

Operational NP Determination –Accounting for Iron Manganese Carbonates and Developing a Site-specific Fizz Rating

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Abstract. Cost effective segregation and the subsequent mitigation of potentially ARD generating waste rock requires an accurate, precise and relatively quick operational determination of the neutralization potential (NP). Due to time constraints, most operators base their NP assessment on an interpretation of laboratory carbon analysis and the Sobek NP procedure. Two of the major factors that confound the interpretation of these tests are the contribution of iron and manganese carbonates and the subjective nature of the Sobek fizz rating procedure. While there are various methods to address these concerns, there is very little data in the literature showing their effectiveness or how to interpret their results.

The primary neutralizing minerals at the site were a variety of carbonate species, which were identified and quantified by energy dispersion x-ray spectrometry and the Rietveld XRD method. The Sobek fizz ratings, and the resulting Sobek-NP and Skousen-NP values, were found to differ widely between the different laboratories. A relatively accurate, precise and quick operational CO₂-based fizz rating has been developed to overcome the shortcomings of the Sobek fizz rating.

Introduction

The neutralization potential (NP) of a waste rock is a difficult parameter to measure due to the variety and reactivity of minerals involved. The presence of Fe and Mn carbonate minerals, such as siderite (FeCO₃) and ankerite (Ca(Fe,Mn)(CO₃)₂) is a concern because the alkalinity produced is offset by the acidity created by Fe and Mn oxidation and no net neutralization of acid is provided (Lapakko, 1994; Price, 1997). Determination of a carbonate NP from an inorganic-C measurement has the potential to overestimate the neutralizing capacity if Fe and Mn carbonates are present. Another concern is that the Sobek et al. (1978) NP procedure does not allow enough time for complete oxidation of released Fe and Mn and may also overestimate the NP measurement. Meek (1981) proposed adding hydrogen peroxide (H₂O₂) during the NP procedure to ensure ferrous iron is oxidized to ferric iron prior to back titration with NaOH to pH of 7.0. Skousen et al. (1997) found that using H₂O₂ provided a more accurate NP result than the standard Sobek method, however, this conclusion was primarily based on results from

samples having 18 to 65% siderite. A confounding problem with both the Sobek and Skousen NP procedures is their reliance on the Sobek rating system for determining acid addition.

Objectives

The objective of this study was to: (1) assess the minerals potentially contributing to operational NP measures; (2) investigate procedures for removing the contribution of iron and manganese carbonates; and (3) recommend a practical operational method for estimating the NP.

Materials And Methods

Samples used for this study represent Takla Sedimentary waste rock from the Kemess South gold-copper orebody, located in north central British Columbia. The Takla Sediments are a heterogeneous mix of very fine-grained, non-foliated sediments, pyroclastics and various alteration types (e.g., hematitic), including graphitic and non-graphitic cherts, cherty and tuffaceous mudstones and porcellanitic tuffs (tuffites). Pre-mining analysis of drill core

samples indicated that the Takla Sediments contained up to 4% sulphide-S with a median of 0.6%, and less than 0.1% sulphate-S. Almost all the sulphide-S occurs as pyrite, typically as disseminated minute euhedra (5 – 100 microns) and as small segregations associated with quartz/carbonate veinlets.

The study samples were primarily obtained from pre-blast drill hole cuttings with the addition of a few samples obtained from diamond drill core. All samples were pulverized in the Kemess laboratory preparation room to minus 200 mesh. Splits of the seventeen samples were sent to commercial laboratories for Sobek-NP and Skousen-NP analyses and to the University of British Columbia for XRD.

Mineralogy

X-ray powder-diffraction (XRD) data was conducted using the Rietveld method and a qualitative check of carbonate minerals was conducted on eight of the samples containing ankerite by scanning electron microscopy /energy-dispersion spectrometry (SEM/EDS) (Raudsepp and Pani, 2001). As an approximate guide, the proportion of Fe and Mn as a % of the cations in ankerite (reported as Fe mole%) was estimated using an equation for cell volume from published crystallographic data on dolomite/ankerite solid solutions. The XRD carbonate mineralogy was used to estimate the contribution of Ca and Mg carbonates to NP (reported as kg CaCO₃/tonne) and was calculated by:

$$\text{CaCO}_3\text{-NP} = (\text{calcite wt.\%} + (\text{ankerite wt.\%} \times (1 - (\text{Fe mole\%} / 100)))) \times 10 \quad (1)$$

and the potential contribution of Fe and Mn carbonates to NP (kg CaCO₃/tonne) was determined from:

$$\text{FeCO}_3\text{-NP} = ((\text{ankerite wt.\%} \times \text{Fe mole\%}) + \text{siderite wt.\%}) / 115.86 \times 100.09 \times 10 \quad (2)$$

where 115.86 and 100.09 are the molecular weights of FeCO₃ and CaCO₃, respectively.

Carbon Analysis

Inorganic carbon was measured with a carbon dioxide analyzer at a commercial laboratory. Carbon analyses are commonly used to calculate a carbonate NP (Carb-NP) because it is a parameter that can be measured operationally. Carb-NP (kg CaCO₃/tonne) is a measure of the maximum theoretical neutralization capacity if all the carbonates reacted like calcite and is calculated by:

$$\text{Carb-NP} = \% \text{CO}_2 \times (100.09/44.01) \times 10 \quad (3)$$

where %CO₂ is the inorganic-C measurement, and 100.09 and 44.01 are the molecular weights of CaCO₃ and CO₂, respectively.

Sobek-NP Procedure

The Sobek NP procedure (Sobek et al., 1978) requires a “fizz” test to be initially performed. One or two drops of 25% HCl is added to a small amount of the sample and the reaction is rated as none, slight, moderate, or strong. Hydrochloric acid is added to 2 g of sample based on the fizz ratings (none = 20 mL 0.1N, slight = 40 mL 0.1N, moderate = 40 mL 0.5N, and strong = 80 mL 0.5N), the mixture is heated, de-ionized water is added, then the mixture is boiled. The amount of acid neutralized by the sample is determined by titrating with 0.1 or 0.5 N NaOH (dependant on the concentration of the acid used) to pH 7.0. The Sobek NP (kg CaCO₃/tonne) is calculated as follows:

$$\text{Sobek-NP} = (\text{mL of acid consumed}) \times 25.0 \times (\text{N of acid}) \quad (4)$$

Skousen Neutralization Potential

The Skousen-NP procedure (Skousen et al., 1997) also uses the four-tiered Sobek rating system to determine acid addition. After acid is added to the sample, de-ionized water is also added and the mixture is boiled. For this study, gravity filtering of the contents was conducted using a 2.5 µm filter. H₂O₂ is added to the filtrate then the solution is boiled and titrated with NaOH to pH 7.0.

Results And Discussion

Results and values derived from the XRD, fizz rating, Sobek-NP and Skousen-NP assays are provided in Table 1.

Mineralogy

The Rietveld XRD results of the carbonate mineralogy showed that FeCO_3 was common in the Takla Sediment waste rock (Table 1). SEM/EDS analysis indicates that Mg is a significant component of ankerite, and that Mg and Ca are significant components of siderite. The presence of Mn was only noted in two of seven ankerite grains. The SEM/EDS results suggest that iron carbonates may provide greater amounts of Ca and MgCO_3 than was accounted for in our FeCO_3 -NP calculation. For samples having calcite as the only carbonate mineral, the CaCO_3 -NP (from XRD data) was similar to Carb-NP (from CO_2 measurements). The comparable results (differences less than 10 kg CaCO_3 /tonne) indicate that the Rietveld method provided an accurate measure of CaCO_3 content.

The common non-carbonate minerals included quartz (37-88%), muscovite (present in all but 2 samples, 1.9-15%) and kaolinite (present in all but 3 samples, 1.3-32%). Chlorite (clinocllore), orthoclase and albite were less common minerals and minor amounts of illite, hematite and barite were noted in a few samples. Chlorite (3.6 to 29%) was only detected in the samples with FeCO_3 -NP <10 kg CaCO_3 /tonne (Table 1), while kaolinite was most common in the samples with FeCO_3 -NP >10 kg CaCO_3 /tonne.

Influence of Fizz Rating on Sobek-NP and Skousen-NP Results

For the same samples, Lab A consistently assigned a lower fizz rating than Lab B. Using a slight fizz rating (Lab A) versus a moderate or strong rating (Lab B) resulted in Sobek and Skousen-NP values being an average of 21 and 29 kg CaCO_3 /tonne lower, respectively. When a moderate fizz rating was used rather than a strong rating, inconsistencies between lab results were much smaller. Moderate versus strong ratings resulted in an average difference between Sobek-NP values of only 5 kg CaCO_3 /tonne, and for Skousen-NP values of 8 kg CaCO_3 /tonne.

Previous studies have also shown that greater acid additions can return greater Sobek and Skousen-NP results (Lawrence and Wang, 1996; Skousen et al., 1997; White et al., 1998) and this study indicates that the Takla Sediments are particularly sensitive to a slight versus moderate assigned fizz rating.

The amount of NP that can be consumed for each of the four Sobek acid additions may be represented in NP units of kg CaCO_3 /tonne. For example, a fizz rating of 'none' requires 20 mL of 0.1N HCl to be added during the procedure. Applying Equation 4, the theoretical amount of NP that can be consumed during a Sobek-NP test using the 'none' fizz rating is 50 kg CaCO_3 /tonne. The theoretical maximum NP consumption for each fizz rating during a Sobek-NP test (none = 50, slight = 100, moderate = 500, and strong = 1000 kg CaCO_3 /tonne) is supported by research. Jambor et al. (2000) conducted Sobek-NP analyses on calcite using acid additions equivalent to 50, 250, and 1000 kg CaCO_3 /tonne that returned values of 49.9, 249 and 926 kg CaCO_3 /tonne, respectively.

The Sobek acid additions as NP equivalents are useful for interpreting Sobek-NP values if the results are accompanied by the assigned fizz rating. From the data set (Table 1), Sobek-NP analyses conducted at Lab B are shown to have received a strong fizz rating for most samples and were therefore provided with enough acid to consume 1000 kg CaCO_3 /tonne. Since the maximum Sobek-NP result returned was 166 kg CaCO_3 /tonne, no sample should have been provided with a rating greater than moderate (500 kg CaCO_3 /tonne). Since Lab A added more reasonable quantities of acid than Lab B for their Sobek and Skousen-NP analyses, only the results from Lab A will be discussed further.

Contribution of Non-neutralizing Carbonate Minerals to Sobek-NP

A comparison of Skousen and Sobek-NP results suggests that iron carbonates have a relatively small impact on the Sobek-NP. For samples containing iron carbonates (i.e., $\text{FeCO}_3\text{-NP} > 10$ kg $\text{CaCO}_3/\text{tonne}$), the Sobek-NP minus Skousen-NP was only 0 to 8 $\text{CaCO}_3/\text{tonne}$ even though the $\text{FeCO}_3\text{-NP}$ values were as high as 74 kg $\text{CaCO}_3/\text{tonne}$. Assuming the Skousen-NP results correctly accounted for the presence of iron carbonates in the study samples, the Sobek-NP is overestimated by 0 to 9% with an average error of 3%. It is considered that most of the Fe and Mn released during the Sobek-NP analysis is oxidized and hydrolyzed prior to the end of the titration. The low levels of Mn within siderite and ankerite (SEM/EDS) may also help explain why these minerals have a low influence on Sobek-NP since Mn oxidizes more slowly than Fe.

Samples having little or no iron carbonates (i.e., $\text{FeCO}_3\text{-NP} < 10$ kg $\text{CaCO}_3/\text{tonne}$) returned Skousen-NP values greater than the Sobek-NP (1 to 7 kg $\text{CaCO}_3/\text{tonne}$). One possible explanation for the higher Skousen-NP values is a loss of leachate during filtering. Based on the difference between the Sobek and Skousen-NP values, 3 to 5% of the total leachate volume may have been lost during the Skousen procedure. Measuring and correcting for any change in leachate volume may have avoided this error.

The relationship between Carb-NP and Sobek-NP appears to be a reliable method of identifying Takla Sediment samples that contain iron carbonates. The Carb-NP was consistently greater than Sobek-NP for all samples having a $\text{FeCO}_3\text{-NP} > 10$ kg $\text{CaCO}_3/\text{tonne}$. For these samples, the potential contribution of non-neutralizing carbonates to Sobek-NP can be conservatively corrected for by reducing the Sobek-NP by 10%.

Contribution of Non-carbonate Minerals to Sobek-NP

Another concern when using the Sobek procedure to predict NP is an excessive contribution by non-carbonate minerals.

Although silicate minerals may provide a source of neutralization (Kwong and Ferguson, 1997; White et al., 1998), they are far less effective neutralizers than carbonates due to their slow dissolution rates. Studies by Jambor et al. (2000 and 2002) measured the Sobek-NP of non-carbonate minerals using a 'none' fizz rating and obtained 0.3 kg $\text{CaCO}_3/\text{tonne}$ for muscovite, 0 kg $\text{CaCO}_3/\text{tonne}$ for kaolinite, 0.8 to 10.3 kg $\text{CaCO}_3/\text{tonne}$ for clinocllore, 0.5-1.4 kg $\text{CaCO}_3/\text{tonne}$ for K-feldspar, 0.5-0.6 kg $\text{CaCO}_3/\text{tonne}$ for albite, and 2 kg $\text{CaCO}_3/\text{tonne}$ for hematite. Results from Jambor et al. suggest that clinocllore was the primary source, but not the only source, of non-carbonate NP when excessive volumes of acid were added during the Sobek procedure at Lab B.

The study exemplifies the potential of non-carbonate minerals to provide an unreasonable amount of the NP when subjected to excessively acidic conditions during the Sobek procedure. To rectify the problem of acid addition, the determination of a relationship between CO_2 measurements and acid addition was pursued.

Development of a CO_2 -based Acid Addition for Sobek-NP Determination

The amount of NP that can be consumed for each of the four Sobek acid additions may be represented as % CO_2 . As previously discussed, the theoretical amount of NP that can be consumed during a Sobek-NP test using the slight fizz rating is 100 kg $\text{CaCO}_3/\text{tonne}$. If the NP of a sample is assumed to be solely from carbonates, and is at least 100 kg $\text{CaCO}_3/\text{tonne}$, then 4.4% CO_2 would be evolved using a slight fizz rating (Equation 3). The theoretical amounts of CO_2 (%) that would evolve from unlimited carbonate for all four Sobek acid additions are provided in Table 2.

Kemess Mine is currently using a CO_2 -based rating system to determine acid addition for the Sobek procedure. The Kemess CO_2 ranges are slightly less than the CO_2 equivalent of each fizz rating (Table 2) to permit a slight excess of acid to be available for dissolution of non-carbonate minerals. The CO_2 rating does not account for the presence of iron carbonates, therefore, an

excessive amount of acid may be added and an overestimation of non-carbonate NP may result. Test work is in progress to measure, and then if necessary minimize, the rate of ankerite and siderite dissolution in the CO₂ assay. In the meantime, the criteria used by the mine to identify potentially acid generating (PAG) Takla Sediments is a net potential ratio (Sobek-NP divided by the acid potential) of ≤ 3 , $> 0.8\%$ sulphide-S or the presence of carbonaceous chert. The net potential ratio criterion of 3 for pre-blast samples was set to account for the concentration of pyrite in fines. The criteria for sulphide-S and carbonaceous chert were derived from an analysis of acid base accounting results that were obtained from an extensive drilling program of the Takla waste rock and should ensure that no PAG rock is incorrectly classified.

Table 2. Sobek Acid Addition as CO₂ Equivalent and the Kemess CO₂ Rating System.

Sobek Fizz Rating	Sobek Acid Addition (kg CaCO ₃ /t)	CO ₂ Equiv. (%)	Kemess CO ₂ Ratings (%)
None	50	2.2	0 – 1.5
Slight	100	4.4	1.5 – 4.0
Moderate	500	22	4.0 – 15
Strong	1000	44	> 15

Measurement of CO₂

The Vortex CO₂ Method (Onggowidgaja, 1999) is being used at Kemess to provide a quick and simple CO₂ measurement. LECO equipment is not utilized since organic carbon from graphitic material would also report to the CO₂ during analysis. The Vortex CO₂ procedure requires a test tube to be filled with 5 mls of 70% HCl and then weighed. A representative sample is crushed to minus 200 mesh and 1.0000 gram is added to the test tube. The test tube containing the sample and acid is vortexed for 60 seconds and then weighed again. Multiplying the difference between the initial weight and the post-vortexed weight by 100 provides an estimate of inorganic-C as %CO₂.

Proposed Refinement of CO₂-based Rating System

Use of a sliding scale for determining acid addition as opposed to the four-tiered Sobek rating may result in more precise Sobek-NP results. Although basing acid addition on CO₂ measurements ensures Sobek-NP results are reproducible, the four-tiered Sobek rating system can result in an unreasonable contribution to NP by non-carbonate minerals. For example, a sample returning 3.9% CO₂ at Kemess would be assigned a slight rating and a small amount of excess acid would be available for non-carbonate mineral dissolution. A sample returning a slightly higher CO₂ value of 4.1% would receive five times as much acid (moderate rating) and the excessive acid may result in an unrealistically high contribution to NP from non-carbonate minerals. By using a sliding scale, the amount of acid available for dissolving non-carbonates could be controlled.

The proposed sliding scale allows a specific volume of acid to be added for carbonate dissolution, then an additional amount of acid added for non-carbonate dissolution. Combining the relationship between the four Sobek acid additions (Figure 1) with Equation 4 allows the acid volume required to dissolve the carbonate minerals to be determined from:

$$\text{Acid for Carbonate Dissolution (mL)} = \%CO_2 \times 22.7 / 25.0 / N \text{ of acid} \quad (5)$$

The acid volume required for measuring non-carbonate NP can be based on mineralogical results and studies specifically designed to evaluate the response of non-carbonate minerals to acid strength and volume. For example, non-carbonate NP would be limited to 20% of a Sobek-NP result by adding 20% more acid than is required for carbonate dissolution. Another approach would be to limit the non-carbonate NP to a specific amount by always adding the same volume of excess acid (e.g. 25 kg CaCO₃/tonne = 10 mL of 0.1N HCl).

Conclusions

A study was undertaken to quantify non-neutralizing Fe and Mn carbonate minerals and to determine their impact on the Sobek-NP and Carb-NP. Based on the study results, the potential contribution by non-neutralizing carbonates is avoided by reducing the Sobek-NP by 10% whenever the Carb-NP is greater than Sobek-NP.

The remaining concern was avoiding a potential overestimation of non-carbonate NP. Based on the results of this study and supported by literature, the solution is to use an acid addition that only slightly exceeds the CaCO₃ and MgCO₃ content. Test work is underway at Kemess to develop an acceptably accurate, precise and quick operational assay for CaCO₃- and MgCO₃-CO₂ for materials potentially containing siderite and ankerite.

A refinement of the CO₂-based rating system has been proposed to avoid potential problems using the four-tiered Sobek rating. Acid addition based on a sliding scale would have the advantage of limiting the contribution of slow weathering non-carbonate minerals to a Sobek-NP measurement, but testing is required to validate the proposed rating system.

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Table 1. XRD Carbonate Quantification Samples with Comparison of Carbonate-NP, Sobek-NP, and Skousen-NP.

No.	XRD Analysis (wt%)				XRD NP			Lab A					Lab B		
	Cal.	Ank	Sid.	Chl.	CaCO ₃	FeCO ₃	Carb-	Fizz	Sobek	Skousen	Skousen	Carb -	Fizz	Sobek	Skousen
					NP	NP	NP	Rating	NP	NP	- Sobek	Sobek	Rating	NP	NP
S1	0.3	7.1	5.9		47	74	138	Mod	102	97	-5	36	Strong	105	99
S2		6.0	5.3		41	62	125	Slight	68	67	-1	57	Strong	82	82
S3	7.2	1.5	4.5		83	42	130	Slight	86	83	-3	44	Strong	101	101
S4	8.7	5.2	2.5		124	35	178	Mod	154	153	-1	24	Strong	166	155
S5		6.4	2.4		42	40	108	Mod	95	87	-8	13	Strong	89	86
S6	0.1	1.7	1.5		14	16	38	Slight	28	28	0	10	Mod	28	28
S7	12.9	4.7	1.1		160	21	173	Mod	168	164	-4	5	Strong	166	168
S8	5.3		0.7	18.1	53	6	41	Slight	51	58	7	-10	Mod	86	101
S9	4.7		0.5	29.3	47	4	54	Slight	64	67	3	-10	Mod	113	134 (Str)
S10	0.4	9.6			79	18	113	Mod	107	107	0	6	Strong	106	107
S11	5.2	2.9			74	6	88	Slight	*	81			Strong	89	86
S12	10.2			3.6	102	0	103	Mod	117	119	2	-14	Strong	99	86
S13	9.6			14.1	96	0	91	Mod	112	113	1	-21	Strong	115	130
S14	8.2			11.5	82	0	83	Slight	*	72			Strong	103	124
S15	7.0			4.3	70	0	72	Mod	74	80	6	-2	Strong	74	83
S16	5.9			7.8	59	0	50	Mod	64	68	4	-14	Strong	64	74
S17	1.8			13.7	18	0	19	Slight	28	29	1	-9	Strong	69	29 (Slight)

Unit for NP measurements is kg CaCO₃/tonne; Abbreviations are: Calcite, Ankerite, Siderite, and Chlorite

* Insufficient sample for analysis; (Str) Strong fizz rating used; (Slight) Slight fizz rating used

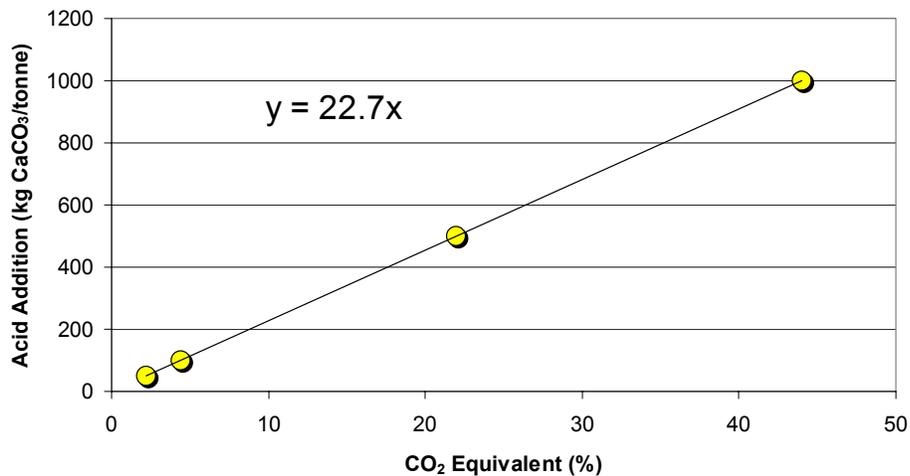


Figure 1. Sobek Acid Additions Versus %CO₂ Equivalent.