### **Comparison of Laboratory and Field-Scale Predictions of Processed Kimberlite Effluent in the Arctic**

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#### ABSTRACT

When assessing mine closure options, predicting mine waste effluent water quality is a particular challenge. Conventional static and kinetic tests on small sample volumes are typically used to assess if the mine wastes will be acid generating. However, using these small scale tests to assess if or when field-scale waste piles will release poor quality effluent requires recognizing scale-up issues between laboratory and field conditions. At the Diavik Diamond Mine, up to 42 million tonnes of fine processed kimberlite (FPK) will be deposited onsite for permanent storage. To evaluate the effectiveness of using lab scale experiments to predict mineral weathering and the evolution of porewater geochemistry from the FPK storage impoundment, testing was done to quantify the relationship between laboratory humidity cells and large field-scale tanks. Three fractions of FPK were used for the humidity cell experiments including fine, medium and coarse grained fractions of slurry-deposited FPK. Humidity cells representing the three grain sizes were run for 80 weeks in duplicate at 4°C and 20°C, following the ASTM method. Concurrently, three 5700 L high-density polyethylene tanks were filled with the same fractions of FPK material as the humidity cells. The three tanks were constructed to represent unsaturated conditions. Each tank was instrumented in detail to measure pore water geochemistry, gas-phase concentrations and oxygen diffusion, matrix pressure and flow, evolution of temperature, as well as to resolve mass and flow balances. Results from this study will be used for closure plans at Diavik and potentially other Diamond mines in the Canadian North.

Keywords: Sulfide oxidation, neutralization, processed kimberlite, humidity cells, scale-up

#### INTRODUCTION

Processed kimberlite (PK) is discharged by slurry to permanent tailings impoundments at diamond mines in Canada's arctic. The Diavik Diamond Mine (Diavik) is an operating mine with two completed open pits and an underground mine. Up to 42 million tonnes of slurry-deposited fine processed kimberlite is expected to be permanently retained on-site in the Processed Kimberlite Containment (PKC) facility. Slurry-deposited PK is defined by Diavik as the < 1 mm fraction of the PK stream and is referred to as fine PK (FPK). Diavik is located 300 km northeast of Yellowknife, NT, Canada (Figure 1) in the remote barren lands of the Canadian arctic. The area is dry and cold with a mean annual temperature, rainfall, snowfall and lake evaporation of -12°C, 164 mm, 187 mm and 271 mm, respectively.

Laboratory humidity cell experiments and *in situ* 5700 L tank experiments were initiated in 2012 to complement an on-going *in situ* PKC experiment at the Diavik Diamond Mine (Moncur and Smith, 2012) to understand better the complexities of using small-scale laboratory experiments for long-term predictions of seepage quality.

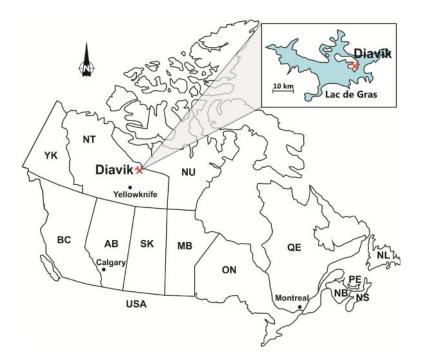


Figure 1 Location of Diavik Diamond Mines in NT, Canada.

#### METHODOLOGY

Processed kimberlite used for this study was deposited by slurry in 2011 on the East Beach of the PKC. Using a track hoe, FPK was excavated and transported by dump truck on August 2, 2012 to the experiment site. The coarse fraction of FPK (CT) was collected near the toe of the containment dam; the fine fraction of FPK (FT) was collected approximately 200 m from the dam, adjacent to the standing pond water; and the medium FPK fraction (MT) was collected mid-way between the pond

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and the containment dam. During FPK excavation every attempt was made to remove the overlying unsaturated FPK to prevent the collection of previously weathered material. Material collected for the tanks was excavated from within the saturated and frost zones of the impoundment.

#### Tank design and construction

Instrumentation was selected to measure pore water geochemistry, gas-phase concentrations and oxygen diffusion, matrix pressure and flow, evolution of temperature, as well as to resolve mass and flow balances, developed after Smith et al. (2013).

Each tank was constructed from a 2 m in height by 2 m diameter 5700 L HDPE tank. The top of each tank was cut off at a 1.8 m and a 44.5 mm drainage hole was drilled in the wall of the tanks near the bottom for drainage. Water discharging from the bottom of each tank was channeled through a 37.5 mm schedule 40 PVC pipe into a sealed sample cell (Figure 2). Overflow from the sample cell discharged into a data-logging tipping bucket rain gauge so that a continuous record of flow could be maintained to provide bulk chemistry and volume of flow from each tank.



Figure 2 Photos showing (A) completed PK tanks, from left to right FT, MT, CT (B) tank bottom drain pipe discharging into the sample cell and tipping bucket (C) instrumentation on tank tops.

Soil water solution samplers (SWSS) were installed in each tank to provide point measurements of pore water solute concentrations by extracting pore water using applied suction from an area of influence around the probe tip. SWSS were installed at depths of 0.25, 0.5, 1.0, 1.5 and 1.7 m.

Decagon Devices ECH<sub>2</sub>O-TE probes were installed in each tank at depths of 0.25, 0.5, 0.75, 1.0 and 1.5 m to provide discrete measurements of electrical conductivity (EC), volumetric water content and temperature. Measurements of volumetric water content through space and time can be used to monitor the wetting front propagation through each tank in which discontinuous zones of ice may form, and may indicate the locations and/or transient nature of any preferential flow paths. Continuous monitoring of EC at various depths will provide an understanding of the evolution of pore water during weathering, precipitation and freeze/thaw events. All ECH<sub>2</sub>O probes were connected to a Decagon Devices EM50 data logger that continually records measurements every 4 hours.

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A thermistor string consisting of 10 thermistors was installed at 0.0, 0.25, 0.5, 0.75, 1.0, 1.25, 1.5 and 1.75 m depths through each tank to allow the determination of spatial and temporal variations in permafrost formation, as well as the quantification of thermal contributions from exothermic sulfide oxidation reactions. Nine of the thermistors were attached to a 37.5 mm PVC stand pipe for support, with the bottom thermistor placed on the tank bottom. The PVC stand pipe was filled with FPK to prevent the conduction of air temperatures down the pipe annulus. Thermistor strings were connected to Lakewood Systems UltraLogger data loggers for continuous measurements.

Gas sampling lines consist of 0.63 mm I.D. low density polyethylene tubing installed at 0.1, 0.2, 0.3, 0.4, 0.5, 0.7, 0.9, 1.1, 1.3, 1.5 and 1.7 m depths within each tank. Internal gas compositions in each tank will be used to understand O<sub>2</sub> gas transport mechanisms, O<sub>2</sub> consumption rates from the oxidation of sulfide minerals, and CO<sub>2</sub> production or consumption rates from the dissolution or precipitation of carbonate minerals. Measured O<sub>2</sub> consumption rates can be used to determine sulfide mineral oxidation rates assuming sulfide oxidation is the only process consuming O<sub>2</sub> (Linklater, Sinclair and Brown, 2005).

Soil moisture 2725ARL Jet FI11 tensiometers were installed in the unsaturated zone of the tanks to measure pressure potential (matrix potential) in the FPK at depths of 0.25, 0.5, 1.0, 1.5 and 1.7 m. Pressure potential measurements will be used to calculate the gradient pressure in order to determine the rate of water movement through the unsaturated zone in the tanks.

Each tank was filled with the assigned FPK fraction in lifts of 0.3 to 0.4 m. Filling the tanks in lifts was necessary to accurately place the instruments and insure material was applied uniformly and compacted. Tanks were filled with FPK to a height of 1.7 m above the tank bottom. During filling of the tanks, a 20 L composite sample from each tank was collected for humidity tests and analyses of mineralogy, grain size, total sulfur and carbon, whole rock analyses, acid-base accounting and neutralization potential. All tanks were filled and instrumented on August 3, 2012 and remain exposed to ambient conditions year-round.

#### Sampling

Water sampling was initiated on August 28, 2012. Porewater was collected into SWSS by applying a vacuum of approximately 70 kPa two days prior to sampling. Water samples were collected from SWSS and drain sample cells using 60 mL polyethylene (PE) syringes. Measurements of pH, Eh and electrical conductivity (EC) were made on unfiltered samples in the field. The resulting water samples were passed through 0.45  $\mu$ m filters and split into three aliquots. One aliquot of water was acidified with 12 *N* trace-metal grade HNO<sub>3</sub> to a pH of <1 for cation analysis, and another aliquot was left unacidified to use for anion analysis. The remaining water was used for alkalinity and NH<sub>3</sub> analyses. Water samples collected for anion and cation analyses were immediately refrigerated until analyses. Determination of major cations and trace elements was performed by inductively coupled plasma–optical emission spectroscopy (ICP–OES) and inductively coupled plasma-mass spectrometry (ICP–MS), respectively. Ion chromatography was used to measure inorganic anion concentrations. Concentrations of O<sub>2</sub> and CO<sub>2</sub> gas were measured from gas lines using a Grafton Model 902 O<sub>2</sub>/CO<sub>2</sub> analyzer. Air temperature and precipitation data from site was provided by Diavik.

#### **Humidity Cells**

Two samples of each the fine-, medium- and coarse fractions of FPK were split from the tank material for an 80 week kinetic testing program. Kinetic testing followed the ASTM (1996) 5744-07

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protocol, with the exception that samples were not crushed. Original size fractions were maintained in order to observe any geochemical variation caused by differences in grain size distributions. One set of fine, medium and coarse humidity cells was operated at room temperature (~22°C) and one set was operated at 4°C. The cells were subjected to a weekly cycle of a 3-day dry period (0% relative humidity), 3-day wet period (>95% relative humidity), and a 1-day flood leach with 500 mL of DI water. Effluent from each humidity cell was analyzed weekly for pH, ORP, acidity, alkalinity and SO<sub>4</sub>. Dissolved metals, Cl and F were analyzed weekly from week 1-20, biweekly from week 20-40, and every six weeks from week 40-80.

#### **RESULTS AND DISCUSSION**

#### Mineralogy

The kimberlite pipes at Diavik are intrusions within supracrustal rocks and late Archean granitoids of the Slave structural province (Moss, Russell, and Andrews, 2008). The pipes are composed of bedded volcaniclastic kimberlite, consisting of both kimberlite and mudstone xenoliths. Processed kimberlite used for this study was composed of olivine, calcite, quartz, garnet, lizardite, biotite, albite, saponite, and both framboidal and massive pyrite. Pyrite grains are mostly encapsulated in fragments of serpentine and aluminosilicate clays. The acid generating potential (AP) and neutralizing potential (NP) of the FPK were calculated following the Modified ABA test described by MEND (1991). The AP values are similar among the three grain size fractions, 5.3 - 7.8 kg CaCO<sub>3</sub> eq/t, however not all of the AP would be available due to the occurrence of some pyrite as locked particles in other mineral fragments. The NP of the samples are similar, 165 - 218 kg CaCO<sub>3</sub> eq/t, and far exceed the acid generating potentials (Table 1). Olivine and calcite were the primary neutralizing minerals.

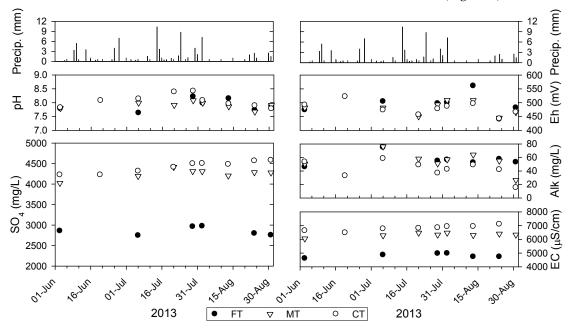
Sieve analyses using sieves +20, -20+35, -35+60, -60+100, -100+120, -120+200, -200+270 and -270 show the differences in grain size between the FPK in the three tanks and humidity cells (Table 1). The FPK from the FT and MT samples were similar with FT having slightly finer FPK, whereas the FPK from CT was coarser with a lower uniformity coefficient (Table 1).

Tank	d10	d60	Uniformity	Sulfur	NP	AP	NNP
			Coefficient	(%)	(kg CaCO₃ eq/t)	(kg CaCO₃ eq/t)	(kg CaCO₃ eq/t)
FT	0.07	0.39	5.8	0.23	218.4	5.3	213
MT	0.08	0.44	5.3	0.34	203.1	7.8	195
СТ	0.18	0.82	4.6	0.26	165.3	6.6	159

**Table 1** Grain size distribution, sulfur content, neutralization potential (NP), acid-generating potential (AP) and net neturalizing potential (NNP) for the tank and humidity cell experiments.

#### Processed kimberlite tanks

The pH from the tank drains were circumnetural to alkaline and showed little variation between tanks during the active period of June 1, 2013 to August 31, 2013 (Figure 3). The Eh and alkalinity trends were similar to that of pH, with all tanks exhibiting oxidized conditions and alkalinity typically >40 mg L<sup>-1</sup> (as CaCO<sub>3</sub>) (Figure 3), consistent with calculated NNP. Concentrations of SO<sub>4</sub> remained relatively constant for each tank but with the FT tank drain having much lower concentrations (Figure 3). The CT tank had slightly higher concentrations than the MT tank (Figure 3). The EC trends for the tanks mirror the SO<sub>4</sub> concentration trends with the highest concentration of dissolved SO<sub>4</sub> observed from the CT tank and the lowest from the FT tank (Figure 3).



**Figure3** Temporal variation of precipitation, pH, SO<sub>4</sub>, Eh, alkalinity and EC from the PK tanks between June 1, 2013 and August 31, 2013.

The pH in the SWSS from all three tanks remained circumneutral to alkaline at all depths and was slightly higher in the MT and CT tanks for 2013 than for 2012 (Figure 4), possibly as accumulated reaction products began to flush through the tanks. Similarly, alkalinity concentrations in all tanks at all depths remained similar for both 2012 and 2013 (Figure 4), consistent with calculated NP values and alkalinity concentrations measured in porewater from the unsaturated zone at the PK facility (Moncur and Smith, 2012). Dissolved sulfate concentration in PK slurry water from the endof-pipe ranged from 59 to 380 mg/L. Porewater concentrations of dissolved sulfate in the tanks approached 5000 mg L-1 and remained relatively constant between 2012 and 2013 sampling events (Figure 4). These elevated dissolved sulfate concentrations suggest that sulfide oxidation is likely occurring throughout the tanks (Lindsay et al., 2015). Depletions in O<sub>2</sub> in the FT and MT tanks were observed for 2012, but O<sub>2</sub> concentrations remained near atmospheric levels in 2013 and for both years in the CT tank (Figure 4). Volumetric water content (VWC) was typically higher in 2012 than in 2013 but varied with depth. The similar peaks and trends between sample years suggest the changes in VMC are not an artefact of sample timing dates (i.e. sampling during a particularly dry or wet time) and may suggest compositional differences by depths that affect wetting front propagation.

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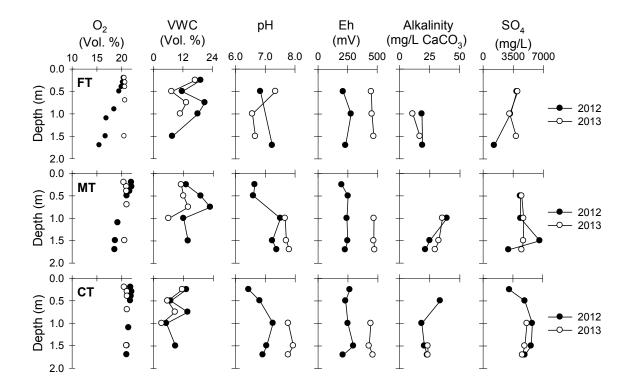


Figure 4 Depth profiles of oxygen gas, volumetric water content (VWC) and selected porewater chemistry from the PK tanks measured in 2012 and 2013.

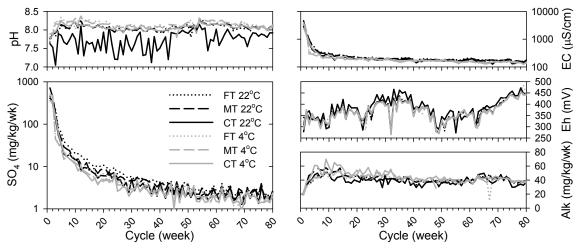
#### Humidity cells

The pH in all humidity cell charges remained circumneutral to alkaline for the duration of the experiment with pH values ranging from 7.1 to 8.4 (Figure 5). The cells in the cold room consistently exhibited higher pH compared to the corresponding cell at room temperature. The room temperature cell with CT material exhibited lower pH and erratic fluctuations compared to the other five cells, possibly as a result of preferential flow paths reducing contact and reaction time with the cell material. However, alkalinity measurements from the same cell did not exhibit the same erratic behavior. Alkalinity remained >60 mg/L (as CaCO<sub>3</sub>), for the duration of the experiment (Figure 5). Conditions within all cells remained oxidized with ORP ranging from 300 to 450 mV, consistent with the tank data.

Sulfate release rates were slightly higher for room temperature cells than for cells operated at 4°C, and in descending order, FT, MT and CT cells (Figure 5). Data from the first three weeks represent flushing of accumulated reaction products. Release rates from week 4 - 24 declined rapidly, and then declined at a more moderate rate from week 24 - 55. Sulfate release rates become more stable after week 55 until the end of the experiment at week 80 (Figure 5). Mass calculations suggest 77-85% of total sulfur and 73-81% of sulfides remain in the test charges after the 80 week experiment; however the decreasing rates suggest available sulfide surfaces are becoming limited by a reduction of available free grains, sulfide armoring or other limiting processes within the test cells.

Preliminary pH and alkalinity tank data are typically consistent with both the humidity cell experiments and the static tests which indicate that there is an excess of neutralization potential

over acid-generating potential. On-going monitoring of the tanks will determine if SO<sub>4</sub> release rates in the tanks follow the observed humidity cell trends of rapidly declining release rates despite >70% of measured sulfides remaining in the samples. Though not directly comparable, the difference between tanks in drain SO<sub>4</sub> concentration trends from humidity cell release rate trends suggest additional processes are occurring *in situ*. These processes need to be identified and evaluated for any scaling calculations.



**Figure 5** Temporal variation of pH, SO<sub>4</sub>, EC, Eh and alkalinity of leachate from the FT, MT and CT humidity cells at 22°C and 4°C.

#### CONCLUSION

Comparing humidity cell reaction rates to the *in situ* tank experiment results are preliminary because the low amount of precipitation and infiltration and short thaw season limits the release of reaction products from the tanks and products are expected to take more than one thaw year to flush from the tanks. On-going monitoring of the tank experiments will permit the calculation of an *in situ* sulfate release rate(s) that accounts for local climatic factors that affect sulfide oxidation. Tank release rates can then be compared to humidity cell release rates to determine the applicability of using the small-scale tests to predict long-term seepage quality from the PKC facility. Diavik will use the results from the tank and humidity cell experiments to predict and refine long-term seepage quality from the PKC facility.

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#### NOMENCLATURE

PK	Processed kimberlite
РКС	Processed kimberlite containment facility
FPK	Fine processed kimberlite
FT	Tank with fine FPK fraction
MT	Tank with medium FPK fraction
CT	Tank with coarse FPK fraction
SWSS	Soil water suction sampler
EC	Electrical conductivity

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