



GEOCHEMICAL ASSESSMENT OF VANGORDA/GRUM WATER TREATMENTSLUDGE

Prepared for:

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1 Introduction

Metal contaminated water collected at the Vangorda Open Pit is currently being treated using the conventional lime water treatment process that was installed and used to treat water pumped from the Grum Pit, the Vangorda Pit and the Little Creek Dam during active mining (pre 1998). Water treatment solids, or sludge, are produced which contain the metals removed from solution during the water treatment process. These solids are accumulated in a settling or clarification pond, located adjacent to the water treatment plant, and excess water is decanted.

Since the clarification pond has a limited storage capacity, the solids have to be removed periodically and disposed elsewhere. Currently, the water license requires that the sludge be disposed on land within the Vangorda waste rock dump. However, other options are available for sludge disposal, including sub-aqueously in the pit lake or separately in a purpose-built facility. Past practice has been to deposit the sludge in the Faro Main Pit (pre 1998) or into the Vangorda Open Pit (post 1998).

A sludge management plan is required for an interim period while the Final Closure and Reclamation Plan (FCRP) is developed and implemented. Selection of the disposal option should be considerate of the geochemical properties of the sludge, the long-term chemical stability, potential physico-chemical controls brought about by the disposal strategy, as well as long-term closure requirements.

For example, co-disposal with waste rock would not be considered if the waste rock is acid generating. Free acid released from the waste rock would react with the sludge and result in remobilization of metals. Similarly, disposal in the pit lake would not be feasible if reducing conditions at depth would affect metal solubility. Or, if there is a risk that the pit lake may become acidic, in pit disposal would not be considered.

Therefore, a laboratory program has been initiated to assess some of the geochemical and physical properties of the water treatment sludge. Section 2 summarises the investigation that has been undertaken, and the test results are summarised in Section 3. Section 4 provides a summary of the conclusions and recommendations.

2 Sludge Characterization Program

2.1 Sampling Program

A series of six sludge samples from the sludge pond at the water treatment facility were obtained by site personnel and shipped directly to Canadian Environmental and Metallurgical Inc. (CEMI) for testing. The samples, each weighing about 2 kg, were sealed in large plastic bag, with little or no evidence of pore water. The samples were designated as follows:

1. Near Upper
2. Near Lower
3. Mid Upper
4. Mid Lower
5. Far Upper
6. Far Lower

The samples were taken from three locations along the line of discharge, approximately diagonally across the storage impoundment from the discharge point. The 'near' sample location was closest to the discharge point, and the 'far' sample furthest from the discharge point. The 'lower' samples were taken at depth and the 'upper' samples are from near surface.

2.2 Test Program

The testing methods adopted and brief descriptions of these methods are provided in the following sections.

2.2.1 Moisture Content

Moisture contents were determined for each sample by accurately determining the weight of a sub-sample of the 'as received' sample, drying it at 105 °C for 24 hours, and accurately determining the dry weight of the sample.

2.2.2 Chemical Analysis

Sub-samples of the as-received samples were dried and submitted for trace and major element analysis by Inductively Coupled Plasma (ICP) and X-Ray Fluorescence (XRF) methods.

2.2.3 Mineralogy

A sub-sample from each sample was submitted for mineralogical examination. Method descriptions are provided in Appendix A. The purpose was to assess how much residual lime remains in the samples, to determine if any secondary calcite has formed and to identify the most abundant mineral phases that may determine long-term water quality effects.

2.2.4 Settling Test

A column settling test was undertaken to assess the possible density that may be reached through natural consolidation of the sludge in a lake environment.

2.2.5 BC SWEP Test

A standard BC SWEP test was completed on a composite sample, prepared from all 6 samples on an equal weight basis, to assess metal solubility for slightly acidic conditions.

2.2.6 Three-Stage Leach Extraction Test

A three-stage distilled water leach extraction test was undertaken to assess metal leachability and cumulative solute release from the samples. These test were undertaken at a solid (as received wt.) to liquid ratio of 1:3, i.e. 150 grams of solids were mixed with 450 ml of water. The water was extracted and analyzed, and the leach extraction of the solids was repeated twice with fresh water.

2.2.7 Saturated Column Test

An anoxic saturated column test was undertaken to assess the potential effects on solute release that might occur should the sludge be disposed of in the pit lake. The test comprised placing approximately 1 kg of sample in a sealed column, filling the column with distilled water and then recycling the porewater continuously until equilibrated conditions are achieved. To ensure anoxic conditions, the column test and recycle were maintained in a nitrogen environment.

2.2.8 Porewater Extraction

Finally, porewater extractions were to have been completed on each sample; however, the samples contained no 'free' porewater and porewater extractions could therefore not be completed.

3 Results

The test results are presented and briefly discussed in the sections below.

3.1 Moisture Content

Even though the samples did not yield any porewater, as shown in the Table 3.1, the moisture content was above 80 %, i.e. less than 20 % solids. The moisture content varied little with depth. It is also noteworthy that the samples at depth consistently had lower moisture contents than the near surface samples.

Table 3.1 Summary of Sludge Moisture Contents

Sample	Sample Weight (‘As Received’) (g)	Dry Weight (g)	Moisture Content (% H ₂ O)	Solids Content (%)	Specific Gravity (Calc.)
Near Lower	200.0	32.5	83.8	16.3	1.17
Near Upper	200.0	27.9	86.1	14.0	1.14
Mid Lower	200.0	38.9	80.6	19.5	1.21
Mid Upper	200.0	32.5	83.8	16.3	1.17
Far Lower	200.0	30.9	84.6	15.5	1.16
Far Upper	200.0	28.7	85.7	14.4	1.15

3.2 Major and Trace Element Analyses

Complete results are provided in Appendix B. The analytical results for the samples are summarised in Table 3.2. As shown by the calculated standard deviations presented in the table, when compared to the averages, the analytical results varied little among the samples, with the exception of copper, cobalt and nickel. These results suggest that the sludge deposit is comparatively homogeneous and does not vary significantly spatially. The sludge is characterized by elevated zinc and manganese, and contains significant concentrations of nickel and cobalt.

The results also indicate that only sulphate sulphur is present in the sludge. Assuming that the sulphate is present predominantly as gypsum, the results suggest that the balance of the calcium is present as a carbonate phase, or insoluble CaO, or un-reacted or Ca(OH)₂. However, based on the sludge pH, it is likely to be a carbonate phase or insoluble CaO, as un-reacted Ca(OH)₂ would have resulted in a very high sludge pH. It is possible that some or most of the magnesium could also be present as a (Ca,Mg)CO₃ mineral phase, which would account for the inorganic carbon content of the sample. These results further suggest that the zinc is present predominantly as a hydroxide.

Table 3.2 Summary of Analytical Results

Parameter	Units	Average	Standard Deviation
Ag	ppm	7.8	0.5
Al	%	0.04	n/a
As	ppm	7.0	4.5
Ba	ppm	52	12
Ca	%	6.2	1.1
Cd	%	0.033	0.003
Co	ppm	977	78
Cr	ppm	26	1.6
Cu	ppm	130	51
Fe	%	0.58	0.083
Mg	%	5.3	0.48
Mn	%	6.8	0.50
Ni	ppm	1,061	83
Pb	ppm	61	7.3
Zn	%	25	2.2
S(T)	%	1.2	0.14
S(SO ₄)	%	1.2	0.16
TIC	%C	3.8	0.35

3.3 Mineralogical Assessment

Analysis of the samples by scanning electron microscope (SEM) showed strong similarities in all samples. The primary observations indicated that the dominant phase consists predominantly of Zn with lesser Mn and minor Si and S. The second most abundant phases contained predominantly calcium. These could not be verified as calcium hydroxide or calcite. These grains were also extremely small and it was difficult to obtain spectra representative of just one phase. One sample contained trace amounts of a clay or mica.

Rietveld X-ray diffraction (XRD) analysis was carried on two different samples, which proved to be virtually identical. The only crystalline phases that could be identified were calcite and quartz. However, from the SEM analysis, these are not the dominant phases. A variety of hydroxides were attempted as fits to the patterns, but none correlated well. The XRD also indicated the presence of abundant amorphous material.

3.4 Settling Test

The results of the settling test illustrated in Figure 3-1. As shown, the sludge was initially suspended to a slurry with a solids density of about 8 % (by wt.). Over a period of 8 days, the slurry settled to about 13.4 % (by wt.) solids, approaching the solids content of 14 % (by wt.) for the 'wettest' as received sample (see Table 3.1).

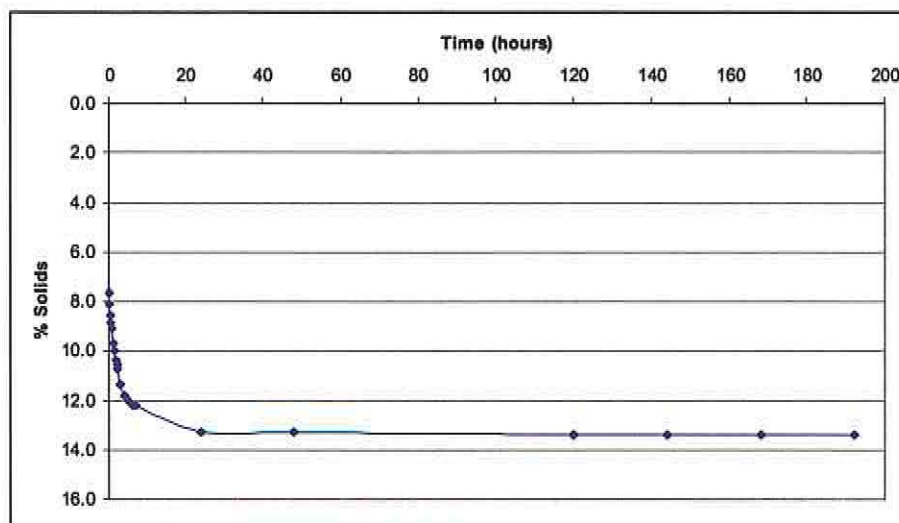


Figure 3-1 Densification Profile of the Suspended Sludge

3.5 BC SWEP Test

The results for the BC SWEP test completed on a representative composite sample of the water treatment sludge are summarised in Table 3.3. Even though the maximum allowable volume of acetic acid was added to the sample during the procedure, the leachate pH was buffered to about 8.2 after 24 hours. As shown in Table 3.3, none of the prescribed SWEP test limits were exceeded in the leachate.

Table 3.3 Summary of BC SWEP Test Results

Dissolved Metals		Result	SWEP Limit
As	mg/L	<2	5
Ba	mg/L	0.1	100
B	mg/L	<1	500
Cd	mg/L	<0.1	0.5
Cr	mg/L	<0.1	5.0
Cu	mg/L	<0.1	100
Hg	mg/L	<0.00005	0.1
Ag	mg/L	<0.1	5.0
Zn	mg/L	0.56	500

3.6 Three Stage Sequential Leach Extraction

Complete analytical results for the three stage sequential leach extraction tests are provided in Appendix C. The leach extraction tests were performed on 150 gram (wet weight) samples using 450 mL of distilled water. Only a limited number of soluble species showed appreciable concentrations in the leachate. These concentrations are summarised in Table 3.4. Because of the similarity among the six leach extraction tests, Table 3.4 summarises the average concentrations detected for each stage of the extraction procedure. Ion

balances calculated for the leachate analyses show a better than 5 % result, verifying the results and major ion composition.

Table 3.4 Summary of Sequential Leach Extraction Leachate Concentrations

Parameter	Units	Concentration		
		Stage 1	Stage 2	Stage 3
pH		8.87	8.71	8.26
Conductivity	uS/cm	1159	714	566
Alkalinity	mgCaCO ₃ eq/L	59	76	89
Sulphate	mg/L	851	486	293
Barium	mg/L	0.01	0.01	0.01
Calcium	mg/L	15.0	8.6	5.8
Magnesium	mg/L	235	143	94
Manganese	mg/L	0.041	0.011	0.011
Silicon	mg/L	0.19	0.14	0.14
Sodium	mg/L	2.2	2.0	2.0
Strontium	mg/L	0.082	0.049	0.035
Zinc	mg/L	0.16	0.07	0.07

The cumulative mass released to leachate of each of the solutes detected was calculated and are summarise Table 3.5. The table also provides the initial solids content of each parameter for comparison.

Table 3.5 Summary of Cumulative Solute Release

Parameter	Units	Initial Solids Content	Cumulative Release (Average)		
			Stage 1	Stage 2	Stage 3
Barium	mg/kg	52	0.2	0.4	0.6
Calcium	mg/kg	61,583	280	441	550
Magnesium	mg/kg	53,100	4413	7084	8849
Manganese	mg/kg	67,850	0.8	1.0	1.2
Strontium	mg/kg	170	1.5	2.4	3.1
Zinc	mg/kg	246,500	3.0	4.4	5.7
Sulphate	mg/kg	36,000	15,963	25,075	30,575

The fractions of the initial solids content leached, expressed as a percentage, are shown in Table 3.6. The results indicate that most of the sulphate is dissolved as MgSO₄ suggesting in fact that very little sulphate is present as gypsum. However, not all of the magnesium is leached which suggests that most of it is bound in other mineral forms. It is possible that the balance of the magnesium is bound as in a carbonate form. The low fraction of calcium leached confirms that most of the calcium is present as calcite (CaCO₃).

Table 3.6 Summary of Fractions Leached

Element	Cumulative Fraction Extracted (%)		
	Stage 1	Stage 2	Stage 3
Barium	0.36	0.73	1.15
Calcium	0.46	0.72	0.89
Magnesium	8.3	13.3	16.7
Manganese	0.001	0.001	0.002
Strontium	0.9	1.4	1.8
Zinc	0.001	0.002	0.002
Sulphate	44.3	69.7	84.9

The leach extractions tests further indicate that, for relatively short contact times and oxidizing conditions, both zinc and manganese remain insoluble.

3.7 Saturated Anoxic Column Test

The porewater of the saturated column test was recycled for a period of 37 days, after which the porewater was extracted and the dissolved parameters analysed. The leachate water quality results are provided in Appendix D, and are summarised in Table 3.7. The table also provides a direct comparison with the first stage results for the sequential leach extraction tests.

Table 3.7 Summary of Anoxic Column Test Results

Parameter	Units	Anoxic Column	Stage 1
Solid to Liquid		~1:5	~1:19
pH		8.15	8.9
Redox.	mV	321	-
Conductivity	µS/cm	1508	1159
Alkalinity	mg CaCO ₃ /L	77.0	58.7
Sulphate	mg/L	1550	851
Ba	mg/L	0.02	0.01
Ca	mg/L	34.8	15
Co	mg/L	0.02	< 0.01
Cu	mg/L	< 0.01	< 0.01
Fe	mg/L	< 0.03	< 0.03
Mg	mg/L	433	235
Mn	mg/L	0.149	0.041
K	mg/L	3	< 2
Si	mg/L	1.36	0.19
Na	mg/L	5	2.2
Sr	mg/L	0.149	0.082
Zn	mg/L	2.45	0.16

The first stage leach extraction test results represent the initial equilibrium conditions under oxidizing (open to the atmosphere) conditions, whereas the

saturated column test results represent equilibrium conditions under anoxic (oxygen excluded) conditions.

A number of significant differences exist between these data sets. First, it should be noted that the solid to liquid ratio between the two tests are significantly different. The solid to liquid ratio for the anoxic column test was much lower than that of the leach extraction, and the contact time was much longer. The column test results are therefore more likely to provide an indication of the equilibrium concentrations that may result in the saturated sludge pore water. Consistent with this is the much higher sulphate concentration (and the conductivity) observed for the anoxic column test.

In contrast to the saturated column, the three stage leach extraction test uses 'fresh' water for each extraction step. If equilibrium constraints exist, the three stage extraction would yield higher overall solute releases (combined for stages 1 to 3). Typically, the overall release of the more soluble parameters such as magnesium would be expected to be similar for the two tests. However, extraction of the sulphate and magnesium in the saturated column did not occur to the level observed for the third stage of the sequential tests. This suggests that there is either a kinetic control on the rate of dissolution, or there is a possible secondary mineral phase that may be limiting the solubility of either or both the sulphate and the magnesium. To test this hypothesis, geochemical speciation modelling of the saturated column test leachate was undertaken, using the MINTEQA2 model. The results of the modelling can be summarised as follows:

- In the first modelling step, the purpose was simply to determine which phases may be present at equilibrium with the sludges. In this step, the pH was fixed at that measured for the column test. The results are as follows:
 - a) At the measured pH, the geochemical modelling confirms the presence of calcite (CaCO_3) and barite (BaSO_4).
 - b) The results suggest that magnesite (MgCO_3) may also be present, which could explain the limited magnesium solubility.
 - c) The modelling further indicated that amorphous zinc hydroxide (Zn(OH)am.) may also be present, albeit that the zinc concentration is below saturation.
 - d) Manganese concentrations are likely limited by an oxy-hydroxide (e.g. manganite MnO(OH)).
 - e) While below detection, the modelling run included cadmium at the detection limit. The results suggest that the solubility of cadmium is likely limited to the solubility of otavite (CdCO_3).
- In the second step, the potential zinc concentration that may result in the porewater at the prevailing pH conditions was estimated by allowing amorphous zinc hydroxide to equilibrate with the porewater. The modelling results indicate that:
 - a) Once fully equilibrated, the zinc concentration in the porewater could increase to about 63 mg/L.
- In a third step of modelling, rather than limiting the porewater pH to that measured, the pH was calculated by allowing the phases that are likely present in the sludge (i.e. calcite, amorphous zinc hydroxide, manganite) to equilibrate. The results indicate that:
 - a) The porewater pH is likely to equilibrate at about 9.3.

- b) At the equilibrated condition, the zinc concentration will be limited to about 0.6 mg/L.

It is concluded from the geochemical modelling that the zinc solubility is pH dependent, and that even a relatively small decrease in pH could result in a significant increase in the zinc concentration. The results also suggest that under their present condition, the porewater pH would buffer to in excess of 9 and the resultant zinc concentration would be about 0.6 mg/L.

3.8 Neutralization Potential

The neutralization potential of the sludge was determined using the Modified Sobek method. The paste pH of the sludge is 9.0 and the neutralization potential is 382.5 kg CaCO₃eq/tonne, measured to an endpoint pH of 8.3.

The carbonate neutralization potential, which represents that calcium and magnesium carbonate buffering capacity, as calculated from the carbonate content is about 313 kgCaCO₃. The difference between the Modified Sobek method NP and the carbonate NP likely represents the dissolution of zinc hydroxides.

A plot of the base titration of the acidified sample for the Modified Sobek NP test, expressed as sample NP, is provided in Figure 3-2. The plot shows a buffering zone from about pH 6 to pH 7, which continues, albeit at a flatter slope through pH 8.3. It is considered that this represents the reprecipitation of zinc that had been dissolved from the sludge. The NP equivalent of the amorphous zinc hydroxide is estimated to be about 378 kgCaCO₃ eq/tonne. This corresponds well with the NP difference between pH 6 and the endpoint (about 323 kgCaCO₃ eq/tonne). It is therefore concluded that the zinc (hydroxide) was dissolved when the sample was acidified, and it starts to reprecipitated (as zinc hydroxide) when the pH increases above 6. It should however be noted that not all of the zinc would have been precipitated at the endpoint of the test.

The porewater of the saturated column test reached an alkalinity of about 77 mg CaCO₃ eq/L. The corresponding results for the leach extraction tests ranged from 58 in the first stage, to 88 mgCaCO₃/L in the third stage. At these concentrations, the porewater pH would be readily buffered by the available carbonate minerals for many (in excess of 500) porewater displacements. This represents a contact ratio of about 3500:1.

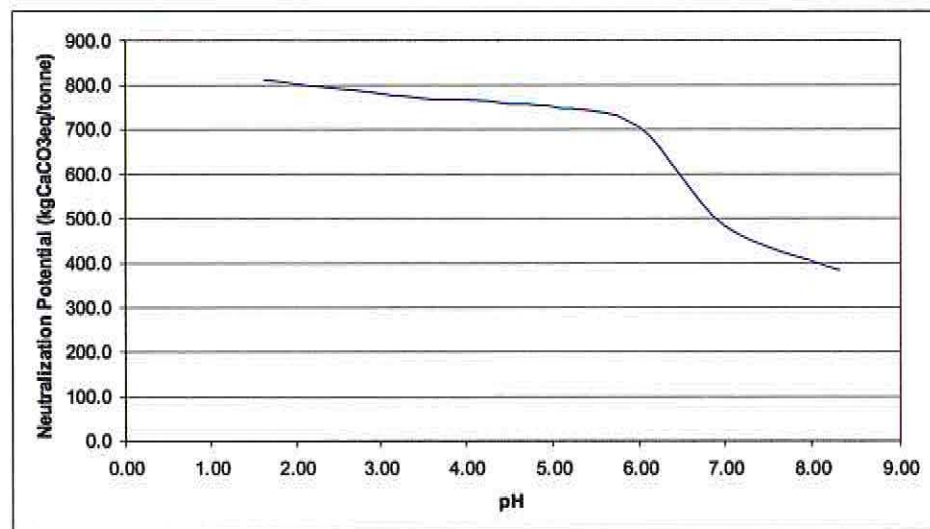


Figure 3-2 NP Representation of Base Titration of Acidified Sample

3.9 Summary

The results of the sludge characterization program can be summarised as follows:

- As received, the sludge samples contained on average 16 % solids. Only the 'wettest' sample, at a moisture content 86 %, showed only minor amounts of free porewater, and none of the remaining samples contained free-draining porewater. Due to this, porewater extraction was precluded from the testing program.
- The sludge comprises predominantly zinc, manganese and calcium. A minor proportion of the secondary minerals present are carbonates, and an even lesser amount is present as sulphates. Mineralogical examination indicated the vast majority of the sludge to be amorphous, hydroxide phases. Calcite was identified in the sludge and probably accounts for most of the carbonate present in the sample.
- A composite sample of the sludge, representing the entire deposit, did not exceed the BC SWEP test criteria.
- The sludge, re-suspended in water, showed poor consolidation properties, and reached a density of about 13.4 % solids after 8 days of consolidation.
- Sequential distilled water leach extraction testing indicated that predominantly sulphate and magnesium would be readily leached from the sludge, and that most of the sulphate is leachable. For the conditions tested, zinc was not readily leachable. The tests however indicated that gypsum is not present at any significant quantities in the sludge.
- Results from the anoxic saturated column test however indicate that zinc may be dissolved to about 2.5 mg /L in a period of 37 days. Geochemical modelling suggests that at the measured pH, concentrations as high as 63 mg/L may result in the porewater.

4 Conclusions and Recommendations

4.1 Conclusions

Based on the test results and geochemical modelling presented herein, it is concluded that:

- The majority of the zinc within the water treatment sludge is present as an amorphous hydroxide, and that the porewater pH is effectively buffered by secondary calcite and magnesite that have formed in the sludge.
- The buffered conditions ensure low zinc concentrations in the sludge porewater as evidenced in the sequential leach extraction test results.
- Under saturated conditions, the zinc concentrations in the porewater increased, as evidenced by the anoxic column test results. However, the pH was slightly lower than the predicted equilibrium pH, which may be a result of the anoxic conditions.
- Sufficient buffering capacity is available in the sludge to ensure carbonate buffering for many pore volume displacements. However, the available buffering capacity may be depleted at a water to solids contact ratio of about 3500:1.
- The neutralization potential determination further indicated that if acidified to a pH of about 6, most of the zinc would be dissolved from the sludge (see Section 3.8).

It is therefore concluded that the sludge should not be disposed of in a location where i) it may be at risk to acidification and/or, ii) the solids to water contact ratio could exceed about 3500 : 1 to prevent excessive release of zinc.

4.2 Recommendations

The interim care and maintenance options for sludge disposal that are being considered include on-land disposal with the waste rock and in-pit disposal, in the pit lake.

Disposal of sludge in the Vangorda waste rock dump, as required by the current water licence, is not recommended because of the known acid generation potential of the waste rock in that dump. Acidification of the sludge in contact with the waste rock could mobilise zinc and increase the solute loading to the receiving environment.

Unless the pH of the Vangorda Pit lake is maintained to above 9.3, the large contact ratio of the pit lake would likely result in the dissolution of zinc to elevated concentrations. The post closure management of the pit lake is currently being investigated by the Type II Mines Office as part of the development of the Final Closure and Rehabilitation Plan, which includes a 'clean pit' flow through option. Such an option would preclude sludge disposal in the Vangorda Pit as a preferred interim care and maintenance option. At the time of writing, the investigation of the feasibility of a flow through pit option was under way, and is scheduled for completion in the

spring of 2004. The feasibility for in pit disposal should be re-evaluated at that time.

It is therefore recommended that the interim care and maintenance sludge management plan be finalised in March of 2004 to ensure conformity with the final closure plan. In the interim, if necessary, it is recommended that a suitable on-land disposal location be identified. As an interim measure, the sludge could be contained within a bermed area, free of potentially acid generating waste rock. As an interim measure, capping and lining would not be foreseen as essential; however, these measures would benefit longer term storage. Rehandling of the sludge may be required once the long term sludge storage strategy has been developed.

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Table A-1
Elemental Analysis of Sludge Samples

Parameter		Sample					
		Near Lower	Near Upper	Mid Lower	Mid Upper	Far Lower	Far Upper
Ag	ppm	7.4	8.2	7.4	7.4	8.2	8.4
Al	%	<0.01	<0.01	0.04	<0.01	<0.01	<0.01
As	ppm	15	<5	5	5	5	5
Ba	ppm	70	40	60	50	50	40
Be	ppm	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Bi	ppm	<5	<5	<5	<5	<5	<5
Ca	%	7.08	5.29	7.11	7.37	4.96	5.14
Cd	ppm	>100	>100	>100	>100	>100	>100
Cd*	%	0.030	0.033	0.030	0.031	0.037	0.036
Co	ppm	935	1005	906	888	1058	1070
Cr	ppm	26	27	25	23	27	27
Cu	ppm	228	130	96	131	98	98
Fe	%	0.65	0.60	0.63	0.65	0.48	0.47
K	%	0.01	0.01	0.03	0.02	0.01	0.01
Mg	%	4.91	5.71	5.06	4.73	5.53	5.92
Mn	ppm	>10000	>10000	>10000	>10000	>10000	>10000
Mn*	%	6.39	7.29	6.10	6.57	7.14	7.22
Mo	ppm	<2	<2	<2	<2	<2	<2
Na	%	0.01	0.01	0.02	0.02	0.01	0.01
Ni	ppm	1011	1097	986	969	1146	1159
P	ppm	30	<10	140	80	60	70
Pb	ppm	74	64	60	52	58	60
Sb	ppm	<5	<5	<5	<5	<5	<5
Sc	ppm	<1	<1	1	<1	<1	<1
Sn	ppm	<10	<10	<10	<10	<10	<10
Sr	ppm	225	143	175	197	135	144
Ti	%	<0.01	<0.01	0.01	0.01	<0.01	<0.01
V	ppm	3	1	5	3	2	2
W	ppm	5240	5850	4990	5040	5850	5900
Y	ppm	2	2	2	2	2	2
Zn	ppm	>10000	>10000	>10000	>10000	>10000	>10000
Zn*	%	23.50	26.40	21.30	23.40	26.90	26.40
Zr	ppm	<1	<1	<1	<1	<1	<1
S(T)	%	1.13	1.51	1.16	1.14	1.21	1.29
S(SO4)	%	1.10	1.50	1.11	1.09	1.17	1.23
TIC	%C	4.24	3.31	3.85	4.03	3.53	3.56

BACKGROUND

A set of six samples was submitted to PetraScience Consultants by Sohan Bosra (CEMI) in July 2003 for mineralogical testing. The samples were described as metal hydroxide sludges from processing at the Faro Mine. The samples are known to contain large amounts of Zn and Mn as well as Ca. Minor sulfate also appears to be present based on geochemical analysis.

Two types of analysis were attempted; 1) scanning electron microscope (with energy dispersive analysis) and 2) X-ray diffraction using full Rietveld analysis. Anne Thompson of PetraScience Consultants Inc. carried out the SEM analysis. Mati Raudsepp, Ph.D. and Elisabetta Pani, Ph.D. completed the XRD analysis in the Dept. Earth and Ocean Sciences, The University of British Columbia.

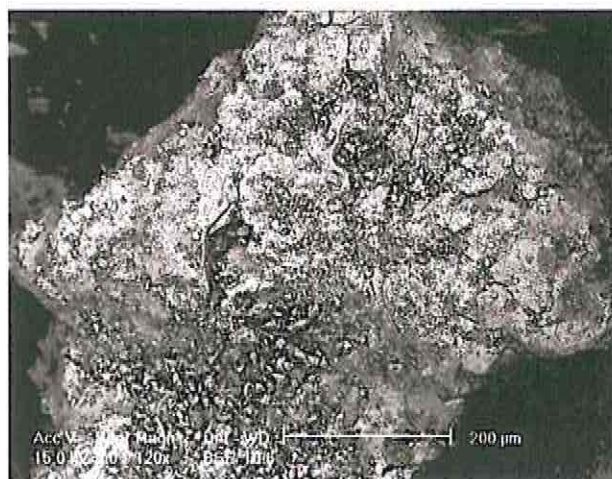
SUMMARY

Five samples were analyzed with the SEM (16381, 16382, 16383, 16384 and 16386), however most of the analytical time was spent on 16381 and 16384. The samples all showed strong similarities with areas of fine grey material (in backscattered images) that appears to be dominated by calcium. These could not be verified as calcium hydroxide or calcite. The dominant material consists of Zn with lesser Mn and minor Si, S. Sample 16381 did appear to have trace amounts of a clay or mica (see spectrum in following pages). These grains were also extremely small and it was difficult to obtain spectra representative of just one phase.

Rietveld analysis was carried on samples 16382 and 16386. These samples proved to be virtually identical. The only crystalline phases that could be identified were calcite and quartz. Clearly from the SEM analysis, these are not the dominant phases. A variety of hydroxides were attempted as fits to the patterns, but none worked. The large hump in the background indicates the presence of amorphous material.

SEM ANALYSIS

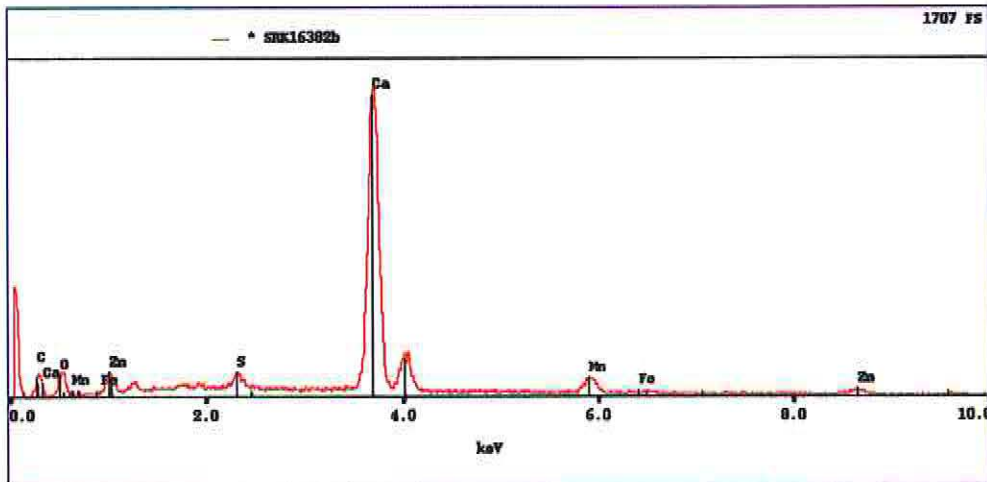
The samples were selected for scanning electron microscope (SEM) analysis in order to characterize particle sizes and attempt to identify minerals present. The samples were analyzed using the SEM in the Earth and Ocean Sciences Department at the University of British Columbia, Vancouver. The SEM is a Philips XL30 with a Princeton Gamma Tech energy dispersion X-ray spectrometer (EDS). Back-scattered electron (BSE) images and EDS spectra are included for each of the three samples.



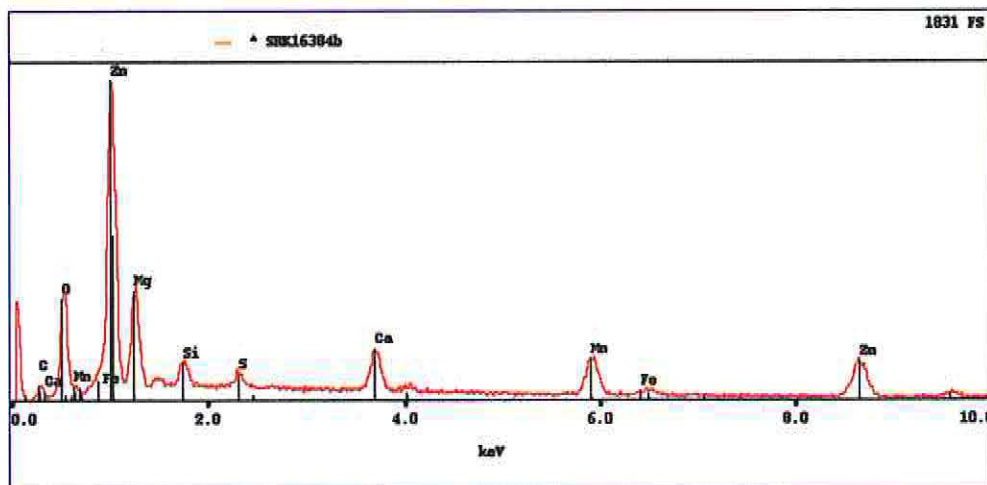
Backscattered image of clot of sludge/powder (Sample 16381) . Lighter areas are rich in Zn, while fine dark grey zones are dominated by Ca.



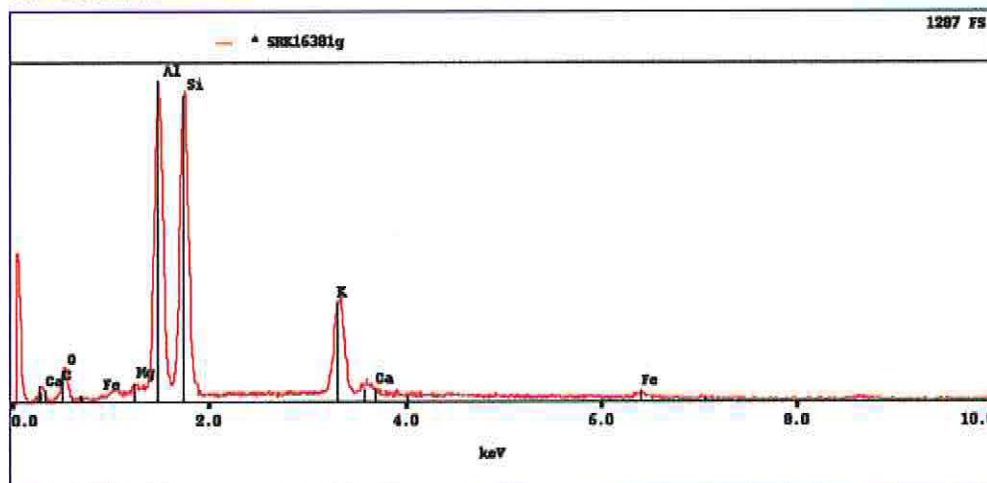
Backscattered image of sludge, detail of above. Grains are extremely small, note scale bar is 50 microns. Individual grains where they can be identified are less than 5 microns and typically 1-2 microns.



Area of typical fine grey material. This material is dominated by Ca which may be present as calcium hydroxide.



Typical spectrum for smooth light grey material. This material is dominated by Zn with lesser Mn and Ca. Minor Si and S are also present, however grain sizes are so fine, that no individual material or mineral can be identified with certainty.



Spectrum from sample 16381 suggesting the presence of mica (K Al Si).

XRD ANALYSIS

Experimental Methods

The particle size of the samples was further reduced to the optimum grain-size range for X-ray analysis ($<5\ \mu\text{m}$) by grinding under ethanol in a vibratory McCrone Micronising Mill (McCrone Scientific Ltd., London, UK) for 7 minutes. Fine grain-size is an important factor in reducing micro-absorption contrast between phases. Samples were pressed from the bottom of an aluminum sample holder against a ground glass slide; the cavity in the holder measures $43 \times 24 \times 1.5\ \text{mm}$. The textured surface of the glass minimizes preferred orientation of anisotropic grains in the part of the powder that is pressed against the glass.

Step-scan X-ray powder-diffraction data were collected over a range $3\text{--}70^\circ 2\theta$ with $\text{CuK}\alpha$ radiation on a standard Siemens (Bruker) D5000 Bragg-Brentano diffractometer equipped with a diffracted-beam graphite monochromator crystal, $2\ \text{mm}$ (1°) divergence and antiscatter slits, $0.6\ \text{mm}$ receiving slit and incident-beam Soller slit. The long sample holder used ($43\ \text{mm}$) ensured that the area irradiated by the X-ray beam under these conditions was completely contained within the sample. The long fine-focus Cu X-ray tube was operated at $40\ \text{kV}$ and $40\ \text{mA}$, using a take-off angle of 6° . X-ray powder-diffraction data were refined with Rietveld program Topas 2.0 (Bruker AXS).

Results

The X-ray diffractograms were analyzed using the International Centre for Diffraction Database PDF2 Data Sets 1-49 plus 70-86 using Search-Match software by Siemens (Bruker). The results of quantitative phase analysis by Rietveld refinement are given in Table 1. **Note that these amounts represent the relative amounts of crystalline phases normalized to 100%. Judging from the nature of the “humpy” background and the presence of a large, wide peak at 34-35 degrees, most of the sample is amorphous. As the samples are poorly crystallized with a poorly defined background, the results should be considered semi-quantitative only.**

The X-ray powder diffraction patterns are shown in Figures 1 and 2. Note that the samples are virtually identical. A few very small peaks could not be fitted. Analysis of the unit cell dimensions of calcite show that it is very close to CaCO_3 in composition. The presence of elemental Zn is likely, but not certain.

TABLE 1. RESULTS OF QUANTITATIVE PHASE ANALYSIS (wt. %)

Mineral	Ideal Formula	CEMI16382	CEMI16386
Quartz	SiO_2	94	94
Calcite	CaCO_3	4	4
Zn?	Zn	2	2
Total		100	100

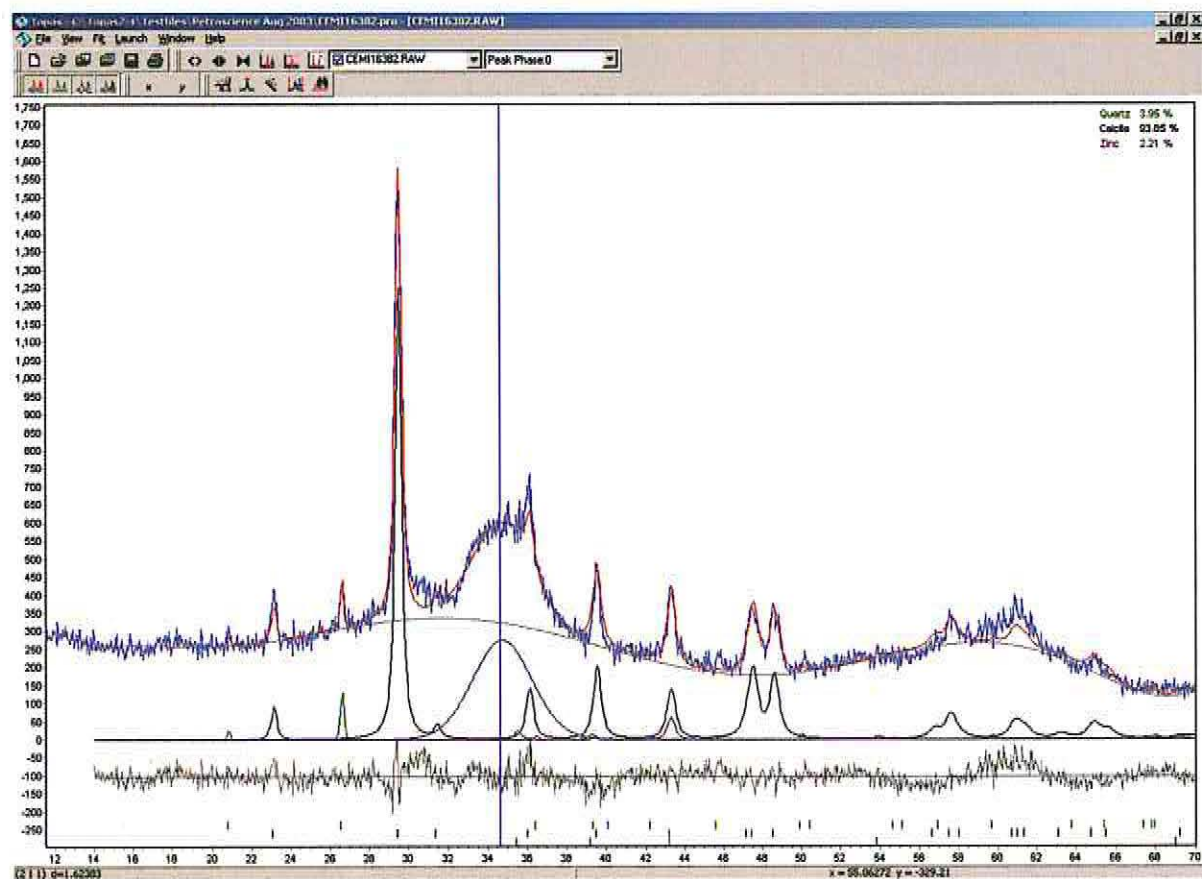


Figure 1: Rietveld refinement plot for sample CEMI16382 (blue line - observed intensity at each step; red line - calculated pattern; solid grey line – background, solid grey line below – difference between observed and calculated intensities; vertical bars, positions of all Bragg reflections). Coloured lines are individual diffraction patterns of all phases.

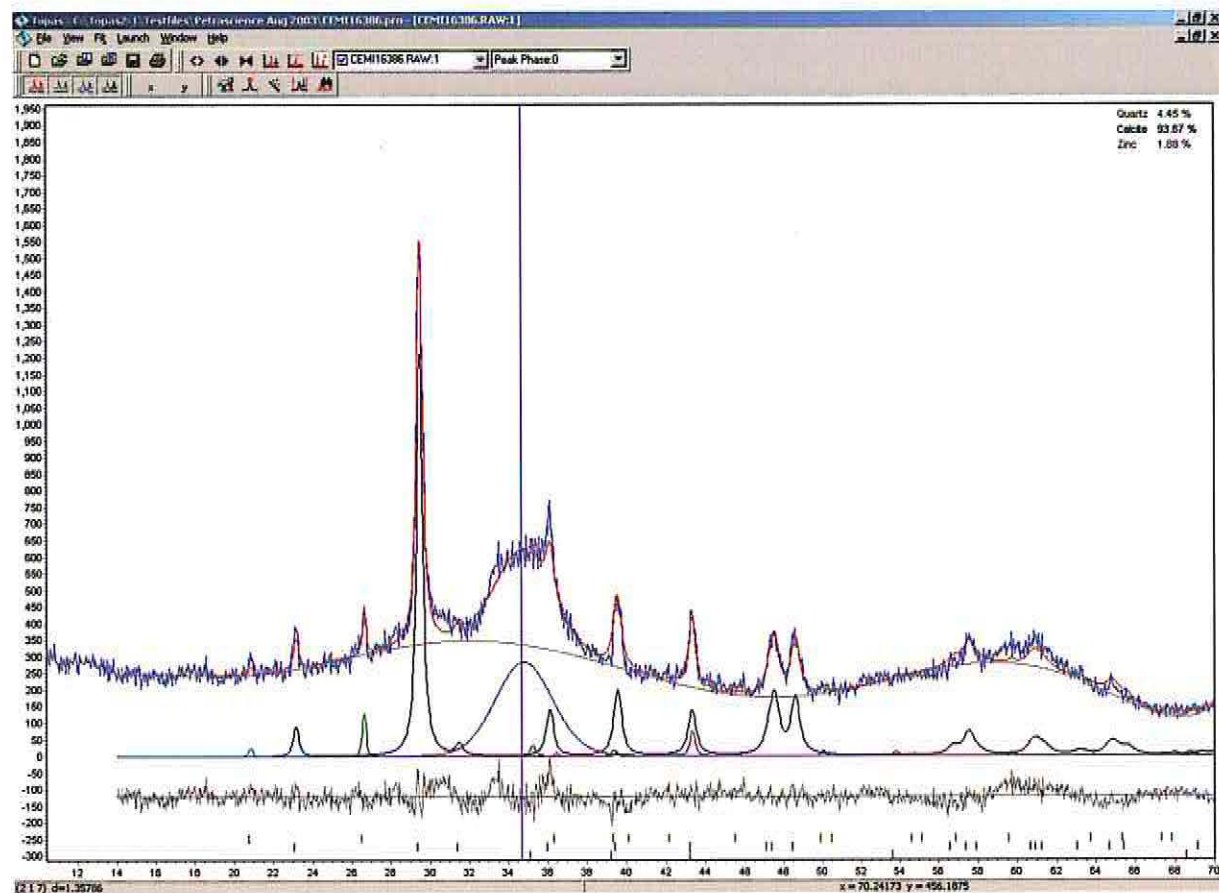


Figure 2: Rietveld refinement plot for sample CEMI16386 (blue line - observed intensity at each step; red line - calculated pattern; solid grey line – background, solid grey line below – difference between observed and calculated intensities; vertical bars, positions of all Bragg reflections). Coloured lines are individual diffraction patterns of all phases.

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Appendix C

Three Stage Sequential Leach Extraction Test Results

Table C-1 Three Stage Leach Extraction Tests - Leachate Parameters

SAMPLE	DISTILLED WATER VOLUME (mL)	WET SAMPLE WEIGHT (g)	pH	CONDUCTIVITY (uS/cm)	ALKALINITY (mg CaCO ₃ /L)	ACIDITY		SULPHATE (mg/L)
						(pH 4.5) (mg CaCO ₃ /L)	(pH 8.3) (mg CaCO ₃ /L)	
N L Stage 1	450	150	8.82	1145	51.5	0.0	0.0	814
N L Stage 2	450	150	8.71	735	79.5	0.0	0.0	473
N L Stage 3	450	150	8.26	531	89.5	0.0	0.0	301
N U Stage 1	450	150	8.82	1122	56.5	0.0	0.0	800
N U Stage 2	450	150	8.81	708	70.5	0.0	0.0	467
N U Stage 3	450	150	8.22	565	88.5	0.0	0.0	289
M L Stage 1	450	150	8.83	1115	53.5	0.0	0.0	800
M L Stage 2	450	150	8.61	679	69.0	0.0	0.0	461
M L Stage 3	450	150	8.26	524	81.5	0.0	0.0	248
M U Stage 1	450	150	8.90	1220	63.0	0.0	0.0	947
M U Stage 2	450	150	8.76	711	74.0	0.0	0.0	508
M U Stage 3	450	150	8.21	565	84.5	0.0	0.0	289
F L Stage 1	450	150	8.89	1120	59.0	0.0	0.0	712
F L Stage 2	450	150	8.68	670	70.5	0.0	0.0	461
F L Stage 3	450	150	8.21	569	86.5	0.0	0.0	301
F U Stage 1	450	150	8.94	1233	68.5	0.0	0.0	1035
F U Stage 2	450	150	8.66	782	91.5	0.0	0.0	546
F U Stage 3	450	150	8.38	642	101.5	0.0	0.0	332

Table C-2 Three Stage Leach Extraction Leachate Analysis

Sample Name:		Units	Near Lower			Near Upper			Mid Lower			Mid Upper			Far Lower			Far Upper		
			Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3
Dissolved Metals																				
Aluminum	Al	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Antimony	Sb	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Arsenic	As	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Barium	Ba	(mg/L)	0.01	0.01	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Beryllium	Be	(mg/L)	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Bismuth	Bi	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Boron	B	(mg/L)	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cadmium	Cd	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Calcium	Ca	(mg/L)	16.6	9.31	5.96	14.9	8.32	5.68	15.6	9.02	5.73	14.6	8.17	5.86	13.5	8.40	5.71	14.5	8.33	5.65
Chromium	Cr	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cobalt	Co	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Copper	Cu	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Iron	Fe	(mg/L)	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Lead	Pb	(mg/L)	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Lithium	Li	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01
Magnesium	Mg	(mg/L)	228	137	93.2	218	132	92.1	214	130	81.5	254	139	91.6	212	133	93.4	286	184	113
Manganese	Mn	(mg/L)	0.047	0.015	0.015	0.032	0.009	0.008	0.046	0.009	0.007	0.038	0.012	0.007	0.039	0.010	0.021	0.042	0.011	0.007
Molybdenum	Mo	(mg/L)	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Nickel	Ni	(mg/L)	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Phosphorous	P	(mg/L)	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3
Potassium	K	(mg/L)	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2
Selenium	Se	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Silicon	Si	(mg/L)	0.18	0.18	0.17	0.15	0.11	0.13	0.25	0.14	0.13	0.17	0.14	0.14	0.17	0.12	0.14	0.19	0.15	0.15
Silver	Ag	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Sodium	Na	(mg/L)	3	<2	<2	<2	<2	<2	2	<2	<2	2	<2	2	2	<2	<2	2	2	<2
Strontium	Sr	(mg/L)	0.100	0.057	0.038	0.086	0.050	0.037	0.076	0.042	0.033	0.075	0.042	0.034	0.074	0.050	0.035	0.080	0.050	0.035
Thallium	Tl	(mg/L)	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Tin	Sn	(mg/L)	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Titanium	Ti	(mg/L)	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Vanadium	V	(mg/L)	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
Zinc	Zn	(mg/L)	0.170	0.070	0.057	0.124	0.064	0.064	0.168	0.073	0.062	0.173	0.086	0.063	0.160	0.062	0.113	0.165	0.078	0.059

Table C-3 Three Stage Leach Extraction - Calculated Solute Release for Parameters above Detection Limits

Sample Name:		Units	Near Lower			Near Upper			Mid Lower			Mid Upper			Far Lower			Far Upper		
			Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3	Stage 1	Stage 2	Stage 3
Aluminum	Al	mg/kg																		
Antimony	Sb	mg/kg																		
Arsenic	As	mg/kg																		
Barium	Ba	mg/kg	0.19	0.38	0.75	0.19	0.38	0.56	0.19	0.38	0.56	0.19	0.38	0.56	0.19	0.38	0.56	0.19	0.38	0.56
Beryllium	Be	mg/kg																		
Bismuth	Bi	mg/kg																		
Boron	B	mg/kg																		
Cadmium	Cd	mg/kg																		
Calcium	Ca	mg/kg	311	486	598	279	435	542	293	462	569	274	427	537	253	411	518	272	428	534
Chromium	Cr	mg/kg																		
Cobalt	Co	mg/kg																		
Copper	Cu	mg/kg																		
Iron	Fe	mg/kg																		
Lead	Pb	mg/kg																		
Lithium	Li	mg/kg																		
Magnesium	Mg	mg/kg	4275	6844	8591	4088	6563	8289	4013	6450	7978	4763	7369	9086	3975	6469	8220	5363	8813	10931
Manganese	Mn	mg/kg	0.9	1.2	1.4	0.60	0.77	0.92	0.86	1.03	1.16	0.71	0.94	1.07	0.73	0.92	1.31	0.79	0.99	1.13
Molybdenum	Mo	mg/kg																		
Nickel	Ni	mg/kg																		
Phosphorous	P	mg/kg																		
Potassium	K	mg/kg																		
Selenium	Se	mg/kg																		
Silicon	Si	mg/kg	3.4	6.8	9.9	2.81	4.88	7.31	4.69	7.31	9.75	3.19	5.81	8.44	3.19	5.44	8.06	3.56	6.38	9.19
Silver	Ag	mg/kg																		
Sodium	Na	mg/kg	56	56	56				38	38	38	38	38	75	38	38	38	38	75	75
Strontium	Sr	mg/kg	1.9	2.9	3.7	1.6	2.6	3.2	1.4	2.2	2.8	1.4	2.2	2.8	1.4	2.3	3.0	1.5	2.4	3.1
Thallium	Tl	mg/kg																		
Tin	Sn	mg/kg																		
Titanium	Ti	mg/kg																		
Vanadium	V	mg/kg																		
Zinc	Zn	mg/kg	3.2	4.5	5.6	2.3	3.5	4.7	3.2	4.5	5.7	3.2	4.9	6.0	3.0	4.2	6.3	3.1	4.6	5.7

Appendix D

Saturated Anoxic Column Test Results

Table D-1 Saturated Column - Test Conditions

Parameter	Units	Value
Contact Time	days	37
Leachate Collected	L	0.200
pH	s.u.	8.15
Redox.	mV	321
Conductivity	µS/cm	1508
Acidity to pH 4.5	mg CaCO ₃ /L	
Total Acidity	mg CaCO ₃ /L	1.0
Alkalinity	mg CaCO ₃ /L	77.0

Table D-2 Saturated Column Test Leachate Analysis and Solute Release

Parameter	Concentration (mg/L)	Extracted (mg/kg)
Sulphate	1550	12788
Al	<0.2	0.17
Sb	<0.2	
As	<0.2	
Ba	0.02	
Be	<0.005	
Bi	<0.3	287
B	<0.1	
Cd	<0.01	
Ca	34.8	
Cr	<0.01	
Co	0.02	0.17
Cu	<0.01	
Fe	<0.03	
Pb	<0.05	
Li	<0.01	
Mg	433	3572
Mn	0.149	
Mo	<0.03	
Ni	<0.05	
P	<0.3	
K	3.0	25
Se	<0.2	
Si	1.36	
Ag	<0.01	
Na	5	
Sr	0.149	1.2
Tl	<0.2	
Sn	<0.03	
Ti	<0.01	
V	<0.03	
Zn	2.45	20