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FINAL PROJECT REPORT

CEC Production Scale-Up for Next Generation Batteries using Liquefied Gas Electrolytes

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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 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.

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

ABSTRACT

This project focused on development of a low-rate initial production line of cylindrical battery cells fabricated using a patented and novel Liquefied Gas electrolyte at South 8 Technologies. The Liquefied Gas electrolyte allows for safer and higher energy Li-ion batteries which operate over a wider temperature range and at a lower cost for applications in energy storage systems, electric vehicles, and defense. Two manufacturing challenges exist to bring this technology to market: (1) electrolyte mixing and (2) electrolyte injection into cells. This project focused on these two aspects to elevate the manufacturing readiness level of the technology. Throughout this project, South 8 developed the necessary process planning, equipment design, and execution towards elevating the manufacturing readiness level on the Liquefied Gas electrolyte technology. Three primary tasks within the project included: (1) gas cabinet design and installation, (2) gas mixing manifold design and construction, and (3) cell injection design and construction. These tasks were fully validated and shown to meet project goals.

Keywords: California Energy Commission, Liquefied Gas Electrolytes, South 8 Technologies, cylindrical battery cells

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Background

Advances in battery technology will be critical to broad use of clean energy in California. The simultaneous push towards electrification and reduction of fossil fuel use requires not only increases in clean energy generation, but even larger increases in energy storage. A significant increase in battery storage capacity will be required to meet the increasing electric demand and meet California's state goal of achieving 85 percent reduction of carbon emissions and 94 percent drop in gasoline consumption by 2045. To achieve this goal while providing benefits to rate payers, a battery technology with low manufacturing cost 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 developed a liquefied gas (LiGas) electrolyte which utilizes fundamental chemical properties to enable high energy density batteries with long cycle life and inherent safety. 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's ratepayers when the technology is commercialized and reaches the market.

Project Purpose and Approach

The core motivation of the project is to enable a LiGas manufacturing process capable of improving energy storage performance with low cost and risk for customers. To demonstrate this, cells of 18650 form-factor shall be manufactured using LiGas formulations. Through several discussions with major customers (utilities, state and federal policymakers, and automotive manufacturers) it is clear the high demand for improved batteries having lower cost and longer cycle life is greatest in grid-storage and electric vehicle markets.

LiGas battery manufacturing may be adopted in state-of-the-art gigafactories by demonstrating production methods that are closely aligned with the existing methods. The construction does not require significant manufacturing changes, with the exception of the liquefied gas electrolyte preparation and injection. The goal of this project is to design, build, and demonstrate an electrolyte production capability of 500 cells per day and demonstrate the capability and scalability for electrolyte injection into 18650 cells for a Low Rate Initial Production.

LiGas battery technology is inherently unique as it utilizes a liquefied gas based electrolyte. While all other components of the cell manufacturing remain the same, the LiGas electrolyte preparation and injection require unique tooling to accommodate the handling of electrolyte during cell manufacturing. Electrolyte production and cell injection in quantities of 500 cells per day, or 8 kWh, manufacturing rate capability was proposed as a threshold for demonstrating the potential for high volume manufacturability with strategic manufacturing partners.

The scope of projects included the design and development of a gas distribution system, a gas flow delivery system, and a LiGas electrolyte injection system. The design and development of these systems required additional in-house expertise in engineering design. Several mechanical and process engineers were recruited during the project to create a custom design for the gas flow and electrolyte injection systems. The design and build of these systems were coordinated with the process development so that the final production line would meet both throughput and accuracy metrics. In addition to a throughput goal of 500 cells per day, an accuracy goal of ± 1percent of the target electrolyte fill mass was proposed.

Several key contributors from outside South 8 Technologies contributed to the facilities' expansion and construction of the gas distribution system. Local contractors selected by the general contractor CRB Group provided the skills necessary for proper installation of the gas cabinet systems and tubing systems that connect the cabinets to the gas delivery manifold. South 8 Technologies' partnership with industrial gas supplier, Matheson Tri-Gas was also utilized for gas cabinet equipment sourcing. Most engineering design was performed in-house, and machining performed by a local machine shop Proto Solutions.

Key Results

A gas delivery system utilizing gas cabinets and a gas manifold was successfully designed and built at South 8 Technologies for preparation of LiGas electrolyte on a scale needed to satisfy a Low-Rate Initial Production goal of five hundred (500), 18650 cells per day. An electrolyte injection demonstration system was built in parallel with a design that is scalable to meet the 500 cell/day goal.

Accurate mass delivery of the liquefied gas solvents is critical to maintaining performance specifications of the LiGas electrolyte batteries. The gas distribution system and manifold demonstrate excellent accuracy when delivering single or multiple gases to a container. Validation tests of individual gas lines show mass accuracy within the ± 1percent target masses. The ± 1percent mass error target was achieved with additional fine tuning of the manifold controllers. For multiple gas fills, the ± 1percent mass accuracy target was achieved in fills totaling 500 grams of liquefied gas solvents. The system was designed using commercially off-the-shelf components and could be replicated following appropriate specifications. The system controls utilize an in-house programmed LabView code which provides automated functionality and ease of use without a need for high level skillset.

While the gas delivery system was built with close resemblance to the proposed design, the injection system was changed considerably. Several critical junctions influenced the decision to change the design of the injection system. The original design utilized a fixture that filled the cell with liquefied gas solvent and also crimped the 18650 cap to the can. Inconsistent cap crimping resulted in cell leakage and influenced the decision to pursue alternative sealing and injection methods. Therefore, an approach utilizing a soldered cap was implemented as a prototype product. This cap required designing a new injection apparatus, which operates independent of the gas delivery manifold. Learnings from this injection system will be utilized in the build of a larger automated system currently under development for South 8

Technologies’ new commercial cell design which will feature hermetic sealing made possible by laser weld designs.

The cell injection system is constructed for delivering measured quantities of LiGas electrolyte to individual 18650 or 21700 cells. The system uses manual controls and convective heating to transfer LiGas electrolyte from a keg to a cell. Each cell injection takes about 5 minutes, which is below the target production rate. More efficient heating and automatic controls will be implemented in future design iterations to enable cell injection time scales of approximately 1 minute, which will meet the project production goal.

The process has provided insight to the potential challenges that may need to be addressed to provide a cell design which is safe and commercially viable. One example is the venting mechanism of the cell upon over pressurization. Prevention of thermal runaway via appropriate venting of the LiGas is a critical hurdle to implementation of next generation Li-ion cells. Combining the intrinsically safe chemical properties of LiGas electrolytes with a safe engineered can/cap design will enable high capacity LiGas cells in grid storage systems. This could shrink the footprint of grid storage systems and provide reliable grid service to California ratepayers.

Utilizing the system designed as part of this project, the LiGas electrolyte has already demonstrated excellent performance metrics including:

- Improved safety with potential to eliminate thermal runaway as demonstrated with 3rd party testing at Sandia National Labs
- Excellent cycle life with >1,000 cycles at 100percent depth of discharge
- World-record temperature performance from -60 to +60 °C
- Excellent fast-charge performance with pathway to <10 min EV charge time
- Ability to increase cell energy by 20percent using thicker electrodes and higher voltage cathodes, enabling cell energy up to 300 Wh/kg and 800 Wh/L
- Potential to lower cost by 20percent down to $80/kWh with the use of cobalt free and low nickel cathodes
- Reduction in battery pack mass, volume, and cost by removing heaters and denser cell packaging due to increased cell safety.

Knowledge Transfer and Next Steps

Maintaining the flexibility to pivot along a technical roadmap allows innovative concepts to be developed. A benefit to pursuing the alternative injection method was the opportunity for intellectual property development. New innovative electrolyte preparation and injection methods were developed, along with novel electrolyte preparation methods. Such an example involves the cell cap, which required modifications from the original crimped design to a soldered design. The knowledge gained will carry into higher throughput manufacturing methods and enable a high-quality product. Intellectual property (patents and trade secrets) will be utilized to transfer knowledge to future licensees of the LiGas technology. South 8 Technologies has already delivered prototype cells to several customers across industry segments (e.g., electric vehicle and cell manufacturers) as a direct result of this project’s outcomes. South 8 Technologies attended several conferences and marketing opportunities to discuss the technology and gained several customers and partners who continue to engage with South 8 Technologies to further technology and business development.

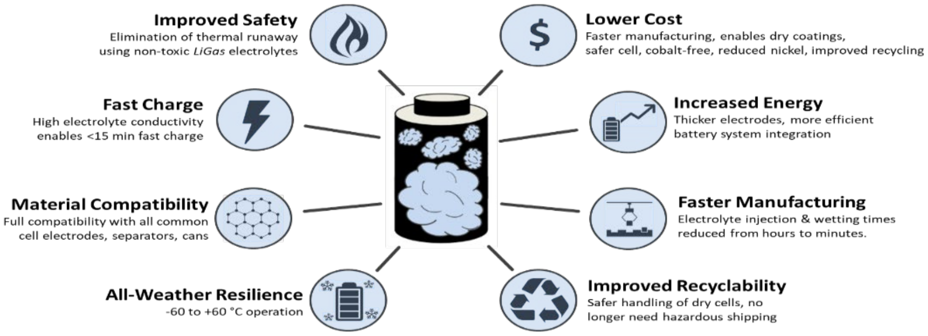
Demonstrated progress towards low-rate initial production (LRIP) of LiGas batteries was achieved, and several research and manufacturing projects continue to further advance the LRIP initiative. A commercially relevant cap design is being developed which will streamline the cell build process. Fundamental work on the electrolyte formulation will improve the intrinsic safety of the cell, lower cost, and facilitate manufacturing. As next generation LiGas products are developed, the flagship product will undergo extensive testing and validation to facilitate adoption across the industry.

CHAPTER 1: Introduction

There is a significant push to lower carbon emissions and curb climate change globally, and California is a leader in this effort through adoption of new technologies toward this end. The electrification of the transportation sector and advancement of renewable power generation is key to this effort and largely depends on the Li-ion battery industry. Both the federal (e.g., Inflation Reduction Act) and state (e.g., increased California Energy Commission (CEC) budget) governments are encouraging these efforts at the private level via research and development (R&D) and scale-up manufacturing grants towards the development of clean tech and particularly battery systems. The Li-ion space is now considered a critical technology to the future U.S. security and the effort to develop technology and manufacturing know-how domestically is enormous.

The Li-ion battery technology, originally developed for consumer electronics (e.g., laptops and cell phones), requires significant improvements in performance and safety and reduction in cost to gain widespread adoption for wider markets including electric vehicles and energy storage systems. South 8 Technologies (South 8) is the first and only to develop a novel Liquefied Gas (LiGas®) electrolyte which improves safety through the elimination of thermal runaway, increases battery performance with increased energy, has fast-charge capability, provides a world-record -60 to +60 °C operating temperature range, and has lower manufacturing cost (Figure 1). These value propositions are highly valued in the industry by electric vehicle and cell manufacturers as well as the Department of Defense. South 8 has already delivered prototype cells to several of these customers 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 goals of lowering carbon emissions through electric vehicle and energy storage system adoption.

Figure 1: Value Propositions of the LiGas Electrolyte



Source: South 8 Technologies

The LiGas electrolyte is fully compatible with all conventional battery materials (e.g., anode, cathode, current collector, separator, salt, etc.), and majority of the conventional manufacturing processes. However, there is no current system which is capable of manufacturing the electrolyte in mass. This project aimed to design, fabricate, and demonstrate a low rate initial production gas manifold which would be capable of mixing the required liquefied gas solvents to form the final electrolyte mixture in quantities sufficient for 500 cells/day production. This would demonstrate the manufacturing processes for the electrolyte mixing which then could be scaled up for future licensees' manufacturing batteries on a larger scale. Further, the electrolyte injection process into the battery was demonstrated as part of this project and shown to be a simple and rapid process which can easily be scaled to high volume manufacturing.

The project was completed on time and on budget. Demonstration of the technology is well underway, and the industry has taken notice of South 8's breakthrough technology. The Gas Manifold enabled by this project is now a centerpiece of the LiGas electrolyte business and has attracted financial investment in a successful Series A financing which closed in Dec 2021.

CHAPTER 2:
Project Approach

Overview:

The team at South 8 Technologies is the first to develop a novel and patented Liquefied Gas Electrolyte, LiGas, chemistry for rechargeable lithium batteries with superior cost performance metrics and value propositions. The proposed nonhazardous chemistry has already demonstrated exceptional safety, power, and cycle life with conventional 4-V cathodes. A novel electrolyte vaporization through cell venting at high temperatures enables the cell to safely shut down and eliminate the potential for thermal runaway and was validated by Sandia National Labs. Similarly, intentional venting and recovery of the solvent is a simple process that will allow for end-of-life recycling for reuse in future devices or other applications.

Key Participants:

The project included contractors and equipment manufacturers and included internal personnel growth. A mechanical engineering team was established at South 8 within a few months after the project kick-off. Significant engineering efforts were dedicated to the gas manifold system design, as well as the LiGas injection system re-design. The mechanical engineering team continued to grow at South 8 with increasing focus on designs and equipment necessary for achieving commercially viable products that can be produced at the low-rate initial production (LRIP) goal of 500 cells per day. Equipment manufacturers include Matheson Tri-Gas, which constructed the gas cabinets and gas panels. Contractors provided services for facility renovations and equipment installations, both in Summer 2020 and Summer 2022. CRB Group served as a general contractor on both occasions with local subcontractors for mechanical, electrical, and fire safety.

Approach:

This project required substantial engineering design to enable the scientific foundation of the LiGas battery fabrication process. Some of the designs included in the original project description were tested and found to be impractical for scaleup to the LRIP goal. Therefore, some tasks associated with the original project description became no longer applicable as the design was restructured.

Of the primary technical tasks (Task 2.1 Gas Storage and Distribution, Task 2.2 Gas Delivery Manifold, and Task 2.3 Cell Injection and Crimp), Tasks 2.1 and 2.2 were accomplished with minor changes to the task breakdown. Task 2.3 required significant approach modifications to achieve the originally described milestones.

This project was divided into the three primary components:

1. Gas Storage and Distribution: Gas cabinets were to be designed, procured, and installed at South 8 facilities to connect to the gas manifold system. These cabinets should be capable of safely containing several different gasses, have switch-over valves to prevent process breaks, and appropriate alarm systems to detect any potential leaks.
2. Gas Delivery Manifold: The gas delivery manifold was the primary focus of the project. The manifold is capable of mixing several gaseous solvents into a electrolyte "keg" in high volume and high accuracy. It has been designed to be fully automated and very user friendly with accuracies within the target +/- 1percent mass. The manifold and the keg have been designed to be able to mix and dispense electrolyte for up to 500 cells/day low-rate initial production. The "keg" is highly mobile and easily sized up for future delivery to customers across industry segments.
3. Cell Injection: South 8 designed, fabricated, and demonstrated a scalable system that is capable of rapid LiGas electrolyte injections into battery cells. This has been demonstrated on both 18650 and 2170 cells. The injection system was designed for prototype cells and the process was demonstrated to achieve +/- 1percent accuracy across several cell sample groups. The learnings from this manual system will be used in the design and scaleup of an automated system currently under development.

Task 2.1: Gas Storage and Distribution

The primary goal of achieving a LRIP process for LiGas batteries requires significant quantities of liquefied gas solvents which are packaged in high pressure cylinders. Ventilated cabinets outfitted with intrinsically safe leak detection and process monitoring are typically used to store and distribute gas cylinders. A previously established partnership with Matheson Tri-Gas provided a platform for scoping the purchase of such ventilated cabinets. A cabinet model with capacity of two (2) gas cylinders was selected to facilitate cylinder changeover and minimize downtime associated with cylinder exchange. The cabinet design was customized with a in-house designed gas distribution panel. Two types of panels were designed, with one specifically for Argon, which is broadly used as a utility gas, a purge gas, and a process gas. Matheson engineers assisted in the design of the panels and selection of the sensors utilized for each of the gas chemistries.

The cabinets were installed to ventilation, fire sprinkler water lines, and process gas line tubing by contractors. These installations were part of the tenant improvement project for the South 8 laboratories. The contributing contractors were determined by a general contractor CRB Group, but the project was managed by South 8. The cabinets were tested and adjusted to draw the rated air flow by the heating, ventilation and air conditioning (HVAC) contractor, Apex Mechanical. South 8 Technologies will stay in compliance with all state and federal laws regulating the handling of the gasses and materials used in manufacturing.

Task 2.2: Gas Delivery Manifold

The original objective for the gas delivery manifold was to have it integrated with the injection system. The changes to the injection process (discussed in next section) resulted in a gas delivery manifold design that could be a standalone system. Instead of connecting to an injection system, the manifold fills the liquefied gas solvents into a large volume reservoir which has a capacity for five hundred (500) 18650 cells. The reservoir could then be installed on the injection system for electrolyte delivery.

Testing of components that would be incorporated on the manifold was performed on the R&D manifold built prior to this CEC project. New pressure and flow controllers were sourced from Brooks Instruments and validated for performance of the research and development (R&D) manifold prior to purchasing a set of eight (8) for the LRIP gas manifold.

The manifold design required detailed specifications which served as a project for the mechanical engineering team at South 8. As a standalone system, the manifold incorporates the gas flow equipment, the electrical and pneumatic controls, and the operator interface. All of this is built into a portable ventilated enclosure which allows easy maneuverability for use on a production floor. The mechanical engineers at South 8 were tasked with designing and building the enclosure, equipment panels, electrical and pneumatic interconnects. The operator interface includes a LabView program which was developed in-house.

Additional mechanical engineers were brought on the team to design and build the electrolyte reservoir system. The reservoir container is a commercially available gas cylinder with off-the-shelf fittings, valves, and thermocouples. The apparatus used for filling the reservoir is a structural support housed in a cryogenic dewar. The filling process uses an ethanol bath chilled with solid carbon dioxide. Therefore, the engineering team crafted a support structure for the reservoir to sit in the cryogenic ethanol bath.

A new process engineer with experience in gas and vacuum processing was hired and assigned the task of validating proper manifold installation and performance. Testing was systematically performed for gas line leak integrity, manifold volume calculations, and gas controller calibrations. A high sensitivity helium leak detector (Agilent PHD-4) was purchased to detect any leaks accurately and efficiently on the gas lines. The manifold has volumetric parameters that are necessary for accurate mass calculations. These parameters are determined by using Argon as an "ideal gas" and calculating manifold line volumes using the ideal gas law. The gas controllers include the set of eight (8) Brooks pressure and flow controllers discussed above. The calibration process involves using the desired process gas and ensuring accurate mass delivery to test vessels. Test vessels are weighed before and after filling from the manifold to ensure ±1percent mass accuracy.

The manifold design enables the LRIP goal of 500 cells per day. Changing the gas delivery from direct-to-injector to a large volume reservoir does not impact the throughput capability. The new process permits easier methods for batch logging, and quality control analysis. A Senior Chemist role was filled to assist in analytical techniques such as gas-chromatography mass spectrometry that will be used to validate the composition of electrolyte batches.

Task 2.3: Cell Injection and Crimp

Early-stage electrolyte injection methods utilized a custom-built injection and crimp apparatus. The apparatus was used to both inject electrolyte to the cell and crimp the cell to make a hermetic seal. This method was used to fabricate several 18650 cells which demonstrated the good performance of the LiGas electrolyte. Unfortunately, the crimping process required multiple stages and provided unreliable sealing.

A mechanical engineer was hired and onboarded by June 2020 and assigned the task of creating an alternative injection and sealing apparatus. An alternative sealing method was first designed so that the injection method could be built to fit.

The use of a crimping method to seal the cell was abandoned in favor of utilizing a custom designed cap assembly with proprietary built in sealing mechanisms and electrical feedthroughs. The cap is hermetically sealed to the 18650 can through a soldering method, therefore the crimping process milestone (M2.3.1) is no longer applicable to the cell fabrication. The original milestone to have no electrolyte leakage when the cell is exposed temperatures up to +60°C was tested with the modified cap design. Due to a high percentage of cells failing this test, the target was reset to +50°C. The root cause analysis revealed the solder integrity was failing at 60°C causing the leakage. The assembly and soldering process involves several trade secrets that are the result of numerous modifications to the process methods. The process has been well tested and the percentage of cells which do not leak electrolyte is already at 85percent and steadily improving. It should be noted that the currently used cap design is not commercially viable, but rather a intermediate design for validating 18650 and 27100 cells prepared with LiGas electrolytes. The commercial cell designs currently under development will utilize hermetic sealing enabled by laser welding techniques.

The injection apparatus was also modified from the proposed design as a result of the modified cell cap and electrolyte injection requirements. The apparatus includes custom designed components for making a hermetically sealed interface with the 18650 cap during injection. The rest of the apparatus is used to transfer a pre-mixed electrolyte formula from a bulk container (keg) to a cell. The pre-mix method was determined in separate tests to be more feasible and still enable high throughput and being highly interrogatable into current manufacturing methods and processes. Therefore, the injector no longer requires connections to the gas delivery manifold.

An additional engineer was hired and tasked with completing the injector design. Critical components included the structural design to support a keg of electrolyte and thermal management to ensure the assembly could maintain a desired temperature. Components that enable tight thermal management of the whole assembly were built into the system.

While the project goal was to achieve a gas manifold capable of electrolyte mixing and production for up to 500 cells per day, the current injector design was not intended to meet the LRIP goal of 500 cells per day. However, the design is scalable, and a next generation injector is in development. The 500 cell per day goal may be achieved using more efficient heating methods, and automated valve controls, however it will likely require duplicate injectors operating in parallel. Injection methods capable of meeting the 500 cell per day goal will be developed based on the design of a commercially viable cell cap.

CHAPTER 3:
Results

Summary

Demonstrating manufacturability is a critical step for any novel material system intended for use in mass production. LiGas based battery cells can be utilized as drop-in replacements for any of the cylindrical battery cell applications. However, the electrolyte formulation and injection methods require new processes to which this project aimed to develop. The project objectives focused on applying the research conducted at South 8 to design and build of electrolyte formulation and injection methods capable of a low-rate initial production target of 500 cells per day. The production methods developed would be transferrable to high volume manufacturing spaces in the electrical vehicle or grid storage markets.

Gas Cabinet Design and Installation

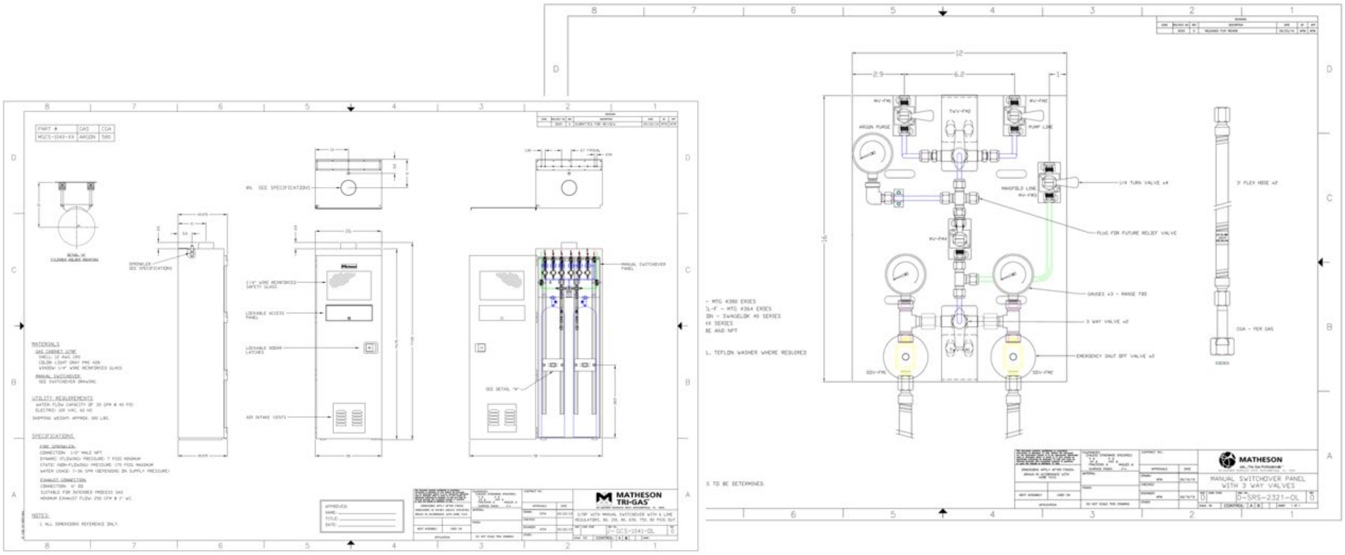
This section describes the design and installation of gas cabinets used for delivery of liquefied gas solvents used in preparation of LiGas electrolytes. Gas cabinets are ventilated enclosures used to improve workplace safety by ventilating the immediate vicinity where gas cylinders are installed. In the event of a gas leak within the cabinet, the gas would be evacuated through the duct work and minimize any risk posed to the operators or proximity workers in the laboratory or manufacturing space.

Design:

A total of eight (8) gas cabinets were specified to provide the capacity of the LRIP project. A base model cabinet was selected from Matheson Tri-Gas and a custom gas panel was prepared for each cabinet. The gas panels were designed at South 8 and assembled by Matheson prior to delivery. Elements important to safety were incorporated in the cabinets including gas sensors and emergency shut-off valves. The gas sensors are tied to emergency shut-off valves connected immediately downstream of the cylinders so that if any hazardous gas leak occurs the gas flow is terminated at the source.

The gas cabinets are built with two different designs (**Figure 2**):

Figure 2: Gas Cabinet Enclosure and Panel Drawings. Finalized Gas Cabinet Design.



Source: South 8 Technologies

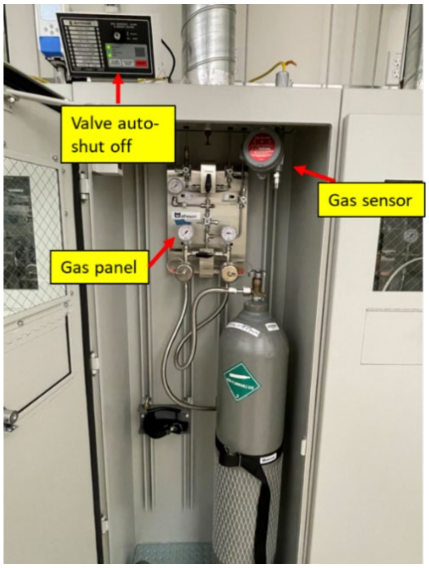
One design is exclusively for the Argon cylinder cabinet (**Figure 3**). The Argon cabinet provides Argon to the gas delivery manifold, and is used as a utility gas for purging, leak checking, and equipment operation. There are six (6) independent gas delivery lines coming from the Argon gas cabinet. Each line is regulated to a specific pressure to accommodate the specific use such as pump & purge, pneumatic controls, emergency shut offs, etc.

Figure 3: Installed Argon Gas Cabinet Panel. Argon Gas Cabinet Panel with Multiple Delivery Lines for Different Pressure Requirements.

Source: South 8 Technologies

The second gas cabinet design is for the gases being used for electrolyte formulations (Figure 4). The panel design used in these seven (7) other cabinets consists of a dedicated Argon purge and vacuum line so that any new cylinder exchange will not contaminate the electrolyte gas delivery manifold.

Figure 4: Installed Electrolyte Gas Cabinet Panel. Electrolyte Gas Cabinet Panel with Gas Sensor and Auto-Shut off Relay.



Source: South 8 Technologies

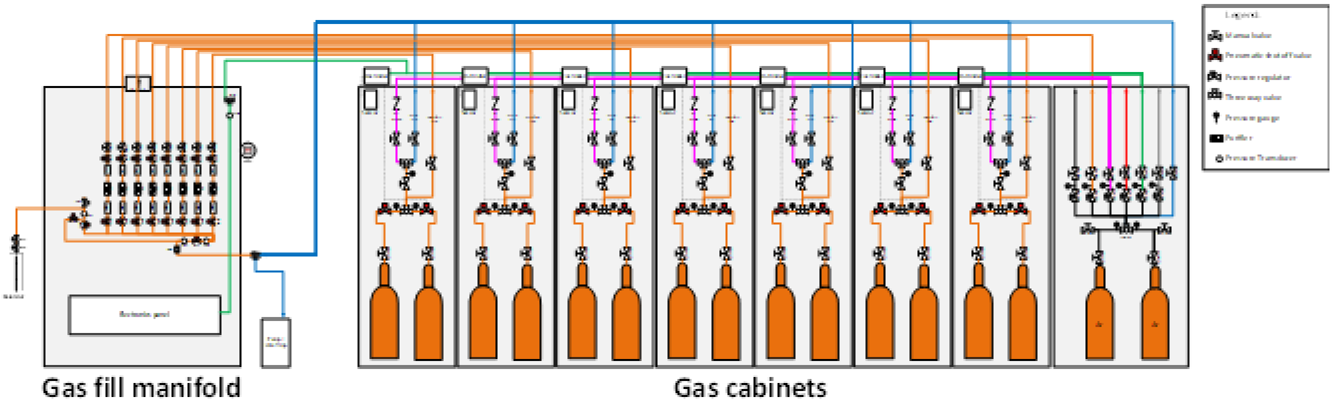
Each cabinet has a capacity for two (2) gas cylinders so that immediate cylinder switchover is possible, thereby minimizing downtime of the LiGas electrolyte preparation process.

Installation:

The cabinets were initially installed in summer of 2021 in South 8's facilities, but connection to the gas manifold was postponed due to a pending facility move. The facility move in 2022 was followed by cabinet installation by third party contractors during the tenant improvement project in the new South 8 facilities as of July 2022. Installation included bolting the cabinets to the floor for stability, connecting them to ventilation, and verifying sufficient air flow through the cabinets. The cabinets also have water sprinkler connections which were installed to the house fire sprinkler water lines.

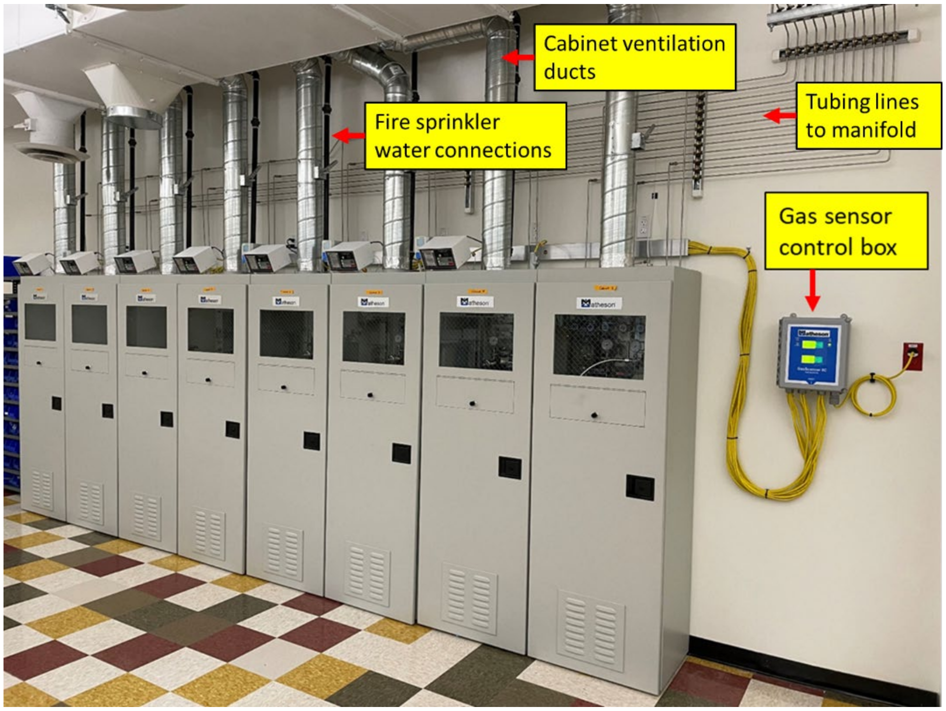
Installation further included connection of the gas delivery lines between the cabinets and the gas manifold (**Figure 5**). The tubing connections were completed through a third-party contractor and helium leak checked by South 8 using a high sensitivity helium leak detector to ensure all fittings were installed properly. South 8 additionally installed the gas sensor controller box provided by Matheson which reads the sensor output and provides signal to the shut-off valve relay boxes. The completed setup is shown in **Figure 6**.

Figure 5: Cabinet to Manifold Tubing Connections. Schematic of the Gas Cabinet to Gas Manifold Tubing Connections.



Source: South 8 Technologies

Figure 6: Eight Gas Cabinet System Installed. Eight Gas Cabinet System fully installed to Ventilation, Fire Sprinkler System, and Manifold Tubing. Gas Sensor Controller Installed to all Gas Cabinet Sensors.



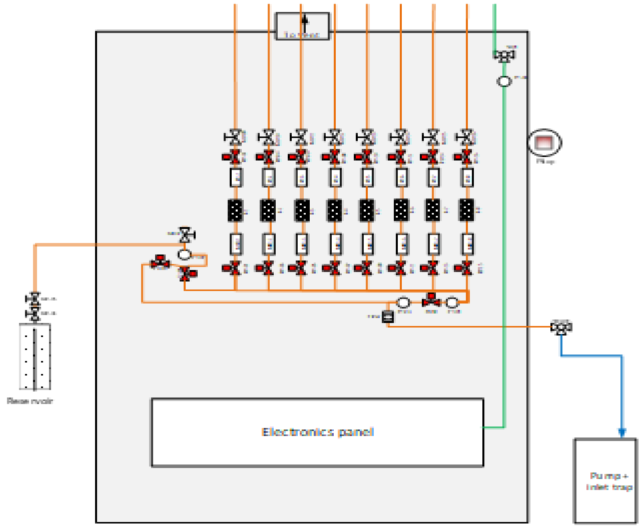
Source: South 8 Technologies

Gas Manifold Design, Build, and Validation

Design:

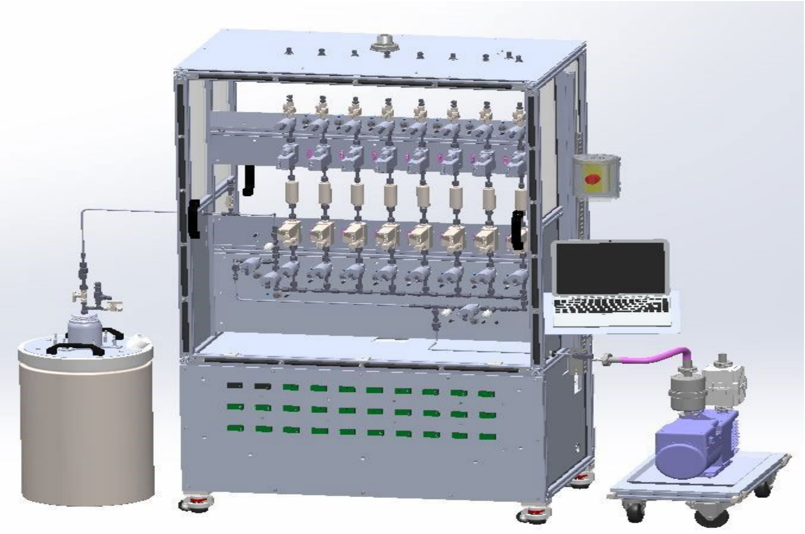
The gas manifold was designed to accommodate up to eight (8) different gases to match the eight (8) gases used in the gas cabinets. A diagrammatic drawing and a 3D CAD drawing are shown in **Figures 7 and 8**. The gas control hardware was modeled from an in-house designed R&D manifold and includes higher flow rates for rapid filling of LiGas electrolyte reservoirs. The operating pressures and flow rates are tunable using pressure and mass flow controllers supplied by Brooks Instruments (pressure controller model SLA5810, mass flow controller model SLA5851). The option to tune these parameters permits optimization of the gas flow to meet the 1percent mass fill accuracy target. In addition to these controllers, each gas line incorporates high purity diaphragm valves (Swagelok, APTech), pressure transducers for monitoring the reservoir and vacuum line pressures (ProSense), and optional purifier slots (Matheson). The gas flow instrumentation is installed on modular aluminum supports and connected to an in-house designed electronics board.

Figure 7: Manifold and Cabinet Schematic. Schematic of the Gas Manifold Connections and Components.



Source: South 8 Technologies

Figure 8: Drawing of Manifold in Enclosure with Electrolyte keg. Drawing of the Final Gas Manifold Design including the Operator Computer, Vacuum Pump, and Electrolyte keg Installed in a Cooling Dewar.



Source: South 8 Technologies

All components except the pump and keg are enclosed in an in-house designed aluminum t-slot framed enclosure. The enclosure has removable panels for easy access to the manifold instrumentation and easy visualization of the instrumentation during operation. The enclosure has a sealed ventilation duct connection that creates negative pressure within the enclosure, minimizing the risk to operators or proximity workers in the event of a gas leak on the manifold. An E-Stop button is built in for an emergency which shuts off all valves and controllers, terminating flow to the reservoir.

The lower section of the enclosure houses the electronics panel (**Figure 9**). The electronics panel communicates between the instrumentation and operator computer. It also is connected to digital readouts for all controllers, and temperature monitors, as well as LED indicators for valve status.

Figure 9: Electrical Panel for Manifold. Image of the Electrical Panel Build under the Manifold

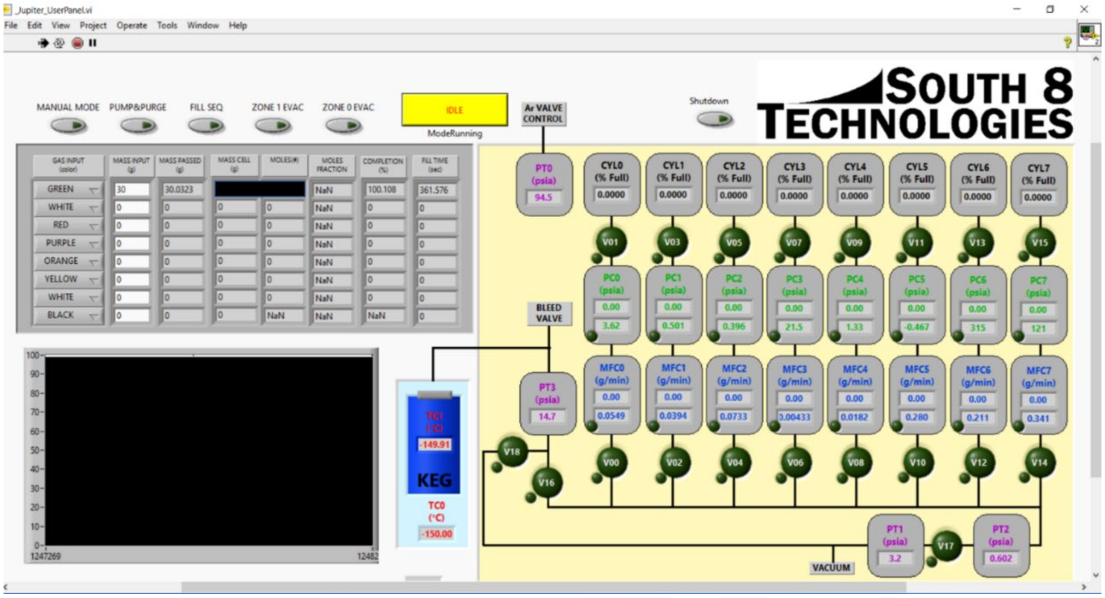
Source: South 8 Technologies

The electrolyte keg is connected to the manifold via an external tubing connector. It is mounted in a cryogenic dewar used to sustain a cold bath below 0 degrees Celsius so that electrolyte gases will condense in the keg. The keg volume is approximately one (1) gallon which is sufficient electrolyte for five hundred (500) fills of typical 18650 cells. The keg is designed for real-time monitoring during the electrolyte preparation process and includes temperature monitors, and pressure transducers. The dewar is custom modified for mounting the keg and can accommodate kegs of smaller or larger sizes.

The standard operating procedure for the manifold uses a LabView program (**Figure 10**) which enables fully automated operation of the gas fill sequence. The LabView program was custom developed in-house and integrates a user panel, instrument communications, gas physical properties tables, and process calculations. The program also enables digital logging of all the process parameters and readouts for historical tracking and troubleshooting. If there is a need for troubleshooting, the LabView platform enables easy modifications of any program code.

The details of the program were seamlessly integrated so that the user panel is limited to a few functions and minimizes the need for extensive operator training. As seen in Figure 9, the user panel is simplified to an electrolyte recipe input table, and five other buttons. Process instrumentation readouts are displayed on a rough diagram and used to assist troubleshooting processes. General operator use would only require attention to sequence progress and keg fill status.

Figure 10: Labview User Panel. Screenshot of the LabView User Panel showing the Control Buttons and Electrolyte Gas Recipe Input.



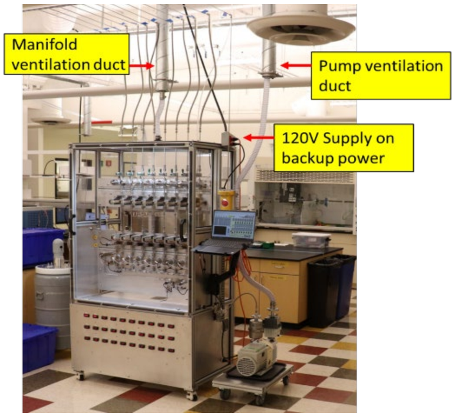
GAS INPUT (color)	MASS INPUT (g)	MASS PASSED (g)	MASS CELL (g)	MOLES(#)	MOLES FRACTION	COMPLETION (%)	FILL TIME (sec)
GREEN	30	30.0323			NaN	100.108	361.576
WHITE	0	0	0	0	NaN	0	0
RED	0	0	0	0	NaN	0	0
PURPLE	0	0	0	0	NaN	0	0
ORANGE	0	0	0	0	NaN	0	0
YELLOW	0	0	0	0	NaN	0	0
WHITE	0	0	0	0	NaN	0	0
BLACK	0	0	0	NaN	NaN	NaN	0

Sources: South 8 Technologies

Build:

The manifold was built as described above, and several additional facilities modifications were required to make the manifold operational. This includes electrical, ventilation, and tubing installation which was performed by third party contractors. As part of the tenant improvement project for our laboratory, electrical and ventilation modifications were included to accommodate the manifold (**Figure 11**).

Figure 11: Facilities Improvements for Gas Manifold. Facilities Improvements for the Manifold include Two Duct Drops for Ventilation of the Manifold and Rough Pump Exhaust, and a 120V Power Supply on a Backup Power Circuit.



Source: South 8 Technologies

The ventilation installation included vent drops for both the manifold and vacuum pump. The manifold duct is sealed so that the manifold enclosure is always under negative pressure and minimizes the possibility of operator exposure in the event of a gas leak. The vacuum pump is connected to a separate duct vent which removes exhaust of the manifold operation. In addition, an electrical drop connected to an emergency backup power circuit was installed for minimizing risk associated with power outages.

The tubing installation was performed by a third-party contractor, Apex Mechanical, according to specifications determined by South 8. The tubing is all 316SS seamless tubing connected by tube fittings joining the gas cabinets to the gas manifold. All parts were sourced from Swagelok and handled to minimize contamination. After completion of the tubing installation, the leak-free integrity was verified by South 8 using pressurized helium and a high sensitivity helium leak detector sourced from Agilent. The completed manifold is shown in Figure 12.

Figure 12: Completed Manifold Vuild. Image of the Completed Manifold Build.

Source: South 8 Technologies

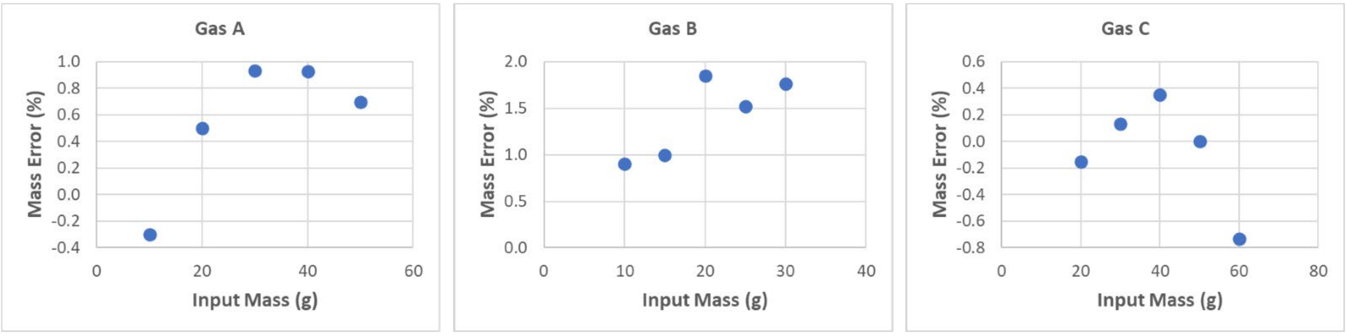
Validation:

The manifold operation was validated by testing the mass accuracy of single and multiple gas fills into test vessels. The standard operating procedure for fills with the LabView user panel was used for filling single gases into evacuated test vessels. The mass of the test vessel is recorded before and after the fill with an accuracy target of 1percent of the expected mass.

The results of single gas fills into a test vessel using three different gas lines are shown in **Figure 13**. **Figure 13** shows the percent mass error versus the expected mass of gas delivered to the test vessel. Gas B shows the best accuracy, with mass errors of less than ±

1percent. Gases A and C show mass errors slightly larger than ± 1percent for some of the input mass targets. Fine tuning of the controllers is expected to bring the errors for these gas lines to within the ± 1percent target. The remaining gas lines on the manifold will be validated is a similar manner with their respective gases.

Figure 13: Single Gas Fill into Test Vessel. Mass Accuracy results of a Single Gas fill into a Test Vessel. The Accuracy is within 2 Percent of the Target Masses.



Source: South 8 Technologies

The results of a multi-gas fill into a keg are shown in **Figure 14**. The results illustrate the iterative process improvements involved in the calibration process. Fill 1 had a fill error of minus 2percent. The gas controllers were then tuned and recalibrated. Fill 2 and Fill 3 were performed after Fill 1 and show excellent performance within ±1percent of the target masses. The 500-gram fills are achieved in approximately 75 minutes which is sufficient for achieving throughput targets of 500 18650 cells per day.

Figure 14: Multi-Gas fill into Keg. Mass Accuracy Results of Multi-Gas fills into a keg.

Gas	Flow rate (g/min)	Fill 1 Mixture	Fill 2 Mixture	Fill 3 Mixture
Gas A	5	100 g	100 g	100 g
Gas B	10	400 g	300 g	300 g
Gas C	5	0 g	100 g	100 g
Fill Error	--	-2 %	-1 %	-1 %

Source: South 8 Technologies

Electrolyte injection

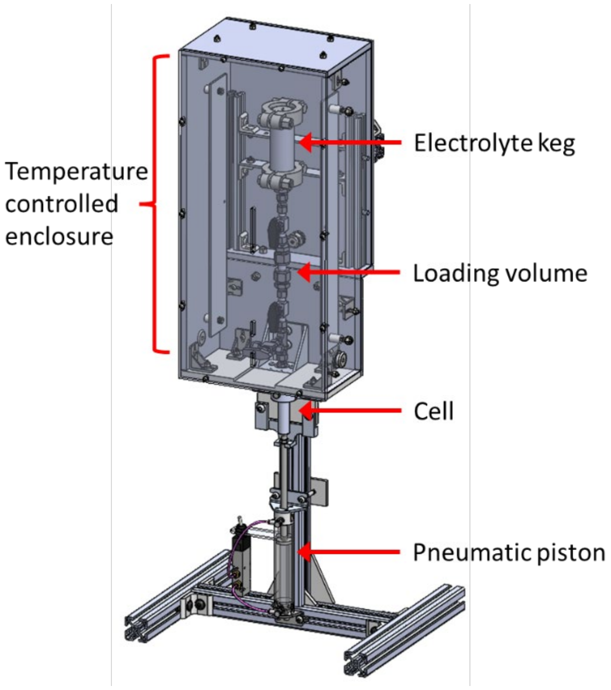
The electrolyte injection system is used to transfer prepared electrolyte from a keg to a battery cell. The system utilizes the thermophysical properties of the electrolyte to deliver pre-determined masses efficiently and accurately into battery cells. This report highlights the non-confidential aspects of the design, build, and validation of the system, while the finer design details will not be disclosed.

Design:

The injection system works under a similar principle as common electrolyte injection systems; there is a pre-mixed keg of electrolyte which is then syphoned off in small portions via a "loading volume" and injected into each cell (**Figure 15**). The loading volume is a measured vessel sized for the appropriate mass of electrolyte to be delivered to the cell. The keg, loading volume, and cell are separated by valves which are actuated so that the loading volume is first filled from the keg and the cell is then filled from the loading volume. The system was designed with focus on critical parameters such as thermal control, and seal integrity, as discussed below.

A drawing of the electrolyte injector system is shown in **Figure 15**. The drawing shows the framework of the temperature-controlled enclosure and the components of the keg to cell transfer. The insulated enclosure provides a uniform temperature for the keg and loading volume. The cell is outside the enclosure and remains at room temperature.

Figure 15: Electrolyte Injection System Drawing. 3D CAD Drawing of Electrolyte Injection System



Source: South 8 Technologies

To better align with standard industry systems, all cells are held at room temperature during the fill process. However, the electrolyte within the keg has slightly varying density depending on temperature. To maintain tight (<1percent) mass distribution in the electrolyte injection step, the keg is held at a moderate temperature (~+40 °C). Because boil off also lowers the temperature of the keg, the temperature is monitored and controlled using electronic controllers.

Additional connections to a vacuum pump and pressurized argon are included in the design but not shown explicitly in **Figure 15**. These connections allow the operator to effectively remove air contamination from the injector headspace and ensure the cell is under vacuum prior to injection. Having the cell under vacuum prior to injection allows maximum wetting of electrodes and capacity output of cells. The design permits the cell to be evacuated prior to gas injection, and subsequently be sealed shut after injection. A proprietary injection head design has been developed to ensure no leakage throughout the process.

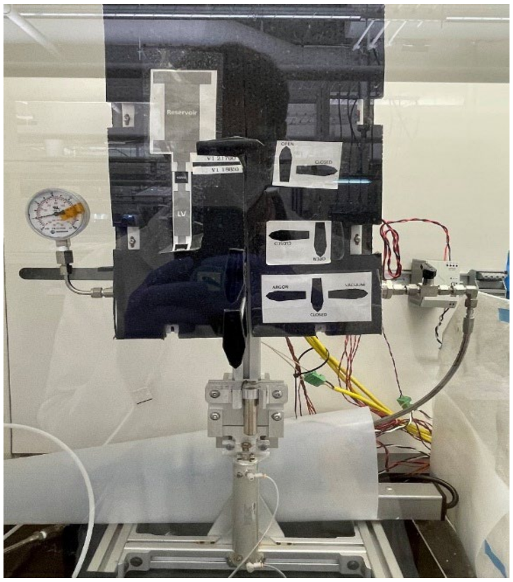
An additional key element to the injector system is the pneumatic piston which provides support for pressing the cell against the injection head. The piston is sized based on the force due to the electrolyte vapor pressure once injected in the cell. The sealing interface uses a secured elastomeric o-ring in the injection head to provide a leak-free seal. Adding the electrolyte to the cell requires a process which forces the electrolyte through a narrow opening on the cell cap and then seals the cap after the electrolyte is transferred. This has been achieved using some proprietary techniques which have been utilized successfully.

This system has been designed with scale up capacity in mind. Ease of use and parallel fills will be critical to the end product of achieving 500 cell/day production capacity. The current progress made to demonstrate the method and design will be scaled up over the next several months and allow for rapid ramp up of our cell production.

Build:

The injection system was built in-house according to the design specifications discussed above. During operation a small quantity of electrolyte vapor is released during as the cell disengages from the injection head. Although the vapor is non-toxic, the system is operated inside a fume hood to minimize risk of operator exposure to the vapor. **Figure 16** shows the system setup in a fume hood with all connections installed. The temperature-controlled enclosure was found to require additional insulation; therefore, a layer of black foam insulation covers the exterior of the enclosure.

Figure 16: Installed Injection System. Image of the Electrolyte Injection System fully Installed including Temperature Controllers, Argon, and Vacuum Connections, and an 18650 loaded in the holder.



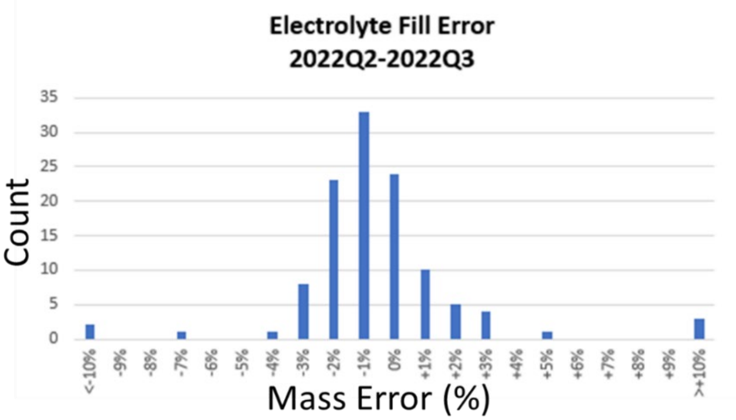
Source: South 8 Technologies

Validation:

The first step to evaluate proper function of the injector system was to ensure the system was hermetically sealed with the cell engaged to the injector head. This was achieved by pressurizing the system and using an electronic leak detector around the injector head o-ring seal. The seal between the injector head and the cell did not show leakage above the detection limit of the leak detector.

The injection process required optimization of parameters such as heater temperature and step timing to achieve reproducible accurate mass injections. Representative data of the accuracy for electrolyte mass transferred to the cell is shown in **Figure 17**. Although most of the fills lie within the project target of ±1percent error, there is still significant room for improvement in spread of mass error. One issue encountered was day-to-day variations in the accuracy. This variation could be attributed to keg electrolyte preparation variability or room temperature variability, either of which could impact the fill masses on a given day.

Figure 17: Fill error for Injection System. Statistical Plot of Mass Error for cell fills on Injection System.



Source South 8 Technologies

Commercial cell production demands that every aspect of the cell be highly consistent within a batch and from batch-to-batch to allow for well-balanced module and battery systems to ensure systems (e.g., grid storage or electric vehicles) perform within spec over the course of their lifetime. The electrolyte is no exception here and customers have shared targets of +/- 1percent mass accuracy in individual components within the mixed electrolyte and +/- 1percent mass accuracy in cell electrolyte injection. This project achieved the mixed electrolyte accuracy (**Figure 14**) and have targets to further tighten these numbers to +/- 0.5percent component mass accuracy. Electrolyte mass injection currently has a spread showing most fills between +/- 3percent electrolyte mass accuracy. This system is continuously being refined for tighter thermal and volume controls to improve this accuracy and allow high repeatability.

Using lessons learned from this project, a new injection system is already being developed and will be functional by mid-2023. This new system will be highly automated, ensure high accuracy, and be capable of up to 500 cells per day production. Between both the electrolyte gas mixing manifold and the electrolyte injection systems, these will be used to demonstrate high-rate capability manufacturing of the LiGas technology and allow tech transfer to commercial cell manufacturers soon, requirements set forth by the cell manufacturing partners that South 8 is already engaged with today.

Larger Scale Production:

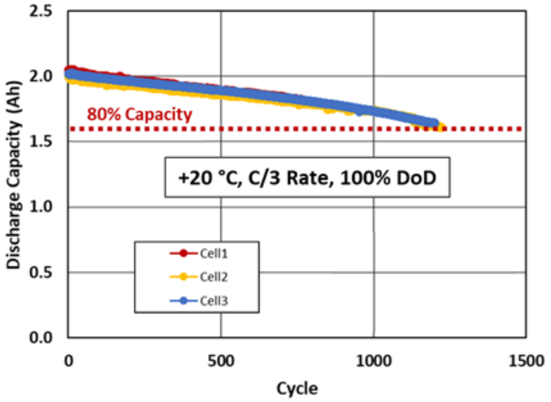
The existing design provides a system which enables efficient and accurate electrolyte injection to single 18650 or 21700 battery cells. However, to achieve the target throughput of 500 cells per day, the system will require improvements that allow faster injections or multiple injections in parallel. The simple and compact design of the system lends itself to reproduction of multiple systems that may operate in parallel. Alternatively, multiple injection ports may be used on a single injector apparatus so that multiple cells are filled in parallel.

Higher throughput production may also be enabled by design improvements for a more efficient process. One such improvement is automation of the injection process. The existing valve system is all manual and requires significant operator interaction for valve actuation. This process has a straightforward path for improvement by replacing manually actuated valves with pneumatically actuated valves that can be controlled by a pneumatic panel and CPU. In addition to saving operator time, it would minimize variability due to human error, improve safety, and improve accuracy. Another improvement would be the thermal control mechanism. The current design requires long thermal equilibration time to ensure accurate electrolyte mass transfer. More efficient and uniform heating of the keg and loading volume would enable faster and more consistent/accurate electrolyte injections, controls which are being designed into the new injection system under design currently.

Cell Performance:

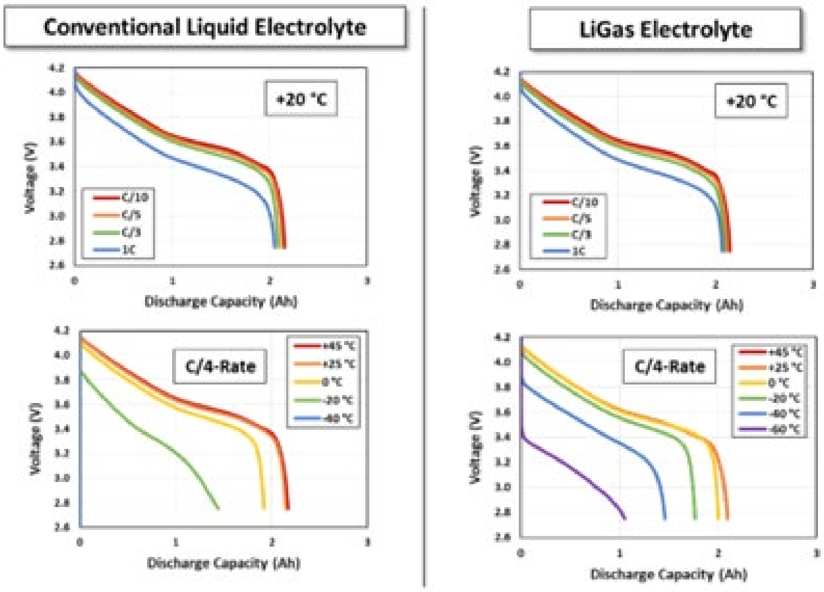
Though not a direct objective of this project, several cells were filled using the LiGas electrolyte using mechanisms developed through this project. 18650 prototypes of 2Ah capacity and having graphite-NMC chemistry were built and tested. **Figure 18** below shows the excellent cycle life of the LiGas cells, having well over 1,200 cycles under 100percent depth-of-discharge. This is very comparable to conventional liquid electrolytes and shows the robustness of the LiGas chemistry. **Figure 19** shows a comparison of the power and temperature performance of two identical cells, one using a conventional liquid electrolyte and the other using the LiGas electrolyte. Both have comparable power, with the LiGas showing slightly higher power output. The temperature performance of the liquid cell is shown to drop considerably at -20 °C and with no performance below this temperature. In contrast, the LiGas cell shows considerably improved low temperature performance, having excellent discharge capacity of ~40percent down to -60 °C. This has been validated by several groups and is the highest performance low-temperature cell available today. **Figure 20** compares three cells undergoing a fast-charging protocol put forth by the Department of Energy (3 cycles C/3 charge, 1 cycle fast-charge). The cell using LiGas electrolyte is shown in red, and the degradation is seen to be far superior to the curves shown in yellow (identical jelly roll, using liquid electrolyte) and blue (commercially available Samsung cell). This demonstrates the excellent fast charging kinetics of the cells using the LiGas electrolyte. This is due to (a) excellent conductivity of the electrolyte and (b) the robust and ionically conductive solid electrolyte interphase (SEI) layer on the graphite anode preventing lithium metal plating and capacity degradation.

Figure 18: LiGas Cell Cycle Life. Cycle Life of 3 LiGas Cells showing >1,200 cycles.



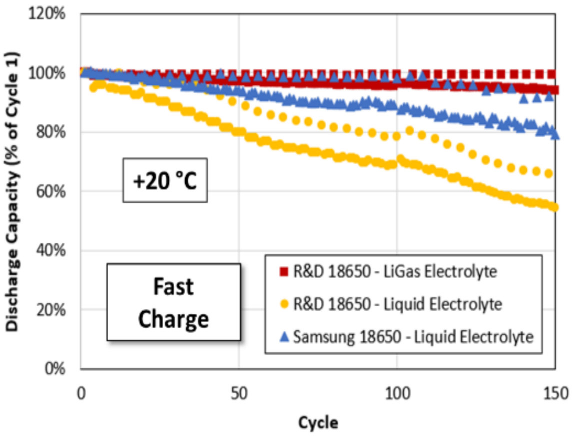
Source: South 8 Technologies

Figure 19: Power and Temperature Performance. Comparison of the Power and Temperature Performance of two Identical Li-ion cells, one using a Traditional Liquid Electrolyte and the other using a LiGas Electrolyte.



Source: South 8 Technologies

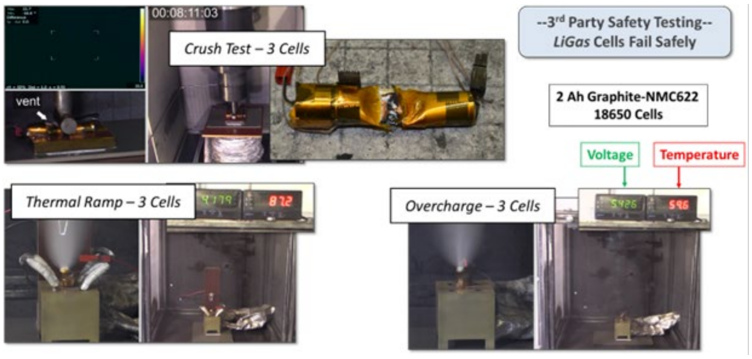
Figure 20: Fast Charge Performance. Fast-Charge Performance Comparison between a LiGas cell (red), an Identical Cell using Liquid Electrolyte (yellow) and a Commercial Samsung Cell (blue).



Source: South 8 Technologies

In addition to the excellent performance of the LiGas cells, the LiGas electrolyte has also demonstrated excellent safety with the elimination of thermal runaway. Under any physical or electrical abuse, the cell may safely vent the non-toxic LiGas solvent leaving nothing behind other than a dry salt. The dry salt itself is non-conductive and so any thermal runaway reactions are therefore shutdown. This has been validated at Sandia National Labs through crush, thermal ramp, and overcharge testing (**Figure 21**). Under these tests, the LiGas cells are shown to vent and fail safely. The inherent safety of the cells is a breakthrough in the industry which is ripe for a new cell chemistry. This enhanced safety can enable a new generation of battery technology which will transform the energy storage industry.

Figure 21: Safety Characterization. Sandia National Labs Testing and Safety Characterization of Li-ion 18650s using LiGas Electrolyte. Testing shows Cells can Vent the Electrolyte fail safely under Physical or Electrical Abuse Conditions.



Source: South 8 Technologies

While the current project was focused on scaling the LiGas electrolyte, demonstration of the LiGas electrolyte technology in prototype cell packages was completed and the chemistry is expected to scale well to more commercial ready Pre-A cells currently under development (**Figure 22**). **Figure 22** also summarizes the typical performance of the prototype cells compared to projected performance of South 8's Pre-A cells.

Figure 22: Images and Comparison of Prototype Cells Utilized During this Project Performance and the More Commercial Ready Pre-A Cells Currently Under Development.

Performance Metric	Prototype Packages	Commercial Ready Package
Cycle Life	1,200	1,500
Temperature Range	-60 to +60 °C	-60 to +60 °C
Maximum Discharge Rate	2C	5C
Cost Projection ($/Wh)	-	80
Specific Energy (Wh/kg)	120	300
Energy Density (Wh/L)	350	800

Source: South 8 Technologies

CHAPTER 4:
Conclusion

In this project, South 8 demonstrated a manufacturing method for LiGas electrolyte batteries utilizing a gas distribution and delivery system and a LiGas electrolyte injection system. The manufacturing development achieved at South 8 reduces the risk of larger adoption of LiGas electrolyte into existing gigafactories. LiGas electrolyte has intrinsic properties that allow safer operation, lower cost, and better performance in lithium-ion batteries.

Through this project, a LRIP of the electrolyte was demonstrated. A TRL of five (5) and Manufacting Readiness Level (MRL) of four (4) were achieved through this project. Using lessons learned from this project, a new cell design and injection system will be deployed in mid-2023, which will raise these levels to TRL six (6) and MRL six (6). This will allow further demonstration projects of the technology at the kWh scale (e.g., microgrid batteries), further raising both TRL and MRL levels. Deployment of these projects will accelerate the adoption of the technology.

The adoption of the LiGas electrolyte technology demands three key achievements: (1) demonstrated improvement in cell performance, (2) demonstrated cell packaging, and (3) demonstrated manufacturability. This project reduced the adoption risk associated with the LiGas electrolyte in all three of these categories. The gas electrolyte mixing, and injection systems demonstrated manufacturability, which allowed the key metrics in cell performance to be demonstrated. The injection system was also a driving force into how the cell packaging must be designed to interface with the manufacturing systems. The future adoption of the technology by larger cell manufacturers will accelerate the commercialization and deployment of the LiGas technology. Once deployed, the technology is expected to lower the cost of battery energy storage systems and electric vehicles considerably. The reduced cost will be realized through: (1) faster manufacturing and lower scrap rate, (2) improved cell energy for lower $/kWh, (3) more efficient and lower cost battery and modules due to improved safety and low temperature performance, and (4) safer cells leading to reduced insurance requirements on system installations. The lower costs realized here will accelerate carbon emission reductions in the state of California and beyond.

The technology demonstrated excellent safety and low temperature performance, making it a highly desirable energy storage device for beach head markets in cold climates. These industries include arctic sensing, microgrids, and the department of defense (DoD). As the DoD currently has a big push to bring battery cell technology back to domestic manufacturing, the LiGas electrolyte is a prime opportunity to achieve these goals. As the technology gains traction within these beachhead markets, the larger energy storage system and EV markets will begin to adopt the technology, reducing the cost to below today’s Li-ion levels (below $100/kWh).

The primary goal of any grid-storage battery is to benefit ratepayers through lower cost and lower carbon emissions by enabling further penetration of renewables and increasing grid efficiency. This will only be possible with lowering the cost of battery technologies through low cost, high cycle life, and high energy. Further, with larger battery systems, safety is of primary importance since systems may be installed near populated or environmentally sensitive areas. With the penetration of South 8’s LiGas electrolyte technology, a lower cost and safer battery will be made to the California grid and beyond. Finally, with increasing renewables (e.g., solar, wind) introduced to the grid, the increased deployment of grid batteries will improve grid reliability for the ratepayer as well.

Project Deliverables

- Cabinet Design Report
- Gas Injection Report
- Final Report (this document)