Process Development and Scale Up of Advanced Electrolyte Materials

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Project ID: ES168

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Overview

Timeline

- Project start date: Oct. 2010
- Project end date: Sept. 2015
- Percent complete: on going

Budget

- Total project funding:
  - $1.2M in FY13
  - $1.45M in FY14
    - $1.2M core funding
    - $250K for silicon anode binder work

Barriers

- Cost: Reduce cost to manufacture materials
- Performance: Optimize for highest purity and maximum performance

Partners

- Scaling materials for:
  - General Motors
  - Lawrence Berkeley National Lab
  - Case Western Reserve University
  - North Carolina University
  - Argonne's Applied R&D Group
- Provided materials to:
  - 24M Technologies, Apple, Cidetek, TIAX, QuantumScape, MIT, Army Research Lab, Pacific Northwest National Lab, Sandia National Lab, Argonne's Materials Screening Group and JCESR battery hub

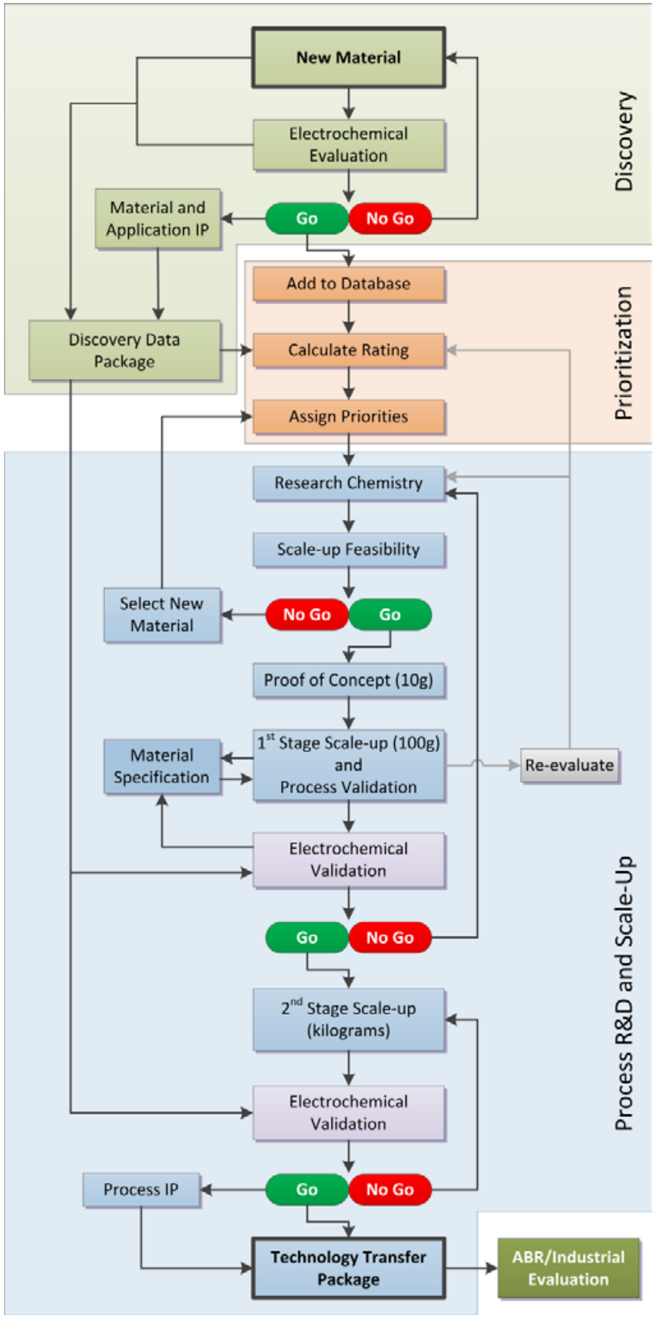
Objectives - Relevance

- The objective of this program is to provide a systematic engineering research approach to:
  - Identify and resolve constraints for the scale-up of advanced battery materials, from the bench to pre-pilot scale with the development of cost-effective process technology.
  - Provide sufficient quantities of these materials produced under rigorous quality control specifications for industrial evaluation or further research.
  - Evaluate emerging manufacturing technologies for the production of these materials.

- The relevance of this program to the DOE Vehicle Technologies Program is:
  - The program is a key missing link between discovery of advanced battery materials, market evaluation of these materials and high-volume manufacturing
    - Reducing the risk associated with the commercialization of new battery materials.
  - This program provides large quantities of materials with consistent quality
    - For industrial validation in large format prototype cells.
    - For further research on the advanced materials.

Approach

- Explore various chemical pathways and determine scale-up feasibility and best scalable route
- Proof-of-concept in a small-scale synthesis (10 g)
- First-stage scale-up and product quality verification, electrochemical performance validation (100 g)
- Develop material specifications that meets electrochemical performance requirements
- Second-stage scale-up and electrochemical performance validation (kilogram scale synthesis)
- Create Technology Transfer Package
- Make fully characterized materials available for industrial evaluation and to the R&D community for basic research with larger samples from a uniform standardized batch

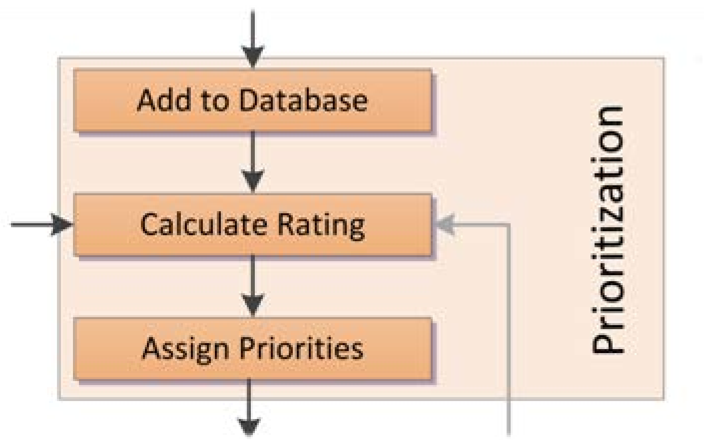


Approach – Materials to Scale

Electrolyte Material	Chemical formula/ full name	Date added to spreadsheet	Organization	IP or patent #	Main Reference	Redox Potential (vs Li^+/Li)	Solubility in Electrolyte (1.2M LiPF6 EC/EMC (3/7 V/V))	Diffusion coefficent ($cm^2 S^{-1}$)	100% Overcharge Cycling Stability	Side Effect Prior Overcharge	Chemical Stability	Ionic Conductivity (in Electrolyte System)	Viscosity (in Electrolyte System)	Applicable Chemistries
ANL-1NM2	$(CH_3)_3SiO(CH_2CH_2O)_2CH_3$	11/1/2010	Argonne National Laboratory	unknown	unknown	4.89	0.9	LiTFSI only	2.5	5	68	0.32	250	$LiMO_2$ (Co, Mn, Ni) $LiMn_2O_4$ $LiFePO_4$
ARL-HFiPP	tri(hexafluoro-iso-propyl)phosphate (C3HF6O)3PO	12/10/2011	Army Research Laboratory	unknown	Cresce & Xu, JES 158 A337 (2011)	unknown	<10%		unknown		Highly Moisture sensitive			LMNO 4.6 V
ARL-PFTBP	tris(perfluoro-tert-butyl)phosphate (C4F9O)3PO	1/12/2012	Army Research Laboratory	unknown	unpublished	unknown	~10%	unknown	unknown	unknown	Moisture sensitive	unknown	Unknown	Protects cathode surface at high potentials
ANL-RS2	2,5-di-tert-butyl-1,4-di-(2-methoxyethoxy)benzene (DBDMB)	11/1/2010	Argonne National Laboratory	ANL-IN-09-082 unpublished	US 2011/0294003	4.00V	0.5M	in progress	200 cycles for $Li/LiFePO_4$ 200 cycles for $Li_4Ti_{12}O_4/LiFePO_4$ 200 cycles for $MCMB/LiFePO_4$	Stable	Stable in air	Excellent	Medium	$LiFePO_4$
ANL-RS21	Confidential - Patent Pending	1/11/2012	Argonne National Laboratory	unknown	unknown	4V	> 0.4M	unknown	150 cycles	stable	stable in the air	excellent	Low	LiFePO4
FRION	Confidential - Patent Pending	2/1/2013	Case Western University	unknown	unknown	unknown	unknown	unknown	unknown	unknown	Moisture sensitive	Excellent	unknown	unknown

Electrolyte Material	Electrochemical Evaluation (Y/N) (attached if Y)	Industrial/Commercial Interest (1-10, best 10)	Material Performance (1-10, best 10)	Intellectual property clear (Y/N)	Scale previously achieved/ commercial (#g or unknown)	Number of Validations/ Syntheses (# or unknown)	Number of Synthetic Steps (# or unknown)	Product commercially available? (Y/N'	Cost (L/M/H)	Safety Hazard (L,M,H)	Raw Materials Readily Availability (Y/N'	OVERALL SCALE SCORE (best score 89)	Commercially available No/Yes (green/red)	Electrochemical Evaluation Yes/No (green/red)	Intellectuall Property Clear (Y/N)	Raw Materials Available (Y/N) (green/red)
ANL-1NM2	Y	8	5	Y	50	2	1	N	L	L	Y	61	No	Yes	Yes	Yes
ARL-HFiPP	Y	10	7	Y	500	5	1	N	L	M	Y	71	No	Yes	Yes	Yes
ARL-PFTBP	unknown	10	7	unknown	5	2	1	N	M	M	Y	57	No	unknown	unknown	Yes
ANL-RS2	Y	9	8	Y	1	5	2	N	L	L	Y	68	No	Yes	Yes	Yes
ANL-RS21	unknown	8	8	Unknown	5	3	1	N	L	M	Y	62	No	unknown	unknown	Yes
FRION	unknown	unknown	unknown	unknown	2	5	unknown	N	unknown	unknown	Y	53	No	unknown	unknown	Yes

- Identify candidate materials of interest
  - Contact ABR/BATT funded researchers
  - Contacted by organizations looking to have a material scaled (GM, Sharp – ARPA-E)
  - Contacted by researchers looking for a material
- Maintain database of the materials
  - Material background
  - Performance characteristics
  - Prioritization criteria
- Prioritize materials – Ranking based on:
  - Level of interest
  - Material performance
  - Readiness to scale



Approach - Milestones

- FY13
  - 4-6 Electrolyte materials to be scaled
    - ANL-RS21
    - ARL-LiPFTB
    - ANL-RS5
    - ANL-RS51
    - CWR-FRION
- FY14
  - 4-6 Electrolyte materials to be scaled
    - LBNL-PEFM
    - LBNL-PFM suite
    - Li-FSI
    - Li-TDI
    - GM-Polymer
- FY15
  - 4-6 Electrolyte materials to be scaled
  - Develop specifications for battery grade materials

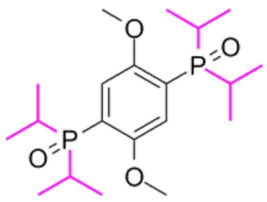
MILESTONE	DATE
ANL- RS21	
Assess scalability of disclosed process	9/28/12
Develop and validate scalable process chemistry (10g scale)	10/30/12
First process scale-up (100g bench scale)	11/30/12
Second process scale-up (1000g pilot scale)	01/10/13
2,320g produced in a single batch, purity >99.5%	
ARL-LiPFTB	
Assess scalability of disclosed process	11/21/12
Develop and validate scalable process chemistry (10g scale)	12/21/12
First process scale-up (100g bench scale)	2/28/13
Second process scale-up (1000g pilot scale)	3/15/13
1,200g produced in a single batch, purity >99%	
ANL- RS5	
Assess scalability of disclosed process	9/28/12
Develop and validate scalable process chemistry (10g scale)	7/31/13
First process scale-up (100g bench scale)	8/30/13
115g produced in a single batch, purity >99%	
ANL- RS51	
Assess scalability of disclosed process	N/A
Develop and validate scalable process chemistry (10g scale)	7/31/13
First process scale-up (100g bench scale)	8/16/13
96g produced in a single batch, purity >99%	
CWU-FRION	
Assess scalability of disclosed process	5/21/13
Develop and validate scalable process chemistry (10g scale)	7/9/13
First process scale-up (100g bench scale)	9/30/13
118g produced in a single batch, purity >97%	

Technical Accomplishments and Progress Overview

- Scalable processes were developed and 5 materials were synthesized.
  - **CWR-FRION** (electrolyte additive) - Case Western Reserve patent pending
  - **ANL-RS5** (redox shuttle) - (2,5-dimethoxy-1,4-phenylene)bis(diisopropylphosphine oxide)
  - **ANL-RS51** (redox shuttle) - (2,5-dimethoxy-1,4-phenylene)bis(diethylphosphine oxide)
  - **Li-TDI** (electrolyte salt) - lithium 4,5-dicyano-2-(trifluoromethyl)imidazol-1-ide
  - **Li-FSI** (electrolyte salt) - lithium bis(fluorosulfonyl)imide
- Work on other materials is in progress.
  - **LBNL-PEFM** (conductive binder)
  - **LBNL-PFM** (conductive binder)
  - **GM-Polymer** (separator modifier)
- Technology transfer packages were created.
- Materials were distributed.
  - Since the program start, 60+ material samples have been sent out. A total amount of around 8,500g of battery grade material has been sampled.

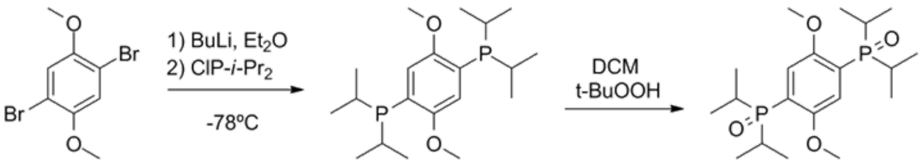
Technical Accomplishments and Progress
Process R&D of ANL-RS5

- Previously scaled ANL-RS2 & RS6 shuttles
  - Bench scale process was suitable only for materials discovery stage.
  - New processes were developed to enable large scale production.



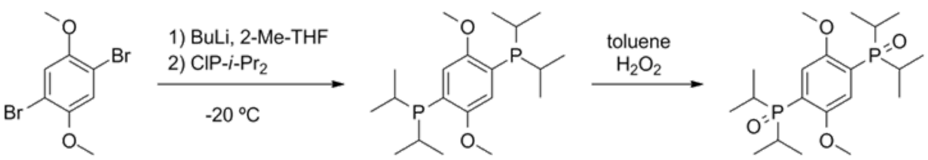
ANL-RS5

- For ANL-RS5:
  - Bench scale process was suitable for small scale only.



ANL-RS5

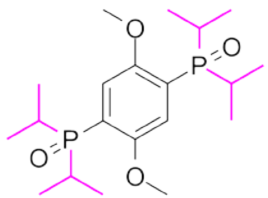
  - Reaction was optimized and simplified, 100 g synthesis was run to secure materials for comparative study – reaction still not suitable for large scale production.



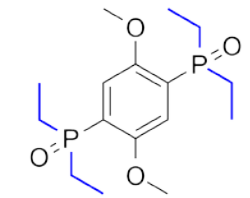
ANL-RS5

  - Alternative reaction paths to synthesize RS5 were needed.

Technical Accomplishments and Progress
Process R&D of ANL-RS5 Analogs

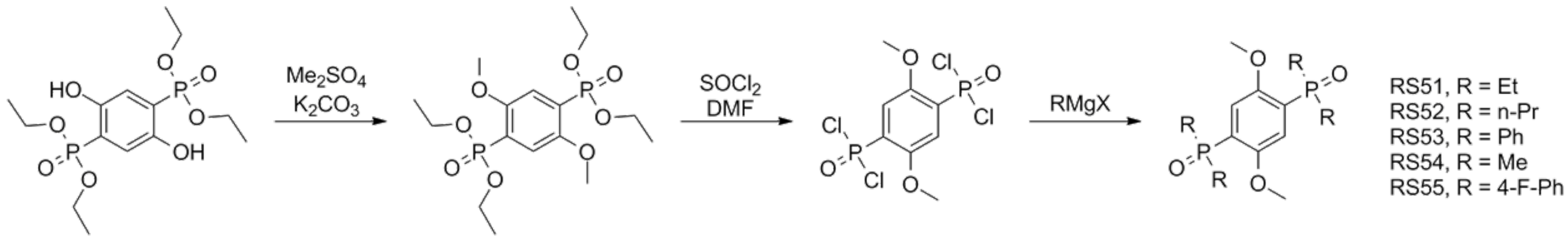


ANL-RS5



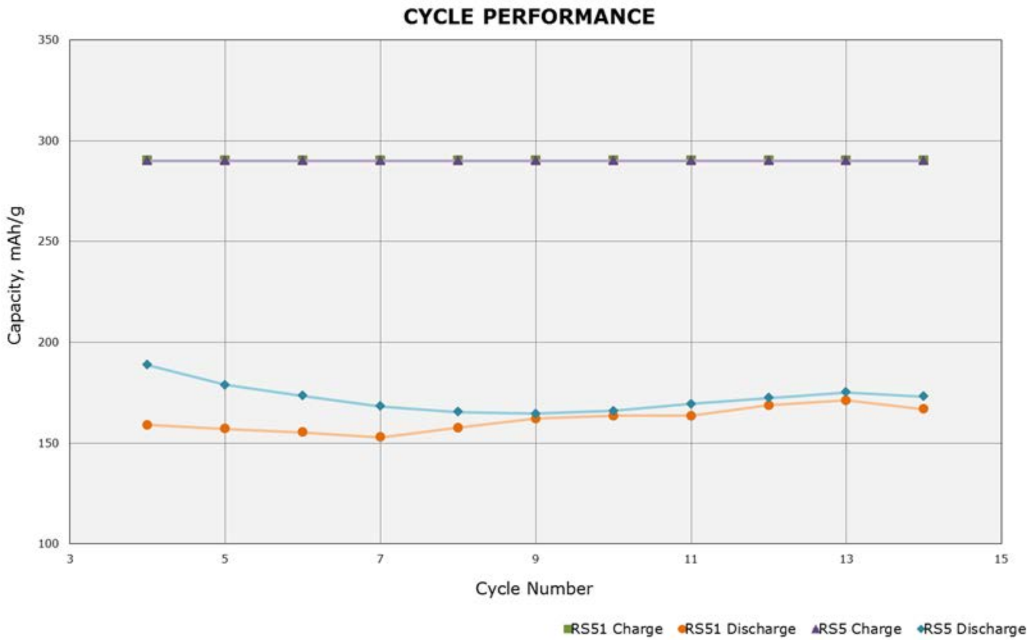
ANL-RS51

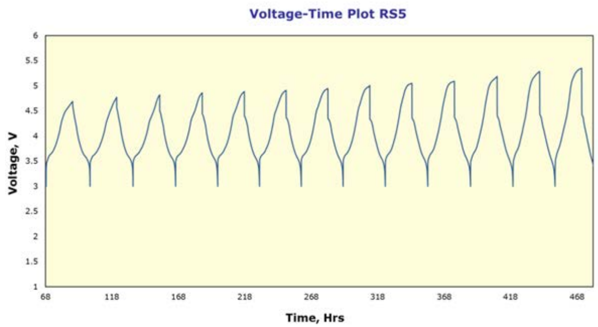
- Evaluated four alternative routes based on analogous chemistry
  - Phosphite addition to benzoquinone
    - Determined to have very low reaction yield
  - Metal catalyzed C-P bond formation/Hirao cross coupling reaction
    - Over 100 parallel reactions – reaction was not sufficiently selective
  - Double lithiation / phosphination of veratrole
    - Over 10 reactions – unable to get sufficient reaction completion
  - Fries rearrangement from phosphorylated hydroquinone
    - Over 80 parallel reactions to map out route and determine working conditions
    - Unable to synthesize RS5! But we were successful in making RS5 analogs
    - Reaction was very scalable, the final step allows for the addition of tunable groups, enabling the synthesis of a suite of shuttles

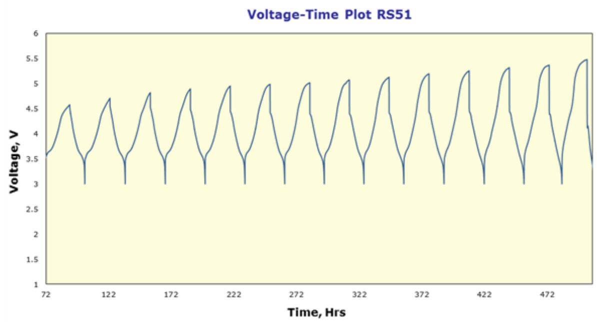


Technical Accomplishments and Progress
ANL-RS5&51 Electrochemical Performance

- RS51 electrochemical performance was comparable to RS5.
- Unexpectedly, RS51 and RS52 show higher solubility in standard electrolyte formulations compared to RS5.
  - RS5 solubility limit in Gen 2 electrolyte = 5%
  - RS51 solubility limit in Gen 2 electrolyte > 15%

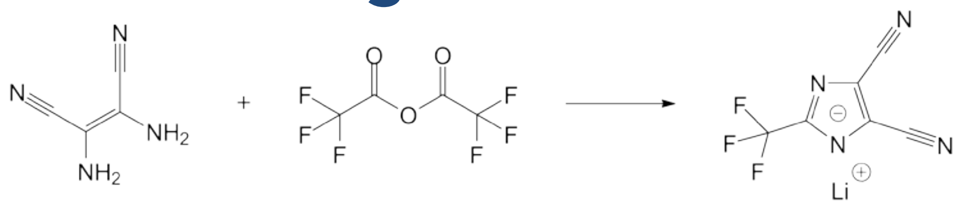






Capacity retention profiles of MCMB/ NCM523 cells containing 5 wt% ANL-RS5 or 5 wt% ANL-RS51 in 1.2M $LiPF_6$ in EC/EMC.

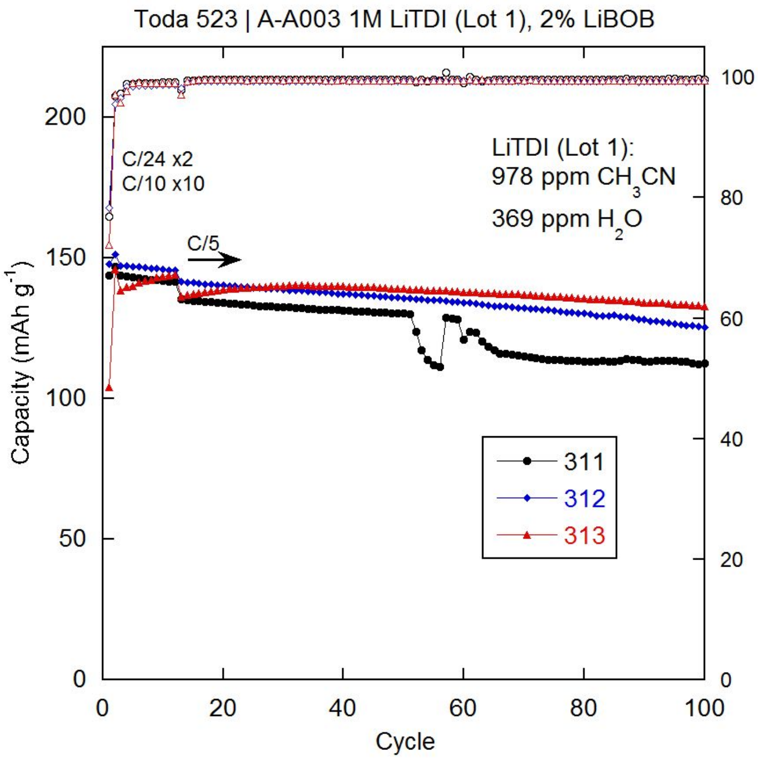
Technical Accomplishments and Progress
Synthesis of Li-TDI

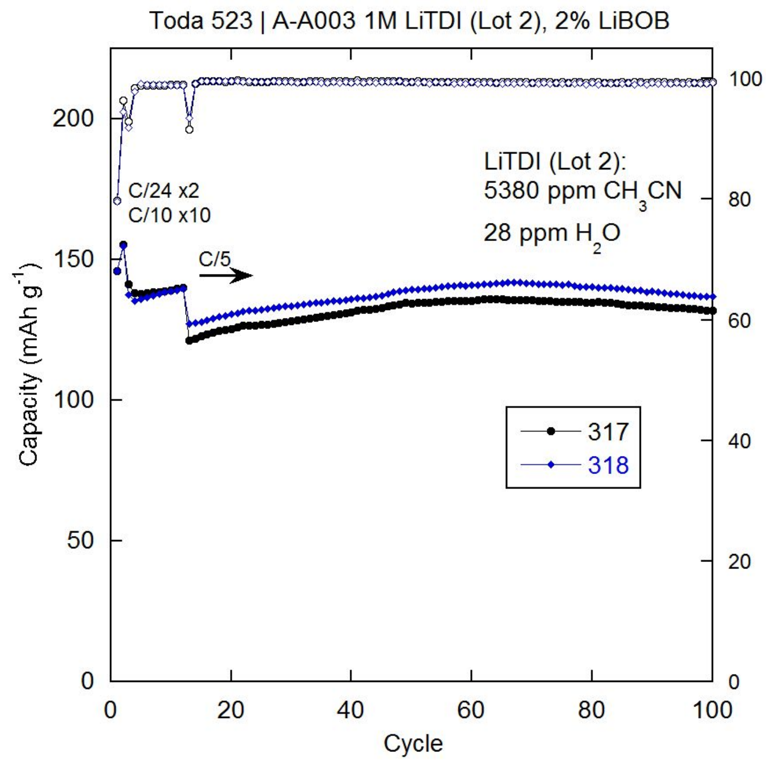


- We received several requests from researchers for Li-TDI which is not commercially available.
- The synthesis was rather straightforward, minor modifications were made to streamline the process:
  - Developed in-process analytical, shortening reaction time by 50%
  - Replaced diethyl ether with MTBE.
  - Modified work-up procedures for shorter exposure time to atmosphere.
- It became evident that slight differences in processing techniques resulted in materials with the same high chemical purity but a different impurity profile and level.
- Unless you were specifically looking for these impurities, they would go unnoticed.

Analysis	Instrument/Method	Results A	Results B
HPLC	Agilent Eclipse Plus C18, 3.5 um, 4.6x100, UV 225, water/ACN gradient	>99.9%	>99.9%
GC/FID	Agilent 7890A Agilent HP-5MS, 0.25 um, 30m x 0.250 mm, 40 to 300 °C, 30 °C /min	978 ppm CH_3CN	5380 ppm CH_3CN
Melting Point	Buchi M-565 Automatic, range method	265-294°C (dec)	265-294°C (dec)
FTIR	Bruker Vertex 70 Attenuated Total Reflection	Consistent with Structure	Consistent with Structure
NMR	Bruker 500 MHz ^{19}F, ^{13}C observed in DMSO-d_6 solution	Consistent with Structure	Consistent with Structure
KF Moisture Titration	KEM MCU-610 Coulometric, WaterMark 1612/1613	369 ppm	28 ppm

Technical Accomplishments and Progress
Electrochemical Evaluation of Li-TDI

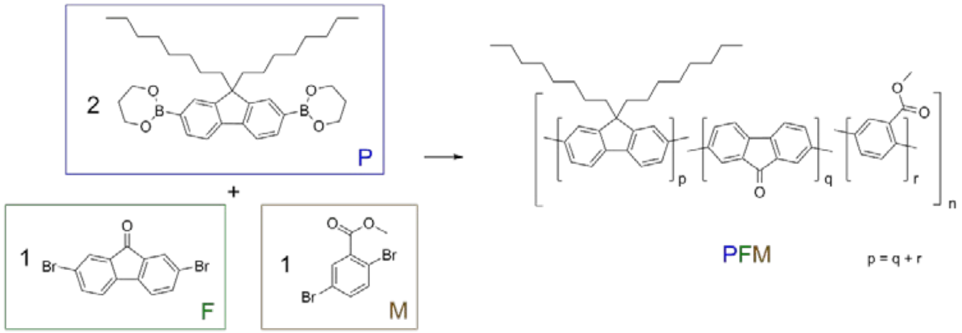




- Performance clearly depends on the trace impurity level!
- Accurate knowledge of the nature and levels of impurities is critical for understanding the material performance.

Materials Cell Cycling:
2032 Coin Cells Charge/Discharge at C/24 x2, C/10 x10 (formation)
Celgard® 2500 Separator Charge/Discharge at C/5
A-C004 (Toda 523 NCM)
A-A003 (CPG-A12)
EC/DEC (3/7 v/v, 15 ppm H2O)
LiBOB (Chemetal) recrystallized from CH_3CN
Analysis by Dennis McOwen, NCSU

Technical Accomplishments and Progress
Process R&D of LBNL-PFM Conductive Binder

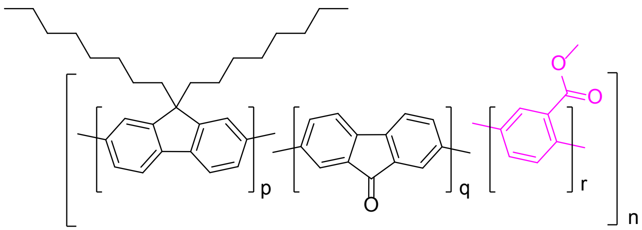


- Synthesis published by Gao Liu et all, Adv. Mater. 2011, 23, 4679-4683
  - Suzuki type co-polymerization in heterophase system catalyzed by Pd(0), 72 hours at 70 °C.
- Several variations of reaction condition were investigated to simplify the procedure while maintaining desired Mn, PDI and purity of the product.

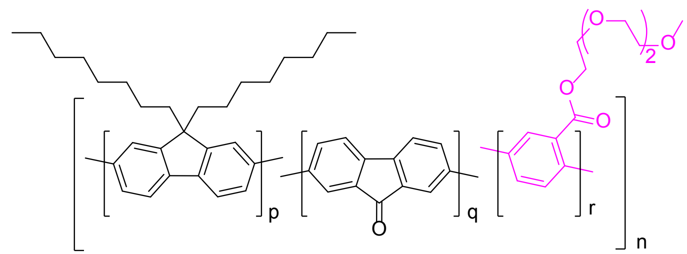
Lot #	Phase Transfer Catalyst	Comments	Polymer Characterization Courtesy of LBNL			
			Mn	Mw	Mp	PDI
PFM, LBNL	Aliquat 336	3rd precipitation, 1/50 ratio, All reagents pre-mixed	36,000	-	-	2.10
KP03018-2	None	2nd precipitation, 1/10 ratio	15,000	37,500	34,500	2.49
KP03018-3	None	3rd precipitation, 1/10 ratio	15,800	39,400	36,400	2.50
KP03028	Aliquat 336	All reagents pre-mixed	28,548	81,761	72,165	2.86
KP03026	Aliquat 336	Slow addition of reagents	32,107	87,078	75,233	2.71
KP03032	Aliquat HTA-1	All reagents pre-mixed	38,197	107,769	86,425	2.82

Technical Accomplishments and Progress
Library of PFM-Type Co-Polymers

- Structurally diverse library of PFM-type co-polymers was synthetized to investigate structure-property relationship.
- All the varied co-monomers were synthetized in-house in a manner that allow for easy scale up.



PFM



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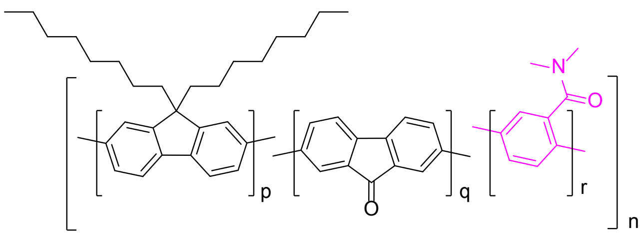

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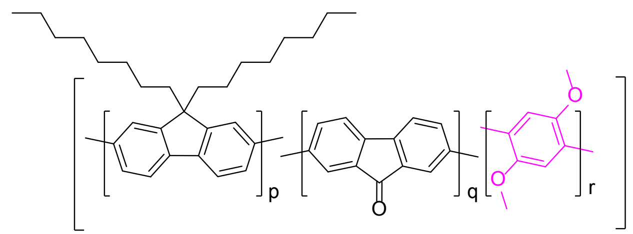

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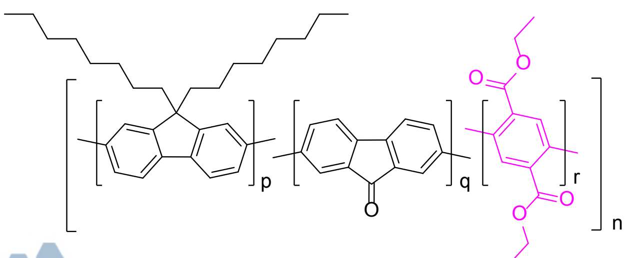

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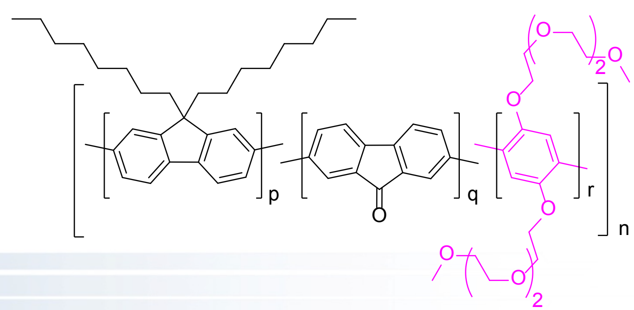


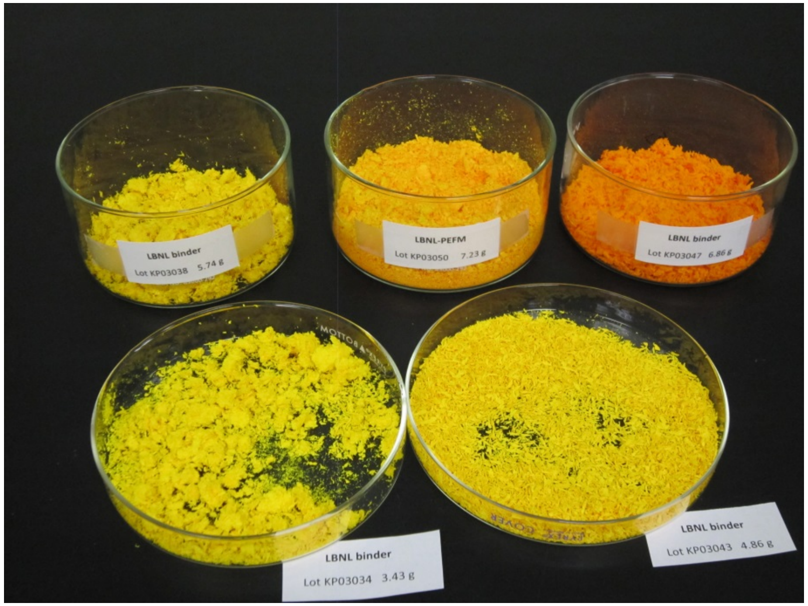
KP03047

Technical Accomplishments and Progress
Library of PFM-Type Co-Polymers

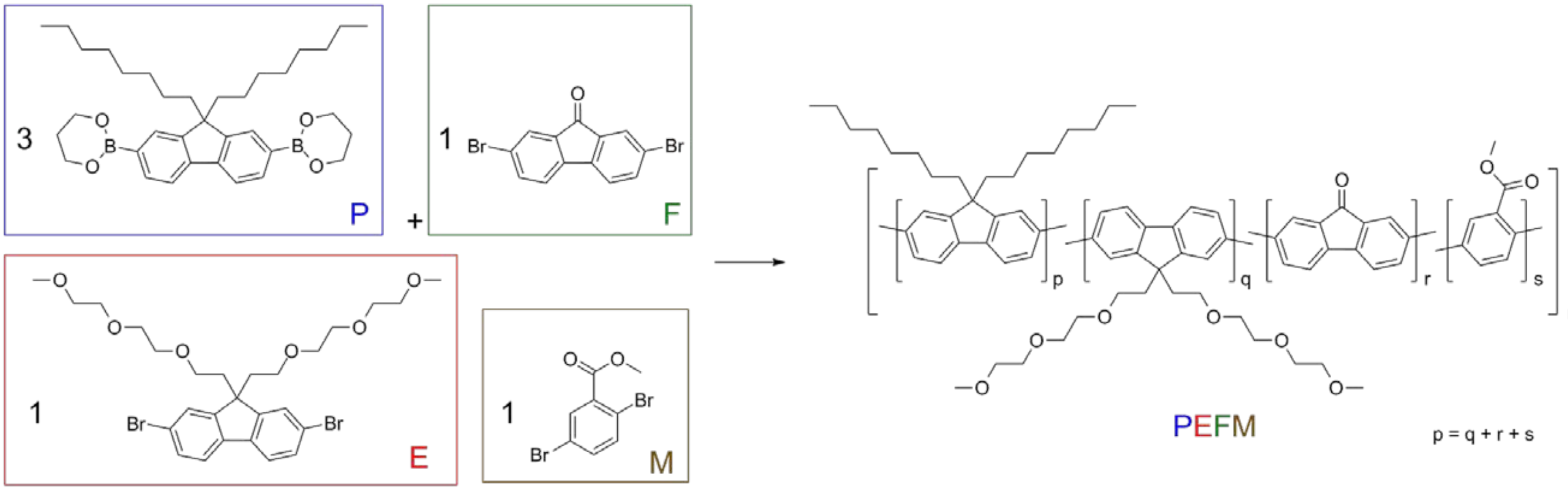
- All the polymeric materials were synthetized in similar condition and all have similar Mn and PDI characteristics.
- The library can be easily expanded to include new materials as needed.
- Samples were submitted for electrochemical testing.

Lot #	Polymer Characterization			
	Mn	Mw	Mp	PDI
PFM, LBNL	36,000	-	-	2.10
PFM, KP03032	38,197	107,769	86,425	2.82
KP03030	37,969	96,586	87,259	2.54
KP03034	33,708	82,351	74,841	2.44
KP03038	38,243	99,272	92,830	2.60
KP03040	23,344	71,718	59,659	3.07
KP03043	32,150	80,013	72,792	2.49

Analytical data courtesy of LBNL



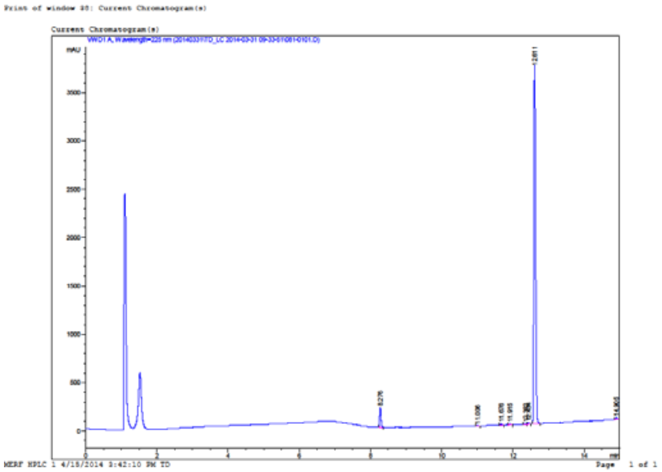
Technical Accomplishments and Progress
Process R&D of LBNL-PEFM Conductive Binder



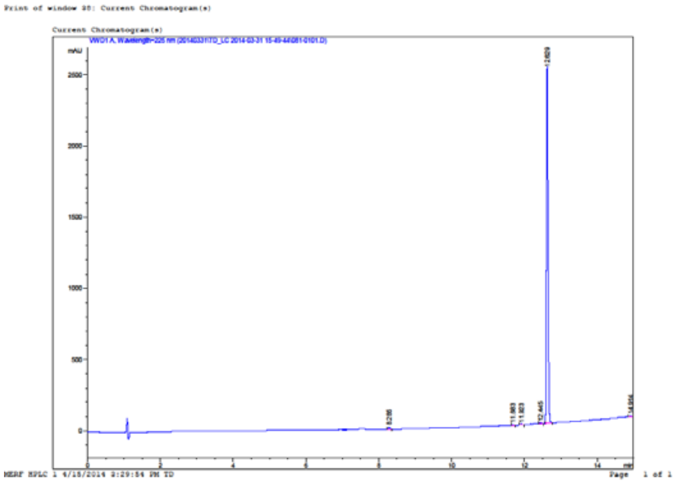
- Synthesis published by Gao Liu *et all*, J. Am. Chem. Soc., 2013, 135, 12048
  - Suzuki type co-polymerization in heterophase system catalyzed by Pd(0), 72 hours at 70 °C.
  - All reagents for the synthesis of PEFM are commercially available – except for "E" (2,7-dibromo-9,9-bis(2-(2-(2-methoxyethoxy)ethoxy)ethyl)-9H-fluorene) .
- Bench scale synthesis of "E" require chromatograph purification, yield 60-80 %.
- Chromatography is not suitable for large scale synthesis!

Technical Accomplishments and Progress
Scalable Procedure for Preparation of "E"

- A new process was developed to synthesize co-monomer "E"
  - THF/potassium t-butoxide was found to be the optimal base/solvent .
  - Replacement of sodium hydride with potassium t-butoxide eliminates hydrogen gas generation.
  - The temperature of the process was lowered from reflux to room temperature.
  - Small excess of the tosylate is used to achieve complete conversion eliminating the undesired mono-alkylated product.
  - The leftover tosylate is hydrolyzed by addition of methanol/sodium hydroxide.
- The material does not require any further purification (> 90% yield and >95% purity).



Crude reaction mixture before hydrolysis



Crude "E" after aqueous work up, >95%

Technical Accomplishments and Progress
Synthesis of LBNL-PEFM

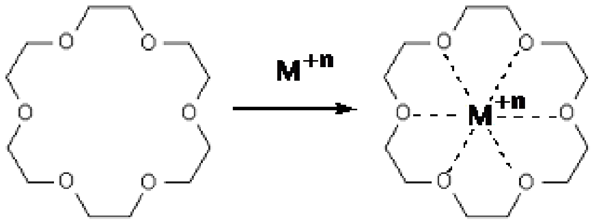
- Various samples of the polymeric material were submitted for electrochemical validation.
- Lot KP03050 was prepared using MERF developed, scalable procedure for synthesis "E" co-monomer.
- Work continues to optimize Mn and PDI.
- Plan to synthesize a kilogram batch for distribution.

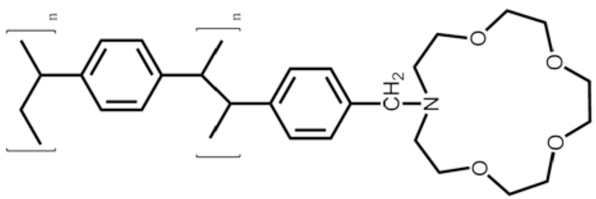
Lot # KP03050

Lot #	Phase Transfer Catalyst	Comments	Polymer Characterization			
			Mn	Mw	Mp	PDI
PEFM, LBNL	Aliquat 336	Published data	34,000	78,200	-	2.30
KP03010	None	All reagents pre-mixed	10,900	22,200	18,900	2.02
KP03012	None	Slow addition of reagents	18,400	51,300	41,700	2.79
KP03050	Aliquat 336	All reagents pre-mixed	22,220	60,606	43,559	2.73
Pending	Aliquat 336	Slow addition of reagents	-	-	-	-
Pending	Aliquat HTA-1	Slow addition of reagents	-	-	-	-

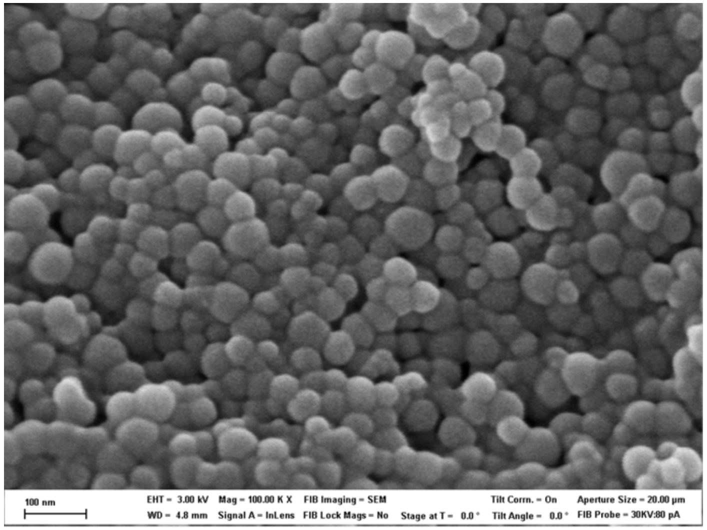
Analytical data courtesy of LBNL

Technical Accomplishments and Progress
Process R&D of GM Separator Modifier





- Concept: to use tethered chelating agents (crown ethers, cryptands, etc.) to trap (“chelate”) transition metal cations that leach from the positive electrode.
- Polymeric, non soluble material is to be attached to or incorporated into the separator.
- Process R&D work has recently begun:
  - Assess scalability of disclosed process
    - Completed
  - Develop and validate scalable process chemistry
    - In progress



Response to Previous Year Reviewer' Comments

- **"More insight into how new materials are identified and introduced to the process would be helpful"**
  - Response: The database of potential materials to scale is populated using various sources; information from current BATT and ABR programs, direct request from researchers and industrial organizations. We developed a set of criteria and a numerical value is assigned to each material. We carry out process R&D and scale up for the material with highest overall score. The set of the criteria covers various aspects ranging from scale up feasibility, electrochemical performance and level of interest from researchers and industrial organizations.

- **"With the level of funding and the simplicity of most of the synthetic routes shown, one might expect more materials to have been screened and scaled"**
  - Response: While the reaction equation may looks simple, the process to develop the optimal condition to enable large scale synthesis matching discovery stage material properties is very time consuming and complex. Bench scale processes and conditions typically can't be used for large scale production. Our goal is to develop scalable, economical, industry-ready process to manufacture battery grade material on a large scale.

- **"Would like to see more outreach to industrial partners"**
  - Response: We have increased our efforts to promote the capabilities of this program and materials available through conferences, a new website (www.anl.gov/merf)and through Argonne's tech transfer office who has been actively promoting to industry. Since the last AMR meeting we have scaled materials for or provided samples to GM, Sharp America, TIAX, 24M Technologies, Cidetek and QuantumScape.

Collaborations

- Materials process R&D:
  - General Motors (Bob Powell)
  - Lawrence Berkeley National Lab (Gao Liu)
  - Argonne National Lab (John Zhang)
  - Case Western University (Dan Scherson)
  - North Carolina University (Wesley Henderson)
- Materials provided for further research:
  - Army Research Lab (Kang Xu)
  - Pacific Northwest National Lab (Wu Xu)
  - Sandia National Lab (Chris Orendorff)
  - JCESR (Lu Zhang)
  - TIAX (Brian Barnett)
  - MIT (Fikile Brushett)
- Electrochemical evaluation of scaled materials
  - Argonne's Materials Screening Group (Wenquan Lu)
  - Argonne's CAMP facility (Andrew Jansen)

Remaining Challenges and Barriers

- New battery materials are continually being discovered and developed.
- There is a strong demand from the research community for high quality experimental materials in quantities exceeding bench scale synthesis.
- A detailed understanding of impurity profiles of experimental materials used in the battery community is needed.
- Battery grade specifications are needed for newly developed battery materials to fully understand cost and capabilities.
- Emerging manufacturing technologies need to be evaluated to further reduce production costs of battery materials.

Activities for Next Fiscal Year

- Manage battery materials database and populate with new candidates of materials of interest to the ABR program.
  - Rank and prioritize the materials.
- Develop scalable process for 4-6 materials and produce kilogram quantities for sampling.
  - Develop scalable process, analytical methods and quality control procedures.
  - Validate the manufacturing process, analytical and electrochemical properties and characterize the impurity profile.
  - Create Technology Transfer Packages for industry.
  - Supply material samples to the research community and industry for evaluation.

Summary

- This program has been developed to provide a systematic approach to process R&D and scale-up, and to provide sufficient quantities of advanced battery grade materials for industrial evaluation.
- Argonne's process R&D program enables industry to carry out large-scale testing of new battery materials and enable scientists to obtain consistent quality, next generation materials for further research.
- Integration of materials discovery with process R&D will expedite the time needed to commercial deployment.
- **Over 60 samples have been presented to collaborating research entities**
- **Since the last AMR Meeting, processes for 5 additional battery materials were successfully developed and materials were produced.**

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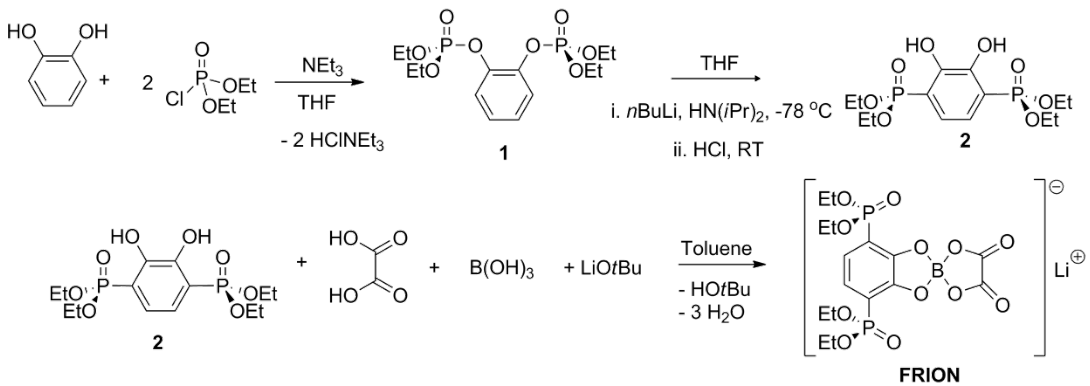
- General Motors
  - Bob Powell
  - Ion Halalay

Technical Backup Slides

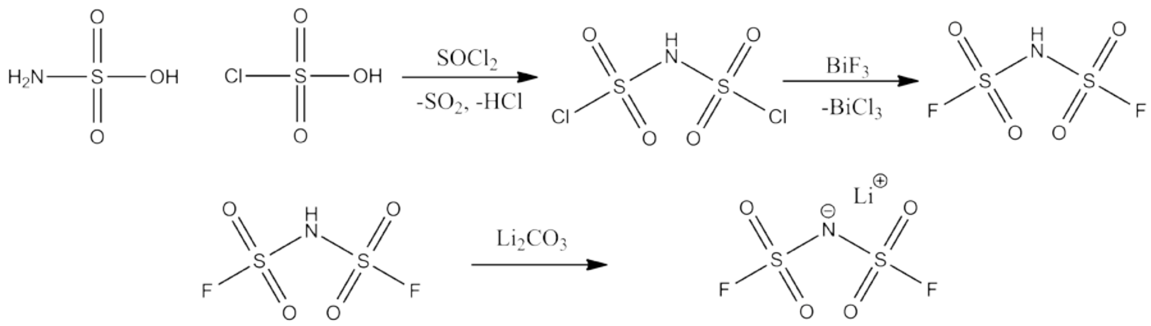
Technical Accomplishments and Progress
Process R&D of CWR-FRION



- FRION is phosphoryl-rich Flame Retardant ION developed by Case Western Reserve for the BATT program.
- Synthesis Modifications:
  - Replaced butyl lithium with commercial non-pyrophoric lithium diisopropylamide solution.
  - The ratio of LDA to substrate was optimized: dramatically simplified work-up procedure.
  - Removed halogenated solvents from the process.
  - No cryogenic reactions.
  - In-process tests developed: all reactions can be followed by HPLC.
- Material is being evaluated at LBNL and SNL in full cell tests.

Analysis	Results
HPLC	>97%
Melting Point	238-241°C
NMR	Consistent with Structure

Technical Accomplishments and Progress
Synthesis of Li-FSI



- Li-FSI is a promising salt candidate with several unresolved issues:
  - Corrosion of Al collector- possibly due to trace (<10 ppm) residual impurities.
  - "Difficult to prepare this salt with high purity in an unspecialized laboratory"[1]
  - Several competing routes are used, exacerbating the purity question.
- Our **ongoing** investigations have found additional scale-related complications due to:
  - Drying of bulk material.
  - Hydrolysis of bulk material.
  - Processing steps that are impractical on large scale.
- Current work is focused on the preparation and trace impurity analysis to better understand contamination/performance relationship.

[1]Hong-Bo Han et. al. Journal of Power Sources 196 (2011) 3623–3632.

Materials Scaled

Solvents	Shuttles	Salts	Additives	Binders
ANL-1NM2	ANL-RS2	Li-DFOB	ARL-HFiPP	LBNL-PFM
ANL-1NM3*	ANL-RS5	Li-TDI	ARL-LiPFTB	LBNL-PEFM
ANL-2SM3*	ANL-RS6	Li-FSI	CWR-FRION	
ANL-1S1M3	ANL-RS21			
	ANL-RS51			
* Out of stock				

For samples and information:

www.anl.gov/merf