

# **Arsenic Speciation in Mine Tailings and Co-existing Pore Water, Lower Seal Harbour Gold District, Nova Scotia, Canada**

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## **ABSTRACT**

Environmental impacts from historical mining and milling of lode gold (Au) deposits in Nova Scotia include elevated arsenic (As) concentrations within the tailings and in downstream receiving environments. The Lower Seal Harbour Gold District (LSH) operated from 1904 to the mid-1940s and produced approximately 395,000 tonnes of tailings. This study examined As speciation in tailings and associated pore waters to better understand the processes that control the mobility of As from these mine wastes.

Within LSH, three geographically and geochemically distinct sample locations were chosen to collect tailings and pore waters, including (1) sub-aerially exposed tailings; (2) shallowly submerged tailings; and (3) tailings that are present in the intertidal zone of Seal Harbour, 2 km downstream of the milling operations.

Arsenic is present in the tailings at Sites 1, 2 and 3 at maximum concentrations of 9,200, 12,000 and 1,040 mg/kg, respectively. Elevated concentrations of As corresponded to either arsenopyrite-bearing sections of the tailings cores, or intervals with abundant As-bearing secondary phases. Pore water analyses indicate high concentrations of As and pH values ranging from 6.2 to 8.0.

Results from this study have helped to clarify the environmental risks associated with these historical mine wastes, and will support better informed land-management decisions.

Key Words: arsenic, arsenopyrite, gold mines, health risks, land-management, tailings

## **Introduction**

Historical mining and milling of lode Au deposits in mainland Nova Scotia from 1862 to the mid-1940s generated more than 3,000,000 tonnes of tailings containing high concentrations of both As and mercury (Hg) (Parsons *et al.* 2004). Tailings from these historic Au mining operations were typically disposed directly into local rivers, swamps, lakes and the ocean (Smith *et al.* 2005). The potential health hazards associated with these mine wastes were first recognized in 1976, when a resident of the Waverley Gold District near Halifax was diagnosed with chronic As poisoning after drinking well water contaminated with tailings runoff (Hindmarsh *et al.* 1977). Since that time, expanding residential developments and recreational activities have continued to increase the likelihood of human exposure to these mine wastes.

In response to earlier studies of As and/or Hg contamination at Au districts throughout Nova Scotia, a multidisciplinary study led by Natural Resources Canada (NRCan) was initiated in 2003 to (1) determine the concentrations, distribution, and speciation of elements near selected mine sites; (2) identify and characterize processes that control the release of elements from the tailings; and (3) quantify the off-site transport of metal(oid)s from the mine wastes, and the transformation and fate of these elements in receiving environments. Detailed speciation and characterization studies were required to better resolve the form, nature and distribution of As in the tailings. Results from studies by NRCan and partners show

significant enrichment of As in the mine tailings (9 mg/kg to 31 wt. %; median 2550 mg/kg;  $n = 482$ ) and associated surface waters (range: 0.2–6580  $\mu\text{g/L}$ ; median 117  $\mu\text{g/L}$ ;  $n = 181$ ) (Parsons *et al.* 2004). These results led to the formation of the Historic Gold Mines Advisory Committee in 2005, which continues to evaluate the potential ecological and human health risks associated with Au mines throughout Nova Scotia, and to develop recommendations for future management of these tailings sites (<http://www.gov.ns.ca/nse/contaminatedsites/goldmines.asp>). Detailed investigations in recent years have clarified the mineralogy and bioaccessibility of As in the tailings (e.g. Walker *et al.* 2009; Meunier *et al.* 2010; Corriveau *et al.* 2011a, 2011b; DeSisto *et al.* 2011; Percival *et al.* in press) and have led to better informed land-management decisions.

The Lower Seal Harbour Gold District (LSH) is a past-producing mine district located along the eastern shore of Nova Scotia (Figure 1). The lode Au deposit at LSH consists of structurally controlled auriferous quartz veins occurring in anticlines and preferentially confined to the lower boundary between the Goldenville Group and the Halifax Group of the Cambro-Ordovician Meguma Supergroup (Malcolm 1929). The mine operated for approximately 40 years during the first half of the 20<sup>th</sup> century. Ore was processed using a 10-stamp mill and Hg amalgamation until 1936, at which time a 181-tonne-per-day cyanide plant was constructed for the treatment of ore using a combination of cyanide methods and barrel amalgamation (Roach 1937, 1940). Approximately 395,000 tonnes of tailings produced during these operations (Blakeman 1978) were slurried directly into surrounding swamps and streams and now extend more than 2 km downstream to where the local watercourse, West Brook, carries tailings and runoff into the Atlantic Ocean at Seal Harbour (Figure 2). Evidence of recreational clam digging was observed in 2003 in the intertidal zone where tailings occur. Studies in 2004 and 2005 confirmed that high concentrations of As are present in bivalve shellfish from this area relative to background sites (Koch *et al.* 2007). In 2011, the LSH property was acquired by a junior mining company, which is now compiling historical records to determine potential Au resources and identify drilling targets (Beatrix Ventures Inc. 2011).

The mineral composition of the ore body at LSH, as summarized by Smith *et al.* (2005), consists of smoky grey quartz veins and grey slate with arsenopyrite (up to 5 wt.%) and minor amounts of galena, pyrite, pyrrhotite, chalcopyrite and sphalerite. Calcite is disseminated in the veins and surrounding wallrock and feldspar is abundant. Other minerals associated with the veins include iron oxides (Fe-oxides), silicates and other minor mineral phases (Goodwin 1935; Newbury 1974; Sangster 1990; Ryan and Smith 1998). The abundance of arsenopyrite in the ore results in high As concentrations in the tailings.

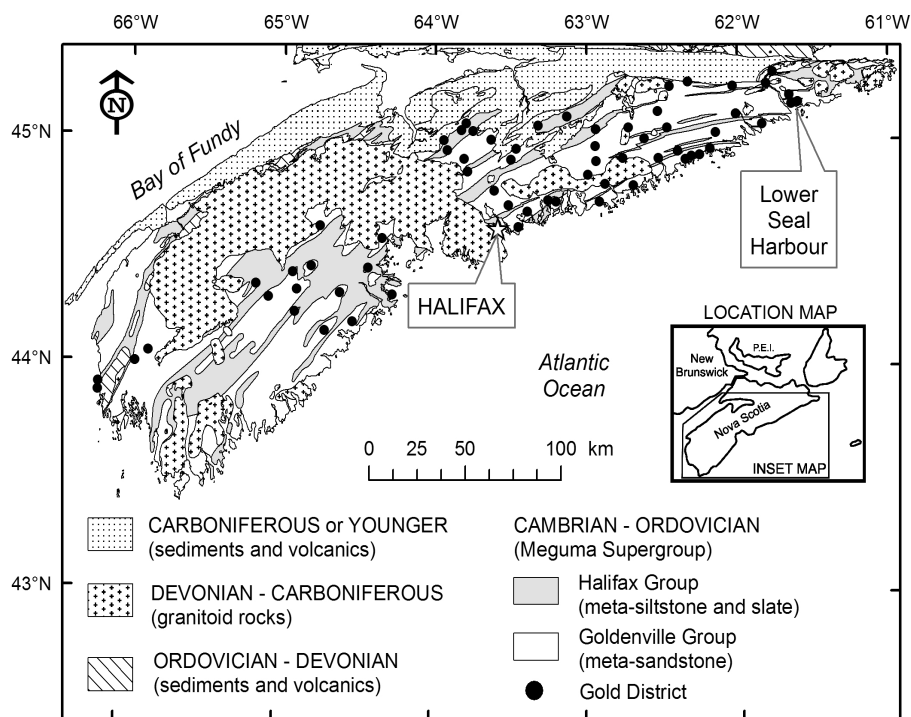


Figure 1. Location map of Lower Seal Harbour.

The main objective of this study was to examine the speciation of As in tailings and pore water in different environmental settings to better understand the processes that control the mobility of As from these mine wastes. Detailed examination of the tailings, interstitial waters and water column (where applicable) were conducted at LSH in 2004 and 2005 to assess the controls on As speciation, mobility, and solubility at the sediment-water interface at the millimetre scale.

## Method

### Study Area

To determine the controls on As speciation and mobility from the historical tailings deposits at LSH, three sites with contrasting environmental conditions were chosen for the collection of tailings and associated pore water samples (Figure 2). Site 1 was located adjacent to an old stamp mill foundation. The site contains amalgamation tailings produced prior to the introduction of the cyanidation Au-recovery process in 1936. Tailings at this site were sub-aerially exposed, variably saturated with water, and underlain by organic-rich wetland sediments below approximately 40 cm depth. There was no standing water cover over the tailings at the time of sample collection.

Approximately 250 m south of Site 1 in a shallow wetland area is Site 2 (Figure 2). Here the tailings originated from the cyanide plant and were shallowly submerged, with about 20 cm of slow-moving water above the tailings at the time of sampling.

The last sample site, Site 3, is located approximately 2 km downstream from Site 2 in the intertidal zone of Seal Harbour. Sediments at this coastal site were a mixture of natural marine sediments and tailings originating from the LSH mill upstream. A channel with As-contaminated runoff from the LSH site crosses the tailings flat adjacent to Site 3 and empties into Seal Harbour. Evidence of clam digging was

observed in 2003 in this intertidal tailings flat, and shell fragments were found within the sediment samples collected for this study.

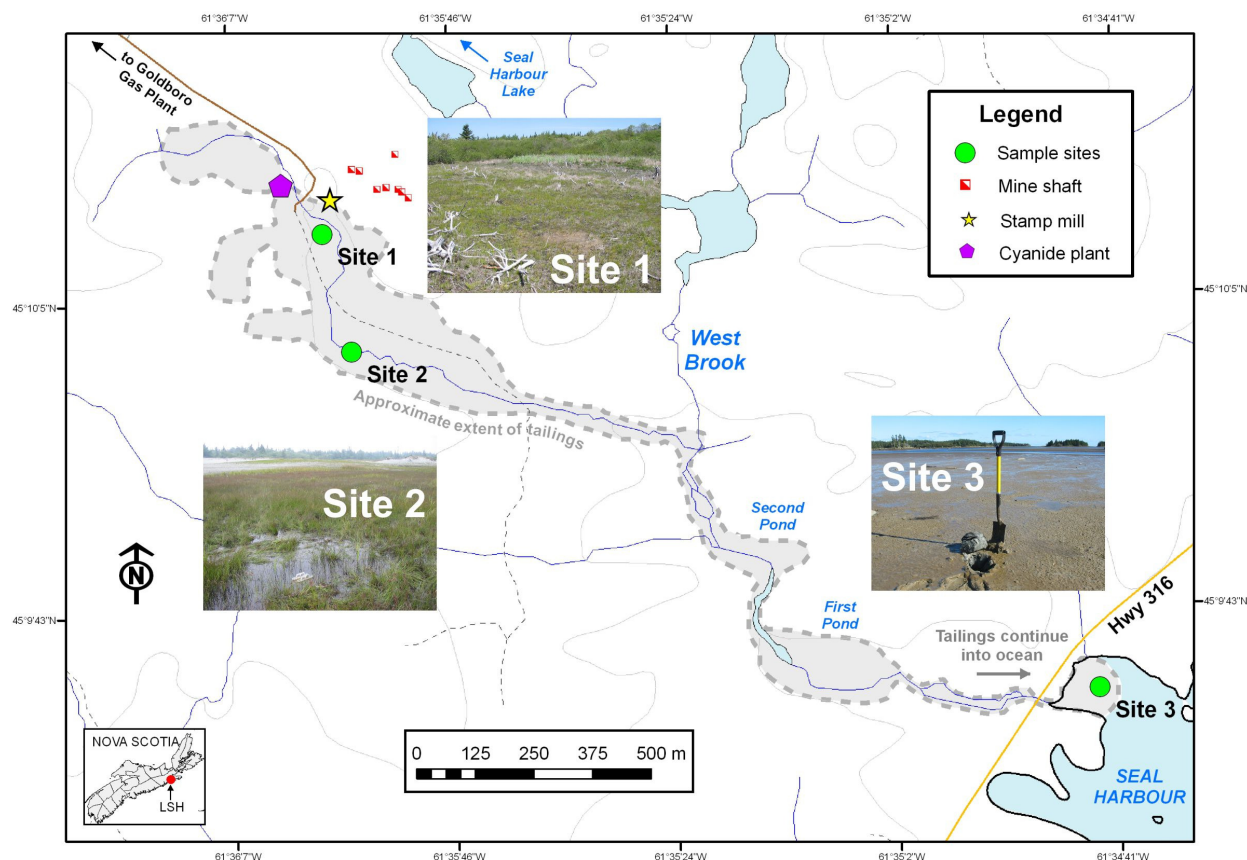


Figure 2. Map showing Site 1, Site 2 and Site 3 tailings and pore water collection locations.

### Sample Collection

Pore water in the tailings and the overlying water (where applicable) were sampled at sub-centimetre resolution *in-situ* using diffusion-based dialysis arrays (peepers) (Hesslein, 1976). As the pore water samples were used to measure redox-sensitive species ( $\text{As}^{3+}$ ,  $\text{As}^{5+}$ ,  $\text{Fe}^{2+}$ ,  $\text{H}_2\text{S}$ ), prior to field deployment, the peepers and their cases were filled with distilled, deionized water and deoxygenated by purging them with ultra-pure nitrogen for 10 days at room temperature.

Peepers were deployed directly into the mine tailings at the three different sites, two peepers per site. The peepers were left to equilibrate with the interstitial waters for  $21 \pm 2$  days. Once collected, the peepers were sealed in cases which were immediately purged with ultra-pure nitrogen to avoid oxidation of the peeper cell waters and covered to avoid photo-oxidation of aqueous ferrous iron. At each site, duplicate tailings cores were collected adjacent to the peepers, using 7.5 cm diameter PVC tubing.

## Analyses

### Solids

Solid samples were sub-sectioned in the field into discrete sample intervals using a sediment core extruder within a nitrogen-flushed glove bag: every 0.5 cm down to 10 cm depth, every 1 cm from 10 to 20 cm depth, and every 2 cm from 20 cm depth to the bottom of each core. Sub-sectioned samples were then frozen in the field and shipped to the laboratory, where they were analysed for metals by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) and organic carbon by a LECO combustion analyzer. Thin sections were also prepared from selected samples for petrographic analyses and electron probe micro-analyses (EPMA).

### Pore Waters

Peeper cell waters were extracted in a nitrogen atmosphere using metal-free pipettes and waters were passed through a 0.45  $\mu\text{m}$  filter. Filtered waters to be analysed for As speciation were transferred to HDPE bottles pre-loaded with 5% EDTA (225  $\mu\text{L}$ ) to preserve As-species (McClesky *et al.* 2004). Samples were stored at 4 °C in the dark for later speciation ( $\text{H}_2\text{AsO}_4^-$ ,  $\text{HAsO}_4^{2-}$  and  $\text{H}_3\text{AsO}_3^0$ ) analysis using high-performance liquid chromatography-ICP-MS.

## Results

### Solids

Solid-phase As concentrations in this study were highest at Sites 1 and 2, with maximum values of 9,700 mg/kg and 12,000 mg/kg, respectively. The highest As concentrations at Site 1 occur in the oxidized zone (0-15 cm), which may reflect post-deposition remobilization of As during weathering of the tailings (Figure 3). Arsenic concentrations in the intertidal tailings flat at Site 3 peaked at 1,040 mg/kg As. Although As concentrations at Site 3 are well above the Probable Effect Level (PEL = 41.6 mg/kg) for As in marine sediments (CCME 2002), the most As-rich sediments at Site 3 were likely buried over time by a layer of cleaner sediment with the continual deposition from West Brook and tidal currents.

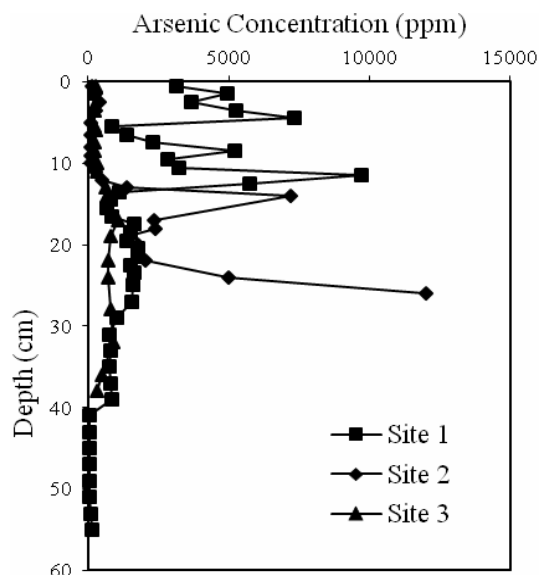


Figure 3. As concentration in solid phase.

Petrographic and EPMA analyses of thin sections were carried out to determine the mineralogical composition throughout the tailings profiles. The presence of pyrite and arsenopyrite grains at each of the three sites was confirmed using EPMA. Arsenic identified in well-oxidized, near-surface samples by EPMA techniques was likely pentavalent, based on bulk micro-X-ray absorption near edge structure ( $\mu$ XANES) and micro-X-ray diffraction ( $\mu$ XRD) spectra findings by Corriveau *et al.* (2011a) within surface tailings at LSH and other historical mine sites within the Meguma Terrane. Here, weathering in the tailings is defined as the alteration of primary minerals through chemical means, primarily the oxidation of sulphides (Smith and Beckie 2003).

At Site 1, analysis of near-surface, medium-brown tailings samples identified arsenopyrite grains with red-brown rims containing variable concentrations of As (Figures 4 and 5). In dark grey tailings at 20 cm depth, both pyrite and pyrrhotite grains were observed, but no reaction rims were evident on the sulphide grains. Immediately above wetland sediments at the base of the tailings (40 cm depth), grey tailings with fine-grained spheroidal pyrite grains, likely secondary phases, were observed.

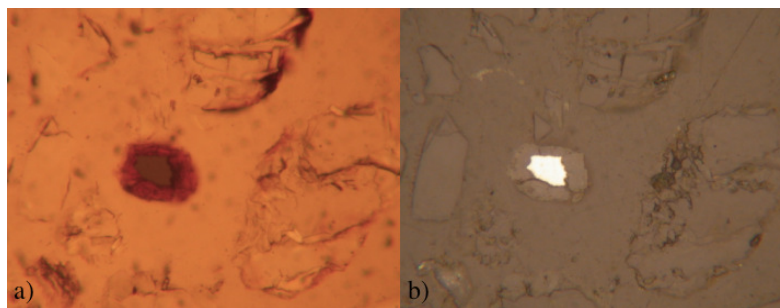


Figure 4. Arsenopyrite grain at 2.5 cm depth with red-brown Ca-Fe-As bearing rim, seen in a) transmitted light and b) reflected light. Field of view is 300  $\mu$ m.

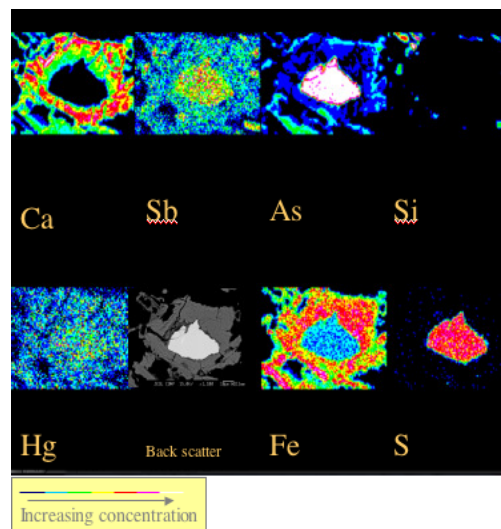


Figure 5. Site 1 Elemental map and back scattered electron image via EPMA of arsenopyrite grain with As-bearing rim at 2.5 cm depth.

At Site 2, brown, coarse-grained tailings at 2.5 cm depth contain red-brown As-bearing cements amongst silicate grains. There was no evidence of sulphide minerals in the near-surface samples. However, at

depth, in coarse-grained, yellow-brown tailings (approximately 10 – 20 cm) and deeper (20+ cm) fine-grained grey tailings, arsenopyrite and pyrite grains were observed with minimal to no evidence of alteration.

Sulphide minerals at Site 3 appeared to have been subjected to the least amount of weathering, as unaltered pyrite and arsenopyrite grains were observed from 17 cm depth to 30 cm depth. Sulphides were less common, yet present, in the near-surface samples. The majority of the sediments sampled throughout the core consist of coarse-grained tailings. At approximately 30 cm depth, the tailings/sediments were finer-grained than at the surface.

#### Pore Water

Tailings pore waters sampled using peepers indicate that oxidizing conditions persist to depths of 10 – 30 cm, 5 cm and 10 cm below the sediment-water interface (SWI) at Sites 1, 2 and 3, respectively. Measured pH values in the pore waters ranged from 6.2 to 8.0.

In this study, the  $\text{As}^{3+}/\text{As}^{5+}$  ratio is used to characterize redox conditions in the pore waters. The upper 30 cm of the dissolved As profiles at Site 1 were dominated by  $\text{As}^{5+}$  (Figure 6). Just above the organic-rich tailings and sediment interface identified at 40 cm, there was a sharp transition to an  $\text{As}^{3+}$  dominated profile and an increase in total As concentration. Profiles of dissolved As at Site 1 indicate strong redox control on the mobility of As. The increase in dissolved As concentrations and percentage of  $\text{As}^{3+}$  near the tailings-sediment interface suggests that  $\text{As}^{3+}$  is more soluble and mobile in these shallow groundwaters as compared to  $\text{As}^{5+}$ .

Total As and As speciation sums were measured from adjacent cells within the same peeper unit, therefore difference between the two profiles may reflect heterogeneity of the two adjacent cells, or differences in the analytical techniques used to measure the dissolved concentrations of As.

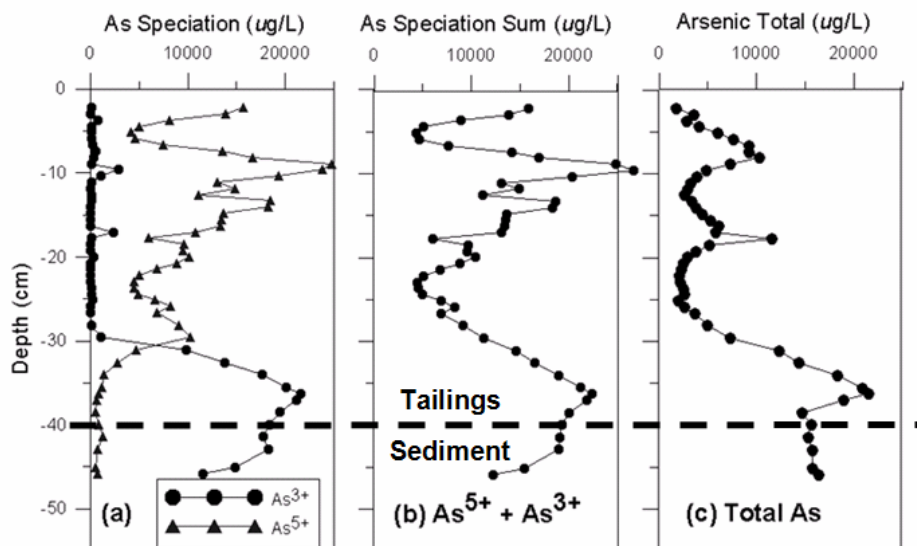


Figure 6. Site 1 dissolved As speciation in pore water.

At Site 2, there were high levels of dissolved As in the pore water relative to the water overlying the SWI (Figure 7). The dissolved As was significantly remobilized in the upper 15 cm of the profile to maximum values exceeding 10,000  $\mu\text{g/L}$ . The dominant As species in this profile switched at just 5 cm depth from  $\text{As}^{5+}$  to  $\text{As}^{3+}$ .

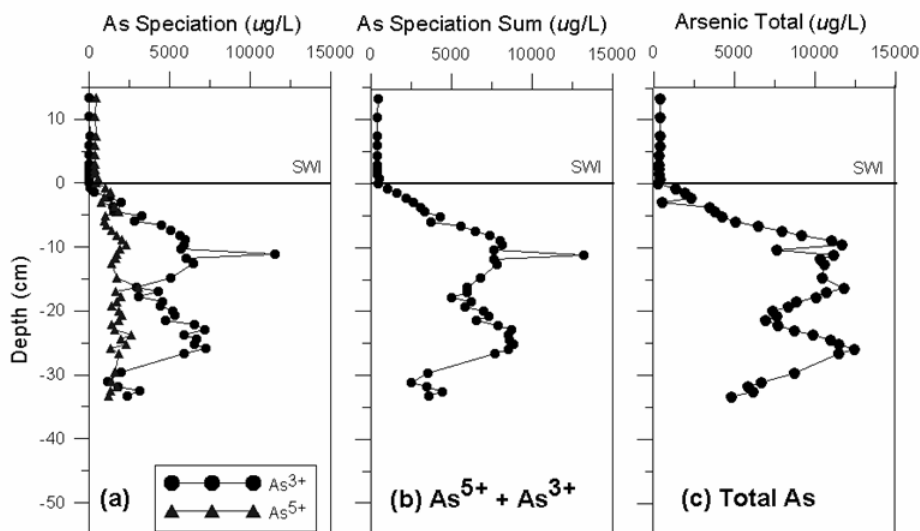


Figure 7. Site 2 dissolved As speciation in pore water.

At Site 3, above the SWI and in the upper decimeter arsenate concentrations dominate the inorganic As species of the sediment/tailing profile suggesting oxidizing conditions across the SWI (Figure 8). However, it is important to note that the peepers were extracted during low tide, at a time when the surface of the sediments was exposed to ambient air. The dissolved As species distribution may differ during high tide conditions as these shallow waters would be very well mixed and oxygenated.

Throughout the remainder of the profile at Site 3,  $\text{As}^{3+}$  and  $\text{As}^{5+}$  alternately dominate the dissolved inorganic species. The switch between dominant As species down-core may be due to a variety of factors, including decomposition of organic-rich material in the marine sediments, bioturbation, and varying amounts of tailings and natural sediments throughout the core. The pore water total dissolved As maxima (1,750  $\mu\text{g/L}$ ) was reached at a depth of 28 cm.

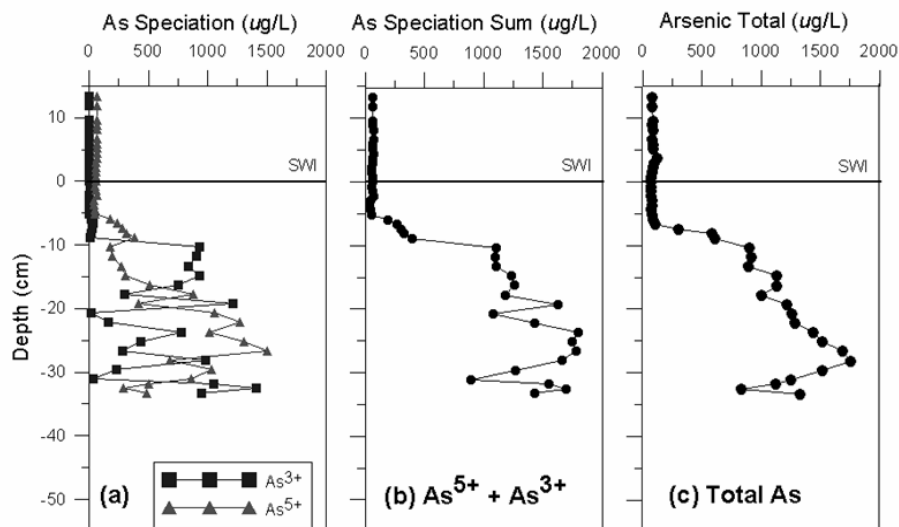


Figure 8. Site 3 dissolved As speciation in pore water.

## Discussion

Examination of the tailings at Site 1 reveals that As concentrations in the pore water were strongly controlled by the variable redox conditions within the tailings. Near surface, the conditions at Site 1 were oxidizing based on the physical appearance of the tailings in the field (e.g., brown in colour), the lack of unweathered primary sulphide minerals such as arsenopyrite, the presence of secondary minerals (as distinct grains, and reaction rims on weathered sulphides), and dominance of  $\text{As}^{5+}$  in the pore water. Maximum total As concentrations within the near-surface tailings and pore water were 9,700 mg/kg and 10,000  $\mu\text{g/L}$ , respectively.

Below approximately 20-30 cm depth at Site 1, the tailings demonstrate reduced conditions based on their grey appearance in the field, increased presence of sulphide minerals including unaltered arsenopyrite, pyrite, and trace amounts of chalcopyrite, and decreasing percentages of  $\text{As}^{5+}$  within the pore water. Maximum As concentrations within the mid-section tailings and pore water were 1,600 mg/kg and 21,000  $\mu\text{g/L}$ , respectively. Elevated dissolved As concentrations at this depth may result from reductive dissolution of As-bearing iron-oxide phases, as a result of increasingly reduced conditions created by low oxygen penetration into the waterlogged tailings. Toward the base of this interval, mixing of tailings with the underlying organic-rich sediments may promote enhanced activity of microbes, leading to further reductive dissolution and  $\text{As}^{3+}$  release.

Within the deepest fine-grained, organic-rich portion of the tailings at Site 1, maximum As concentrations within the tailings and pore water were 140 mg/kg and 16,000  $\mu\text{g/L}$  respectively. Rounded grains of iron sulphide interpreted to be authigenic in origin were present within this portion of the tailings; however, they do not contain As. The mixing of tailings with the underlying organic-rich sediment appears to lead to high concentrations of As in the tailings pore water, possibly through reductive dissolution and desorption reactions, or stabilization of high As levels in solution via organic acids.

The near-surface environment within the tailings at Site 2 is also a zone of sulphide oxidation, demonstrated by the absence of visible arsenopyrite, the presence of secondary phases, and the dominance of  $\text{As}^{5+}$  within the pore water. The sudden change from oxidizing to reducing conditions at 5 cm and elevated As concentrations in the pore water (3,400 to 12,400  $\mu\text{g/L}$ ) below 5 cm, corresponds to the region within the tailings where As-bearing sulphides were present. Reductive dissolution of the Fe-oxyhydroxides containing sorbed As at this depth results in increased concentrations of As. High levels

of As in the tailings pore water relative to the overlying surface water indicates that the tailings will continue to be a long-term source of As to the surrounding aquatic environment.

Arsenic concentrations within the sediments at Site 3 range from 74 to 1040 mg/kg. These concentrations exceed the Probable Effect Level for the Protection of Aquatic Life of 41.6 mg/kg (CCME 2002). The biological uptake of inorganic As by soft-shelled clams in Seal Harbour has been documented in recent studies (Koch *et al.* 2007), which suggests that As in the tailings pore waters is indeed bioavailable. Oxidizing conditions were demonstrated at the SWI by the dominance of As<sup>5+</sup> until 10 cm depth; however, there is no evidence of oxidation of As-bearing sulphide minerals. Elevated dissolved As concentrations in the pore water (up to 1,750 µg/L) below 10 cm depth at Site 3 correspond to the region of elevated As within the sediments. Variability in As concentrations within the sediments at Site 3 is likely due to mixing of tailings with natural marine sediments and the processes of precipitation and flocculation as As-rich waters from West Brook mix with seawater in Seal Harbour.

Arsenic released from arsenopyrite oxidation in the near-surface environments at Sites 1 and 2 is partially re-precipitated in the form of reaction rims on sulphide grains, and as cements between silicate grains. No scorodite (FeAsO<sub>4</sub>•2H<sub>2</sub>O) or other secondary As minerals were identified at LSH in this study, although many secondary phases have been characterized elsewhere in historical Au mines tailings throughout Nova Scotia using synchrotron-based methods (e.g. Walker *et al.* 2009, Corriveau *et al.* 2011a, DeSisto *et al.* 2011). Near-neutral pH values in the pore water throughout LSH suggest the dissolution of carbonate minerals (such as ankerite and calcite, present in the host rock) is sufficient to consume the acidity released by sulphide-mineral oxidation.

## Summary

The study of three geographically and geochemically distinct locations within LSH has demonstrated various factors that exert control on As mobility from historical arsenopyrite-rich Au mine tailings. Within the intertidal zone of Seal Harbour, As concentrations in the tailings and pore water were lower than at the mine site, likely as a result of the distance between the mine site and the harbour, the mixing of transported tailings with natural sediments, and the dilution of dissolved As in the marine waters. The presence of relatively unaltered arsenopyrite grains in these intertidal tailings flats also suggested that the tailings are relatively unreactive under these geochemical conditions. However, the mixing of As-contaminated freshwater with marine waters resulted in As-bearing flocculated material which may be relatively reactive near the sediment-water interface.

Uptake of inorganic As by bivalves in this environment may pose a threat to humans inadvertently consuming locally harvested shellfish. Koch *et al.* (2007) studied the bioaccessibility of softshell clams and seaweed harvested from LSH. Their study showed that contaminated clams in the area contained almost exclusively inorganic As, whereas seaweed contained predominantly organic forms of As. The high concentrations and bioaccessibility of As in both clams and seaweed suggests that they should not be used for human consumption. As a result of these findings, the Seal Harbour area is now closed for the harvesting of clams and seaweed.

The presence of shallow overlying waters and waterlogged tailings areas reduced oxygen penetration into the tailings, creating a stable environment for As-rich sulphides. However, other reactions (e.g. reductive dissolution of Fe-oxides) in these environments are still releasing As and other elements from the tailings to overlying surface waters. Also, when waterlogged As-bearing sulphide-rich tailings are in contact with organic-rich sediments, there appears to be an increase in As released to the tailings pore water. The results of this study show that these Au mine wastes have remained reactive for decades following their disposal and will continue to act as a long-term source of As to downstream environments.

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