



# Bioaccumulation of Tl in otoliths of Trout-perch (*Percopsis omiscomaycus*) from the Athabasca River, upstream and downstream of bitumen mining and upgrading<sup>☆</sup>

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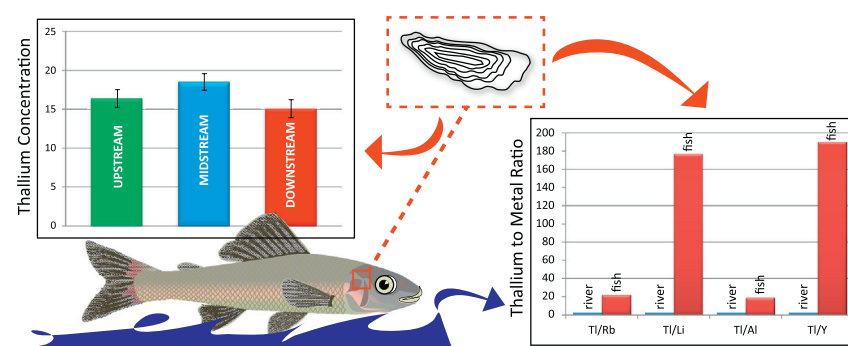
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## HIGHLIGHTS

- Concentrations of trace elements were determined in fish otoliths using ICP-QMS.
- Compared to river water, Tl shows the greatest enrichment of any element.
- Thallium dissolved in the Athabasca River is at natural, background levels.
- The enrichment of Tl in the fish otoliths is a natural process.
- There is no significant difference in enrichment upstream or downstream of industry.

## GRAPHICAL ABSTRACT



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## ABSTRACT

It has been suggested that open pit mining and upgrading of bitumen in northern Alberta releases Tl and other potentially toxic elements to the Athabasca River and its watershed. We examined Tl and other trace elements in otoliths of Trout-perch (*Percopsis omiscomaycus*), a non-migratory fish species, collected along the Athabasca River. Otoliths were analyzed using ICP-QMS, following acid digestion, in the metal-free, ultraclean SWAMP laboratory. Compared to their average abundance in the dissolved (<0.45  $\mu\text{m}$ ) fraction of Athabasca River, Tl showed the greatest enrichment in otoliths of any of the trace elements, with enrichments decreasing in the order Tl, Sr, Mn, Zn, Ba, Th, Ni, Rb, Fe, Al, Cr, Ni, Cu, Pb, Co, Li, Y, V, and Mo. Normalizing Tl in the otoliths to the concentrations of lithophile elements such as Li, Rb, Al or Y in the same tissue reveals average enrichments of 177, 22, 19 and 190 times, respectively, relative to the corresponding ratios in the water. None of the element concentrations (Tl, Li, Rb, Al, Y) or ratios were significantly greater downstream of industry compared to upstream. This natural bioaccumulation of Tl most likely reflects the similarity in geochemical and biological properties of  $\text{Tl}^+$  and  $\text{K}^+$ .

<sup>☆</sup> The authors declare no competing financial interest

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Summary of main findings: Thallium is enriched in fish otoliths, relative to the chemical composition of the river, to the same degree both upstream and downstream of industry.

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## 1. Introduction

Based upon snowpack sampling, it was suggested that open pit mining and upgrading of bitumen in northern Alberta “releases the 13 elements considered priority pollutants (PPE) under the Environmental Protection Agency’s Clean Water Act, via air, soil and water, to the Athabasca River and its watershed” (Kelly et al., 2010, PNAS 107:16178, line 1). The priority pollutants listed by Kelly et al. (2010) refer to Ag, As, Be, Cd, Cr, Cu, Hg, Ni, Pb, Sb, Se, Tl and Zn. The appearance of Tl on this list of elements of concern is especially disconcerting, as it is more toxic to humans than either Cd, Hg or Pb (Peter and Viraraghavan, 2005). Subsequent studies of precipitation in the area have further extended the list of elements of concern to many other trace metals and metalloids (Bari et al., 2014; Guéguen et al., 2016). Fish is an important component of the traditional diet of First Nations communities in northern Alberta, and concerns about fish health (Tetreault et al., 2003; Schwab et al., 2015; Arens et al., 2017; Parrott et al., 2018) as well as fish taste (Barona et al., 2011) are of particular importance. Concerns have also been expressed about impacts of trace elements on other aquatic organisms including invertebrate communities (Gerner et al., 2017) and mussels (Pilote et al., 2018). Examination of existing, long-term monitoring data revealed elevated concentrations of Al and Cu in the Athabasca River during periods of episodic acidification (pH < 6.5) at snowmelt (Fiera (Biological Consulting Ltd), 2013). Recently it was suggested that Tl and other trace elements are similarly affected (Alexander et al., 2017).

As part of a multi-disciplinary study on the physics, chemistry, and biology of groundwater-surface interactions in the lower reaches of the Athabasca River, we collected Trout-perch (*Percopsis omiscomaycus*) to explore its use as a biomonitor of potentially toxic trace elements; early work had suggested that this fish could be used as a sentinel species for studying environmental change in this watershed (Spafford, 1999). Also known as the Sand Minnow, and with features resembling both the trout and the perch (Kocovsky et al., 2014), this small fish (typically not >10 cm long) is non-migratory (Natureserve, 2013), providing an opportunity to use the fish to indicate water quality conditions at discrete locations along the river. Here, we examine the trace element chemistry of otoliths, the small, primarily calcium carbonate (aragonite) structures that form extracellularly within the inner ear of the fish (Degens et al., 1969; Campana, 1999). Otoliths are metabolically inert so that otolith material is not reabsorbed after deposition, and formation occurs continually in concentric layers of proteins and calcium carbonate about the nucleus (Campana and Neilson, 1985). Because of these characteristics, some trace elements, primarily those that are non-essential to fish, have been found to reflect environmental levels (Milton and Chenery, 2001; Arslan and Secor, 2005; Halden and Friedrich, 2008; Friedrich and Halden, 2010, 2011; Arnold et al., 2015; Thomas et al., 2017).

## 2. Materials and methods

### 2.1. Study sites

Trout-perch (*Percopsis omiscomaycus*) were collected from 12 sites on the Athabasca and 2 sites on the Clearwater Rivers during October of 2014. Fish harvest locations were based on an earlier geophysical survey of terrain conductivity: this had identified zones of elevated conductivity which are thought to reflect inputs of saline groundwaters of natural and/or industrial origin which occur commonly along the

Athabasca River between Fort McMurray and the Firebag River (Gibson et al., 2011). While the fish collection was taking place, a water sampling campaign was underway at the same sites on the river, with much of the trace element data already published (Cuss et al., 2018; Donner et al., 2017; Javed et al., 2017; Shotyk et al., 2017a).

### 2.2. Field collection

Trout-perch were collected using a 20 ft Smith-Root electrofishing boat (Model SR-20H) equipped with a 7.5 GPP electrofishing system by conducting multiple transects of approximately 1 km in length starting from the site locations shown in Fig. 1. The GPS coordinates of the sites are provided in the Supporting information (SI Table 1). Along with fish sampling, water temperature, turbidity, conductivity and pH were measured. Trout-perch were euthanized in accordance with an approved animal care protocol (AUP00001111), frozen at  $-20^{\circ}\text{C}$ , and shipped back to the University of Alberta for processing.

### 2.3. Sample preparation

For work on trace elements, 56 Trout-perch were defrosted in the lab, then measured for fork length and weight. Sagittal otoliths were extracted, cleaned with deionized water, and allowed to air dry overnight. No tumors were found in any of the fish. All subsequent sample handling and analysis was undertaken in the metal-free, ultraclean SWAMP lab, within polypropylene, Class 100 clean air cabinets (including the autosampler for the ICP-MS), using procedures similar to those described elsewhere (Shotyk et al., 2017a) for the study of trace elements in the dissolved fraction of the Athabasca River. After weighing, otolith samples were transferred to acid-cleaned, 4 ml PFA (perfluoroalkoxy alkane) tubes, then 200  $\mu\text{l}$  of concentrated nitric acid was added and allowed to react for 30 min. The acid employed was distilled twice using sub-boiling distillation in high purity quartz (Duopur, MLS, Leutkirch, Germany). After adding 800  $\mu\text{l}$  of DI/RO water (18.2 M $\Omega$ ·cm; Milli-Q Element, Millipore, USA), the samples were further diluted to contain 1 mg/ml, then analyzed using ICP-MS.

### 2.4. Analysis of trace elements using ICP-MS

Samples were analyzed using a quadrupole ICP-MS (ICAPQc, Thermo Fisher Scientific, Waltham MA, USA) in Kinetic Energy Discrimination mode, with helium (He) as the collision gas. Stock standard solutions were purchased from Spex CertiPrep (Metuchen, NJ, USA). Multi-element solutions (1 & 2A) as well as single element solutions (Mo, Bi, Re & Sb) were diluted as appropriate to create the calibration curves. A linear regression was confirmed for all selected isotopes ( $R^2 > 0.99$ ). Indium-115 was used as the internal standard, with instrument parameters of 80 sweeps and 30 ms dwell time for all isotopes.

### 2.5. Limits of detection, accuracy, and precision

The limits of detection (LODs) for trace elements in otoliths were based on the daily instrument response (cps) to diluent blanks (2% HNO<sub>3</sub>). For each isotope, the LOD was calculated using the average instrument response (cps) from 5 blank replicates plus 3 times the associated standard deviation. The mass/volume LODs were converted to mass/mass LODs by applying the sample dilution factor. The LODs obtained ( $\mu\text{g}/\text{kg}$ ) were Al, 480; Ba, 3.1; Co, 0.7; Cr, 2.1; Cu, 15; Fe, 230; Li, 1.1; Mn, 3.2; Ni, 17; Pb, 0.2; Rb, 0.9; Sr, 5.1; Tl, 0.6; V, 1.6; Y, 0.2; Zn, 210.

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