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# Static tests response on 5 Canadian hard rock mine tailings with low net acid-generating potentials

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# article info abstract

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It is crucial for mining operators to predict the acid-generating potential of their mine wastes as early as possible in a mine development project, because of the high remediation costs of acid-generating tailings and the risks of environmental issues associated with an incorrect classification of the wastes. However, many tailings having low net acid-generating potentials fall into the uncertainty zone of the static test. Different chemical and mineralogical static test results are compared in this paper for 5 Canadian hard rock mine tailings having low net acid-generating potential, in order to help determine which method is more appropriate for such tailings. Static test methods showed significant result variations (NNP or NP/AP) for each tailings tested, demonstrating the need to develop tools to identify the most appropriate technique for a given mine waste. Thus, static test selection guidelines were developed based on mineralogical considerations for each test. A modification to the Lawrence and Scheske method based on the Paktunc CNP method is proposed in order to improve its accuracy, which enables to account for the presence of oxidizable cations (such as iron and manganese) within the minerals.

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# 1. Introduction

Mine drainage arises as a result of water percolating through various components of a mining complex, such as tailings impoundments and waste rock piles. Acid-mine drainage (AMD) occurs when mine drainage comes into contact with sulfide minerals that have oxidized due to exposure to water and oxygen. The generation of AMD is known to be catalyzed by bacteria under acidic conditions (e.g. [Blowes et al., 2003;](#page-11-0) [Johnson and Hallberg, 2003; Nordstrom, 2000\)](#page-11-0). AMD appears when the neutralization capacities of the carbonate and silicate minerals contained in the mine waste cannot counterbalance the acidity produced by sulfide oxidation. AMD is typically characterized by high acidity, low pH and high concentrations of heavy metals and sulfates (e.g. [Blowes et al., 2003; Nordstrom and Alpers, 1999\)](#page-11-0).

Once mine tailings have been identified as acid generating a remediation strategy must be implemented at the mine in order to prevent sulfide oxidation within the tailings, and to facilitate effluent treatment. Typical costs for the reclamation of an acid-generating mine tailings impoundment in Canada are between 100000 \$ and 250 000 \$ per hectare, while they are generally between 2000 \$ and

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20 000 \$ per hectare for tailings that are not generating contaminated drainage ([Aubertin et al., 2002](#page-11-0)). Because of the high remediation costs of potentially acid-generating tailings, it is crucial for mining operators to accurately predict the acid-generating potential of their mine wastes as early as possible in the mine development project. This allows for the early selection of an appropriate waste management and remediation method. Many prediction techniques are available, generally classified as static or kinetic depending on the time scale of the experimental procedure ([Blowes et al., 2003; Morin and Hutt,](#page-11-0) [1997](#page-11-0)). The static tests generally take less than a week to perform, while the kinetic procedures can last from a few weeks to years. The first tests used to characterize the acid-generating potential of a given mine waste are usually the static tests, which are relatively quick and inexpensive; the present study focuses on these tests.

Many static prediction techniques are available to evaluate the acid-generating potential of mine tailings ([Adam et al., 1997; Day,](#page-11-0) [1991; Duncan and Bruynesteyn, 1979; Lapakko, 1994a; Lawrence](#page-11-0) [and Wang, 1996, 1997; Lawrence et al., 1989; Skousen et al., 2002;](#page-11-0) [Sobek et al., 1978; Weber et al., 2004, 2005\)](#page-11-0). These procedures generally estimate the global acid-generating (AP) and acid-neutralizing (NP) potentials and are often called acid–base accounting (ABA) tests [\(Lawrence and Wang, 1996\)](#page-11-0). Static test procedures can be separated in two distinct classes; chemical methods and mineralogical methods. Chemical methods require experimentation and testing in a chemistry lab, while mineralogical methods are mainly based on the mineralogical composition of the tailings.

The acid-generating potential (AP) and neutralizing potential (NP) are defined separately in the different static test procedures

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considered in this study. The difference between the two parameters is known as the net acid-neutralizing potential ( $NNP = NP-AP$ ); the NP/AP ratio is also used to interpret the results of static test. Tailings with low net acid-generation potential may generate contaminants from sulfide oxidation nonetheless and have a significant environmental impact, with acidic conditions or with near-neutral conditions (contaminated neutral drainage; e.g. [Nicholson, 2004; Plante et al.,](#page-11-0) [2011\)](#page-11-0). Although the extent of their environmental impact may not always be as important as for highly acidic tailings, their management may call for the implementation of control and remediation measures comparable to those of highly acidic tailings.

In recent years, research related to static tests has focused (amongst other issues) on silicate mineral contributions to the NP (e.g. [Jambor et al., 2002, 2007; Li, 2000; Sherlock et al., 1995](#page-11-0)), the effect of iron-bearing carbonates on NP determination and methods to account for the presence of iron within neutralizing minerals [\(Bennett et al., 2000; Frostad et al., 2003; Jambor et al., 2003;](#page-11-0) [Lapakko, 1994b; Malmström et al., 2000; Paktunc, 1999a; 1999b;](#page-11-0) [Weber et al., 2004\)](#page-11-0). Additionally there has been considerable interest in static test result extrapolation from the lab to the field ([Bethum et](#page-11-0) [al., 1997; Feasby et al., 2001; Frostad et al., 2000; Lapakko, 1994b;](#page-11-0) [Liao et al., 2007; Miller et al., 2003\)](#page-11-0). Several studies have compared various static NP determination methods ([Kwong and Ferguson,](#page-11-0) [1997; Lawrence and Wang, 1996; Paktunc, 1999b\)](#page-11-0). These comparisons highlight some important considerations which need to be addressed in the choice of an appropriate NP determination method. These considerations include sample mineralogy, such as silicate vs carbonate neutralization, iron content (or other oxidizable/hydrolysable elements) of neutralising minerals, previous oxidation of the sulfides, and the sulfide content of the sample.

This paper compares the results of a number of different chemical and mineralogical NP determination procedures for 5 Canadian hard rock mine tailings with low net acid-generating potential. The limitations of static test procedures for these particular tailings located near or within the uncertainty zone are highlighted. These comparisons will help determine which method would be more appropriate for a given low net acid-generating potential tailings. Given the the economic and environmental impact of a misclassification of the acid-generating potential, this research aims to develop practical selection tools for mine operators.

# 2. Materials and methods

Several characterization techniques were used to assess the static test responses on the different tailings studied. These tailings samples were chosen for their low net acid-generating potentials, close to or within the uncertainty zone. The following presents a brief description of the methods used to characterize the samples and the main characterization results.

# 2.1. Static tests

This section describes the different chemical and mineralogical procedures considered in this research to evaluate the NP and AP. Tables 1 and 2 compare the chemical and mineralogical NP and AP determination procedures, including details about their calculation and main characteristics.

#### 2.1.1. Chemical NP procedures

Some of the NP procedures are defined as chemical because they involve some level of chemical reaction. The method proposed by [Sobek et al. \(1978\)](#page-11-0) (original Sobek test) was the most popular NP determination approach for many years. In this test, excess acid is added according to different fizz ratings representing different neutralizing potentials. The aqueous suspension of the sample is allowed to react with neutralizing minerals while heating for 1–2 h.

#### Table 1

The different Sobek procedures examined in this study.



The residual acid is then back-titrated to a defined end-point of pH 7. Different modifications of the original Sobek test have been proposed in order to try to better reflect the real conditions in which AMD occurs (see Table 1 for a comparison of the modified procedures to the original test). The modified Sobek I method (proposed by [Lawrence et al., 1989](#page-11-0)) eliminates boiling from the procedure and works at room temperature for longer times (24 h instead of 1–2 h in the original Sobek) and suggests a titration end-point pH of 8.3. [Lawrence and Wang \(1996, 1997\)](#page-11-0) (Modified Sobek II, in Table 1) suggested a different set of hydrochloric acid volumes and normality prescribed by the fizz ratings, and proposed modifications in the manner by which the acid is added. The different Sobek test versions use Eq. [\(1\)](#page-2-0) for NP calculation. The AP calculation according to the Modified Sobek I and II methods is carried out using the  $S_{\text{sulfide}}$  content (Eq. [\(2\)](#page-2-0), in [Table 2\)](#page-2-0), while the original Sobek test uses the  $S<sub>total</sub>$  content in the calculation. [Lawrence and Wang \(1996\)](#page-11-0) provided evidence that the original Sobek procedure is generally more aggressive than the modified Sobek II procedure; consequently, the original Sobek results are generally higher than the modified Sobek II results. The authors explain that the original Sobek procedure generally overestimates the NP because of the high temperatures involved (near 100 °C, Table 1) even if reaction times are lower (1–2 h in the original Sobek vs 24 h in the modified II Sobek). The Sobek II modified procedure ([Lawrence and Wang, 1996](#page-11-0)) was selected for the analysis of the tailings samples in this study for these reasons.

The standard carbonate NP method (CNP; e.g. [Frostad et al., 2003](#page-11-0)) consists of measuring the total inorganic carbon and converting the value into equivalent  $CaCO<sub>3</sub>$  (see Eqs. [\(3\) and \(4\)](#page-2-0) in [Table 2](#page-2-0)). [Lawrence and Wang \(1996, 1997\)](#page-11-0) showed that the modified Sobek II procedure usually gives slightly higher NP results than the CNP because of the silicates contribution to the NP in the modified Sobek II procedure, which is not accounted for in the CNP method. The validity of this method is limited also by the contribution of carbonates other than calcite, such as dolomite, magnesite, siderite and ankerite. Since siderite (FeCO<sub>3</sub>) is considered as a non neutralizing carbonate mineral [\(Coastec Research Inc., 1991; Skousen et al., 1997](#page-11-0)), it may become necessary to take its presence into account when calculating the CNP. The corrected CNP (CCNP) is determined using the standard CNP along with the siderite content ( $\%$ FeCO<sub>3</sub>, in wt. $\%$ , evaluated by XRD in the present study) according to Eqs. [\(5\) and \(6\)](#page-2-0) presented in [Table 2.](#page-2-0) The validity of this correction method is limited by the accuracy and precision of the siderite contents measurements. A similar NP correction was reported by [Frostad et al. \(2003\),](#page-11-0) where the Fe molar ratio is considered when calculating the contribution of Ca and Mg carbonates from quantitative XRD mineralogy.

# 2.1.2. Mineralogical NP approaches

The mineralogical approaches of NP evaluation are rarely employed in practice, mainly because of limited quantification capabilities. However, the latest advances in mineralogical quantification, such as Rietveld fitting of XRD data [\(Rietveld, 1993; Taylor and Hinczak,](#page-11-0)

# <span id="page-2-0"></span>Table 2

# NP methods considered in this study.



[2001\)](#page-11-0), or the ModAn program [\(Paktunc, 1998, 2001](#page-11-0)), enable sufficient mineralogical precision for the successful use of mineralogical NP determinations. Two mineralogical approaches are compared in this study: the [Lawrence and Scheske \(1997\)](#page-11-0) and the [Paktunc \(1999b\)](#page-11-0) carbonate NP.

The [Paktunc \(1999b\)](#page-11-0) method calculates the mineralogical AP based on the sulfide minerals content while the [Lawrence and](#page-11-0) [Scheske \(1997\)](#page-11-0) method uses the  $S_{\text{sulfide}}$  content. The [Lawrence and](#page-11-0) [Scheske \(1997\)](#page-11-0) mineralogical method (based on previous work from [Kwong, 1993](#page-11-0) and [Sverdrup, 1990\)](#page-11-0) calculates the NP from the sum of the individual contribution of the neutralizing minerals composing the material, based on their proportions and relative reactivity (see Eq. [\(7\)](#page-2-0) in [Table 2](#page-2-0)).

The Paktunc carbonate NP (or Paktunc CNP) sums the individual contributions of carbonates to the NP based on their concentration and composition. The Paktunc CNP method accounts for the presence of iron (or other oxidizable cations such as manganese) and its subsequent oxidation and hydrolysis reactions, an acid-generating process which ultimately decreases the overall NP of the host mineral (see Eqs. [\(\(8\)](#page-5-0) [and \(9\)](#page-5-0) in [Table 2\)](#page-2-0).

# 2.2. Physical, mineralogical and chemical characterization methods

The tailings volumetric particle size distribution was determined by laser diffraction (e.g. [Merkus, 2009; Xu, 2000\)](#page-11-0) for sizes between 0.05 and 879 μm using a Malvern Instruments Mastersizer S analyzer. The tailings specific gravity  $(G_s)$  was determined using a Micromeritics AccuPyc 1330 pycnometer [\(Allen, 1990](#page-11-0)), while the specific surface  $(S_s)$ was determined with a Micromeritics Gemini surface analyzer using the nitrogen BET adsorption isotherm ([Brunauer et al., 1938](#page-11-0)).

The tailings chemical composition was determined by ICP-AES following an acid digestion using concentrated nitric  $(HNO<sub>3</sub>)$ , hydrofluoric (HF) and hydrochloric (HCl) acids, as well as liquid bromide ( $Br<sub>2</sub>$ ). Sulfate (SO $<sup>2</sup><sub>4</sub>$ ) content was determined using a 40% HCl extrac-</sup> tion followed by ICP-AES determination of the extracted sulfur (modified from [Sobek et al., 1978](#page-11-0)). Silicon content was determined by a sodium peroxide/sodium hydroxide (Na<sub>2</sub>O<sub>2</sub>/NaOH) fusion using a Claisse Peroxide Fluxer followed by dissolution in diluted HCl and ICP-AES measurement of silicon. The inorganic carbon content  $(C_{\text{inorg}})$  was determined using a LECO furnace with a  $\pm$  0.05 to 0.1 wt.% precision.

The tailings mineralogy was determined with a Bruker A.X.S. Advance D8 XRD using a cobalt X-ray source. Mineral quantification was performed using the Rietveld quantification method [\(Rietveld,](#page-11-0) [1993; Taylor and Hinczak, 2001\)](#page-11-0) with a 0.5 to 1% absolute error on tailings samples pulverized to approximately  $90\% < 10 \mu m$  with a McCrone Micronizing mill. A Savitzky–Golay smoothing filter [\(Savitzky and Golay, 1964\)](#page-11-0) was applied on the raw XRD data in order to accentuate the signal-to-noise ratio in XRD data. Acquisition runs were performed at 0.005°/s from 5 to 70° (2Θ) for regular scanning, and at 0.001°/s for higher resolution runs. Only well crystallized phases are detected with this technique because only crystalline phases effectively diffract X-rays. Therefore, the mineralogical quantification from XRD data does not take into consideration the amorphous phases that may be present within the tailings. Many secondary minerals formed in acid-mine drainage conditions are amorphous and therefore not detected by XRD (e.g. [Cravotta, 1994; Hakkou et al., 2008\)](#page-11-0).

Estimation of the calcium, iron, magnesium, and manganese content of ankerite minerals was done on polished sections of the tailings samples using a Hitachi S-3500N Scanning Electron Microscope (SEM) coupled with an Oxford Instruments EDS (Energy Dispersive X-ray Spectroscopy) probe. The analyses were performed using 20 kV, 120–130 μA, a 25 Pa vacuum, a working distance of 15 mm and a detector dead time of approximately 25–40%. The ankerite composition was determined by analyzing between 6 and 10 ankerite particles from each tailings site by EDS (since MAT-M1 and MAT-M2 were generated from the same tailings material, their ankerite composition are assumed to be the same). The proportions of calcium, magnesium, manganese and iron were deducted from these analyses, with the oxygen dosed stoichiometrically. The ankerite compositions were obtained with approximately 10% accuracy on the stoichiometric coefficient using this method.

# 2.3. Materials

## 2.3.1. Tailings sample preparation

All five tailings come from the Abitibi-Témiscamingue region of Québec, Canada. Sample UQ-8 was sampled in a tailings impoundment and weathered in a laboratory kinetic test ([Villeneuve, 2004](#page-12-0)) prior to the present study, while samples GRE-M1, LAR-M3, MAT-M1 and MAT-M2 were processed via desulfurization (froth flotation using xanthates and MIBC frother; see [Benzaazoua et al., 2000](#page-11-0) for more details) in order to obtain acid-generation properties close to or within the uncertainty zone of static tests interpretation.

#### 2.3.2. Physical properties

The  $D_{10}$ ,  $D_{50}$  and  $D_{90}$  values taken from the grain size distributions are presented in Table 3 along with specific gravity  $(G_s)$  and specific surface areas  $(S_s)$ . The grain size distributions of the studied tailings are typical of fine tailings from hard rock mines ([Bussière, 2007;](#page-11-0) [Vick, 1983](#page-11-0)), with  $D_{10}$  values ranging approximately from 1 to 4  $\mu$ m and 80 to 95% passing 80  $\mu$ m. G<sub>s</sub> values are between 2.7 and 3.1 for all tailings studied.  $S_s$  values are similar for tailings LAR-M3, MAT-M1 and MAT-M2, from 1.5 to 1.9  $m^2/g$ , while GRE-M1 and UQ-8 tailings have significantly higher  $S_s$  values with 3.24 and 5.89 m<sup>2</sup>/g respectively. The higher  $S_s$  value of UQ-8 is related to a significant proportion of secondary oxides generated by sulfide oxidation prior to sampling.

#### 2.3.3. Chemical and mineralogical properties

Table 3 presents the chemical composition of the tailings studied. All five tailings studied have relatively low sulfide contents, with the highest value at 5.72 wt.% (for UQ-8). The low sulfate contents are consistent with fresh tailings (less than  $0.57$  wt.%  $S_{\text{sulfate}}$ ), except for sample UQ-8 which was partly oxidized (1.37 wt.% Ssulfate). The inorganic carbon  $(C_{\text{inorg}})$  content of the tailings reflect the levels of carbonate minerals in the different tailings and ranges from 0.21 to 3.06 wt.%.

[Table 4](#page-4-0) presents the mineralogical composition of the tailings samples. Sample UQ-8 is composed of around 50 wt.% quartz and albite, with approximately equal amounts of pyrite, dolomite, ankerite

Table 3

Chemical composition and physical properties of the tailings studied (element concentrations in wt.%).

Element (wt.%)	UQ-8	GRE-M1	LAR-M3	MAT-M1	MAT-M2
Al	3.81	7.51	5.87	3.16	2.99
$C_{\text{inorg}}$	2.79	1.39	0.21	3.06	2.75
Ca	3.43	2.89	1.68	3.79	3.60
Cu	0.012	0.030	0.023	0.026	0.028
Fe	17.1	11.5	2.83	16.6	17.9
Mg	1.65	2.87	0.73	3.15	2.94
Mn	0.456	0.196	0.043	0.434	0.409
Na	1.80	1.36	1.37	1.01	1.01
Ni	0.005	0.017	0.008	0.007	0.007
Pb	0.008	0.034	0.046	0.028	0.028
$S_{total}$	7.09	1.09	0.816	1.90	2.85
$S_{\text{sulfate}}$	1.37	0.202	0.15	0.57	0.234
$S_{\text{sulfide}}$	5.72	0.89	0.666	1.33	2.62
Si	n/a	53.09	68.81	53.29	46.82
Zn	0.007	0.159	0.108	0.234	0.276
$Ca + Mg + Mn$	5.54	5.96	2.45	7.37	6.95
$G_{S}$	3.04	2.90	2.78	3.03	3.07
$S_s$ (m <sup>2</sup> /g)	5.89	3.24	1.54	1.90	1.49
% under 80 µm (%)	94.6	94.0	89.2	90.3	83.6
$D_{10}$ ( $\mu$ m)	1.08	3.92	3.46	2.04	2.35
$D_{50}$ ( $\mu$ m)	11.88	20.38	22.43	15.68	22.78
$D_{90}$ ( $\mu$ m)	54.8	63.84	83.27	78.45	104.5

<span id="page-4-0"></span>Table 4 Mineralogical composition of the tailings studied.

Mineral (wt.%)	$UO-8$	GRE-M1	LAR-M3	MAT-M1	MAT-M2
Ouartz	23.2	37.4	55.0	43.6	43.0
Albite	26.2	13.1	17.9	5.8	5.1
Chlorite	6.1	18.0	4.8	13.9	10.8
Muscovite	5.3	15.8	11.3		
Paragonite		6.9	6.6		
Phlogopite mica				4.1	4.2
Hornblende	3.8				
Calcite	2.9	2.8	1.0	4.0	3.6
Ankerite	6.1	4.6	1.8	8.9	8.3
Dolomite	7.7				
Pyrite	7.5	1.5	1.6	2.6	5.1
Magnetite				5.9	8.0
Siderite	7.5			11.2	11.8
Gypsum	3.7				
Total	100.0	100.1	100.0	100.0	99.9

and siderite (between 6.1 and 7.7 wt.%), and 2.9 wt.% calcite. The presence of gypsum (3.7 wt.%) results from the previous oxidation of this material in the field. Sample GRE-M1 is mainly composed of quartz, chlorite, muscovite and albite (for more than 80 wt.%), with minor amounts of ankerite (4.6 wt.%) and calcite (2.8 wt.%) and trace amounts of pyrite (1.5 wt.%). Approximately 55 wt.% of the LAR-M3 sample is composed of quartz, with albite and muscovite accounting for nearly 30 wt.% together; trace amounts of ankerite (1.8 wt.%), calcite (1.0 wt.%) and pyrite (1.6 wt.%) were also detected. Since samples MAT-M1 and MAT-M2 were obtained by desulfurization of the same material at two different levels, their global mineralogy are similar; the pyrite content of MAT-M1 (2.6 wt.%) is about half the pyrite content of MAT-M2 (5.1 wt.%). The balance of these samples is mostly composed of quartz (more than 43 wt.%) and chlorite (10 to 14 wt.%), with minor amounts of ankerite (between 8 and 9 wt.%) and calcite (between 3.6 and 4 wt.%).

The ankerite compositions in Table 5 show significant variations between mine sites, with stoichiometric coefficients varying from 0.25 to 0.37 for Mg and from 0.57 to 0.68 for iron, while Mn composition show little difference, varying from 0.06 to 0.08 in the samples studied. No ankerite composition analysis was performed on the LAR-M3 sample because of its low concentration (1.8 wt.%). As a result using a hypothetical ankerite composition for the LAR-M3 sample will not significantly affect the results.

## 3. Results and interpretation

#### 3.1. Static test results

The chemical and mineralogical static test results are presented in [Tables 6 and 7.](#page-5-0) [Table 6](#page-5-0) presents the mineralogical NP calculations



Ankerite composition in the tailings samples.

from the Lawrence–Scheske and Lawrence–Scheske–Paktunc methods. [Table 7](#page-5-0) shows the static test results from all methods considered in this study.

The mineralogical results in [Table 6](#page-5-0) suggest that 72 to 95% of the NP is provided by carbonate minerals for the samples studied, depending on the method considered. Therefore, 5 to 28% of the NP is provided by silicate minerals. Sample UQ-8 has the highest NP contribution from carbonates (94–95%) while sample LAR-M3 has the lowest (72–76%). The main carbonate NP sources are ankerite (12.2– 86.6 kg CaCO<sub>3</sub>/t) and calcite (10.0–40.0 kg CaCO<sub>3</sub>/t) for all tailings, in addition to dolomite (83.6 kg  $CaCO<sub>3</sub>/t$ ) for the UQ-8 sample. The main silicate NP source is chlorite  $(3.2-12.1 \text{ kg } CaCO<sub>3</sub>/t)$  for all studied samples.

Different criteria were proposed to evaluate the acid-generating potential based on NNP values. [SRK \(1989\)](#page-11-0) and [Miller et al. \(1991\)](#page-11-0) suggested the following ABA interpretations: acid generation is uncertain for NNP values between  $-20$  and 20 kg CaCO<sub>3</sub>/t, acid generation is likely for NNP values below  $-20$  kg CaCO<sub>3</sub>/t, and acid generation is unlikely for NNP values above 20 kg  $CaCO<sub>3</sub>/t$ . Another useful tool to evaluate the AMD production potential using static tests is the NP to AP ratio ([Price, 2005](#page-11-0)). Typically, the material may be considered non acidgenerating if  $NP/AP > 2$ , uncertain if  $2 > NP/AP > 1$  and acid generating if NP/AP<1 ([Price, 2009\)](#page-11-0).

The NP results in [Table 7](#page-5-0) vary significantly for a given sample depending on the method employed. On the other hand, AP results are similar for both the chemical and the Paktunc AP methods for all tailings studied, except for UQ-8 where the difference is more significant.

## 3.2. Static test comparisons

The different NP results are compared with each other in [Fig. 1.](#page-6-0) The 1:1 line is shown for each comparison plot.

# 3.2.1. CCNP vs modified Sobek II NP

The CCNP results are higher than the modified Sobek II results for all tailings studied ([Fig. 1a](#page-6-0)), except for LAR-M3 for which the results are very similar. Discrepancies between these methods may arise as a result of incorrect siderite quantification for CCNP calculation and the oxidation/hydrolysis of iron released during the modified Sobek II procedure. In addition, it can be seen that the results from the oxidized UQ-8 sample show the greatest deviation from the 1:1 relationship, probably because dissolution of acidic salts (hydroxides and sulfates) during the test diminishes the modified Sobek II NP results [\(Weber et al., 2004](#page-12-0)), and because dolomite in UQ-8 sample may not dissolve completely [\(Kwong and Ferguson, 1997](#page-11-0)). Therefore, the dolomite contribution to the overall UQ-8 NP is not fully taken into account in the modified Sobek II method.



# <span id="page-5-0"></span>Table 6

individual contributions to the NP for mineralogical methods taking all minerals into account.



#### 3.2.2. Paktunc CNP vs modified Sobek II NP

The modified Sobek II NP results are close to the Paktunc CNP results that take into account the presence of oxidizable cations for all tailings, except for UQ-8 ([Fig. 1](#page-6-0)b) where the modified Sobek II NP is significantly lower than the Paktunc CNP. This difference is again mainly due to (1) the presence of soluble acidic salts [\(Weber](#page-12-0) [et al., 2004](#page-12-0)) and (2) the possible incomplete dissolution of dolomite during the modified Sobek II procedure. The modified Sobek II NP results are slightly higher than the Paktunc CNP for the GRE-M1, MAT-M1 and MAT-M2 tailings, probably because the silicates contribution to the NP is only accounted for in the modified Sobek II method.

# 3.2.3. CCNP vs Paktunc CNP

The Paktunc CNP results are lower than the CCNP results for all tailings except LAR-M3, for which the results are similar [\(Fig. 1c](#page-6-0)). These results are explained by the ankerite iron deduction from the

Table 7 static test results from different methods on the 5 studied tailings samples.

Parameter	<b>UQ-8</b>	GRE-M1	$IAR-M3$	MAT-M1	MAT-M2			
Modified Sobek II NP	64.2	71.7	13.6	93.9	91.0			
Paktunc CNP	143.5	53.6	19.8	88.5	81.2			
Lawrence-Scheske NP	180.8	89.2	35.6	143.1	131.2			
<b>CNP</b>	232.5	115.8	17.5	255.0	229.2			
<b>CCNP</b>	167.7	115.8	17.5	158.2	127.3			
$L-S-P NP$	160.9	76.1	30.9	117.2	107.0			
AP	199.4	27.7	24.5	54.5	81.8			
Paktunc AP	125.1	25.0	26.7	43.4	85.1			
Modified Sobek II NP-chemical AP								
<b>NNP</b>	$-135.2$	44.0	$-10.9$	39.4	9.2			
NP/AP	0.3	2.6	0.6	1.7	1.1			
Paktunc CNP-Paktunc AP								
<b>NNP</b>	18.4	28.5	$-6.9$	45.1	$-3.9$			
NP/AP	0.7	1.9	0.8	1.6	1.0			
Lawrence-Scheske NP-chemical AP								
<b>NNP</b>	$-18.6$	61.5	11.1	88.6	49.4			
NP/AP	0.9	3.2	1.5	2.6	1.6			
CCNP-chemical AP								
<b>NNP</b>	$-31.7$	88.1	$-7.0$	103.7	45.5			
NP/AP	0.8	4.2	0.7	2.9	1.6			
Lawrence-Scheske-Paktunc NP-chemical AP								
<b>NNP</b>	$-38.5$	48.4	6.4	62.7	25.2			
NP/AP	0.8	2.7	1.3	2.2	1.3			

NP in the Paktunc CNP method. The difference between the CCNP and the Paktunc CNP results increases with ankerite content.

# 3.2.4. Paktunc CNP vs Lawrence–Scheske NP

The Lawrence–Scheske NP results are systematically higher than the Paktunc CNP results for the tailings studied ([Fig. 1d](#page-6-0)), mainly because the iron content is not accounted for in the Lawrence– Scheske method, and because the silicates contribution to the NP is taken into account in the Lawrence–Scheske NP.

# 3.2.5. Lawrence–Scheske NP vs modified Sobek II NP

All of the studied samples have a Lawrence and Scheske NP higher than the modified Sobek NP ([Fig. 1e](#page-6-0)). All studied samples contain ankerite, which partially explains the higher NP results from the Lawrence–Scheske method than from the modified Sobek II procedure.

The Lawrence–Scheske method can be modified in order to take the iron content of neutralizing minerals into account, by incorporating the "ci" parameter from the Paktunc CNP method. This modified Lawrence– Scheske–Paktunc method (Eq. (7)) combines the characteristics of both methods, accounting for the silicates contribution to the overall NP and the non-oxidizable cations content of the minerals:

$$
NP = 1000 \ kg/t \cdot M_{CaCO_3} \cdot \sum_{i=1}^{n} \frac{C_{Mi}R_ic_i}{M_{Mi}}
$$
 (7)

In Eq. (7), NP stands for neutralization potential (kg  $CaCO<sub>3</sub>/t$ ), M<sub>CaCO3</sub> is the calcite molar mass (100.09 g/mol),  $M_{Mi}$ ,  $C_{Mi}$  and  $R_i$  are respectively the molar mass (g/mol), the weight content (wt.%) and the reactivity factor (unitless), and the  $c_i$  is the sum of the stoichiometric coefficients of non-oxidizable cations in the carbonate. The results from the Lawrence–Scheske–Paktunc method are shown in [Fig. 1](#page-6-0)e. It appears that the NP results obtained by this method are closer to the modified Sobek II results than for the original Lawrence–Scheske version.

Sample UQ-8 still shows a greater modified Sobek NP than the Lawrence–Scheske NP, despite the correction for the iron content of the carbonate minerals. As mentioned previously, discrepancy can be explained by incomplete dolomite dissolution ([Kwong and](#page-11-0) [Ferguson, 1997\)](#page-11-0) and the presence of acid salts ([Weber et al., 2004](#page-12-0)) that both lower the modified Sobek II NP result. An unweathered UQ-8 sample [\(Villeneuve, 2004; Villeneuve et al., 2003, 2009](#page-12-0)) gave a Sobek II NP result of 180 kg  $CaCO<sub>3</sub>/t$ , which is closer to the NP estimated by the mineralogical methods (143.5 to 180.8 kg CaCO<sub>3</sub>/t). Since the XRD mineralogical quantification of the fresh UQ-8 sample is close to that of the weathered UQ-8 sample (aside from the presence of gypsum), one can assume that the lower modified Sobek NP result

<span id="page-6-0"></span>

Fig. 1. Comparison of NP results from different methods on the studied samples: a) CCNP vs modified Sobek II NP, b) Paktunc CNP vs modified Sobek II NP, c) CCNP vs Paktunc CNP, d) Paktunc CNP vs Lawrence–Scheske NP and e) calculated NP for Lawrence–Scheske NP and Lawrence–Scheske–Paktunc NP vs modified Sobek II.

on the weathered UQ-8 sample is mainly attributed to the presence of soluble acidic oxidation product salts rather than to incomplete dolomite dissolution. The absence of such acidic salts from the XRD data collected from the UQ-8 sample suggests that these salts are amorphous (e.g. [Cravotta, 1994; Hakkou et al., 2008](#page-11-0)) and/or at concentrations under the detection level. Dolomite dissolution during the modified Sobek II procedure is verified in Section 4.1.

# 4. Discussion

# 4.1. Mineralogical evolution during modified Sobek II test

As seen in the previous section, subtle changes in a static test method or interpretation may lead to a significant change in the acid-generating nature prediction statement, particularly for low acid-generating tailings. To obtain an accurate acid-generating potential classification, some hypotheses related to the modified Sobek II procedure must be validated:

- 1. Dolomite dissolves only partially during the modified Sobek II test;
- 2. Siderite does not contribute to the NP determination during the modified Sobek II test;
- 3. Ankerite and calcite are completely dissolved during the modified Sobek II test;
- 4. Neutralizing silicates contribute at least partially to NP in the modified Sobek II test.

The mineralogical composition of the samples after submission to the modified Sobek II test procedure was evaluated by XRD. The comparison of XRD diffraction patterns of the samples before and after submission to a modified Sobek II procedure enable a better



Fig. 2. XRD evidence of carbonates fate during the modified Sobek II procedure (d-spacing between 2.70 and 3.10 Å for a) UQ-8, b)GRE-M1, c)LAR-M3, d) MAT-M1 and e) MAT-M2).

appreciation of the mineral dissolution during the modified Sobek II test, particularly for carbonates. Fig. 2a–e shows XRD data of major carbonate peaks (d-spacing ranging from 2.70 to 3.10 Å) from samples before and after the modified Sobek procedure. The major calcite peak ( $d = 3.035$  Å) disappears after the modified Sobek II procedure, indicating complete calcite dissolution in all samples studied (Fig. 2a–e). The ankerite and dolomite major peaks (respectively 2.89 and 2.88 Å) disappear after the procedure, except for the UQ-8 sample (Fig. 2a). While both ankerite and dolomite are detected by XRD in this sample, the GRE-M1, MAT-M1 and MAT-M2 samples only contain XRD-detectable ankerite (Fig. 2b, d and e respectively). Observation of ankerite and dolomite secondary peaks is necessary to appreciate the dissolution level of these minerals because their peaks are overlapped. The secondary ankerite and dolomite XRD peaks of the UQ-8 tailings are shown in [Fig. 3.](#page-8-0) A significant decrease in the ankerite secondary peaks can be seen at  $d=2.199$  and 1.812 Å on [Fig. 3a](#page-8-0) and b respectively. The decrease in dolomite secondary peaks at  $d = 2.191$  and 1.785 Å is not as significant. The remaining secondary dolomite peaks suggest incomplete dolomite consumption during the modified Sobek procedure.

The XRD data shown in Figs. 2 and 3 were acquired with a single acquisition run at 0.005°/s steps. A higher definition XRD pattern using two acquisition runs at 0.001°/s was undertaken on the UQ-8 sample after the modified Sobek II procedure (see [Fig. 4](#page-8-0)a–c) in order to clarify the interpretations regarding dolomite dissolution. The major dolomite and ankerite peaks are overlapped and still present after the modified Sobek II digestion procedure (2.88 and 2.90 Å respectively, [Fig. 4](#page-8-0)a). The secondary dolomite peaks are detected (2.191 and 1.785 Å, shown respectively on [Fig. 4](#page-8-0)b and c), but the presence of the secondary ankerite peaks is questionable (2.199 Å and 1.812 Å, shown respectively on [Fig. 4](#page-8-0)b and c). Thus, the ankerite is most likely dissolved while dolomite is still present, which means that the peak at  $2.88-2.90 \text{ Å}$  [\(Fig. 4a](#page-8-0)) is attributable mainly to dolomite.

The major siderite peaks shown in Fig. 2 are practically unaltered during the modified Sobek II procedure for samples UQ-8, MAT-M1 and MAT-M2. Thus, it appears that siderite does not seem to dissolve in the conditions of the modified Sobek II procedure. On the other hand, Siderite is believed to be consumed during the original Sobek procedure without enough time for iron to oxidize and hydrolize,

<span id="page-8-0"></span>

Fig. 3. XRD data showing secondary ankerite and dolomite peaks before and after the modified Sobek II procedure on tailings UQ-8 sample (d-spacing for a) 2.15–2.25 Å and b) 1.77–1.83 Å).

therefore overestimating the NP obtained by the original Sobek procedure when siderite is present ([Jambor et al., 2003](#page-11-0)). To this regard, the modified SObek II method is best suited for siderite-containing samples.

The main silicates found in the studied tailings are presented in [Table 4.](#page-4-0) The neutralizing silicates identified by [Jambor et al. \(2002,](#page-11-0) [2007\)](#page-11-0) that are also found in the studied samples are chlorite (approx. 6 kg CaCO<sub>3</sub>/t when pure), mica/phlogopite (approx. 8 kg CaCO<sub>3</sub>/t when pure), and mica/muscovite (approx. 1 kg  $CaCO<sub>3</sub>/t$  when pure). These proposed mineral NP should only be considered as indicators of the most reactive minerals to acid digestion, because they were determined using the original Sobek procedure. The neutralizing silicate minerals within in the studied tailings do not appear to be significantly altered during the modified Sobek procedure, as suggested by XRD data. No muscovite peak decrease is observed for the studied tailings, except for LAR-M3 [\(Fig. 5](#page-9-0)a). A slight chlorite peak alteration is observed for all tailings except MAT-M2 suggesting that chlorite is slightly altered by the procedure, as illustrated by the GRE-M1 sample in [Fig. 5b](#page-9-0). The major phlogopite peak is not significantly different in shape and size before and after the modified Sobek II procedure, as illustrated in [Fig. 5](#page-9-0)c for the MAT-M2 sample. However, it is possible that the silicates alteration in the static test become negligible to the bulk of the particle after it is micronized for XRD analysis. Therefore, it is possible that silicate alteration is greater than what can be deduced from XRD data as interpreted in the present study.

In summary, the following hypotheses were verified by the XRD data obtained in this study:

1. Dolomite only partially dissolves during the modified Sobek II test, while calcite and ankerite seem to be completely dissolved; however, this needs to be verified further on more samples because only one sample contained dolomite.



Fig. 4. XRD data showing the main and secondary ankerite/dolomite peaks with multiple data acquisition runs on tailings UQ-8 after the modified Sobek II procedure (d-spacing for a) 2.70–3.10 Å, b) 2.15–2.25 Å and c) 1.77–1.83 Å).

- 2. XRD data interpretation does not detect significant alteration of siderite during the modified Sobek II procedure, unlike in the original Sobek procedure test, where siderite is dissolved ([Jambor et al.,](#page-11-0) [2003\)](#page-11-0), probably due to the higher digestion temperature;
- 3. Calcite and ankerite are completely dissolved during the Sobek II procedure;
- 4. Slight chlorite and muscovite alteration were caused by the modified Sobek II procedure. Therefore, neutralizing silicates partially contribute to the NP as determined by this method.

# 4.2. Static test selection

Mineralogical investigations are necessary for an appropriate static test selection and in the results interpretation. Methods only taking carbonates into account (such as CNP, Paktunc CNP) should be avoided when silicate minerals provide a significant proportion of a material's NP. The silicates contribution to the NP can be crucial: [Heikkinen and Räisänen \(2008\)](#page-11-0) observed a particular case in which

<span id="page-9-0"></span>

Fig. 5. XRD evidence of neutralizing silicate minerals fate during the modified Sobek II procedure (d-spacing for a) 3.20–3.50 Å, b) 6.70–7.50 Å and c) 9.50–10.50 Å).

the CNP procedure suggests that the tailings are likely acidgenerating, while the [Lawrence and Scheske \(1997\)](#page-11-0) method suggests that they are not. When iron-bearing neutralizing minerals provide a significant proportion of a material's NP, methods not accounting for the released iron oxidation and hydrolysis from neutralizing minerals should also be avoided. Particular attention should be given to the presence of acidic salts resulting from sulfide oxidation, which may decrease the apparent NP in Sobek-like methods. [Weber et al. \(2004\)](#page-12-0) argued that sulfide-rich materials (particularly when containing framboïdal pyrite) and materials containing secondary acidic salts resulting from previous sulfide oxidation decrease the apparent NP of samples subjected to a Sobek-like procedure because of the acidity and iron released by the oxidation products. Paste pH measurement provides a qualitative idea of the presence of acidity within the sample.

Finally, particular care must be observed for tailings with significant dolomite content, as Sobek-like methods appear to incompletely dissolve this mineral. These precautions become even more important for samples close to or within the uncertainty zone of static test interpretation, as is often the case for low net acid-generating materials.

Guidelines for an appropriate NP method selection for low net acid-generating tailings may be drawn from comparisons between NP results from the studied samples and the literature. These guidelines are summarized in the flowchart shown in [Fig. 6.](#page-10-0) The following presents the static test results interpretation with regards to the flowchart in [Fig. 6](#page-10-0).

# 4.2.1. UQ-8

The NNP values for the UQ-8 tailings vary between  $-135$  kg  $CaCO<sub>3</sub>/t$  (modified Sobek II NP–chemical AP) and 18.4 kg  $CaCO<sub>3</sub>/t$ (Paktunc CNP and AP), while the NP/AP ratio varies from 0.3 to 1.7. The UQ-8 tailings may be classified as likely AMD generating or within the uncertainty zone, depending on the methods employed in the static test interpretation.

Since UQ-8 contains a significant amount of dolomite, the Sobek II procedure should be avoided. In addition, methods not accounting for the ankerite and siderite content of the UQ-8 tailings should be avoided. Therefore, the Lawrence–Scheske, CNP and the CCNP (which only take siderite into account) methods should not be used for the UQ-8 tailings. Finally, methods not taking the silicates into account are acceptable for the UQ-8 tailings because the neutralizing silicates are expected to contribute 5–6% of the overall NP (see [Table 6](#page-5-0)). Thus, two approaches are more appropriate to evaluate the net acid-generating potential of UQ-8 using the guidelines:

- 1. The Paktunc CNP–Paktunc AP couple, which classifies the sample in the uncertainty zone with the NNP (18.4 kg  $CaCO<sub>3</sub>/t$ ) but acidgenerating with the NP/AP ratio (0.7).
- 2. The Lawrence–Scheske–Paktunc NP–chemical AP couple classifies the sample as potentially acid-generating with the NNP  $(-38.5 \text{ kg } CaCO<sub>3</sub>/t)$  and the NP/AP ratio (0.8).

Hence, it is expected that the true NNP value would be between these two boundaries.

# 4.2.2. GRE-M1

All methods considered in this study classify the GRE-M1 sample as non acid-generating based on NNP values between 28.5 and 88.1 kg CaCO<sub>3</sub>/t. The NP/AP values correspond to uncertain and to non-acid generating with values between 1.9 and 4.2. The GRE-M1 sample contains ankerite and a contribution of 18–21% from neutralizing silicates (mainly from chlorite; see [Table 6\)](#page-5-0). The CCNP, the Paktunc CNP and the Lawrence–Scheske NP methods could be considered for the GRE-M1 tailings because of the low contributions from silicates compared to other minerals, while the significant contribution of ankerite to the NP favours the use of methods considering the iron content of minerals.

The remaining approaches (Sobek II–chemical AP, Paktunc CNP– Paktunc AP and Lawrence–Scheske–Paktunc–chemical AP methods) classify the GRE-M1 material as non acid-generating with the NNP criteria with values between 28.5 and 48.4 kg  $CaCO<sub>3</sub>/t$ . The NP/AP ratio of the Sobek II–chemical AP and Lawrence–Scheske–Paktunc NP–chemical AP methods (2.6 and 2.7 respectively) consider the GRE-M1 tailings non-acid generating. The Paktunc CNP–Paktunc AP NP/AP ratio (1.9) falls just at the limit of the uncertainty zone but does not account for the silicates contribution to the NP. Thus, this material may be considered as non-acid generating.

#### 4.2.3. LAR-M3

The LAR-M3 tailings are classified in the NNP uncertainty zone for all methods considered with values between  $-10.9$  and 11.1 kg  $CaCO<sub>3</sub>/t$ . However, they fall between the acid-generating and uncertainty zones with the NP/AP ratio (0.6–1.5). The LAR-M3 tailings are mainly composed of silicates with less than 3 wt.% calcite and ankerite which provide 72–76% of the NP [\(Table 6](#page-5-0)), while the silicates are believed to contribute for approximately 24 to 28% of the overall NP [\(Table 6\)](#page-5-0). Therefore, methods not accounting for the presence of

<span id="page-10-0"></span>

Fig. 6. Static test selection flowchart for low-NP, low-AP mine tailings.

iron or silicate contributions should be avoided for these tailings. Thus, the preferred methods for the LAR-M3 tailings are the Sobek II–chemical AP and Lawrence–Scheske–Paktunc–chemical AP methods that give NNP values and NP/AP ratios within the uncertainty zones. Despite the use of the flowchart, the static test assessment of the LAR-M3 tailings is still uncertain.

## 4.2.4. MAT-M1

The MAT-M1 tailings are considered non acid-generating with the NNP values of all methods considered in this study, with values between 39.4 and 103.7 kg  $CaCO<sub>3</sub>/t$ . The NP/AP ratios for this sample range from 1.6 to 2.9. The MAT-M1 tailings contain calcite, ankerite and siderite, as well as neutralizing silicates providing between 12 and 14% of the overall NP [\(Table 6](#page-5-0)). Therefore, methods not considering the iron content of minerals (Lawrence–Scheske NP, CNP, CCNP) must not be used for these tailings, but methods not taking silicates into account (Paktunc CNP, CNP, CCNP) may still be used. This leaves the Sobek II–chemical AP, Paktunc CNP–Paktunc AP and the Lawrence– Scheske–Paktunc NP–chemical AP methods that classify the MAT-M1 tailings as non acid-generating with the NNP criteria (respectively 39.4, 45.1 and 62.7 kg  $CaCO<sub>3</sub>/t$ ). The NP/AP ratios of these methods (respectively 1.7, 1.6 and 2.6) consider the MAT-M1 tailings to lie between the uncertainty and non acid-generating zones.

#### 4.2.5. MAT-M2

According to NNP criteria, the MAT-M2 material is considered either non acid-generating (Lawrence–Scheske NP–chemical AP, CCNP– chemical AP, Lawrence–Scheske–Paktunc NP–chemical AP) or within the uncertainty zone (modified Sobek II–chemical AP, Paktunc CNP– Paktunc AP). The NP/AP ratio considers this tailings sample within the uncertainty zone (1.0–1.6 NP/AP ratios).The MAT-M2 tailings contain the same minerals as MAT-M1 but in different amounts: calcite, ankerite and siderite, with neutralizing silicates providing between 11 and 14% of the overall NP ([Table 6\)](#page-5-0). AS for MAT-M1, methods not accounting for the iron content of minerals (Lawrence–Scheske NP, CCNP) should be avoided, while methods not taking silicates into account (Paktunc CNP, CNP, CCNP) may still be used. The Paktunc

CNP–Paktunc AP and Sobek II–chemical AP methods classify the MAT-M2 tailings as uncertain with the NNP value ( $-3.9$  and 9.2 kg CaCO<sub>3</sub>/t respectively), while the Lawrence–Scheske–Paktunc NP–chemical AP couple classifies the MAT-M2 tailings just above the uncertainty zone with the NNP value (25.2 kg  $CaCO<sub>3</sub>/t$ ).

In summary, it has been demonstrated that proper method selection and interpretation based on mineralogical data can reduce the range of static test results and refine their interpretation for tailings having low net acid-generating potentials. However, one method alone rarely is perfectly suited for a material as methods rarely account for all characteristics of a given mineralogy.

#### 5. Conclusion

Static tests are an important tool in the optimization of mine waste management at the mine site. A misclassification of the AMD generation potential for a given waste can have important economical and environmental impacts. In this study, different static tests were used to assess the NP of mine wastes and were compared for 5 Canadian hard rock mine tailings with low net acid-generation potentials. Significant differences in NP results were obtained for the tailings studied depending on the method used, demonstrating the need to develop tools for appropriate method selection. Results suggested that dolomite does not completely dissolve during a modified Sobek II test, while ankerite and calcite do dissolve. It was also demonstrated that siderite is not dissolved by the modified Sobek II test and that the main neutralizing silicates appear to be only slightly altered by the test. A modification to the Lawrence and Scheske method (called the Lawrence–Scheske–Paktunc method), which takes into account the presence of oxidizable cations such as iron and manganese in the minerals, was also suggested to improve the precision of this method.

Guidelines were issued in order to improve static tests selection for low net acid-generating tailings. The tool is based on the mineralogy of the tailings and on the characteristics of each static test. However, it is recommended to perform kinetic tests (ex. humidity cells or column tests; see [Morin and Hutt, 1997,](#page-11-0) and [Demers et al., 2010](#page-11-0), for more

<span id="page-11-0"></span>details) to better define the AMD generating potential for results located within or close to the uncertainty zone.

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