31	0.42	0.34	<b>22.3</b>	<b>8.26</b>	<b>18.8</b>	nd
Site 18	1	<DL	<DL	0.18	<DL	<DL	<b>11.6</b>	<b>5.37</b>	<b>4.23</b>	0.22
Site 15	5	<DL $\pm$ 0.01	<DL	0.30 $\pm$ 0.10	0.58 $\pm$ 0.41	0.19 $\pm$ 0.15	<b>15.7 <math>\pm</math> 9.39</b>	<b>9.23 <math>\pm</math> 1.69</b>	<b>18.9 <math>\pm</math> 6.6</b>	nd-0.18
Site 9	1	0.16	<DL	0.39	1.31	0.42	<b>26.5</b>	<b>16.3</b>	<b>34.8</b>	0.15
Site 24	1	0.13	<DL	0.25	1.00	0.38	<b>23.5</b>	<b>13.6</b>	<b>18.3</b>	0.13
Site 10	1	<DL	<DL	0.38	1.20	0.59	<b>29.5</b>	<b>14.9</b>	<b>21.0</b>	nd
Site 20	1	0.19	<DL	<DL	<DL	<DL	0.24	0.88	0.38	0.20
Site 4	1	<DL	<DL	0.19	0.39	0.25	<b>12.2</b>	<b>6.89</b>	<b>5.63</b>	nd
Site 8	5	0.23 $\pm$ 0.12	<DL	0.23 $\pm$ 0.06	0.64 $\pm$ 0.22	0.58 $\pm$ 0.33	<b>20.8 <math>\pm</math> 3.9</b>	<b>9.83 <math>\pm</math> 1.81</b>	<b>17.8 <math>\pm</math> 4.4</b>	0.35 $\pm$ 0.32
Site 7	1	0.13	<DL	0.19	0.70	0.68	<b>14.1</b>	<b>8.17</b>	<b>20.3</b>	nd
Site 6	1	0.14	<DL	0.40	0.67	0.53	<b>23.4</b>	<b>11.6</b>	<b>16.6</b>	0.32
Site 5	1	0.14	<DL	0.40	0.68	0.17	<b>20.2</b>	<b>9.75</b>	<b>23.6</b>	nd
Site 3	1	<DL	<DL	<DL	<DL	<DL	<b>2.95</b>	1.14	<b>5.48</b>	nd

CCME ISQG is Interim Sediment Quality Guidelines (CCME, 1998-2001). PEL is Probable Effects Level (CCME). Samples and guidelines are in  $\mu\text{g}/\text{kg}$ -dry weight. Average  $\pm$  Standard Deviation are reported for sites with n=5 and nd means not detected.

**Table 9:** Organic carbon (%), Kjeldahl nitrogen (mg/kg-dw) and phosphorus (mg/kg-dw) in sediment samples collected in November, 2014.

<b>Site #</b>	<b>n</b>	<b>Organic Carbon (%)</b>	<b>Kjeldahl Nitrogen (mg/kg-dw)</b>	<b>Phosphorus (mg/kg-dw)</b>
Site 17	1	3.0	3350	1490
Site 18	1	3.9	3690	1120
Site 15	5	3.0 ± 0.5	3214 ± 587	1343 ± 127
Site 9	1	2.9	2990	1270
Site 24	1	2.5	2580	1310
Site 10	1	2.8	3350	1280
Site 20	1	0.2	340	430
Site 4	1	2.3	2190	870
Site 8	5	2.8 ± 1.3	2586 ± 1168	937 ± 253
Site 7	1	2.2	2200	860
Site 6	1	3.7	3760	1300
Site 5	1	3.6	3030	1310
Site 3	1	0.8	930	530

All organic carbon, Kjeldahl nitrogen and phosphorus reported in Table 9 were tested at RPC, Fredericton, NB

## REFERENCES

- Bonham-Carter, G.F. 2005. Introduction to the GSC MITE Point Sources project; in *Metals in the Environment Around Smelters and Rouyn-Noranda, Quebec and Belledune, New Brunswick: Results and Conclusions of the GSC MITE Point Sources Project*, (ed.) G.F. Bonham-Carter; Geological Survey of Canada, Bulletin 584, 14p.
- Buchman, M.F. 2008. NOAA Screening Quick Reference Tables, NOAA OR&R Report 08-1, Seattle WA, Office of Response and Restoration Division, National Oceanic and Atmospheric Administration, 34 p. [Accessed Mar-09-2015] [http://archive.orr.noaa.gov/book\\_shelf/122\\_NEW-SQuiRTs.pdf](http://archive.orr.noaa.gov/book_shelf/122_NEW-SQuiRTs.pdf)
- Canadian Council of Ministers of the Environment (CCME). 1998-2001. Canadian Environmental Quality Guidelines Summary Table – Sediment Quality Guidelines for the Protection of Aquatic Life. [Accessed Ma-09-2015] <http://st-ts.ccme.ca/>
- Canadian Council of Ministers of the Environment (CCME) 1991-2009. Canadian Environmental Quality Guideline Summary Table- Soil Quality Guidelines for the Protection of Environmental and Human Health. [Accessed Mar-20-2015] <http://st-ts.ccme.ca/en/index.html>
- Dimond, J.B., A.S. Getchell, and J.A. Blease. 1971. Accumulation and persistence of DDT in a lotic ecosystem. *J. Fish. Res. Bd. Canada* 28; 1877-1882.
- Environmental Protection Agency (US EPA). 1994a. Method 200.7 Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry (Revision 4.4, 1994) In: *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online)*.
- Health Canada, 2009. Healthy living, Mercury and Human Health (online). <http://www.hc-sc.gc.ca/hl-vs/iyh-vsv/environ/merc-eng.php> (accessed March 26, 2015).
- Keachie, P.P. and Côté, R.P. 1973. Toxic pollutants in the Saint John River Basin. Saint John River Basin Bd.,Fredericton, N.B. MS Rept. T16.
- Kidd, S., R.A. Curry and K.R. Munkittrick (Eds) 2011. *The Saint John River: A State of the Environment Report*. Canadian Rivers Institute, University of New Brunswick.
- Montaser, A and Golightly, D.W. (Eds.). 1992. *Inductively Coupled Plasmas in Analytical Atomic Spectrometry*. 2nd Revised and Enlarged Edition. September 1992. 1040 p.
- New Brunswick Department of Environment (NBENV). 2008. *New Brunswick Groundwater Chemistry Atlas: 1994-2007*. Science and Reporting Branch, Sciences and Planning Division, Environmental Reporting Series T2008-01.
- Stone, K., 2009. Canadian Minerals Yearbook (CMY) – 2009 (online). <https://www.nrcan.gc.ca/mining-materials/markets/canadian-minerals-yearbook/2009/8476> (accessed March 26, 2015).

Travers, I. 1976. Mercury in the Saint John River Basin. Environment Canada Report EPS-5-AR-76-10.

Wuana, R.A. and Okieimen, F.E., 2011. Heavy Metals in Contaminated Soils: A Review of Sources, Chemistry, Risks and Best Available Strategies for Remediation. International Scholarly Research Network Ecology. 2011:1-20.

US EPA. 1994b. Method 3640A Gel Permeation Cleanup (Revision 1, 1994) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1996a. Method 3620C Florisil Cleanup (Revision 3, 1996) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1996b. Method 8082 Polychlorinated Biphenyls by Gas Chromatography (Revision 0, 1996) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1996c. Method 8270C Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (Revision 3, 1996) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1996d. Method 3660B Sulphur Clean Up (Revision 2, 1996) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1998a. Method 6010C Inductively Coupled Plasma-Atomic Emission Spectrometry (Revision 3, 2007) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 1998b. Method 7473 Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry. (Revision 0, 1998) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 2007a. Method 3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils. (Revision 1, 2007) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).

US EPA. 2007b. Method 3545A Pressurized Fluid Extraction (Revision 1, 2007) In: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846 Online).