The Role of Barium Carbonates in Acid Base Accounting: Evidence from the Macmillan Pass Zn-Pb-Ag Deposits and the Mactung W Deposit

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ABSTRACT

The ability to accurately quantify the neutralization potential (NP) of tailings is critical for designing mine waste management systems. However, there is a lack of literature on the NP of samples containing naturally occurring barium carbonate minerals. In this study, fifteen samples from the Macmillan Pass zinc-lead-silver deposits and the Mactung tungsten skarn deposit were analysed using chemical and mineralogical acid-base accounting methods. Chemical methods included the modified ABA and Net-Acid Generation (NAG) tests. Mineralogical methods included the use of automated mineralogy by scanning electron microscope (SEM) and electron microprobe (EMP) analysis to calculate mineralogical carbonate-NP. The Macmillan Pass samples contained average carbonate concentrations of 16 wt.% siderite ($FeCO_3$,), which does not contribute to NP, 1.1 wt.% barytocalcite ($Ba_{1.02}Ca_{0.96}Mn_{0.01}Sr_{0.01}(CO_3)_2$), 1 wt.% witherite ($Ba_{0.93}Sr_{0.06}Mn_{0.01}CO_3$), less than 0.5 wt.% ankerite ($Ca_{1.02}(Mg_{0.65}Fe_{0.3}Mn_{0.03})(CO_3)_2$), and less than 0.5 wt.% calcite ($Ca_{0.99}Mg_{0.01}CO_3$). Calcite was the sole contributor to NP in the Mactung samples with an average concentration of 6.5 wt.%. The average modified ABA NP was 18 and 64 kg $CaCO_3$ eq/t for Macmillan Pass and Mactung, respectively. The average mineralogical NP was 16 and 65 kg $CaCO_3$ eq/t for Macmillan Pass and Mactung, respectively. Barytocalcite and witherite provided averages of 7.5 and 4.8 kg $CaCO_3$ eq/t of neutralization capacity, respectively, for Macmillan Pass. The mineralogical and modified ABA results were also compared to the NAG capacity in units of kg H_2SO_4/t. The comparison of chemical and mineralogical NP showed that barium carbonates do contribute to NP in Macmillan Pass samples.

Key Words: neutralization potential, acid rock drainage, automated mineralogy, net-acid generation test, barytocalcite, witherite

INTRODUCTION

Understanding the potential for acid rock drainage (ARD) from mine tailings is necessary to design management systems that minimize risk. ARD can occur when tailings contain iron sulfide minerals that are exposed to oxygen and water, resulting in sulfide oxidation reactions. ARD can be mitigated, however, if neutralizing minerals are present and can neutralize the acidity produced by the iron sulfide oxidation reactions. The minerals with the greatest neutralizing capacity include non-Fe/Mn carbonate minerals, such as calcite and dolomite. Some silicate minerals, such as olivine and wollastonite, may contribute to NP but are less reactive (Jambor et al. 2002). Static prediction techniques, such as acid base accounting (ABA) or net-acid generation (NAG) tests, are used to predict the ARD potential of tailings by comparing the expected acid production to the neutralization capacity. ABA measures the acid potential (AP), which is a measure of the acidity produced by a sample, and neutralization potential (NP), which is a measure of alkalinity released when a sample undergoes weathering (Jambor et al. 2007). ABA results are typically expressed using measurements of $CaCO_3$ equivalent, which is the amount of acidity that can be neutralized by calcite dissolution (Morin and Hutt, 2006). Calcite ($CaCO_3$) is the carbonate mineral with the greatest neutralizing capacity, due to its solubility, but other carbonate minerals contribute to neutralization as well. The neutralizing capacity of a carbonate mineral is affected by both solubility and the cations that comprise the mineral. Carbonate minerals containing cations that oxidize and produce acid (such as Fe, Mn) are not expected to contribute to the net NP (Dold, 2017). The neutralizing effectiveness of dolomite

(CaMg(CaCO$_3$)$_2$), magnesite (MgCO$_3$), ankerite ((Ca,Mg,Fe)(CO$_3$)$_2$) and siderite (FeCO$_3$) has been well studied, but there are few studies on the NP of barium carbonates such as witherite (BaCO$_3$) and barytocalcite (CaBa(CO$_3$)$_2$) (Blowes et al. 2014; Busenberg and Plummer, 1986; Castillo et al. 2015; Chang, 1965; Chou et al. 1989; Dold, 2017; Paktunc, 1999; Pokrovsky and Schott, 2002; Skousen et al. 1997; Zendelska et al. 2022). Chou et al. (1989) determined that witherite has a similar dissolution rate to calcite. Castillo et al. (2015) showed that BaCO$_3$ increased pH when added to acid or alkaline mine drainage for remediation. These studies demonstrate that barium carbonates in mine tailings have the potential to contribute to NP.

The purpose of this study was to investigate the NP of barium carbonate minerals in two mineral deposits located in Northern Canada: barium carbonates in simulated tailings from the Macmillan Pass deposits, compared to the NP of calcite-bearing simulated Mactung tailings. Macmillan Pass and Mactung are advanced exploration projects near each other and are owned by the same company, Fireweed Metals (JDS Energy & Mining Inc., 2018). Blending tailings from the two sites has been considered to mitigate ARD, but the mineralogy must be well-understood. The Macmillan Pass deposits are a series of sulfide-hosted zinc (Zn) – lead (Pb) – silver (Ag) deposits in the Yukon (Figure 1). The primary ore minerals are sphalerite ((Zn,Fe)S) and galena (PbS) and the tailings are expected to contain pyrite (FeS$_2$) barite (BaSO4), quartz (SiO$_2$), and carbonate minerals, including siderite, ankerite, cerussite (PbCO$_3$), barytocalcite, and witherite (JDS Energy & Mining Inc., 2018; Magnall et al. 2020; Magnall et al. 2014; Surrette et al. 2023). The Macmillan Pass tailings are expected to be classified as potentially acid generating (PAG) due to the lack of net neutralizing carbonates compared to the concentrations of pyrite. ARD is evident on the property from previous underground development during exploration (Woloshyn et al. 2011).

Figure 1. Location map for the Macmillan Pass and Mactung projects

Mactung is a tungsten (W) skarn deposit located approximately 10 km from Macmillan Pass (Figure 1) on the Northwest Territories and Yukon border. The primary ore mineral is scheelite (CaWO$_4$) and the tailings are expected to contain pyrrhotite (Fe$_{(1-x)}$S), silicates and calcite (Kirkham Geosystems Ltd., 2023; Surrette et al. 2023). While calcite is present in the Mactung tailings, the tailings are expected to be

classified as PAG due to the abundance of pyrrhotite. Since Mactung is a skarn deposit containing calcite and is close to Macmillan Pass, blending the Macmillan Pass and Mactung tailings is being considered to leverage the neutralization potential of calcite to mitigate ARD from Macmillan Pass. The Mactung tailings, however, are known to contain pyrrhotite, so environmental desulfurization to remove pyrrhotite from the Mactung tailings was investigated as a further mitigation measure.

MATERIALS AND METHODS

Materials and Sample Preparation

Core and outcrop samples of ore-bearing material were collected from Macmillan Pass and Mactung in 2022 (Table 1). The samples were shipped to Queen's University for sample processing and analysis, and subsamples of Macmillan Pass core (MP-C) were prepared into thin sections for analysis by scanning electron microscope (SEM) with automated mineralogy software (mineral liberation analysis, MLA; Surrette, 2024). Preparation of the remaining Macmillan Pass and Mactung samples followed similar procedures to create simulated feed and tailings products. The samples were crushed to 100% passing 6 mesh using laboratory-scale jaw and roll crushers and homogenized and split using a rotary sample splitter. Grinding was conducted in a 1 kg Titan laboratory rod mill with stainless steel grinding media at 60% solids w/w. The target grind size was 80% passing 50 μm for Macmillan Pass and 80% passing 150 μm for Mactung based on preliminary flowsheets developed for each site (JDS Energy & Mining Inc., 2018; Wardrop Engineering Inc., 2009). The post-grinding products were termed Macmillan Pass Feed (MP-F) and Mactung Feed (MT-F) as they represent the feed material for ore recovery. The MT-F sample was also used to simulate the tailings that will be produced at Mactung, as scheelite ($CaWO_4$), the ore mineral, is a stable and relatively insoluble oxide mineral, which does not contribute to the acid or neutralization potential of a sample.

Table 1. Sample descriptions

Sample ID	Site	Sample Type and Preparation	Number of Samples
MP-C	Macmillan Pass	Core – polished thin section	9
MP-F	Macmillan Pass	Feed – crushing and grinding	2
MP-T	Macmillan Pass	Tailings – crushing, grinding and flotation (sphalerite and galena)	4
MT-F	Mactung	Feed – crushing and grinding	3
MT-DS	Mactung	Desulfurized feed – crushing, grinding, magnetic separation and flotation (pyrrhotite)	3
MT-MP	Mactung and Macmillan Pass	20% Mactung Feed (MT-F) and 80% Macmillan Pass Tailings (MP-T)	3

The ore minerals at Macmillan Pass, sphalerite ($(Zn,Fe)S$) and galena (PbS), could contribute to the AP of the samples, so flotation was conducted to recover sphalerite and galena to simulate Macmillan Pass tailings. The flotation process involved a four-stage lead rougher circuit and a two-stage zinc rougher circuit. The resulting product was termed Macmillan Pass Tailings (MP-T). A blend of MT-F and MP-T was created to investigate the impacts on the AP and NP. A ratio of 5:1 MP-T to MT-F was used based on estimates of expected tailings volumes from each deposit. Flotation was also conducted on MT-F samples to recover pyrrhotite ($Fe_{(1-x)}S$) for environmental desulfurization. The process involved separation of

magnetic pyrrhotite using a hand-magnet and a two-stage sulfide rougher circuit. The resulting product was termed Mactung Desulfurized (MT-DS). Additional details about the Macmillan Pass and Mactung flotation work is available in Surrette et al. (2023). Samples of each product were split into 3 subsamples for analysis by SEM-MLA for mineralogical characterization, modified ABA, and NAG testing.

Mineralogical Characterization

Mineralogical characterization by SEM-MLA was conducted on polished epoxy grain mounts of MP-F, MP-T, MT-F, MT-DS, and MT-MP samples. Analyses were performed by the author using a Thermo Scientific™ (formerly FEI) Quanta™ 650 Field Emission Gun (FEG) environmental (E)SEM at Queen's University. The primary MLA mode used was x-ray modal (XMOD) analysis to assess modal mineralogy. A mineral reference library was developed based on literature and manual investigation and is available in Surrette (2024). The modal mineralogy was validated by comparing the MLA assigned phase to manually collected energy dispersive spectroscopy (EDS) spectra of a subset of grains.

Electron microprobe (EMP) analysis was conducted on polished thin sections of MP-C samples to determine the compositions of carbonate minerals and the iron content of sphalerite. The JEOL JXA-8320 electron microprobe at Queen's University was used for analysis under wavelength dispersive mode. Operating conditions used were 15 kV accelerating potential, 10 nA beam current, and a 3-10 μm beam for carbonate mineral analyses and a focused beam for sphalerite analyses. Thirty carbonate grains and 9 sphalerite grains were analysed at 2 to 4 spots, resulting in a total of 158 carbonate mineral composition analyses and 48 sphalerite composition analyses. Siderite, rhodonite, dolomite, synthetic ZnS, strontianite, and witherite were used as calibration standards for the carbonate mineral analyses. Synthetic ZnS, synthetic CdS, synthetic pyrrhotite, and synthetic digenite were used as calibration standards for the sphalerite analyses.

Acid Base Accounting

Mineralogical and chemical acid base accounting methods were used to determine the neutralization and acid potentials of the Macmillan Pass and Mactung samples. Mineralogical AP and mineralogical carbonate NP were calculated based on the modal mineralogy determined by SEM-MLA. The mineralogical method involved calculating the AP based on the amount of sulfide-sulfur calculated from the weight percent (wt.%) of iron sulfide minerals in the sample and the NP based on the wt.% of carbonate minerals in the sample, including a correction for iron-bearing carbonate minerals (Dold, 2017, Frostad et al. 2003, Karlsson et al. 2018, Lapakko, 1994, Paktunc, 1999). Manganese concentrations in the carbonate minerals in the Macmillan Pass samples were low (less than 1 oxide wt.%) and not included in calculations. The wt.% of carbonate minerals were converted to a $CaCO_3$ equivalent value based on the concentrations of cations determined by EMP analysis. Using the assumption that one mole of calcite neutralizes the two moles of acid produced from the oxidation of one mole of sulfur, a calculation factor of 31.25 was used to determine the AP of pyrrhotite and pyrite and a factor of 31.25/2 was used to determine the AP of chalcopyrite, arsenopyrite and sphalerite. A detailed description of this method is available in Surrette et al. (2024). NP results for two scenarios (1) assuming barium carbonates provided full neutralization capacity and (2) assuming barium carbonates provided no NP were compared to the chemical ABA NP to assess the impact of barium carbonates.

The chemical ABA NP method used was the modified ABA test (Lawrence and Wang, 1996). The AP was calculated by multiplying the amount of sulfide-sulfur by a factor of 31.25. The amount of sulfide sulfur was determined by calculating the difference between the measured total sulfur (by induction furnace; LECO) and sulphate sulfur (by acid leach). The NP was determined by adding a known amount of HCl to 2 g of sample, allowing the sample to react, then titrating back to a pH of 8.3 using NaOH (0.1 N).

Net-Acid Generation Testing

Single addition NAG tests following the procedure outlined by Stewart et al. (2006) and AMIRA (2002) were used to compare with the ABA classifications. The NAG test involved adding 150 mL of hydrogen peroxide to 1.5 g of sample and allowing the mixture to react at room temperature. The mixture was then heated to help the reaction proceed, followed by cooling and measurement of the pH (the NAG pH). The mixture was then titrated back to a pH of 4.5 and 7 using NaOH (0.1 N).Samples with high sulfide concentrations may not be fully oxidized during a single addition NAG test due to the catalytic breakdown of hydrogen peroxide (AMIRA, 2002). This could result in uncertainty in the classification when compared with the mineralogical or modified ABA classifications.

RESULTS AND DISCUSSION

Mineralogical characterization of Macmillan Pass and Mactung samples is shown in Figure 2. The MP-F samples consisted primarily of pyrite (7.6 wt.%), sphalerite (14 wt.%), barite (32 wt.%), and silicate (20 wt.%), and carbonate minerals (18 wt.%). MP-T samples had similar compositions of pyrite (6.3 wt.%), barite (42 wt.%), silicate minerals (26 wt.%), and carbonate minerals (21 wt.%); however sphalerite concentrations were reduced by flotation to an average of 0.2 wt.%. The dominant silicate minerals in the Macmillan Pass samples were quartz (average 16 wt.%), followed by illite (average 2.6 wt.%) and orthoclase (average 1.8 wt.%), with other silicate minerals having concentrations less than 1 wt.%. MT-F samples consisted primarily of pyrrhotite (21 wt.%), silicate minerals (65 wt.%), and calcite (5.5 wt.%). Desulfurization reduced pyrrhotite concentrations from 21 wt.% to an average of 2.3 wt.% in the MT-DS samples, and the remaining composition was dominated by silicate minerals (84 wt.%) and calcite (7.5 wt.%). The dominant silicate minerals in the Mactung samples were actinolite and quartz. The blended MT-F and MP-T samples (MT-MP) had compositions dominated by pyrite and pyrrhotite (15 wt.%), silicate minerals (47 wt.%), barite (21 wt.%) and carbonate minerals (12 wt.%).

Multiple types of carbonate minerals were identified in the Macmillan Pass samples, including ankerite-dolomite, barytocalcite, calcite, cerussite, siderite, and witherite. Calcite was the dominant carbonate mineral in the Mactung samples, allowing the Mactung samples to be used as a point of reference to compare with the more complicated mineralogy of the Macmillan Pass samples. The distribution of types of carbonate minerals is shown in Figure 3. Carbonate mineralogy of the MP-F and MP-T samples was dominated by siderite (average 13 wt.% and 17 wt.%, respectively), which does not usually contribute to net NP (Skousen et al. 1997). Cerussite was identified in MP-F and MP-T samples, but later EMP analyses indicated that both cerussite and anglesite were present and these phases cannot be distinguished by MLA. Therefore, cerussite was not included in mineralogical ABA calculations because the concentrations could not be properly determined. Barytocalcite (0.8 wt.% in MP-F samples and 1.3 wt.% in MP-T samples) and witherite (0.7 wt.% in MP-F samples and 1.1 wt.% in MP-T samples) were the most abundant carbonate minerals that contribute to NP in the Macmillan Pass samples. Blending MT-F material with MP-T increased calcite concentrations to an average of 2.1 wt.%, although the MT-MP samples were still dominated by siderite (8.4 wt.%).
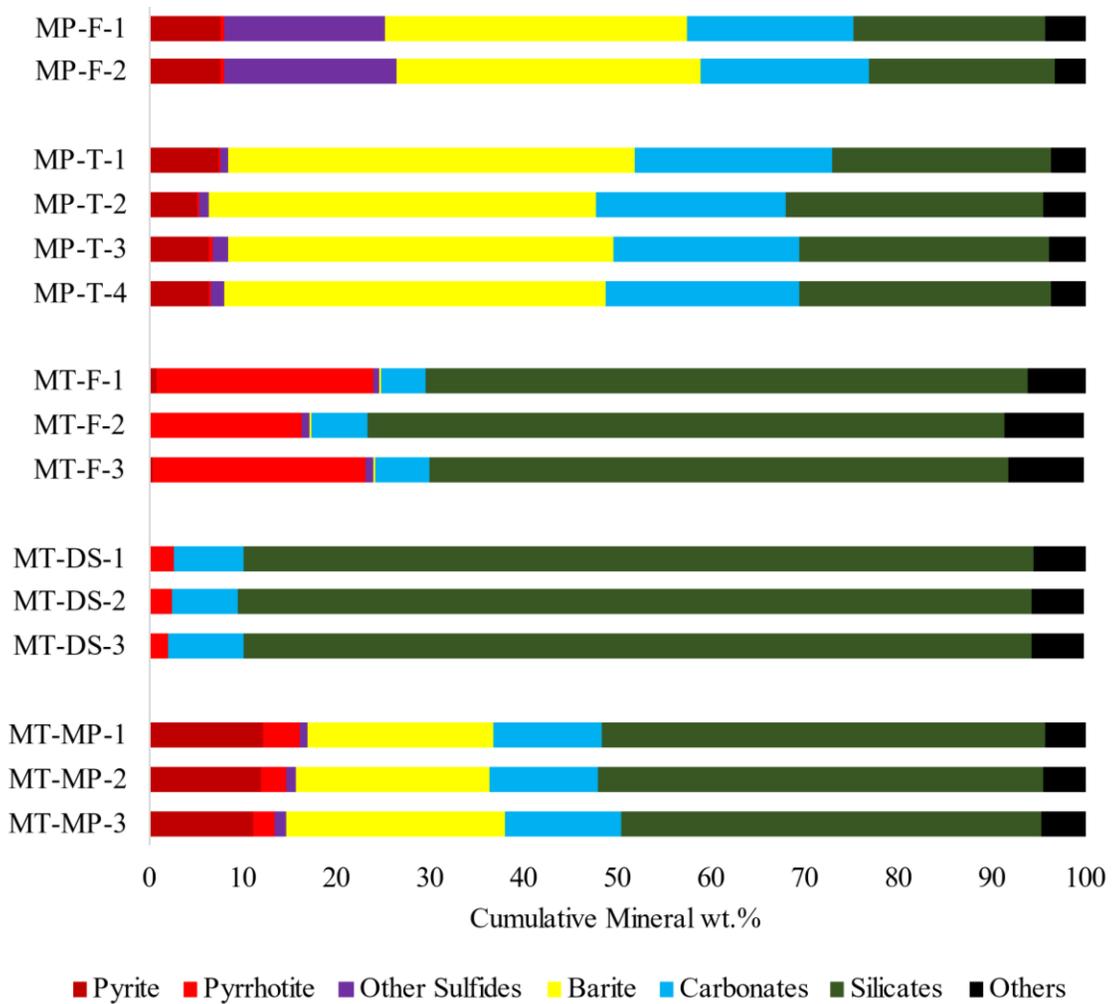
Figure 2. Modal mineralogy of Macmillan Pass (MP-F, MP-T), Mactung (MT-F, MT-DS), and blended (MT-MP) samples. Other sulfides include sphalerite, galena, chalcopyrite and arsenopyrite. Carbonates include calcite, dolomite-ankerite, siderite, witherite, and barytocalcite. Silicates include quartz, actinolite, clays, feldspars, and other minor silicates.
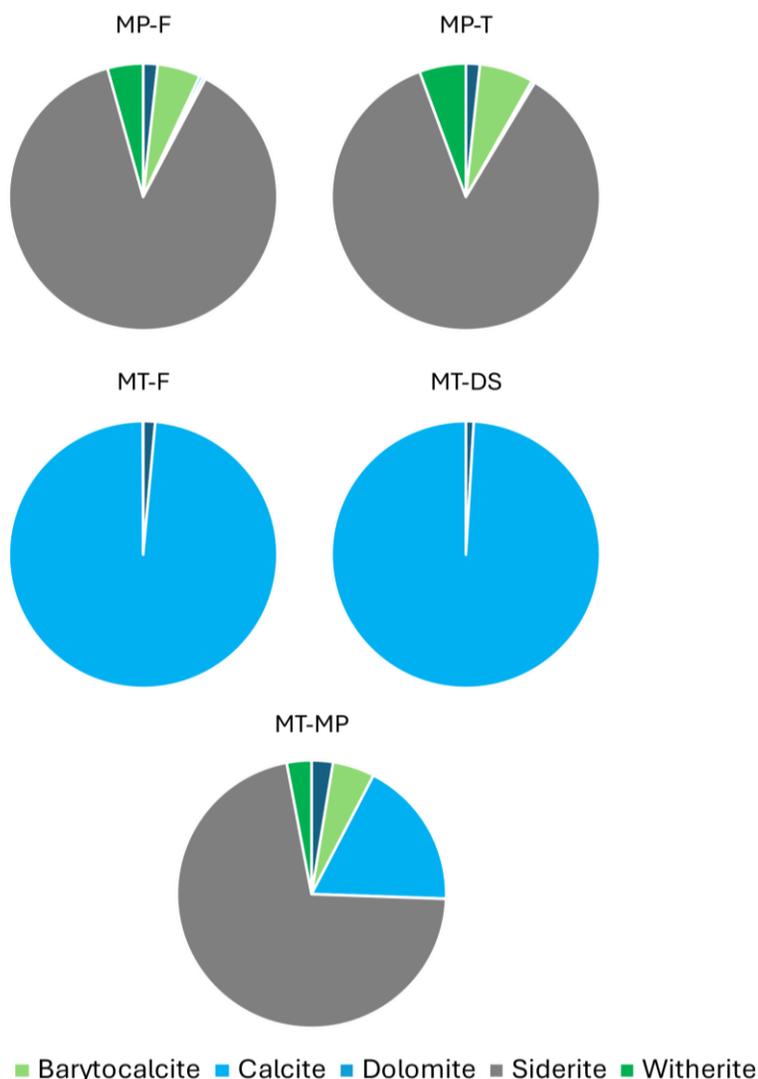
Figure 3. Average distribution of carbonate minerals in Macmillan Pass, Mactung and MT-MP blend samples

The average composition of each carbonate mineral and sphalerite was determined by EMP analyses of the Macmillan Pass core samples (MP-C). The results are shown in Table 2. The carbonate mineral compositions were used to calculate the mineralogical NP. The iron content measured in sphalerite was determined to be insignificant, so sphalerite was not included in mineralogical AP calculations.

Table 2. Mineral formulas of Macmillan Pass carbonate minerals and sphalerite determined by EMP

Mineral	Number of Analyses	Average Formula
Ankerite-dolomite	20	$Ca_{1.02}Mg_{0.65}Fe_{0.3}Mn_{0.03}(CO_3)_2$
Barytocalcite	15	$Ba_{1.02}Ca_{0.96}Mn_{0.01}Sr_{0.01}(CO_3)_2$
Calcite	11	$Ca_{0.99}Mg_{0.01}CO_3$
Siderite	11	$Fe_{0.89}Mn_{0.05}Zn_{0.03}Ca_{0.02}Mg_{0.01}CO_3$
Witherite	24	$Ba_{0.93}Sr_{0.06}Mn_{0.01}CO_3$
Sphalerite	35	$Zn_{0.99}Fe_{0.01}S$

Mineralogical carbonate NP and AP results are plotted in Figure 4 (A). The MP-F, MP-T, MT-F and MT-MP samples were classified as PAG and the MT-DS samples were classified as non-potentially acid generating (NPAG; NPR > 2). Barytocalcite had the greatest contribution to mineralogical NP for the MP-F (5.2 kg $CaCO_3$ eq/t) and MP-T (8.6 kg $CaCO_3$ eq/t) samples, followed by witherite (3.4 kg $CaCO_3$ eq/t for the MP-F samples and 5.7 kg $CaCO_3$ eq/t for the MP-T samples). Calcite was the greatest contributor to mineralogical NP in the MT-F, MT-MP, and MT-DS samples. The concentration of calcite increased in the MT-MP samples compared to the MP-T samples, resulting in an increase in mineralogical NP, but the addition of pyrrhotite from the MT-F material also increased the AP.
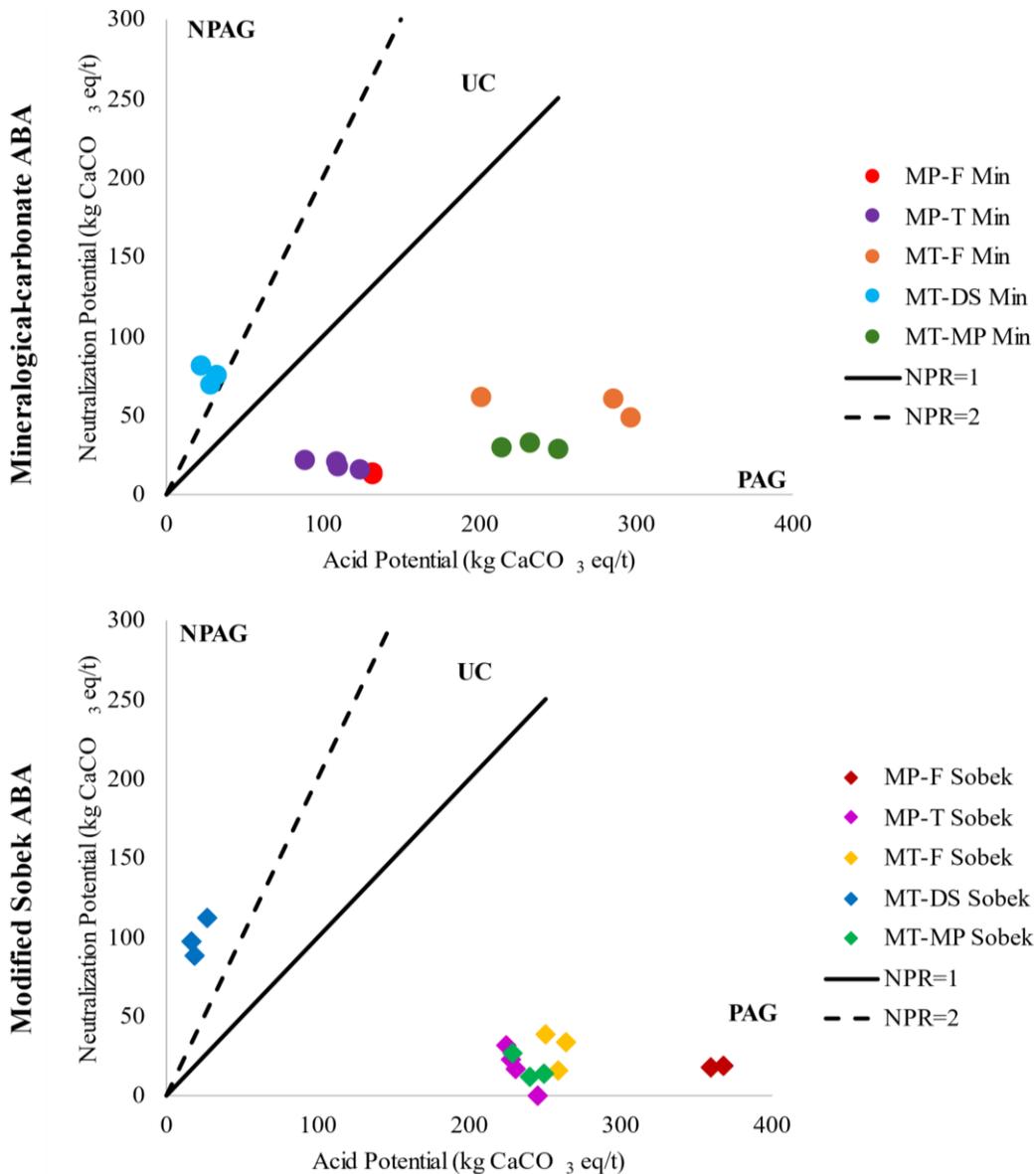

Figure 4. (A) Mineralogical-carbonate NP vs. AP and (B) modified Sobek ABA NP vs. AP for Macmillan Pass and Mactung samples

The modified ABA tests resulted in classifications of PAG for MP-F, MP-T, MT-F, and MT-MP samples and NPAG for MT-DS samples, which was in agreement with the mineralogical ABA results. The high concentrations of barite, however, resulted in an overestimation of AP because barite is relatively insoluble. Therefore, the sulfur associated with barite was not captured in the sulfate-sulfur measurement

and was assumed to be sulfide-sulfur (Donkervoort et al. 2023). This caused the modified Sobek AP values for the Macmillan Pass samples (MP-F, MP-T, and MT-MP) to be greater than the mineralogical AP values. Modified ABA results are plotted in Figure 4 (B). NP values ranged from 18.6 to 18.9 kg $CaCO_3$ eq/t for MP-F samples and ranged from 0 to 31.9 kg $CaCO_3$ eq/t for MP-T samples. Mactung samples (MT-F and MT-DS) presented similar mineralogical and modified Sobek AP and NP values. The increase in calcite concentration in the MT-MP sample compared to the MP-T sample did not have a measurable impact on the modified ABA AP or NP.

The mineralogical-carbonate NP was also calculated excluding barium carbonates to assess the difference barium carbonates make in NP (Table 3). With the exception of one sample (MT-T-1), excluding barium carbonates from the NP determination of the Macmillan Pass samples caused the NP to be substantially underestimated.

Table 3. Comparison of calculated mineralogical-carbonate NP values that included and excluded barium carbonates (barytocalcite and witherite) with modified Sobek NP results.

Sample ID		Mineralogical-carbonate NP (kg $CaCO_3$ eq/t)		Modified Sobek NP (kg $CaCO_3$ eq/t)
		Including Ba Carbonates	Excluding Ba Carbonates	
Macmillan Pass - Feed	**MP-F-1**	13	3.6	18.9
	MP-F-2	12	3.6	18.6
Macmillan Pass - Tailings	**MP-T-1**	15	3.0	0
	MP-T-2	21	4.9	31.9
	MP-T-3	17	3.1	22.7
	MP-T-4	20	4.6	17.3
Mactung - Feed	**MT-F-1**	48	48	34
	MT-F-2	61	61	16
	MT-F-3	59	59	38.6
Mactung - Desulfurized	**MT-DS-1**	74	74	97.6
	MT-DS-2	69	69	88.4
	MT-DS-3	81	81	112
Mactung (Feed) - Macmillan Pass (Tailings) Blend	**MT-MP-1**	28	23	13.8
	MT-MP-2	32	27	27.2
	MT-MP-3	29	22	12.4

The mineralogical and modified ABA results were converted to units of kg H_2SO_4/t and compared to the NAG pH results from the NAG tests (Figure 6). The NAG and ABA tests agreed on the NPAG classification of the MT-DS samples, and the PAG classification of the MP-F and MT-F samples, but disagreed on the classification of the MP-T and MT-MP samples (UC: uncertain). The MP-T and MT-MP samples were classified as uncertain when comparing the ABA and NAG results because the net acid production potential (NAPP) determined by the ABA tests was greater than 0, indicating a PAG classification, but the NAG pH measured during the NAG tests were greater than 4.5, indicating a NPAG classification. This is likely due to the high sulfide concentration which can cause the acid forming potential to be underestimated due to the breakdown of hydrogen peroxide prior to complete oxidation of all sulfide minerals (AMIRA, 2002). Further examination using a sequential or kinetic NAG test could be used to assess the uncertainty in classification of the MP-T and MT-MP samples.
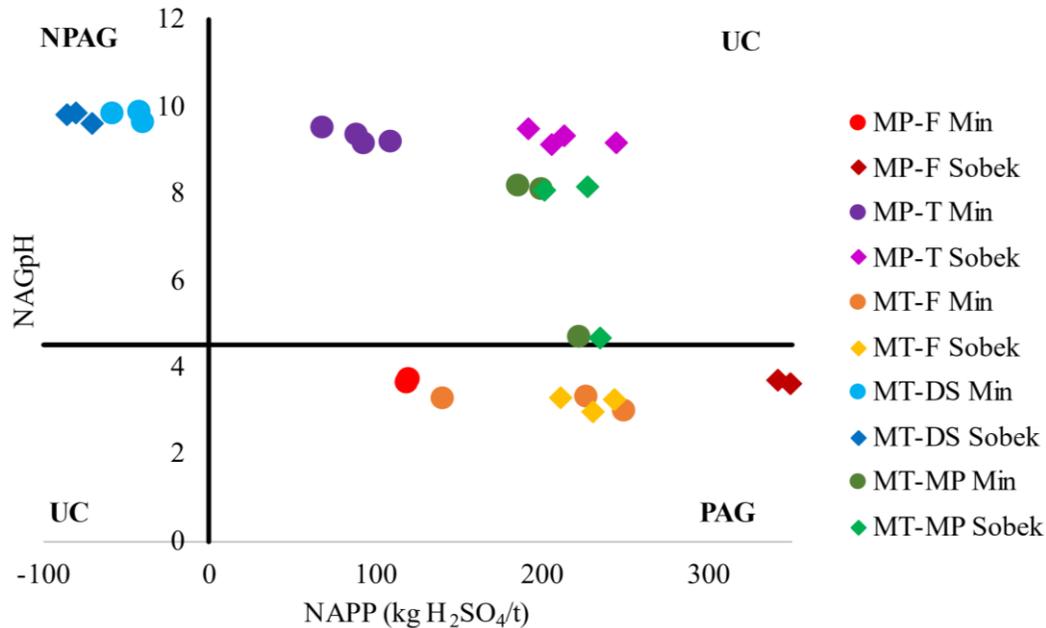
Figure 6. Comparison of NAG pH and NAPP determined by mineralogical and modified Sobek ABA for Macmillan Pass and Mactung samples

CONCLUSIONS

The ARD potential of Macmillan Pass and Mactung feed and tailings was preliminarily investigated to assess the contribution of barium carbonates to neutralization potential. Mineralogical ABA, modified ABA, and NAG tests were conducted on MP-F, MP-T, MT-F, MT-DS, and MT-MP samples to compare results across the three methods and between samples. Macmillan Pass samples contained various carbonate minerals including ankerite-dolomite, barytocalcite, siderite and witherite. Mactung samples contained calcite as the dominant carbonate mineral. Based on mineralogical ABA, barytocalcite and witherite were the dominant contributors to the NP of Macmillan Pass samples. The contribution of barytocalcite and witherite to NP was also indicated by modified ABA. Calculations of mineralogical NP excluding barium carbonates showed that the NP was substantially underestimated compared to the modified Sobek NP, demonstrating that witherite and barytocalcite contribute to the net NP of the Macmillan Pass samples. Although neutralizing capacity was provided by barytocalcite, witherite, and minor ankerite-dolomite in the Macmillan Pass samples, the samples were classified as PAG by mineralogical and modified ABA and as PAG or uncertain by NAG tests due to the concentrations of pyrite in the samples. The addition of calcite to the Macmillan Pass tailings by blending with Mactung feed did not result in a substantial increase in neutralization potential due to the increase in pyrrhotite concentrations from the Mactung material. Blending Macmillan Pass tailngs with desulfurized Mactung tailings could be a way to mitigate this issue Future work should include detailed kinetic testing as well as the investigation of samples that contain barium carbonates with limited barite, as the high barite concentrations influenced modified Sobek ABA measurements.

ACKNOWLEDGEMENTS

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