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U.S. Army Research, Development and Engineering Command

# Pure Form of LiBOB Salt and the Purification Process Producing Such Form



***TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.***

**Inventor: Dr. Kang Xu**

ARL 09-33

APPROVED FOR PUBLIC RELEASE

February 16, 2011

The invention describes the synthesis and purification of a new lithium salt, bis(oxalato) borate (LiBOB).

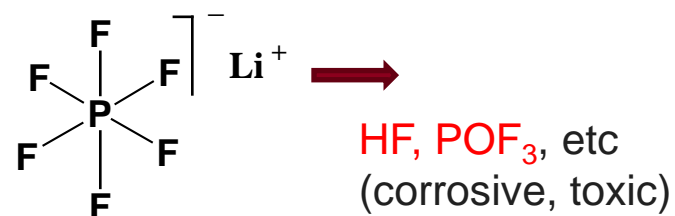
Due to inherent limitations, there is interest in replacing LiPF<sub>6</sub> salt. LiBOB is viewed as a good option because:

- No P-F bond, does not attack organic components
- Does not decompose thermally into HF (as LiPF<sub>6</sub> does); CO<sub>2</sub> as benign products

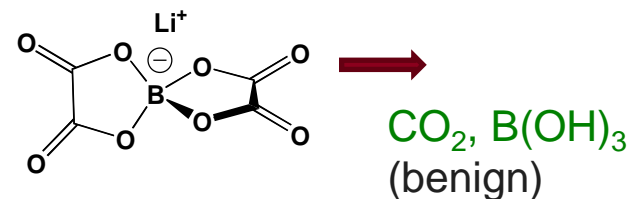
*However, "purified" LiBOB is required to maximize performance benefits. This is currently difficult and expensive to achieve.*

❖ The core technology provided by this invention is the purification procedure, the quality-control standard and the resulting pure form of LiBOB obtained from this process.

❖ This pure form of LiBOB is a distinct compound as compared with other available commercial products.



SOA Electrolyte contains **LiPF<sub>6</sub>**

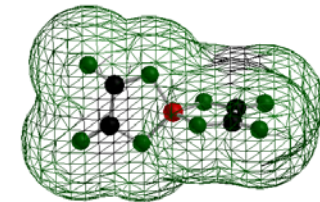


Thermally Stable Electrolyte contains **LiBOB**

## The innovation of preparing pure form of LiBOB and the QC

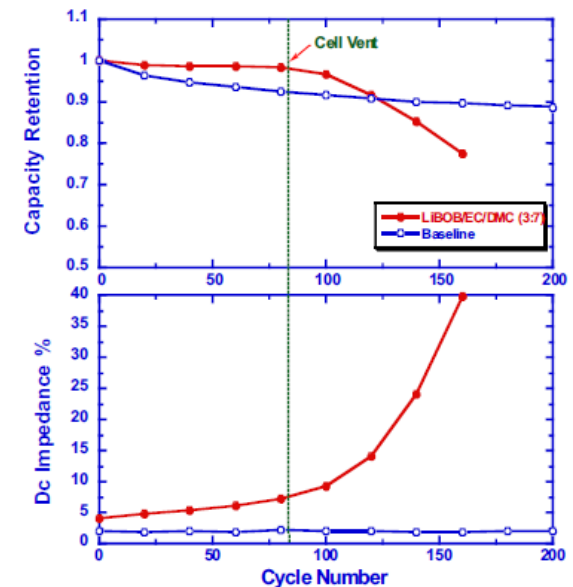
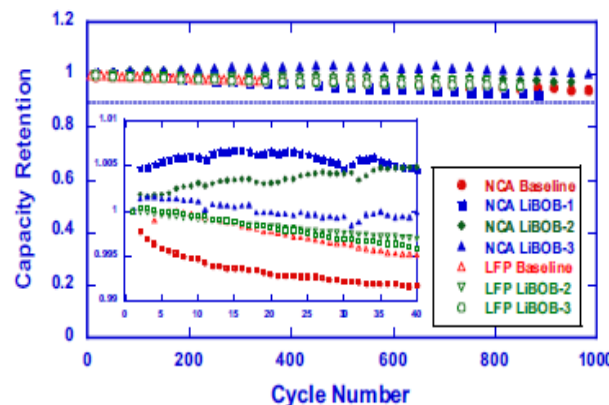
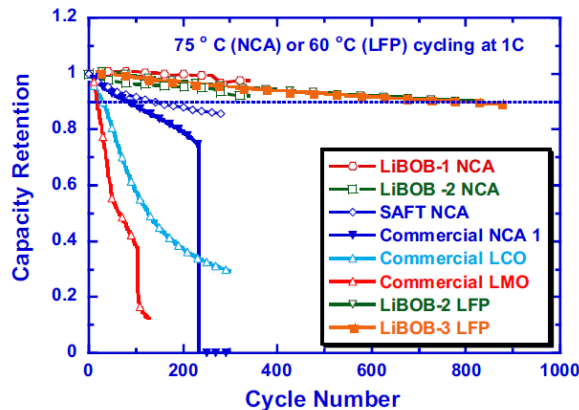
- Impure LiBOB from commercial source cannot support high temperature operation
- The pure form of LiBOB can support Li ion batteries operating at elevated temperatures up to 80 °C
- It also improves safety under abusive over-charge and high-temperature storage

LiBOB



Impure LiBOB does not support HT operation

Pure Form of LiBOB supports HT operation of Industry Li Ion Cells for > 1000 cycles



High temperature stability is critical for battery packs in electrified vehicles

- SOA electrolyte fail to do so
- Become dangerous over 60 °C due to HF production

The pure form LiBOB can widen service temperature range of Li ion batteries

- Dramatically improves capacity retention at both room and high temperature up to 80 °C
- Significantly reduces cell impedance

[Video - SOA Electrolyte \(LiPF<sub>6</sub>\) w/o LiBOB](#)

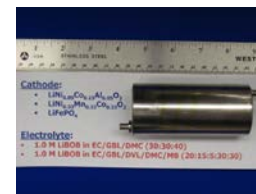
[Video - Electrolyte with pure LiBOB](#)

The invention of the process provides easy production of high purity of LiBOB and its effective Quality Control

Safety advantage over SOA electrolytes

- LiBOB allows large format Li ion cells with higher safety than SOA electrolyte salt LiPF<sub>6</sub>
- Higher stability for both over-charge and HT abuses

8 Ah Li ion cell



Battery Pack in Prius

This invention holds a number of advantages over the current state-of-art:

- Enables the high temperature application of Li ion battery
  - Demonstrates excellent stability at high temperature; up to 80°C
- Is well suited for harsh environments of Hybrid Electric Vehicles (HEV)
- Provides superior performance vs. existing commercial LiBOB; maintains 95% energy density after 1,000 cycles
  - at 75 °C capacity retention ~90% at 1000<sup>th</sup> cycle while most SOA failed before 400<sup>th</sup> cycle
  - at 60 °C capacity improved by 15% vs. SOA at 2000<sup>th</sup> cycle
- Establishes purification process and standard; nearly 100% pure
- Improves safety of Li ion battery under both over-charge and HT abuses
- Open system accommodates a variety of cathode chemistries

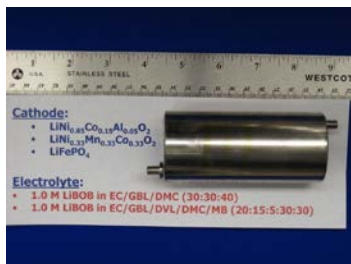
## Method of preparation of these novel additives



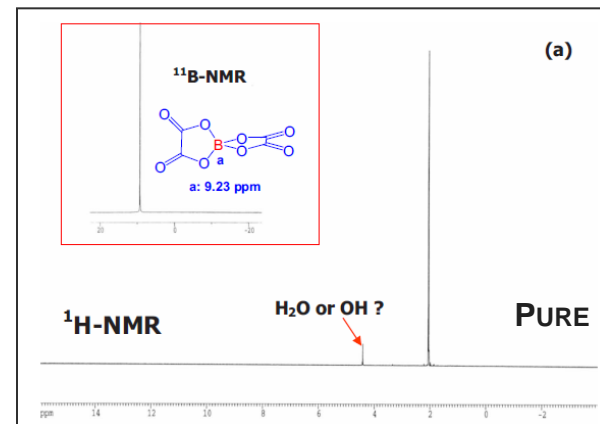
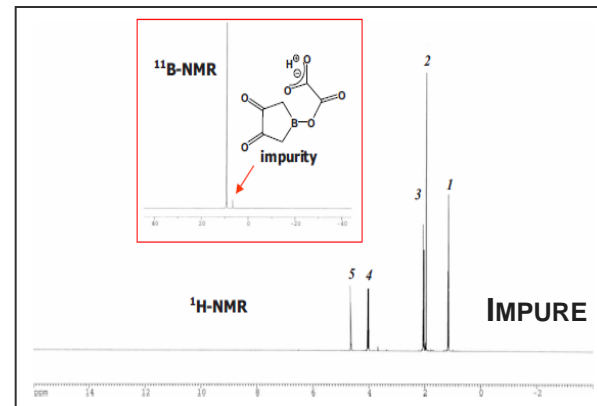
Evaporation/Precipitation  
Recrystallization



Soxhlet Extraction



Coin Cells  
Industry Cells (8 Ah)



Structural characterization/QC



- Military hybrid electric vehicle applications to reduce fuel consumption and reduce the need for dangerous logistical refueling operations
- Soldier Power in hot climate
  - Battery life significantly improved

**80°C = 176 °F**







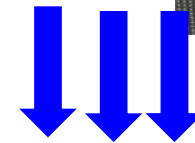
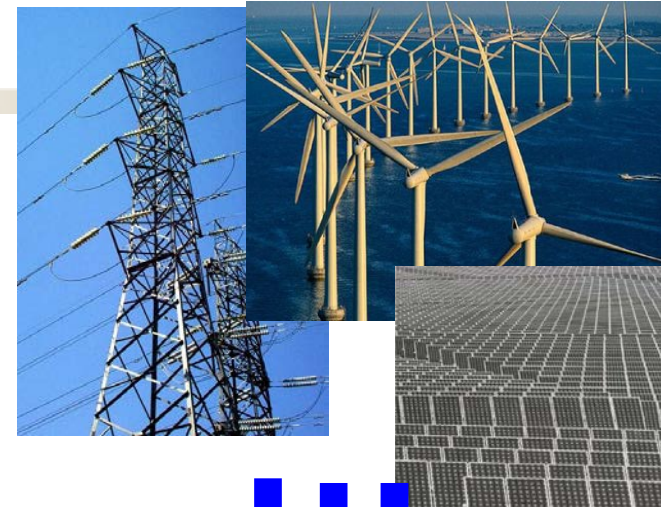
## Two major markets

- Electric Vehicle, Hybrid Electric Vehicle
- Large scale stationary energy storage

The invention provides high temperature stability of Li ion battery.

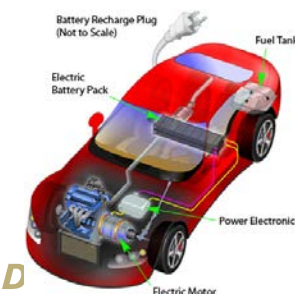
In particular, the invention benefits Li ion battery high temperature applications/environments such as those found in hybrid electric vehicles (HEV).

The purification method developed is also useful for producing other salts that have the BOB anion, such as NaBOB,  $\text{Mg(BOB)}_2$  or other metal salts as additives, ionic liquid for double layer capacitors and batteries, etc.



Energy Storage for Grid Stabilization

Electrified Vehicular Power Systems





# Technology Agreements



A patent license and CRADA is sought.

The current technology is TRL 6 and will benefit from a collaboration between the inventor team and the commercialization partner in order to speed the development to the market. This would most readily be done through a license agreement/CRADA.

A provisional patent application has been filed.