

Electrolytes for Use in High Energy Lithium-Ion Batteries with Wide Operating Temperature Range

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Overview

Timeline

- Start Date = October 2009
- End Date = October 2014
- Percent complete = 50%

Budget

- Total project funding
 - 875K total (~ 175K/year)
 - Contractor share = 0K
- Funding received

FY'10 = 175K

FY'11 = 175K

FY'11 = 170K

Barriers



Barriers addressed

- Enhance low temperature performance
- Define performance limitations that limit life
- Develop long life systems stable at high voltage

Partners

Univ. Rhode Island (Brett Lucht)

(Analysis of harvested electrodes, on-going collaborator)

• Argonne Nat. Lab (Khalil Amine)

(Source of electrodes, on-going collaborator)

• LBNL (John Kerr, Li Yang)

(Evaluation of novel salts)

• Loker Hydrocarbon Institute, USC (Prof. Surya Prakash)

(Fluorinated Solvents and novel salts)

A123 Systems, Inc.

(Electrolyte development, on-going collaborator)

• Quallion, LCC.

(Electrolyte development, on-going collaborator)

Yardney Technical Products

(Electrolyte development, on-going collaborator)

• Saft America, Inc.

(Collaborator, industrial partner under NASA program)

NREL (Smith/Pesaran)

(Supporting NREL in model development by supplying data)

• Hunter College/CUNY (Prof. Steve Greenbaum)

(Analysis of harvested electrodes)

Sandia National Lab (Orendorff/Nagasubramanian)

(Assessment of electrolyte safety)



Objectives

- Develop advanced Li-ion electrolytes that enable cell operation over a wide temperature range (i.e., -30 to +60°C).
- Improve the high temperature stability and lifetime characteristics of wide operating temperature electrolytes.
- Improve the high voltage stability of these candidate electrolytes systems to enable operation up to 5V with high specific energy cathode materials.
- Define the performance limitations at low and high temperature extremes, as well as, life limiting processes.
- Demonstrate the performance of advanced electrolytes in large capacity prototype cells.

Milestones

Month/Year	Milestone or Go/No-Go Decision
Sept. 2012	Milestone: Prepare and characterize experimental laboratory cells containing Gen-3 electrolytes, designed to operate over a wide temperature range in high voltage systems (i.e., LiNiMnCoO ₂ and LiNi _{0.5} Mn _{1.5} O ₂), and identify performance limiting characteristics (Sep. 12)
Sept. 2012	Milestone: Demonstrate improved performance of second generation electrolytes over a wide temperature range (-30° to +60°C) compared with baseline electrolytes, in experimental and prototype cells (Sep. 12).

Technical Approach



Electrolyte Development Approach:

- Optimization of carbonate solvent blends
- Use of low viscosity, low melting ester-based co-solvents
- Use of fluorinated esters and fluorinated carbonates as co-solvents
- Use of "SEI promoting" and thermal stabilizing additives
- Use of novel, alternative lithium based salts (with USC, LBNL)

• Electrolyte Characterization Approach:

- Ionic conductivity and cyclic voltammetry measurements
- Performance characteristics in 300-400 mAh three electrode cells
 - MCMB/LiNi $_{0.8}$ Co $_{0.2}$ O $_2$, MCMB/LiNi $_{0.8}$ Co $_{0.2}$ AlO $_2$, Graphite/LiNi $_{1/3}$ Co $_{1/3}$ Mn $_{1/3}$ O $_2$, and Graphite/LLC-LiNiCoMnO $_2$
 - Use of high specific energy electrode materials (from in-house NASA program and DOE)
 - Electrochemical Impedance Spectroscopy (EIS) Measurements as function of temperature, high temperature storage, and cycle life
 - DC Tafel and linear (micro) polarization measurements on electrodes
 - Ex-situ analysis of harvested electrodes (URI and Hunter College)
- Performance characteristics in coin cells
 - Evaluation of electrolytes in conjunction with high voltage cathodes

Performance evaluation in prototype cells

- Yardney, A123, Saft, and/or Quallion Cells (0.300 mAh to 7 Ah size prototype cells)
- Cells will be procured and/or obtained through on-going collaborations

Summary of Technical Accomplishments



- 1) Demonstrated improved wide operating temperature performance with methyl propionate-based electrolytes (i.e., methyl propionate) in Quallion prototype cells.
 - Demonstrated wide operating temperature performance in large capacity (12 Ah) cells containing methyl propionate-based electrolytes (with and without FEC as additive).
 - Excellent performance was observed down to temperatures as low as -50°C, with over 75% of the room temperature capacity being delivered up to 2C rates with both formulations.
 - Currently evaluating a number of Quallion prototype 0.25Ah cells with methyl propionate-based electrolyte possessing varying FEC content and LiBOB as an additive.
 - An electrolyte consisting of LiPF₆ in EC+EMC+MP (20:20:60) + 0.10M LiBOB has been demonstrated to have good performance down to -50°C as well as excellent cycle life performance at +40°C in prototype 0.25Ah cells manufactured by Quallion, LCC.
- 2) Demonstrated good high temperature cycle life and improved low temperature performance LiFePO₄-based cells (A123) using methyl butyrate-based electrolytes:
 - Cells containing 1.2M LiPF₆ EC+EMC+MB (20:20:60) with the addition of 4% FEC or 2% VC demonstrated to have wide operating temperature range (i.e., -60°C to +60°C).
 - Electrolytes previously investigated in NCO-based three-electrode cells were shown to have improved kinetics at low temperature and improved resilience to high temperatures.
 - The cells were observed to perform well down to -60°C, with 80% of the room temperature capacity being delivered using a C/10 rate.

Summary of Technical Accomplishments



- 3) Performed electrochemical characterization of Graphite/Toda 9100 LiNiCoMnO₂ three electrode experimental cells with methyl butyrate-based electrolytes.
 - Using Tafel polarization measurements on each electrode (which possess heavy loading), it is evident the NMC-based cathode displays poor lithium kinetics (limiting electrode).
 - The MB-based electrolyte displays improved rate capability at low temperature compared to an all carbonate-based formulation.
 - Electrochemical measurements suggest that the cathode kinetics of this system is contributing to the poor rate capability at low temperature rather than being dominated by electrolyte type.
- 4) Evaluated a number of methyl butyrate-based electrolytes in Conoco A12 Graphite/Toda HE5050LiNiCoMnO₂ three electrode cells (Argonne materials):
 - The MB-based formulations containing LiBOB delivered the best rate capability at low temperature, which is attributed to improved cathode kinetics. Whereas, the use of lithium oxalate as an additive lead to the highest reversible capacity and lower irreversible losses.
 - At lower temperature and higher rates, the advantages of utilizing the high voltage system diminishes when compared a standard NCA material, again attributed to the relative cathode kinetics.
 - Of the different cathodes, the LLC-NMC electrodes (received from Argonne) displayed much slower lithium de-intercalation kinetics compared to the NCA electrodes (attributed to poor charge transfer resistance of the electrodes), which is exacerbated at lower temperatures.

Summary of Technical Accomplishments

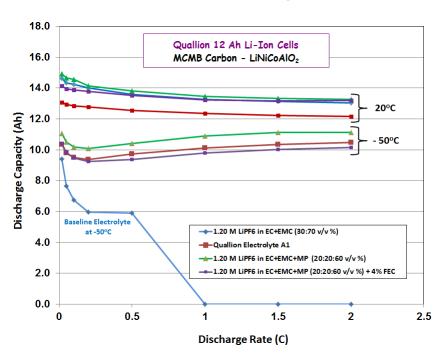


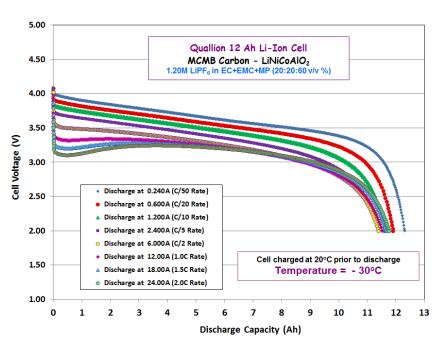
- 5) Investigated the life characteristics of a number of wide operating temperature range electrolytes in various high voltage systems.
 - Characterized a number of electrolytes in various systems, including (a) Li₄Ti₅O₁₂ (LTO)/LiNi_{0.5}Mn_{1.50}O₂ (LMNO) (b) Conoco A12 Graphite/Toda HE5050 LiNiCoMnO₂ and (c) MPG-111 Graphite/Toda 9100 LiNiCoMnO₂ (coin cell studies).
 - The incorporation of LiBOB was observed to improve the life characteristics of the methyl butyrate-based systems, displaying comparable life characteristics to all carbonate blends.
 - A number of formulations were observed to perform well in the Li₄Ti₅O₁₂ (LTO)/LiNi_{0.5}Mn_{1.50}O₂ (LMNO), providing improved cycle life over the baseline.
- 6) Characterization of Conoco Graphite/LiNi_{1/3}Mn_{1/3}Co_{1/3}O₂ (NMC) Coin Cells with low flammability electrolytes.
 - In collaboration Sandia National Laboratory (Chris Orendorff and Mani Nagasubramanian), we are currently evaluating a number of Conoco Graphite/LiNi_{1/3}Mn_{1/3}Co_{1/3}O₂ cells containing electrolytes with low flammability that were developed under a NASA-funded program.
 - Thus far, preliminary results suggest that many triphenyl phosphate-containing electrolytes show good compatibility with the system. However, in contrast to the higher voltage systems that we have studied, the incorporation of LiBOB does not provide benefit.
 - This is to understand the electrochemical performance in these systems, as Sandia National Lab is planning to perform the safety studies (ARC)



Technical Accomplishments Quallion Prototype 12 Ah Li-Ion Cells Wide Operating Temperature Electrolytes

Discharge Characterization at Various Temperatures



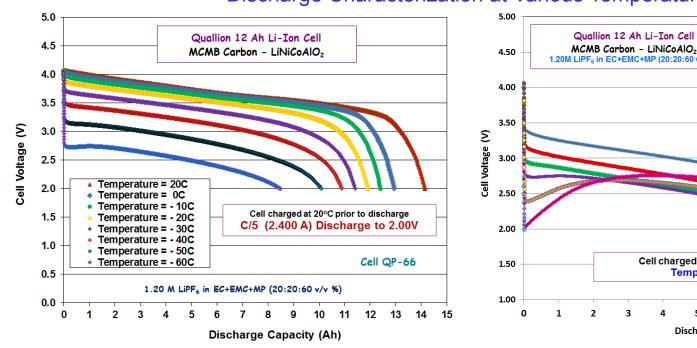


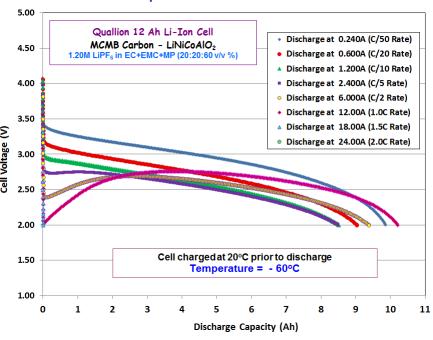
- Methyl propionate-based electrolyte was previously demonstrated to have dramatically improved rate capability compared to the baseline DOE formulation (i.e., 1.2M LiPF₆ in EC+EMC (30:70) in 0.25 Ah cells.
 - Performance successfully demonstrated in larger capacity prismatic 12 Ah cells.
 - ➤ Quallion collaboration supported by NASA SBIR Program. (H. Tsakamoto, M. Tomcsi, M. Nagata, and V. Visco)



Technical Accomplishments Quallion Prototype 12 Ah Li-Ion Cells Wide Operating Temperature Electrolytes

Discharge Characterization at Various Temperatures





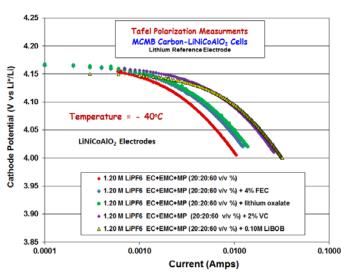
- Methyl propionate-based electrolyte demonstrated to have good performance down to -60°C, whereas the baseline electrolyte displays negligible capacity under these conditions.
- Currently performing life tests (~ 50% DOD) in which the capacity, impedance, and rate capability at low temperature will be periodically measured.
 - It is anticipated that the addition of FEC will improve the life characteristics.
 - ➤ Quallion collaboration supported by NASA SBIR Program. (H. Tsakamoto, M. Tomcsi, M. Nagata, and V. Visco)

Technical Accomplishments Quallion Prototype 0.25 Ah Li-Ion Cells

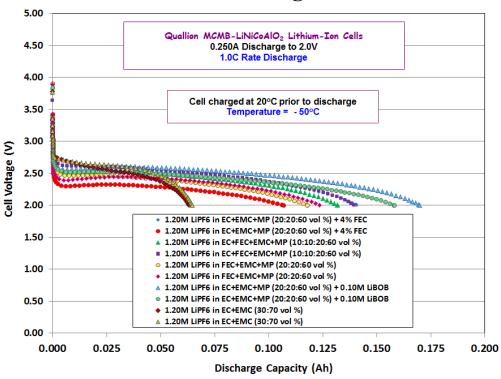


Discharge Characterization at Low Temperatures

- We are currently evaluating a number of 0.30 Ah
 Quallion cells with the following JPL electrolytes:
- 1.2M LiPF6 in EC+EMC+MP (20:20:60) + 4% FEC
- 1.2M LiPF6 in EC+FEC+EMC+MP (10:10:20:60)
- 1.2M LiPF6 in FEC+EMC+MP (20:20:60)
- 1.2M LiPF6 in EC+EMC+MP (20:20:60) + 0.10M LiBOB



C Rate Discharge at -50°C



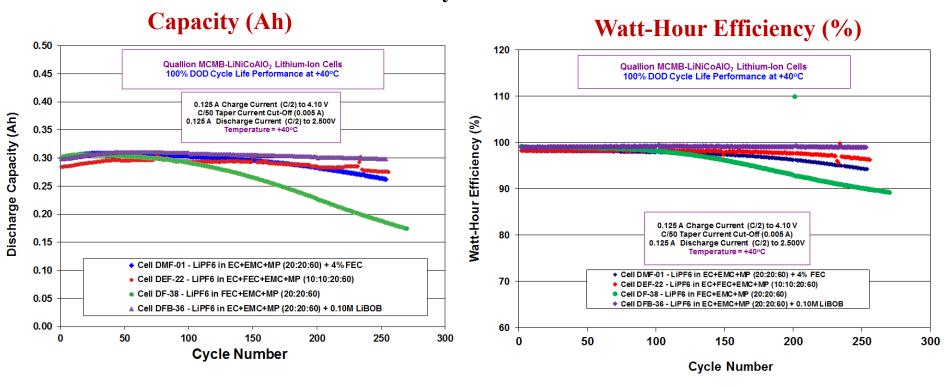
- Electrolytes selected for evaluation include methyl propionate-based electrolytes with increasing FEC content and the use of LiBOB as an additive.
- All electrolytes are observed to provide improved performance at high rates at very low temperatures compared with the baseline electrolytes, with the LiBOB outperforming all formulations.
 - The incorporation of LiBOB has been attributed to increase cathode kinetics at low temperatures,
 - As determined by Tafel Polarization measurements performed on 3-electrode cells.





Cycle Life Performance at High Temperature

100% DOD Cycle Life at +40°C



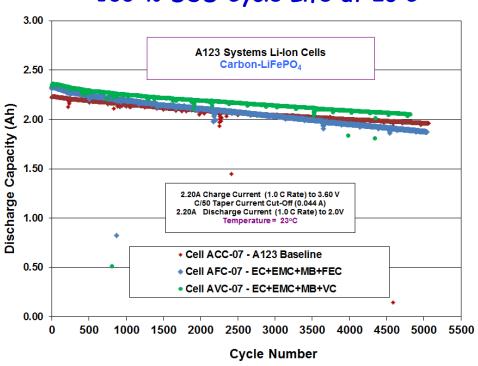
- Thus far, the cells containing the MP-based wide operating temperature range electrolytes provide good life at 40°C, with the LiBOB outperforming all formulations.
 - Increased degradation was observed when FEC was used as the only cyclic carbonate present.
 - Additional life tests will be performed at higher temperatures, as well as with baseline electrolytes.

Technical Accomplishments

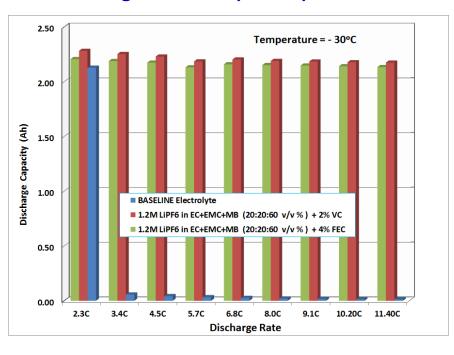


A123 2.20 Ah High Power Lithium-Ion Cells

100 % DOD Cycle Life at 23°C



Discharge Rate Capability at -30°C



- Cells were demonstrated of supporting >11C discharge rates at -30°C, with over 90% of the room temperature capacity being delivered.
- Impressive life characteristics were observed at 23°C, with \sim 5,000 cycles being demonstrated at 23°C (100 % DOD cycling). The methyl butyrate-based electrolytes delivers comparable performance to the baseline electrolyte.

Technical Accomplishments



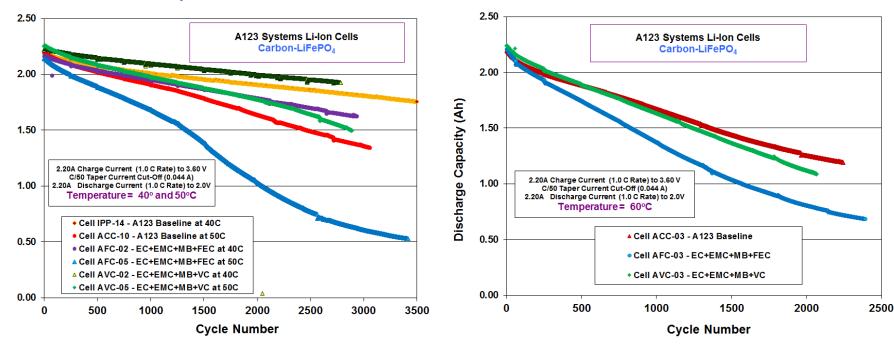
A123 2.20 Ah High Power Lithium-Ion Cells

High Temperature Cycling Performance

100 % DOD Cycle Life at 40-50°C

Discharge Capacity (Ah)

100 % DOD Cycle Life at 60°C



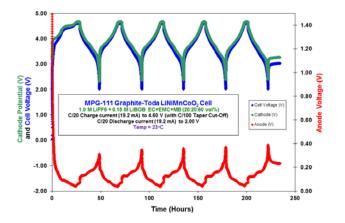
- Good life characteristics were observed at up to 50°C with the EC+EMC+MB+VC system, outperforming the baseline electrolyte with >2,500 cycles being demonstrated.
- Reasonable performance was obtained at 60°C, with cells possessing the EC+EMC+MB+VC formulation displaying increased degradation compared to the cells containing the baseline electrolyte (over 2,000 cycles being demonstrated).



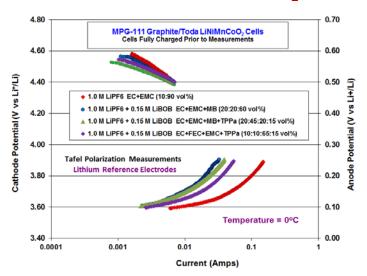


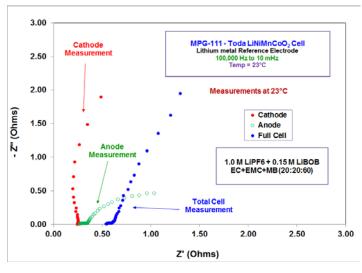
Characterization of Three Electrode MPG-111 Graphite/Toda 9100 LiNiCoMnO₂ Cells

Electrolyte Type	Charge Capacity (mAh/g) 1st Cycle	Discharge Capacity (mAh/g) 1st Cycle	Irreverisible Capacity (mAh/g) (1st Cycle)	Couloumbic Efficiency (1st Cyle)	Charge Capacity (mAh/g) 5th Cycle	Reversible Capacity (mAh/g) 5th Cycle	Cummulative Irreverisible Capacity (mAh/g) (1st-5th Cycle)	Couloumbic Efficiency (5th Cycle)
1.0 M LiPF6 + 0.15 M LiBOB EC+EMC+MB (20:20:60 vol%)	312.06	232.67	79.39	74.56	242.34	238.18	92.53	98.28
1.0 M LiPF6 EC+EMC (10:90 vol%)	318.34	233.51	84.84	73.35	245.63	239.85	107.37	97.65



- Upon performing Tafel polarization measurement on each electrode (which possess heavy loading), it is evident the NMC-based cathode displays poor lithium kinetics (limiting electrode).
- EIS measurements suggests that the charge-transfer kinetics of the cathode contribute significantly to the overall cell impedance and poor rate capability.

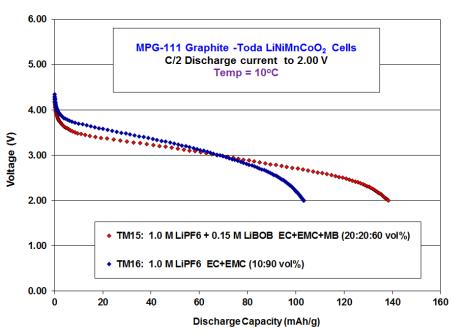








Characterization of Three Electrode MPG-111 Graphite/Toda 9100 LiNiCoMnO₂ Cells



- The MB-based electrolyte displays improved rate capability at low temperature compared to an all carbonate-based formulation.
- Electrochemical measurements suggest that the cathode kinetics is dominating the poor rate capability at low temperature rather then electrolyte type.
- Improved low temperature rate capability will most likely be achieved with lower loading cathode (resulting in diminished specific energy of the cell).

Electrolyte Type			iPF ₆ + 0.15 N C+MB (20:20:		1.0 M LiPF6 EC+EMC (10:90 vol%)			
Temperature	Current (mA)	Capacity (Ah)	Capacity (mAh/g)	Percent (%)	Capacity (Ah)	Capacity (mAh/g)	Percent (%)	
23°C	C/20	0.3898	238.18	100.00	0.3920	239.85	100.00	
	C/10	0.3639	222.36	93.36	0.3718	227.54	94.87	
	C/5	0.3351	204.77	85.97	0.3398	207.94	86.70	
	C/2	0.2815	172.00	72.21	0.2321	142.04	59.22	
10°C	C/20	0.3289	200.97	84.38	0.2998	183.44	76.48	
	C/10	0.3159	193.00	81.03	0.2952	180.65	75.32	
	C/5	0.2841	173.55	72.87	0.2569	157.18	65.53	
	C/2	0.2264	138.36	58.09	0.1688	103.29	43.06	
'0°C	C/20	0.2912	177.92	74.70	0.2680	164.00	68.38	
	C/10	0.2725	166.51	69.91	0.2606	159.49	66.50	
	C/5	0.2393	146.23	61.40	0.2068	126.53	52.75	
	C/2	0.1839	112.35	47.17	0.1247	76.28	31.80	

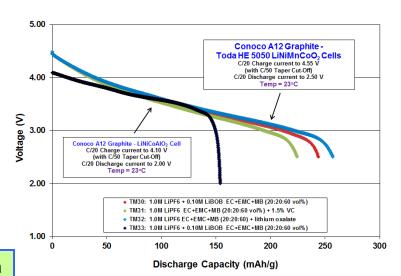


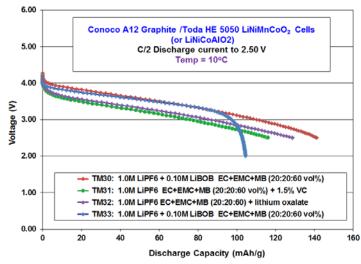


Characterization of Three Electrode Conoco Graphite/Toda HE5050 LiNiCoMnO₂ Cells

Electrolyte Type	Charge Capacity (mAh/g) 1st Cycle	Discharge Capacity (mAh/g) 1st Cycle	Irreverisible Capacity (mAh/g) (1st Cycle)	Couloumbic Efficiency (1st Cyle)	Reversible Capacity (mAh/g) 5th Cycle	Cummulative Irreverisible Capacity (mAh/g) (1st-5th Cycle)	Cathode Type
1.0M LiPF6 + 0.10M LiBOB EC+EMC+MB (20:20:60 vol%)	323.57	246.10	77.47	76.06	243.87	138.98	Argonne NMC (Toda HE 5050)
1.0M LiPF6 EC+EMC+MB (20:20:60 vol%) + 1.5% VC	325.89	233.46	92.43	71.64	224.33	211.93	Argonne NMC (Toda HE 5050)
1.0M LiPF6 EC+EMC+MB (20:20:60) + lithium oxalate	337.29	261.84	75.45	77.63	257.10	125.28	Argonne NMC (Toda HE 5050)
1.0M LiPF6 + 0.10M LiBOB EC+EMC+MB (20:20:60 vol%)	182.62	161.05	21.57	88.19	153.90	91.64	Argonne NCA (LiNiCoAlO ₂)

- A number of methyl butyrate-based electrolytes have been evaluated in the high voltage system involving the LLC-NMC (received from Argonne) and compared with a baseline NCA system.
- The MB-based formulations containing LiBOB delivered the best rate capability at low temperature, which is attributed to improved cathode kinetics. Whereas, the use of lithium oxalate as an additive lead to the highest reversible capacity and lower irreversible losses.
- At lower temperature and higher rates, the advantages of utilizing the high voltage system diminishes, again attributed to the relative cathode kinetics.



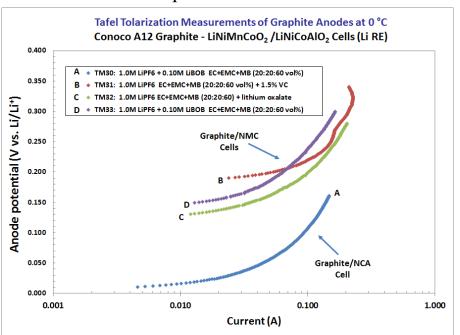




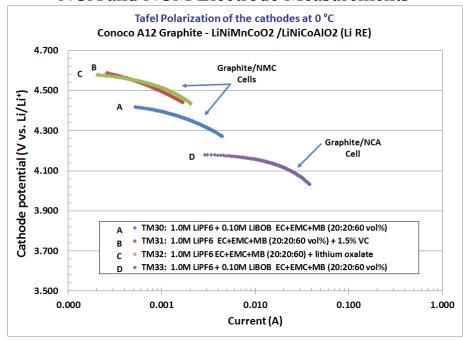
Characterization of Three Electrode Conoco Graphite/Toda HE5050 LiNiCoMnO₂ Cells

Tafel Polarization Measurements at 0°C

Conoco Graphite Electrode Measurements



NCA and NCM Electrode Measurements



- When Tafel polarization measurements were performed each electrode (using 3-electode cells), both the NCA and NMC electrodes displayed poorer lithium kinetics compared to the anode.
- Of the different cathodes, the LLC-NMC electrodes (received from Argonne) displayed much lower lithium de-intercalation kinetics compared to the NCA electrodes (attributed to poor charge transfer resistance of the electrodes), which is exacerbated at lower temperatures.
- Of the electrolytes evaluated, the MB-based system with LiBOB displayed the best cathode kinetics.

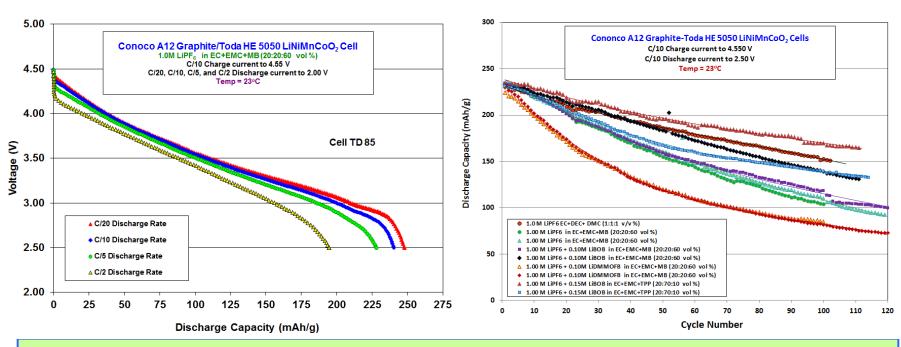
Technical Accomplishments



Characterization of Conoco Graphite/Toda HE5050 LiNiCoMnO₂ Coin Cells

Discharge Rate Capability at 23°C

Cycle Life Performance at 23°C



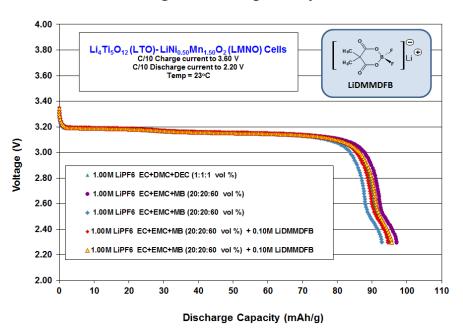
- The discharge rate capability and cycle life performance of a number of cells containing methyl butyrate-based electrolytes was also evaluated at 23°C in coin cells.
- The presence of LiBOB in the methyl butyrate-based systems was observed to improve the cycle life characteristics. However, the use of a lithium malonate-based salt (LiDMMOFB provided by LBNL/URI) did not improve the performance.
- An electrolyte with low flammability containing triphenyl phosphate (developed under a NASA funded program), was observed to provide improved cycle life performance compared to the baseline.

Technical Accomplishments

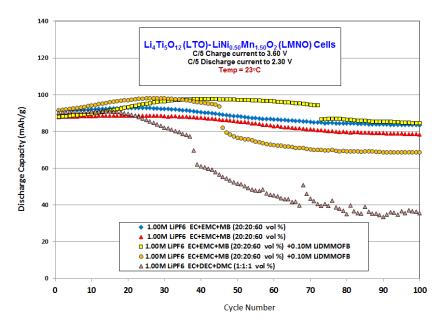


Characterization of Li₄Ti₅O₁₂ (LTO)/LiNi_{0.5}Mn_{1.50}O₂ (LMNO) Coin Cells

Discharge Rate Capability at 23°C



Cycle Life Performance at 23°C

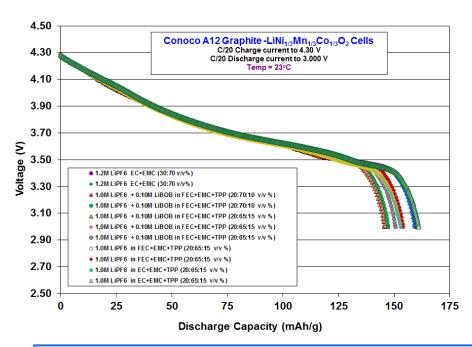


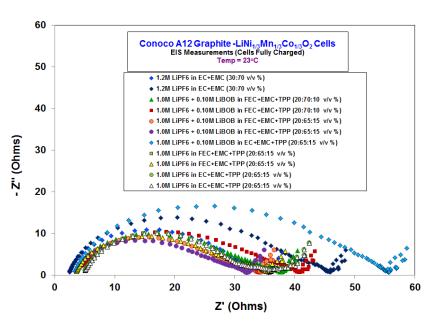
The discharge rate capability and cycle life performance of a number of Li₄Ti₅O₁₂ (LTO) /LiNi_{0.5}Mn_{1.50}O₂ cells containing methyl butyrate-based electrolytes was also evaluated at 23°C.
 In contrast to the LCC-NMC system, the use of a lithium malonate-based salt (LiDMMOFB provided by LBNL/URI) did not adversely affect the cycle life performance, suggesting that the anode interactions influenced the overall performance (i.e., related to possible filming or metal dissolution).





Characterization of Conoco Graphite/LiNi_{1/3}Mn_{1/3}Co_{1/3}O₂ (NMC) Coin Cells





- In collaboration Sandia National Laboratory (Chris Orendorff and Mani Nagasubramanian), we are currently evaluating a number of Conoco Graphite/LiNi_{1/3}Mn_{1/3}Co_{1/3}O₂ cells containing electrolytes with low flammability that were developed under a NASA-funded program.
 - DOE has expressed interest in having Sandia perform safety testing on 18650-cells containing the JPL/NASA developed electrolytes that have been demonstrated to be compatible with NMC-system. Thus far, preliminary results suggest that many triphenyl phosphate-containing electrolytes are compatible with the system, displaying comparable performance to the baseline.

Summary



- Met programmatic milestones for program.
- Demonstrated improved performance with wide operating temperature electrolytes containing ester co-solvents (i.e., methyl butyrate) containing electrolyte additives in A123 prototype cells:
 - Previously demonstrated excellent low temperature performance, including 11C rates at -30°C and the ability to perform well down to -60°C.
 - Excellent cycle life at room temperature has been displayed, with over 5,000 cycles being demonstrated.
 - Good high temperature cycle life performance has also been achieved.
- Demonstrated improved performance with methyl propionate-containing electrolytes in large capacity prototype cells:
 - Demonstrated the wide operating temperature range capability in large cells (12 Ah), successfully scaling up technology from 0.25 Ah size cells.
 - Demonstrated improved performance at low temperature and good cycle life at 40°C with methyl propionate-based electrolyte containing increasing FEC content and the use of LiBOB as an additive.
- Utilized three-electrode cells to investigate the electrochemical characteristics of high voltage systems coupled with wide operating temperature range electrolytes:
 - From Tafel polarization measurements on each electrode, it is evident the NMC-based cathode displays poor lithium kinetics (being the limiting electrode).
 - The MB-based formulations containing LiBOB delivered the best rate capability at low temperature, which is attributed to improved cathode kinetics. Whereas, the use of lithium oxalate as an additive lead to the highest reversible capacity and lower irreversible losses.

Future Work

- Continue the investigation of the use of additives in conjunction with ester-based wide operating temperature range electrolytes.
 - Expand study to include other potential additives.
 - Continue to study the cycle life behavior and high temperature resilience more thoroughly.
 - Correlate trends in electrochemical data with charge/discharge behavior
 - Identify performance limiting processes at extreme temperatures
 - Explore the use of fluorinated esters, especially with high voltage systems, including trifluoroethyl butyrate, ethyl trifluoroacetate, and trifluoroethyl acetate.
 - Continue studying the wide operating temperature range systems in conjunction with ABR developed electrodes, which were recently received from Argonne, focusing upon improving the low temperature capability of these systems.
- Continue the investigation of alternate lithium-based electrolyte salts
 - Continue investigating the use lithium hexafluoroisopropoxide (and other analogues), lithium malonate borate-based salts (in collaboration with Li Yang, John Kerr and Brett Lucht), as well as other LiBOB derivatives to determine their impact in high voltage systems.
 - Explore the utility of these salts in conjunction with alternate solvent mixtures and explore concentration effects.
 - Investigate the impact that these additives have upon the high temperature resilience of lithium ion cells.
- Continue the assessment of candidate electrolytes in high capacity prototype cells.
- Demonstrate the safety performance of JPL-Low Flammability electrolytes in 18650 cells, including ARC testing performed at Sandia National Lab.

Collaborations



- Quallion, LCC: Provided prototype cells, on-going collaborator (NASA SBIR Phase II)
- A123 Systems, Inc.: Provided prototype cells with DOE developed electrolytes, on-going collaborator
- Argonne Nat. Lab (Khalil Amine): ANL provided electrodes for evaluation (on-going collaborator)
- LBNL (Li Yang and John Kerr): LBNL provided novel lithium malonate-based salt
- Yardney Technical Products: Electrolyte development (on-going collaborator)
- Univ. Rhode Island (Brett Lucht): Analysis of harvested electrodes, (on-going collaborator)
- Saft America, Inc.: Collaborator, industrial partner under NASA program
- NREL (Smith/Pesaran): Supporting NREL in model development by supplying prototype cell data.
- Sandia Nat. Lab. (Orendorff/Nagasubramanian): Future safety testing of low flammability electrolyte/cells, electrode source

Publications and Presentations

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