



## Evaluation of metal partitioning and mobility in a sulfidic mine tailing pile under oxic and anoxic conditions



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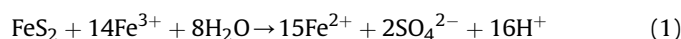
### ABSTRACT

Mining-influenced water emanating from mine tailings and potentially contaminating surface water and groundwater is one of the most important environmental issues linked to the mining industry. In this study, two subsets of Callahan Mine tailings (mainly comprised of silicates, sulfides, and carbonates) were collected using sealed containers, which allowed keeping the samples under anoxic conditions during transportation and storage. Among the potential contaminants, in spite of high concentrations of Cu, Mn, Pb, and Zn present in the solid mine tailings, only small amounts of Mn and Zn were found in the overlying pore water. The samples were subjected to leaching tests at different reduction-oxidation (redox) conditions to compare metal and S mobilization under oxic and anoxic conditions. It was observed that Cd, Cu, Mn, Pb, S, and Zn were mobilized at higher rates under oxic conditions, while Fe was mobilized at a higher rate under anoxic conditions in comparable constant pH experiments. These results suggest that metal mobilization is significantly impacted by redox conditions. When anoxic metal mobilization assessment is required, it is recommended to always maintain anoxic conditions because oxygen exposure may affect metal mobilization. A sequential extraction performed under oxic conditions revealed that most of the metals in the samples were associated with the sulfidic fraction and that the labile fraction was associated with Mn and moderate amounts of Pb and Zn.

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### 1. Introduction

Metal mobility is a major environmental risk related to the mining industry and occurs when mining influenced water (MIW) exits from active or inactive mine sites. MIW is generated under aerobic conditions when sulfidic minerals (e.g. pyrite, sphalerite, galena) react with oxygen and water. Pyrite (FeS<sub>2</sub>) oxidation is a complex multistep process in which the main acting pyrite oxidizing agent is ferric oxide and not oxygen (Johnson and Hallberg, 2005; Stumm and Morgan, 1981) as depicted in Equation (1):



As a consequence of this oxidation, the MIW acidity increases, bringing metals into solution. Active mine sites maintain a low water

bed with the objective of limiting MIW generation by pumping. But the water bed returns to the original (higher) level when mining operations cease. The MIW exits the mined areas through run off, adits and boreholes. MIW discharge may continue for years from spoil heaps, mining material surface piles, and mine tailings, and could contain high metal concentrations depending on the disaggregation and nature of the minerals present (Johnson and Hallberg, 2005).

Although sampling and preparation (transport, drying, particle size reduction, and storage) of mine tailings, soil, and sediments are usually performed while the sample is in contact with ambient air, several researchers have applied anoxic conditions for sampling storage and preparation, since the contact with oxygen significantly influences experimental results due to changes in the sample geochemistry (Johnston et al., 2009; Novák and Wieder, 1992; Priezel et al., 2009). Priezel et al. (2009) demonstrated different inorganic S and Fe speciation in well-aerated and wetland soils using extended X-ray absorption fine structure (EXAFS). Differences in S and Fe speciation occurred because of pretreatment (drying, crushing, and homogenizing) and storage of anoxic samples. The authors acknowledged that the speciation changes can occur rapidly unless anoxic conditions are permanently maintained

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during sample storage and preparation. Other metals can also be affected by introduction of oxic conditions to previously anoxic solid samples. Petersen et al. (1997) observed Cd, Cu, and Zn mobilization when anoxic sediments were aerated due to the oxidation of sulfidic minerals mediated by microbial activity (a control experiment with killed bacteria did not show the same level of metal mobility). Likewise, Barrett and McBride (2007) demonstrated that Zn–Cd sulfide suspensions quickly underwent oxidative dissolution after exposure to air. With all these possible reactions, a better understanding of how sulfidic minerals behave under anoxic and oxic conditions would be beneficial when conducting treatability studies, making decisions about remediation, water treatments, and long-term fate and transport metal assessments.

The aim of the present study was to evaluate metal mobility of the mine tailings and water from the Callahan Mine site when leaching under anoxic and oxic conditions. For this reason, the regulatory tests Synthetic Precipitation Leaching Protocol (SPLP) and Toxicity Characterization Leaching Procedure (TCLP) were conducted under oxic and anoxic conditions, while the Constant Acidity Leaching Procedure (CALP) was conducted at two different oxidative reduction potential (ORP) conditions at constant pH. Metal mobilization in the batch tests was also compared to the metal content of the overlying water collected with the mine tailings. A sequential extraction was used to determine metal associations and their potential mobility in the operationally defined fractions.

## 2. Materials and methods

### 2.1. Mine tailings sampling

Samples of the mine tailings were collected at the Callahan Mine site (Fig. 1), which is located in the Cape Rosier peninsula in Hancock County, ME. The site was an intertidal open-pit mine with an area of 61 ha (150 acres), in which Zn, Cu, Pb, and Ag were intermittently mined from 1880 to 1972 (mindat.org, 2012). Five million tons (4.5 million tonnes) of non-ore waste and 800,000 tons (725,000 tonnes) of ore waste were removed from the mine and accumulated in two waste rock piles within the property (Piatak et al., 2007).

The tailings were collected at two different locations (wells A and B), at three different depths at each location, using a split spoon auger drill. In each case, the contents of the split spoon were directly transferred into a stainless steel apparatus and immediately purged with nitrogen gas to ensure an anoxic environment during transportation. Dissolved oxygen was measured in two water samples of well A (both at 7 m depth) and in three water samples of well B (14–15 m depth) collected in 0.5 L bottles using an Orion 810 Dissolved Oxygen (DO) meter. The samples used for anoxic experiments were processed in a Type C Anaerobic Chamber (Coy Laboratory Products, Grass Lake, Michigan).

### 2.2. Sampling apparatus

The sampling apparatuses were cylindrical stainless steel containers (14.7 cm ID × 15.2 cm H) built by Cluxton Instruments Inc. (Martinsville, OH), having a total volume of 2.58 L. The cover was secured with an external rim which allowed the pressurization of the vessel. The cover included a 3-way valve to allow the use of nitrogen to displace the air in the headspace of the sampling apparatuses through a relief valve (set to 3.6 psi). The sampling apparatuses were kept pressurized during sample transportation.

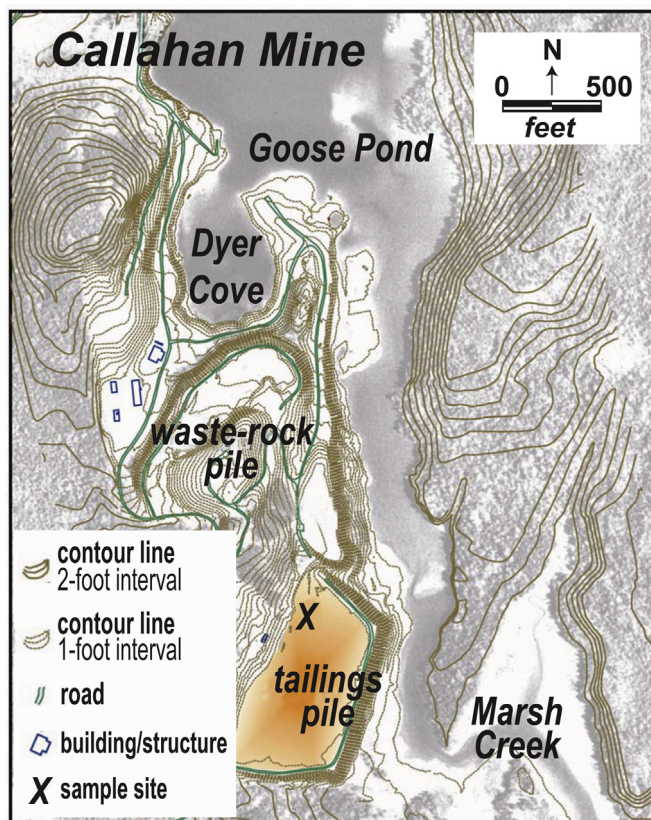


Fig. 1. Location of the mine tailings pile in the Callahan Mine site. Figure taken from Piatak et al. (2007).

### 2.3. Mine tailings characterization

The mine tailings were characterized for moisture using ASTM Method D2216-10 and for solid pH by U.S. EPA Method 9045D, both in as-received conditions. The rest of the analyses were performed on oven-dried mine tailings (105 °C, overnight exposed to ambient air). Particle size distribution (PSD) was measured according to ASTM Method D6913-04 with sieve mesh sizes of 2, 0.422, 0.251, 0.178, and 0.075 mm. Metal content was measured by acid digestion using EPA Method 3051 and by ICP-AES analysis (EPA Method 6010B using an Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) IRIS Intrepid, Thermo Scientific, MA). X-ray diffraction (XRD) was performed using an X'Pert-MPD PW3040/00 Diffractometer (PANalytical, Westborough, MA) using Cu K $\alpha$  radiation, at 2°/min ranging from 5° to 160° and the data analysis was performed using HighScore Plus® software. The overlying water was recovered by decanting the collected samples (~3.5–4 kg of total mass in each case) into a beaker inside the anaerobic chamber without the application of pressure or centrifugation. This overlying water was analyzed for pH by EPA Method 9040C and acid digested following EPA Method 3015 and then analyzed for metal content using ICP-AES. X-ray photoelectron spectrometry (XPS) was performed on a QUANTERA II photoelectron spectrometer from PHI using the < 2-mm dried mine tailings without any further treatment. Each sample was dispersed uniformly on double-sided tape affixed to a 5-mm-diameter stainless-steel platen. Briefly, this XPS uses a monochromatized Al-K $\alpha$  radiation (1486.6 eV) focused on to the sample with an incident angle of 45°. Most XPS analyses were performed at least in duplicate. Coefficients of variation for the results were typically less than 10% for target elements. The surface composition determined by XPS probed the outer 2–10 nm of the

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