A Paleoenvironmental Study Tracking Eutrophication, Mining Pollution, and Climate Change in Niven Lake, the First Sewage Lagoon of Yellowknife (Northwest Territories)

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ABSTRACT. Niven Lake was the first wastewater disposal site for the City of Yellowknife (Northwest Territories, Canada), receiving domestic sewage for more than 30 years. Here, we used a high-resolution sediment core to track past sewage inputs to Niven Lake by comparing changes in sedimentary sterols and three diagnostic ratios for human fecal contamination, as well as biological assemblages and overall lake production, with the known history of sewage inputs to the lake from 1948 to 1981. Coprostanol, often considered the best indicator of human fecal contamination, increased by ~8% between depths of 7.5 cm and 5 cm (~1950 to 1981) and was more reliable in tracking sewage contamination than diagnostic sterol ratios. Muted responses in subfossil diatom and chironomid assemblages were noted during the time of sewage inputs, and similar responses have been reported in other eutrophic Arctic sites, as well as in many macrophyte-dominated shallow lakes in general. More marked shifts in diatoms and chironomids occurred a decade after the end of sewage inputs, in the 1990s, a time that closely aligned with the warmest years on record for Yellowknife. This post–sewage era response was indicative of anoxia and possibly of positive feedback from internal phosphorus loading. The response may have been facilitated by recent climate warming, resulting in a lagging recovery from eutrophication. Changes in the diatoms and chironomids of Niven Lake were also indicative of metal pollution, suggesting that the lake has experienced the compounding effects of arsenic contamination from nearby gold mining.

Key words: sewage lagoon; Arctic; paleolimnology; sterols; stable nitrogen isotopes; diatoms; chironomids; shallow lakes

RÉSUMÉ. Le lac Niven était le premier site d'évacuation des eaux usées de la ville de Yellowknife (Territoires du Nord-Ouest, Canada) et il a reçu des eaux domestiques pendant plus de 30 ans. Ici, nous avons utilisé une carotte de sédiments à haute résolution pour analyser les anciens apports en eaux usées du lac Niven en comparant les changements dans les stérols sédimentaires et trois rapports diagnostiques pour la contamination fécale humaine, ainsi que les assemblages biologiques et la production générale du lac, selon les antécédents connus d'apports en eaux usées du lac de 1948 à 1981. Le coprostanol, souvent considéré comme le meilleur indicateur de contamination fécale humaine, augmentait d'environ 8 % à des profondeurs se situant entre 7,5 cm et 5 cm (~1950 à 1981) et était plus fiable pour évaluer la contamination par les eaux usées que les rapports diagnostiques des stérols. Des réponses atténuées dans les diatomées subfossiles et les assemblages chironomidés ont été notées dans les apports en eaux usées, et des réponses semblables ont été signalées dans d'autres sites eutrophiques de l'Arctique ainsi que dans de nombreux lacs peu profonds dominés par les macrophytes en général. Des changements plus marqués dans les diatomées et les chironomidés ont eu lieu une décennie après la fin des apports en eaux usées, dans les années 1990, une période étroitement liée aux années les plus chaudes à Yellowknife. La réponse après la fin de l'évacuation des eaux usées révélait une anoxie et possiblement une réaction positive à partir de la charge de phosphore interne. Cette réponse a peut-être été facilitée par le réchauffement récent du climat, provoquant ainsi un retard dans le rétablissement après l'eutrophisation. Les changements touchant les diatomées et les chironomidés du lac Niven témoignaient également de la pollution par les métaux, ce qui suggère que le lac a connu les effets conjugués de la contamination à l'arsenic provenant de l'exploitation minière à proximité.

Mots clés : étang de stabilisation; Arctique; paléolimnologie; stérols; isotopes d'azote stables; diatomées; chironomidés; lacs peu profonds

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INTRODUCTION

Sewage lagoons, or water bodies used to decontaminate sewage, are an inexpensive and often cost-efficient method of wastewater treatment for communities that cannot make use of large wastewater treatment facilities. This is especially true for northern communities that find it difficult to implement more complex waste-management infrastructure because of their remote location and extreme climate. Thus, untreated sewage may be released directly into sewage lagoons, coastal bays, or other freshwater systems. The effectiveness of sewage lagoons in treating the wastewater of Arctic communities has been of interest for decades (e.g., Miyamoto and Heinke, 1979) and is still closely monitored today as new risks and challenges arise (Gunnarsdóttir et al., 2013). Yet few intensive limnological studies of wastewater-related cultural eutrophication have been undertaken in Arctic regions even though interest in northern resources, which has been growing in recent decades and will likely continue to grow, has led to expansion of northern populations (AMAP, 2010). After their useful lifetime as sewage receptacles has passed, abandoned lagoons can be assessed for recovery from eutrophication, as well as from contamination by sewagerelated pollutants and pathogens (Squires, 1982; Heinke and Smith, 1986; Ferguson Simek Clark, 1990a, b).

Niven Lake (62.4612° N, 114.3695° W; Fig. 1) was the primary receptacle of wastewater for the community of Yellowknife from 1948 until 1981, when the current lagoon system at Fiddlers Lake was constructed (Squires, 1982). Throughout the 1950s and into the 1960s, the lake was considered effective in providing what was deemed to be acceptably clean effluent that flowed down a ravine and into "Back Bay," a portion of Yellowknife Bay in Great Slave Lake (reviewed by Heinke and Smith, 1986). However, in the decade after territorial government operations were moved from Ottawa to Yellowknife in 1967, the population of Yellowknife more than tripled (from ~3000 to ~10000 residents by 1977). Niven Lake became overloaded, and it was determined that the effluent was no longer being sufficiently detoxified (Grainge, 1971; Yamomoto, 1975; Bell et al., 1976). Once Niven Lake was no longer used as a sewage lagoon, studies of the public and environmental health risks associated with it were initiated, and remediation options were considered, since the area around Niven Lake was attractive for residential development (Squires, 1982; Heinke and Smith, 1986; Ferguson Simek Clark, 1990a, b). Because of logistical and cost constraints, the City of Yellowknife opted to allow Niven Lake to recover naturally from sewage inputs, after confirming that this course of remediation posed no serious risk to public or environmental health (Ferguson Simek Clark, 1990a). The Niven Lake area was developed shortly after 2008 and is now in the centre of a prosperous and growing residential district.

For this study, we measured the stanols, coprostanol and epicoprostanol, in dated lake sediments to trace human sewage inputs to Niven Lake over time, as they have been

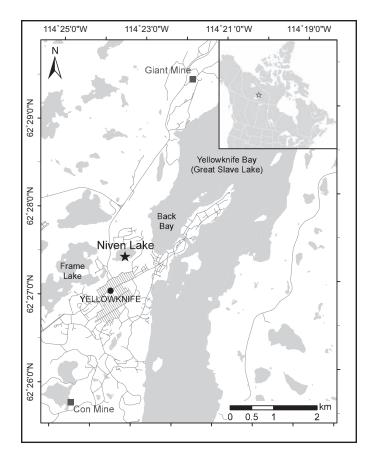


FIG. 1. Map showing the location of Niven Lake in a residential neighbourhood of Yellowknife, Northwest Territories. Niven Lake drains Frame Lake (immediately to the west), and water from Niven Lake flows into Back Bay, which is a part of Yellowknife Bay of Great Slave Lake. Giant Mine (1948–2004) and Con Mine (1938–2003) likely influenced Niven Lake with the release of arsenic trioxide dust. The inset shows the location of Great Slave Lake within Canada.

shown to track human fecal contamination (Leeming et al., 1996; Bull et al., 2002; Carreira et al., 2004; Campos et al., 2012). We also explored whether changes in stable nitrogen isotope ratios, which can be enriched by human waste (Kendall et al., 2007; Vane et al., 2010), were recorded in the sediment profile. In addition, we tracked the limnological responses of the lake biota to past sewage inputs, as well as other environmental stressors, using subfossil algal and invertebrate assemblages. Finally, we estimated past trends of overall lake production using spectrally inferred sedimentary chlorophyll-*a* concentrations (Michelutti and Smol, 2016).

In higher mammals, anaerobic bacteria in the gut convert cholesterol to coprostanol, which once in the aquatic environment can undergo further microbial reduction to yield epicoprostanol (Bull et al., 2002). Furthermore, approximately 10 or more diagnostic ratios using sterols and stanols have been published for identifying human fecal contamination in different media, including sediments, river and lake water, marine water, and landfill leachate (reviewed in Furtula et al., 2012). Here we use a combination of sterol ratios to provide a robust analysis of the extent of sewage contamination in Niven Lake. There is a relatively limited understanding of the conditions that favour the preservation of sterols and stanols in sediments (Korosi et al., 2015). However, anaerobic conditions may slow sterol degradation processes, which may also be especially restricted in cold climates (Ogura et al., 1990). Our application of multiple ratios may inform future studies using sterols as a proxy for sewage contamination, especially in Arctic lakes, by comparing temporal trends in the ratios with the known history of sewage discharge into Niven Lake, as well as information about the limnological conditions under which they were likely deposited.

Subfossil diatom and chironomid assemblages have commonly been examined for studies of eutrophication, as diatom species are responsive to changing nutrient (phosphorus) conditions (Hall and Smol, 2010), and chironomid assemblages often shift with altered oxygen dynamics, specifically the decrease in bottom-water dissolved oxygen that typically accompanies eutrophication (Little et al., 2000; Luoto and Salonen, 2010). However, the response of subarctic and Arctic lakes to excess nutrient inputs can be variable and less predictable than in temperate latitudes, with a notable absence of large cyanobacterial blooms (Schindler et al., 1974), a more subdued and delayed response in diatom taxa that favours periphytic types (Douglas and Smol, 2000), and increases in chironomid production only after climate-mediated reductions in ice cover (Antoniades et al., 2011). The relatively muted responses to cultural eutrophication in northern lakes have been attributed to the dominant influence of prolonged ice cover and short growing seasons (Michelutti et al., 2007; Smol and Douglas, 2007), reinforcing the idea that the extreme climate restricts many aspects of environmental change in Arctic freshwaters.

Tracking Niven Lake's environmental past using sediment archives allowed us to assess eutrophication and contamination, as well as possible trajectories of recovery, from a continuous and time-integrated perspective. In addition, we considered the possibility of multiple environmental stressors acting on Niven Lake. Two gold mines operated near Yellowknife's city limits: Con Mine from 1938 to 2003 and Giant Mine from 1948 to 2004. Both mines released copious amounts of arsenic trioxide dust from the roasting of gold-bearing arsenopyrite ore during the early phases of the operations (Hocking et al., 1978; Indian and Northern Affairs Canada, 2007). Using the sediment record from a lake within the Giant Mine lease boundary, Thienpont et al. (2016) determined that the resultant metal pollution caused the disappearance of keystone invertebrate grazers and planktonic algal groups during the time of peak operational activities, with little to no biological recovery after closure of the mines. Therefore, the objectives of this study were to track historical sewage loading to Niven Lake in the sediment record using multiple proxies, while attempting to evaluate the effects of historical metal contamination, as well as the additional

stressor of recent climate warming known to affect this area (Schindler and Smol, 2006; Coleman et al., 2015).

Since only sparse qualitative observations of Niven Lake's pre-impact conditions exist, these paleoenvironmental data will place the possible recovery of Niven Lake in important historical context, allowing us to evaluate the extent of recovery. We hypothesized that the relatively short growing season and shallow depth of Niven Lake were important in determining the response of diatoms and chironomids to eutrophication and that the compounding effects of mining pollution and recent warming may result in a unique response not often recorded elsewhere in studies of Arctic eutrophication.

SITE DESCRIPTION

Niven Lake is approximately 0.07 km² in open-water surface area with a maximum depth of just over ~1.5 m, as measured in July of 2015 (Fig. 1). Niven Lake is currently in the middle of a residential subdivision and is surrounded by a walking path separated from the open water by some trees and 5–10 m of thick marshland. Macrophytes cover more than 95% of the lake's main basin from the bottom to the water surface, making the lake entirely littoral with very little pelagic habitat and only some small pockets of bare sediment. During the July 2015 sampling, the lake also had a bloom of filamentous green algae floating on its surface. Niven Lake currently freezes nearly to the bottom in winter. The lake has no fish, but in the summer it hosts many migrating birds traveling to Arctic breeding grounds, and some ducks nest in the marshes surrounding the water.

Niven Lake drains a catchment that is relatively large compared to its surface area: it covers almost 4 km², including Frame Lake from the west and runoff from the northern part of Yellowknife's downtown from the south. Niven flows into Back Bay (a portion of Yellowknife Bay of Great Slave Lake between Old Town and the mainland to the west) through a natural ravine at its northeastern edge that was modified when it became a sewage lagoon in 1948 (Heinke and Smith, 1986). During the first few years that the lake was used as a sewage lagoon, a dam was built at one end of a narrow reach in order to raise water levels and increase holding capacity. After sewage inputs ended in 1981, the dam was removed. The sewage inlet built in 1948 consisted of a utilidor that carried wastewater out into the lake from the approximate middle of the southeastern edge. In 1963, in response to deteriorating water quality and impending anoxia, a primary cell $(32 \times 18 \times 2.4 \text{ m})$ was built between Niven Lake and the sewage inlet in order to allow sludge to settle with clear overflow to the main basin (Heinke and Smith, 1986). Niven Lake is close to both historically operated gold mines in Yellowknife, with Giant Mine ~4 km to the North and Con Mine ~3 km to the south (Fig. 1).

METHODS

Water Chemistry

On 23 July 2015 and 12 July 2016, water samples were taken from just below the surface of Niven Lake at the approximate centre of the basin using Nalgene bottles. Surface water pH and specific conductance were also measured on site using a YSI probe. Water chemistry parameters were measured at the Taiga Environmental Laboratory in Yellowknife using the standard U.S. Environmental Protection Agency Methods and the Standard Methods for the Examination of Water and Wastewater (American Public Health Association, 2005). However, total phosphorus concentrations were measured using inductively coupled plasma mass spectroscopy (ICP-MS) because of the suspected interference of arsenate with the colorimetric signal of phosphate (a problem in the arsenic-rich lakes around Yellowknife), which can lead to artificially high measurements of phosphorus using standard colorimetric assays. A profile of dissolved oxygen, specific conductance, and water temperature was also taken using a YSI meter at approximate depths of 0 m (just below the surface), 0.5 m, and 0.9 m (near the sediment-water interface) on 23 July 2015.

Sediment Sampling and Dating

A 30 cm sediment core was retrieved from Niven Lake from the centre of the southwestern half of the basin on 23 July 2015, using a UWITEC[©] gravity corer (Uwitec, Mondsee, Austria). The core was extruded into 0.5 cm intervals using a modified Glew (1988) extruder. Subsamples of every second centimetre from 0 to 20 cm were freeze-dried and used for ²¹⁰Pb dating with an Ortec high-purity Germanium gamma spectrometer (Oak Ridge, Tennessee) at the University of Ottawa. The resultant radioactivity profiles were developed into a chronology using the Constant Rate of Supply (CRS) model (Appleby, 2001) with ScienTissiME software (ScheerSoftwareSolutions, Barry's Bay, Ontario). The CRS model was deemed appropriate because the organic-rich sewage inputs were highly likely to change sedimentation rates, and ²¹⁰Pb ages were verified using the circa 1963 peak in ¹³⁷Cs from the height of atomic bomb testing (see online Appendix 1: Fig. S1).

Sterols and Stanols

Sedimentary sterols and stanols were examined every 1 cm until 12 cm, and then every 2 cm until 20 cm depth to fully capture pre-sewage conditions according to the ²¹⁰Pb dates. Analytical methods were modified from Birk et al. (2012) and Cheng et al. (2016). Freeze-dried sediment (~0.1 g dry weight) samples were sonicated with 10 mL dichloromethane for 10 min with activated cleaned copper, and this procedure was repeated three

more times. All extracts were combined and concentrated to 1.0 mL under a gentle flow of nitrogen at room temperature. The concentrated extract was transferred to a 6 mL liquid chromatography solid phase extraction (LC-Si SPE) column, which was preconditioned with 6 mL of dichloromethane. The SPE columns were eluted with 30 mL dichloromethane for cleanup, which was concentrated to 1.0 mL under nitrogen. For derivatization, the eluted sterols and stanols were dried completely under a gentle flow of nitrogen before adding 100 µL 99:1 BSTFA+TMCS (N,O-Bis(trimethylsilyl) trifluoroacetamide and Trimethylchlorosilane), then heated at 60°C for two hours. We added 900 µL of toluene to the derivatized samples and 10 µL of an internal standard. Sterols and stanols were quantified by gas chromatography mass selective detector (GC-MSD) with a capillary column of Agilent 19091J-433 HP-5 5% phenyl methyl siloxane. The initial oven temperature was set at 80°C and initial time was 1.5 min, then equilibrated for 0.5 min. The oven ramp was set as follows: first an increase to 265°C at 12°C/min, then an increase to 288°C at 0.8°C/min, then an increase to 300°C at 10°C/min and kept at that temperature for 12 min. The transfer line temperature was 280°C (Cheng et al., 2016). For quality control, all GC-MSD results were amended to the internal standard d14-p-terphenyl done by MSD ChemStation D.02.00.275.

For every five samples, an experimental blank was run simultaneously. Cholesterol and stigmastanol were detected in some blanks with concentrations no more than 10% of samples. The blank values were subtracted from corresponding samples. Limit of quantification was defined as a signal-to-noise ratio of three. Signals below that ratio were regarded as not quantified and were discarded. The nine sterols measured were coprostanol $(5\beta$ -cholestan-3 β -ol), epicoprostanol $(5\beta$ -cholestan-3 α -ol), coprostanone (5_β-cholestan-3-one), cholesterol (cholest-5en-3-ol), 5α-cholestanol (5α-cholestan-3β-ol), cholestanone $(5\alpha$ -cholestan-3-one), stigmastanol $(5\alpha$ -stigmastan-3 β -ol), and sitosterol (β-sitosterol). Previously published diagnostic sterol ratios and their associated threshold values for determining human fecal contamination were compared with values calculated for the Niven Lake core, particularly during the period of known sewage inputs, in order to compare their effectiveness in tracking sewage inputs. We considered three ratios (reviewed by Furtula et al., 2012): coprostanol/(coprostanol+5 α -cholestanol), coprostanol/ cholesterol, and epicoprostanol/coprostanol. Values greater than 0.7 and below 0.3 have been considered either indicative or not indicative of human fecal contamination for coprostanol/(coprostanol+5α-cholestanol) (Grimalt et al., 1990; Carreira et al., 2004; Vane et al., 2010), while values between these criteria are considered ambiguous. Similarly, threshold values to indicate human fecal contamination are above 0.5 (yes) and below 0.3 (no) for coprostanol/cholesterol (Fattore et al., 1996; Patton and Reeves, 1999; Tse et al., 2014), and less than 0.2 (yes) and greater than 0.8 (no) for epicoprostanol/coprostanol (Froehner et al., 2009). Ratios for the Niven Lake sediment core were determined using sterol concentrations, and the sterol changes through time were described in percent relative abundances of the total sterol concentration.

Elemental Analysis and Stable Nitrogen Isotopes

Elemental and isotopic analyses of all samples were performed at the G.G. Hatch Stable Isotope Laboratory at the University of Ottawa, Ottawa, Ontario. Samples and standards were submitted to an elemental analysis (EA) to determine the elemental composition of carbon and nitrogen using the CE EA1110 Elemental Analyzer, using the detailed methods outlined in Brazeau et al. (2013). Sample amounts used for the isotopic analyses were based on the results of the EA. Sediments were weighed accordingly into tin capsules (\sim 3 mg) with 5 mg tungsten oxide (WO₃). Calibrated internal standards were prepared as a reference with every batch of samples. Samples were combusted at 1800°C in an elemental analyzer (EA 1110, CE Instruments, Italy) and the gases produced were run through an isotope ratio mass spectrometer (Delta-Plus Advantage IRMS, ThermoFinnigan, Germany) using a Conflo III Interface (Thermo, Germany). The data were reported using Delta notation (δ) in per mil (∞), which is defined as $\delta = [(Rx)]$ $(-R_{std})/R_{std}$ *1000, where R is the ratio of the abundance of the heavy to light isotope, "x" denotes sample, and "std" is an abbreviation for standard. The routine precision of the analyses was 0.20‰.

Spectrally Inferred Chlorophyll a

Sedimentary concentrations of chlorophyll-*a*, estimated using visual reflectance spectroscopy (VRS), were used to track trends in overall primary production of Niven Lake and thus its eutrophication during use as a sewage lagoon, as well as its possible recovery since 1981. The methodology generally followed Wolfe et al. (2006) and Michelutti et al. (2010), as reviewed by Michelutti and Smol (2016). Freezedried sediments were sieved using a 120 μ m mesh and placed in cuvettes to be run for absorbance using a FOSS NIR System Model 6500 spectrometer. The spectra in the range of 650–700 nm were analyzed for the area under the curve using RStudio[®] (version 1.0.136) every 1 cm from 0 to 15 cm, which approximates the concentration of chlorophyll-*a* and its main diagenetic products in mg/g (hereafter VRS-chla, Michelutti and Smol, 2016).

Diatoms and Chironomids

Approximately 0.2 g of wet sediments every cm from 0 to 15 cm were digested for subfossil diatom assemblages using standard methods described by Battarbee et al. (2001). Subsamples were treated with a 1:1 molar ratio solution of concentrated sulfuric acid (H_2SO_4) and nitric acid (HNO_3) and heated in a ~80°C water bath for two hours. Concentrated acid in the diatom slurries was

aspirated and rinsed over several days to reach a neutral pH, and various concentrations of the neutral slurry were plated and mounted on slides using Naphrax[®]. A minimum of 400 diatom valves were enumerated from each sample at $1000 \times$ magnification using primarily Krammer and Lange-Bertalot (1986–91). Zones of the diatom assemblage were determined using the cluster analysis technique, constrained incremental sum of squares (CONISS; Grimm, 1987), and the number of important zones was identified using broken stick analysis (Bennett, 1996). CONISS analyses were performed using the rioja package (Juggins, 2015) in RStudio[®] (version 1.0.136).

Subfossil chironomid assemblages were analyzed on the same sediment intervals as were the diatoms in order to capture pre- and post-sewage conditions. Following the methods described in Walker (2001), $\sim 0.02 - 0.05$ g of wet sediment was deflocculated in 80 mL of 5% potassium hydroxide (KOH) solution at \sim 70°C for 20–30 min and then rinsed through a 100 µm sieve into a beaker using deionized water. Sediments were picked for chironomid head capsules and Chaoborus mandibles, which were placed on slides that were mounted permanently using Entellan. The minimum of 50 head capsules suggested by Quinlan and Smol (2001) was exceeded for all intervals. Chironomid head capsules were identified to the species-type where possible using Larocque and Rolland (2006), Brooks et al. (2007), and Andersen et al. (2013). Taxa were plotted using C2 (version 1.7.4) and arranged by tribe or subtribe. CONISS analyses were also performed on the chironomid assemblage profile, but zones were found to be not important by broken stick analysis (Bennett, 1996).

Climate Data

Temperature data for the City of Yellowknife were obtained from Environment and Climate Change Canada. Mean annual temperature data were calculated for every year from 1944 to 2015 that had an average temperature for all 12 months (n = 47 years). We calculated mean summer temperature data every year from 1943 to 2015 that included average temperatures for June, July, and August (n = 58 years) and used these means to calculate the summer mean. We also calculated an overall average across each time period for comparison and assessed data for a directional trend over time using the linear regression function in Sigma Plot 10.0.

RESULTS

Water Chemistry

Niven Lake was hypereutrophic in July of 2015 and 2016 (Table 1) with an average unfiltered total phosphorus (TP-u) of 96 μ g/L and average total filtered nitrogen (TN-f) value of 1.72 mg/L. Water concentrations of chlorophyll-*a* were measured in July 2015 with a value of 2.35 μ g/L. The

TABLE 1. Lakewater concentrations of nutrients, major ions, and arsenic for Niven Lake taken on 23 July 2015 and on 12 July 2016. The dash indicates unavailable data.

Water chemistry	2015	2016
$TN-f (mg/L)^1$	1.78	1.66
$TN-u (mg/L)^1$	2.00	1.64
$TP-f(\mu g/L)^{1}$	93	49
$TP-u (\mu g/L)^1$	119	73
Chlorophyll a (µg/L)	2.35	-
DOC $(mg/L)^1$	28.2	25.3
Alkalinity (mg CaCO ₃ /L)	89.6	86
pН	9.98	9.89
Specific cond. $(\mu S/cm)^1$	569	561
Calcium (mg/L)	35.6	36.9
Sodium (mg/L)	52.6	51.9
Magnesium (mg/L)	19.7	23.6
Potassium (mg/L)	3.4	4.8
Sulfate (mg/L)	35	40
Chloride (mg/L)	110	104
Dissolved arsenic (µg/L)	47.0	44.1
Total arsenic (µg/L)	41.7	44.2

¹ TN = total nitrogen, TP = total phosphorus, "-u" indicates unfiltered measures, "-f" indicates filtered measures, DOC = dissolved organic carbon, and specific cond. = specific conductance.

average pH of both sampling times was 9.9, and the average specific conductance was 565 microSiemens per centimetre (μ S/cm), a level that was also reflected in the elevated concentrations of major ions in the water (Table 1). Finally, arsenic concentrations were notably high, with values in 2015 and 2016 of 40–50 µg/L compared to the drinking water-quality guideline of 10 µg/L (Health Canada, 2006) and the 5 µg/L guideline for the protection of aquatic life (Canadian Council of Ministers of the Environment, 2001).

The dissolved oxygen (DO) depth profiles from Niven Lake on 23 July 2015 showed a gradient from supersaturation at the water surface (DO = 10.3 mg/L, 105% saturation) to saturated at mid-depth 0.5 m below surface (DO = 7.7 mg/L, 85% saturation) to hypoxic conditions near the sediment-water interface 0.9 m below the surface (DO = 1.6 mg/L, 18% saturation) (Table 2). Specific conductance also showed a differential from the surface downwards, increasing by 200 μ S/cm from 266 μ S/cm to 466 μ S/cm, with the bottom waters approximately equal to mid-depth (0.5 m) values. A gradual change in temperature was also notable in Niven Lake with temperatures of 18°C, 17°C, and 16°C at 0, 0.5, and 0.9 m depth, respectively.

Sterols and Stable Nitrogen Isotopes

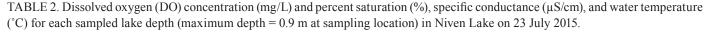
Both the pre- and post-industrial sterol and stanol profiles of Niven Lake were dominated by the quantified plant sterols, sitosterol and stigmastanol (Fig. 2). Over time, the dominance of these plant sterols switched from stigmastanol to sitosterol, and this change was particularly marked at ~1999 (3 cm). Prior to ~1950 (7.5 cm), the sterol and stanol sedimentary profile was relatively consistent, represented on average by 13% coprostanol, 2% epicoprostanol, 3% coprostanone, 7% cholesterol, 14% 5α-cholestanol, 1% cholestanone, 15% sitosterol and 45% stigmastanol (Fig. 2). At 6 cm (~1971), epicoprostanol and coprostanol both increased and represented ~22.5% of the total sterols, with coprostanol increasing from 10% to ~18%. This peak was short, and both stanols had declined by ~1981 (5 cm). Additionally, coprostanone peaked at 6 cm (~1971), reaching its maximum abundance of 4.2% through the sedimentary profile. A correction to sedimentary total carbon content did not alter these trends in human fecal markers.

The ratio of coprostanol/(coprostanol+5 α -cholestanol) peaked at 6 cm (~1971), reaching a maximum value of 0.56 (Fig. 2). The mean for this ratio was 0.48 prior to sewage disposal in Niven Lake and 0.36 after sewage diversion in the 1980s (at 5 cm depth), with all values remaining between the indicator values for the presence (> 0.7)and absence (< 0.3) of human fecal contamination. The coprostanol/cholesterol ratio was more variable through time, with ratios varying from 1.5 to 3.05, and all values prior to 4 cm (~1999) indicated human fecal contamination (> 0.5). However, from a coprostanol/cholesterol ratio equal to ~1.5 at 10 cm, there was a peak in values at 6 cm, consistent with peaks in other human fecal marker ratios (Fig. 2). Only the most modern sediments at 3 cm, 1 cm, and 0 cm had coprostanol/cholesterol values below the lower threshold (< 0.3), indicating no human fecal contamination. The epicoprostanol/coprostanol diagnostic ratio is less clear in capturing the period of sewage inputs to Niven Lake, with values of 0.15 in the pre-sewage part of the core and increasing to almost 0.2 during sewage inputs. Ratios below 0.2 indicate human fecal contamination, and values exceeded 0.2 at 4 cm (~1991), with a sharp spike to just over 0.3 and a decrease in the upper sediment intervals back to a value of $\sim 0.2 - 0.25$.

Stable isotopes of nitrogen (δ^{15} N) increased gradually across the entire core from the bottom (~1.5‰) to the surface (~4‰), with no notable changes during the period of wastewater inputs to Niven Lake (Fig. 2). This trend did not coincide with trends in the relative abundances of animal sterols but was more similar to the gradual increase of the phytosterol, sitosterol.

Diatoms and Chironomids

The diatom assemblage of Niven Lake was characterized by two distinct zones that are separated by the point of greatest turnover at 3-4 cm depth, or approximately the mid-1990s, as determined using CONISS and broken stick analysis (Fig. 3A). However, changes in diatom relative abundances began as early as 7-8 cm (~1940s). The earliest assemblage (8–15 cm) can be considered pre-impact, as this portion of the core was not affected by mining or municipal waste disposal. The pre-impact diatom assemblage of Niven Lake was co-dominated by the cosmopolitan species *Achnanthidium minutissimum*, as well as the epiphytic species *Brachysira neoexilis* and



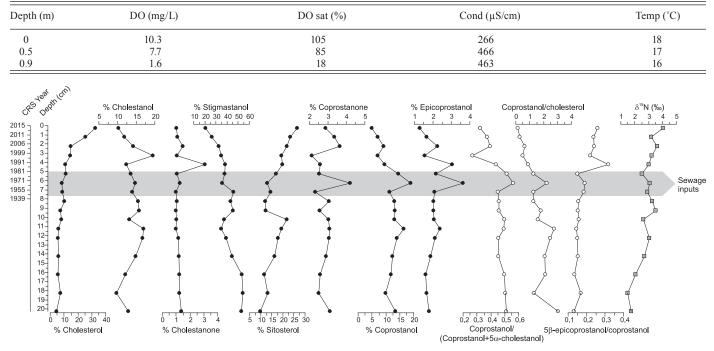


FIG. 2. Relative abundances (%) of sterols and stanols, as well as stable nitrogen isotopes ($\delta^{15}N$ in ‰), measured in the sediment core from Niven Lake (Yellowknife, Northwest Territories). The three lines with white circles show different diagnostic ratios for determining human-sourced fecal contamination in lake sediments (reviewed by Furtula et al., 2012). The shaded grey area represents the approximate duration of sewage inputs to Niven (1948–81).

Encyonopsis microcephala. Beginning around 8 cm, some of these taxa and some of the rare species began to show subtle relative decreases, and by 3 to 4 cm, all of these species had virtually disappeared from the record. *Navicula* taxa remained stable for the entire record.

Simultaneously, at ~8 cm, several species appeared (or appeared more commonly) at low relative abundances in the diatom assemblage, including *Fragilaria mesolepta*, *Nitzschia amphibia*, *Eolimna minima*, *Sellaphora seminulum*, *Planothidium lanceolata*, and *Stephanodiscus* species (primarily *S. hantzschii*). Most of these species dramatically increased above the 3-4 cm depth and became the dominant species that made up the second zone of the diatom stratigraphy from 3-4 cm (the mid-1990s) to the surface (2015). For example, *Fragilaria mesolepta* reached 40% relative abundance in the topmost interval. *Stephanodiscus* sp. had a peak relative abundance of ~5% around the 3 cm depth and decreased again thereafter.

Changes in sedimentary VRS-chla concentrations were subtle throughout the core (Fig. 3A). In the pre-impact sediments, VRS-chla concentrations maintained values around ~0.095 mg/g at 15 cm and slowly increased to ~0.11 mg/g by the approximate time of the onset of sewage inputs around 7.5 cm, or ~1950 (Fig. 3A). This slow increase continued through the period of sewage inputs, with the highest values throughout the entire record occurring at ~5 cm, or 1981, at the end of sewage discharge into Niven Lake. Subsequently, VRS-chla concentrations decreased back to values of ~0.095 mg/g by 3.5 cm (early 2000s) and

then remained stable until 1.5 cm ($\sim 2008-09$), at which point a secondary increase to just over 0.1 mg/g was noted in the most recent sediments.

The chironomid assemblage of Niven Lake was not dominated by any particular taxon, but instead had low abundances ($\sim 5\% - 10\%$) of 21 species types or groups (Fig. 3B). CONISS determinations showed that the largest groupings of the chironomid assemblage occurred between 3 and 4 cm, which was the same for the diatom assemblage. However, broken stick analysis (Bennett, 1996) indicated that groups in the chironomid assemblage were not more probable than random distributions, highlighting the subtle and gradual nature of any visible changes in the chironomid assemblage compared to the drastic changes seen in the diatoms. The most notable changes were decreases in the Tanytarsini by 3-4 cm (1990s), especially the cold-indicator, Micropsectra insignobilous-type. At the same time, an increase in Chironomus plumosustype to 5% relative abundance and a moderate increase in *Psectrocladius sordidellus/psilopterus*-type occurred. The abundance of chironomid remains in the core was calculated as head capsules per gram of dry sediment (HC/g dry sed) and gave an idea of the overall production of chironomids in Niven Lake through time. Chironomids were extremely abundant in the Niven Lake sediment core with ~3000 head capsules in 1 gram of dry sediment. A notable increase in chironomid production from ~3000 HC/g dry sed to ~5000 HC/g dry sed occurred at 6 cm (1969), followed by a sharp decline back to initial concentrations.

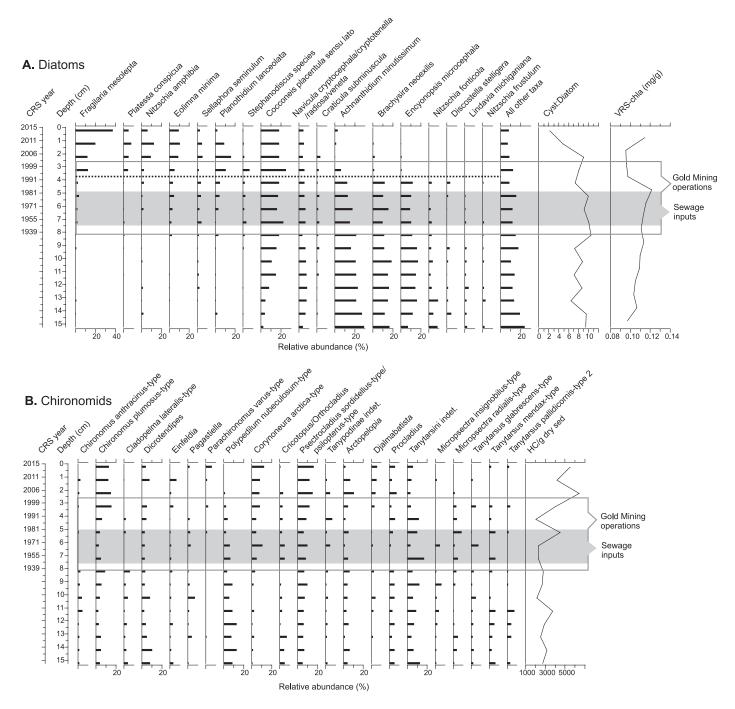


FIG. 3. A) The subfossil diatom assemblage (%) in the sediment core from Niven Lake (Yellowknife, Northwest Territories). Line graphs at the right show the chrysophyte cyst to diatom valve ratio and sedimentary concentrations of chlorophyll *a* (mg/g) measured using visual reflectance spectroscopy (VRS-chla). The dotted line indicates the transition between CONISS groupings. B) The subfossil chironomid assemblage (%) and the number of head capsules per gram of dry sediment (HC/g dry sed). The shaded grey area on each stratigraphy represents the approximate duration of sewage inputs to Niven Lake (1948–81), and the grey box outline shows the overlapping operational periods of the Con Mine (1938–2003) and Giant Mine (1948–2004).

Yellowknife Temperature Records

The mean annual temperature (MAT) of the City of Yellowknife from 1993 to 2015 was consistently above the average MAT from 1944 to 2015 (n = 47 years), and an increase in MAT over this time period was also significantly described by a positive linear regression

 $(R_{adj}^2 = 0.42, p < 0.0001, Fig. 4)$. A similar trend was also apparent for the mean summer temperature of Yellowknife with summer temperatures from 1996 onwards exceeding the ~70-year average from 1943 to 2015 (n = 58 years) (Fig. 4). Summer temperatures over this time were also significantly described by a positive linear regression $(R_{adj}^2 = 0.41, p < 0.0001, Fig. 4)$.

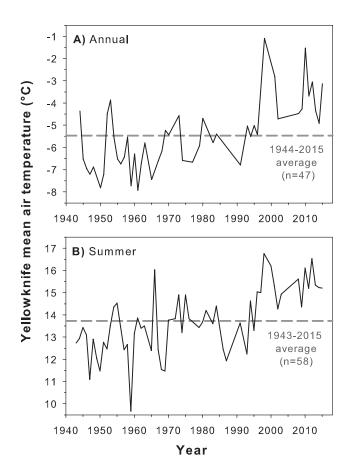


FIG. 4. Mean air temperatures for the City of Yellowknife (Northwest Territories) including A) annual means for select years between 1944 and 2015 and B) summer means of June, July, and August for select years between 1943 and 2015. Data were included for years that had all monthly mean air temperatures for annual averages (n = 47 years) or all June/July/August monthly means for summer averages (n = 58 years), and overall temperature averages for the time periods are shown as grey dashed lines. Data from Environment and Climate Change Canada.

DISCUSSION

Tracking Sewage Using Sterols and $\delta^{15}N$

The phytogenic sterol and stanol compounds, sitosterol (10%-20%) and stigmastanol (50%-60%), dominated the early sedimentary record (Fig. 2), indicating that Niven Lake was likely relatively productive or received abundant plant matter from its catchment before it was used for sewage disposal from 1948 to 1981. Surprisingly, coprostanol comprised on average 13% of the measured sterol composition of Niven's presumed pre-impact sediments (before 7.5 cm, or ~1950 according to our ²¹⁰Pb dates). This percentage may be considered high for pristine and uncontaminated sediments, where in situ production of coprostanol has been shown to be negligible (Nishimura, 1982; Leeming et al., 1996). Coprostanol is a product of cholesterol formed in the intestines, and humans have been found to excrete approximately 10 times as much coprostanol as other animals (Leeming et al., 1996), making large increases in coprostanol an effective

indicator of human fecal contamination in some instances. In addition, the values of two of the three previously published diagnostic sterol ratios (coprostanol/cholesterol and epicoprostanol/coprostanol) also suggest a human impact on Niven Lake in the pre-sewage sediments (see methods for threshold criteria). Our sedimentary sterol evidence of early human impact may be corroborated by the history of the Weledeh Yellowknives Dene First Nation, who documented the occupation of the area around Great Slave Lake by the T'satsaot'ine people for centuries before Yellowknife became a mining town in the 1930s (Weledeh Yellowknives Dene, 1997). In fact, the Niven Lake region was bisected by many traditional community trails that were used to move between camps, along shores, and as hunting trails (Weledeh Yellowknives Dene, 1997). Sedimentary sterol analysis has tracked prehistoric human activity elsewhere as well, including the arrival of humans ~2250 calendar years before 1950 in northern Norway, indicated by an abrupt increase in sedimentary coprostanol and its epimer epicoprostanol (D'Anjou et al., 2012).

The duration of sewage inputs to Niven Lake was also tracked by sedimentary sterols and stanols (Fig. 2). The 8% increase in the relative importance of coprostanol in Niven's sediments (to ~18% of the overall composition) between 7.5 and 5 cm (the 1950s to the 1980s) coincides with the main use of the lake as the primary (and, until 1975, the only) sewage lagoon of the City of Yellowknife from 1948 to 1981 (Heinke and Smith, 1986). Smaller increases (1% - 2%) also occurred at this time in coprostanone and epicoprostanol (Fig. 2), both of which are naturally reduced under low dissolved oxygen and thus were unsurprising, as hypoxia had been reported in Niven Lake during the sewage era (Heinke and Smith, 1986). Decreases in the relative abundances of coprostanone, coprostanol, and epicoprostanol occurred by 5 cm (~1981), matching the historical cessation of sewage inputs to Niven Lake. However, the sterol signal of sewage contamination may be muted in the sediment record because of the 1963 construction of a primary cell (a small dug-out area) between the sewage inflow and the main basin of Niven Lake, where solid waste settled out, allowing for a relatively clear overflow of water to the lake (Heinke and Smith, 1986). Sterol and stanols are hydrophobic and will rapidly attenuate in the water column by binding to organic matter (reviewed by Korosi et al., 2015), and thus it is likely that much of the sterol input may have resided in the sludge of the primary cell, with only some inputs making it into the sediment of Niven Lake. Despite this, however, the sediment record still appears to subtly track the known sewage inputs to Niven Lake, suggesting sterol analysis can track even small changes in the sediment record.

Trends in the ratio of coprostanol/(coprostanol+5 α cholestanol) best recorded fecal contamination of Niven Lake, peaking in value at 6 cm (~1971) and decreasing thereafter. The increase coincided with the height of sewage disposal to Niven Lake (Heinke and Smith, 1986), and the decrease may be indicative of the distribution of 50% of the sewage load into nearby Kam Lake from 1975 to 1981 (Squires, 1982). However, values for this ratio fell in the ambiguous range between previously published threshold values for determining human fecal matter contamination (Grimalt et al., 1990; Carreira et al., 2004; Vane et al., 2010), suggesting that these thresholds for coprostanol/(coprostanol+5a-cholestanol) cannot diagnose all sedimentary sewage contamination. The Niven Lake $coprostanol/(coprostanol+5\alpha-cholestanol)$ values likely remained in the ambiguous range because of the similar concentrations of coprostanol and 5α -cholestanol. However, the fairly stable relative abundance of 5a-cholestanol during sewage inputs and the nearly 10% increase in coprostanol signify that sterol analysis still tracked sewage inputs to Niven Lake. Our analysis of Niven Lake suggests that changes in coprostanol are the most reliable indicator of sewage inputs and should be prioritized above previously published sterol ratio thresholds.

Changes in the other two sterol ratios were less clear than the trend in coprostanol/(coprostanol+5 α -cholestanol). The lack of changes in these ratios may be due to variable concentrations of cholesterol, in the case of the coprostanol/ cholesterol ratio, and to increases in both epicoprostanol and coprostanol, in the case of the epicoprostanol/coprostanol ratio. As cholesterol is the most ubiquitous animal sterol and may come from various sources (including birds, Cheng et al., 2016), this ratio may be driven by other aspects of the environment in and around Niven Lake. The epicoprostanol/coprostanol ratio has also traditionally been used to indicate either the degree of treatment of sewage or its age in the environment (Mudge and Ball, 2006), as coprostanol can be reduced to epicoprostanol in anoxic conditions over time. Thus, sediments with combined high coprostanol/cholesterol and low epicoprostanol/coprostanol ratios should be a strong indication of active high sewage input, and such a combination of ratios peaked at 6 cm (~1971) in the sediments. Therefore, overall, our analysis of sedimentary sterols in Niven Lake highlighted that it is important to consider both relative changes in different sterol compounds, as well as changes in multiple diagnostic ratios, as much is still unknown about the dynamics of sterol compounds under various conditions in lakes. especially in northern regions (Korosi et al., 2015).

Stable nitrogen isotopes did not track sewage inputs to Niven Lake, showing no apparent trends during the period of sewage inputs, even though these isotopes have been shown to track sewage contamination in some freshwater systems (Vane et al., 2010). The $\delta^{15}N$ of Niven Lake's sediments remained low with values common in pristine and non-impacted sediments (Blais et al., 2005). Even though animal or sewage waste can have a relatively high $\delta^{15}N$ signature of more than 10‰ (Heaton, 1986), overall municipal wastewater can have a complex $\delta^{15}N$ signal because human excrement makes up only a component of the entire discharge, which also includes water from sinks and tubs, food scraps, and other domestic waste. The $\delta^{15}N$ in a lake is also mediated by primary production and other aspects of the N-cycle (Leng et al., 2005), which may drastically change with eutrophication. The unresponsive sedimentary $\delta^{15}N$ suggests that the use of sterol-based proxies may be more effective in tracking human wastewater inputs.

Effects of Sewage, Mining, and Climate Warming on Niven Lake

The early sediments of Niven Lake were dominated by high abundances of benthic epiphytic diatoms (*Brachysira neoexilis* and *Encyonopsis microcephala*) and macrophyteassociated littoral chironomids (*Polypedilum nubeculosum*type, *Psectrocladius* sp., and *Dicrotendipes*) (Fig. 3), substantiating the high proportions of phytogenic sterols in this portion of the core (Fig. 2). Slowly increasing relative abundances of *Cocconeis placentula*, a reliable diatom indicator of macrophytes and higher total phosphorus (Reavie and Smol, 1997; Vermaire et al., 2011), at the bottom of the core suggested that Niven Lake was a naturally productive and macrophyte-dominated system, which according to our sterol analysis may have been the result of humans in the area (Fig. 2).

The species characteristic of the pre-impact diatom assemblage in Niven Lake began decreasing in relative importance in the 1940s (7–8 cm), and more substantially by the mid-1990s (3-4 cm) (Fig. 3A). During the sewage era, the pre-impact diatom assemblage was replaced by taxa characteristic of higher nutrient concentrations or heavy organic matter pollution, such as Nitzschia amphibia, Eolimna minima, Sellaphora seminulum, and Stephanodiscus hantzschii (Kelly et al., 2005). S. hantzschii, the main species comprising Niven Lake's Stephanodiscus group and a well-established indicator of eutrophication (Hall and Smol, 1992, 2010; Hadley et al., 2010; Reavie and Kireta, 2015) appeared at ~5% relative abundance around the time of sewage inputs and declined thereafter. However, the increase in planktonic *Stephanodiscus* species in Niven Lake was somewhat muted, as is common for diatoms experiencing eutrophication in the Arctic, where ice cover, a short growing season, and colder temperatures may override the effects of nutrient additions (Douglas and Smol, 2000; Michelutti et al., 2007; Stewart et al., 2014). In addition, Niven Lake is very shallow and dominated by aquatic macrophytes—a limnological setting in which various feedback mechanisms of vegetation (e.g., stabilizing sediments, providing shelter to zooplankton grazers) provide resilience to the more striking eutrophicationrelated ecological changes (e.g., algal blooms) that are typically recorded in deeper lakes (Scheffer, 1998). However, once a threshold of nutrient loading is crossed, this resilience can be lost, and the negative consequences of eutrophication can occur (Scheffer, 1998). In fact, given Niven Lake's shallow and macrophyte-dominated nature, the increase in the planktonic S. hantzschii was perhaps more ecologically significant than the percentage data may indicate. Similarly, there were virtually no changes in the chironomid species assemblage during the period of sewage inputs, which has also been noted elsewhere in shallow eutrophic Arctic sites (Stewart et al., 2013, 2014). Only very subtle increases in VRS-chla (an indicator of overall primary production) were detected in the sediments representative of the sewage era, which may also be due to the resistance of shallow macrophyte-dominated systems to algal blooms and the influence of the cold climate.

In contrast, the major turnover in the diatom assemblage occurred later in the 1990s (at 3 to 4 cm), a decade after the end of sewage inputs (Fig. 3A), according to our CONISS (Grimm, 1987) and broken stick (Bennett, 1996) analyses. This change consisted of increases in the meso- to eutrophic taxon, Fragilaria mesolepta, as well as hypereutrophic species, N. amphibia, E. minima, and S. seminulum (Kelly et al., 2005). The turnover of the assemblage to these species suggest that Niven Lake exhibited a further signal of eutrophication after the cessation of sewage inputs in 1981, a change that was more drastic than those during the onset or height of wastewater discharge. Additional evidence of the post-sewage eutrophication of Niven Lake includes recent increases in sedimentary VRS-chla (overall primary production) in the early 2000s, as well as increases in Psectrocladius, a strongly macrophyte-associated chironomid genus, which suggests further expansion of macrophyte growth in Niven Lake from 1991 (4 cm) onwards. Furthermore, modern phosphorus concentrations from Niven Lake in 2015 and 2016 showed that the lake was still hypereutrophic (Table 1), and the current lakewater pH of nearly 10 was elevated compared to the circumneutral pH of Niven Lake in 1990 (Ferguson Simek Clark, 1990a). A recent pH higher than in 1990 may be a direct result of increased primary production and associated CO₂ uptake that would have shifted the pH buffering reaction to the alkaline.

The continued eutrophication of Niven Lake into the 1990s and 2000s could be linked to internal phosphorus loading, or the release of phosphorus from the sediments, which can occur under low oxygen conditions and create a positive feedback cycle (Orihel et al., 2017). The more than twofold increase in the hypoxia- and anoxiatolerant species, Chironomus plumosus-type, by 1991 (4 cm) suggests that oxygen depletion in Niven Lake was likely occurring after the end of sewage inputs. Our July 2015 water column depth profile of dissolved oxygen demonstrated decreasing concentrations and saturation with depth, where hypoxic conditions (1.6 mg/L) prevailed near the sediment-water interface (Table 2). Biological oxygen demand (BOD) measurements in the 1960s indicated that Niven Lake had also previously been reported as on the verge of becoming anaerobic (Heinke and Smith, 1986). Furthermore, the increase in the anaerobic degradation products of coprostanol, such as epicoprostanol, in the sediment record at this time (Fig. 2) supports the likelihood that Niven Lake had low oxygen conditions during the height of sewage inputs and in the decades after. As Niven Lake was dominated by macrophyte cover from

the sediments to the water surface during sampling, it may be that, despite its shallow depth, Niven experienced reduced water-column mixing because of the stagnating effect of extensive macrophyte growth throughout the lake, potentially leading to oxygen depletion (and possibly internal phosphorus loading) lower in the water column.

The response of diatoms and chironomids in the 1990s indicative of post-sewage eutrophication could have been facilitated and/or exacerbated by recent climate warming, as there have been notable increases in mean annual and mean summer air temperatures over the past \sim 70 years (Fig. 4). In particular, annual and summer temperatures remained consistently above the 70-year average by the 1990s, the timing of which agrees with other climatedriven regional changes such as lake expansion occurring post-1986 in the Mackenzie Bison sanctuary southwest of Yellowknife (Korosi et al., 2017). Recent climate warming has reduced ice cover in Arctic lakes and prolonged the growing season in sensitive Arctic regions, leading to related changes in lake functioning (Douglas et al., 1994; Griffiths et al., 2017). A longer and warmer growing season for Niven Lake may have promoted greater lake stagnation (i.e., reduced mixing of dissolved oxygen) and algal production (increasing BOD), exacerbating the cycle of anoxia and possible internal phosphorus loading that may be inferred from our multiple paleo-proxies.

A critical review of internal phosphorus loading in Canadian lakes has suggested that recent climate warming may be an important driver of the release of phosphorus from sediments, even in lakes that continuously or repeatedly mix throughout the year, because of an increase in thermal stratification events (Orihel et al., 2017). Our point measurements of oxygen and specific conductance in Niven Lake on 23 July 2015 displayed a gradient in values over less than a metre of depth, supporting the idea that stratification may facilitate the conditions under which internal phosphorus loading may occur. Lakewater pH was also found to be one of the three most important drivers of internal phosphorus loading in Canadian lakes (in addition to oxygen and trophic status), with alkaline conditions promoting the release of phosphorus from sediments (Orihel et al., 2017). Niven Lake was alkaline in 2015 and 2016, with an average pH of 9.9. meaning that in addition to the measured hypoxia and hypereutrophic conditions, Niven Lake was a likely candidate for the occurrence of internal phosphorus loading according to the meta-analysis of Canadian lakes (Orihel et al., 2017). A paucity of data on the prevalence of internal phosphorus loading exists for Arctic lakes (Orihel et al., 2017), though our data suggest that this process may be an important consideration in shallow and eutrophic Arctic lakes.

To further complicate matters, some biological changes in Niven Lake were also indicative of metal pollution, namely from gold mining and its consequent release of arsenic trioxide dust to the atmosphere (Indian and Northern Affairs Canada, 2007). Niven Lake was affected by gold mining activities in the area, as the arsenic concentration in the water was elevated at values of ~60 μ g/L in 1990 (Ferguson Simek Clark, 1990a). Our modern water chemistry data also documented legacy pollution from regional gold mining, with total arsenic concentrations between 41 and 44 μ g/L (Table 1), which were much higher than the arsenic guideline of 5 μ g/L for the protection of aquatic life (Canadian Council of Ministers of the Environment, 2001). For Niven Lake, legacy arsenic contamination may also be controlled by the hypoxia/anoxia associated with its use as a sewage lagoon, since arsenic can be released from the sediments under anoxic conditions (Andrade et al., 2010).

Niven Lake was first used as a sewage lagoon during the same year (1948) that the Giant Mine opened. The onset of the increases in E. minima and S. seminulum, which may indicate eutrophication because of their high TP optima (Kelly et al., 2005), may also reflect mining pollution, as both taxa have been previously associated with tolerating metal pollution (Pérès et al., 1997; Morin et al., 2008). Furthermore, Niven Lake sediments tracked decreases in the Tanytarsini, which also declined in Pocket Lake on Giant Mine property, a lake severely affected by atmospheric metal contamination (Thienpont et al., 2016), as well as in other studies of mining contamination (Ilyashuk et al., 2003; Doig et al., 2015). Tanytarsus species, in particular, all of which were effectively lost to the Niven Lake assemblage by the end of mining operations in 1999, have been shown to be intolerant to trace metal contamination (Johnson et al., 1992) (Fig. 3B; Indian and Northern Affairs Canada, 2007). Interestingly, however, many of the *Tanytarsus* species-types in the Niven Lake core also have high dissolved oxygen optima (Luoto and Salonen, 2010), low temperature optima (Larocque and Rolland, 2006), or both, suggesting that these species likely experienced the compounding stressors of not only trace metal pollution, but also anoxia and recent climate warming. Finally, the relative increase in Psectrocladius may indicate that macrophyte growth increased in Niven Lake in the 1990s, but it was also noted to increase and co-dominate the chironomid assemblage of highly impacted Pocket Lake during mining operations (Thienpont et al., 2016). This makes *Psectrocladius* a likely candidate for tolerating the metal pollution of the Giant Mine in this region.

Recovery in Niven Lake

Some recovery from eutrophication and arsenic contamination is evident in the sediment record and water chemistry of Niven Lake. For example, our total filtered nitrogen (TN-f) measurements in July 2015 and 2016 of 1.78 and 1.66 mg/L, respectively, are lower than ammonium-N measurements (4.58 mg/L, an average of four sampling sites) made on 23 June 1990 during an environmental survey of Niven Lake (Ferguson Simek Clark, 1990a). Though these measurements represent different fractions of the nitrogen in Niven Lake, ammonium-N comprises

part of TN-f, and thus some chemical recovery of the lake is evident. The total phosphorus (TP) concentrations from June 1990 were extremely high (~3000 µg/L) (Ferguson Simek Clark, 1990a), even for Arctic sewage lagoons (Douglas and Smol, 2000) and may have been affected by measurement error. High arsenic concentrations may interfere with the measurement of phosphorus in these systems, as arsenate and phosphate have the same colorimetric effect, yielding TP values that are artificially high when measured using standard colorimetric assays. Therefore, we measured phosphorus concentrations in Niven Lake using ICP-MS (see water chemistry methods) and cannot identify the extent of reductions in phosphorus concentrations since the 1990 sampling event. Moreover, Niven Lake may be experiencing internal phosphorus loading from the sediments to the water column, which can delay chemical and biological recovery from eutrophication (Orihel et al., 2017). The lake also had a decrease in the concentration of arsenic from 65 µg/L in 1990 (Ferguson Simek Clark, 1990a) to $\sim 40 \ \mu g/L$ in 2015 (Table 1), reflecting the end of gold mining operations in the area.

The increase in the diatom F. mesolepta to $\sim 40\%$ relative abundance near the surface of the Niven Lake sediment core may indicate moderate recovery from eutrophication, as this species has a lower TP optimum compared to hypereutrophic species, N. amphibia, E. minima, and S. seminulum (Kelly et al., 2005), in the years immediately prior (the 1990s). This recovery was also supported by a subtle decrease in the VRS-chla concentrations after 5 cm or ~1981 (Fig. 3A), tracking a decrease in the overall primary production, though it did again increase in the early 2000s. However, Niven Lake has clearly not returned to pre-impact conditions in the past 35 years since the end of sewage inputs. Here, the interplay of eutrophication, anoxia, and arsenic contamination is evident in the sediment record, and the added "threat multiplier" of climate warming (Smol, 2010) may be a barrier to the full recovery of Niven Lake from eutrophication, as has been the case in other heavily human-impacted Arctic lakes (Moiseenko et al., 2009; Antoniades et al., 2011).

CONCLUSIONS

Niven Lake's use as a sewage lagoon for three decades was tracked in the sediments, albeit subtly, by the deposition of higher amounts of coprostanol, which is considered the best indicator of human fecal contamination (Korosi et al., 2015). The analysis of multiple sterol ratios indicated that previously published threshold values do not hold true during the known period of sewage inputs to Niven Lake. The sewage inputs were best represented by the trend in coprostanol/(coprostanol+5 α -cholestanol); however, changes in the relative abundance of coprostanol appear to be the most reliable indicator of human sewage contamination and should be prioritized above diagnostic sterol ratios. During the period of sewage inputs, subfossil diatom assemblages tracked sewage inputs with subtle increases in planktonic *Stephanodiscus* species, and in particular the well-known eutrophication indicator, *S. hantzschii* (Reavie and Kireta, 2015). The chironomid assemblage demonstrated little change during the time of sewage inputs, which together with the subtle shifts in the diatoms, equated to a muted response typical of Arctic systems that are generally controlled by the overarching effect of a short growing season and prolonged ice cover (Douglas and Smol, 2000; Antoniades et al., 2011). In addition, shallow lakes dominated by aquatic macrophytes, such as Niven Lake, are also known to often be resistant to threshold-type changes linked to eutrophication as a result of a variety of feedback mechanisms (Scheffer, 1998).

Interestingly, the greatest period of biotic change in Niven Lake occurred in the 1990s, approximately a decade after sewage inputs ended, coinciding with the onset of the warmest years on record for Yellowknife. The nature of the marked changes in diatoms and chironomids were consistent with lagging symptoms of eutrophication such as oxygen depletion and internal phosphorus loading, both of which may have been facilitated by climate warming. A longer growing season and warmer temperatures can promote the development of anoxia and thus the positive feedback cycle of internal phosphorus release, causing a lag in recovery despite the end of sewage inputs. Finally, changes in both the diatoms and chironomids were also consistent with metal(loid) pollution, as Niven Lake was affected by arsenic contamination from the nearby Giant Mine. Overall, Niven Lake may be showing some signs of chemical recovery from sewage loading since the early 1990s, but the additional stressor of recent climate warming appears to have complicated biological recovery as the lake experiences a novel climatic regime, as well as legacy metal pollution. The lagging recovery of Niven Lake from eutrophication may be due primarily to internal phosphorus loading, a problem that occurs across Canada and is particularly poorly understood in the Arctic (Orihel et al., 2017). Overall, this work adds to the growing body of research that suggests the management of eutrophic Arctic freshwaters must consider the effects of recent warming, particularly with regard to complications such as internal phosphorus loading.

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APPENDIX 1: SEDIMENT CHRONOLOGY

A supplementary file to the online version of this article is available at:

http://arctic.journalhosting.ucalgary.ca/arctic/index.php/ arctic/rt/suppFiles/4720/0

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