



## ENERGY RESEARCH AND DEVELOPMENT DIVISION

# FINAL PROJECT REPORT

# Advanced Lithium-ion Chemistry for Safer and Greener Electric Vehicle and Energy Storage Systems

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# PREFACE

The California Energy Commission's (CEC) Energy Research and Development Division supports energy research and development programs to spur innovation in energy efficiency, renewable energy and advanced clean generation, energy-related environmental protection, energy transmission, and distribution and transportation.

In 2012, the Electric Program Investment Charge (EPIC) was established by the California Public Utilities Commission to fund public investments in research to create and advance new energy solutions, foster regional innovation, and bring ideas from the lab to the marketplace. The EPIC Program is funded by California utility customers under the auspices of the California Public Utilities Commission. The CEC and the state's three largest investor-owned utilities— Pacific Gas and Electric Company, San Diego Gas and Electric Company, and Southern California Edison Company—were selected to administer the EPIC funds and advance novel technologies, tools, and strategies that provide benefits to their electric ratepayers.

The CEC is committed to ensuring public participation in its research and development programs that promote greater reliability, lower costs, and increase safety for the California electric ratepayer and include:

- Providing societal benefits.
- Reducing greenhouse gas emission in the electricity sector at the lowest possible cost.
- Supporting California's loading order to meet energy needs first with energy efficiency and demand response, next with renewable energy (distributed generation and utility scale), and finally with clean, conventional electricity supply.
- Supporting low-emission vehicles and transportation.
- Providing economic development.
- Using ratepayer funds efficiently.

Advanced Lithium-ion Chemistry for Safer and Greener Electric Vehicle and Energy Storage Systems is the final report for Contract Number EPC-20-016 conducted by South 8 Technologies, Inc. The information from this project contributes to the Energy Research and Development Division's Electric Program Investment Charge Program.

For more information about the Energy Research and Development Division, please visit the <u>CEC's research website</u> (<u>www.energy.ca.gov/research/</u>) or contact the Energy Research and Development Division at <u>ERDD@energy.ca.gov</u>.

# ABSTRACT

The team at South 8 Technologies (South 8) is the first to develop a novel and patented Liquefied Gas Electrolyte, LiGas<sup>®</sup>, chemistry for advanced Lithium-ion batteries with superior cost performance metrics and value propositions that make it uniquely suited to the future of energy storage systems and electric vehicles. The proposed non-hazardous chemistry not only lowers the cost to California ratepayers through the operational life of the electric vehicle or energy storage system due to increased cell performance, but also lowers recycling costs at end-of-life. The improved safety inherent to the cell chemistry furthermore reduces potential for cell-to-cell thermal propagation, which lowers downtime and improves grid resilience. Lastly, the innovation improves fast charge capabilities and the operation temperature range of the cell.

Through the course of this California Energy Commission project, the team at South 8 improved cycle life (>2,000 cycles), temperature (operation from -76°F [-60°C] to 140°F [60°C]), and power performance (2C-Rate) of the LiGas technology while also reducing the cost, vapor pressure, and global warming potential of the electrolyte chemistry. These cell level improvements were made by modifying the electrolyte composition, improving from South 8's "Gen 1" chemistry at the start of the project and finishing with the team's "Gen 3" chemistry.

During the duration of the project, further improvements were made to the cell form-factor hardware (mechanical packaging), reducing the overall cell height (9-percent reduction) and mass (5-percent reduction) from prior to the contract start. An improved LiGas injection system was also designed and implemented. The semi-manual system is now capable of filling an individual cell within nine minutes (including handling, vacuum, injection, and seal), and the time is being further reduced. The actual wetting time within the cell was measured to be a maximum of five minutes but was essentially complete within 60 seconds.

**Keywords:** Lithium-ion, Li-ion, lithium, battery, safety, low temperature, liquefied gas, LiGas, South 8

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## Background

Advances in battery technology will be critical to the future broad consumption of clean energy in California. The simultaneous push toward electrification and a reduction in fossil fuel use requires not only more clean energy generation, but also even larger increases in energy storage. A significant increase in battery storage capacity will be required to both meet increasing electric demand and meet California's state mandate to achieve an 85-percent reduction in carbon emissions and a 94-percent drop in gasoline consumption by 2045. To achieve this goal while providing benefits to utility ratepayers, a battery technology with low manufacturing costs and improved performance must be developed. The battery technology must be intrinsically safe to provide low-risk adoption in both grid-storage systems and electric vehicles. South 8 Technologies (South 8) developed a liquefied gas (LiGas) electrolyte, which uses fundamental chemical properties to enable high-performance battery cells with longer cycle life, wider temperature range, and inherently greater safety than conventional Li-ion batteries. LiGas electrolytes can be made from materials that are currently manufactured at low cost and may be implemented as a drop-in replacement for existing electrolytes, requiring minor capital investments in gigafactory production lines. The technology is an attractive solution for low-cost energy storage for applications such as energy storage systems and electric vehicles, which will benefit California ratepayers when the technology is commercially available.

## **Project Purpose and Approach**

Safety is critical to all battery applications. Energy storage systems have suffered from fires, creating downtime, property damage, higher insurance premiums, injury, and in the worst cases, loss of life. Similarly, there is hesitation around EV adoption due to not only safety concerns, but also range anxiety (the ability to charge quickly), high cost, and operation temperature.

The purpose of this project was to further develop the LiGas electrolyte chemistry for ultimate use in energy storage system and electric vehicle applications that reduce carbon emissions and save California ratepayers money. Unlike conventional Li-ion battery electrolytes that use solvents that are liquid at room temperature and atmospheric pressure, the LiGas electrolyte uses solvents that are gaseous at room temperature and atmospheric pressure. However, these gaseous solvents may be easily stored under pressure in a liquid state within a battery cell; while under pressure and in the liquid phase, LiGas solvents may solubilize lithium salts, creating a LiGas electrolyte. Unique attributes of the LiGas chemistry include high safety, a wide temperature operation window, fast-charge capability, and the potential to reduce battery installation costs through lower-cost cells and greater reliability. Technical goals included cycle-life metrics, high-power performance, wide operation temperature performance, fast charge, and cell energy, all of which were achieved. Together, these performance attributes would allow electric vehicles to charge faster, drive further, accelerate faster, and

maximize performance across all weather environments. Project outcomes additionally included cell-level safety characterization, which supported South 8's improved safety performance. Lastly, demonstration electrolyte capture at end-of-life recycling was completed, which will further drive down costs and reduce carbon emissions.

To achieve project goals, the research team was expanded from ~10 to ~35 employees (all residing in California and primarily with engineering backgrounds) through the course of the project to support product development and achieve project goals. Work was primarily done within South 8, while safety characterization was completed out of state with third parties. Further cell validation was also performed with several customers at customer locations to demonstrate the technology.

## **Key Results**

Through the course of this project, the team at South 8 accelerated development using 18650 cells with a graphite anode and nickel-manganese-cobalt (NMC811) cathode. These cells met the following performance targets.

- 1. **Cycle Life:** Cells have demonstrated >1,000 cycles (projected to be 2,000 cycles) at full depth of discharge at room temperature and cycle life projected to be >1,500 cycles at +113°F (45°C).
- 2. **High Power and Fast Charge:** Cells have demonstrated high power and greatly improved performance over liquid equivalent cells (95-percent capacity retention for LiGas versus 57 percent for liquid electrolyte cells over 150 fast-charge protocol cycles with 15-minute charge times).
- 3. **Low-Temperature Performance:** More than 70-percent capacity retention as low as –76°F (–60°C) while maintaining high voltage
- 4. **Improved Safety:** Reduced burn time and no thermal propagation when compared with conventional liquid electrolyte cells
- 5. **Demonstrated Recyclability:** Demonstrated ability to vent and collect LiGas electrolyte at end of life, with a rough demonstration showing 56-percent collection of the LiGas electrolyte solvent, which may be purified, rebalanced, and reused in future Li-ion battery cells.

## **Knowledge Transfer and Next Steps**

Throughout the course of this project, South 8 worked with customers and partners to further develop the LiGas chemistry. Soliciting customer feedback was valuable to better use South 8's development resources in a targeted and focused manner to improve the technology for rapid adoption. South 8 has sampled cells to numerous customers ranging from automotive manufacturers and power tool makers to the United States Department of Defense. South 8's value propositions both increase value today and develop the technology to maximize future value. For example, customers agree South 8's LiGas electrolyte is the best performing low-temperature battery cell available. However, several of these customers also require other

performance metrics (for example, high-temperature operation or high power) to develop their products. Understanding the customer perspective in use-case scenarios helped South 8 better define the technology roadmap to deliver more value to customers.

South 8 has been building relationships throughout the gas and electrolyte industry to better prepare the business for future scaling. The LiGas chemistry uses off-the-shelf materials to formulate the advanced electrolyte technology, so working with established suppliers that ensure a stable future supply is important. These relationships are critical for ensuring that South 8 selects low-cost chemistries that can be sourced domestically, and which will not be regulated in the future. Since South 8 plans to be an electrolyte provider, an understanding of geographic logistics (where both customers and suppliers reside) will be critical to minimizing future shipping costs. Partnering with established gas suppliers will help enable South 8 to scale quickly and continue to foster these relationships.

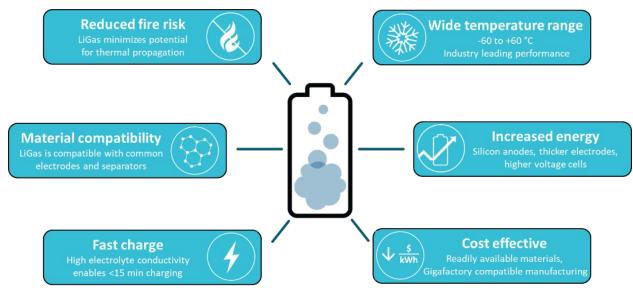
South 8 has developed a healthy intellectual property portfolio, with 25 issued patents across the globe covering electrolyte composition, electrolyte blending, electrolyte injection, and cell design. Furthering South 8's intellectual property is a primary objective for the business to continue to grow and succeed in the energy storage industry.

South 8 has demonstrated excellent cell performance in the 18650 form-factor and has attracted customers that would be excited to adopt the LiGas technology in the near future for low temperature and high safety applications, such as power for U.S. soldiers or engine cold-start in defense applications. These beachhead markets will serve as a platform for South 8 to establish itself as a market leader in low temperature and highly safe batteries. Engaging with these defense applications will require transferring manufacturing technology for the LiGas electrolyte, electrolyte injection systems, and cell housings to South 8's cell manufacturing partners, namely mid-tier cell providers in the United States. South 8 has already begun multiple engagements with partners to this end. A growing portfolio of end-use customers will also ensure business success and ability for South 8 to grow to the next phase in which the company will provide products to energy storage system and electric vehicle markets.

# CHAPTER 1: Introduction

California is a leader in its efforts to adopt new technologies to ease the state's successful transition to clean energy. The electrification of the transportation sector and advancement of renewable resource power generation are key to this effort and largely depend on the Lithiumion (Li-ion) battery industry. Both the federal (for example, the Inflation Reduction Act of 2022) and state (for example, increased California Energy Commission [CEC] budget) governments are encouraging these efforts at the private level through research and development (R&D) and manufacturing scale-up grants. The Li-ion space is now considered a critical technology to future United States (U.S.) security, and the effort to develop technology and manufacturing know-how domestically is enormous. There is a planned order of magnitude increase in Li-ion production in the U.S. from today's ~100 gigawatt-hours (GWh) to ~1,000 GWh by 2030 (Volta Foundation, 2023).

Li-ion battery technology, originally developed for consumer electronics (for example, laptops and cell phones), requires significant improvements in performance, safety, and cost reduction to gain widespread adoption in wider markets including electric vehicles (EV) and energy storage systems. South 8 Technologies (South 8) is the first and only company to develop a novel liquefied gas (LiGas<sup>®</sup>) electrolyte, which improves safety through the reduction of cellto-cell thermal propagation, increases battery performance with increased energy, has fastcharge capability, provides a world-record –76 degrees Fahrenheit (°F) (–60 degrees Celsius [°C]) to +140°F (60°C) operating temperature range, and has lower manufacturing cost than standard electrolytes (Figure 1). These value propositions are highly valued in the industry by EV and cell manufacturers as well as the U.S. Department of Defense. South 8 continues to deliver cells to the industry with increasingly better performance as a direct result of this project's outcomes. The outcomes of this project will enable South 8 to deliver a breakthrough new battery technology to deliver on California's climate mandates to lower carbon emissions through EV and energy storage system (ESS) adoption.



### Figure 1: Value Propositions of the LiGas Electrolyte

The project started at technology readiness level (TRL) 3 and required further product development to achieve market adoption. This included modifying the electrolyte chemistry to enable equivalent or improved performance over a variety of metrics when compared to conventional liquid electrolytes. The low temperature performance remains one of South 8's most differentiating metrics, but this quality needs to be accompanied by other basic performance metrics. These include improving cycle-life performance to >1,000 cycles and increasing durability at high temperatures while maintaining good low temperatures, power, and safety metrics. To accomplish this, South 8 iterated on the LiGas electrolyte chemistry with a focus on delivering higher performance cells. South 8 evolved the electrolyte from a "Gen 1" to a "Gen 3" electrolyte, which improved all metrics including life, temperature performance, power, global warming potential (GWP), and vapor pressure. This improvement was achieved by replicating the overall gas solvent composition. With a focus on materials that were low cost, non-toxic, non-corrosive, and lower pressure, the Gen 3 chemistry is a competitive contender to successfully enter the commercial market.

Additional goals for this project included conducting safety characterization on cells, which was done at third-party laboratories. Heating, abnormal charge, and nail penetration characterization tests were conducted. These tests showed comparable results to conventional liquid cells (with the exception that nail penetration showed faster cooling time by about 50 percent), showing the potential for less thermal propagation between cells.

Lastly, the project also called for a demonstration of recycling LiGas electrolyte, a process impossible with today's liquid electrolytes. Success here would lead to lowering the cost of future battery systems and reducing carbon emissions by reducing the manufacture of material components. While the project goal was to demonstrate the ability to capture >70 percent of the electrolyte mass, a rudimentary system yielded only 56 percent captured mass. With improvements in battery pack design and setup fixturing, >70 percent should be achievable both in the lab and on a larger scale.

The project was successfully completed on budget and with a 12-month contract extension requested due to COVID-19 related delays. The success of this project led to a second financing round for the business in 2023, with strategic partners Lockheed Martin and a German automotive group alongside current investors LG Energy Solution, Anzu, and Foothill Ventures. The project also facilitated multiple contracts from the U.S. Department of Defense, totaling over \$7 million to date, to enable safer and higher performance batteries for end-use cases.

# CHAPTER 2: Project Approach

The first portion of this project focused on improving cell performance. This was done by improving the LiGas performance on the graphite anode and high-nickel-manganese-cobalt (NMC) cathodes separately, then bringing them together in a full-cell construction in an 18650 form-factor cell. The full 18650 cell then underwent safety characterization testing by a third party. Lastly, a small 5-cell battery pack was constructed to demonstrate the potential for end-of-life recycling and decommissioning. These five tasks were:

- Task 1) Graphite Anode Development.
- Task 2) High-Nickel Cathode Development.
- Task 3) Full Form-Factor 18650 Device Development.
- Task 4) Safety Testing.
- Task 5) End-of-Life Recycling Decommissioning Demonstration.

A summary of the final technical data provided in this report is summarized in Table 1.

Task #	Task Title	Task Deliverables and Outcomes
1	Graphite Anode Development	First cycle coulombic efficiency (charge efficiency) >90%
		Cycle life performance >1,000 cycles
		Power performance up to 2C
		Temperature performance from –76°F (–60°C) to +140°F (60°C)
2		Cycle life on NMC811 up to 4.5 volts (V), projected to reach >1,000 cycles
	High-Nickel Cathode Development	Power performance up to 5C
		Temperature performance from –76°F (–60°C) to +140°F (60°C)
		Power testing on thick cathodes of 148 microns ( $\mu$ m) thickness.
		Cycle life demonstrated >1,000 cycles
		Fast charge protocol demonstrated >150 cycles
	Full Form-Factor	Power performance up to 5C
3	18650 Device Development	Temperature performance from –76°F (–60°C) to +140°F (60°C)
		Cycle life performance at +113°F (45°C), projected to be >1,000 cycles
		Injection time to be seconds, full wetting to be <5 minutes

#### Table 1: Summary of Task Deliverables and Outcomes from this Project

Task #	Task Title	Task Deliverables and Outcomes
4		UL1642 (Underwriters Lab Standard Test 1642) Heating Test of 18650 Cells with positive outcome
	Safety Testing	UL1642 Abnormal Charge Test of 18650 cells with positive outcome
		SAE2452 (Standard American Engineering Test 2452) nail penetration test of 18650 cells with positive outcome
5	End-of-Life Recycling Decommissionin g Demonstration	Demonstration of capture of >56% LiGas solvent at the cells' end-of-life through heating of a 5-cell battery pack with no thermal propagation

Note: "C-Rate" is defined as the rate at which a cell may be charged or discharged fully. E.g., 1C = 1 hour,  $2C = \frac{1}{2}$  hour, C/2 = 2 hours, etc.

### **Task 1: Graphite Anode Development**

**The Goal:** A goal of Task 1 was to reach a first-cycle efficiency of >90 percent and a cycle life of 1,000 cycles to 80 percent nominal capacity at full depth-of-discharge, and 1C rate on the graphite anode. This will ultimately constitute a product-ready level of performance on the graphite anode. Graphite anodes were also characterized for performance. Power performance was tested at various C-rates (C/10, C/3, C/2, 1C, 2C, and 5C), at room temperature. Temperature performance was tested from  $-76^{\circ}F(-60^{\circ}C)$  to  $+140^{\circ}F(60^{\circ}C)$  in  $68^{\circ}F(20^{\circ}C)$  steps at the C/2 rate.

**The Approach:** A design-of-experiments (DOE) was conducted to screen various additives at varying concentrations. The base electrolyte chemistry (composed of several liquefied gas solvents and salts) and the formation cycle rate were fixed, while the DOE was performed on additives. This initial DOE was focused on first-cycle coulombic efficiency (charge efficiency) and cycle life of the graphite anode. When the most promising additive formulations were established, a second DOE was completed that optimized the full electrolyte composition for first-cycle coulombic efficiency (charge efficiency), cycle life, power capability, operation temperature performance, and pressure reduction.

While these tests were planned to be primarily performed in a coin-cell form-factor in half-cell configuration (with Li metal counter electrode), it was determined early on that development could be accelerated by focusing on 18650 form factors. This had the benefit of consistent cell builds and performance, which are representative of a commercially viable form factor. While there was an upfront cost associated with purchasing the 18650 cells from vendors, there were cost savings from lower labor at South 8 to build several smaller cells with potentially less illustrative performance.

### Task 2: High-Nickel Cathode Development

**The Goal:** A goal for this task was to demonstrate a high-nickel cathode (for example, nickelmanganese-cobalt, NMC811) capable of 1,000 cycles at  $\geq$ 4.2 V to 80 percent nominal capacity cycling at full depth of discharge, performance that is representative of a product-ready cathode. Cycle-life performance was conducted at +68°F (20°C) at 4.2 V, 4.3 V, 4.4 V, and 4.5 V (in coin cells using a Lithium metal counter electrode) to maximize voltage and available capacity out of the cathode. Power performance was tested at various C-rates of C/10, C/3, C/2, 1C, 2C, and 5C at room temperature. Temperature performance was tested from -76°F (-60°C) to +140°F (60°C) in 68°F (20°C) steps at the C/2 rate. Cathodes of thickness >120 µm were also be evaluated and compared to traditional cathodes ( $\sim$ 65 µm thick) for C-Rate and life comparison.

**The Approach:** Electrodes were sourced from established manufacturers. South 8 did not prepare coatings but rather relied on several partners to provide coated and calendared electrodes. Using the most promising electrolyte formulations from Task 1, the NMC811 and high-Ni NCA electrodes were tested for coulombic efficiency, C-Rates, and temperature performance, in addition to cycle life at various maximum voltage limits to maximize capacity and cell energy. Thick cathodes were sourced from other project partners that developed their own thick-electrode technology.

Like Task 1, it was planned that these tests would be primarily performed in a coin-cell form factor in half-cell configuration (with Lithium metal counter electrode). However, it was determined early on that development could be accelerated by focusing on 18650 form factors. The electrodes were therefore tested in 18650 rather than coin-cell form-factor.

### Task 3: Full Form factor 18650 Cell Development

**The Goal:** Full form-factor 18650 cells were constructed using a graphite anode and highnickel cathode and tested for performance with a capability of 1,000 cycles at  $\geq$ 4.2 V to 70 percent nominal capacity cycling at full depth-of-discharge, performance representative of a product-ready cell. Cycle life performance was conducted at +68°F (20°C) and 1C rate. Power performance was tested at various C-rates (C/10, C/3, C/2, 1C, 2C, 5C), at room temperature. Temperature performance was tested from -76°F (-60°C) to +140°F (60°C) in 68°F (20°C) increments. Fast-charge testing was conducted to demonstrate cell charge to 80-percent capacity in 15 minutes. Electrolyte injection processes were examined with a goal of demonstrating full-cell wetting within five minutes. While cell energy life may be further optimized in the future, a goal for this project was to demonstrate that the technology could reach 200 watt-hours (Wh) per kilogram (Wh/kg), which is an excellent early data point to demonstrate the technology for further investment, development, and deployment. This project successfully hit this energy density in 18650 form-factor cells.

**The Approach:** South 8 runs a custom-designed and operational electrolyte injection pilot line in-house. Because of this unique tool, South 8 was able to accelerate cell chemistry development in the 18650 form factor, which is an ideal platform to demonstrate new cell chemistry performance and safety metrics. This pilot line, made possible by the CEC's 2018 SCALE-UP project, was a critical and valuable tool in this current project's success.

South 8 sourced 18650 dry cells using standard graphite and high-nickel cathodes, typically in groups of 100 to 1,000. These cells were in a jelly-roll form factor already wound with a separator, inserted into the can, bottom welded, and shipped to South 8 under dry or inert atmosphere. The cells were received by South 8, vacuum dried again, injected with the LiGas

electrolyte, and sealed. Cells were tested to meet described performance goals. Cell design iterations were made to meet performance metrics. Fast-charge performance was tested by using an ARPA-E (United States Department of Energy) suggested protocol.

## Task 4: Safety Testing

**The Goal:** South 8 conducted safety performance characterization including thermal ramp, abnormal charge, and nail penetration. The tests showed several cells passing the UL1642 characterization standard. Some cell failures provided useful results to help improve future iterations of the cell or electrolyte design. In addition, a small module with at least five cells was tested via thermal ramp to demonstrate that there is no cell-to-cell thermal propagation.

**The Approach:** South 8 Technologies worked with third-party safety characterization labs to conduct safety characterizations on South 8 cells. The safety testing closely followed UL1642 or SAE (Society of Automotive Engineers) guidelines. For each cell test, between 3 and 5 cells were tested. Each test had cell data recorded including voltage, temperature, and applicable video and photo footage that monitored the cell. Further, pack-level testing was conducted to demonstrate that thermal propagation does not occur from cell-to-cell within a pack. This was conducted with cells in close proximity (no cell spacing) for maximum pack-energy density and worst-case-scenario analysis.

Initial safety characterization was conducted at Sandia National Laboratories. The cells tested were in a prototype form factor, which was state-of-the-art at the beginning of this project. These tests satisfied safety characterization requirements for this project. However, South 8 chose to also perform safety characterization on the more relevant "pre-A" (higher TRL product) form factor 18650 cells, which were ready toward the end of the project. The safety characterization studies outlined in the proposal varied slightly from the actual tests conducted. Actual safety characterization tests conducted included nail penetration, over charge, and thermal ramp. This was due to increased knowledge about how the LiGas cells behave, and feedback from tests that customers deemed most critical. South 8 focused resources on the tests that were most relevant and critical to the technology's success. The project manager was informed about these changes throughout the project.

### Task 5: End-of-Life Recycling Decommissioning Demonstration

**The Goal:** The goal was to demonstrate the improved recyclability of the LiGas electrolyte technology. This was accomplished by demonstrating a simple method of venting a small pack of Li-ion cells assembled in Task 3. The project team established a goal of >70 percent by mass of vented gasses to be captured into a separate vessel to demonstrate the potential of electrolyte recycling or scrubbing.

**The Approach:** A pack of at least five cells was assembled and installed inside a simple metal enclosure. The enclosure featured electrical connections to test the entire pack, a heating element, and an opening through which the gaseous materials were captured. The pack was first tested to demonstrate electrical functionality. The pack was then heated via the internal heater to approximately +194°F (90°C); when the LiGas electrolyte vapor pressure increased enough to open the cell vent, the gaseous materials were then quickly vented from the cell

and out through the port on the battery pack. The gas port was hooked up to a chilled cylinder in which the gaseous vapors were captured in liquid form. Each cell vented eventually, and all gaseous solvents were captured safely. At this time, a valve to the capture cylinder was closed and the heater was turned off. Once the cells cooled and were determined to be safe to handle, they were disassembled in atmosphere and the cathode was extracted from the cell. Lastly, the mass of gaseous solvent captured was compared to the known mass of the solvent to ensure that most of the solvent was captured. The demonstration was successfully completed, with success determined by the dismantling of cells without any spark, flame, or other event, as well as the collection of >50 percent of the liquefied gas solvent mass.

This task was important to demonstrate a step improvement in the potential recyclability of future Li-ion cells using advanced LiGas technology. The industry is continuously seeking improvements in recycling of battery materials to lower costs, improve supply chain security, and reduce environmental impacts. The LiGas technology is uniquely positioned to provide what is now a focused effort on cathode materials for electrolyte materials. This demonstration was an important step towards this goal.

# CHAPTER 3: Results

### **Task 1: Graphite Anode Development**

Enabling the graphite anode was the last hurdle to overcome in the commercialization of the Li-ion battery in 1990. The difficulty now is identifying a suitable electrolyte system that does not degrade under charge conditions and keeps the graphite anode intact. The use of ethylene carbonate as a co-solvent in liquid electrolytes was identified as the key to a stable solid electrolyte interphase (SEI), which is formed on the graphite anode and protects both it and the electrolyte from decomposition during charge and discharge. Furthermore, the formation of this SEI layer requires that the lithium from the cathode be sacrificed to create a stable layer, which reduces the energy of the cell. Minimizing this lithium use, or maximizing the cell's first-cycle coulombic efficiency, is critical for a high performing cell.

The LiGas electrolyte is the first ethylene carbonate free electrolyte with high-performance metrics that are either comparable to or improved upon conventional liquid electrolytes. Through chemical permutations and the design of experiments, South 8 developed novel chemistries that form SEI layers on the graphite anode. These SEI layers created high performance cells with low impedance for high power, which were stable across many cycles and across wide temperature ranges.

South 8 made the first batch of 18650 cells with ~88.2 percent first cycle coulombic efficiency. The second batch with improved processing and chemistry improved this to 91.2 percent, as shown in Figure 2.

With the Gen 1 LiGas electrolyte, cycle-life testing at room temperature was conducted. Cells were cycled at 100 percent depth of discharge at the C/3 rate, shown in Figure 3. The cell shown reaches 80 percent discharge capacity at >1,000 cycles, an excellent result that shows the robustness of the formed SEI layer and the LiGas electrolytes in general. These performance metrics are important for meeting growing customer requirements.

Power testing was also conducted with the Gen 1 LiGas electrolyte on a graphite anode halfcell in coin cell form factor. Power performance was excellent, showing >95-percent capacity retention up to the 2C-discharge rate, shown in Figure 4. This shows the SEI layer created in the LiGas electrolyte enabled low impedance and high power, capable of sustaining fastcharge applications.

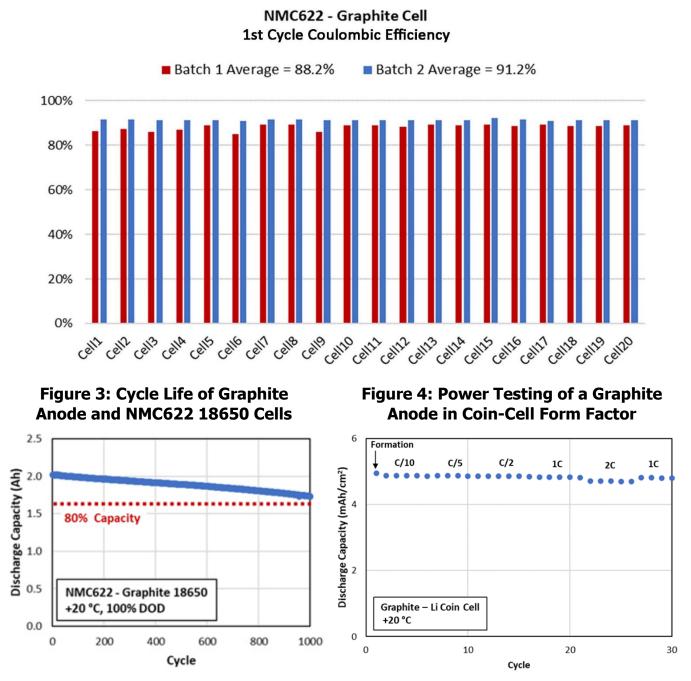
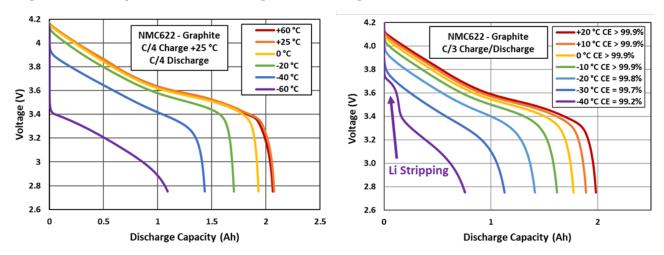


Figure 2: Coulombic Efficiency of Graphite Anode and NMC622 18650 Cells

Temperature testing was also conducted on 18650 form-factor cells using a graphite anode and NMC622 cathode. A wide temperature range was demonstrated, showing excellent discharge performance from  $-76^{\circ}$ F ( $-60^{\circ}$ C) to  $+140^{\circ}$ F ( $60^{\circ}$ C) at the C/4 rate, shown in Figure 5. With a discharge capacity retention of ~50 percent at 140°F ( $60^{\circ}$ C), the LiGas electrolyte demonstrates a world record as well as the uniqueness of the LiGas electrolyte in extending the operational range of batteries down to ultra-low temperatures. Furthermore, preliminary examination of cell rechargeability at low temperatures was conducted. It was shown that charging down to  $-22^{\circ}$ F ( $-30^{\circ}$ C) at the C/3 rate is possible, shown in Figure 5. At lower temperatures or higher rates, it was observed that there may be a lithium plating risk. The impressive improvement in low-temperature rechargeability will be important to customers in many industries.





#### **Task 2: High-Nickel Cathode Development**

While more attention is typically paid to the SEI on the anode surface, electrolytes must also not decompose on the cathode surface. This is generally accomplished through the formation of robust cathode electrolyte interphase (CEI) layers on the surface of the cathode. The LiGas electrolyte has shown to be exceptionally robust against oxidation reactions on the cathode surface and it forms low impedance CEIs, allowing for high power and good cycle life.

Figure 6 shows LiGas life performance on NMC811 half-cell coin cells at the C/2 rate at +68°F (20°C) at varying voltages versus a lithium metal anode. The stable cycle life shows the electrolyte is incredibly resistant against oxidation up to high potentials, which is beneficial in creating higher capacity and higher energy cells. The power performance of the cathode was also evaluated in the LiGas electrolyte at +68°F (20°C), showing ~80 percent discharge capacity retention up to 5C (Figure 7). This is an excellent result, sufficient for most applications including the EV and ESS industries.

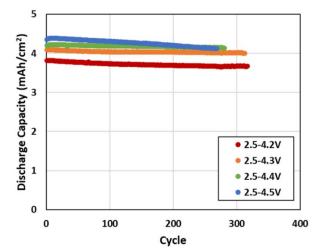


Figure 6: Cycle Life of NMC811 Cathode Coin Cells at Varying Voltages

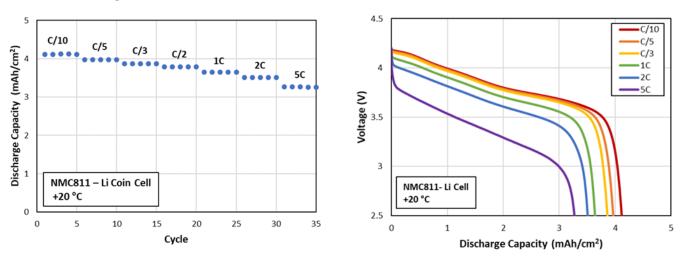
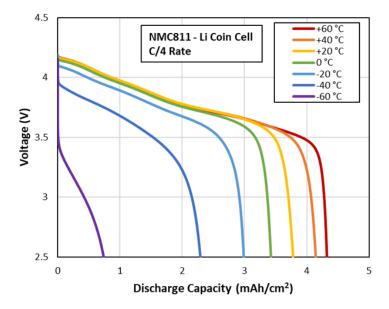


Figure 7: C-Rate Performance of NMC811 Cathode Coin Cells

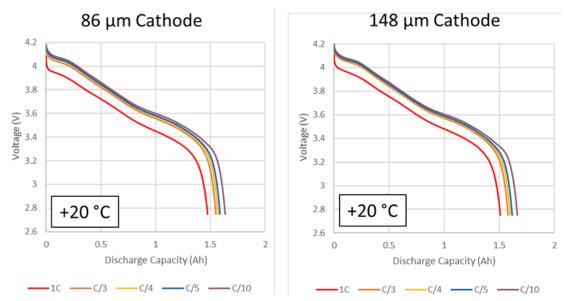
It is generally recognized that the cathode is the rate-limiting electrode at low temperatures. This was evaluated with the LiGas electrolyte on an NMC811 cathode in the coin-cell form factor at the C/4 rate, shown in Figure 8. The LiGas electrolyte performed well down to  $-40^{\circ}$ F ( $-40^{\circ}$ C) with good voltage hold-up during discharge and  $\sim$ 50-percent capacity retention. At 140°F ( $60^{\circ}$ C), performance was diminished as expected on the coin cell and the aggressive power rate. As seen elsewhere in the report, 18650 form factors held up significantly better due to lower cell impedance and some limited self-heating during discharge. The high-temperature performance is excellent, as expected for the LiGas electrolyte.





Conventional cells using liquid electrolytes are limited in how thick the electrodes (anode and cathode) can be coated. This is due to the relatively high viscosity and low conductivity of the electrolyte, which is unable to penetrate thick electrodes so would not deliver needed power. Due to the ultra-low viscosity and moderate pressure of the LiGas electrolytes, thick electrodes

are expected to behave well in this system. Figure 9 shows two comparative 18650 graphite-NMC622 cells, with cathodes coated with similar technology and active material recipe but to different thicknesses (86 and 148  $\mu$ m) at different power rates. Generally, cathode thickness is limited to ~80  $\mu$ m in conventional cells. These cells behaved similarly, showing little impact on the power performance due to electrode thickness, which illustrated the excellent wettability of the LiGas electrolyte. This can have implications for higher-energy cells (due to increased active material) and lead to lower costs due to a shorter coating time in cell manufacturing. This could potentially lead to between a 7-percent and 10-percent increase in cell energy when optimized for thickness and density, an excellent gain to improve cell energy across the board.



### Figure 9: 18650 Power Testing with Thick Electrodes

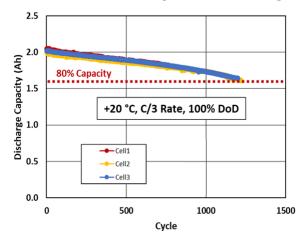
### Task 3: Full Form-Factor 18650 Cell Development

While Task 1 and Task 2 focused on LiGas development in coin cell form factor, they were also supplemented with 18650 builds. Scaling quickly to larger form factors is important since this allows "real life" performance checks, which may bring a multitude of newer challenges including transition metal dissolution, expansion and contraction of electrodes, safety issues, cell balancing, electrolyte volume usage, and other factors.

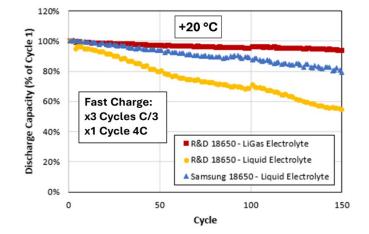
South 8 worked with third-party toll manufacturers to build 18650 "dry cells." These dry cells contain standard anode and cathode coatings (active material, conductive carbon, binder) on standard current collectors (aluminum for cathode, copper for anode), and standard separator membranes (typically polyethylene and/or polypropylene). Graphite was used for the anode active material, and the cathode current collector used was NMC622 or NMC811 for varying builds.

One of the most challenging aspects of any new electrolyte chemistry is to achieve high cycle life in a full-cell design. South 8's LiGas chemistry had an impressive start in showing high cycle life with the Gen1 LiGas chemistry, shown in Figure 10. Three cells mapped very closely on each other, showing >1,200 cycles to 80-percent discharge capacity. The consistency shows the quality of both the dry-cell builds and the LiGas chemistry itself.

#### Figure 10: Cycle Life Performance of 18650 Cells with Gen1 LiGas at 100 Percent Depth of Discharge



Fast-charge performance characterization was also conducted to determine its application for advanced EVs. Two sets of cells were characterized with identical dry cells, but one was filled with conventional liquid electrolyte and the other with Gen 1 LiGas electrolyte. Both cells were put through identical fast-charge protocols (issued by United States Department of Energy). This protocol called for three cycles at the C/3 charge rate, then a 15-minute fast charge at 4C for every fourth cycle. Figure 11 shows that the cell with LiGas far outperformed the cell with the liquid-based electrolyte, showing 95-percent capacity retention for LiGas versus 57 percent for liquid electrolyte cells, over 150 fast-charge protocol cycles. This impressive performance is thought to be due to a few factors including lower impedance of the SEI layer on the graphite anode (which leads to less lithium plating), high electrolyte conductivity, and better wetting on the electrodes. Because the liquid cells in this case were R&D cells and the quality of the cell build was possibly not as high as in a commercial cell; an off-the-shelf commercial 18650 cell was procured and put through an identical fast-charge protocol. The LiGas cell still outperformed the commercially available cell. This demonstrated the advantages of the LiGas chemistry when it came to fast-charge applications for the EV industry. With range anxiety a serious concern with EV end-use customers, fast-charging will be needed to ensure that they get maximum distance on their charging without long charge times.



### Figure 11: Fast-Charge Performance on 18650 Cells Using Gen 1 LiGas

While a considerable amount of characterization was conducted with the Gen1 electrolyte on varying cathodes and anodes, the Gen3 electrolyte was superior in many aspects including power, low- and high-temperature performance, and cycle life. Figure 12 shows the power, temperature, and cycle life performance of the Gen 3 LiGas electrolyte using a 2.7 Ah 18650 cell with a graphite anode and NMC811 cathode. Power performance up to 1C was excellent, showing ~95-percent discharge capacity retention compared to C/10 capacity. At low temperatures, -40°F (-40°C) and -76°F (-60°C) show ~80-percent and ~70-percent discharge capacity retention, respectively, at the C/3 discharge rate when compared to +68°F (20°C). Further, the voltage hold up was excellent, allowing for high-power output even at these low temperatures. This is remarkable performance at low temperatures, which would appeal to customers in need of low-temperature operation devices.

The cycle life at +68°F (20°C) and +113°F (45°C) was also evaluated on cells using the Gen 3 LiGas electrolyte at full depth-of-discharge. The +68°F (20°C) performance was projected to hit >1,500 cycles while the  $+113^{\circ}F$  (45°C) performance was projected to hit >1,000 cycles. The performance at elevated temperatures was a significant improvement over the Gen1 chemistry. The Gen 1 LiGas chemistry suffered at elevated temperatures due to a phase separation phenomenon. The development of the Gen3 electrolyte focused on improving this issue, and resulted in a higher performance electrolyte capable of increased temperature operation among other performance improvements. This elevated temperature cycle life is highly desirable for most applications where the cell resides in ambient temperatures or experiences elevated temperatures during discharge. This improved performance expands for further applications through the industry, including ESS and EVs.

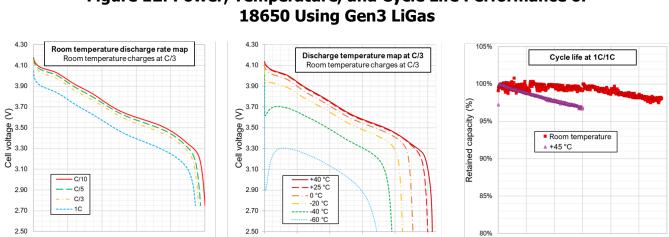


Figure 12: Power, Temperature, and Cycle Life Performance of

Typical liquid electrolytes are relatively viscous and require mechanical pistons to force the electrolyte into the micro pores within the cell to wet all electrode surfaces. Several vacuum and injection steps were used to ensure that no gas remained within the cell, which would prevent full wettability.

40%

60%

Discharge capacity (% of +25 °C)

80%

100%

0

100

200

300

Cvcle

400

500

600

0%

20%

40%

60%

Discharge capacity (% of C/10)

80%

100%

0%

20%

One additional benefit of the LiGas electrolyte is simplifying the injection process. Due to its ultra-low viscosity and inherent pressure, the LiGas electrolyte can be injected into a cell

relatively quickly without the need for mechanical pumps to force the electrolyte into the cell. While a vacuum is still pulled to remove any gas within the cell prior to injection, any residual gas simply solubilizes into the LiGas solution, allowing for full wettability of electrode surfaces.

South 8 upgraded LiGas injection hardware to use a semi-automated system capable of filling cells in nine minutes, including loading, vacuum, fill, seal, disengage, and unload. Performance optimization continues to improve this already impressive design. Using this new system, electrolyte fill times were evaluated. 18650 cells were hooked up to an impedance monitor and impedance measurements were taken at the 1 kilohertz rate from the moment of injection (results in Figure 13). As shown, the impedance quickly dropped to 27 milli-ohm (m $\Omega$ ), then settled down to a final impedance of 24 m $\Omega$  over the course of ~5 minutes. This shows that the injection itself was rather quick, but the full wetting and residual gas solubilization settled after a few minutes' time. This is an impressive result considering the longer times for liquid electrolyte injection and multiple vacuum and injection steps involved. This will help gigafactories in the future save on floor space, capital expenditures (few machines) and higher throughput and reliability (full wettability), ultimately leading to cost savings.

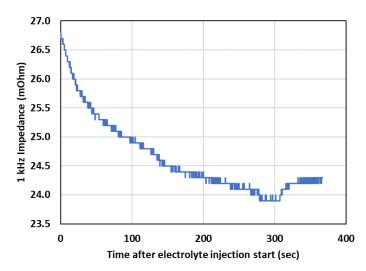


Figure 13: Impedance Measurements During LiGas Injection into 18650 Cell

### Task 4: Cell Safety

Cell safety is a critical test that is run routinely with customers, alongside electrochemical performance. Safety characterization may include any number of evaluations including heating, overcharge, short circuit, and other factors. As part of this research project, safety characterization tests were planned and conducted. Cells with Gen 1 LiGas electrolyte were shipped to third parties for safety characterization. Three tests were conducted: UL1642 heating test, UL1642 abnormal charge test, and SAE 2452 nail penetration.

For UL1642 heating tests, cells were ramped to  $+140^{\circ}F$  (60°C), charged to full voltage (4.2 V), cooled to  $+68^{\circ}F$  (20°C), then ramped back up to  $+266^{\circ}F$  (130°C) and held for 10 minutes. The UL1642 test standard states that a cell is compliant with no thermal event (venting is acceptable). It was shown the LiGas cells conform to test standards and only showed a vent

event at around +212°F (100°C), as designed into the cell. Cell data and photos from this test are shown in Figure 14. Liquid comparison cells showed similar conformance (data not shown).

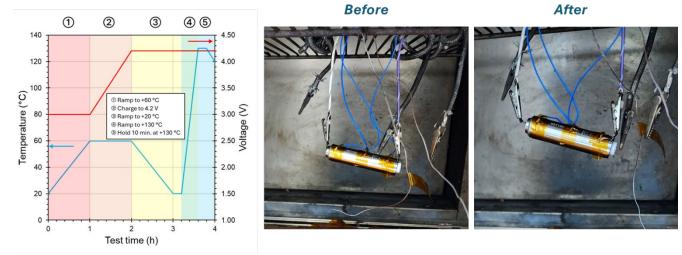
For UL1642 abnormal charge, cells were discharged and held at +68°F (20°C), then charged at three times the maximum charge rate and observed for seven hours prior to discharge. The UL1642 test standard states that a cell is compliant with no thermal event, disassembly, or vent. It was shown that the LiGas cells conform to test standards with no adverse effects from the test profile. Cell data and photos from this test are shown in Figure 15. Liquid comparison cells showed similar conformance (data not shown).

For SAE 2452 nail penetration test, cells were taken to full charge, then penetrated with a 3 millimeter-diameter sharp-tipped nail at 4 centimeters/second velocity into the center of the can side wall until full penetration. Thermocouples were placed at various locations around the cell to observe temperature before, during, and after nail penetration. It was shown that both liquid and LiGas cells go into thermal runaway (as expected). Liquid comparison cells were equipped with a vent integrated into the lid while the LiGas cell was equipped with a vent on the bottom portion of the cell hardware. Both vents opened during the thermal event and, in the case of the LiGas cell, debris was ejected from the cell through the bottom vent. This vent was not equipped with a screen to catch debris, but this feature will be integrated in future designs. Cell data and photos from this test are shown in Figure 16.

Thermal runaway events can occur in single cells due to defects or abuse. Significant engineering goes into battery pack assemblies to mitigate this risk and prevent cell-to-cell propagation. Reducing the heat output from a single cell to a level where it cannot propagate to the next cell is a primary focus area to reduce risks of injury or property damage. During the nail penetration testing, the time it took for the cells to cool to +212°F (100°C) (when thermal propagation risk is lessened) was 780 and 400 seconds for the liquid and LiGas cells, respectively. The 51-percent reduction in cooling time considerably reduced the potential for thermal propagation, which would result in safer battery packs.

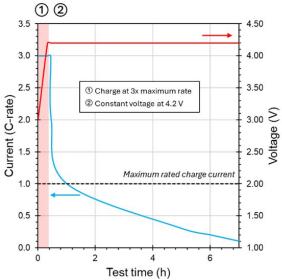
More effort is being dedicated to ensure that LiGas cells are the safest in the industry. This will require further design efforts on the hardware as well as improvements to the LiGas chemistry, to reduce pressure and side-wall fracture before vent opening and reduce flammable components to decrease the possibility of ignition. Future safety characterization is planned beyond this project to continue to provide best-in-class safe cells.

#### Figure 14: UL1642 Heating Test of LiGas 18650 Cell



Cells vent at <100 °C as designed

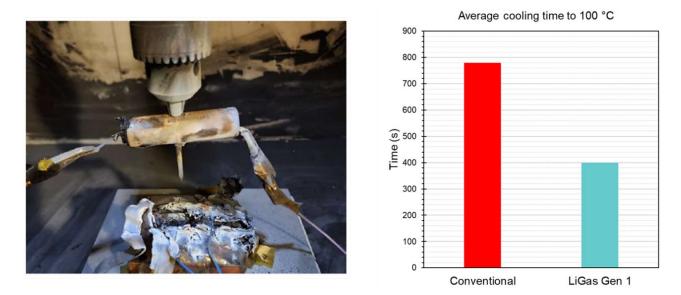
### Figure 15: UL1642 Abnormal Charge Test of LiGas 18650 Cell





Cells intact, no vent or damage Success = no fire after 7-hour charge

#### Figure 16: SAE2452 Nail Penetration Test of LiGas 18650 Cell and Heat Output from Comparison Liquid Cell



#### Task 5: End-of-Life Recycling and Decommissioning Demonstration

The LiGas electrolyte is unique in several ways in terms of safety, performance, and manufacturing. One additional area of benefit is the ability to easily capture and recycle the LiGas electrolyte. While liquid electrolytes incinerate with the rest of the Li-ion cell, the LiGas solvent may simply be vented from the cell with gentle heating, then captured. This is similar to common refrigerant capture from both commercial and residential air conditioning systems in place today. Once the gas is captured, it may be routed to a refrigerant recycling center, distilled, and purified into singular components for reuse. This will further reduce the cost of LiGas battery cell manufacturing in the future.

A five-cell battery pack has been built and underwent a recycling demonstration, shown in Figure 17. The cells were discharged, placed into a housing, and temperatures were monitored on three cells with thermocouples. The housing was secured with a lid, and an elastomer gasket provided an air-tight seal. The thermocouples and cell voltage monitoring wires were fed through a leak-tight epoxy feedthrough. A separate gas port allowed collection of the vented gas.

The battery pack was heated via strip heaters to a temperature of +266°F (130°C). Each cell's vent opened in the span of approximately five minutes, and the vented gas was collected into a separate chilled vessel through condensation.

Each cell carried ~3.68 grams (g) of gas solvent, giving a total of 18.4 g. A total of 10.31 g of gas was collected from the 5 cells, yielding a 56-percent collection efficiency. The remaining gas was expected to remain in the battery pack and the connecting tubes. A larger battery pack is expected to yield more efficient gas collection due to its higher cell count to empty space ratio, leaving comparatively less dead space in the system and allowing better collection efficiency. Though lower than the 70-percent target collection efficiency, this was a rudimentary setup with several avenues to improve upon in the future.

This proof-of-concept recycling demonstration effectively showed that it is possible to collect and capture the gases from a battery pack after use by simply heating the system. This will lower the cost and simplify the recycling of Li-ion cells for the industry.

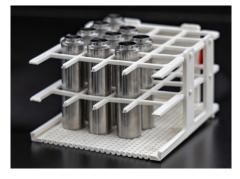


## Figure 17: Five-Cell Battery Pack Used for Recycling Demonstration

## **Additional Project Outreach**

This CEC research project helped advance South 8's technology maturation, allowing further outreach into the community. To date, South 8 has shipped to 16 customers for cell evaluation. A photo of South 8's "Pre-A" 18650 cells is shown in Figure 18. Customer feedback has been invaluable in building a "requirements list" to support product evolution. Attendance at conferences and trade shows is also an important method to share South 8's story, and there have been numerous speaking opportunities (Plug Volt, International Battery Seminar, Advanced Automotive Battery Conference, and others). Regular online marketing posts, including a popular 2023 post showing a flashlight operating in dry ice at  $-94^{\circ}F$  ( $-70^{\circ}C$ ) for over 18 hours. There was also a demonstration of this at the Advanced Automotive Battery Conference, which was eye-catching and engaged attendees.

Travel to partners, both domestic and international, is regularly conducted to continue building relationships through the automotive, cell manufacturing, and gas manufacturing value chain. South 8's intellectual property has also grown. With over 17 different patent families and 25 patents issued globally, South 8's IP portfolio will soon be formidable. South 8 is currently engaging interested parties to install LiGas injection systems in their cell-manufacturing facilities. This is the first step in South 8's ultimate business of selling LiGas electrolyte to cell manufacturers globally and bringing this CEC-supported technology to market.



### Figure 18: Image of South 8's Pre-A 18650 Cells

# CHAPTER 4: Conclusion

The environmental push toward a clean energy future will require an enormous increase in energy storage systems to transition the transportation sector to electrification. This transition is well underway in California, the United States, and globally. A global effort will require leadership on the international stage by those who control the supply chains and the manufacturing that power this energy revolution. Today the U.S. manufactures roughly four percent of batteries globally while Asia (primarily China) manufactures roughly 84 percent of battery manufacturing capacity (Benchmark, 2021). Asia also dominates the raw materials supply chain, including the electrolyte, where the U.S. produces only 2 percent of the global market supply (Bloomberg, 2019).

For the U.S. to maintain its leadership, resources should not solely focus on the current state of art, but rather strategically focus on new innovations, which can scale and reach cost-parity quickly, and with improved performance. South 8's LiGas electrolyte offers the opportunity to achieve these goals. The technology has been demonstrated to provide superior performance to the incumbent technology. The electrolyte may be simply integrated into today's gigafactories since it maintains most common manufacturing processes. Those processes may be made more cost-effective through higher speeds and lower scrap rates. While some cell hardware modifications are required, they are minimal and, with the push toward larger cellform factors (for example, 46XX for EVs), are less burdensome.

The U.S. is investing heavily into the battery materials supply chain (for example, the "Infrastructure Investment and Jobs Act," a \$1.2 trillion investment in U.S. infrastructure, signed into law in 2021). Several of these programs focus on a "catch up" strategy when investing in current state-of-art Li-ion battery cell technologies; additional investment has also been made in innovative solutions (for example, silicon anode technologies). The LiGas solution benefits from an already established gas supply chain in the U.S. and South 8 is focused on delivering a high-performance electrolyte that leverages materials already domestically produced in large quantities. This leads to a cost-effective solution that is already engaged with all the major gas manufacturers in the nation and is planning both future projects and collaborations with these groups to forge a successful path to expanding commercialization.

This CEC research project raised the technology and manufacturing readiness levels in a demonstrated battery cell, which is now being sampled to customers throughout the industry. By demonstrating this successful technology, South 8 is better able to finance future projects, which will help California both lower carbon emissions and benefit ratepayers throughout the state. With the technology demonstrated, South 8 is focusing on the next stage of electrolyte manufacturing scale-up. South 8 has already engaged engineering, procurement, and construction firms to provide a concept study for a hypothetical future electrolyte manufacturing facility. This pilot facility will target an electrolyte production capacity of roughly

1 GWh and could be located in California (pending project planning and future financing opportunities). It is likely South 8 will partner with leading gas manufacturers to leverage their knowledge and experience in gas blending, storage, and shipping to scale rapidly. With a demonstrated pilot system, the technology may rapidly be scaled further for penetration into the wider ESS and EV markets to provide higher volumes of electrolyte to the industry.

When the LiGas enters the wider market, ratepayers are expected to benefit from reduced electricity costs at home, in commercial buildings (through the higher performance and efficiencies that the LiGas electrolyte provides), and for both personal and fleet EVs. This will happen through a combination of technology enhancements (higher energy, longer cycle life, fast-charge capability) and safety improvements (fewer battery fires, which lead to lower cost of ownership and insurance burdens on ESS installations). Beyond the pilot phase, manufacturing the LiGas electrolytes may benefit from co-location with gigafactories; South 8 intends to remain headquartered in San Diego, California, so job growth in that area is expected for both R&D and future pilot manufacturing.

South 8's business success, and adoption of the CEC-supported technology, could benefit from future policy and financing opportunities within California. This could include incentives for manufacturing within the state (for example, grant opportunities, tax breaks) and larger R&D tax credits focused on energy technologies. The U.S. lacks battery expertise in comparison with Asian counterparts. Promoting further education in science, technology, engineering, and mathematics fields focused on energy storage and generation will benefit California for years to come in growing the workforce, which are sorely needed to both power this industry into the future and maintain a competitive edge on the global technology stage.

# **GLOSSARY AND LIST OF ACRONYMS**

Term	Definition
18650 form factor	Cell having dimensions 18mm diameter and 65mm height
°C	degrees Celsius
CEC	California Energy Commission
CEI	cathode electrolyte interphase
C-Rate	C-Rate is defined as the rate at which a cell may be charged or discharged fully. For example, $1C = 1$ hour, $2C = \frac{1}{2}$ hour, $C/2 = 2$ hours, etc.
DOE	design-of-experiments
ESS	energy storage system
EV	electric Vehicle
°F	degrees Fahrenheit
g	gram
GWh	gigawatt-hour
GWP	Global warming potential
kHz	kilohertz
LiGas	liquefied gas
mΩ	milli-ohm
NMC	nickel-manganese-cobalt
R&D	research and development
SAE	Society of Automotive Engineers
SEI	solid electrolyte interphase
South 8	South 8 Technologies
TRL	technology readiness level
μm	microns
U.S.	United States
V	volts
Wh	watt-hours
Wh/kg	watt-hours per kilogram

# References

- Benchmark Mineral Intelligence. 2021. *Lithium-Ion Battery Megafactory Assessment*. Available at https://www.benchmarkminerals.com/market-assessments/gigafactory-assessment/.
- Bloomberg New Energy Finance. 2019. "Battery Components Manufacturing Asset Map." Available at www.bnef.com.
- Volta Foundation. 2023. *Battery Report*. Available at https://volta.foundation/battery-report-2023.

# **Project Deliverables**

Project deliverables for this contract are available at <u>pubs@energy.ca.gov</u>.

- X1 report on Graphite Anode development
- X11 quarterly reports throughout the course of the project (dated June 30, 2021; September 30, 2021; December 31, 2021; March 31, 2022; June 30, 2022, September 30, 2022; December 31, 2022; March 31, 2023, June 30, 2023; September 30, 2023; and December 31, 2023)
- Final Report (this document)