Oil & Natural Gas Technology

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Quarterly Report
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Post Retort, Pre Hydro-treat Upgrading of Shale Oil

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDICES
None

EXECUTIVE SUMMARY

After 2 quarters of primarily setting up the laboratory, the 3rd quarter can be characterized as one with extensive experimentation.

Both upgrading reactor and electrolysis endeavors were focused on sodium as the active alkali metal. Sulfur and nitrogen reduction were found using sodium as the active using 2 different shale oil sources, but the process is far from optimized. Also, on the electrolysis side, after changing the anolyte solvent, and moderately raising the temperature, sodium removal from a sodium polysulfide solution electrolytically ran very well. Improvement in electrolysis is attributed to greater polysulfide solubility. Lithium remains to be evaluated but our lithium membrane is much improved so we are in a good position for that work in the next quarter.

A technician with prior gas chromatography experience joined our team primarily to assist with reactor runs and analysis. An offer has been made to a Senior Ceramic Technician to be dedicated to our membrane fabrication, characterization and improvement.

We have had issues with our CHNS analysis which we expect to resolve in the next quarter with assistance from Perkin Elmer.

Expenditures to date are approaching our cumulative budget target and are about 12% low.

PROGRESS, RESULTS, DISCUSSION

Task 1.0: Project Management Plan

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

Task 2.0: 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.

Sub task 2.1: Analytical Capability

Analytical Laboratory Set up

The laboratory set up was complete in the second quarter. The analytical laboratory scientist traveled for attending a workshop on Perkin-Elmer Inductive Coupled Plasma for gaining scientific understanding of the instrument such that results can be better interpreted. The Perkin-Elmer 2400 CHNS analyzer needed a modification to feeding mechanism. The modification has been accomplished. New standards have been ordered to serve better for CHNS analysis.

Sub-task 2.2: Upgrading Reactor and Separation Setup

Experimental

Several different experiments were performed on upgrading of Red Leaf shale oil (RL), (Oil shale Exploration Company shale oil (OSEC) and San Joaquin heavy crude oil (SJ). All the experiments were performed with using sodium as the alkali metal, 200 gm of oil as raw material and 1000 psig pressure. Upon reaction, the product was subjected to centrifuging to separate the solid fraction (sulfides and possibly heavy metals) from liquid. Table 1 below shows the list of different experiments.

Table 1: List of reactor experiments Performed

Experiment Number	Feedstock	Details		
		Temperature (^{o}C)	Quantity of sodium (g)	Reaction time (Min)
1	RL	375	6.0	30
2	RL	275	6.0	60
3	RL	125	6.04	120
4	RL	125	None	60
5	RL	375	None	120
6	RL	375	3.0	60
7	RL	275	3.0	60
8	RL	375	2.0	60
9	OSEC	375	2.0	60
10	OSEC	375	1.0	60
11	SJ	375	6	30

Results and Discussions
CHNS measurements were performed on raw material samples and product samples Product samples from experiment 1, 6, 9, 10 and 11. Compositional analysis for all the three raw material samples is shown in Table 2 below and Table 3 lists analysis results for products.

Table 2: C, H, N and S Composition of Raw Materials

Raw material	C (%)	H(%)	N (%)	S (%)
RL	85.48	12.33	1.48	0.25
OSEC	84.83	12.74	0.47	0.84
SJ	85.70	11.27	0.76	1.54

Table 3: C, H, N and S Composition of Products

Experiment number	C (%)	H (%)	N (%)	S (%)
1	85.79	12.97	0.72	0.04
6	84.88	12.30	1.29	0.11
9	85.77	12.99	0.39	0.51
10	85.33	12.81	0.40	0.62
11	85.61	11.51	0.77	0.37

SimDist (Simulated Distillation)
All the samples obtained from experiments 1 through 11 were subjected to SimDist analysis using HP 6890 GC. Both the shale oil samples being very light, more than 95% of material boiled in the 9 through 13 carbon range. Hence the impact of upgrading process could not be quantified towards heavy to light hydrocarbon conversion. For San Joaquin heavy crude, it was observed that after the upgrading process, boiling point was reduced by 41 ^{o}C at 60% mass boil -off indicating improvement in oil quality.

Conclusion
1. San Joaquin heavy crude sulfur content was reduced by 75% while nitrogen remained unchanged. A reduction in boiling point by 41 ^{o}C at 60% mass boil-off, indicated improvement in oil quality.
2. Red leaf shale oil nitrogen was reduced by 51% while sulfur content was reduced by 84%.
3. OSEC shale oil sulfur was reduced by 26% and 40% in two different experiments (40% sulfur reduction had twice the amount of Na) while both the experiments showed 17% reduction in nitrogen content
4. A different GC column would be required for finer resolution.

Schedule
The progress of Task 2 is as per schedule.

Task 3.0 – 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.

Subtask 3.2 – Seal testing

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

EPDM (Ethylene Propylene Diene Monomer) O-ring seals (3 inch in diameter and 0.07 inch thick) were used in the Test Cells to seal the cathode and anode chambers. Data obtained from Test Cell runs indicate that even after only one or two days of exposure of the O-rings to the anolyte and catholyte solutions, they start showing signs of wear. By far, the largest damage is in the anode O-ring, which is the one exposed to the corrosive polysulfide salts. The damage is in the form of deformation, stretching, and weight loss. However, no leaks of electrolyte were observed during cell operation. Long term testing of several O-ring materials will be conducted in the near term future to sort out the best candidates.

In the Test Cells, 1.5” in diameter Nasicon and Lisicon wafers are sealed to a stainless steel mounting plate by a high temperature and chemical resistant epoxy. This material, called Duralco 4461, is manufactured by Cotronics Corp. In a different program at Ceramatec, researchers have verified that this material is stable in contact with sodium metal up to 200°C. Our short term testing experience using this type of seal in the polysulfide environment of the anolyte has proven very satisfactory. Similarly to the O-ring seals, aggressive long term testing will be done in the future with this and other sealing materials.

Subtask 3.3 – 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.

Electrolysis Test Cell Design

Designed and fabricated an electrolytic test cell. The cell is ideal for quick scouting experiments due to its easiness of assembly and disassembly. Figures 1 and 2 show side views of the cell with all the components. The cell fits 1.5” in diameter Nasicon or Lisicon wafers, which are placed in between adjustable cathode and anode electrodes. The electrodes-to-membrane distance can be varied by simply tightening or loosening the four screws of the anode electrode assembly, and by moving up/down the "spider" cathode piece. While the anode compartment volume is constant, the cathode compartment volume can be increased or decreased by moving the "spider" cathode piece up or down, respectively. The hermiticity of the cathode and anode compartments is accomplished by two O-ring seals, which are compressed against the membrane mounting plate. The cell can be run in batch mode by preloading known volumes of catholyte and anolyte, or in

continuous mode with respect to the anolyte. In the continuous mode, a stream of anolyte is flown in and out the anode chamber from a reservoir tank. The cell can be operated above ambient temperature inside a lab oven. In the continuous mode, the anolyte solution can be heated up externally in a reservoir tank. Figure 3 shows a photograph of a fully assembled cell with the anode and cathode electrical wires visible.
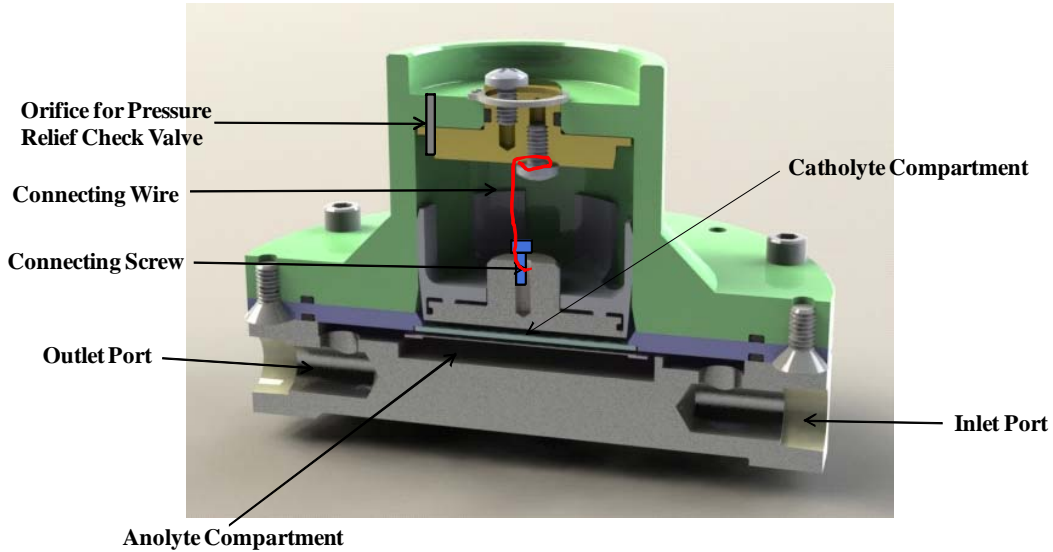

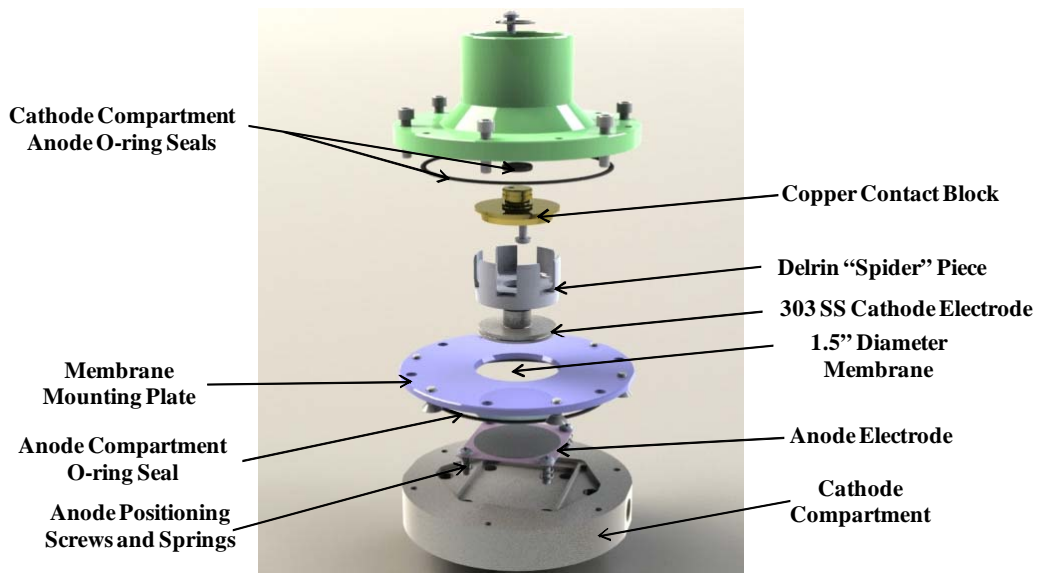**Figure 1.** Solid Works model of the Electrolytic Test Cell

Figure 2. Solid Works model of the Electrolytic Test Cell with all its components
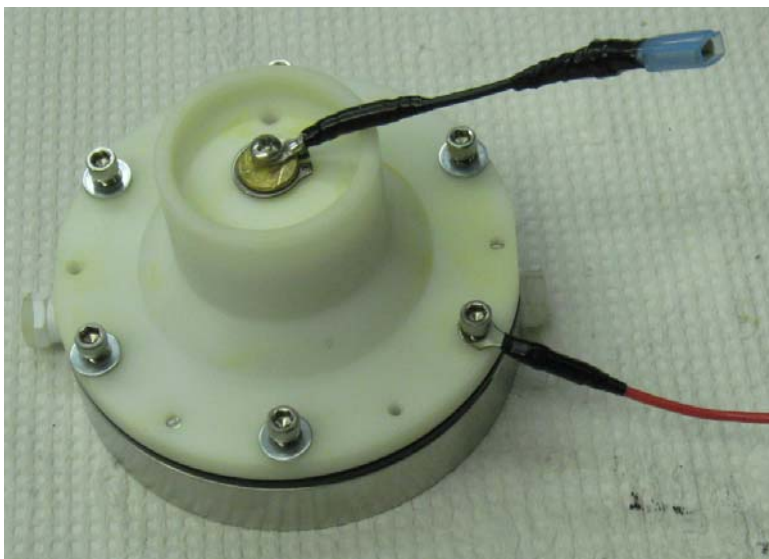
Figure 3. Photograph of an assembled Electrolytic Test Cell

Anolyte Circulating/Heating System Design

Designed and assembled a circulating/heating system to operate the anode electrolyte of the new test cell in continuous or semi batch modes (Figure 4). The system consists of an insulated 2 liter PTFE reservoir tank, where the polysulfide containing anolyte is stored and heated-up to the test cell operating temperature with the help of a submergible heater. A variable speed peristaltic pump is in charge of circulating the anolyte to and from the reservoir tank through the cell's anolyte compartment. A by-pass circuit allows to accurately adjust the anolyte flow rate within the range 5-500 ml/min. A PID controller controls the temperature of the anolyte in the tank within ±0.5ºC of accuracy from the setpoint value, using the test cell's temperature as the measured feedback variable.
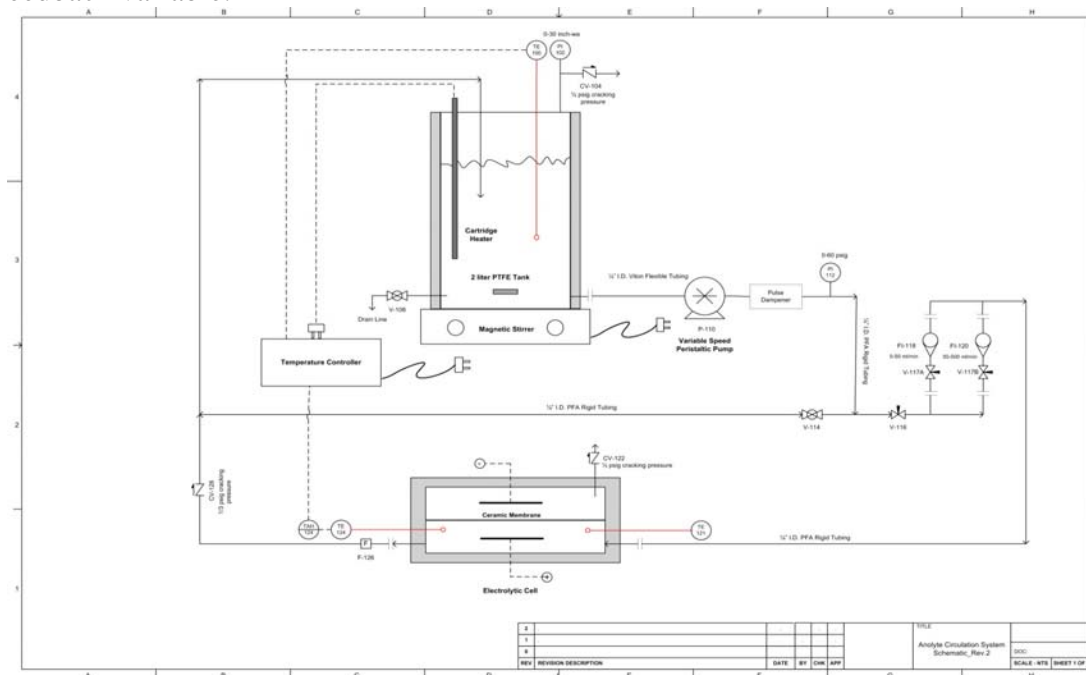

Figure 4. Schematic of the anolyte circulating/heating system

Electrolysis Lab Instrumentation Procurement
Ordered a 16-channel potentiostat/galvanostat testing instrument from Arbin Instruments (model BT2000). This device will allow us to conduct advanced electrochemical measurements of up to 16 test cells simultaneously. The instrument will be installed and setup in Lab 214.

Subtask 3.4 – 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.

Survey of Organic Electrolyte Solvents
A survey has been conducted to identify organic solvents that can be used as basis for the catholyte and anolyte solutions in the electrolytic cells. The general physical properties that make a good solvent for this application include high boiling point, high ionic conductivity, electrochemical stability within a large voltage window, thermo stability, relatively high flash point, and low viscosity. In addition, high alkali metal polysulfide solubility is desired in the anolyte solution to maximize the recovery efficiency of the electrolytic process. Critical properties that the catholyte solvent needs to meet are lack of reactivity with the alkali metal and large solubility of the supporting electrolyte. A supporting electrolyte that contains cations of the alkali metal is needed to increase the ionic conductivity of the catholyte solvent and to support the desired redox reaction. Table 1 lists some potential solvents that meet some of the requirements mentioned above.

Unfortunately, reliable data of ionic conductivity, polysulfide and supporting electrolyte solubilities or electrochemical stability of the solvents is very scarce in the open literature. Therefore, we have started a study to measure these physical properties to better down select our optimum solvents. During this reporting period, we have tested a glyme (Tetraethylene Glycol Dimethyl Ether, Tetraglyme), three amides (Formamide, Methylformamide, and Dimethylacetamide), and a cyclic carbonate (Propylene Carbonate). Figure 5 reports the measured solubility of Sodium Tetrasulfide (Na_2S_4) in the solvents at ambient temperature. Clearly, the amide solvents show a much higher solubility than Tetraglyme and Propylene Carbonate. Among the amides, Methylformamide presents the highest solubility of the three. One negative aspect of the amides is their reactivity with the alkali metals. Formamide showed the strongest reactivity among the three tested. This solvent was actually the only one of the tested that ignited the small sample of sodium metal added during the reactivity test. On the other hand, both Tetraglyme and Propylene Carbonate seem to be very stable in the presence of sodium metal.

Figure 6 shows the measured ionic conductivity of Methylformamide, Tetraglyme, and Propylene Carbonate at ambient temperature. Methylformamide is over two orders of magnitude more ionically conductive than the other compared solvents.

The conductivity of the solution of 0.75M Na_2S_4, dissolved in Methylformamide, is still eight times larger than that of Tetraglyme with 1M NaI and two times higher than Propylene Carbonate with 1M NaI (Figure 7). This result suggests that Methylformamide is an excellent solvent choice to be used as anolyte. However, its reactivity with the alkali metal makes it a less desirable option as catholyte in the cell.

Figure 8 shows the conductivity of Tetraglyme with different concentrations of supporting electrolytes Sodium Iodide (NaI) and Sodium Tetrafluoro Borate (NaBF4). It should be noted the large solubility of both salts in the solvent and the significant increase in ionic conductivity with salt concentration in solution. Sodium Iodide is cheaper and more readily available than Tetrafluoro Borate. So for the time being and until a better solvent+supporting electrolyte pair is discovered, Tetraglyme+NaI and Propylene Carbonate+NaI will the preferred catholytes used in the Test Cells. The look for better solvents and supporting electrolytes is an on-going objective of this research work.

Table 4. Physical Properties of selected organic solvents

Solvent	Class	Boiling Point	Density	Viscosity	Dielectric Constant	Ionic Conductivity	Reactivity with Alkali Metal?
		(°C)	(g/cm3)	(cP)		µS/cm	
Tetraglyme	Glymes	275	1.01	4.1	Unknown	0.2	None
Diglyme	Glymes	162	0.94	1.2	7.2	Not measured	None
Formamide	Amides	210	1.13	3.3	111	Not measured	High
Methylformamide	Amides	180	0.998	1.6	182	721.2	Low
Dimethylformamide	Amides	153	0.94	0.8	36.7	Not measured	Low
Dimethylacetamide	Amides	166	0.94	0.8	37.8	Not measured	Low
Propylene Carbonate	Carbonates	242	1.2	2.5	64	3.1	None
Ethylene Glycol	Glycols	197	1.11	13.5	37.7	Not measured	High
Propylene Glycol	Glycols	188	1.03	56	32	Not measured	Unknown
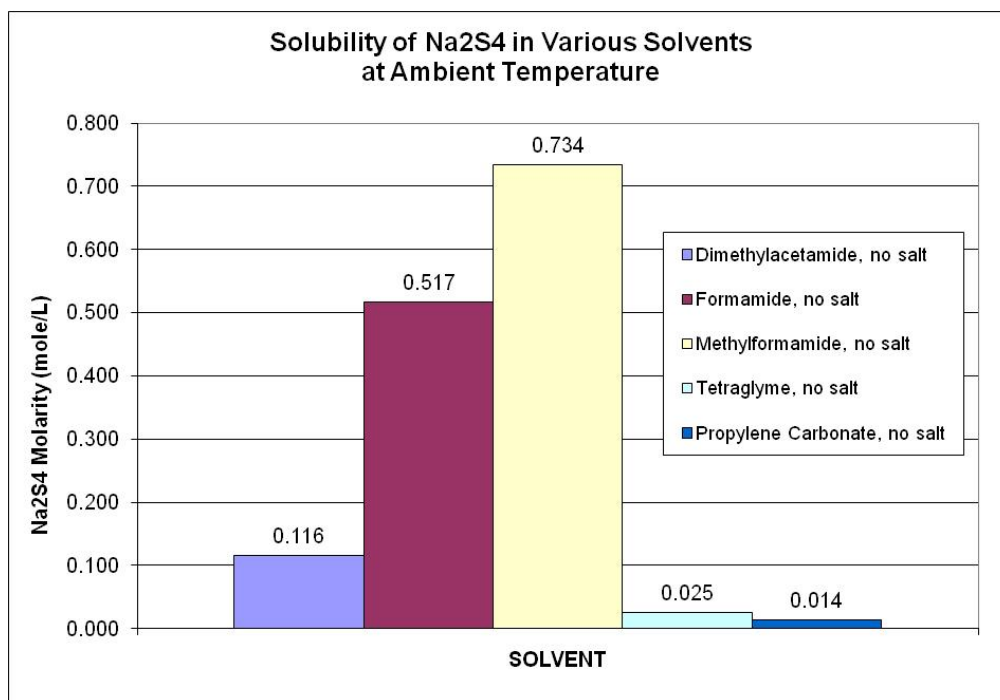
Figure 5. Solubility of Sodium Tetrasulfide (Na_2S_4) in selected solvents at ambient temperature
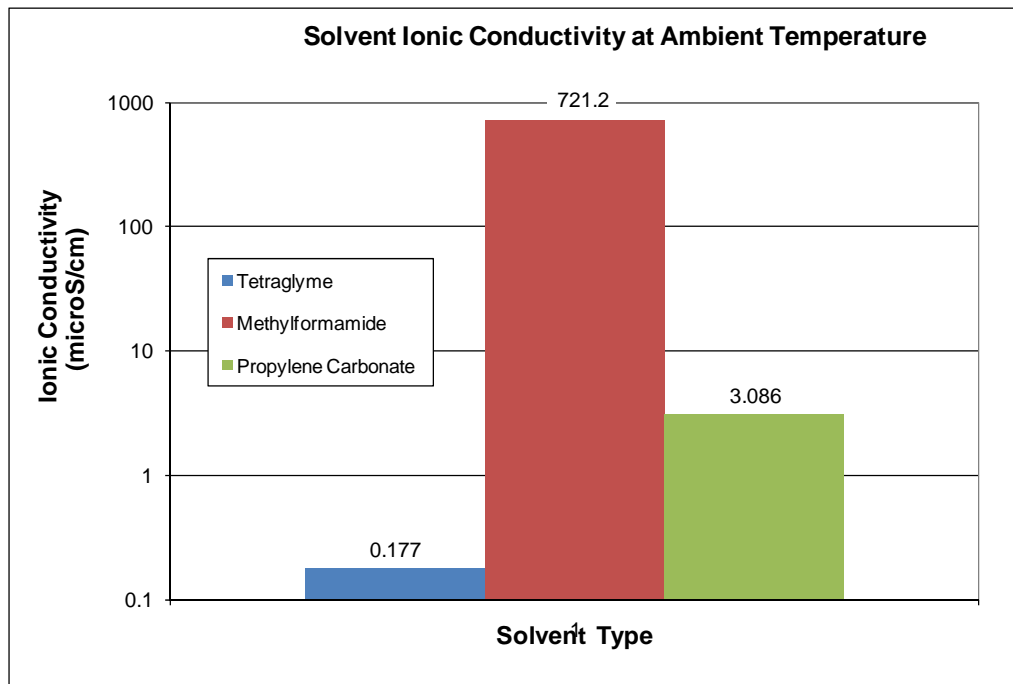
Figure 6. Ionic conductivity (log scale in microS/cm) of Tetraglyme, Methylformamide, and Propylene Carbonate at ambient temperature

Figure 7. Ionic conductivities (log scale in microS/cm) of Tetraglyme, Propylene Carbonate with supporting electrolyte (NaI), and Methylformamide with Sodium Polysulfide (Na_2S_4) at ambient temperature
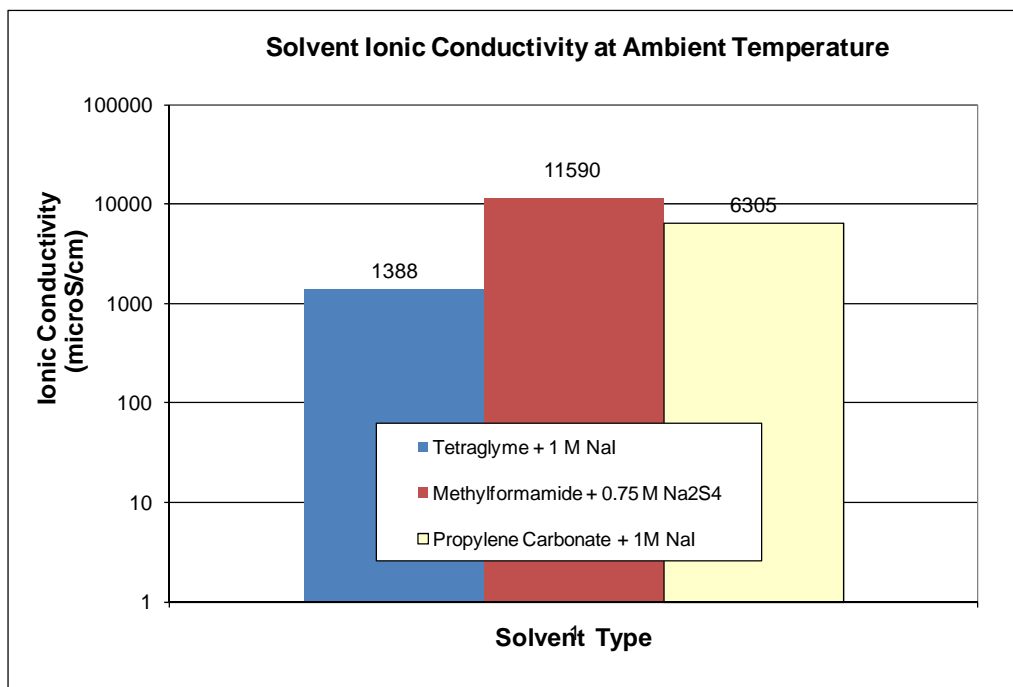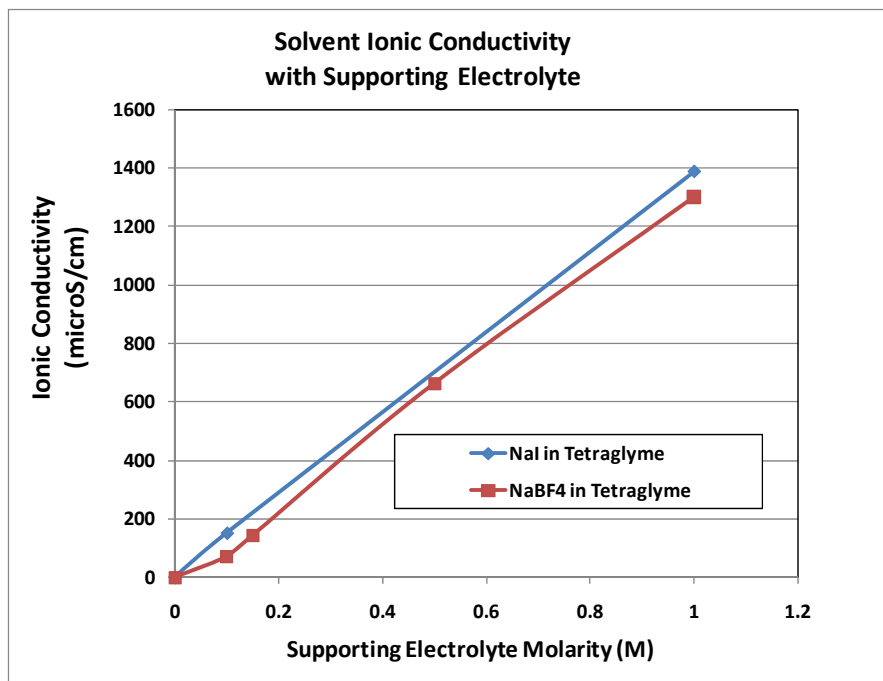

Figure 8. Ionic conductivity (in microS/cm) of Tetraglyme versus concentration of supporting electrolytes (NaI and $NaBF_4$) at ambient temperature

Implementation of Cyclic Voltammetry Technique

Cyclic Voltammetry has been applied to determine the adequacy and the electrochemical stability of different solvents and supporting electrolytes selected for this program. In this technique, the DC voltage is sweep between an initial and a final value, at a given sweep rate, while the DC current through the cell is measured and recorded. The main advantage of this method is that it shows the presence of undesirable electrolytic active species that can compete with the desired redox reactions undergone by the alkali metals (sodium or lithium) and polysulfide ions. Also, it can be used to measure the overall resistance of a test cell from the slope of the linear region of the I vs. E curve. For example, Figure 9 shows a voltammogram obtained from one of our test cells, with the main sodium reduction peak $Na^+/Na_{(s)}$ starting at 2.7 volts. The reduction peak of an unknown contaminant is present at around 1.5 volts. Fortunately, the effect of the contaminant on the selectivity of the desired reaction is very small as evidenced by the relative height of the peaks. The overall cell resistance was calculated to be equal to 73 ohms.
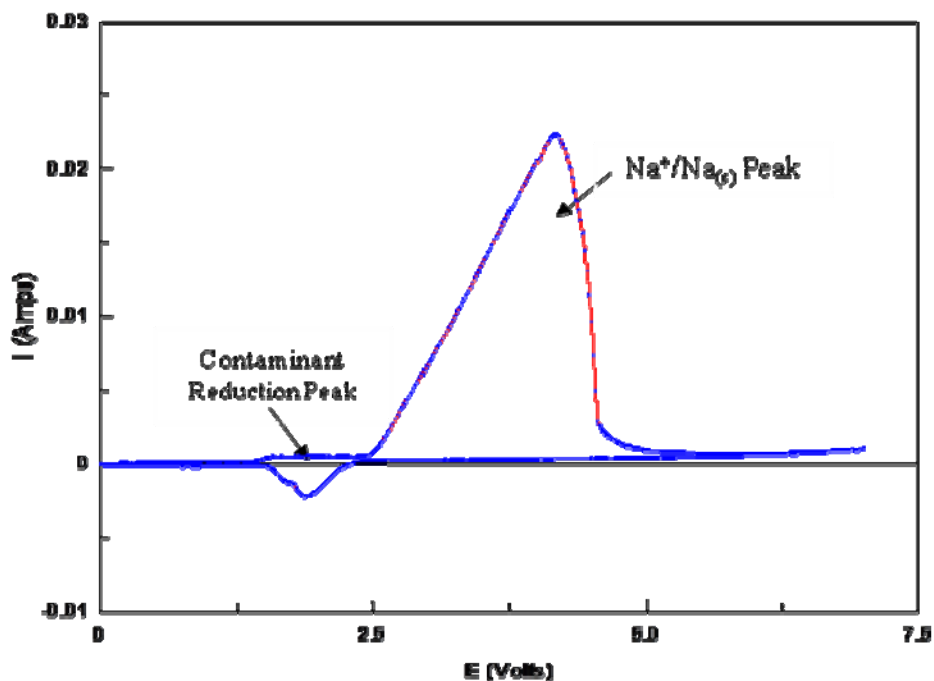
Figure 9. Cyclic voltammogram of a sample test cell

Test Cell Runs Results

A total of eight Test Cells of the new design have been assembled and run during this reporting period. Table 2 lists the construction properties of each of the cells including: anolyte and catholyte type; anode and cathode electrode materials and their distance to the membrane; and membrane type. The main goal of these initial test cell runs was to support the work of finding appropriate catholyte and anolyte systems and to study their effect on the cell's performance. The compatibility of the Nasicon GY wafers with the organic solvents and their reliability during the runs were also studied. Table 3 lists the operating conditions and summarizes the observa-

tions and main conclusions of each of the runs. The following are the most important findings learned from these first set of tests.

1.   The selection of the appropriate electrode materials is critical to avoid undesired reactions with the electrolyte components. Figure 10 shows pictures of the Nickel anode electrode [A] and the anode side of the Nasicon GY wafer after only seven hours of cell operation. Nickel was oxidized forming an unknown black insoluble substance that coated both the electrode and the membrane. We replaced Nickel with a more inert metal such as Titanium and the problem disappeared.

2.   As explained in the survey of electrolyte solvents section above, the use of proper catholyte and anolyte solutions is very important in terms of electrochemical stability, poly-sulfide solubility, and overall ionic conductivity. For example, Test Cell #20100630, which was assembled with an anolyte consisting of Methylformamide+0.75M Na_2S_4, had an overall ionic conductivity 6 times larger than Test Cell #20100616, which was assembled with Tetraglyme+0.03M Na_2S_4+1M NaI. As a result, the first Test Cell was able to run at a current density 10 times larger than the second one.

3.   Most of the wafers, disassembled from cells that successfully produced sodium, show extensive cracking with sodium metal visible in the cracks as displayed in Figure 11[B] and Figure 12[A] and 12[B]. We are investigating the failure mechanism of these wafers.

4.   Using Cyclic Voltammetry, we have observed a significant surface electrode deactivation as manifested by a decrease in the peak current and a shift in the peak potential of successive voltage sweeps. We believe that the rate of electron transfer at the electrode surface is hindered by the precipitation of insoluble salts on the surface. We are currently investigating the source of this problem and ways to prevent it.

5.   An interesting finding relates to the way in which sodium metal has plated to the cathode electrode in different cell runs. In one particular case (Figure 11[A]) a bright and shinning deposit of sodium was observed, whereas in others cases more granular and porous deposits turned out (Figure 12 [C]).

Table 5. Electrolytic Test Cells assembled and tested during the reporting period

Cell ID#	Anolyte	Anode Electrode	Membrane	Catholyte	Cathode Electrode
20100513	0.02M Na2S4 in TG	Nickel mesh 1/8" to membrane	Nasicon GY, t=0.71 mm	1M NaI in TG	303 SS disk 1/4" to membrane
20100519	0.01M Na2S4+0.1 M NaI in TG	Nickel solid foil 1/4" to membrane	Nasicon GY, t=1.50 mm	1M NaI in TG	303 SS disk 1/8" to membrane
20100524	0.03M Na2S4+0.1 M NaI in TG	Titanium mesh 0.07" to membrane	Nasicon GY, t=0.69 mm	0.1M NaI in TG	303 SS disk 1/8" to membrane
20100527	1M NaI in TG	Titanium mesh 0.07" to membrane	Nasicon GY, t=0.74 mm	1M NaI in TG	303 SS disk 1/8" to membrane
20100601	0.03M Na2S4+0.15 M NaBF4 in TG	Titanium mesh 1/8" to membrane	Nasicon GY, t=0.72 mm	0.15M NaBF4 in TG	303 SS disk 1/4" to membrane
20100604	0.03M Na2S4+0.1 M NaI in TG	Titanium mesh 0.07" to membrane	Nasicon GY, t=1.52 mm	0.1M NaI in TG	303 SS disk 1/8" to membrane
20100616	0.03M Na2S4+1 M NaI in TG	Titanium mesh 0.07" to membrane	Nasicon GY, t=1.48 mm	1M NaI in TG	303 SS disk 1/8" to membrane
20100630	0.75 M Na2S4 in Methylformamide	Titanium mesh 0.07" to membrane	Nasicon GY, t=1.38 mm	1M NaI in TG	303 SS disk 1/8" to membrane

Table 6. Summary and conclusions of the Test Cell runs conducted during the reporting period

Cell ID#	Run Conditions	Run Summary	Conclusions
20100513	Temperature=50°C Cte DC Current=0.4 mA	7 hours total run time. Cell never reached steady state conditions. Voltage continuously increased during run until it reached the 6 V overvoltage limit. Black solid residue formed on Nickel anode electrode. Sodium was not recovered from cathode electrode.	Nickel electrode is not electrochemically stable. Supporting electrolyte is needed in anolyte to increase electrical conductivity.
20100519	Temperature=50°C Cte DC Current=1mA	9 hours total run time. After 6 hours, voltage started increasing until it reached overvoltage limit. Black solid residue formed on Nickel anode electrode. Sodium was not recovered from cathode electrode.	Confirmed that Nickel electrode is not electrochemically stable in this system.
20100524	Temperature=50°C DC Current=2mA for 13.5 hr; 1 mA for 19.5 hours; 0.5-0.2 mA for last 6 hours	38 hours total run time. DC current reduced in steps after reaching voltage limit in previous step. 32 mg of sodium recovered from cathode electrode. Sodium plated preferentially in one half of the electrode. Large side to side crack in Nasicon membrane. Sodium metal observed along crack in wafer.	Titanium is a better anode electrode choice than Ni. Cell overvoltage increases with run time, perhaps due to electrode surface passivation.
20100527	No sodium sulfide in anolyte Temperature=50°C DC Current=2mA for 1 hr; 1 mA for 8.5 hours;	9.5 hours total run time. Sodium was not observed in cathode electrode.	The purpose of this run was to verify if iodide could be oxidized in the absence of polysulfide ions in the anolyte. Even though the cell run, sodium was not recovered from the cathode. The results are inconclusive.
20100601	Temperature=50°C DC Current=1 mA for 3 hr; 1.5 mA for 10.5 hours	13.5 hours total run time. After 8 hours of operation, cell voltage started to increase until reached voltage limit. No sodium detected in cathode electrode. Salt cristals cover the electrode surface instead.	The purpose of this run was to study effect of replacing NaI with NaBF4 in the anolyte and catholyte solutions as supporting electrolyte. Even though the cell run, sodium was not recovered from the cathode.
20100604	Temperature=50°C DC Current=1 mA	8 hours total run time. Cell voltage started increasing right from the beginning of test until it reached voltage limit. Around 2 hours run time, a leak was observed. The run was paused, the leak fixed and additional anolyte was added. No sodium was detected in cathode electrode.	The purpose of the run was to replicate run conditions of cell 20100524. It is possible that the leak exposed the cathode to external moisture, which migh have reacted the small amount of sodium formed during the run. It was decided to run future cells inside dry box to minimize moisture exposure.
20100616	Cell run inside dry box Ambient Temperature DC Current=0.5 mA	72 hours total run time. Cell voltage remained relatively stable at 3.25 V for the first 57 hours and then suddently increased to 7.45 V for remaining of run. Overall cell resistance (from cyclic volammetry) equal to 447 ohms. 28 mg of granular sodium recovered from cathode. A large part of the sodium fell off from electrode surface into membrane. Nasicon membrane showed extensive cracking on cathode side. Yellowish permanent decoloration of the membrane on anode side.	Cell achieved stable longest run time at very low current density. Plating of granular sodium might be advantageous for easier recovery. Cyclic voltammetry shows
20100630	Cell run inside dry box Ambient Temperature DC Current=5 mA	7 hours total run time. Cell voltage remained stable at 3 V for 6.75 hrs until sudden increase that reached overvoltage limit and terminated run. Overall cell resistance (from cyclic volammetry) equal to 74.6 ohms. 30 mg of sodium recovered from cathode. Extensive cracking of Nasicon membrane. One large crack ran radially from side to side and through the membrane. Sodium metal visible in some of the larger cracks.	Successful first run using methylformamide solvent as anolyte. Sodium sulfide is significantly more soluble in methylformamide. As a result, no supporting electrolyte is added to anolyte to increase electrical conductivity. This anolyte allowed the cell to run at a current density 10 times higher than with Tetraglyme based anolyte.

Membrane improvement
A Lipon coating was successfully applied to a Lisicon membrane by a contractor.

Task 4.0 – Analysis

Subtask 4.1 – Develop upgrading process model

Ceramatec shall evaluate several process variables in Task 2 which will impact the effectiveness of impurity removal, saturation, coking, and alkali metal utilization. In addition, the shale oil source and initial composition is likely to be a factor. Extent of hydrogen donation gas added and presence of hydrotreating catalyst will affect the extent of upgrading. A preliminary model based on the data from Task 2 will be constructed.

No progress on this task to date. Plan to begin in the 4th quarter.

Subtask 4.2 – 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.

No progress on this task to date. Plan to begin in the 4th quarter.

Subtask 4.3 – 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.

No progress on this task to date.

Task 5.0 – Reporting

The 3rd Quarterly Report is presently submitted.

CONCLUSION

Our conclusion at this point is that levels of both sulfur and nitrogen can be removed from shale

oil with the process tested. More optimization is required. So far process has been more effective

with shale oil than with Heavy Oil.

COST STATUS

The monthly costs of the 3rd quarter are shown in Table 7, along with the projected costs stated in the Project Management Plan.

Table 7. Project costing profile for the 3rd Quarter

		Apr-10		May-10		Jun-10		Q3	
		Projected	Actual	Projected	Actual	Projected	Actual	Projected	Actual
Direct Labor			31,867.58		32,505.40		28,631.49		93,004.47
Benefits	32%		10,197.63		10,401.73		9,162.08		29,761.44
Overhead	41%		13,065.71		13,327.21		11,738.91		38,131.83
Total Burdened Labor			55,130.92		56,234.34		49,532.48		160,897.74
Direct Materials / Spec Test Equipment			79,966.60		37,548.72		44,156.68		161,672.00 -
Travel							2,745.24		2,745.24
Subtotal			135,097.52		93,783.06		96,434.40		325,314.98
G&A	29%		39,178.28		27,197.09		27,965.98		94,341.34
Total monthly		72,109.29	174,275.80	80,768.99	120,980.15	82,998.66	124,400.38	235,876.94	419,656.32

Our costs in the 3rd quarter were higher than the projection because there was a lag in ordering equipment in the 1st quarter that carried through. Overall the project is behind in spending by approximately 12% over the first 9 months.

Figure 10 shows a plot of the total monthly costs and the initially projected costs versus time and figure 11 shows the cumulative monthly costs versus time. Also shown in figure 11 is the fraction of actual over planned cumulative expenses.
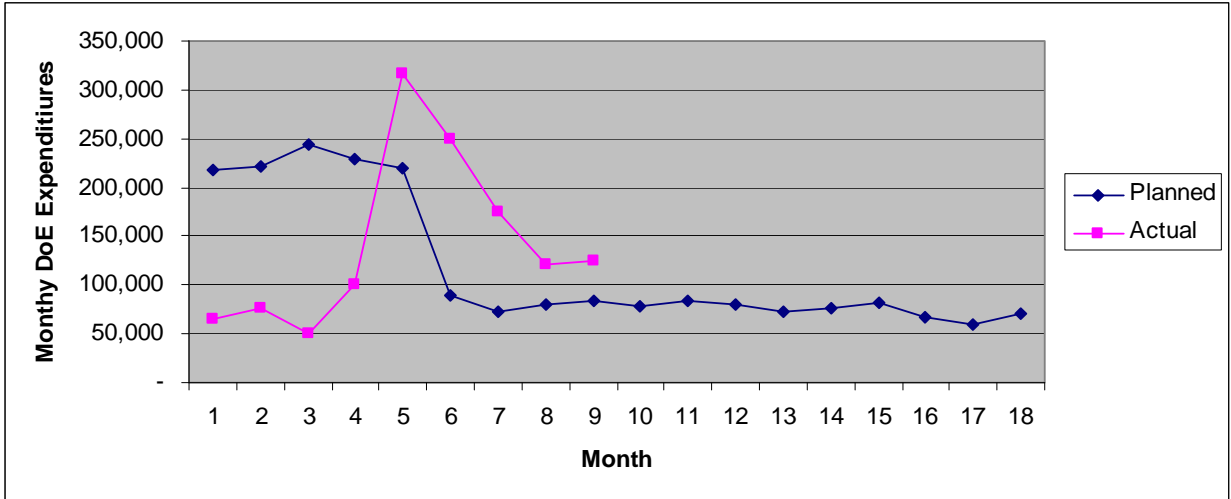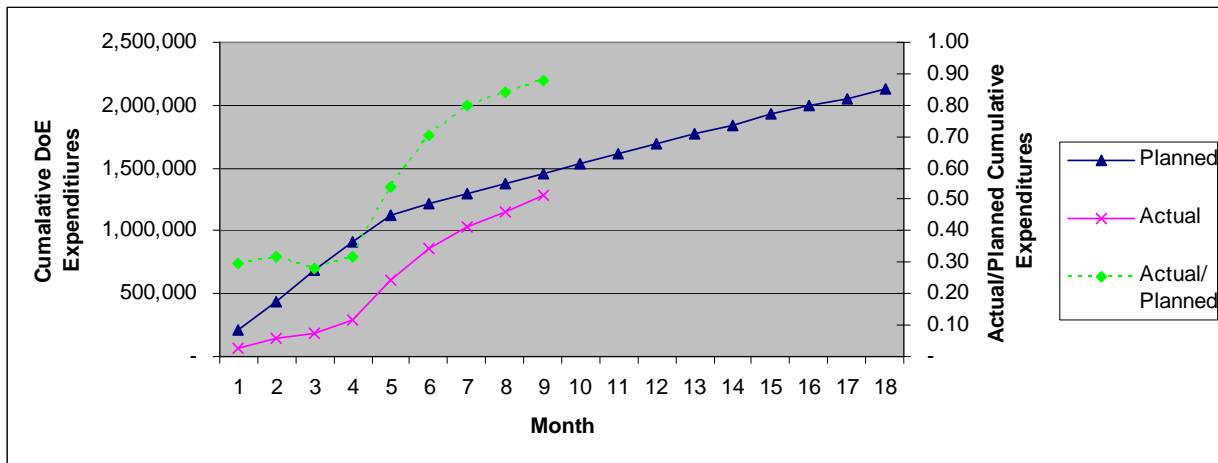
Figure 10: Projected and actual monthly costs over time

Figure 11: Projected and actual cumulative costs over time

MILESTONE STATUS

Table 8: Milestone log for 3rd Quarter

Mile stone 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 re-view. 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		Process runs underway
5	3.1.1	Complete membranes for Phase 1	9/29/09	7/5/10	9/29/09		Membrane fabrication has exceeded demand for fabri-cation. Mechanical character-ization will be complete 10/30/10
6	3.3	Cells ready for opera-tion	4/13/10	2/28/11	4/13/10		Cells were ready for opera-tion on time. Initial cells test-ing began running 4/26/10 when sufficient sodium poly-sulfide was synthesized.
7	4.3	Preliminary cost model complete	2/8/11	3/14/11			

Milestone 5 was scheduled for completion in the 3rd quarter but is being pushed out to the 5th quarter. The delay was necessary to bring the membranes to an improved level of leak tightness before characterizing in terms of physical properties.

ACCOMPLISHMENTS

- ✓ Reactor process runs have begun

- ✓ Our more conductive Lisicon membrane has been fabricated where the material was impermeable

- ✓ A Lipon coating was successfully applied to a Lisicon membrane.

- ✓ Sodium was recovered from a sodium polysulfide mixture through electrolysis with a Nasicon membrane

PROBLEMS OR DELAYS

Our standards for CHNS has been unsatisfactory. Testing and standards are being reviewed by Perkin Elmer. In the meantime samples have been sent to an outside lab for analysis. These analyses will confirm our methodology when our new standard arrives.

PRODUCTS

No products to report at this time.

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