

Geochemical and Mineralogical Controls on Metal(loid) Mobility in the Oxide Zone at Prairie Creek, Northwest Territories

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Abstract

Prairie Creek is an unmined high grade Zn-Pb-Ag deposit in the southern Mackenzie Mountains of the Northwest Territories. The upper portion of the primary quartz-carbonate-sulphide vein mineralization has undergone extensive oxidation, forming high grade zones rich in smithsonite (ZnCO_3) and cerussite (PbCO_3). This weathered zone represents a significant resource and a potential component of mine waste material. This study is focused on the characterization of the geochemical and mineralogical controls on metal(loid) mobility under mine waste conditions, with particular attention to the metal carbonates as a potential source of trace elements to the environment. Analyses were conducted using a combination of microanalytical techniques and the elements of interest included Zn, Pb, Ag, As, Cd, Cu, Hg, Sb and Se.

Results include the identification of minor phases previously unknown at Prairie Creek, including cinnabar (HgS), acanthite (Ag_2S), metal arsenates, and Pb-Sb-oxide. Anglesite (PbSO_4) may also be present in greater proportions than suggested by previous work. Smithsonite is the major host for Zn but this mineral also contains elevated concentrations of Pb, Cd, Cu, Fe, and Mn, while cerussite (which may be removed as Pb concentrate) also hosts Zn, Cu and Cd. Variable concentrations of Fe, As, Sb, Hg, Ag, and Se are also present in smithsonite and cerussite (listed in approximately decreasing order). A significant proportion of

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the trace metal(loid)s may also be hosted by other secondary minerals associated with mineralization. Processing will remove significant sources for these elements, but the original mine plan calls for leaving the smithsonite fraction as tailings. Significant Hg and Ag could remain in tailings from cinnabar and acanthite that is trapped within smithsonite grains.

In a mine waste setting, near-neutral pH will encourage retention of trace metal(loid)s in solids. Regardless, oxidation, dissolution and mobilization is expected to continue in the long term, which may be slowed by saturated conditions, or accelerated by localized flow paths and acidification of isolated, sulphide-rich pore spaces.

Introduction

The Prairie Creek Zn-Pb-Ag deposit is located 500 km west of Yellowknife in the southern Mackenzie Mountains and is surrounded by the recently-expanded Nahanni National Park Reserve. The district contains several types of carbonate-hosted mineralization, including stratabound replacement massive sulphides, quartz-carbonate sulphide veins, and classic Mississippi Valley-type (Paradis, 2007; Paradis, this open file). The quartz-carbonate-sulphide veins and the stratabound replacement sulphides have undergone extensive oxidation and alteration, forming zones rich in smithsonite and cerussite, which is referred to as the “oxide zone”. There is a wide range of trace elements associated with the oxide zone of the deposit, including Ag, Cu, As, Sb, Cd, Se, and Hg, some of which are of potential economic or environmental significance.

The objective of this research project was to understand how these trace elements are mineralogically hosted in the oxide zone of the Prairie Creek deposit. The results have implications for assessing the economic value of the oxide zone and for predicting the geochemical controls on metal(loid) concentrations in drainage from future mine waste.

The site was developed in the 1980s but was never mined. The current owner, Canadian Zinc Corporation, intends to bring the property into production (Canadian Zinc Corporation, 2010, 2014). Although acid rock drainage is not anticipated due to the substantial amount of carbonate associated with the mineralization, metal(loid) leaching occurs from the portal and is predicted from geochemical tests (MESH Environmental, 2008). Post-production plans include underground deposition of all tailings as paste backfill. Results from this research have been used to predict the leaching behaviour of paste backfill made with the oxide zone tailings.

This research formed part of the MSc thesis of Drew Stavinga in the Department of Geological Sciences and Geological Engineering at Queen’s University and detailed results are available therein (Stavinga, 2014).

Results/Data Analysis

Field Sampling

Fieldwork in August 2013 resulted in 29 samples of surface exposures of vein mineralization, 19 samples of the main quartz-carbonate-sulphide vein from the 930 level of the underground workings, 83 samples from on-site core, and 3 samples from the ore stockpile representing the 870 underground level. Samples were chosen based on the presence of “oxide zone” mineralization and, where available, elevated concentrations of the elements of interest. Seven additional samples of surface exposures were supplied by the Northwest Territories Geoscience Office (NTGO) and 10 thin sections from the Geological Survey of Canada (GSC) collection were analysed. Sample locations are shown in Stavinga (2014).

Analysis

Lithogeochemistry

A subset of 53 samples, chosen to represent various degrees and styles of oxidation, were digested via *aqua regia* and analysed for 45 elements by inductively coupled plasma - optical emission spectrometry (ICP-OES) at AGAT laboratories in Vancouver. A non-sulphide leach using ammonium acetate (for Pb) and ammonium chloride/ammonium acetate (for Zn) was used to obtain Pb and Zn concentrations that were considered to represent cerussite and smithsonite, although this was not explicitly tested. Samples with Ag concentrations greater than 500 ppm were subjected to Fire Assay Fusion with Gravimetric finish. Complete results, including results on blanks, replicates and reference materials are available in Stavinga (2014).

Detailed Mineralogical and Trace Element Analysis

Polished thin sections were made from 38 samples selected on the basis of elevated concentrations of elements of interest, presence of Pb or Zn carbonates and location within the deposit. These were examined by petrographic microscopy, and analysed by scanning electron microscopy (SEM), electron microprobe analysis (EMP), Laser Ablation - Inductively Coupled Plasma - Mass Spectrometry (LA-ICP-MS), X-ray diffraction (XRD), synchrotron based trace element mapping, synchrotron-based grain-scale micro-X-ray Diffraction (μ XRD), micro-X-ray Fluorescence (μ XRF), and SEM-based quantitative mineralogy using Mineral Liberation Analysis (MLA). The results of μ XRD analysis provided the general mineral classifications that were applied to the results of MLA. Further details on analytical methods can be found in Stavinga (2014).

Results

Lithogeochemistry

The complete analytical results and correlation matrices for the lithogeochemical analyses can be found in Stavinga (2014). In general, most samples of the oxidized main quartz-carbonate-sulphide vein material showed higher average concentrations of Ag (422 ppm), As (1587 ppm), Cu (8786 ppm), Cd (1374 ppm), Hg (491 ppm), Pb (16.4%), and Sb (3563 ppm) when compared to the adjacent

host rock. Except for two samples, Se was mostly below detection limit (<50 ppm). Total Zn concentrations are relatively consistent between the vein (15.8%) and the immediately adjacent host rock of the hanging wall (15.2% for a sample within 10 cm), but are lower in the footwall. Non-sulphide Zn follows a similar pattern, with an average concentration of 9.9% and 8.2% in the vein and hanging wall, respectively. Iron concentrations were found to be similar among the footwall (0.4%), hanging wall (0.4%), and vein material (0.6%). Although Cd and Hg have higher average concentrations in the vein than the host rocks, their values generally fall within the range of concentrations occurring within the hanging wall, with the exception of a few outliers. Digestion by non-sulphide leach found non-sulphide Pb concentrations are generally higher (average 8.1%) in the mineralized vein, are slightly lower in the hanging wall (average 3.6%), and much lower in the footwall (average 0.3%).

Mineralogy

Table 1 lists all minerals identified through a combination of analytical techniques, including SEM, EMP, XRD and synchrotron-based μ XRF and μ XRD. The most common and abundant minerals identified consist of the primary sulphides (galena, sphalerite, pyrite) and sulfosalts (tetrahedrite-tennantite), the host rock and gangue minerals (calcite, dolomite, quartz), and the secondary metal carbonates (smithsonite, cerussite, malachite, azurite). The list of positively identified minerals provides at least one host for almost every element of concern. Elements without an obvious host, notably Cd and Se, likely occur as minor or trace substitutions and possibly as a tentatively identified phase (i.e. greenockite, tiemannite). Tentatively identified phases, 26 in all, were distinguished by SEM but did not diffract well enough to be firmly identified by μ XRD.

Figure 1 is a box plot showing elemental concentrations on a logarithmic scale. Analyses of the metal carbonates and anglesite (a Pb-sulphate oxidation product of galena) by EMP and LA-ICP-MS indicate that they host many of the elements of concern. Some elements are preferentially hosted by certain minerals over others; for instance Ag and Se are higher in the Pb-carbonate and Pb-sulphate than in the Zn-carbonate, while the opposite is true for Cd. Anglesite also contains higher concentrations of Ag, As, Cu, Sb and Se than the Pb and Zn carbonates. Other secondary oxidation products include goethite, which replaces pyrite and is also a common inclusion in smithsonite. Detectable concentrations of metal(loid)s, primarily Pb, Sb, Zn, As, and Cu, are concentrated along the edges of goethite grains or evenly dispersed throughout them. Lead is the predominant metal of interest hosted by goethite, in both pyrite replacement rims and smithsonite inclusions.

In many cases, elements of interest are hosted by secondary minerals present as small inclusions within smithsonite and cerussite, as illustrated in Figure 2. Previous work (MESH Environmental, 2008; pHase Geochemistry, 2010)

Table 1. Minerals identified by one or more microanalytical technique within the quartz-carbonate-sulphide vein and stratabound massive sulphide-type mineralization. An additional 26 minerals were tentatively identified by SEM but not confirmed by μ XRD (Stavinga, 2014).

Mineral	Formula
<i>Positively Identified Phases</i>	
<i>Major Minerals</i>	
Galena	PbS
Pyrite	FeS ₂
Sphalerite	(Zn,Fe)S
Tennantite-Tetrahedrite	(Cu,Ag,Fe,Zn) ₁₂ As ₄ S ₁₃ - (Cu,Fe,Ag,Zn) ₁₂ Sb ₄ S ₁₃
Anglesite	PbSO ₄
Azurite	Cu ₃ (CO ₃) ₂ (OH) ₂
Calcite	CaCO ₃
Cerussite	PbCO ₃
Dolomite	(Ca,Mg)(CO ₃) ₂
Malachite	Cu ₂ CO ₃ (OH) ₂
Smithsonite	ZnCO ₃
Quartz	SiO ₂
<i>Minor minerals</i>	
Acanthite	Ag ₂ S
Bournonite	PbCuSbS ₃
Cinnabar	HgS
Covellite	CuS
Goethite	FeO(OH)
Bindheimite	Pb ₂ Sb ₂ O ₆ (O,OH)
Hydrozincite	Zn ₅ (CO ₃) ₂ (OH) ₆
Barite	BaSO ₄
Christelite	Zn ₃ Cu ₂ (SO ₄) ₂ (OH) ₆ ·4H ₂ O
Adamite	Zn ₂ (AsO ₄)(OH)
Arsentsumebite	Pb ₂ Cu(AsO ₄)(SO ₄)(OH)
Bayldonite	Pb(Cu,Zn) ₃ (AsO ₄) ₂ (OH) ₂
Beaudantite	PbFe ₃ ³⁺ (AsO ₄)(SO ₄)(OH) ₆
Duftite	PbCu(AsO ₄)(OH)
Mimetite - Clinomimetite	Pb ₅ (AsO ₄) ₃ Cl
Segnitite	PbFe ₃ ³⁺ H(AsO ₄) ₂ (OH) ₆

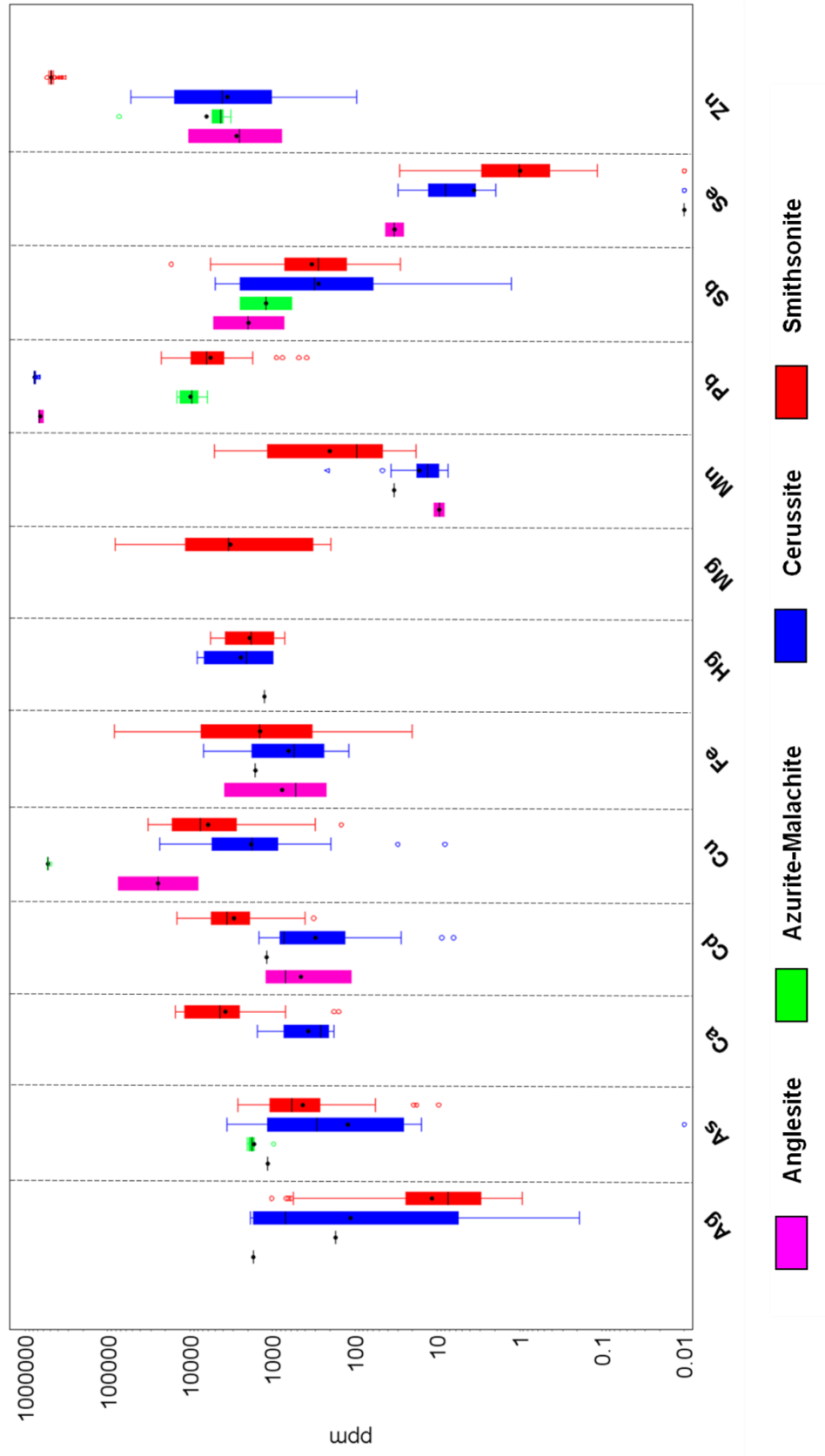


Figure 1. EMP and LA-ICP-MS results for selected elements of interest in secondary oxide minerals. EMP concentrations below detection limit and LA-ICP-MS concentrations greater than one order of magnitude above the calibration curve limit are omitted.

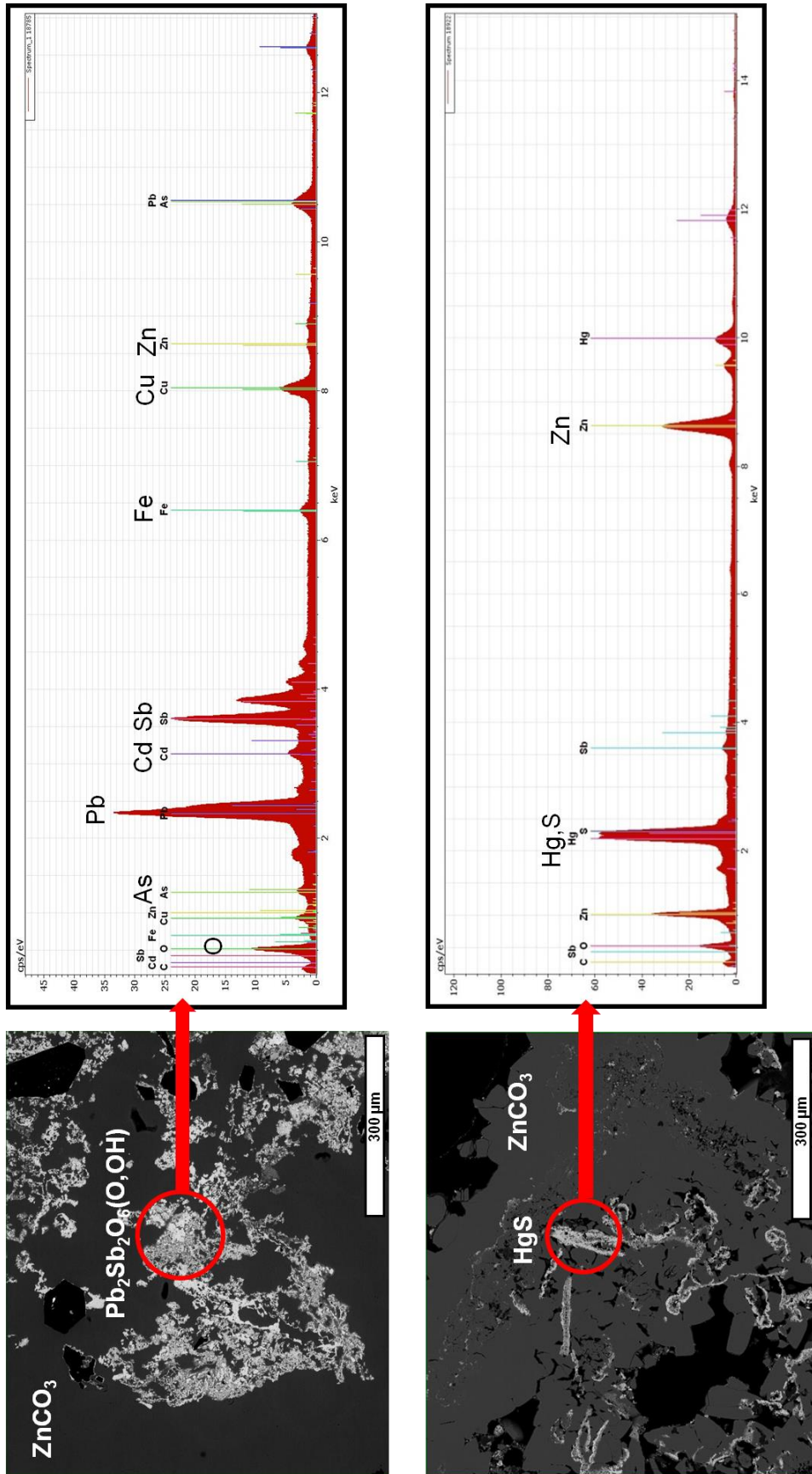


Figure 2. Backscattered-electron images and energy-dispersive spectra showing elements of interest hosted in mineral inclusions included in smithsonite (A) Pb - Sb - O phase, (B) Hg - S phase. Mineral composition based on μXRD .

attributes dissolution of the metal carbonates as the primary source of mobilized metal(loid)s from simulated tailings material. However, our results indicate that some of the metal(loid)s are actually hosted in minerals other than smithsonite and cerussite.

Trace element mapping employing synchrotron-based μ XRF illustrates the textural relationship of minerals hosting the elements of interest. For example, Figure 3A and 3B shows arsenate minerals forming rims along the edges of other minerals. Both Cd (Figure 3C) and Cu (not shown) are concentrated within discrete bands in smithsonite.

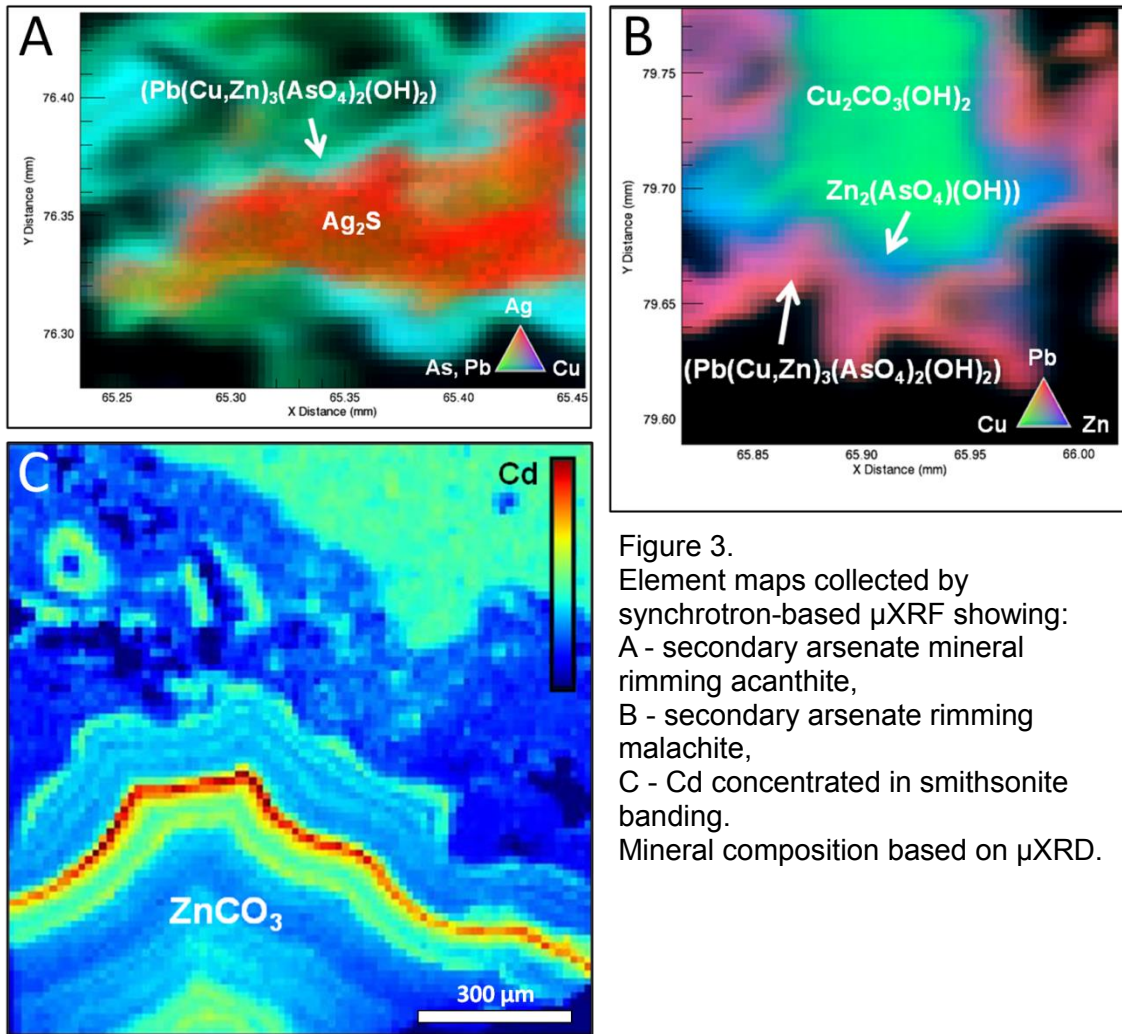


Figure 3. Element maps collected by synchrotron-based μ XRF showing: A - secondary arsenate mineral rimming acanthite, B - secondary arsenate rimming malachite, C - Cd concentrated in smithsonite banding. Mineral composition based on μ XRD.

Mineral mapping of thin sections by SEM-MLA provides information on mineral proportions, grain sizes and mineral associations (Figure 4). These results reveal that anglesite is present in greater proportions than previously thought (Stavinga, 2014). The other secondary phases identified may also exist in high enough proportions to influence metal(loid) mobility. The grain sizes of some, namely cinnabar and acanthite, were found to be relatively fine and hosted primarily by smithsonite (Figure 4C). Textural evidence of dissolution of the metal carbonates and other secondary phases is also seen in strongly weathered material.

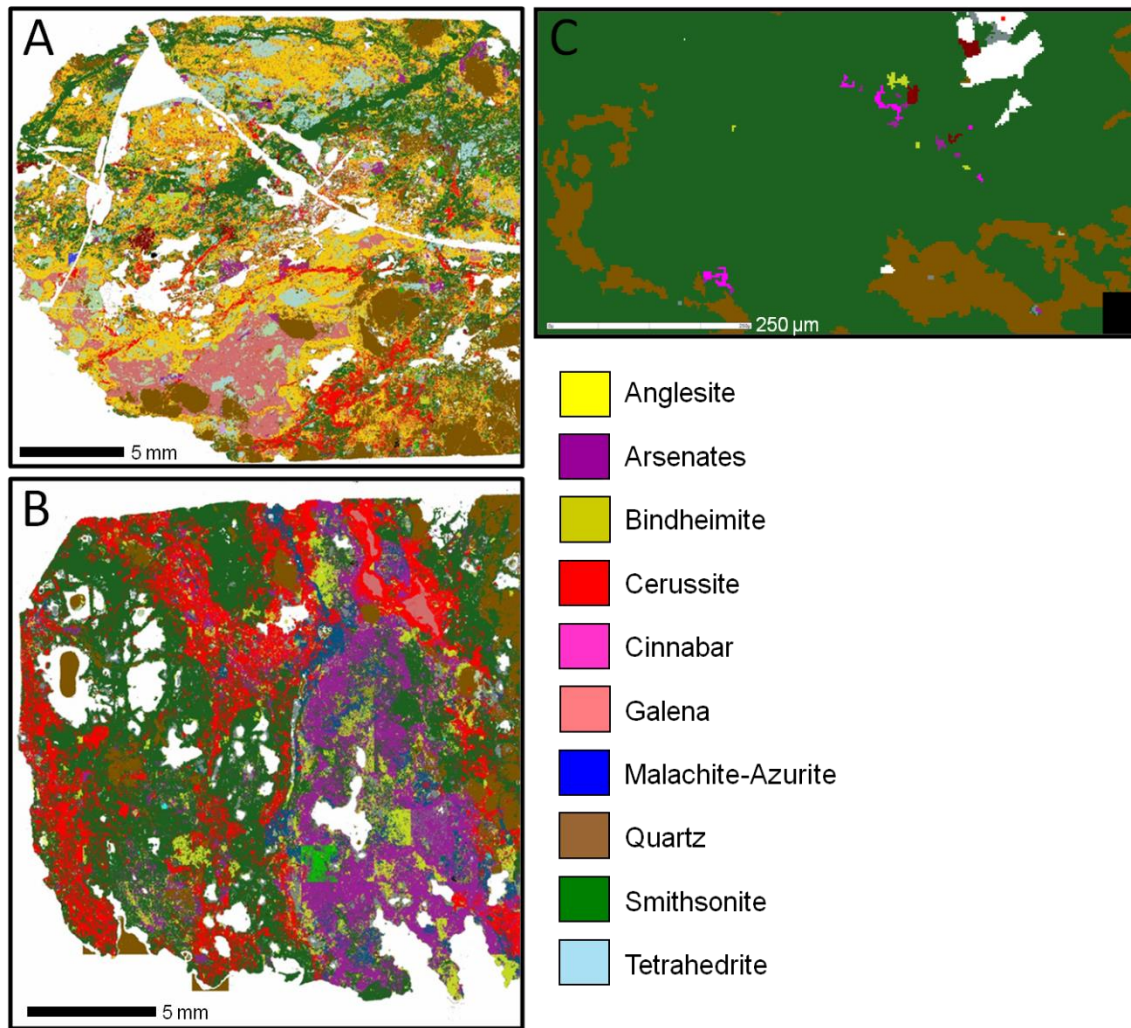


Figure 4. Examples of mineral maps generated by SEM-MLA used for calculating mineral proportions and observing grain size and textures. A - Strongly oxidized sample of quartz-carbonate-sulphide vein from the underground mine containing abundant anglesite (yellow), sample A930-11, B - Strongly oxidized sample of quartz-carbonate-sulphide vein from surface, containing abundant arsenate (purple) and bindheimite (yellow-green), sample Z6-S7, C - Cinnabar and associated arsenate and bindheimite inclusions in smithsonite, sample Z7-S6a.

Discussion

Comparison of Analytical Techniques

Overall, in the context of this research project, analysis by EMP proved to be most useful for determining the elemental composition of specific minerals. However, LA-ICP-MS did prove essential in quantifying trace element concentrations, which were subsequently used to estimate the average concentration of elements within a particular mineral, and the distribution of elements. Characterization of texture and mineral associations was best accomplished using the SEM, while MLA contributed to its understanding and proved to be the best tool for estimating relative proportions and specific element

speciation. Synchrotron-based analysis proved instrumental in identifying unknown phases, while giving important insight into the distribution of elements of concern and their valence states that would not otherwise have been known (i.e. Hg and Se associations, and Sb valence; see Stavinga (2014) for details regarding Sb oxidation state measured by synchrotron-based X-ray absorption). Each analytical technique complemented the others, and their combined use greatly increased the quality of the results achieved.

Trace Element Geochemistry and Speciation of the Oxide Zone

The distribution of elements amongst minerals in each thin section analyzed by MLA is estimated by combining the estimated average concentrations for each mineral (Table 2) with the modal mineralogy calculated by the MLA software (details in Stavinga, 2014). Thus, the major sources of elements of interest are revealed for each sample (Figure 5). Reconciliation assays that compare the total concentration of each element calculated by MLA for a thin section agree fairly well with the overall whole rock geochemical results, usually falling within the range of measured total element concentrations. The heterogeneous nature of the mineralization, however, means a direct comparison of concentrations between the same samples is less reliable, resulting in small to large differences as the material used for each analysis could vary in mineral proportions. Nevertheless, this suggests that speciation analysis of a large suite of thin sections could give concentrations that are relatively representative of the overall geochemistry of the deposit.

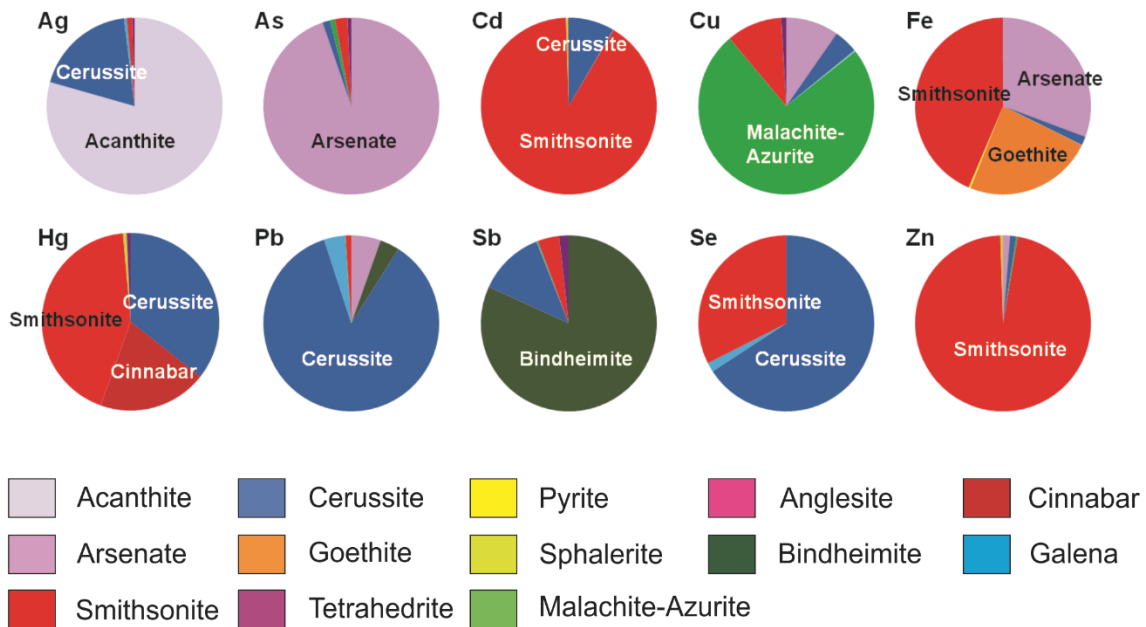


Figure 5. Distribution of elements between different minerals for a single thin section of mineralized material, based on SEM-MLA mineral maps and measured average element concentrations from EMP and LA-ICP-MS analyses.

Table 2. Concentration ranges from EMP and LA-ICP-MS analysis of primary sulphides and sulfosalts and secondary oxide minerals. Values below detection limit are listed as '< [Detection Limit]'. Values one order of magnitude above or below the ICP-MS calibration curve limit are listed as '>[Limit]' and '<[Limit]', respectively. No Hg standard was available for LA-ICP-MS.

Mineral	Method	Ag ppm	As ppm	Ca ppm	Cd ppm	Cu ppm	Fe ppm	Hg ppm	Mg ppm	Mn ppm	Pb ppm	Sb ppm	Se ppm	Zn ppm
Galena	EMP	-	<290	-	<550 - 555	<564 - 1500	<363 - 981	<491	-	-	84.9% - 87.3%	<889	-	<433 - 8312
	LA-ICP-MS	25 - >2000	4 - 384	-	3-140	21 - >4000	55 - 2.7%	-	-	2 - 30	>4000	12 - 3956	1 - 18	20 - >5000
Sphalerite	EMP	<324	<659	-	936 - 5516	<485 - 3968	<262 - 1.5%	<497 - 3869	-	-	<1294 - 2668	<647	-	64.4% - 70.1%
Tennantite-Tetrahedrite	EMP	<323 - 3.3%	1.5% - 17.7%	-	1295 - 9615	32.6% - 42.7%	<286 - 2360	1972 - 6892	-	-	<1289 - 6%	3.8% - 25%	-	7.1% - 10.3%
Pyrite	EMP	<323	<445	-	<361	<507	46.8% - 47.2%	<491	-	-	1481 - 2699	<705	-	<384
Bournonite	EMP	<323	2.4% - 2.7%	-	<361 - 520	13.1% - 13.6%	<286	<491 - 506	-	-	43.3% - 43.5%	20.6% - 20.9%	-	<384 - 1363
	EMP	<385 - 1021	<356 - 2650	157 - 1.5%	<301 - 1.4%	<452 - 3.3%	<277 - 8.3%	<593 - 5679	<182 - 8.1%	<275 - 5026	761 - 2.2%	<235 - 1.7%	<603	34.5% - 54.9%
Smithsonite	LA-ICP-MS	1 - 593	10 - >2600	-	1497 - >1600	146 - >4000	20 - 8.3%	-	-	18 - 3117	384 - >4000	28 - >5000	<0.02 - 29	>5000
	EMP	<578 - 915	<407 - 3558	<160 - 1530	<453 - 1475	<605 - 2.4%	<420 - 3025	<675 - 8139	<157	<408	70.7% - 79.5%	<327 - 1709	<560	<597 - 5.3%
Cerussite	LA-ICP-MS	0.2 - >2000	<0.3 - >2600	-	6 - >1600	8 - >4000	119 - 6954	-	-	7 - 219	>4000	>5000	<0.02 - 30	96 - >5000
	EMP	<578 - 3944	<578 - 3944	<160 - 1530	692 - 1191	<605 - 7.5%	<420 - 521	<675 - 1254	<157	<408	60.1% - 68.6%	<327 - 5187	<560	782 - 1%
Anglesite	LA-ICP-MS	1709 - >2000	1140 - >2600	-	112 - >1600	>4000	225 - 3815	-	-	8 - 11	>4000	>5000	26 - 43	2505 - >5000
Malachite	EMP	<385	973 - 2047	-	<425	50.3% - 54.5%	<319	<593	-	-	<1349 - 1.5%	<557 - 582	<603	3177 - 7.3%
Azurite	LA-ICP-MS	172	>2600	-	1180	>4000	1611	-	-	33	>4000	2453	<0.02	>5000

Sulphide Oxidation Under Alkaline Conditions

Oxidation of the sulphides is occurring at Prairie Creek despite the alkalinity and effective buffering capacity offered by the carbonate host rocks. Although sphalerite oxidation decreases with increasing pH, it begins to increase again above pH 7, primarily due to the influence of the oxidant O₂ (Ziping et al., 2012). Oxidation may also be aided in part by armouring of the host rock by gypsum and hydrous ferric oxide, inhibiting fast neutralization of the acidic solution produced by oxidation of the sulphides and allowing the establishment and stability of an acidic pH within the oxidation zone (Reichert and Borg, 2008). At Prairie Creek, dissolution and alteration is more apparent with sphalerite as opposed to galena, likely due to anglesite and cerussite rims protecting the galena from further oxidation (Stavinga, 2014). This may explain why in highly oxidized samples the only trace of original sulphides is usually galena.

Prediction of Metal(loid) Mobility from Mine Waste

Samples analyzed in this study were collected from quartz-carbonate-sulphide veins and stratabound replacement massive sulphides *in situ*, except samples collected from drill core, which have been stored at surface for up to 22 years, and the 870 level samples, which have been undergoing weathering in the ore stockpile for approximately 30 years. Little mineralogical difference is observed between the samples collected directly from the orebody and surface showings and those from stored drill core and the stockpile. When mining commences, the final tailings will be ground to a fine grain size (~80% at < 80 µm), increasing their potential reactivity, and for paste backfill, tailings will be mixed with binders and cement. Most of the Pb and Zn sulphides will be removed, and Pb and Zn oxides (cerussite and smithsonite) may be removed as well. The mine plan calls for underground storage of all tailings as paste backfill. This may or may not include the smithsonite fraction, depending on the final mine plan. It has been suggested in previous studies (MESH Environmental Inc., 2008) that the metal carbonates will likely dissolve and contribute to the alkalinity and dissolved metal(loid) content of the mine water, with smithsonite supposedly more likely to dissolve than cerussite. This is due to the tendency of cerussite to alter to anglesite rather than dissolve and release Pb ions (Sato, 1992); the release of Zn ions from smithsonite should therefore occur at a greater rate. Except in strongly weathered surface showings, samples usually show only minor dissolution of the metal carbonates under the current *in situ* conditions. However, should dissolution of the metal carbonates occur, Zn, Pb and other trace elements of concern would be released into the pore waters, increasing concentrations significantly if conditions do not favour their attenuation. The dissolution of metal carbonates, in addition to the oxidation of sulphides, is therefore likely to be a major factor controlling the mobility of the trace elements.

Infiltration of the tailings by mine water may generate similar reactions to that experienced by the ore stockpile from rainwater infiltration. The mineralogical characterization by Skeries (2013) of the sediments present in the stockpile,

which has been exposed for 30 years, may indicate which minerals are likely to dissolve. The sulphides, metal carbonates (smithsonite, cerussite) and anglesite may also dissolve, as indicated by observed dissolution textures. However, the formation of secondary rims on sulphides, such as cerussite and anglesite on galena and goethite on pyrite, should slow their oxidation and release of metal(loid)s. Goethite is the most common Fe-(oxy)hydroxide formed under alkaline conditions (Bigam, 1994) and is a predominant precipitate in the ore stockpile and, along with Mn-oxide coatings, in the waste rock pile (Skeries, 2013). It is therefore likely to remain stable in the tailings, possibly increasing in content along with Mn-oxides, and, through attenuation, act as an efficient immobilizer of the metal(loid)s of concern. Significant amounts of precipitated azurite, chertelite, hydrozincite and possibly aurichalcite also coat the mine adit walls, signifying these phases act as major controls on Zn and Cu mobility once they migrate out of the vein.

Potential flooding of the mine workings after production ends at Prairie Creek would result in the saturation of the backfilled tailings. Exposure to oxygen would be limited, and further oxidation of remaining sulphides would be slowed. However, the oxide tailings will include non-sulphide metal-hosting minerals and their geochemical behaviour under water-saturated conditions is uncertain (Stavinga, 2014).

Implications for Exploration

The focus of this research was to understand how trace elements of economic value and environmental concern are mineralogically hosted in the oxide zone of the quartz-carbonate-sulphide veins and stratabound replacement sulphides at Prairie Creek. Our results show that detailed mineralogical analyses using multiple techniques revealed the presence of Ag (a valuable commodity) hosted in acanthite and Hg (an environmental concern) hosted in cinnabar. Overall, however, the metal carbonates smithsonite and cerussite are the major sources for the elements of concern, and a small, even local decrease in pH may result in metal(loid) release (Stavinga, 2014). There is also the possibility that saturation of tailings used for paste backfill could release metal(loid)s through reductive dissolution of waste from the smithsonite-cerussite “oxide” zone. Mine planning (Canadian Zinc Corporation, 2010) includes consideration of a three-concentrate scheme where most of the smithsonite would go to the tailings. The SEM-MLA calculations suggest that this fraction would carry a significant concentration of Ag, Hg, Cd and other trace elements. Canadian Zinc Corporation is currently considering revising their mine plan to ship Zn carbonate (i.e. smithsonite) concentrate for this and other reasons (Alan Taylor, VP Exploration, personal communication, 2014).

Future Work

This project is complete except for publication of journal articles, and conference presentations (see Stavinga et al., 2014a, 2014b). The MSc thesis is available at <http://library.queensu.ca/research/format/finding-theses>.

The results were presented at the annual meeting of the Geological Society of America in October 2014, and at the Geoscience Forum in November 2014. We expect to publish the results in *Geochemistry: Exploration, Environment and Analysis*.

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