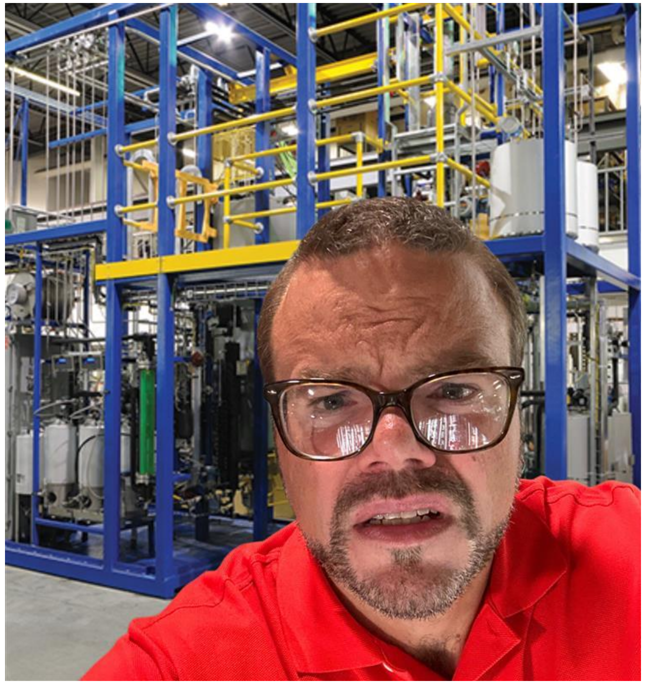
Heat of Mixing and Me – Tools for making life a little easier.

A Reactive Chemicals Story



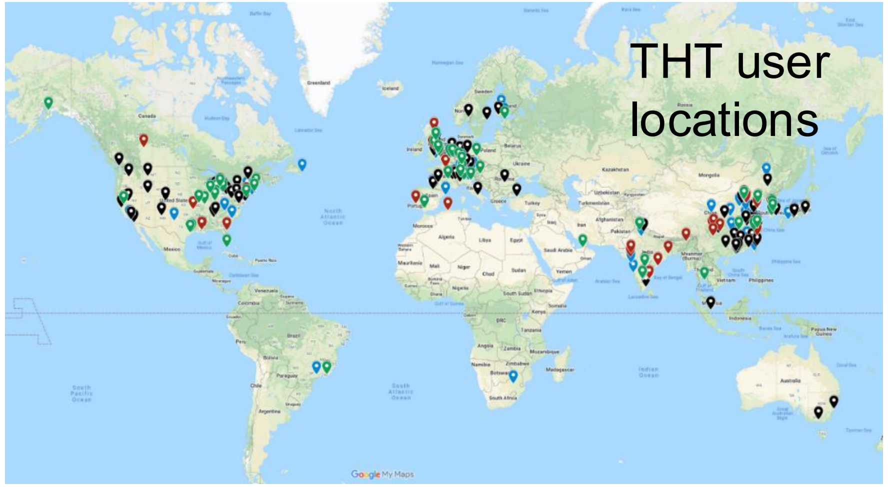
Introduction to THT

- Thermal Hazard Technology (THT) is a manufacturer of specialized calorimeters for the chemical and lithium battery industries

- THT was founded in the late-90s with 4 employees. Prior to this the founders worked as distributors for the original Columbia Scientific Industries CSI-ARC outside North America.

- THT's headquarters located in the village of Bletchley, located in Milton Keynes UK and currently employs 50+ staff members. THT also has sales offices in India and the USA, with distribution support worldwide.

Judges Scientific plc



- In May 2020, THT was acquired by Judges Scientific plc. The THT name is retained but the company is now part of the Judges group.

- In 2023, THT moved to a new dedicated 15,000 sq. facility (still in Milton Keynes UK)

- Coming Soon in 2025 – Testing and support/service facility located in Charlotte, NC.

I wonder what exciting problems ill get to solve today?!?!

Uhhh…So….the OCEAN TANKER filled with our products has indicated that our material is slowly heating up while in transit…in the middle of the ocean. (TRANSPORTATION)

Uhhh…So….the WAREHOUSE storing our product caught fire. We had stacks of pallets containing organic materials arraigned tightly together in a <u>very</u> organized fashion…in the summer. (PACKAGING & STORAGE)

Uhhh…So….the R&D Manager wants to advance this project by increasing the synthesis capacity of our new material as soon as possible. There is a “MEETING” in a couple weeks and they want this milestone met… It involves dramatically higher volumes of hazardous solvents. (R&D SCALE)

Being in Process Safety also means emergency response.

These are some examples of the REAL problems we face…

Its not always a chemical plant or production facility that has the process safety event.

I ran a DSC, but it was a heterogenous mixture and was very difficult to load a representative sample. Data was useful but we are still worried there could be more activity we are missing.

A variety of testing needs typically found across your business...

R&D would like to mix Chemical A & B on a heated stir plate. We know they have some potential for heat generation based on functional groups but unsure on extent.

Reaction Engineering has taken over the project and is ready to scale project from the bench to a 500ml sealed glass reaction vessel. How much heat and pressure will be generated during the various additions?

The PHA just identified hazards related to potential mixing of fluids from post-cleanout pressure vessel heels when not mitigated or dried properly.

If we accidently load the wrong solvent in the reaction mixture, what will happen?

As others mention, there are a number of excellent calorimeters to help answer simple and complex thermodynamic problems. We consider our various tools like DSC, TSu, ARC, VSP, Reaction Calorimeters, among others. All with advantages!

Uhhh…So… a low-cost screening version of a reaction calorimeter would be nice.

µRC micro Reaction Calorimeter

THT

Temperature:
-20 to 170°C
Peltier based

Modes of operation:
Isothermal
Scanning
Titration
Heat capacity

Scanning rate:
0.05°C/min to 2.0°C/min

Baseline noise:
From 5µW

Dynamic range:
5µW to 700mW

Stirring Speed:
0 – 400rpm

Cell Volume:
1.5ml

Cell Type:
Removable glass vial, High pressure steel vial

Injection Volume:
1 to 250µL (0.1µL/sec to 25µL/sec)

Measurement Principle:
Power compensation

Connection to PC:
USB / Windows software

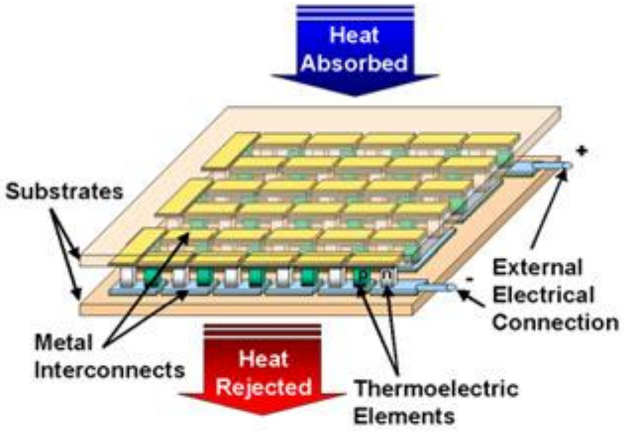
Footprint:
15 x 40 x 35cm

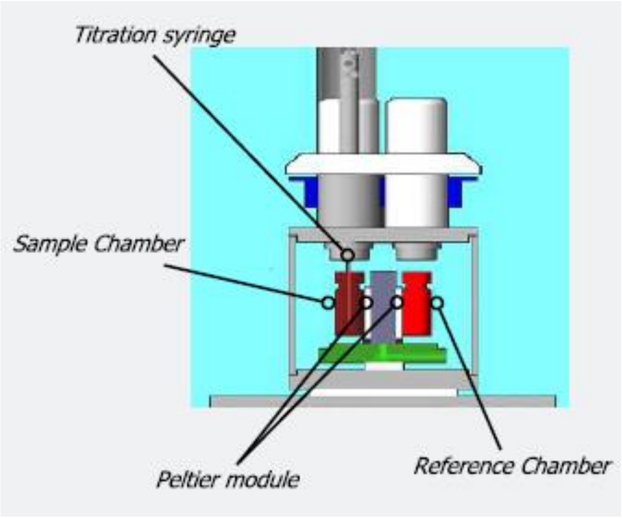
Titration Calorimetry
Isothermal Calorimetry
Scanning Calorimetry
Pressure Measurement
ALL IN ONE INSTRUMENT

https://www.thermalhazardtechnology.com/contentfiles/downloads/32.pdf

Measurement principle

Peltier Module

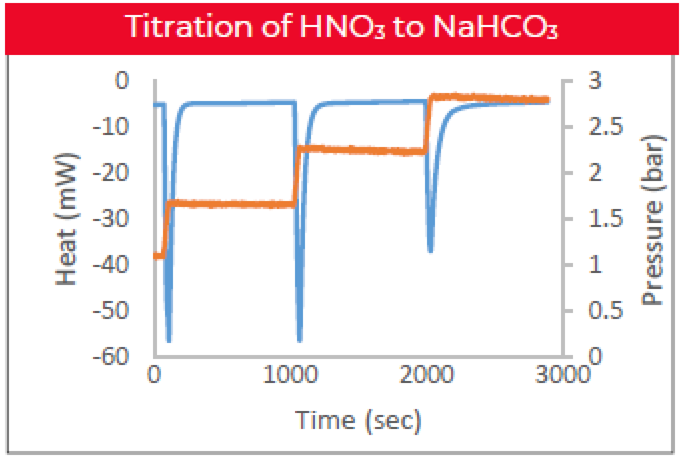


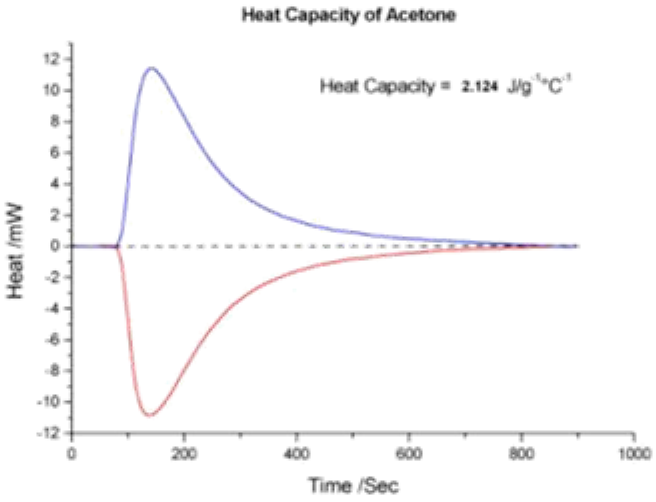


At equilibrium, the temperature of both cells will be equal and no additional power is added or removed.

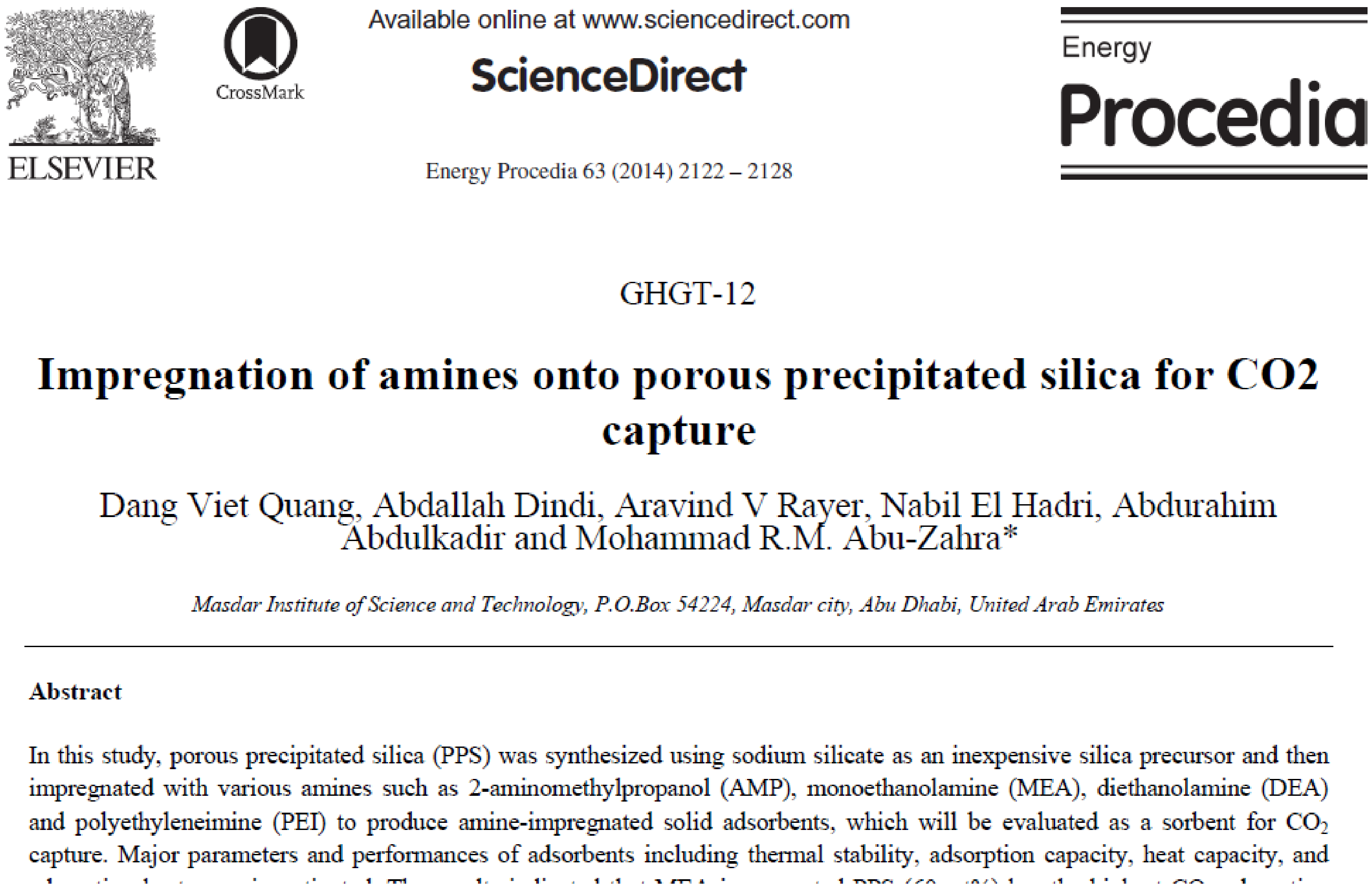
An exothermic sample causes the temperature of the sample cell to increase. In response to this increase, the control algorithm removes power from the sample cell to return and maintain its temperature to that of the reference cell.

The power added to or removed from the sample cell during this control procedure is equal to the power absorbed or produced by the sample.





uRC Process Safety anecdote – no data, just a good story for Testing decision making.



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Impregnation of amines onto porous precipitated silica for CO2 capture

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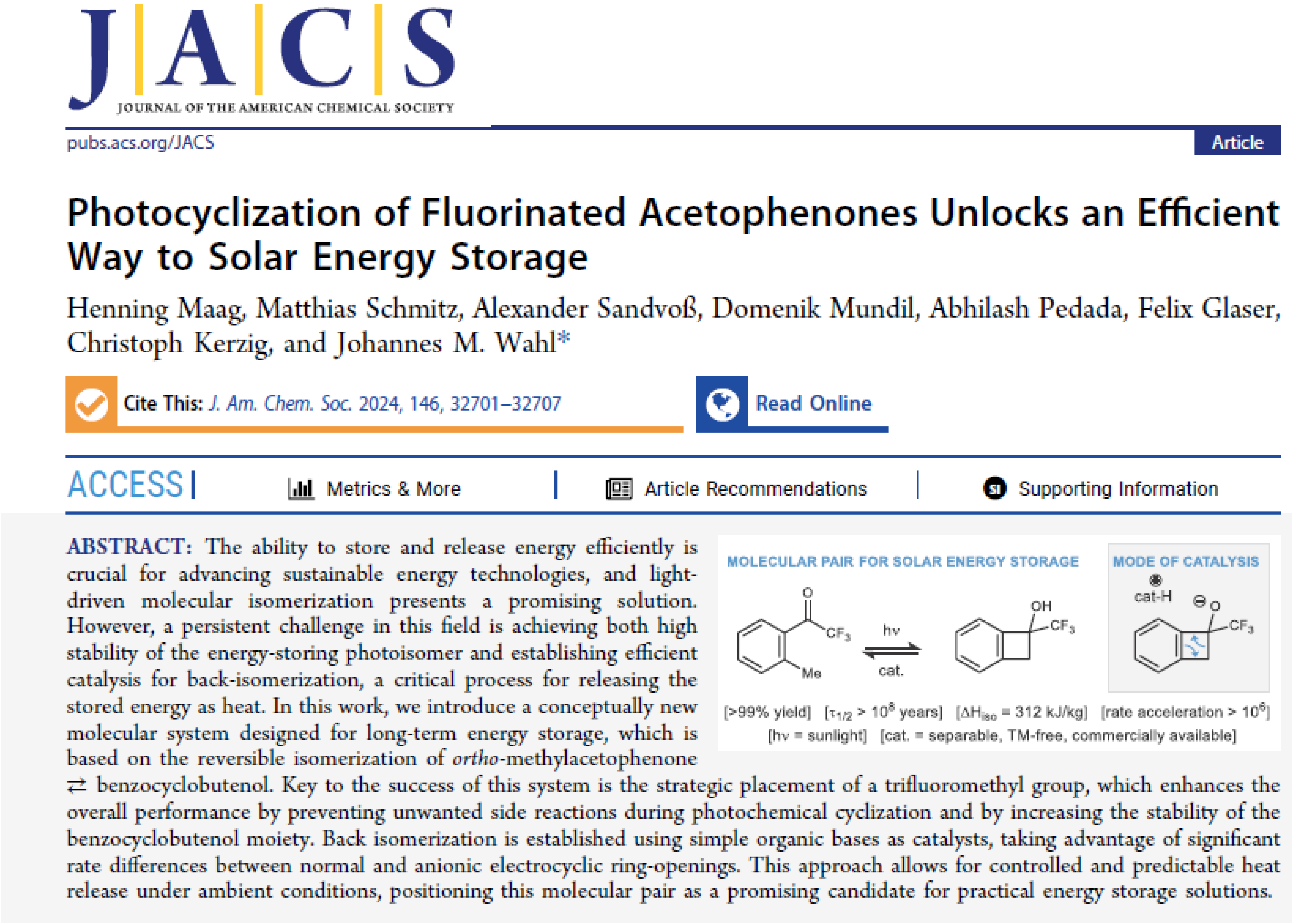
Abstract

In this study, porous precipitated silica (PPS) was synthesized using sodium silicate as an inexpensive silica precursor and then impregnated with various amines such as 2-aminomethylpropanol (AMP), monoethanolamine (MEA), diethanolamine (DEA) and polyethyleneimine (PEI) to produce amine-impregnated solid adsorbents, which will be evaluated as a sorbent for CO_2 capture. Major parameters and performances of adsorbents including thermal stability, adsorption capacity, heat capacity, and



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Article

Photocyclization of Fluorinated Acetophenones Unlocks an Efficient Way to Solar Energy Storage

Henning Maag, Matthias Schmitz, Alexander Sandvoß, Domenik Mundil, Abhilash Pedada, Felix Glaser, Christoph Kerzig, and Johannes M. Wahl*

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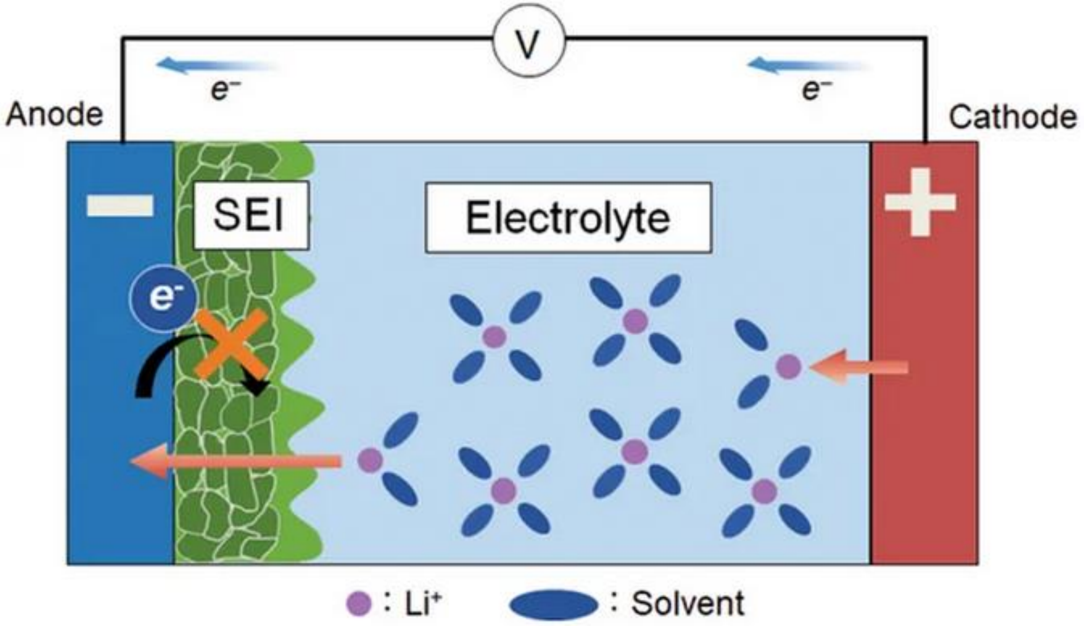
ABSTRACT: The ability to store and release energy efficiently is crucial for advancing sustainable energy technologies, and light-driven molecular isomerization presents a promising solution. However, a persistent challenge in this field is achieving both high stability of the energy-storing photoisomer and establishing efficient catalysis for back-isomerization, a critical process for releasing the stored energy as heat. In this work, we introduce a conceptually new molecular system designed for long-term energy storage, which is based on the reversible isomerization of *ortho*-methylacetophenone ⇄ benzocyclobutenol. Key to the success of this system is the strategic placement of a trifluoromethyl group, which enhances the overall performance by preventing unwanted side reactions during photochemical cyclization and by increasing the stability of the benzocyclobutenol moiety. Back isomerization is established using simple organic bases as catalysts, taking advantage of significant rate differences between normal and anionic electrocyclic ring-openings. This approach allows for controlled and predictable heat release under ambient conditions, positioning this molecular pair as a promising candidate for practical energy storage solutions.

Micro Reaction Calorimeter used in Li-Ion Battery Research.

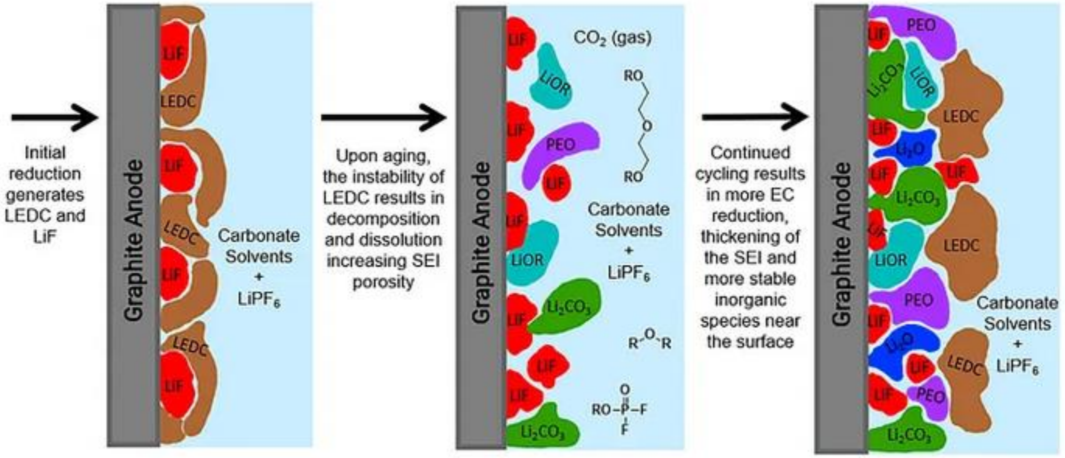
TRIGGER WARNING Here comes a crash course on Li Ion Battery SEI… from a sales rep. 😲

Solid Electrolyte Interphase (SEI) is a layer of material that forms between the Anode and liquid electrolyte. SEI generation is an essential and unavoidable process in a Li-ion cell.

The most difficult obstacle inhibiting improvement of lithium-based battery performance is the electrode/electrolyte interface.



[1]Schematic Diagram Illustrating SEI layer in a Li-Ion Cell


Initial reduction generates LEDC and LiF

Upon aging, the instability of LEDC results in decomposition and dissolution increasing SEI porosity

Continued cycling results in more EC reduction, thickening of the SEI and more stable inorganic species near the surface

[1] Schematic Illustration of Initial formation and further growth of SEI layer

Several publications upcoming related to battery research initiatives using uRC (Dhyllan Skiba – MIT Gallant Lab).

Project goals are to identify direct measurement of thermodynamic driving forces for the solvation of Li+ in polar aprotic solvents.

Why is this important? SEM, XRF and other methods used offer qualitative, not quantitative data at understanding SEI layer.

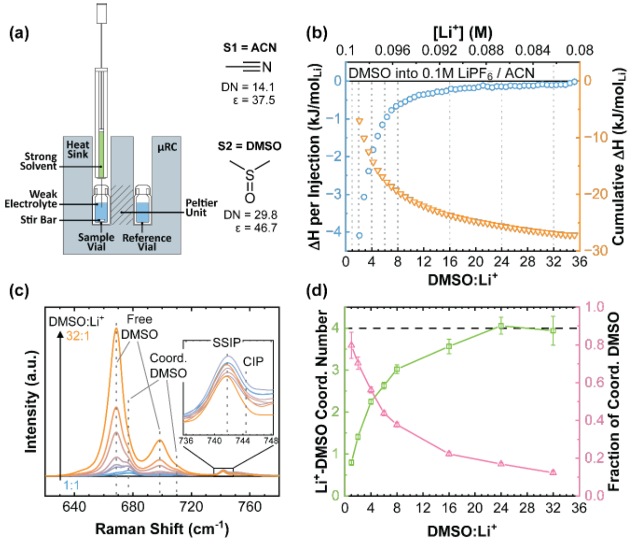
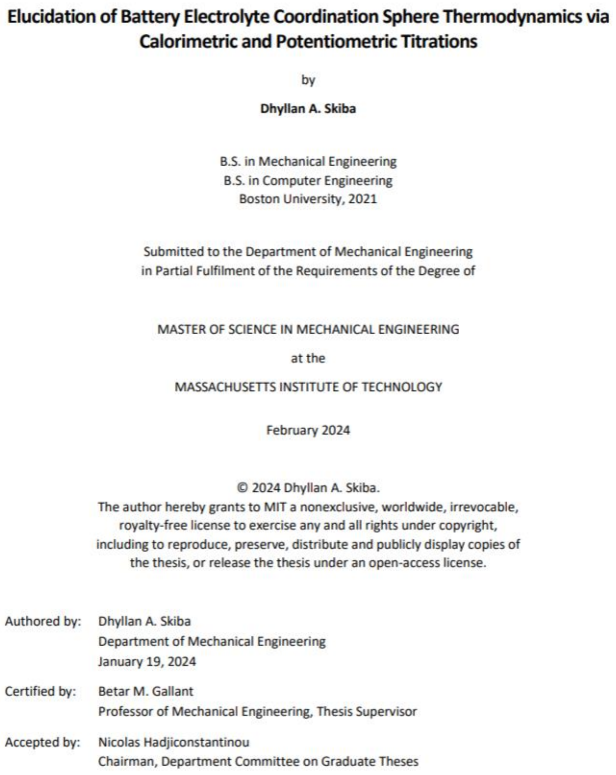


Figure III.5: ***Calorimetric titration method for an exemplar solvent displacement experiment with DMSO as the strong solvent and 0.1 M $LiPF_6$ / ACN as the weak electrolyte. Solvent displacement supported by Raman spectroscopy. (a)*** *Micro Reaction Calorimeter (μRC) setup measures reaction heats. Weak solvent (S1) and strong solvent (S2) chemical structures, donor numbers (DN) in kcal/mol, and dielectric constants (ε).* ***(b)*** *Measured enthalpy change per injection and cumulative enthalpy change vs the ratio of DMSO:Li^+. Measurement is corrected for solvent mixing. Dashed lines indicate mole ratios where Raman samples were taken.* ***(c)*** *Raman spectra of the electrolyte at select DMSO:Li^+ ratios throughout the titration. Vibration modes related to free and coordinated DMSO are labeled, and an enlarged view of the PF_6^- coordination-sensitive vibration modes are shown with labels for solvent-separated ion pairs (SSIP) and contact ion pairs (CIP).* ***(d)*** *Average coordination number of DMSO to Li^+ and the fraction of all DMSO in a coordinated state from deconvolution and integration of data in (c).*



Elucidation of Battery Electrolyte Coordination Sphere Thermodynamics via Calorimetric and Potentiometric Titrations

by

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B.S. in Mechanical Engineering
B.S. in Computer Engineering
Boston University, 2021

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in Partial Fulfilment of the Requirements of the Degree of

MASTER OF SCIENCE IN MECHANICAL ENGINEERING

at the

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

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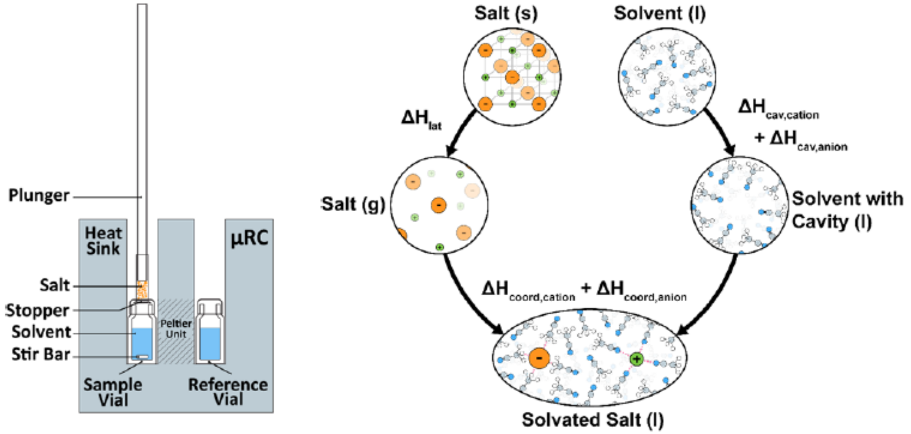

Figure III.1: ***Enthalpy of solution microcalorimetry setup, and a schematic of the contributions to the heat released/absorbed during dissolution.*** *On the left is a diagram of the power compensation microcalorimeter (μRC) apparatus with plunger for the addition of salt into the sample vial. On the right, an illustration of the processes that occur during salt dissolution including lattice breaking, solvent cavity formation, and ion-solvent coordination.*

Thank you P2SAC!

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