

**APPENDIX 4-V
LABORATORY INVESTIGATION OF
HEXAVALENT SELENIUM REMOVAL FROM
KERR WASTE ROCK LEACHATE WATER**



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Laboratory Investigation of Hexavalent Selenium Removal from Kerr Waste Rock Leachate Water

USING BIOTEQ'S SELEN-IX PROCESS AND BIOLOGICAL REDUCTION OF SELENIUM

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BioteQ Environmental Technologies Inc.
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1 Executive Summary

BioteQ's Selen-IX process was employed to remove selenium (Se) from Kerr waste rock leachate water. The proposed process includes Se(VI) removal from the leachate water using ion exchange (IX) during which a Se-rich regenerant is produced. The regenerant is further treated by biological reduction of selenium then recycled to the IX process, which allows for reduction in operating costs. The laboratory work demonstrated the capacity of Selen-IX to remove selenium from Kerr waste rock leachate water and confirmed the potential for low operating costs. The experiments were performed in three stages:

- Identifying the key parameters that affect selenium removal efficiency from Kerr waste rock leachate water using Selen-IX process.
- Maximizing the selenium removal efficiency through Selen-IX process and generating sufficient spent regenerant.
- Treating the spent regenerant and reducing selenite/selenate to solid elemental selenium.

The following conclusions were made based on the experimental results obtained in the laboratory:

- Key parameters were identified that affect the selenium removal efficiency of the Selen-IX process with respect to Kerr waste rock leachate water. Key parameters include regenerant and regenerant strength, as well as resin type, and flow rates, and temperature.
- Over 95% of selenium adsorbed onto resins was successfully stripped off by 2 to 3% solutions of Na_2SO_4 .
- The Selen-IX process was optimized so effluents with Se(VI) concentrations less than 1 ppb were achieved.
- The selectivity of the Se(VI) capture is very high. No other major constituents in the water (Al, Ca, Mg, or Mn) were removed. Nitrate is captured with selenium but was only present in trace quantities in the feed water tested. In the field the Kerr waste rock leachate water is expected to contain variable quantities of nitrate due to the variable environmental leaching rate of blasting residue. The impact of the change in nitrate concentration will be very limited on the Selen-IX process and may only increase the total bio-solids sludge production negligibly.
- Basic design parameters were obtained for preliminary design of a Selenium Treatment Plant.
- Upstream iron removal to the Selen-IX process was determined to be necessary.
- Proof of concept studies were completed on reducing selenium oxyanions to elemental selenium using BioteQ's bioreactor.

Advanced development is under way at BioteQ to test alternate physicochemical methods for selenate reduction. The methods are based on the physico-chemical Se(VI) reduction using a variety of chemical catalysts that accelerate the reduction of Se(VI). This will reduce the required equipment footprint compared to the biological system and produce selenium laden solids that are stable over a wide range of storage conditions including both anoxic and oxic environments commonly encountered in storage facilities.



2 Introduction

Rescan has been retained by Seabridge Gold to identify selenium removal technologies that will be required by the EIS. Rescan contacted BioteQ to investigate the selenium removal and inquired about the applicability of BioteQ's Selen-IX selenium removal process to treat a segregated surface run-off stream from the Kerr waste rock dump. BioteQ's approach to treatment of the stream from the Kerr waste rock dump is summarized briefly in the following.

Billions of tonnes of waste water are generated annually from active and inactive mine sites across Canada. If untreated, the drainage could seriously harm plants, wildlife, fish and consequently human beings. Waste water is generally treated for neutralizing acidity and removing suspended solids as well as metal. Among all contaminants, Se has received great attention recently, particularly due to the narrow window of concentration that exists between Se deficiency and toxicity. Se has chemical properties similar to that of sulfur, which makes its remediation challenging in presence of sulfur containing species. Se can be found in four oxidation states and in both organic and inorganic forms. Among these forms, selenite (SeO_3^{2-}) and selenate (SeO_4^{2-}) are highly soluble in water and do not normally form compounds with common metal cations. Both selenite and selenate are considered toxic since they are bioavailable. Selenite is more reactive than selenate and can be reduced to $\text{Se}(0)$, which is more favourable, environmentally.

Successful and economic removal of selenium to low concentrations in mine impacted water requires a high selectivity for selenite/selenate over sulphate. Potential benefits of BioteQ's Selen-IX selenium removal process are: Selen-IX selectivity for removal of selenium from streams containing sulphate and other ions, low capital cost due to reduction of required hydraulic capacity for selenium reduction due to the concentration factor from IX regeneration, low operating cost due to recycling of regenerant, waste minimization and modularity of the Selen-IX process. This document summarizes the corresponding laboratory test results and conclusions on the applicability of BioteQ's Selen-IX process to the run-off stream from the Kerr waste rock dump. The Kerr waste rock leachate water was treated to remove selenium in solution to below 1ppb from an initial concentration of >100ppb. The laboratory testing focused on the selenium capture and the selenium reduction.

3 Objectives

The objectives of the proposed test program include the following:

1. Assess the treatability of the wastewater stream:
 - a) Confirm the feasibility of using the Selen-IX process to meet Seabridge Gold discharge limits (1 ppb in the effluent for transfer to the water storage facility);
 - b) Confirm the anticipated final effluent water quality;
2. Confirm the feasibility of process options to reduce captured selenite/selenate to produce solid elemental selenium with recycle of regenerant chemicals:
 - a) Biological and non-biological reduction processes will be tested
 - b) Evaluate the quantity and stability of the solid selenium product
3. Confirm the deportment of non-selenium constituents in the selenium removal process; and,
4. Provide preliminary equipment sizing, reagent consumption and pricing information

4 Materials and Methods

All test work was done at 21-22°C unless otherwise expressly noted. Testing for total and dissolved metals as well as anions was done by ALS Global. Metals analysis was done by CCMS and ICP-OES and anion analysis was done by ion chromatography.

4.1 Feed Reception and Assay

The Kerr waste rock leachate was received on October 23rd 2012 in a 200L drum. The drum contents were mixed and a 120mL sample was taken and assayed for metal and anion composition.

Table 1 Anions as tested by ALS

Anions	Feed composition mg/L
Chloride (Cl)	17
Nitrate (as N)	0.25
Sulfate (SO4)	1140

Table 2 Total and dissolved metals as tested by ALS

Total Metals	Feed composition mg/L	Dissolved Metals	Feed composition mg/L
Aluminum (Al)-Total	31.6	Aluminum (Al)-Dissolved	28.6
Arsenic (As)-Total	0.0218	Arsenic (As)-Dissolved	0.0189
Barium (Ba)-Total	0.0882	Barium (Ba)-Dissolved	0.0799
Calcium (Ca)-Total	60.9	Calcium (Ca)-Dissolved	53.3
Cobalt (Co)-Total	0.418	Cobalt (Co)-Dissolved	0.382
Copper (Cu)-Total	21.0	Copper (Cu)-Dissolved	19.3
Iron (Fe)-Total	219	Iron (Fe)-Dissolved	198
Magnesium (Mg)-Total	22.3	Magnesium (Mg)-Dissolved	21.1
Manganese (Mn)-Total	14.3	Manganese (Mn)-Dissolved	13.0
Selenium (Se)-Total	0.115	Selenium (Se)-Dissolved	0.103
Silicon (Si)-Total	6.75	Silicon (Si)-Dissolved	6.36
Silver (Ag)-Total	0.00201	Silver (Ag)-Dissolved	0.00060
Sodium (Na)-Total	15.4	Sodium (Na)-Dissolved	14.2

There were some notable differences between the expected feed composition which was communicated to BioteQ and the feed which was received; the concentrations of nitrate, calcium and iron were lower than expected. The selenium concentration was in the right range and there were no major unexpected constituents in the water.

4.2 Ion Exchange Resin Selection

For the experiments in this section small IX columns were used to compare relative performance of 4 resins under maximal loading conditions. This allowed selection of the best resin based on a

combination of selenium selectivity and resin capacity. The small columns had the following characteristics:

- 1.8 cm Internal diameter
- 13 cm column height
- 35mL column volume
- 8 cm resin bed height
- 20 mL resin bed volume

The columns were set up as follows. Ion exchange resin was housed in the PVC column and the feed was pumped through the column from the top down. The spent feed was collected from the outlet at the base of the column. Wash water and regenerant solutions were pumped through in a similar fashion and collected separately.

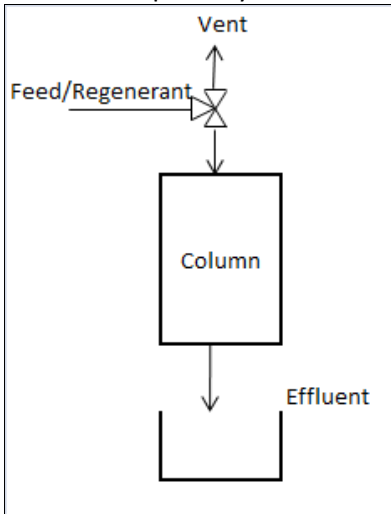


Figure 1 Small IX column schematic

The tests were conducted under the following conditions:

- Feed volume: 1L
- DI water wash volume: 0.1L
- Regenerant volume: 0.1L
- Regenerant: NaCl
- Resin in chloride form
- Flow Rates
 - Load & wash: 16BV/hour
 - Regenerate: 10 BV/hour

The hydraulic contact time of the regenerant with the resin was 6 minutes for each stage of regeneration. For a 20mL bed volume 1 BV/hour translates to 0.33mL/min; 16BV/hour is 5.33mL/min and 10BV/hour is 3.33mL/min. Four resins were tested during the course of stage 1 experimentation.

4.3 Resin Capacity Determination and Regeneration Characteristics

The apparatus used in stage 2 testing was the large IX column which has the following characteristics:

- 2.5 cm Internal diameter
- 130 cm column height
- 650 mL column volume
- 100 cm resin bed height
- 500 mL resin bed volume

The tests were conducted under the following conditions:

- Loading & rinse/wash rate: 21BV/hour & 25BV/hour
- Regeneration rate: 3BV/hour
- Regenerant: NaCl and Na₂SO₄
- Total bed volumes tested: 500mL & 1000mL

4.4 Ion Exchange Regenerant Treatment

The apparatus used for biological reduction of selenium consisted of a 26L metal bioreactor which is equipped with:

- A variable speed motor driving the impeller
- An external pressure gauge (0-10 inches H₂O)
- A external heater
- A pressure regulated nitrogen feed
- A pressure relief gas scrubber
- An inline pH probe
- An inline temperature probe

The bioreactor was inoculated with 6L of biosolution that was collected from an operating full scale BioteQ bioreactor. The bioreactor was kept under anaerobic conditions by constant positive pressure nitrogen feed. The bioreactor was monitored continuously to ensure the health of the bacteria population. The bioreactor was monitored, fed, and sampled on a daily basis. The biosolution was sampled daily for ammonia, sulphide, and selenium concentrations and weekly for lower and upper titration limits. The headspace was sampled daily with Dräger tubes to measure percent composition of hydrogen sulphide gas and carbon dioxide gas. Internal pH and temperature were measured daily from the inline monitors. From the above measurements the bioreactor consumption of: acetic acid, selenium, sulphur, sodium bicarbonate, and nutrients is determined.

The bioreactor is operated at:

- Positive pressure: 1-10 inches H₂O
- pH:7.5-8
- Temperature: 25-30°C
- Operational biosolution volume: 15L

5 Results and Discussion

5.1 Stage 1 (Ion Exchange Resin Selection)

Detailed Stage 1 experimental results that were reported to Rescan under a separate cover on November 7th, 2012 (Appendix 3) The findings of Stage 1 testing are summarized in the following:

Relatively large volumes of feed and long contact times were used for the stage 1 testing. The long contact times were accomplished by re-circulating the effluent through the system. The relative volume of feed to effluent was such that the composition of the effluent and that of the feed were very similar due to the excess of feed used. This was done to saturate the resin while keeping feed concentrations of all the absorbable species which would maximize any selectivity effects; as well as indicate relative resin capacity.

Three regenerant strengths are listed because each loaded column was regenerated with all three regenerant in order of increasing strength to determine which strength of regenerant would be needed to best regenerate the resin. This method of triple regeneration was predicated on the assumption that anything regenerated by one regenerant will also be regenerated by any stronger regenerant of the same type.

Table 3 % selenium, sulphate, and nitrate removal in stage 1 tests

Resin	Se removal %	SO4 removal %	NO3 removal %	Se capacity µg Se/mL resin
IX 1	53%	31%	58%	2.895
IX 2	55%	34%	62%	3.005
IX 3	74%	66%	62%	4.06
IX 4	61%	48%	62%	3.35

As seen in Table 3 on completion of stage 1 testing it was found that each resin that was tested captured appreciable quantities of selenium.

Table 4 Cumulative Regeneration Results for Stage 1 Tests

Resin	Weak Regenerant	Medium Regenerant	Strong Regenerant
	Cumulative Se(VI) regenerated	Cumulative Se(VI) regenerated	Cumulative Se(VI) regenerated
IX 1	82%	90%	97%
IX 2	90%	100%	103%
IX 3	41%	88%	93%
IX 4	59%	80%	84%

Three strengths of regenerant were used in succession to regenerate each loaded resin. The cumulative results of the regenerations can be seen in Table 4. As is apparent, different regenerant strengths are required to regenerate different resins.

Additionally, every resin could be regenerated and the loaded selenium can be removed from the resin by the regenerant. Further details on the results can be found in Appendix 3. The resin performance was differentiated by ease of regeneration, selectivity and capacity. The best resin was selected on the merits of high capacity, good selectivity for Se(VI) over NO₃ (and to a lesser degree SO₄), and good regeneration potential.

5.2 Stage 2 (Resin Loading and Regeneration Profile Determination)

Both the lead lag and the iron removal are included in the flowsheet (Appendix 1) and the equipment sizing and layout (Appendix 2). The resin selected in stage 1 was used in stage 2 and the total resin capacity for selenium, the breakthrough curve, and elution curve were determined. The total selenium capacity of the resin was found to be 6.7mg Se(VI)/L resin. The breakthrough curves depend on loading rate, and resin form (chloride or sulphate) as well as feed composition. Flow rates and regenerant strengths were tuned for maximal performance by the Selen-IX process.

Table 5 Feed and effluent concentrations for the Selen-IX process

	units	Feed	Effluent Grab
Selenium	µg/L	99.4	0.73
Nitrate	mg/L	0.23	<0.05
Sulphate	mg/L	1150	1090
Calcium	mg/L	58.3	59.0
Aluminum	mg/L	29.7	29.5
Manganese	mg/L	14.3	14.0
Magnesium	mg/L	22.6	22.6

The effluent grab sample taken at 7.5 BV (Table 5) shows that selenium is very effectively removed while none of the other major constituents (Al, Ca, Mg, Mn) were removed in any significant proportion. This demonstrates great selectivity of the selected resin. Under a separate cover submitted to Rescan on January 15th 2013 (Appendix 4) it was shown that single column experiments can result in selenium concentrations of less than 1ppb. However using the lead-lag operation setup it is possible to achieve even lower concentrations of selenium for a greater number of bed volumes of operation. This was done during the lead-lag test in the lab where selenium concentrations below the detection limit (0.5ppb) were achieved in the effluent.

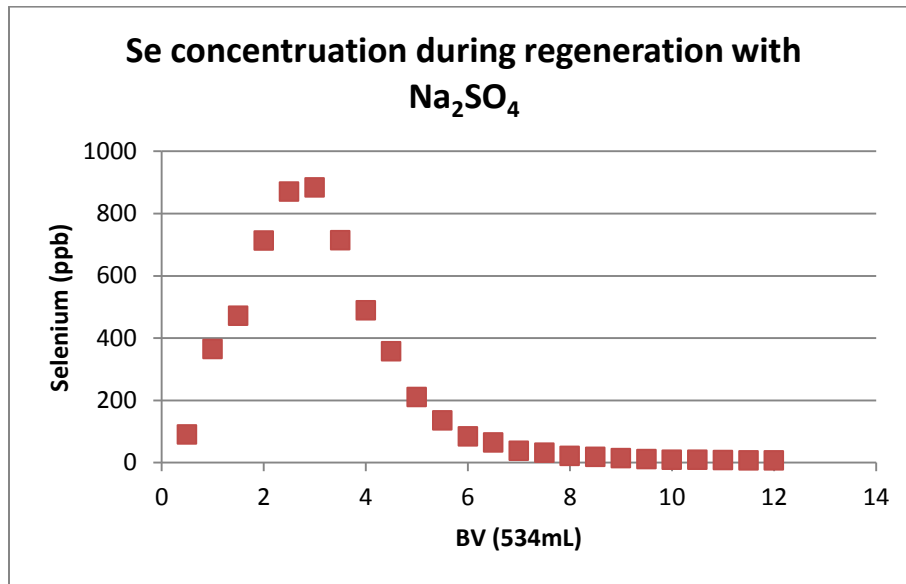


Figure 2 The concentration of Se(VI) in the spent regenerant from the lag column during regeneration with Na₂SO₄

The regeneration of the lag column, Figure 2 The concentration of Se(VI) in the spent regenerant from the lag column during regeneration with Na₂SO₄Figure 2, reports low peak selenium concentrations due to the fact that as a lag column it was not fully saturated with selenium. Under normal operation a lag column would be transitioned into lead operation to fully load it with selenium before regeneration. This would yield a much higher peak, and average, selenium concentration in the regenerant.

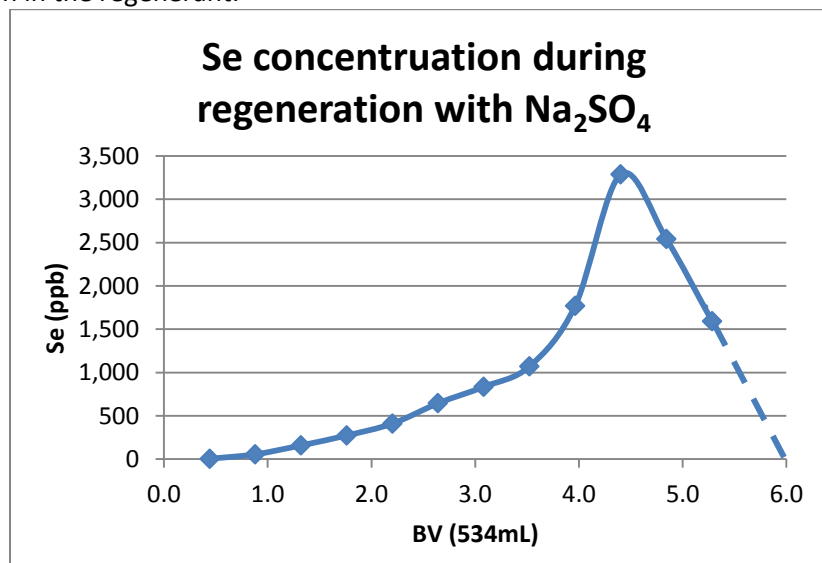


Figure 3 Concentration of Se(VI) in the spent regenerant from the lead column during regeneration with Na₂SO₄

During stage 2 testing it was determined that iron negatively impacted selenium removal and regeneration which led to the recommendation for the inclusion of an iron removal stage in the design up stream of the ion exchange process. Despite the impact of the iron on the efficacy of the system selenium removal was observed to be 98% with regeneration of 95% of the loaded

selenium using only 2-3% solutions of NaCl or Na₂SO₄. Selenium is effectively captured by the resin despite the high sulphate concentration present in the water.

The testing was done in a lead-lag configuration to allow for more selenium loading on the lead column. This increased selenium loading before regeneration allows for higher selenium concentrations in the regenerant and a greater concentration factor. The lag column acts as a polisher for the lead column effluent leading to more effective selenium removal and better regeneration characteristics.

Due to limited feed volume there was only enough feed to conduct one lead-lag column test. That test was conducted without iron removal so there was ~150 ppm Fe in the feed. Even under these adverse conditions after 7.5 BV the effluent selenium concentration was still only 0.73 ppb Se. Operating under iron free conditions with sufficient feed would provide the opportunity to further optimize the operating conditions and achieve even better selenium removal efficiency.

At the conclusion of the stage 2 testing the proposed process description is as follows. Feed water containing some suspended solids, up to 115 ppb of dissolved hexavalent selenium and 200 ppm of dissolved ferric iron enters the plant and reports to the feed tank, where it is mixed with caustic soda in order to raise the solution pH enough to remove the dissolved ferric iron in the form of solid ferric(III) oxyhydroxide. After reaction in the feed tank, the dilute slurry stream reports to the iron removal stage for solid-liquid separation, which serves a dual purpose with respect to the IX stage; avoiding interference of ferric iron with selenium removal and removing suspended solids that can reduce the efficiency or cause plugging of the IX columns. The ion exchange circuit also includes multimedia filtration where fine particulates are captured from the iron stage clarifier overflow. Ion exchange columns, operating in carousel configuration will be used to remove the selenium from the filtered solution. The resin will be regenerated using a sodium sulphate (Na₂SO₄) solution to strip selenium from the resin. Upon exiting the column the regenerant reports to the selenium reduction stage. This will be achieved by either biological reduction or physico-chemical methods. The selenium is removed as a solid product, and the regenerant solution is then recycled to the regenerant storage tank. Further details are available in "the proposed plant layout", a separate cover submitted to Rescan on November 26, 2012, and is also available in Appendix 2.

5.3 Stage 3 (Biological Reduction of Selenium)

The biological reduction of selenium in the bioreactor has been monitored for 20 days at the time of writing. Given the low concentrations of selenium and small absolute weights of selenium in the system it is impractical to quantify production of metallic selenium directly. Reduction of selenium in solution is measured by determining the removal of selenium from solution. Every day the bio-solution is sampled and then bioreactor is fed more selenium. Based on the carefully quantified volumes added and subtracted as well as the feed concentration the 'expected selenium concentration' is calculated; this is the concentration of selenium that would be observed in a sample of bio-solution if no selenium reduction took place. The 'measured selenium' is the selenium concentration that was actually measured in the bioreactor.

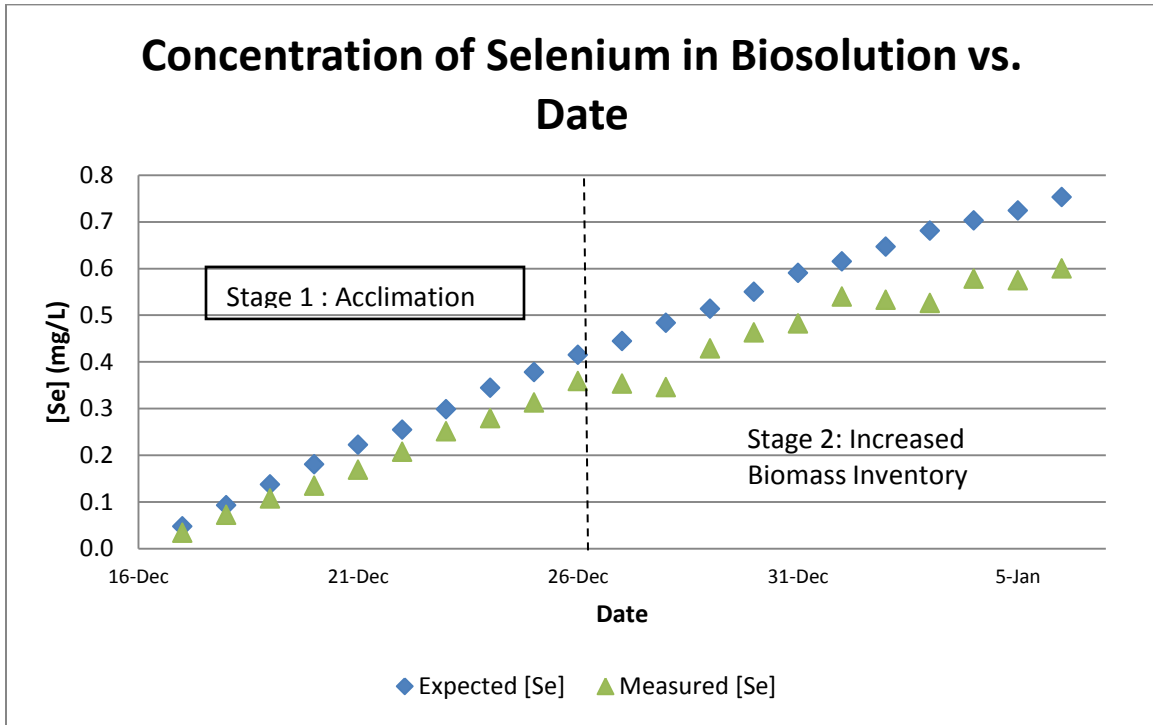


Figure 4 Expected selenium concentration, assuming no reduction, and measured selenium concentration vs. time

The expected concentration curve and the measured concentration curve (Figure 4) are diverging which shows that selenate is being reduced to metallic selenium in the bioreactor. The increasing rate of selenium removal is due to a combination of the increasing biomass inventory and the acclimation of the bacteria to metabolizing selenium. The rate of selenate reduction is proportional to the biomass inventory and therefore as the biomass in the bioreactor increases over the normal course of operations the selenium reduction increases. The bioreactor was inoculated and initially operated using elemental sulfur. At the beginning of the selenium reduction testing the bioreactor was switched to being fed selenium feedstock. The low initial selenium concentration, as expected, resulted in limited selenium reduction due to the bacteria not finding selenium as their major food source (Figure 4: stage 1). There is a selenium concentration threshold above which the bacteria adapts to selenium as their primary food source and the system approaches steady-state.

The initial results from the bioreactor for biological reduction of selenium demonstrate that bacteria consume selenium and remove it from the Kerr waste rock leachate water IX regenerant. Further investigation by BioteQ into easier simpler systems for reducing Se(VI) is ongoing. This work includes investigation into selenium reduction catalysts to accelerate the reduction as well as experimentation with physic-chemical reduction of Se(VI) to Se(IV) or Se(0). Alternative methods of selenium removal will be considered in comparison to the biological selenium reduction process. These methods strive to be superior to the biological system in both operational and economic considerations.

Table 6 Process battery limits for selenium removal process

Stream	Value
Feed Stream	216 m ³ /h
Caustic Soda	93 kg/h
Sludge to HDS	3.2 m ³ /h @ 3.8 wt.% solids
Make-up Na ₂ SO ₄	550 kg/h
Effluent	216 m ³ /h
Se Product	16.7 kg/month as elemental Se(VI) in sludge (sludge mass to be determined)

The battery limits (Table 6), with respect to the Se product and associated solids, were determined based on estimates made during stage 3 testing.

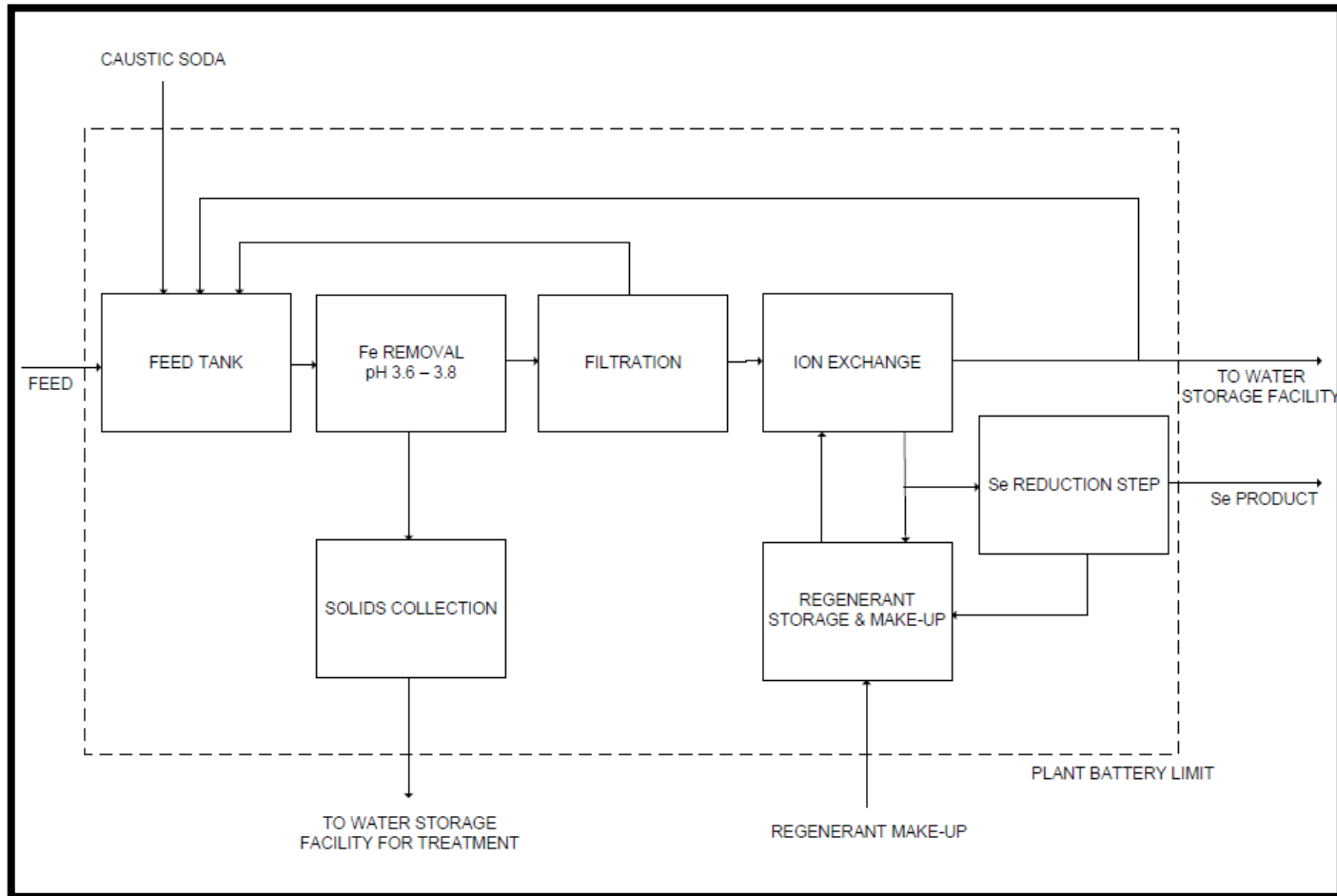
The preliminary estimate for sludge production for the biological selenium reduction and reduced selenium is 4.3 tons of sludge per year (11.8kg/day) and were arrived at as follows:

- 1) Based on battery limits selenium reduction will be 16.7 kg Se/month
- 2) 0.075kg biomass sludge will be produced per kg Se(VI) sludge
- 3) Sludge is estimated to be 5% solids
- 4) From 1), 2) and 3) wet weight sludge can be calculated from
 - a. $16.7 + 0.075 * 16.7 = 0.05(X)$
 - b. Where X is the wet weight sludge
 - c. Wet weight sludge = 4.3 tons /year
 - i. Assuming sludge density of 1g/cm³

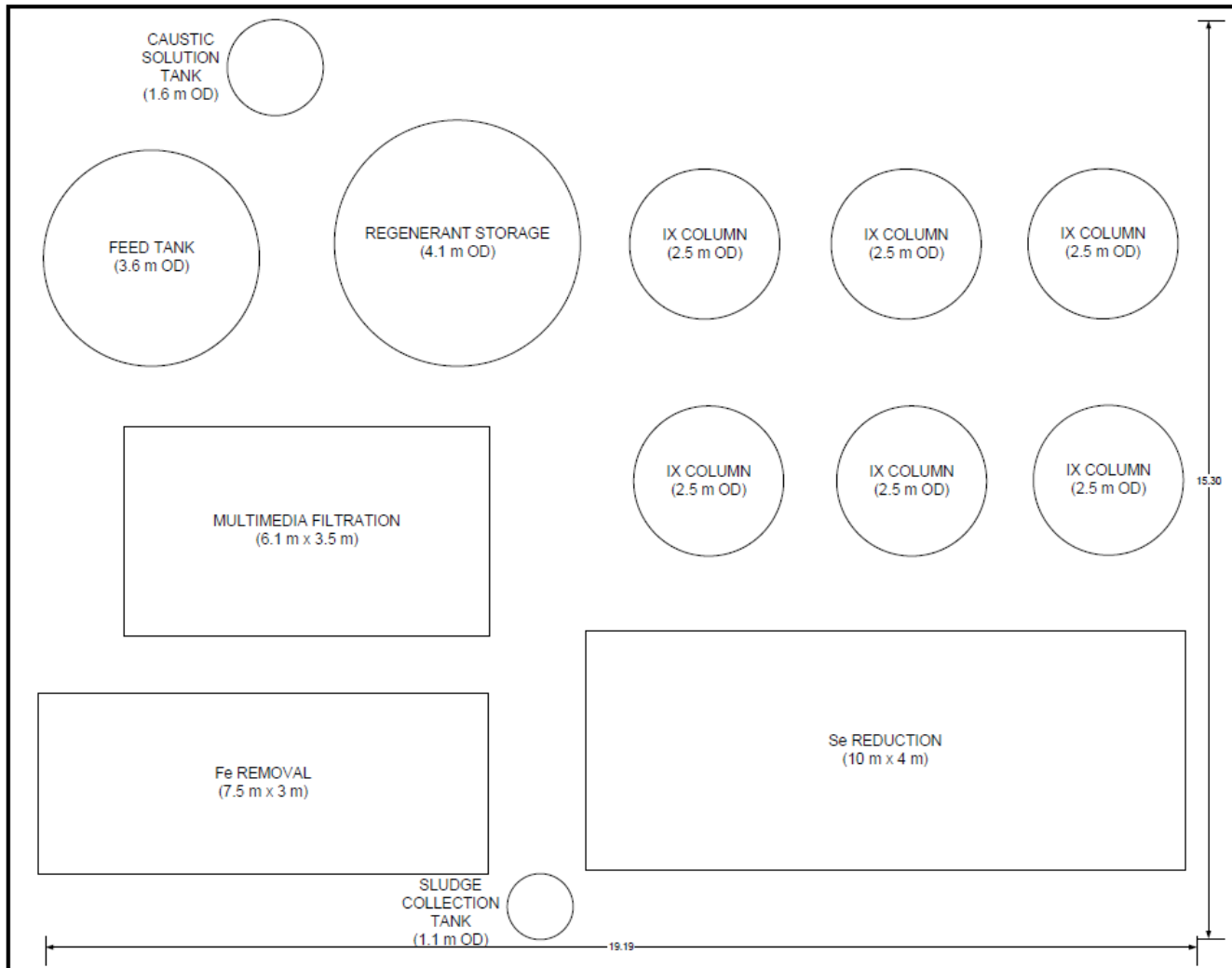
6 Conclusions

1. Removal of Fe upstream of Selen-IX is important to prevent deportment in the IX
2. The Selen-IX process proved to be effective in removing selenium to below 1ppb and successfully met the selenium concentration requirements.
3. The final effluent composition after the Selen-IX process has been established.
4. The proof of concept trial of biological reduction of selenium from the Kerr waste rock leachate water, in BioteQ's bioreactor, was successfully executed.
5. The projected quantity of the selenium biosludge product was outlined.
 - a. Based on the battery limits it was estimated that 4.3 tons per year (11.8 kg/day) of solid selenium laden biosludge would be produced.
6. The deportment of non-selenium constituents in the selenium removal process was determined to be minimal.
 - a. Sulphate is not removed by the proposed IX process and simply passes through the treatment plant.
 - b. Partial removal of nitrate was observed but nitrate was present only in trace quantities in the feed water. Quantities of nitrate are variable due to the environmental leaching rate of blasting residue. However, variation in nitrate concentration is not a concern to the Selen-IX process.
 - c. The other major constituents in the Kerr waste rock leachate water (Al, Ca, Mg, Mn) were not removed by the Selen-IX process.

Appendix 1: Flow Diagram



Appendix 2: Layout Diagram



Appendix 3 Stage 1 progress memo

Feed Characterization

- Table 7 summarizes the analytical results from Kerr waste rock leachate water and only shows results that were relevant and above detection limits.
- Table 4 summarizes the mole ratio of competing ions to selenium

Table 7 Feed Characterization

	Units	Kerr waste rock leachate water
Physical Tests		
Hardness (as CaCO ₃)	mg/L	220
pH	pH	2.64
Conductivity	mS	1.41
Anions and Nutrients		
Chloride (Cl)	mg/L	17
Fluoride (F)	mg/L	1.31
Nitrate (as N)	mg/L	0.25
Nitrite (as N)	mg/L	0.032
Sulfate (SO ₄)	mg/L	1140
Dissolved Metals		
Aluminum (Al)	mg/L	28.6
Arsenic (As)	mg/L	0.0189
Barium (Ba)	mg/L	0.0799
Beryllium (Be)	mg/L	0.0025
Cadmium (Cd)	mg/L	0.0364
Calcium (Ca)	mg/L	53.3
Chromium (Cr)	mg/L	0.0168
Cobalt (Co)	mg/L	0.382
Copper (Cu)	mg/L	19.3
Iron (Fe)	mg/L	198
Lead (Pb)	mg/L	0.0314
Lithium (Li)	mg/L	0.0127
Magnesium (Mg)	mg/L	21.1
Manganese (Mn)	mg/L	13.0
Molybdenum (Mo)	mg/L	0.0241
Nickel (Ni)	mg/L	0.162
Potassium (K)	mg/L	1.52
Selenium (Se)	mg/L	0.103
Silicon (Si)	mg/L	6.36
Sodium (Na)	mg/L	14.2
Strontium (Sr)	mg/L	0.623
Tin (Sn)	mg/L	0.0012
Titanium (Ti)	mg/L	0.667
Uranium (U)	mg/L	0.00198
Zinc (Zn)	mg/L	3.18

Table 8 Mole Ratio of Competing Ions to Selenium in Feed

SO ₄ /Se	mole ratio	9049
NO ₃ /Se	mole ratio	3.04

Treatability Test on the feed sample

- Four different anion IX resins were tested;
- 20 mL of resin was used in the small column setup
- The hydraulic contact time of the feed with the resin during loading was effectively 15 minutes over four cycles.
- The loading flow rate was 15 – 16 BV/hr
- There were three stages of regeneration. The three stages were conducted sequentially and were to investigate various recipes for resin regeneration;
- The hydraulic contact time of the regenerant and the resins were 6 minutes for each stage of regeneration.
- The regeneration flow rate was 10 BV/hr

Kerr waste rock leachate water Results

- From Table 9, IX resins 1 and 2 had higher selectivity for Se/SO₄ than IX resins 3 and 4;
- However IX resins 3 and 4 had better selectivity for Se/NO₃ and higher total resin capacity which resulted in a higher overall recovery of selenium.
- IX resin 3 had the best Se(VI) removal and the best Se(VI) capacity with a Se:SO₄ selectivity that was not much worse than the other IX resins. The vast majority of the selenium was regenerated by stage 2 regeneration.
- **IX resin 3 was selected for stage 2 testing**

Table 9 IX Loading Results

Resin	pH	Se mg/L	Se removal %	SO4 mg/L	SO4 removal %	NO3 mg/L	NO3 removal %	Se capacity µg Se/mL resin	Se/SO4 removed mol/mol	Se/NO3 removed mol/mol
-	-	-	-	-	-	-	-	-	-	-
Feed	2.69	0.109	-	1200	-	0.26	-	-	-	-
IX 1	2.57	0.0511	53%	828	31%	0.11	58%	2.895	0.00019	0.303
IX 2	2.59	0.0489	55%	792	34%	<0.10	62%	3.005	0.00018	0.295
IX 3	2.50	0.0278	74%	404	66%	<0.10	62%	4.06	0.00012	0.399
IX 4	2.60	0.0420	61%	628	48%	<0.10	62%	3.35	0.00014	0.329

Table 10 IX Cumulative Regeneration Results

Resin	Stage 1			Stage 2			Stage 3		
	Cumulative Se(VI) regenerated	Cumulative SO4 regenerated	Cumulative NO3 regenerated	Cumulative Se(VI) regenerated	Cumulative SO4 regenerated	Cumulative NO3 regenerated	Cumulative Se(VI) regenerated	Cumulative SO4 regenerated	Cumulative NO3 regenerated
IX 1	82%	94%	34%	90%	97%	147%	97%	100%	455%
IX 2	90%	99%	18%	100%	101%	54%	103%	102%	196%
IX 3	41%	54%	18%	88%	91%	54%	93%	93%	126%
IX 4	59%	77%	20%	80%	93%	68%	84%	94%	137%

Table 11 Relative Regeneration Results by Stage (colour coded)

Resin	Stage 1			Stage 2			Stage 3		
	Se(VI) regenerated	SO4 regenerated	NO3 regenerated	Se(VI) regenerated	SO4 regenerated	NO3 regenerated	Se(VI) regenerated	SO4 regenerated	NO3 regenerated
IX 1	82%	94%	34%	8%	3%	113%	7%	3%	307%
IX 2	90%	99%	18%	10%	1%	36%	4%	1%	142%
IX 3	41%	54%	18%	47%	37%	36%	5%	2%	72%
IX 4	59%	77%	20%	21%	16%	48%	4%	2%	69%

Table 11 summarizes regeneration results for each stage of regeneration relative to the total amount loaded (calculated based on difference in feed and spent feed concentrations) and displays it on a colour coded scale where 0% regeneration is Red, 100% regeneration is Green, and the maximum result is Yellow. Values between these three points are shades between red and green or green and yellow.

Appendix 4: Stage 2 progress memo

Subject: KSM PROJECT: SELENIUM REMOVAL FOR KERR WASTE WATER ROCK LEACHATE- STAGE 2 RESULTS SUMMARY

Summary

This memorandum summarizes the laboratory test results obtained by BioteQ during Stage 2 testing of the novel Ion Exchange (IX) process for the removal of hexavalent selenium from Kerr waste rock leachate with the intent to provide a preliminary design basis for water treatment that needs to be included in the EIA report prepared by Rescan for Kerr waste rock leachate.

Stage 2 Objectives

- Investigate the removal efficiency of hexavalent selenium using IX resin #3 (selected based on the results of Stage 1 tests) in a flow through packed bed column large enough to provide the following:
 - » Loading and regeneration profiles to confirm effluent chemistry and regenerant solution chemistry for Stage 3 investigation; and
 - » Preliminary sizing of the treatment plant and mass balances;
- Produce sufficient volume of the spent IX regenerant solution to allow testing of biological selenium reduction in Stage 3 of the test program.

Stage 2 Conclusions and Recommendations

1. The Stage 2 was successful and all of the objectives of Stage 2 were met in that the flow-through column tests using Resin #3 showed that:
 - a. the IX process, described in the design bases in Table 13 can produce effluent with Se(VI) concentration less than 1 ppb;
 - b. Over 95% of selenium adsorbed onto resins was successfully stripped off by 2 to 3% solutions of Na₂SO₄ and NaCl;
 - c. Basic design parameters were obtained for preliminary design; and
 - d. Sufficient volume of the spent regenerant was generated during the tests in order to allow Stage 3 of the test program to be initiated.
2. Iron present in the Kerr waste rock leachate appears to negatively interfere with the removal of selenium. Consequently, the proposed water treatment should include an iron removal step upstream of the IX process.

Stage 2 Methodology and Results

This document specifically discusses the IX process. The term 'effluent' refers to water that will be directed towards the Water Storage Facility (WSF) following the IX process.

Table 12 summary of feed and effluent grab samples for 2m packed bed IX test with resin in sulphate form

	units	Feed	Effluent Grab
Iron	mg/L	174	17
Selenium	µg/L	99.4	0.73
Nitrate	mg/L	0.23	<0.05
Sulphate	mg/L	1150	1090

Table 12 shows the effective uptake of selenium by contrasting the concentration in the feed and the effluent grab sample. The grab sample was taken after 7BV of loading. A grab sample is taken directly from the column effluent at a specific time, unlike a composite sample which is sampled from a homogenized collection of column effluent over time.

Notes on Stage 2 Test Methodology

- Samples of Kerr waste rock leachate were analyzed and the speciation confirmed that the water contained an average of 107 ppb (~100%) of selenium in the hexavalent form;
- Resin #3 was selected for Stage 2 testing based on the results of Stage 1 conducted on the Kerr waste rock leachate sample;
- 1" ID packed bed columns were used;
- Resin bed height was 1 m and the volume of resin in the column, i.e. Bed Volume (BV), was approximately 500 mL;
- 2 columns in series were used to investigate the effect of the resin bed height;
- Flowrate during resin loading was varied between 11 and 33 BV/hr;
- Resin regeneration was completed using 2 to 3% solutions of NaCl and Na₂SO₄; and
- The flowrate during regeneration was 3 BV/hr.
- The presentation of the Stage 2 column tests is based on the use of the dimensionless number of Bed Volumes (BV) instead of time to show concentration profiles for various solution constituents recorded over time during resin loading and regeneration. The use of the dimensionless BV facilitates the process scale up in that the concentration profiles expressed in BV will not change when the process is scaled up to the full scale plant.

Design Basis for Conceptual Design of the IX process

Table 13 summarizes the design basis of the IX water treatment which is based on the results from stage 1 & 2. This design basis is a preliminary conceptual design.

Table 13 Design Basis for Kerr waste rock leachate Treatment

Design Parameter	Value
Pre-treatment	Fe removal using lime or caustic
Ionic form of IX resin	Sulfate
Resin bed height (total)	3.6 m
Number of columns in series	Two (lead/lag configuration)
Resin capacity	6.7 mg Se(VI)/L of resin
Resin loading rate	21 bed volumes per hour
Resin regenerant	2-3% w/w Na ₂ SO ₄ solution
Regenerant volume	6 bed volumes per cycle
Regeneration bulk loading rate	3 bed volumes per hour

Table 14 Process Streams at Battery Limits

Stream	Value
Feed Stream	216 m ³ /h
Caustic Soda	93 kg/h
Sludge to HDS	3.2 m ³ /h @ 3.8 wt.% solids
Make-up Na ₂ SO ₄	550 kg/h
Effluent	216 m ³ /h
Se Product	16.7 kg/month as elemental Se(VI) in sludge (sludge mass to be determined)

Process Description

Feed water containing some suspended solids, up to 107 ppb of dissolved hexavalent selenium and 200 ppm of dissolved ferric iron enters the plant and reports to the feed tank, where it is mixed with a caustic soda solution. Caustic soda is added to the feed in order to raise the solution pH enough to remove the dissolved ferric iron. After reaction with caustic soda, ferric iron forms solid ferric(III) oxyhydroxide. After reaction in the feed tank, the dilute slurry stream reports to the iron removal stage. Although lime or potentially also limestone could be used instead of caustic for the pH adjustment, caustic was selected due to the fact that the feed water is expected to contain an elevated level of sulfate and the addition of a calcium based alkali would increase the risk of scaling in the IX process and probably also a higher load of suspended solids reporting to the iron removal stage which would negatively affect the sizing of the solid-liquid separation in the iron removal stage.

The iron removal stage has a dual purpose including 1) the removal of ferric iron at pH 3.6-3.8, and 2) removal of all other suspended solids upstream of the ion exchange process for the removal of hexavalent selenium. Ferric iron is removed upstream of the ion exchange circuit in order to avoid the negative interference of ferric iron with the removal of selenium by IX observed during lab testing including plugging caused by solids entrapment in the resin bed, the delay in selenium elution during resin regeneration, and possible reduction in resin capacity. Suspended solids need to be removed from the feed in order to avoid plugging of the IX resin beds. The iron removal stage consists of a ballasted flocculation unit, which allows solids to settle out of solution with minimum settling area hence equipment footprint requirement. The

sludge produced in the clarifier unit is pumped out to the sludge collection tank before ultimately reporting to the water storage facility. The clarified overflow reports to the ion exchange stage for selenium removal. The iron removal stage won't significantly impact other metal concentrations because of the low pH of operation, 3.6-3.8, and minimal co-precipitation effects.

The ion exchange circuit comprises multimedia filtration and ion exchange. The multimedia filters capture fine particulates from the iron removal stage clarifier overflow and are periodically back flushed using the filter discharge. The backflush reports to the plant feed tank. The ion exchange process operates with two parallel trains of packed bed columns. Each train is arranged into a 3 column carousel configuration where the plant feed passes through two columns in series while the third column in each train is undergoing regeneration. The two columns treating the plant feed in series are referred to as the lead and lag columns. When the lead column switches to regeneration, the lag column becomes the lead, and the freshly regenerated column becomes the lag. The treated effluent from the lag column is directed to the water storage facility. Once the breakthrough point has been reached (i.e. the point just before the selenium discharge criterion is reached) the resin must be regenerated. During regeneration, a sodium sulphate (Na_2SO_4) solution is fed to the column, which strips selenium from the resin into the regenerant solution. Upon exiting the column, a small portion of the spent regenerant solution is returned directly to the regenerant storage tank, while the remainder reports to the selenium reduction stage. After regeneration, the column is full of regenerant solution, which is displaced by the feed water when the column switches back to the loading phase. The solution initially exiting the column reports to the regenerant storage tank, while the last portion is directed to the feed tank. This is because the incoming feed will dilute some of the regenerant solution contained within the column, and it is important to prevent dilution of the regenerant solution, which would result in increased regenerant consumption.

The portion of the spent regenerant that does not report directly to the regenerant storage tank reports to the selenium reduction stage for selenium removal. This will be achieved by either biological reduction or physico- chemical methods. Various methods and catalysis for reduction of Se(VI) to Se(IV) or metallic Se(VI) are being perused. Catalysis is being investigated to shorten retention times. The selenium is removed as a solid product, and the regenerant solution is then recycled to the regenerant storage tank.

Because a portion of the regenerant is lost to the feed tank, Na_2SO_4 solution must be continuously added to the regenerant storage tank to make up the lost regenerant.

Please refer to the Block Flow Diagram in Appendix 1 for the process schematic. Table 13 shows the process streams at the plant battery limits.

Appendix 5: Stage 3 progress memo

Subject: KSM PROJECT: SELENATE REMOVAL FROM THE IX REGENERANT GENERATED DURING KERR WASTE ROCK LEACHATE WATER TREATMENT

As per BioteQ's initial proposal for the laboratory testwork submitted to Rescan and discussed with Seabridge Gold, and in conjunction with the results of the Stage 1 and 2 of the test program reported to Rescan under separate covers (Appendix 3 & 4), the Se(VI) removal process proposed for the treatment of the Kerr waste rock leachate water using IX will generate a stream concentrated in Se(VI) called the spent IX regenerant. In order to avoid costly disposal of the spent regenerant, the Stage 3 of the test program for the KSM project focuses on the proof of concept testing of the Se(VI) removal from the spent IX regenerant stream. The successful removal of Se(VI) from this stream would then allow a recycle of the treated stream back to the IX process thus minimizing the chemical cost associated with IX regeneration.

The state of the art Best Available Technology (BAT) for the removal of Se(VI) from waste water is biological reduction of selenate. The technology has been commercialized [1-2]. BioteQ's BioSulfide® process which is commercially used for large scale production of hydrogen sulphide uses the same bacteria that are used for selenate reduction [3]. Consequently, in Stage 3 of the project, samples of the selenate-bearing IX regenerant were fed to the bioreactor and the removal of selenium from this stream was monitored. The results of Stage 3 up until January 7th, 2013 will be presented in detail in the final report.

Further to the Stage 2 update and the preliminary plant lay-out submitted to Rescan on November 26, 2012, the equipment footprint required for the treatment of 216 m³/h of Kerr waste rock leachate water is calculated based on the commercial size biological selenate reduction system, i.e. approximate hydraulic loading rate of 11 gpm/ft² and hydraulic retention time of 30 minutes. The biomass yield through the process used by BioteQ for biological treatment of selenate is estimated to be approximately 0.075 kg VSS/kg of Se(VI) based on the composition of the solution provided for lab testing (230 ppb nitrate, 1150 ppm sulphate, 174 ppm iron and 107 ppb Se(VI)). Unthickened waste biomass sludge stream produced in the anaerobic bioreactors can be estimated to contain approximately 5% biosolids on a dry weight basis [4]. Based on the above and assuming the selenium reduction rate of 16.7 kg Se(VI)/month, a total of approximately 4.3 tons/year of sludge is projected to be produced by the bioreactor. This sludge needs to be disposed of or stored safely.

There is little information available on the long-term stability of the bioreactor sludge which may re-oxidize and release selenium back into solution which represents a risk and poses unanticipated difficulties. Furthermore, the calculated 4.3 tons of sludge is only a projection. The actual biomass production rate will depend on nitrate and nitrite loading in the Kerr waste rock leachate water, temperature, and electron donor fed into

the bioreactor. Another drawback worth mentioning is the sophisticated control systems required for operation of the bioreactor unit with all its ancillaries in order to ensure high plant availability and overall reliability, especially in cold and remote areas. Therefore, advanced development is under way at BioteQ to test alternate physicochemical methods for selenate reduction.

BioteQ 's approach to the physico-chemical Se(VI) reduction is to use a variety of chemical catalysts that accelerate the reduction of Se(VI) which is thermodynamically favored under certain process conditions. The physico-chemical catalytic method employs reactors and ancillary equipment that will occupy a smaller footprint than the biological system and produce selenium laden solids that are stable over a wide range of storage conditions including both anoxic and oxic environments commonly encountered in storage facilities. One of the main benefits of the physico-chemical approach is the elimination of the complex operability issues associated with the biological selenium reduction systems.

References

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- [2] Golder Associates Inc., Report # 08-1421-0034 Rev. 2, July 2009.
- [3] Private communication, Teck Ltd., 1998.
- [4] Wastewater Engineering Treatment and Reuse, 4th Edition, Metcalf & Eddy, Inc.