

Oil & Natural Gas Technology

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Quarterly Report January 2012 – March 2012 Post Retort, Pre Hydro-treat Upgrading of Shale Oil

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1. EXECUTIVE SUMMARY

In the 10th quarter considerable progress was made. Additional runs with methane and hydrogen were conducted on coker diesel. The tests results indicate that sulfur removal is independent of the reactor headspace gas. However, better yields are achieved with hydrogen as this gas seems to be a better radicals capping agent than methane. A new Athabasca bitumen sample from the Peace River region in Alberta was received and tested in our lab. The composition of this feedstock is similar to the previously tested materials from the Mc. Kay River and Cold Lake areas. Our technology was able to upgrade this oil from an API of 8 to 20, removing 95% of the sulfur, while keeping the process volumetric yield over 90%. Comparison of Simulation Distillation results have shown a reduction of the residual fraction (>524°C) content of as received oil from 51% down to 19% in the upgraded material.

Meanwhile the electrolysis process is still looking very encouraging. A solids pre-treatment step, to improve the efficiency of the extraction of sodium sulfide by an organic solvent, has been developed and tested during this reporting period. The thermal pre-treatment step converts the hydrocarbons in the solids to an insoluble material (e.g. coke). The long term test cell logged 5694 hours of operation with Na₂S₄ at a current density of 95 mA/cm². Unfortunately, the cell failed after loss of temperature control as a result of a power outage. Post-mortem analysis of the NaSICON membrane revealed little or no degradation of both the cathode and anode side surfaces. A new long term test cell was started in February. A total of 1020 hours of operation have been logged so far. The performance of the cell matches very closely the previous long term cell.

2. PROGRESS, RESULTS AND DISCUSSION

2.1 Task 1.0 -- Project Management Plan

The PMP was updated within 30 days and submitted to the Project Manager (Quarter 1).

2.2 Task 2.0 -- Upgrading Development

It is explained in detail in 6th Quarterly report.

2.3 Task 3.0 -- Electrolysis Development

It is explained in detail in 6th Quarterly report.

2.4 Task 4.0 -- Analysis

It is explained in detail in 6th Quarterly report.

Budget Period 2

2.5 Task 5.0 – Upgrading Development

This task is related to developing the process of treating shale oil, or heavy oil at elevated temperature and pressure in the presence of an alkali metal, either sodium or lithium and also a hydrogen source, either hydrogen gas or methane (natural gas) to form an oil stream with reduced levels of sulfur, nitrogen and heavy metals and also in the process reducing the viscosity and increasing the API gravity. The object here is to determine the impact of various reaction parameters on product quality.

2.5.1 Subtask 5.1 – Analytical Capability

Analytical laboratory set up

There are no major changes in analytical capability.

2.5.2 Subtask 5.2: Upgrading Reactor and Separation setup

The set up was modified to measure inlet and outlet gas flow rates so that precise mass balance can be established. The modified reactor is shown in Figure 1 below.

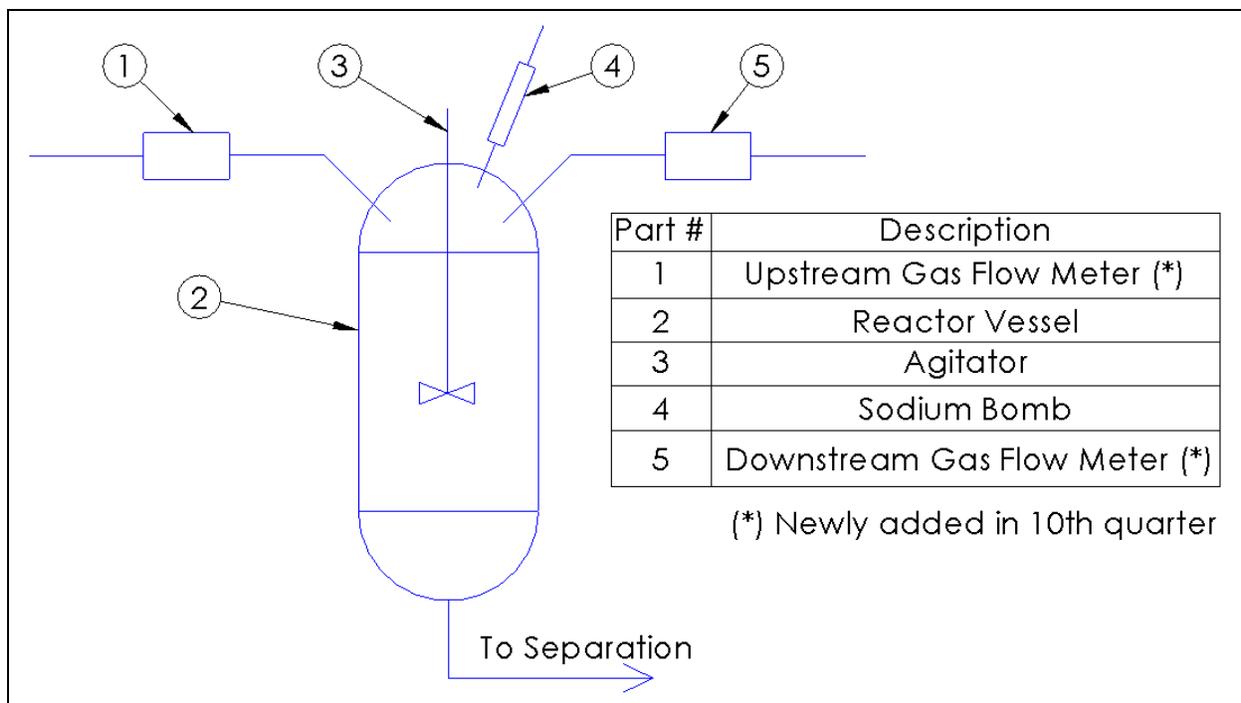


Figure 1: Reactor set up with added accessories

Results and Discussions

Additional experiments were performed with Bitumen and Coker Diesel as feedstocks. The bitumen specifications are shown in Table 1 below.

Table 1: Specifications of Bitumen

C	H	N	S	API	TAN	Ni	V	Fe
83.7	10.03	0.4	5.1	7.97	5.2	77	213	3

The various different runs are listed in Table 2 below. The results of the runs are shown in Table 3. As observed from Table 3, upto 99.6% sulfur was removed from the bitumen (Run 17) in presence of hydrogen. API of the upgraded product ranged from 14.5 to 21.4. The liquid fraction mass yield ranges from 64% to 89%. The hydrogen consumption numbers ranged from 270 SCF/Bbl to 400 SCF/Bbl. These hydrogen consumptions numbers are far lower compared to more than 1000 SCF/Bbl required for catalytic

Table 2: List of upgrading experiments for Bitumen

Run ID	Gas	Actual Na/ Theoretical Na	Reaction Time (Min)	Reactor Pressure (Psig)	Reactor Temp. (C)
B11	H2	0.98	122	1364	381
B12	H2	1.10	121	1179	390
B13	H2	1.15	121	1258	390
B14	H2	1.08	119	1211	391
B15	H2	1.03	120	1174	391
B16	H2	1.19	117	1251	390
B17	H2	1.25	125	1283	390

Table 3: Results of bitumen upgrading

Run ID	Sulfur moved	Re-	Nitrogen Removed	Liquid Fraction Mass Yield	API	Hydrogen Consumption	Light ends (C1-C6) Formed
	%		%	%		scf/barrel	scf/barrel
B11	93.8%	-	-	82.9%	15.6	270	57
B12	82.4%		8.8%	82.4%	14.5	364	91
B13	99.8%		90.2%	67.7%	25.0	345	147
B14	85.0%		-	84.5%	14.6	312	61
B15	91.2%		8.2%	89.0%	15.0	361	55
B16	96.7%		26.3%	71.7%	17.5	384	161
B17	99.6%		64.5%	64.1%	21.4	407	171

Sulfur Removal from Coker Diesel

Coker diesel was treated in the upgrading reactor with Nitrogen and hydrogen as cover gases respectively. The initial coker diesel has a sulfur content of 1.8%. The experiments were performed at 280 C and 1300 psig gas pressure. Table 4 shows the detailed results. The results show upto 97.3% sulfur removal using methane as a cover gas. The liquid fraction yield was in the range of 89 – 96.4%. These results are very encouraging.

Table 4: Results of Coker diesel desulfurization

Run ID	Gas	Actual Na/ Theoretical Na	Sulfur Removed (%)	Liquid Fraction Yield (%)	VOL	API
CR7	H2	1.05	84.9	96.4		33.9
CR8	CH4	1.05	85.9	90.2		34.4
CR9	H2	1.50	96.7	96.4		35.7
CR10	CH4	1.50	97.3	89.7		36.0

Schedule

The progress of Task 2 is as per schedule.

2.5.3 Subtask 5.3: Continuous Reactor Set Up

The new 1.8L reactor was uncrated and moved outside of the lab where it will be installed. This is a scaled up version of the smaller batch reactor but is floor standing with a pneumatic vessel lift and includes a bottom drain for the continuous operation. Schematics for the gas and liquid plumbing have been created and most of the required items to operate this as continuous reactor have been ordered with about 60% received. Oil charging pumps, several Swagelok fittings and valves still need to be specified and purchased. Figure 2 shows the picture of continuous reactor.



Figure 2: New reactor for continuous operation

2.5.4 Subtask 5.4: Space requirements for the continuous reactor operation

Due to space constraints in the current upgrading lab it was necessary to procure a larger space for this reactor and its associated processes. This space needed to be on the ground floor to facilitate replenishing the raw materials in large containers and of sufficient size to accommodate future growth. Two adjacent laboratories were identified and selected and initial floor plans were made. The reactor lab is currently being remodeled. Please see below for floor plan. Figure 3 shows the floor plan for the operation of continuous reactor.

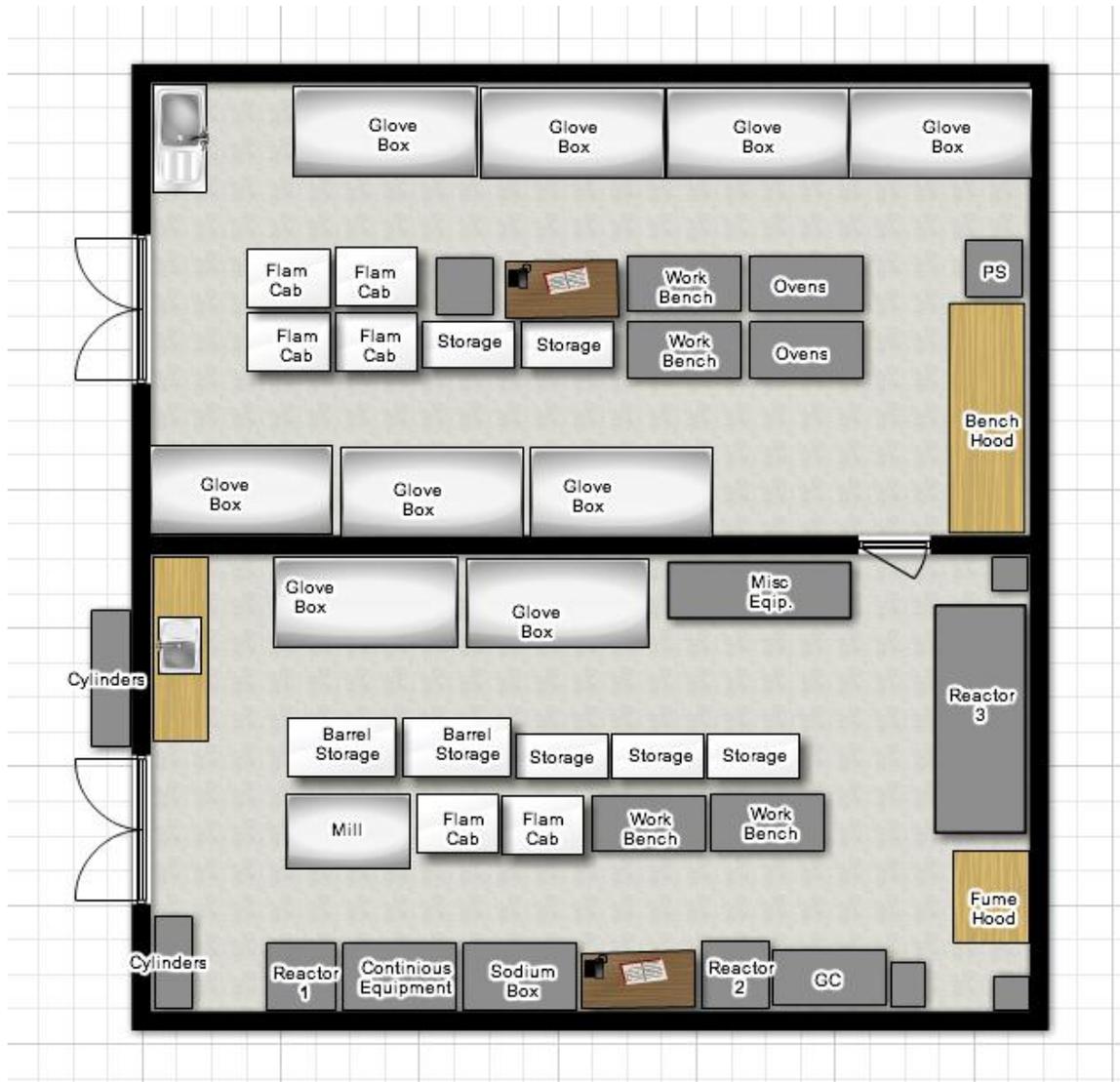


Figure 3: Floor plan for the operation of continuous reactor

2.6 Task 6.0 - Electrolysis development

To reduce the overall cost of the upgrading process, an electrolysis process will be developed to regenerate sodium or lithium from the respective polysulfide. The process will feature ceramic ion conductive membranes developed at Ceramatec. The energy cost to regenerate the alkali metals from the polysulfide is expected to be about half that of producing the metals from their respective chlorides.

2.6.1 Subtask 6.1 – Membrane fabrication

The Recipient shall fabricate and characterize sodium conductive and lithium conductive membranes.

2.6.2 Subtask 6.2 – Seal testing

Ceramatec shall evaluate various seal approaches for compatibility with the alkali metal and the metal polysulfide at various temperatures.

2.6.3 Subtask 6.3 – Cell design and set up

Ceramatec shall design benchtop cells for two types of operation, one where the alkali metal is molten and one where it plates onto a current collector. Reactors and catholyte transfer means will be provided to prepare alkali metal sulfide of differing composition and transfer to the cell. The cells will be designed to accommodate multiple reference electrodes, operate at various elevated temperatures. The cells will have features designed to facilitate sulfur removal and be designed to operate within a dry enclosure.

2.6.4 Subtask 6.4 – Cell operation

The Recipient shall operate cells under various conditions including variation of the current density, electrode gap, temperature, electrolyte, polysulfide order, and alkali metal. Current will be measured as a function of applied voltage. Periodically cell operation will be interrupted and cell contents analyzed to determine current efficiency. In Phase 1 the alkali metal polysulfide will be synthesized from alkali metal and sulfur and will not contain appreciable impurities which may flow through from an actual upgrading process as will occur in Phase 2.

Long term test cell results

Na Recovery Cell 20120504 completed 5694 hours (237 days) of continuous operation during this quarter. Table 5 and Table 6 summarize the properties of the cells under test and their overall performance parameters. Unfortunately, the NaSICON membrane failed after loss of temperature control in the anolyte solution as a result of a power outage. The end result was that DC current was applied to the cell as sodium started to freeze on the cathode side of the membrane. The failure occurred due to cracking caused by the formation sodium metal dendrites on the surface of the membrane. A new system interlock, to prevent that DC current is applied to the

cell below a settable anolyte temperature, has been implemented. Analysis of the failed NaSICON disk showed that the active area of the membrane was significantly smaller (1.13 cm² vs. 1.79 cm²) than originally thought because of the larger area taken by the glass seal on the cathode side of the cell. As a result, the cell was operated at an estimated current density of 95 mA/cm² as opposed to the 60 mA/cm² documented in previous quarterly reports. Figure 4 shows the total operating voltage at 95 mA/cm² and the cell's open circuit voltage throughout the life of the cell. As reported before the anolyte needed to be periodically replaced (every 1000 hours, approximately) due to the steady increase of the operating voltage caused by the aging of the solvent itself. Although sodium tetra sulfide (Na₂S₄) was used as the preferred polysulfide for most of the testing time sodium sulfide (Na₂S) was tested for 583 hours (between 5045 h and 5628 h in Figure 4). The results of the test with Na₂S were found to be in general very positive. However, over time we discovered the formation of a solid precipitate that tended to accumulate on the anode electrode surface and on the NaSICON membrane. This caused the overall cell voltage to increase due to higher electrode contact resistance. The cell voltage would return to its normal lower value once the solids were removed from the anode surfaces. We believe that the presence of undesirable impurities in the sodium sulfide raw material, which get oxidized and compete with Na₂S during cell operation, is the cause of this unexpected problem. Sodium tetra sulfide was used again in the last part of the cell testing. A detailed post-mortem analysis of the NaSICON membrane was conducted. SEM and EDS surface analysis (see Figure 5) of both anode and cathode sides, as well as of cross sectional cuts have shown very little damage to the membrane after almost 5700 hours of operation. No significant amounts of impurities have been detected in the samples analyzed with their elemental atomic composition virtually identical to that of pristine NaSICON. Moreover, the X-Ray Diffraction scans showed no change in the crystalline structure of the material. Figure 6 displays the XRD profiles of the cathode and anode sides of the long term tested membrane, as compared to the profile of a brand new membrane. The main conclusion of the test is that we have proved excellent long term performance of the NaSICON material with little or no corrosion against molten sodium and sodium sulfide dissolved in an organic solvent.

A new long term sodium recovery cell (*Na Recovery Cell 20120213*) has been started in February. The properties of the cell are listed in Table 6. A total of 1020 hours of operation have been logged so far. Figure 7 shows the total operating voltage and the cell open circuit potential versus total elapsed time for the cell.

Table 5: Summary of sodium recovery test cells run in long-term mode during reporting mode

Cell ID#	Anolyte	Anode Electrode	Membrane	Catholyte	Cathode Electrode	Seal Type	Operating Conditions
Na Recovery 20110504	Na ₂ S ₄ in MF Na ₂ S in MF	Platinized Ti mesh (1.1" diam.)	NaSICON GY (1 mm thick, 1.13 cm ² active area)	Molten Na	Molten Na, Ti rod current collector	Silica-Boria Glass	Temperature=130C Anolyte agitation Cte Current=95 mA/cm ²
Na Recovery 20120213	Na ₂ S ₄ in MF	Platinized Ti mesh (1.1" diam.)	NaSICON GY (1 mm thick, 1.67 cm ² active area)	Molten Na	Molten Na, Ti rod current collector	Silica-Boria Glass	Temperature=130C Anolyte agitation Cte Current=60 mA/cm ²

Table 6: Summary of performance of sodium recovery test cells run in long-term

Cell ID	Total Run Time	Current Density	Avg. Voltage	Avg. OCV	Status
	Hours (days)	mA/cm^2	V	V	
Na Recovery Cell 20110504	5694* (237*)	95	3.11	2.20	Membrane failure due to loss of heat after power outage
Na Recovery Cell 20120213	1020* (42.5*)	60	3.26	2.15	Cell still in operation

* Total time includes short time periods to measure the cell OCV

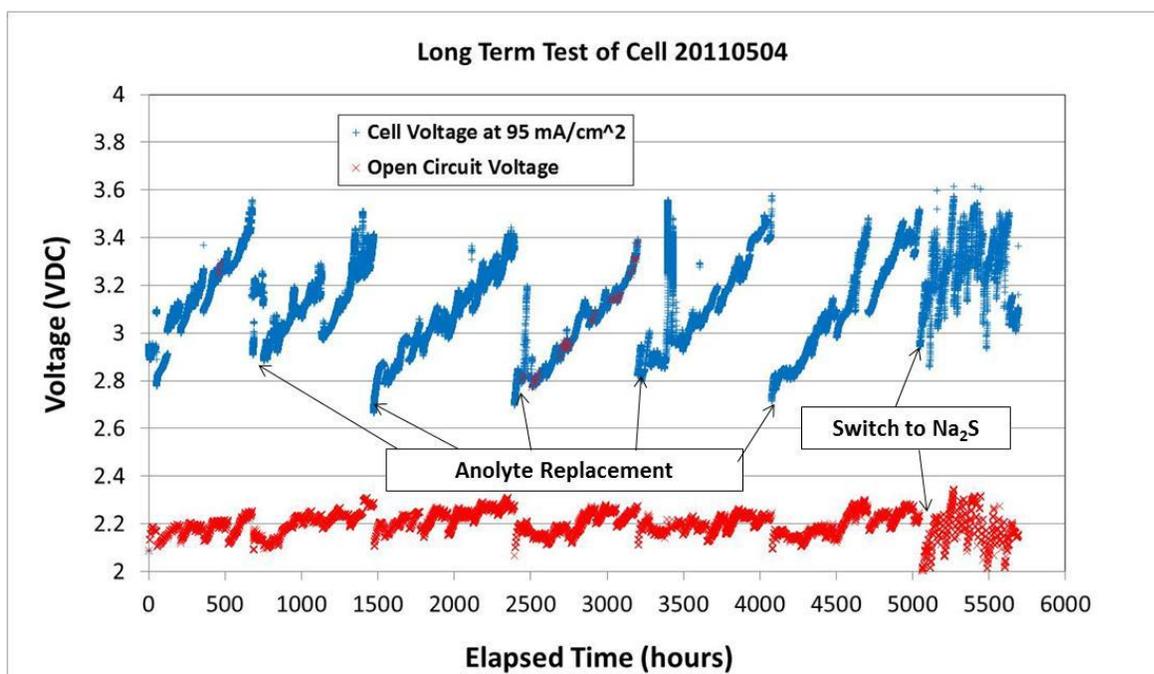


Figure 4: Cell voltage at 95 mA/cm^2 and Open Circuit Voltage (OCV) versus run time for Na Recovery Cell 20110504. Run time includes time periods for OCV measurements (no DC current applied).

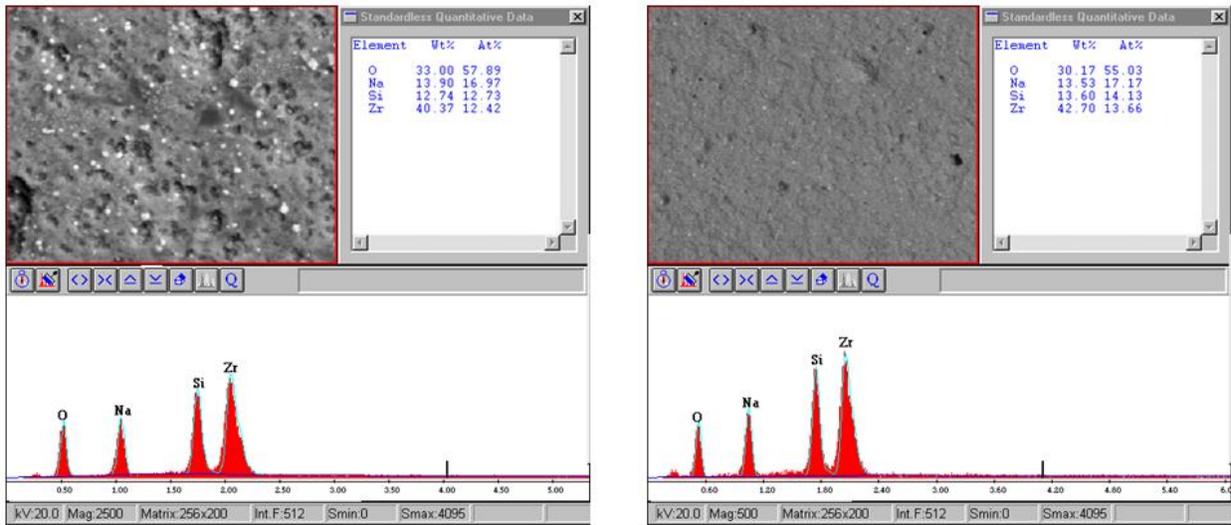


Figure 5: SEM and EDS analysis of the cathode (A) and anode (B) sides of the membrane recovered from *Na Recovery Cell 20110504*

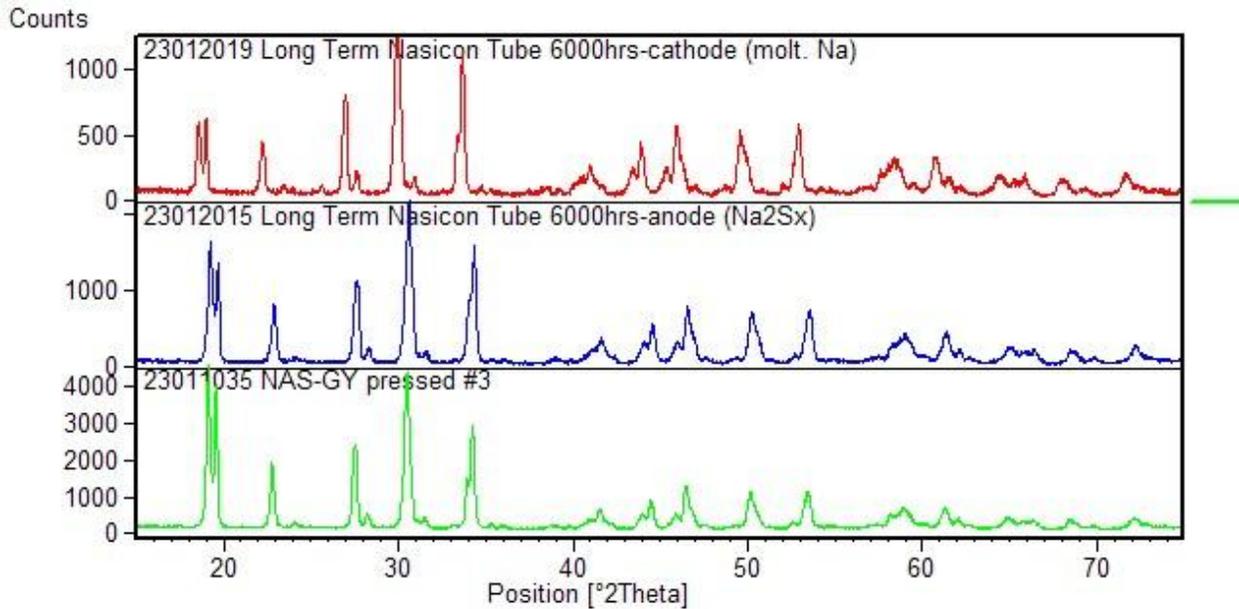


Figure 6: Comparison of X-Ray Diffraction (XRD) scans of the cathode and anode sides of the NaSICON membrane, recovered from *Na Recovery Cell 20110504*, with pristine NaSICON material.

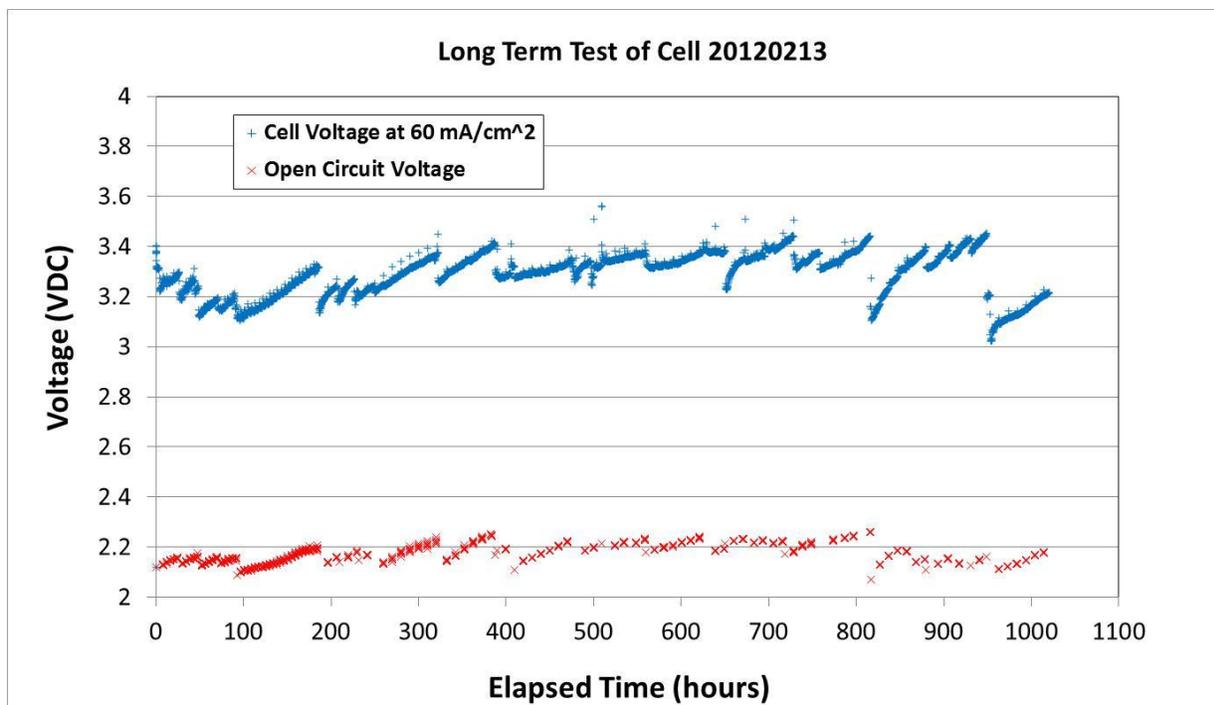


Figure 7: Cell voltage at 60 mA/cm² and Open Circuit Voltage (OCV) versus run time for *Na Recovery Cell 20120213*. Run time includes time periods for OCV measurements (no DC current applied).

Sodium recovery test cells results using solids from processed oil samples:

A solids pretreatment step, to improve the efficiency of the extraction of sodium sulfide by an organic solvent, has been developed and tested during this reporting period. The pretreatment step converts the hydrocarbons in the solids to an insoluble material in the organic solvent (e.g. coke). The schematic in Figure 8 shows the different steps needed to process the separated solids from the upgrading reactor to the anolyte solution. Sodium metal is recovered in the electrolysis cells by electrolyzing the sodium sulfide dissolved in the anolyte.

Solids obtained from two different upgrading runs (with H₂ and CH₄, respectively), using Athabasca bitumen as feedstock material, were used to test two Na recovery cells. Table 7 summarizes the properties of the cells. Figure 9 shows the current density vs. cell voltage plot for cell *Na Recovery B5 20120113* prior to the start of the test. As it can be observed in the plot, the total cell voltage, at the optimum commercial current density of 65 mA/cm² and cell temperature of 130°C, was equal to 2.95 VDC. The cell was run for a total of 67.5 hours in batch mode until most of the sodium was recovered. The cell was operated at 60 mA/cm² for 49 hours, and at 30 mA/cm² for the remaining 18.5 hours of the test. Figure 10 shows the total cell voltage measured during the test. In Figure 11, we have plotted the two components of the total cell voltage (the over potential and the Nernst potential) for the testing period at 60 mA/cm². The Nernst potential is the thermodynamic decomposition potential of the sodium sulfide/sodium polysulfides, which is mainly a function of the cell temperature and the sodium polysulfide concentration. The plot shows that it increases linearly with elapsed time. This is a consequence of the fact that higher

order polysulfides (x in Na_2S_x increases with time) are formed, which have higher oxidation potentials. On the other hand, the cell over potential represents the ohmic potential losses through the anolyte, NaSICON membrane, electrodes, etc. The magnitude of the over potential is directly proportional to the current density. Figure 11 shows that the over potential remained basically constant at around 1 volt throughout the test. This is a clear evidence that no cell deactivation occurred during the test. Figure 12 displays pictures of the anolyte solution from cell *Na Recovery B5_20120113* before and after the test. The post-test anolyte solution appears clear without any solids precipitate. The darker yellow coloration of the anolyte is a sign of the presence of sodium polysulfides in solution, as opposed to the less colored starting liquid which is essentially sodium sulfide (Na_2S).

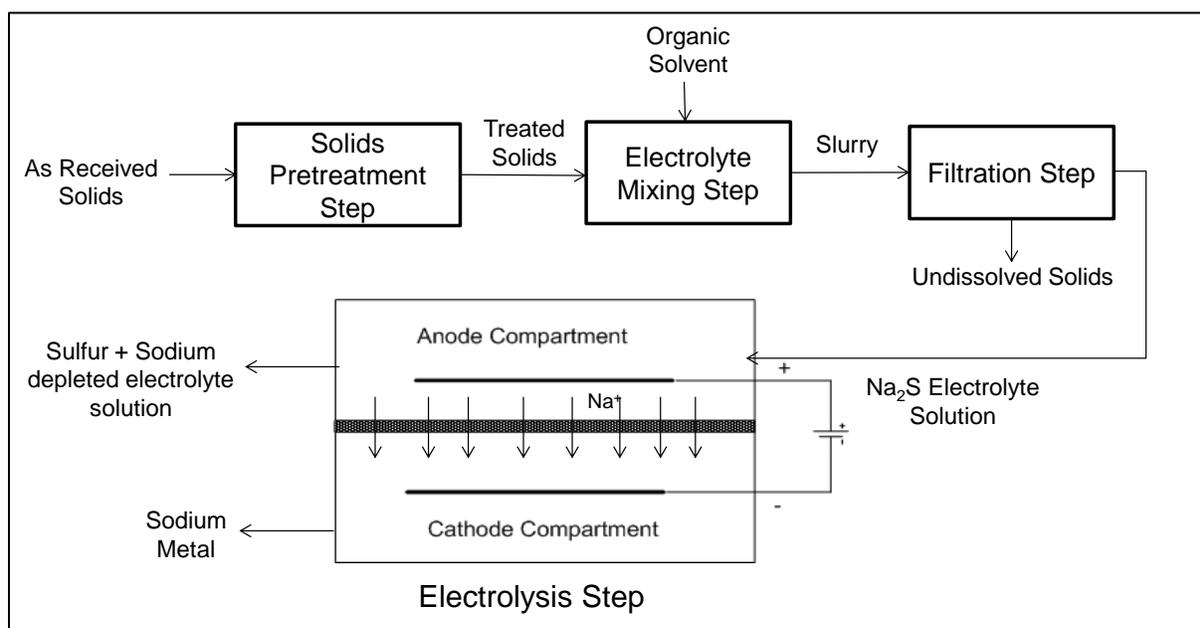


Figure 8: Solids processing and electrolysis schematic

Table 7: Sodium recovery test cells, using pre-treated solids from Athabasca Bitumen runs, assembled and tested during the reporting period

Cell ID#	Anolyte	Anode Electrode	Membrane	Catholyte	Cathode Electrode	Cell Operating Conditions
Na Recovery B5 20120113	242g of 4.2 wt% Pre-treated BS5 Solids (Athabasca Bitumen run with H2) in MF	Platinized Ti mesh (1.1" diam.) 3 mm from membrane	NaSICON GY, Pressed Disk (1 mm thick) Active area=1.8 cm ²	Molten Na	Molten Na, Ti rod current collector	Temperature=130C Cte Current= 60 and 30 mA/cm ²
Na Recovery BS6 20120113	300 g of 2.4 wt% Pre-treated BS6 Solids (Athabasca Bitumen run with CH4) in MF	Platinized Ti mesh (1.1" diam.) 3 mm from membrane	NaSICON GY, Pressed Disk (1 mm thick) Active area=1.7 cm ²	Molten Na	Molten Na, Ti rod current collector	Temperature=130C Cte Current=60, 30, and 20 mA/cm ²

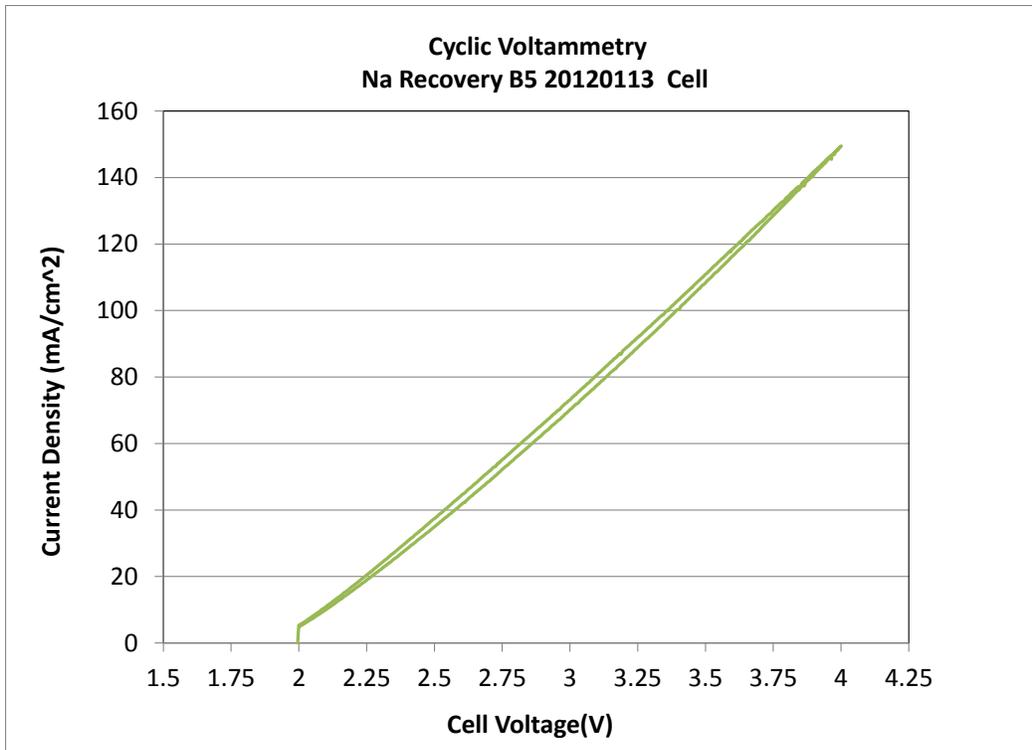


Figure 9: Current density vs. cell voltage for sodium recovery test cell *Na Recovery B5_20120113* prior to test

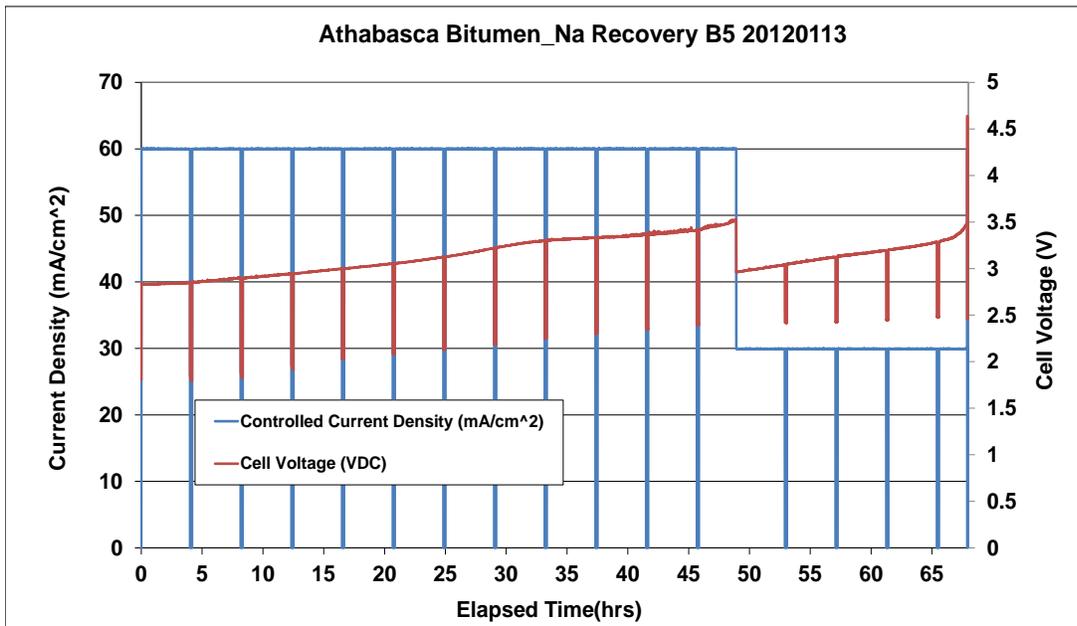


Figure 10: Cell voltage and current density versus elapsed time for sodium recovery test cell *Na Recovery B5_20120113*

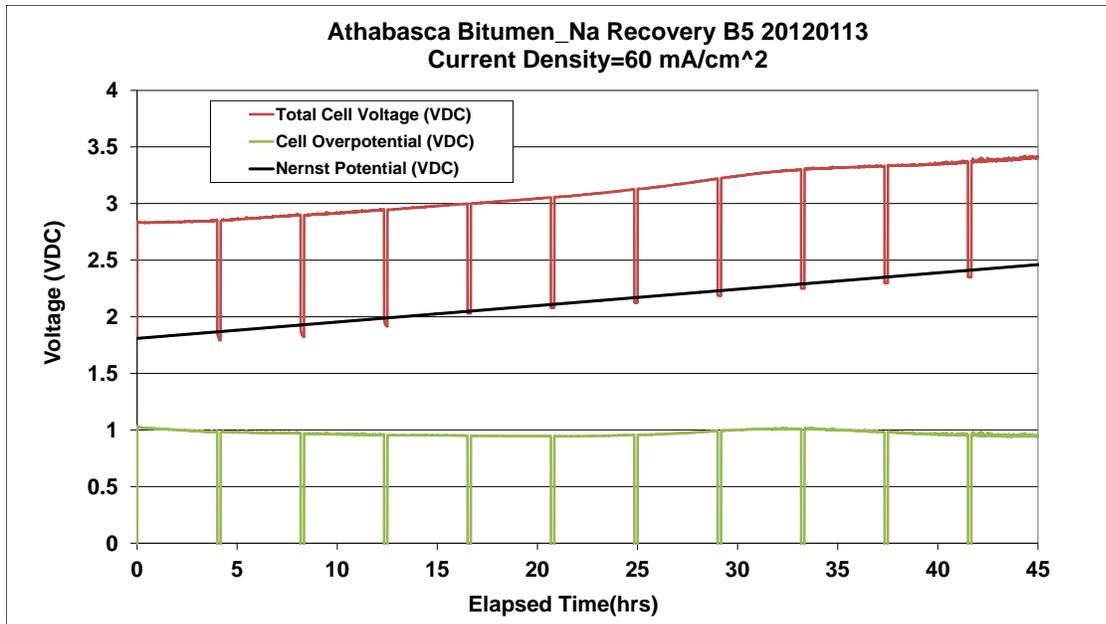


Figure 11: Total cell voltage, cell over-potential, and Nernst potential for sodium recovery test cell *Na Recovery B5_20120113* during the first 45 hours of the test at 60 mA/cm² current density

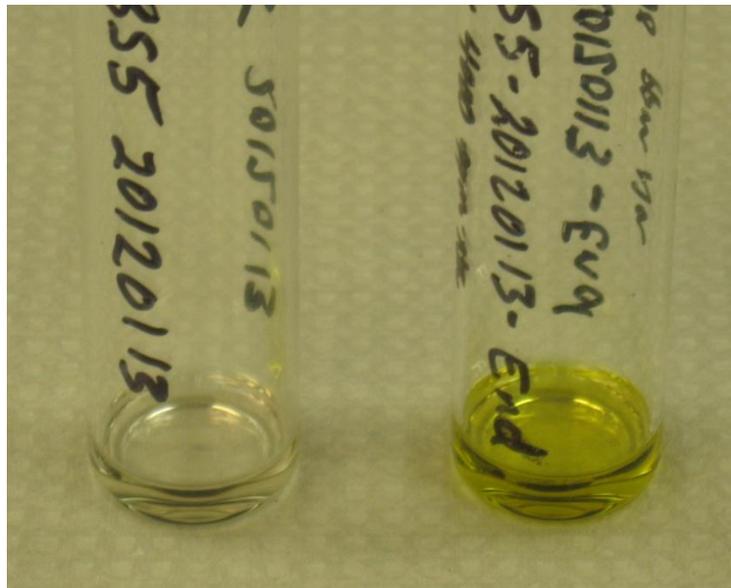


Figure 12: Pictures of the analyte solution of cell *Na Recovery B5_20120113* before (left) and after (right) the test

Task 7.0 – Analysis

Subtask 7.1 – Develop electrolysis process model

Ceramatec shall analyze data from Task 3 and a performance model will be developed. Various factors such as membrane thickness, type of alkali metal, electrode configuration and cell design would be included in the model considerations.

Subtask 7.2 – Preliminary cost analysis

The Recipient shall incorporate the models from Subtasks 4.1 and 4.2 into a preliminary cost model. Based on the preliminary cost analysis, a selection will be made between sodium and lithium as the most promising alkali metal for further pursuit in Phase 2. Sodium was selected because of higher efficiency of removing sulfur and lower cell voltage in electrolysis compared to Lithium.

3. CONCLUSION

Our conclusion at this point is that high levels of both sulfur and nitrogen can be removed from shale oil or heavy oil with the process tested. Nitrogen removal has been less successful with Shale Oil 2 which of the three oils was the least controlled prior to receipt in terms of storage in air and subject to ambient thermal cycles. Both methane and hydrogen are effective in removal of sulfur, nitrogen, heavy metals, and increasing API gravity.

4. COST STATUS

The monthly costs of the 10th quarter are shown in Table 8.

Table 8: Project revised costing profile for the 10th Quarter

Note: Benefits, Overhead, & G&A rates char	Jan-12		Feb-12		Mar-12		Q10		
	Projected	Actual	Projected	Actual	Projected	Actual	Projected	Actual	
Direct Labor		38,049.48		35,775.38		34,142.15		107,967.01	
Benefits	29.76%	11,323.53		10,646.75		10,160.70		32,130.98	
Overhead	43.71%	16,631.43		15,637.42		14,923.53		47,192.38	
Total Burdened Labor		66,004.43		62,059.55		59,226.39		187,290.37	
Direct Materials / Spec Test		18,843.49		11,187.40		21,743.74		51,774.63	
Equipment						-		-	
Consulting		15,119.52		13,956.48		13,956.48			
Travel		1,085.46		238.00		-		1,323.46	
Subtotal		101,052.90		87,441.43		94,926.61		283,420.94	
G&A	31.44%	31,771.03		27,491.58		29,844.92		89,107.53	
Total monthly		-	132,823.93	-	114,933.01	-	124,771.52	-	372,528.47

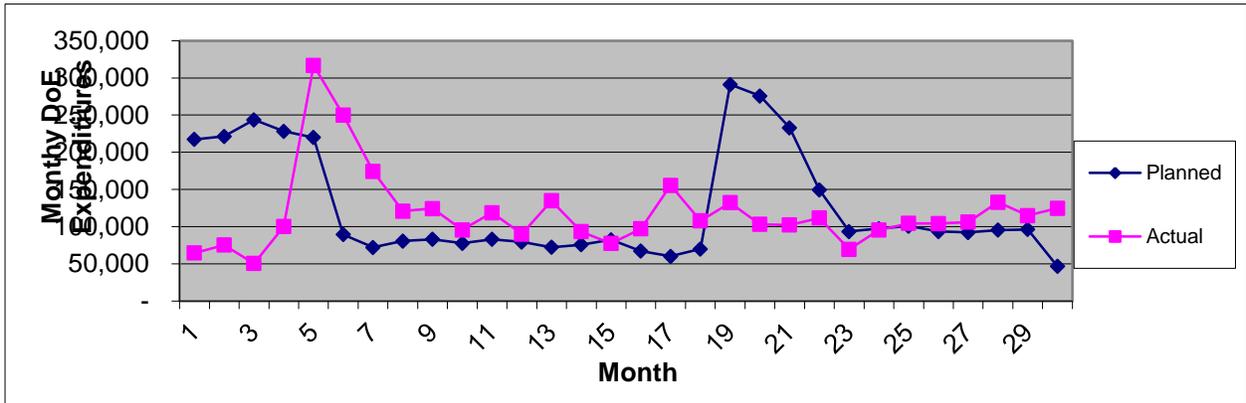


Figure 13: Projected and actual monthly costs over time

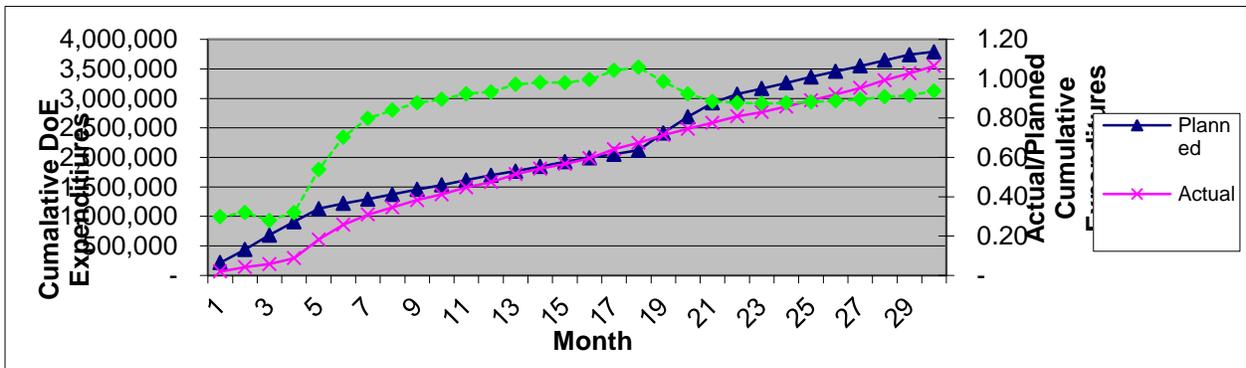


Figure 14: Projected and actual cumulative costs over time

5. MILESTONE STATUS

New milestones need to be set for the current Budget Period.

Table 9: Milestone log for 6th Quarter

Milestone No.	Task / Sub-task	Project Milestone Description	Planned Start Date:	Planned End Date:	Actual Start Date:	Actual End Date:	Comments
1	1	Updated PMP	9/29/09	10/29/09	9/29/09	10/26/09	
2	2.1	Analytic capability established	9/29/09	3/1/10	9/29/09	3/23/10	Analytical capability has been established as stated in the PMP. Operators have been trained on GC. ICP and CHNS are operational
3	2.2	Complete upgrading exp. Setup	9/29/09	3/29/09	9/29/09	3/26/10	Upgrading set-up has been completed including HAZOP and pre-start up safety review. The reactor set up has been ready to be operational as of Friday, March 26, 2010.
4	2.3	Complete process runs	3/30/10	1/3/11	3/26/10	3/31/11	Process runs underway
5	3.1.1	Complete membranes for Phase 1	9/29/09	7/5/10	9/29/09	9/20/10	Membrane fabrication has exceeded demand for fabrication. Mechanical characterization was complete on September 20, 2010.
6	3.3	Cells ready for operation	4/13/10	2/28/11	4/13/10	3/31/11	Cells were ready for operation on time. Initial cells testing began running 4/26/10 when sufficient sodium polysulfide was synthesized.
7	4.3	Preliminary cost model complete	2/8/11	3/14/11	1/4/11	3/18/11	A preliminary cost model was completed and reviewed internally. Adjustments were recommended by the reviewers and additional cases suggested for updated cost models.

6. ACCOMPLISHMENTS

- Additional experiments using a Coker Diesel feedstock were conducted in this quarter. A total 98% sulfur removal of the as received material was achieved. Sulfur removal was independent of the type of headspace gas used (H_2 , CH_4 , or N_2).
- Two Athabasca bitumen feed stocks from the regions of Mc. Kay River and Peace River in Alberta were tested. In both cases, we were able to upgrade the API of receiving oil from 8 up to 20, while removing up to 95% of the sulfur. Volumetric liquid yields in excess of 90% were obtained.
- A large lab space has been identified and assigned for this project. Plans for the installation of the new 1.8 liter upgrading reactor in this laboratory have been made.
- Our long term electrolysis test cell completed 5694 hours at a current density of 95 mA/cm². The cell failed due to loss of temperature control following a power outage.
- A new long term test cell using Na_2S_4 has been initiated. The cell has logged a total of 1020 hours of operation.
- A thermal pretreating process, to improve the efficiency in the separation of sodium sulfide from the solids generated in the upgrading reactions, has been developed and tested.
- Electrolysis of pretreated sodium sulfide containing solids, obtained from a H_2 upgrading run of Athabasca bitumen, has been successfully demonstrated.

7. PROBLEMS OR DELAYS

Our simdist software is not working right now and is preventing us from obtaining results.

8. PRODUCTS

No products to report at this time.

9. LIST OF APPENDICES

None

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