Photo: Pauline Teasdale

WATER QUALITY IMPROVEMENT

Concentrations of Medications, Hormones and Other Emergent Contaminants in the St. Lawrence and Three of Its Tributaries

Issue

A significant number of prevalent pharmaceutical and personal care products (PPCPs), such as hydrating creams for the skin, shampoo and toothpaste, are transferred to the water during showers and other hygiene practices. Other PPCPs, such as oral medications, are eliminated in large part in human waste and also wind up in domestic waste water. The disposal of these substances by manufacturing industries and the improper disposal of unused products (e.g., medication flushed down the toilet) are other sources of PPCPs in industrial and municipal waste water.

In Quebec, as elsewhere, sampling done throughout the last decades has revealed the presence of new contaminants in waterways, as well as new effects on aquatic organisms. In the St. Lawrence River downstream of Montréal, for example, observations of freshwater mussels and fish show that they have been feminizing (Aravindakshan et al. 2003; Blaise et al. 2003). This phenomenon has been attributed to the presence of natural and synthetic hormones in the water, as well as chemicals known as "endocrine disruptors" that can act as hormones.

Municipal waste water treatment plants remove some of the PPCPs and other complex substances; however, this removal is incomplete and varies depending on the substances and the type of treatment. Portions of these substances can still be found in treatment plant final effluent, which is released into receiving waterways such as the St. Lawrence River.

The presence of PPCPs in waterways downstream of urban centres has been a known reality for more than 30 years. Hormones, such as estrogen and testosterone, have also been detected, as well as metabolites such as cholesterol. These substances, common to most organisms that are part of the animal kingdom, can also be found downstream of livestock farms.

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Urban agglomerations, such as Montréal, are sources of PPSP and other contaminants of emerging interest in streams.

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Since the late 1990s, government authorities have increased their water surveillance mandate to include an increasing number of contaminants of emerging interest, several of which are endocrine disruptors. Quebec's Ministère du Développement durable, de l'Environnement et de la Lutte contre les changements climatiques (MDDELCC) and Environment Canada have conducted sampling to confirm the presence of many of these substances in the St. Lawrence and some of its tributaries. This sheet presents the results obtained by these two agencies with respect to PPCPs, hormones and a few other emerging contaminants. This is the second publication on these substances; the first mainly dealt with the presence of these products in drinking water and effluent from waste water treatment facilities (MDDEP 2011).

Methodology

The data from Environment Canada and the MDDELCC in this study are taken from 11 sampling sites: 8 on the St. Lawrence River and 1 at the mouth of the Ottawa, Richelieu and Saint-Maurice rivers. The locations of these sampling sites, as well as the main releases of treated waste water in the study area, are presented in Figure 1.

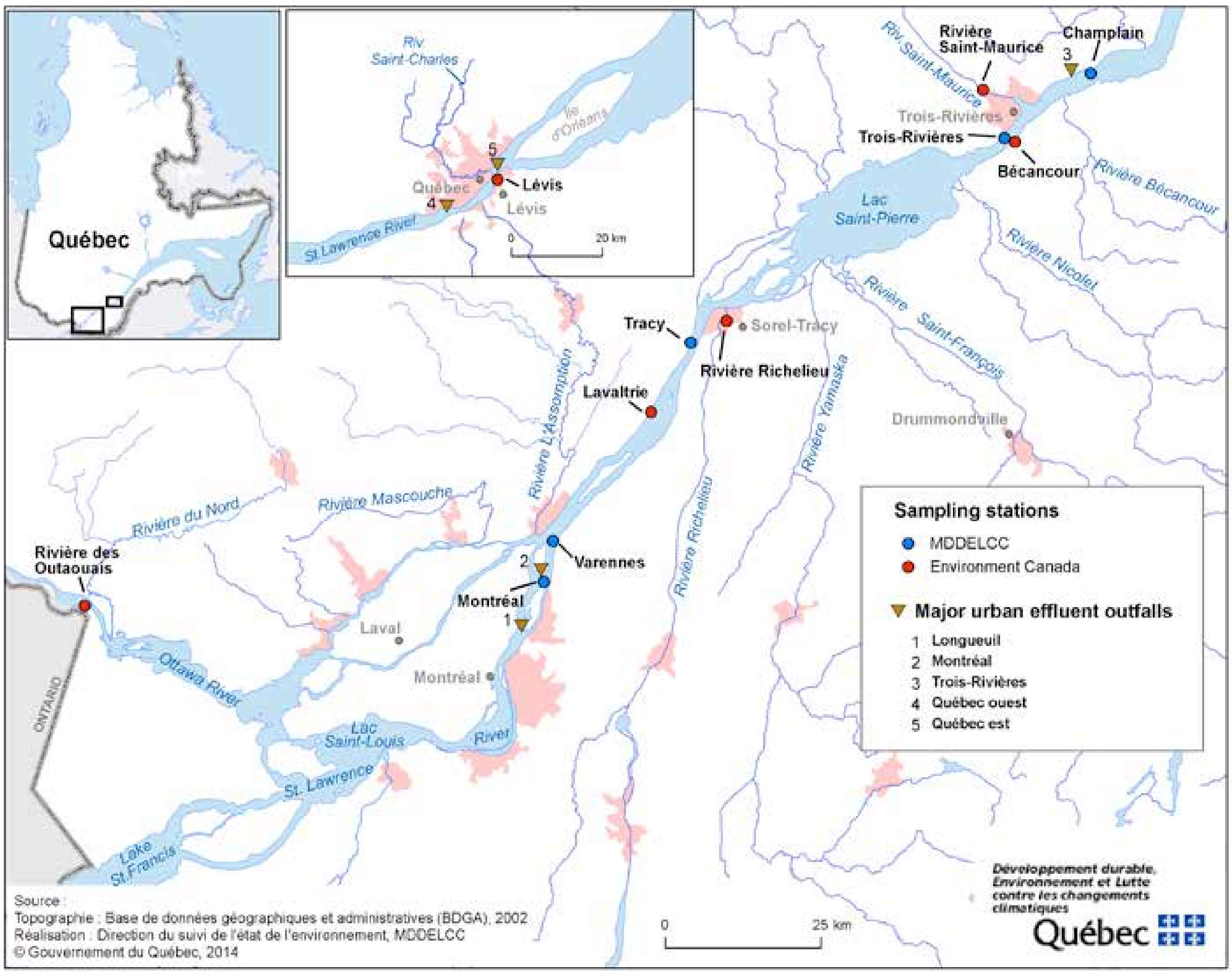

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Figure 1. Location of sampling stations.

Except for the Saint-Maurice River, which was sampled 4 times, Environment Canada's sites were sampled 10 to 14 times from November 2006 to March 2010. The MDDELCC sites were sampled 6 times on a monthly basis, from May to October 2009. The analysis of contaminants for both agencies was done in the laboratories of the MDDELCC's Centre d'expertise en analyse environnementale du Québec, with detection limits in the order of nanograms per litre (ng/L). The specific detection limits for each of the products analyzed are listed in Table 1.

This sheet presents results obtained for 44 substances: 30 PPSPs, 6 hormones, cholesterol and 3 of its derivatives, caffeine, triclosan and chlorophenol (2 antibacterial agents), and bisphenol A (a plasticizer). For almost all of these substances, there are no water quality criteria against which the measured concentrations can be compared in order to evaluate the potential negative effects on aquatic organisms. For this reason, the analytical results are compared instead against scientifically documented concentrations leading to toxic effects, as well as values obtained elsewhere, in other waterways, in North America and in Europe.

Comparison against toxic effects thresholds is one method of evaluating, as a first estimate, whether concentrations measured in the St. Lawrence and its tributaries can be damaging to the organisms living there. Comparisons with concentrations measured in other water bodies aim to determine the level of contamination of the St. Lawrence in relation to other large water bodies. The detection limits of the current study (ng/L) are low enough to permit these comparisons, since the vast majority of toxicological studies and environmental monitoring of PPCPs have been done at concentrations of the same order of magnitude or higher.

Overview

Table 1 summarizes the results for the 44 substances analyzed. Of these substances, 21 had concentrations greater than the detection limit: 4 analgesics/anti-inflammatories, 5 antibiotics, 5 hormones, cholesterol and its 3 degradation products, caffeine, triclosan, and bisphenol A.

Table 1. Substances analyzed and summary of results.

Substance	MDL (ng/l)	Number of samples	Frequency of detection (%)	Concentration interval (ng/l)
Analgesics/anti-inflammatory agents				
Acetaminophen	20	56	80	< 20–500
Ibuprofen	6	91	63	< 6–90
Naproxen	20	91	29	< 20–83
Salicylic acid[1]	55	91	16	< 55–130
Diclofenac	5	91	0	–
Fenoprofen	7–11	91	0	–
Indometacin	10	91	0	–
Ketoprofen	6	91	0	–
Antibiotics				
Chlortetracycline	10–40	56	5.4	< 10–270
Tylosin	2	56	3.6	< 2–34
Erythromycin	20–200	56	1.8	< 20–210
Sulfamethoxazole	10	56	1.8	< 10–10
Tetracycline	20–100	56	1.8	< 20–700
Monensin	40–200	56	0	–
Narasin	50–100	56	0	–
Norfloxacin	10	56	0	–

Table 1. Substances analyzed and summary of results (continued).

Substance	MDL (ng/l)	Number of samples	Frequency of detection (%)	Concentration interval (ng/l)
Antibiotics				
Oxytetracycline	20	56	0	–
Roxithromycine	10	56	0	–
Sulfadimethoxine	4	56	0	–
Sulfamethazine	5	56	0	–
Sulfamethizole	5	56	0	–
Sulfathiazole	20	56	0	–
Trimethoprim	10	56	0	–
Antilipemic agents and other medication				
Clofibric acid	5	91	0	–
Bezafibrate	9	91	0	–
Fenofibrate	10–11	91	0	–
Gemfibrozil	5	91	0	–
Carbamazepine (antiepileptic)	5	91	0	–
Fluoxetine (anti-depressant)	10	56	0	–
Pentoxifylline (treatment of lameness)	23	91	0	–
Hormones and cholesterol				
Estradiol-17ß[2]	1–1.5	104	7.7	< 1–11
Estrone[2]	0.5–0.7	104	5.8	< 0.5–5.6
Estriol[2]	2–2.5	104	3.8	< 2–17
Testosterone[2]	4–5.1	104	1.9	< 4–9.8
17A-ethynylestradiol (contraceptive)	2–2.5	104	1.9	< 2–3.1
Mestranol (contraceptive)	8	91	0	–
Cholesterol	0.5–0.7	104	95	< 0.5–1000
Coprostan-3-ol[3]	4–5.1	104	83	< 4–360
Coprostan-3-one[3]	4–5.1	104	46	< 4–91
Coprostan[3]	1–1.7	104	16	< 1–20
Others				
Caffeine	13	91	85	< 13–950
Triclosan (disinfectant)	6	91	47	< 6–34
Chlorophene (disinfectant)	7–13	91	0	–
Bisphenol A (plasticizer)	0.5–2.5	103	80	< 0.5–90

MDL: method detection limit

1. Analytical results include acetylsalicylic acid and one of its metabolites, salicylic acid, that cannot be differentiated with the analytical method used.
2. From a natural (human or animal) or synthetic source (contraceptive or hormonal therapy).
3. Derived from cholesterol.

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Analgesics/anti-inflammatories

Acetaminophen (e.g., Tylenol™) is the most frequently detected analgesic, detected in 4 out of 5 samples, followed by ibuprofen (e.g., Advil™, Motrin™) in two thirds of samples, naxopren (e.g., Anaprox™) in a third of samples, and salicylic acid (e.g., Aspirin™) in 1 out of 6 samples. The fact that acetaminophen and salicylic acid are found in the St. Lawrence and its tributaries is not surprising, as they are both on the list of the 10 pharmaceutical products most sold through prescription in Canada. These substances are also available over the counter, and the amount sold in this manner is certainly much higher than that sold under prescription.

Figure 2 shows the concentrations measured at different sampling stations for the 4 detected analgesics/anti-inflammatories. Acetaminophen has higher concentrations than the other products, with a median per station of 77 to 250 ng/L, except for upstream of Trois-Rivières, where 5 samples out of 6 are below the 20 ng/l detection limit. For the 3 other substances, the highest median per station is 61.5 ng/L (naproxen at Lavaltrie). Except for this value, all of the medians for naproxen and salicylic acid are below detection limits.

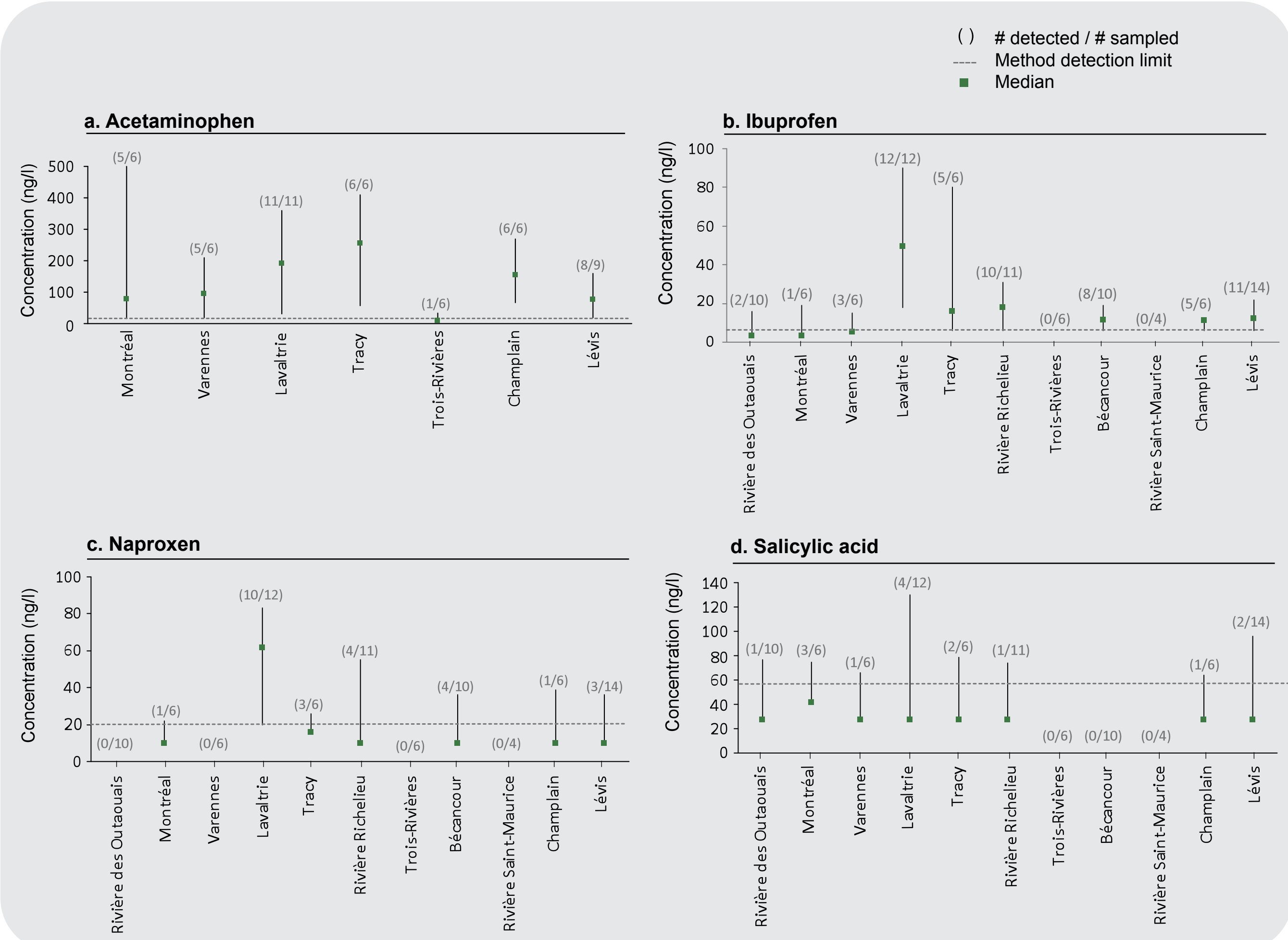

Figure 2. Minimum, maximum and median concentrations of analgesic and anti-inflammatory products most often detected at the monitoring sites on the St. Lawrence River and at three of its tributaries.

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The Greater Montréal area increases the concentrations of acetaminophen and ibuprofen in the St. Lawrence. Figure 2 shows how the detection frequencies and concentrations of these substances are higher downstream of the urban area (at the Lavaltrie and Tracy stations) than upstream (at the Ottawa River and Montréal stations). Though less noticeable, Trois-Rivières has a similar effect: the values at the Champlain station are higher than those at the Trois-Rivières station. At Lévis, concentrations are similar to those measured downstream of Trois-Rivières.

The Varennes station is situated 5.5 km downstream of the Montréal waste water outfall. However, the weak concentrations measured at that station for all of the analyzed substances indicate that it was outside of the city's effluent dispersion plume.

Figure 2 shows that the naproxen concentration profile is similar to that of acetaminophen and ibuprofen, except that the effect from Trois-Rivières is hardly noticeable. For salicylic acid, there is hardly any difference between the stations.

Except for ibuprofen and naproxen in the Richelieu River, the concentrations in the three tributaries are relatively low (Figure 2). In the Saint-Maurice River, at the intake for Trois-Rivières, all of the analyzed results are below the detection limit.

Studies were carried out on the toxicity of analgesics/anti-inflammatories, using standardized tests employed frequently in aquatic toxicology. These laboratory tests aimed to establish the concentration of the contaminant required to provoke inhibition of growth or reproduction, or to cause the death of the studied organisms. Protocols for this purpose were developed for different organisms, including the unicellular alga *Scenedesmus spicatus*, the crustacean *Daphnia magna* and the Japanese medaka (*Oryzias latipes*). Studies show that the concentrations of analgesics/anti-inflammatories required to provoke these types of toxic effects in these organisms are on the order of µg/L or mg/L (Santos et al. 2010; Corcoran et al. 2010). These concentrations are higher than those measured in the St. Lawrence and its tributaries, which are on the order of ng/L. However, at these lower concentrations, the possibility of subtle or indirect effects cannot be ruled out, as is explained below in the Perspectives section.

Photo: Christian DeBlois, CEAEQ

The technician puts the samples for the analysis of antibiotics in a liquid chromatograph coupled to a mass spectrometer in tandem. Analyses are performed at the Centre d'expertise en analyse environnementale du Québec (CEAEQ) of the MDDELCC.

The concentrations of analgesics/anti-inflammatories detected in the St. Lawrence and its tributaries are in the same order of magnitude as those reported in waterways throughout the United States, in Germany and in Canada. In these countries, however, at detection limits similar to those in this study, diclofenac, fenoprofen, indomethacin and ketoprofen were also detected (Figure 3).

Antibiotics and Other Medications

Of the 15 analyzed antibiotics, only 5 were detected, and only in a few samples. Of the 56 samples analyzed for these substances, chlortetracycline was detected in 3 samples and tylosin in 2, while erythromycin, sulfamethoxazole and tetracycline were detected once each.

Chlortetracycline, erythromycin and tetracycline were not often detected, but as shown in Table 1, the samples that were positive for this substance showed relatively elevated concentrations, of 270, 210 and 700 ng/L respectively. These maximum values are still lower, however, than those required to induce the toxic effects measured by the aforementioned standard tests; these concentration thresholds are on the order of hundreds of µg/L or mg/L (Santos et al. 2010; Corcoran et al. 2010). However, at lower concentrations, the possibility of more subtle or indirect effects cannot be ruled out, for the reasons discussed below in the Perspectives section.

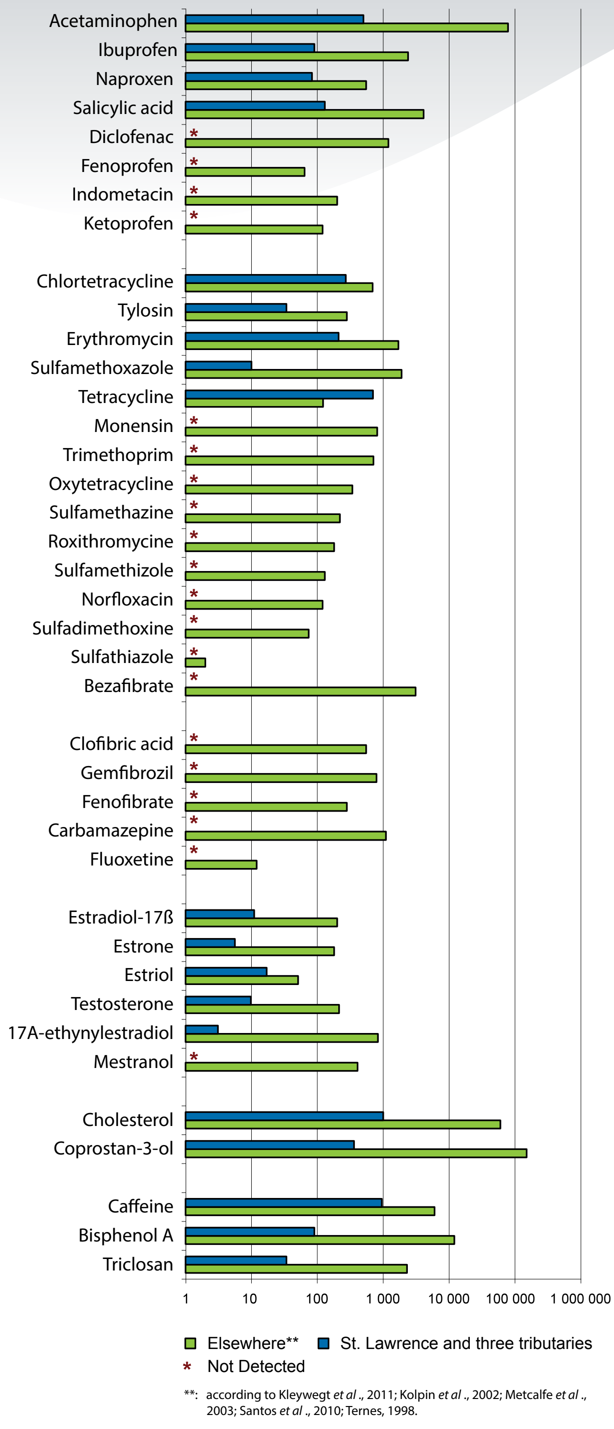

Figure 3. Maximum concentrations (ng/L) of medications and hormones measured in the St. Lawrence River and its tributaries under study, as well as elsewhere in North America and Europe.

The current study included measures of four lipid depressors, medications that reduce the amount of cholesterol in the blood: clofibric acid, bezafibrate, fenofibrate and gemfibrozil. The analyses also included carbamazepine, an anti-epileptic drug, and fluoxetine, an antidepressant. None of these products was detected in the St. Lawrence and its three tributaries, although they have been detected frequently in other areas of North America and Europe at higher concentrations than the detection limits of this study (Figure 3).

Hormones and Cholesterol

As with antibiotics and other medications, hormones were detected in few samples, specifically in 0 to 8% of samples, depending on the hormone. The detection limits for the hormones were low (between 0.5 and 8 ng/L), but these substances were also present at very low concentrations, the maximum obtained being 17 ng/L for estriol (Table 1). In total, there were 22 detections, found at 5 sampling stations. No hormones were detected upstream of Montréal (at the Ottawa River and Montréal stations). The number of detections is a bit higher downstream of Montréal (Lavaltrie) and at Québec City.

Hormones of natural origin—17-estradiol, estrone and testosterone—account for 20 of the 22 detections. Synthetic hormone 17A-ethynylestradiol, notably used in contraceptives, was only detected 2 times, at Lévis, with concentrations of 3.0 and 3.1 ng/L. These concentrations were higher than the criteria for water quality for this substance for British Columbia (0.5 and 0.75 ng/L, for chronic and acute exposure respectively) and the European Commission (0.035 ng/L). Because the detection limit for this analysis method (2.0 or 2.5 ng/L, depending on the sample) is higher than the criteria, it is possible that samples exceeding the criteria went by undetected.

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Cholesterol is a fat found in all animals. It is both a cellular constituent and a key element for various metabolic pathways. Coprostan, measured here in different forms, is a by-product of partial digestion of cholesterol by living organisms, including humans. Taking into account the omnipresence of cholesterol in the animal world, it is not surprising that it was detected in almost all of the samples (95%) in this study, with concentrations slightly higher downstream of Montréal. Median concentrations at 9 of the 11 stations in this study were between 37 and 111 ng/L, and the maximum concentrations ranged between 96 and 429 ng/L (Figure 4). Concentrations were higher at Lavaltrie and downstream of Montréal (median of 213 and maximum of 1450 ng/L), and lower at Trois-Rivières (median and maximum of 0.25 and 14 ng/L respectively).

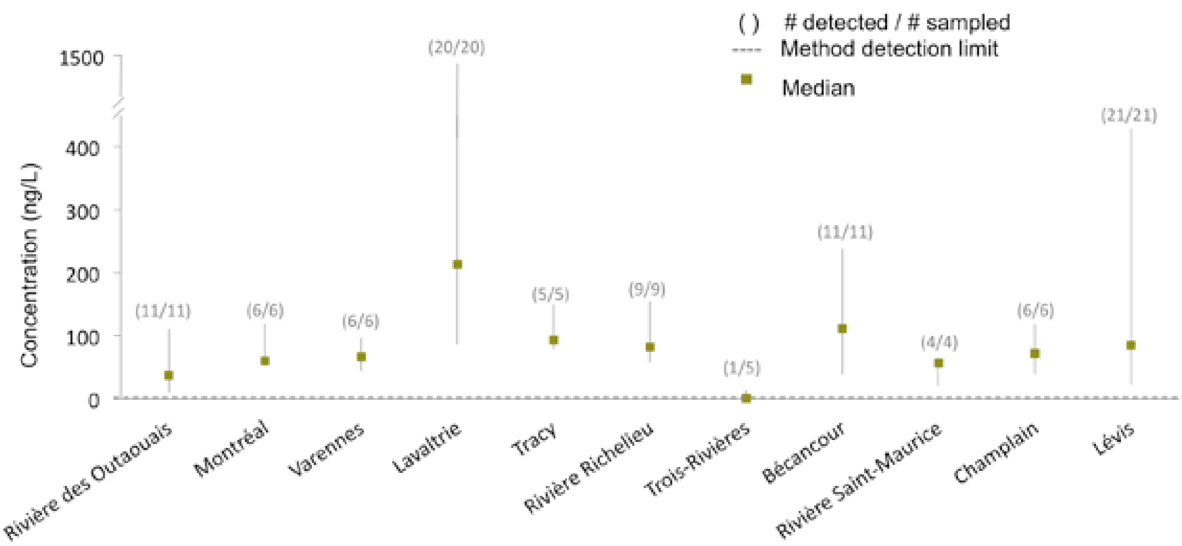

Figure 4. Minimum, maximum and median concentrations of cholesterol and its derivatives at the monitoring sites on the St. Lawrence River and at three of its tributaries.

Caffeine

Caffeine is a component of certain medications; however, the consumption of coffee and other caffeinated beverages contributes the most to its presence in surface water. Present in 85% of samples, this product was detected more frequently and in stronger concentrations than all other substances except acetaminophen, cholesterol and one of its derivatives. Caffeine concentrations varied from below the detection limit (13 ng/L) to 950 ng/L, with medians per station that varied from below the detection limit to 495 ng/L. As with other substances, detection frequencies and concentrations tended to be higher downstream of Montréal, Trois-Rivières and Québec City (Figure 5).

Caffeine concentrations measured in the St. Lawrence and the three tributaries under study were much lower than those that can be directly toxic to aquatic organisms, i.e., on the order of hundreds of mg/L for acute toxicity and dozens of mg/L for chronic toxicity (EPA 2012). However, as with the other detected substances and as explained at the end of this sheet in the Perspectives section, the possibility of subtle or indirect effects cannot be ruled out.

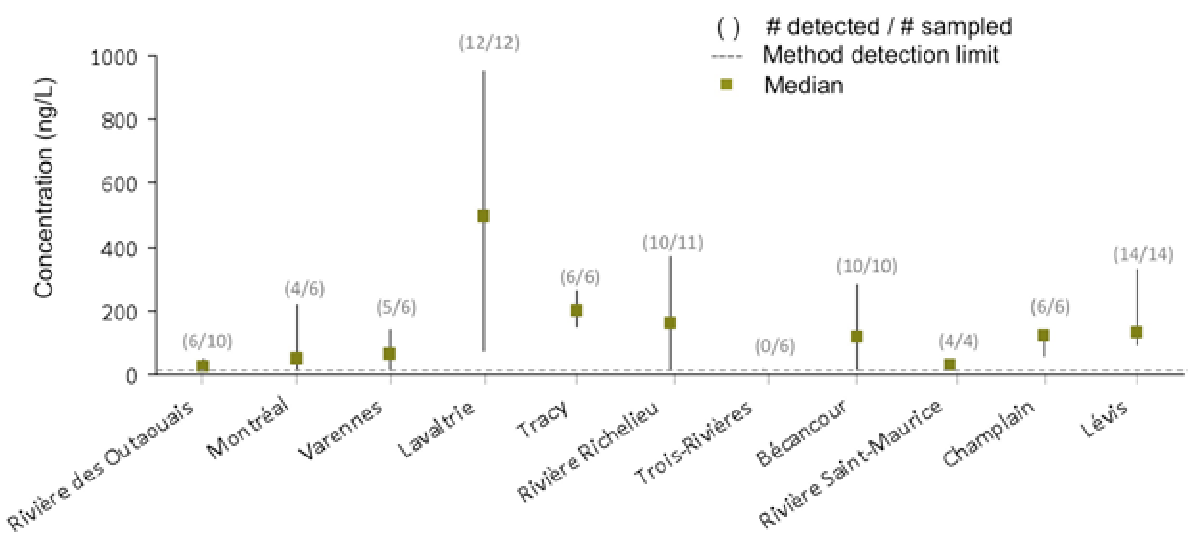

Figure 5. Minimum, maximum and median concentrations of caffeine at the monitoring sites on the St. Lawrence River and at three of its tributaries.

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Triclosan

Triclosan is an antibacterial and antifungal conservation agent used in medications and in many personal care and cleaning products: creams and lotions for the face, hands and body; deodorants; perfumes; sunscreen; shaving products; shampoo; cleaning or disinfecting products for the hands; toothpaste; etc. It is also used to control the growth of micro-organisms on fabrics, paper, leather, plastic and rubber. This substance was assessed under the *Canadian Environmental Protection Act, 1999*. The conclusion was that this product is found in the Canadian environment in concentrations possibly harmful to aquatic and terrestrial organisms. As a result, the federal government has committed to take measures to reduce the concentrations of triclosan in the environment.

Triclosan was detected in almost 50% of samples taken from the St. Lawrence and its tributaries under study, with concentrations varying from the detection limit to 34 ng/L. Detection frequencies and concentrations are low upstream of Montréal and in the tributaries, and higher downstream of Montréal and upstream and downstream of Trois-Rivières and Québec City (Figure 6). The maximum concentration of triclosan measured in this study (34 ng/L) is below the effects threshold for aquatic organisms (115 ng/L) found by Environment Canada in its assessment of this substance (Health Canada and Environment Canada 2012), which noted effects on amphibian thyroid functions at concentrations between 30 and 300 ng/L. Of the 91 samples analyzed for triclosan in this study, only 1, 34 ng/L, fell into this concentration range.

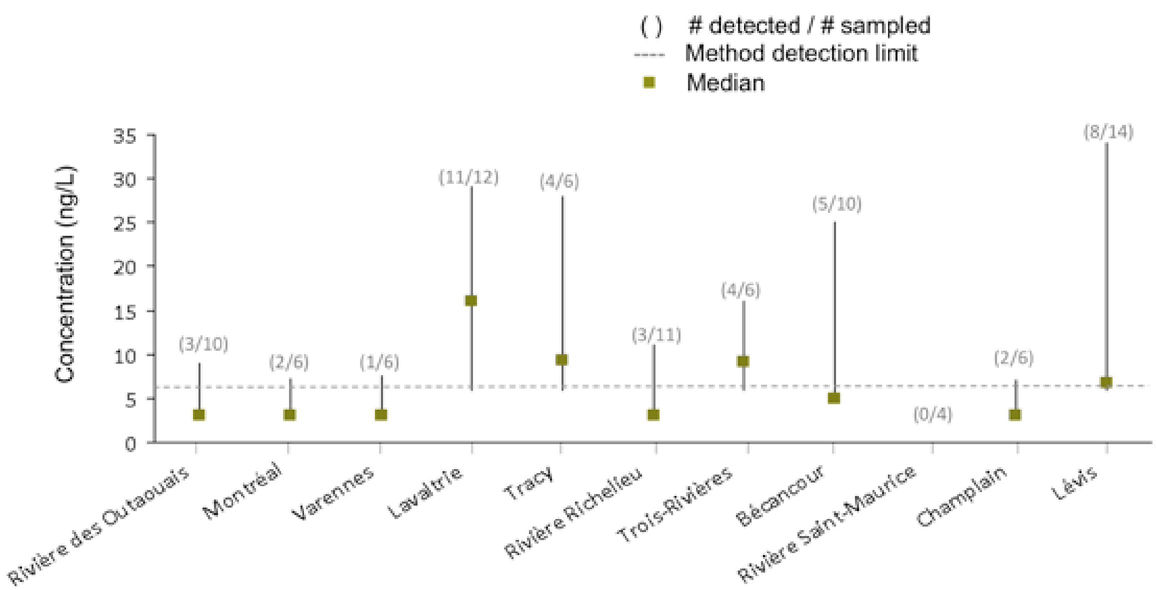

Figure 6. Minimum, maximum and median concentrations of triclosan at the monitoring sites on the St. Lawrence River and at three of its tributaries.

Bisphenol A

Bisphenol A is used primarily for the production of polycarbonate and epoxy resin plastics. These plastic substances are used in the manufacturing of a host of products: compact disks, containers for food and beverages, water pipes, packaging for electronics, electrical equipment, interior coatings of cans, concrete, automobile pieces, etc. This substance is produced and used in large quantities and was assessed under the *Canadian Environmental Protection Act, 1999.* The assessment concluded that this product is toxic, leading to measures aiming to reduce the exposure of individuals and ecosystems to this substance (Environment Canada and Health Canada 2008). One of these measures, effective March 11, 2010, was to ban bisphenol A in baby bottles in order to reduce the exposure of newborns to this substance.

Bisphenol A was detected in 80% of samples taken from the St. Lawrence and its three tributaries under study, with concentrations ranging between the detection limit of 0.6 ng/L and 90 ng/L. The medians by station were between 0.3 and 11 ng/L. The pattern of higher concentrations downstream of Montréal, Trois-Rivières and Québec City, found for other substances, is only evident here for the maximum concentrations (Figure 7). We recorded maximums of 90, 42 and 46 ng/L at Lavaltrie, Bécancour and Lévis respectively. These concentrations are all much lower than the MDDELCC chronic criteria of 20 µg/L for the protection of aquatic life (MDDEP 2009) and lower than the "estimated no-effect concentration" of 175 ng/L retained by the federal government in its evaluation of bisphenol A.

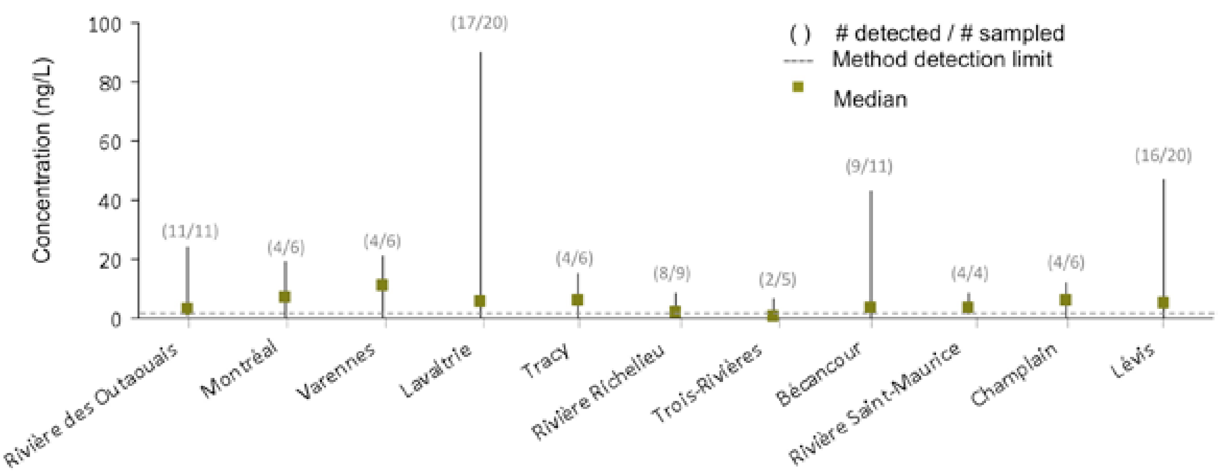

Figure 7. Minimum, maximum and median concentrations of bisphenol A at the monitoring sites on the St. Lawrence River and at three of its tributaries.

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Perspectives

Many PPCPs, hormones and other emerging contaminants of interest analyzed in this study were detected in the St. Lawrence and the three tributaries under study, at concentrations on the order of nanograms per litre. In general, concentrations observed in the study were comparable or inferior to those measured in other waterways in North America or Europe and are similar to those measured in other waterways in Quebec (MDDEP 2011). In the St. Lawrence, numbers and concentrations of detected substances are higher downstream of or close to the Montréal, Québec City and Trois-Rivières urban areas (Figure 8).

Following treatment, Montréal's waste waters are rejected in the St. Lawrence River, near île aux Vaches. The yellow arrows indicate the location of the dispersion plume of the treated waters.

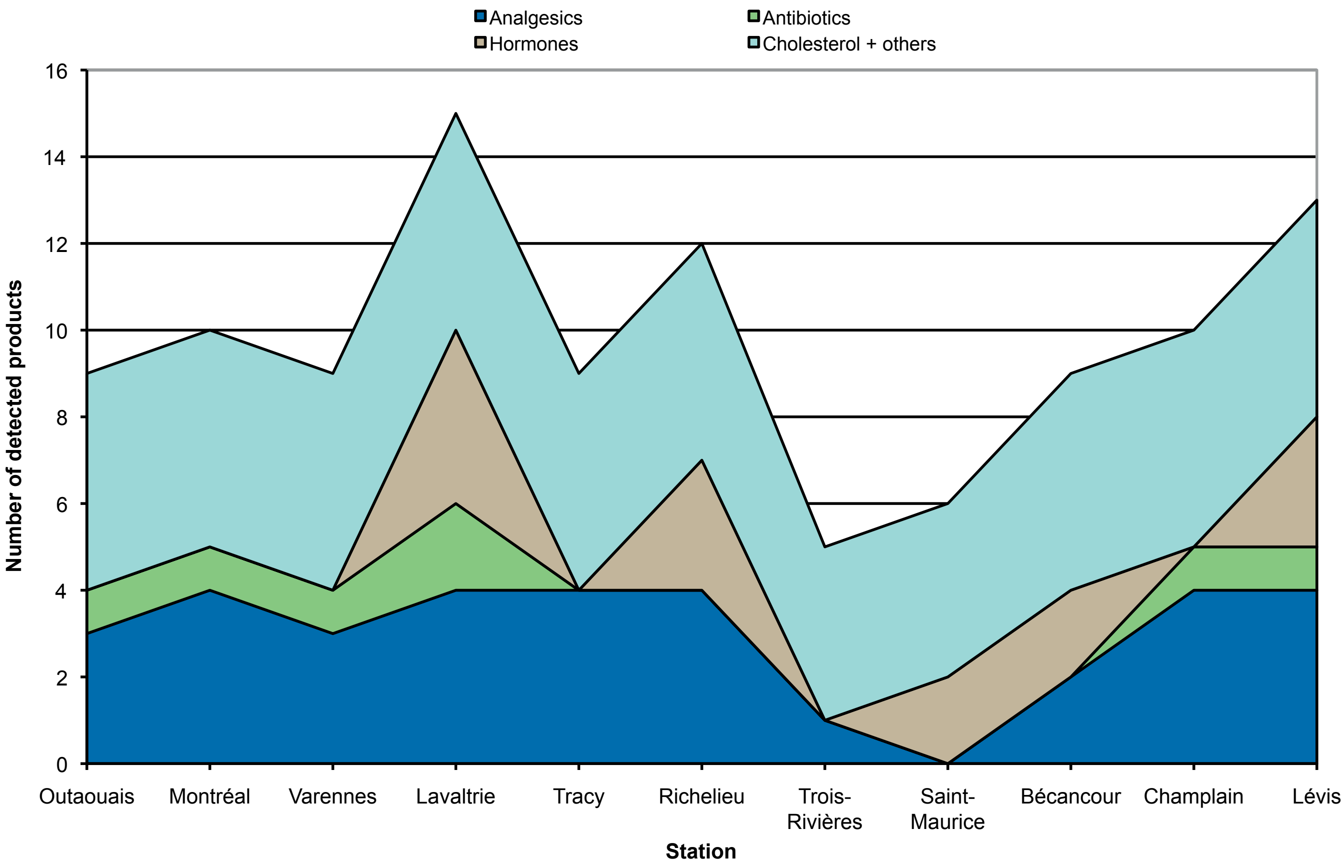

Figure 8. Number of products detected by category at the different sampling stations.

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The concentrations of PPCPs and other emerging contaminants measured in this study are lower than those that can have toxic effects, as measured by standard toxicity tests. However, this observation should not be considered as proof of the safety of the detected substances, for the following reasons:

- Studies have shown that PPCPs and other emerging contaminants can have effects on fish and other aquatic organisms, even when they are present in low concentrations in the water. For example, exposing the Japanese medaka to ibuprofen at a concentration of about 1 µg/L was enough to cause an increase in liver weight in female fish and a reduced liver weight in males (Flippin et al. 2007). Furthermore, 1.8 µg/L of benzodiazepine, an anti-anxiety medication, caused changes in the behaviour of European perch: exposed specimens became more active, less sociable and foraged more (Brodin et al. 2013). These types of effects and the concentrations required to induce them are not known for all PPCPs and other emerging contaminants.

- Little is known about the combined action of the different substances present in the environment. Taken individually, each substance could be found at a concentration lower than the effects threshold; however, the additive or synergistic action of the various products could lead to effects on organisms. It is important to remember that downstream of urban centres, fish and other aquatic organisms are exposed to many PPCPs, hormones and other substances simultaneously: surfactants (e.g., nonylphenol ethoxylates), flame retardants (e.g., PBDEs), protective coatings (e.g., PFOS, PFOA), etc. Many of these products are endocrine disruptors whose combined effects are not well known.

- In essence, this study concerned itself with parent compounds only. In the water, these substances can transform and be found in forms that are still toxicologically active but undetectable by our standard methods of analysis. There is no analysis method capable of detecting all of the by-products of the breakdown of PPCPs and other emerging contaminants. In fact, not all of these by-products are known.

Owing to recent technological developments, it is now possible to detect and monitor these new substances of concern in the environment. Detection and monitoring programs must be implemented or sustained, as the case may be, in order to determine the chemical behaviour and fate of emerging contaminants in the aquatic environment, and close to urban regions in particular.

Key Measures

The establishment of water quality criteria for a greater number of emerging contaminants is crucial to a proper assessment of the risks associated with the presence of these substances in surface waters. Water quality criteria are thresholds or recommendations that permit us to assess whether different water uses are compromised by the presence of a substance. The criteria are not standards and therefore have no legal force. However, they still serve as a reference for evaluating aquatic ecosystem health. Any substance with a concentration exceeding the criterion could have an undesirable effect on an aspect of the ecosystem. There are few emerging contaminants for which water quality criteria are available, as the toxicity and endocrine actions of these substances are not well known. Among the substances covered in this study, there are no available criteria for 17A-ethinylestradiol and bisphenol A.

The comparison of the concentrations observed here against those obtained in other parts of the world for the same substances is another way to evaluate the chemical state of our waterways. However, this method does have its limits, as the studies in question may have different objectives, concern different types of water bodies, or use different methods of analysis. For these reasons, comparison must be done with caution, and concern itself with the order of magnitude of concentrations rather than specific values.

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WATER QUALITY IMPROVEMENT

Written by : **David Berryman**

Direction du suivi de l'état de l'environnement
Ministère du Développement durable, de l'Environnement et de la Lutte contre les changements climatiques

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