**CESI Laboratory Safety Plan**

 **Pressurized Oxygen Conducting Electrochemical Test Stand (POCET)**

Washington State University

Department of Mechanical Engineering

Engineering Teaching and Research Laboratory 310

**Lab Principle Investigator:**

Dr. Dustin McLarty

**Last Updated:**

October 2, 2018

Version 3

Last revised by John Drazin

**Authors:**

Ryan Hamilton, Jeff Collins, Dr. John Drazin, Dr. Dustin McLarty

**Contents**

[1 Scope of Work 4](#_Toc526336029)

[2 CESI Lab Safety Information 4](#_Toc526336030)

[2.1 Lab Commitment 4](#_Toc526336031)

[2.2 Personnel Participation 4](#_Toc526336032)

[2.3 Communication of Safety 4](#_Toc526336033)

[3 Identification of Safety Vulnerabilities 4](#_Toc526336034)

[3.1 Oxygen System’s Safety 8](#_Toc526336035)

[3.2 Hydrogen System’s Safety 9](#_Toc526336036)

[3.3 Nitrogen and Argon System’s Safety 11](#_Toc526336037)

[3.4 Gas Delivery Summary 11](#_Toc526336038)

[3.5 Exhaust Gas System’s Safety (CO, CO2) 11](#_Toc526336039)

[3.6 Exhaust Gas Summary 12](#_Toc526336040)

[3.7 Fuel Cell Sealing Assembly Safety 12](#_Toc526336041)

[3.8 Fideris Equipment Rack 14](#_Toc526336042)

[3.9 Furnace Safety 15](#_Toc526336043)

[3.10 Laboratory Power Safety 15](#_Toc526336044)

[3.11 Hydrogen System’s Safety 15](#_Toc526336045)

[3.12 15](#_Toc526336046)

[4 Description of the POCET Equipment 15](#_Toc526336050)

[4.1 Fideris Equipment Rack 16](#_Toc526336051)

[4.2 Delivery Module with Purge (Hydrogen) 16](#_Toc526336052)

[4.3 Delivery Module (CO/CO2) 16](#_Toc526336053)

[4.4 Delivery Module with Purge (Oxygen) 16](#_Toc526336054)

[4.5 Delivery Module with Purge (Nitrogen) 17](#_Toc526336055)

[4.6 Emergency Stop and Power Supply 17](#_Toc526336056)

[4.7 1kW Electric Load Bank 17](#_Toc526336057)

[4.8 Temperature Controller 17](#_Toc526336058)

[4.9 Pressure Control Module 17](#_Toc526336059)

[4.10 Liquid Delivery Module 17](#_Toc526336060)

[4.11 Liquid Pressure Delivery System 18](#_Toc526336061)

[4.12 Exhaust Gas Module Controller 18](#_Toc526336062)

[4.13 Exhaust Gas Module 18](#_Toc526336063)

[4.14 Humidifier Assembly 18](#_Toc526336064)

[4.15 Heat Exchanger 18](#_Toc526336065)

[4.16 Furnace 19](#_Toc526336066)

[4.17 Gantry 19](#_Toc526336067)

[4.18 Gas Regulators 19](#_Toc526336068)

[5 Standard Operating Procedures 19](#_Toc526336069)

[5.1 Purpose and Applicability 19](#_Toc526336070)

[5.2 PROCESS/EQUIPMENT OVERVIEW 20](#_Toc526336071)

[6 Equipment Integrity 21](#_Toc526336072)

[7 Safety Audit Schedule and Procedures 21](#_Toc526336073)

[8 External Safety Reporting and documentation 21](#_Toc526336074)

[8.1 Safety Reporting 21](#_Toc526336075)

[9 Safety Plan Approval 22](#_Toc526336076)

[10 References 22](#_Toc526336077)

1. Scope of Work

The following document is meant to ascertain a multitude of safety risks associated with the creation of a research laboratory sized prototype gas pressurized solid oxide fuel assembly and a plan to mitigate or eliminate the risks associated with the apparatus during routine scientific operation. The review will cover the gas inlet system, gas exhaust system, SOFC assembly, furnace assembly, and computer controls. In addition standard operating procedures have been addressed for different proposed experiments.

1. CESI Lab Safety Information

## Lab Commitment

The Washington State University (WSU) Clean Energy Systems Integration (CESI) lab is committed to create, maintain and enhance a safe and healthful environment for all individuals associated with the lab, including students, faculty, staff, and visitors.

## Personnel Participation

Laboratory personnel are integral to creating a safe environment for themselves, visitors and future personnel. Preventing injuries and keeping the lab in operation are major concerns. Personnel are required to know and comply with safety guidelines and policies for activities undertaken. It is important for personnel to report unsafe conditions to the Principal Investigator, immediate supervisor, or the Environmental Health & Safety Department.

## Communication of Safety

Safety in the CESI lab is communicated through: the laboratory safety plan, equipment specific safety plans, project specific safety plans, personnel training, design reviews emphasizing safety concerns, internal and external audits of equipment and ongoing projects, and reporting of both positive and negative safety related events.

### Laboratory Layout



**Figure 1:** Room Layout and Locations of Emergency Supplies

The facility contains a fuel cell assembly housed in a high pressure furnace that provides the heat necessary for proper operation of the fuel cell. All electronics that control and monitor the experiment were designed by Fideris. Gas to the furnace is regulated by mass flow controllers located within the Fideris equipment rack.

All components exposed to high pressures are equipped with burst discs that will break the fluid circuit if pressures exceed 165 psi. The gas supply lines and fittings are rated for 8000psi, and the furnace is rated for 200 psi. Therefore, the burst discs will rupture before any of the equipment approaches a critical pressure.

In the event of power loss or malfunction, the system is designed to automatically stop the flow of gasses into the experiment. The system has been outfitted with auxiliary power units (APUs) that will allow the equipment to be properly shut down in case of a power outage.

 Compressed gas cylinders are located in the closets directly outside of the lab, labeled 310A and 310B. Oxygen, Nitrogen and Argon are located in 310A. 310B is exclusively for Hydrogen. General compressed gas safety posters are mounted in each closet. The lab is equipped with gas detection equipment for Methane, Hydrogen and Carbon Monoxide. If these gasses are detected in the room, an alarm will sound.

A fire extinguisher is located on the wall next to the eyewash station (Figure 1). Safety Data Sheets are kept in a binder near the computer used to interface with experiment.

## Lab Training Procedures

Laboratory training takes place on two levels: general lab conduct and safety, equipment specific safety procedures. Training is administered by the lab principal investigator, or a seasoned graduate student. Initial personnel training is conducted upon a new members joining of the lab. Equipment/experiment specific training for lab personnel is required for participation in activities involving potentially hazardous equipment. The potential new operator(s) will be paired with a mentor(s) that have been trained on the equipment/experiment. The first step of the equipment/experiment training is for the new operator to research the procedures and history of equipment/experiment and ask questions on anything which is unclear. An observation step, where the mentee observes the mentor performing the various operations/procedures with the opportunity to ask questions, occurs prior to any equipment operation. The new operator demonstrates proficiency via oral examination and hands-on demonstration of the operating procedures. This marks the completion of the equipment/experiment training and they are allowed to operate equipment/experiment.

## Safety Reviews and Documentation

When new experiments are designed or new equipment is brought into the lab potential hazards are analyzed and documented following the identification of safety vulnerabilities (ISV) procedures, followed by the development of an incident mitigation plan, experiment/equipment operating procedures, incident response procedures, and procedures for auditing the experiment/equipment safety throughout its lifetime.

## Management of Procedure Changes

To make changes to any experiments, the following procedures and documentation must be developed, reviewed, and approved prior to implementing the change. When a change is proposed, it is necessary to review ISV and SOP’s associated with the experiment to make sure no new hazards have been created and that operational procedures remain current.

A suggested management of change process is listed below. It is recommended that this process be followed, but a different process can be used if it makes sense for your experiment, and the process is agreed upon by all stakeholders.

1. A need to change EXPERIMENT is identified.
2. The change is discussed with at least two knowledgeable members of the lab to get a second opinion on the necessity of the change. Details of what should be changed and how are discussed.
3. A proposal for change is created, stating the need for change and details of what the change will include. This proposal will include:
	1. Relevant engineering standards,
	2. Necessary sizing calculations,
	3. Details of implementation of the change, and
	4. How the change affects this document, including Safety Failure/Hazards matrix and operating procedures.
4. The full proposal is discussed with the PI and experiment operators. If it is agreed upon the details of the change, the change is implemented, otherwise the change is discarded or is re-designed. The PI has the final decision on approval.
5. Implement the changes. Document these changes (i.e. the proposal) in the experiment folder on the CESI lab drive. Communicate the implementation and completion of the changes with others in the lab through the proper lab Slack Channel.
6. If procedures are affected by the change, update this document with new operating procedures. Detail any changes or updates to the document in the changelog at the end of the document.
7. If new maintenance / safety concerns arise from the change, note them in the proper areas in this document. Statement of Project Experience
8. Identification of Safety Vulnerabilities

 This section assesses potential hazards associated with the scientific operation of a prototype gas pressurized oxygen conducting electrochemical test stand (POCET) stand during routine high temperature/high gas pressure electrical power generation operation and enumerates how the potential hazards have been mitigated or eliminated to ensure safe operation. Figure 1 below is the failure modes and effects analysis matrix resulting from using risk binning tools for likelihood of detecting an incident, frequency the incident is expected to occur, and severity of the potential hazard to assess risk.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **System** | **Safety Vulnerability** | **D** | **F** | **S** | **Risk** | **Mitigation Strategy** | **Failure Strategy** |
| Oxygen | Leak | 3 | 3 | 5 | Low | Set oxygen pressure regulator to 200 psi. Pressurize gas lines and snoop before experiments. Monitor gas inlet pressure to Fideris oxygen fluid delivery module with pressure switch. Monitor performance and temperature of cell to detect leaks within assembly. | If the inlet gas pressure drops below 200 psi, the cell temperature spikes, or cell performance is lost the oxygen flow will be stopped and the line will be purged with Argon. The system will be put into the parked state with diluted hydrogen at the anode and nitrogen at the cathode. System operators will be notified through the experiment monitoring program. |
| Hydrogen | Leak | 3 | 3 | 5 | Low | Set hydrogen pressure regulator to 200 psi. Pressurize gas lines and snoop before experiments. Monitor gas inlet pressure to Fideris hydrogen fluid delivery module with pressure switch. Monitor performance and temperature of cell to detect leaks within assembly. Set hydrogen detector to 5% of the lower flammable limit in air. | If the inlet gas pressure drops below 200 psi, the cell temperature spikes, or cell performance is lost the oxygen flow will be stopped and the line will be purged with Argon. Oxygen and hydrogen flows will be stopped. System operators will be notified through the experiment monitoring program. |
| DAQ/ monitoring | Electrical fire or break | 3 | 1 | 1 | Routine | All wires will be insulated. All handling of electrical wires will be made in a de-energized state. Room fire detector will detect the presence of smoke. | The experiment will be shut down. |
| Furnace | Combustion of furnace metal due to runaway heating | 1 | 1 | 5 | Routine | The cell temperature is monitored and furnace will never be operated above 1000 C.  | If a spike in temperature occurs the oxygen and hydrogen lines will be purged with Argon and any damage will be assessed. |
| Handling insulation material | 1 | 1 | 1 | Routine | Wearing safety glasses, nitrile gloves, lab coats, and face masks. | Seek appropriate medical attention. |
| Compressed Gas Cylinder | transport | 1 | 3 | 1 | Routine | Use material handling devices whenever possible, do not lift more than 50 lbs. get help to move heavy cylinders, do not bend and twist while lifting. Use the upright spin roll to move cylinders by hand where necessary. Secure cylinders to transport device ensure valve cap is in place. Use a transport device or vehicle suitable to transport cylinders. | Seek appropriate medical attention. |
| Combustion during connection and opening/ closing | 3 | 1 | 7 | Low | Do not use oil on threads, use correct gages, stand to side and open valve slowly | Follow emergency procedures. |
| Combustion during storage | 3 | 1 | 7 | Low | Store in designated storage areas, secure cylinders to prevent falling over, protect from direct sunlight, and segregate flammable gases and oxidizers with a fire barrier for by a distance of 20 feet. Move cylinders to storage when cylinders are not in use. Store with caps on, assure cylinders are labeled correctly and have a full or empty tag, Assure storage area is properly posted including the hazard information for the cylinders. | Follow emergency procedures. |
| Test Stand | Unattended experiment | 5 | 3 | 5 | Low | Experiment will be monitored remotely. Experiment monitoring system will notify laboratory personnel and shut down the experiment if gas flow, temperature, or cell performance deviations occur. | Laboratory personnel will communicate to assess the experiment condition. |
| Safety equipment calibration |  |  |  | An equipment calibration strategy needs to be developed. |

**Figure 1:** Project Failure Modes and Effects Analysis Matrix

## Oxygen System’s Safety

The pressurized POCET apparatus utilizes pressured high purity oxygen gas which will be supplied commercially in rupture proof gas bottles pressurized to ~2200 psig. Under the pressurized experiments, the planned operating POCET oxygen pressure will be 150 psi. Oxygen system safety for this project is guided by the standards developed by the American Society for Testing and Materials, and the National Aeronautics and Space Administration 1 2. Further oxygen systems safety is guided by the report developed by the Idaho National Lab for their high temperature electrolysis project 3.

High pressure oxygen at elevated pressures poses the following issues:

1. Increased flammability of combustible materials that come in contact with hot pressured oxygen gas causing fires.
2. Thermal runaway inside the POCET assembly due to direct combustion with hydrogen/fuel gas causing furnace temperature overheating and possible explosion.
3. Enriching the environment with oxygen in excess of 23.5% by volume potentially increasing flammability of the surroundings due to gas line leaks.

The oxygen gas issues have been mitigated using the following methods:

1. All oxygen gas supply lines have oil free connections as well as an oil free regulator to prevent a fire and all connections have been tested to be leak tight. The stainless steel piping had been scrubbed prior to installation to remove internal contamination. Inconel metal was found to not ignite near its melting point at oxygen pressures near 500 psi 4. The auto ignition temperature of bulk nickel alloys is 1149.85-1369.85$°C$ in oxygen at pressures greater than 200 psi and the minimum burn pressure of Inconel 625 is 1000psi in oxygen based on the promoted ignition of metals test 2. Type 410 and 430 stainless steels were found to ignite at temperatures between 1300-1360 $°C$ in oxygen pressures up to 300 psi 4. The ignition temperature of bulk stainless steel 430 is 1348.85-1365.85$°C$ in oxygen at pressures from 14.7-103 psi and the minimum burn pressure of 430 stainless steel is 500psi in oxygen based on the promoted ignition of metals test 2. Thus, the metals in the POCET were chosen to be below the auto ignition range during nominal operations.
2. Oxygen combined with fuel gasses such as hydrogen will increase flammability, lowering the activation energy (temperature or electric charge) required to initiate an exothermic reaction. The 1,000oC POCET operating temperature is sufficient to auto ignite the fuel and oxygen gas supplied to the fuel cell if the gasses are not maintained separate. Should oxygen and hydrogen interact directly, based upon the maximum design hydrogen delivery rate (5 slpm or 83.33 mL/s) and water’s heat of formation, a direct interaction may release 839 joules per second. Sufficient energy for heating, but not a contributor to catastrophic failure in an open (exhausted) system. In addition, in the event of temperature increase above the operating temperature or a significant loss of power, the computer controls will stop the POCET operation and purge the oxide cell area with argon gas to prevent/exhaust any direct combustion which can be performed in under 1 minute at 10 slpm.
3. Oxygen enriched atmospheres are defined as those containing more than 23.5% oxygen by volume. The worst case leak rate of oxygen into the laboratory is 1 slpm per cell under test. With a single cell it would take about 3 days and 18 hours for the room to reach the enriched oxygen limit of 23.5%. This assumes 209.5 $m^{3}$ room with initial atmospheric oxygen levels of 20.9% and no fresh air brought into the room. Oxygen leaks upstream of the gas delivery module are detected using the pressure switch on the fluid delivery module. If the inlet gas pressure drops below 200 psi, the cell temperature spikes or cell performance is lost the oxygen flow will be stopped and the line will be purged with Argon, then the system will be put into the parked state with .4 slpm nitrogen and .1 slpm hydrogen at the anode and .5 slpm nitrogen at the cathode. Currently there is no leak detection downstream of the cell.

Based on the safety features implemented, the oxygen system poses little risk to the environment and lab personnel. It could cause some internal damage to the metal structure in the event of a faculty cell assembly, but computer controls have been implemented to prevent catastrophic damage to the metal assembly.

## Hydrogen System’s Safety

The pressurized POCET apparatus utilizes pressured high purity hydrogen gas which will be supplied commercially in rupture proof gas bottles pressurized to ~1800 - 2200 psig. Under the pressurized experiments, the planned operating POCET hydrogen pressure will be 150 psi.

High pressure hydrogen at elevated pressures poses the following issues:

1. Hydrogen can combust when in the presence of oxygen and an explosion is possible when pressurized.
2. Thermal runaway inside the POCET assembly due to direct combustion with oxygen gas causing furnace temperature overheating and possible explosion.
3. Hydrogen leaking to the environment increasing flammability of the surrounding air.

The hydrogen gas issues have been mitigated using the following methods:

1. Hydrogen gas mixed with air can create an explosive hazard. This potential hazard will be mitigated by confining the hydrogen gas within a fully sealed flow system. The tubing and components through which hydrogen will flow will be fully leak-checked before operation. Hydrogen gas sensors were installed in the laboratory to detect hydrogen gas leaks.
2. Oxygen combined with fuel gasses such as hydrogen will increase flammability, lowering the activation energy (temperature or electric charge) required to initiate an exothermic reaction. The 1,000oC POCET operating temperature is sufficient to auto ignite the fuel and oxygen gas supplied to the fuel cell if the gasses are not maintained separate. Should oxygen and hydrogen interact directly, based upon the maximum design hydrogen delivery rate (5 slpm or 83.33 mL/s) and water’s heat of formation, a direct interaction may release 839 joules per second. Sufficient energy for heating, but not a contributor to catastrophic failure in an open (exhausted) system. In addition, in the event of temperature increase above the operating temperature or a significant loss of power, the computer controls with stop the POCET operation and purge the cell area with Argon gas to prevent/exhaust any direct combustion which can be performed in under 1 minute at 10 slpm.
3. The worst case leak rate for a single cell test is 1 slpm. It would take 5 days and 19 hours for the room to reach the lower flammable limit of hydrogen in air, 4%. This assumes 209.5 $m^{3}$ room with no fresh air brought into the room.

Based on the safety features implemented, the hydrogen system poses little risk to the environment and lab personnel. It could cause some internal damage to the metal structure in the event of a faculty cell assembly, but computer controls have been implemented to prevent catastrophic damage to the metal assembly.

## Nitrogen and Argon System’s Safety

The pressurized POCET apparatus utilizes pressured high purity nitrogen and argon gas which will be supplied commercially in rupture proof gas bottles pressurized to ~1800 - 2200 psig.

High pressure argon and nitrogen gas at elevated pressures poses the following issues:

1. Oxygen displacement in the event of a leak from the bottle, gas lines, or POCET assembly leading to possible asphyxiation.

The argon and nitrogen issues have been mitigated using the following methods:

1. With a single bottle of gas, a gas line leak would take 3 days to empty the bottle. In addition, the lab and surrounding rooms are ventilated with fume hoods and attached to the environment turning over the entire air volume several times per hour..

## Gas Delivery Summary

Supply gasses are secured upright and stored in a ventilated enclosure, each gas is attached to a gas appropriate regulator (fuel gas vs oxidizing gas) set to the supply side system operating pressure (200 psig). Regulators are in turn attached to 316 stainless steel tubing and Swagelok fittings. The oxygen delivery system tubing and fittings are designated cleaned for oxygen use. Directional control valves are installed in-line after the gas leaves the regulator to prevent backflow. The gas delivery system’s construction is appropriate for operating pressures and temperatures as documented. Gas is delivered to gas specific control modules further regulating the pressure (150 psig) with their own secondary regulator and the quantity of gas supplied (5 to 10 slpm or less for hydrogen and oxygen respectively) to the POCET. Fuel and oxidizing gas are maintained separate in the fuel cell assembly via thermiculite gaskets, keraglass tape, and balanced gas pressure, interacting only electrochemically via the solid oxide fuel cell.

## Exhaust Gas System’s Safety (CO, CO2)

The pressurized POCET apparatus produces CO or CO2 gases which will mixed with the unreacted fuel and may be under pressure up to 150 psig.

High pressure CO or CO2 at elevated pressures poses the following issues:

1. Asphyxiation from gas build up due to leak that displaces oxygen.
2. Carbon Monoxide combustion to Carbon dioxide.

The CO or CO2 issues have been mitigated using the following methods:

1. The exhaust gas pipes are piped to the laboratory exhaust vent which lowers the concentration by 30 to 50x such that they will not accumulate. If there is a leak, the exhaust pipes are directly beneath the laboratory exhaust and remove the leaking gases to prevent build up as well. CO and CO2 gas monitors were installed in the laboratory to detect for leaks.
2. Carbon monoxide is flammable from 12.5% to 74% in air by volume. The auto ignition temperature of carbon monoxide in air is 609oC which is why it is piped separating from the leftover oxygen until it reaches the laboratory exhaust when the gases have cooled below the auto ignition temperature. Carbon dioxide is not flammable in air or oxygen, but can form a weak acid when dissolved in water.

## Exhaust Gas Summary

After heating within the POCET to support electrochemical interaction across the fuel cell, exhaust gasses are cooled by heat dissipation to incoming supply gas (routed via internal concentric tubing to the exhaust gas), and the atmosphere. Typically, the dissipative cooling is adequate to reduce exhaust gas temperatures below 100oC; however, exhaust gasses also pass separately through water cooling, which may be employed to lower exhaust gas temperatures below 100oC, as required by the exhaust gas handling module’s specifications. Exhaust tubing maintains a high to low elevation gradient from the fuel cell discharge to the exhaust handling module to a drip leg, where condensed water may leave the system before the fuel and oxidizing gas are discharged to the building’s hazardous exhaust. A blower is installed upstream of the exhaust gas streams comingling, to enhance dilution (30 to 50 x dilution with room air).

## Fuel Cell Sealing Assembly Safety

The solid oxide fuel assembly is comprised of a commercial ceramic anode, cathode, and electrolyte bonded together. This is then bonded to a stainless steel plate with keraglass tape to separate the fuel and oxygen gases. The steel plate and fuel cell is sandwiched between two other stainless plates to collect the current as well as provide a airway to flow the gases over the anode and cathode. This sealing is performed by compressing thermiculite gaskets to 10 MPa at room temperature.

The fuel cell assembly apparatus poses the following safety issues:

1. The keraglass could fail or not seal or the fuel cell develops a crack during standard operation causing the fuel and oxygen gases come into direct contact at high temperature and pressures resulting in direct combustion and increased furnace temperatures.
2. The thermicullite gaskets could fail or not seal causing oxygen and/or fuel gases to leave the fuel cell assembly which could result in direct combustion or furnace degradation.
3. Increased pressure from a leak could cause one of the rupture disks to break releasing gases into the lab.

The fuel cell assembly apparatus issues have been mitigated in the following ways:

1. In the event of the keraglass seal failing or the fuel cell breaking, the fuel and oxygen gases would come into direct contact resulting in loss of electrical power generation and increased furnace temperatures. In the even this occurs the computer controls have been established to crash the experiment and flush argon through both the fuel and oxygen lines to stop the direct combustion and prevent furnace damage. This can happen in under a minute at 10 slpm of argon gas which is fast enough to prevent furnace heating to 1300⁰C.
2. In the event of the thermicullite seals failing, fuel gas and/or oxygen gas at elevated temperatures and pressures would leak into the furnace chamber which could result in direct combustion or furnace damage. Thus argon/nitrogen gas is purged at 150 psig around the fuel cell assembly to prevent the oxygen/fuel gases from accumulating to cause direct combustion and furnace damage. In addition, in the event of lost electrical power generation, the computer program is designed to turn off and purge the oxygen and fuel lines with argon gas to prevent/exhaust direct combustion.
3. The increased pressure (resulting from increased temperature) within the POCET, may rupture the 165 psig rupture disk, potentially releasing combusting fuel and oxidizing gas to the building’s hazardous exhaust system. Upon purging, the maximum quantity of fuel gas occupying the supply tubing is 0.285 L, or 0.02 grams hydrogen. Assuming hydrogen’s lower heating value (water will be converted to vapor only), the remaining fuel will contribute 2.8 kJ (669 cal) energy to the hazardous exhaust system, insufficient energy to initiate a building material fire, particularly if this energy is dispersed. Hazardous exhaust ductwork is steel and not subject to combustion at low energies. Acoustic ceiling tiles (Hytone ™ Fissuretone II) are constructed from wet formed mineral fiber and also not subject to combustion at low energies.

## Fideris Equipment Rack

The Fideris equipment stand comprises of multiple gas flow controllers, temperature controllers, pressure controller, liquid delivery system (pressurized) and a power load bank connected the experiment controlling computer. The components of the stand and its accompanying software was provided by Fideris Tessol.

The Fideris equipment rack poses the following safety issues”

1. One or more of the gas delivery flow controllers could fail preventing the nominal operation of the POCET.
2. The furnace temperature controller could fail preventing the furnace from heating and lose of experimental run.
3. The 1kW load bank could fail resulting in up to 1kW of electrical energy being routed to the rest of the POCET equipment connected to the load bank leading to dangerous work conditions.
4. The liquid delivery controller could fail preventing the humidity from being added to the gas line.

The Fideris equipment rack issues have been mitigated in the following ways:

1. If one of the gas lines read a pressure below the experimental conditions, the program will stop and purge the oxygen and fuel lines with argon gas. Low pressure lines do not constitute a safety issue for the POCET, but may damage the fuel cell which may need to be replaced.
2. If the furnace controller fails, no power will be supplied to the furnace elements and the furnace will cool to room temperature without control at a maximum rate of 100 ⁰C per hour. The furnace cooling faster than 2 ⁰C per min could cause the fuel to break and need to be replaced prior to another experiment, but the metal assembly and furnace can handle this drastic temperature change without ill effects.
3. Due to high power loads up to 1000W of electrical energy being generated all components of the POCET stand including the furnace gas delivery, gas exhaust, and all Fideris equipment modules have been grounded to the laboratory’s building earth ground to prevent the buildup of electricity on the components which could be transferred to a researcher during an experiment.
4. If the liquid controller fails, no water vapor will be injected into the vapor phase during an electrolyzer operation, then no output power would be generated. As such, the computer software would then turn off the experiment and cool the furnace while purging the gas lines with argon gas.

## Furnace Safety

A 6kW furnace is placed inside the POCET stand to generate the 1000+ ⁰C temperatures needed for the fuel cell to generate measureable quantities of electrical power. The Engineering Teaching and Research Laboratory (ETRL) building is equipped with fire sprinklers. In the event of a fire, the fusible link melts/distorts actuating the sprinkler. Only those sprinklers with fusible links affected by the fire will actuate. Similarly, additional sprinklers will engage if the fire spreads, until sufficient water is applied to extinguish the fire.

The furnace poses the following safety issues:

1. One of the heating elements could fail at elevated temperatures.

The furnace issues have been mitigated in the following ways:

1. In the event of the furnace losing power or the ability to maintain the experimental temperature, the furnace will cool to room temperature, faster than programmed, but not faster than manufactured. Thus the furnace cooling unexpectedly poses no risk to the furnace or the metal fuel assembly. However, the brittle fuel cell may crack under the faster cooling, but can be replaced prior to another experiment.

## Laboratory Power Safety

The POCET stand requires external laboratory building power to operate and in the event of an unscheduled power out could damage some of the equipment.

The furnace is not supplied with an uninterrupted backup supply (UPS) so it will cool when power is lost, but will not damage the furnace as it was intended to cool without supplied power. The fideris equipment stand and modules are supplied with an UPS power that has sufficient power to operate for a minimum of 6 hours which is sufficient to safety stop the experiment and gas delivery and allow the apparatus to cool down. The exhaust blower is also supplied with an UPS so any leaking gases will not accumulate inside the lab during a power outage.

1. Description of the POCET Equipment

The following are descriptions of the various components in the POCET.

## Fideris Equipment Rack

All equipment contained in this rack controls and monitors temperature and flow conditions of the experiment, which the user controls through the Fideris Test Suite Software. A brief description of each component and its location is listed below.

At the top of the rack are two auxiliary power units (APUs). The power cord from the wall is first connected to the APUs, and then from the APUs to the rest of the equipment in the rack. In case of power outage, the APUs will continue to supply power for up to 40 minutes, allowing the user to properly shut down all equipment.

## Delivery Module with Purge (Hydrogen)

This unit is located directly below the APUs, labeled H2. This module controls Hydrogen flow rates using Bronkhorst EL-FLOW mass flow controllers. If the system loses power or if the user disables the flow using either the computer interface or Emergency Stop, this module has a fail-safe mechanism that will purge all hydrogen lines with inert gas. All Delivery Modules will automatically stop the flow of gasses into the unit in the event of a power outage

## Delivery Module (CO/CO2)

Second from the top is another gas Delivery Module that controls mixing of gasses. It will blend CO and C02 at a maximum flow rate of 0.1 slpm (standard liters per minute.) It is currently not in use, but will possibly be used in later phases of the experiment. When this happens, the SOP will be updated.

## Delivery Module with Purge (Oxygen)

Third from the top is the Oxygen flow controller, labeled O2. It is identical in operation to the Hydrogen Delivery Module, but it has been upgraded to accommodate flow rates up to 10 slpm. It may also be used with standard air, but a calibration constant would need be inserted in the Fideris Test Suite software.

## Delivery Module with Purge (Nitrogen)

Fourth from the top is the Nitrogen Delivery Module. The normal operation provides up to 10 slpm, but it has been upgraded to accommodate gas flow rates of up to 135 slpm if the user chooses to upgrade the mass flow controller at a later time.

## Emergency Stop and Power Supply

Fifth from the top is the Emergency Stop/Power Supply. This must be turned on before the rest of the unit can be powered on. Pushing the red stop button on the right hand side will shut down the operation and all gas flows.

## 1kW Electric Load Bank

Sixth from the top is the Electronic Load Bank. This module monitors the electric loads generated by the fuel cell. It has 10 voltage inputs for monitoring cell voltage. It can monitor up to 55V and 200A. It also monitors stack voltage using the large brass positive and large gray negative terminals on the back, and is equipped with a 250A current shunt. Voltage accuracy is +/- 0.1%. Current accuracy is +/- 0.15%. High temperature voltage and power cables that connect the unit have been assembled by us.

## Temperature Controller

Seventh from the top is the 6kW temperature controller. It powers and controls the temperature of the furnace and protects it from overheating. In addition, it displays temperature readings of thermocouples placed inside of the furnace, the Humidifier Assembly (see 5.3.2) and includes four temperature monitoring channels for the thermocouples placed by the user.

## Pressure Control Module

Eighth from the top is the Pressure Control Module. It uses inert gas to pressurize the furnace up to 150 psi. It contains a mechanical failsafe mechanism to avoid going over the set pressure. Set point, pressure and status are displayed on the front panel of the module.

##  Liquid Delivery Module

Ninth from the top is the Liquid Delivery Module that supplies gas to the Humidifier Assembly (see 5.3.2). It contains a mass flow controller to provide a maximum flow of 10 grams per minute. For best results, the liquid should be degassed and pressurized to at least 25 psi by the Liquid Pressure Delivery System (see 5.3.1j).

##  Liquid Pressure Delivery System

Tenth from the top is the Liquid Pressure Delivery System. It degasses, pressurizes and supplies a controlled flow rate of deionized water to the Liquid Delivery Module (see item 5.3.1i), and also pressurizes the water stream as specified by the operator up to a maximum of 185 psi.

##  Exhaust Gas Module Controller

Eleventh from the top is the Exhaust Gas Module Controller. After exhaust gasses from the furnace have been cooled through the Heat Exchanger (see item 5.3.3), the Exhaust Gas Module Controller provides necessary backpressure for the system to properly operate and also monitors the pressure difference between components.

##  Exhaust Gas Module

The bottom module houses the exhaust gas pipe system, which routes all exhaust gasses to the ventilation system. It can handle fully saturated wet gas flows of 47 slpm of fuel and 316 slpm of oxidant at temperatures up to 100C. All gas lines are equipped with burst discs that will rupture if the pressure exceeds 165 psi. Exhaust will be a combination of hot gas and liquid water.

##  Humidifier Assembly

The Humidifier Assembly is located directly below the furnace on the base of the red furnace stand. It provides 1kW of power to boil deionized water from the Liquid Pressure Delivery System, and delivers steam to the furnace at temperatures up to 150C.

##  Heat Exchanger

The Heat Exchanger is the aluminum box located on the back of the red furnace stand. It cools exhaust gasses by flowing cold water around the outside of the exhaust pipes. The water is supplied by the recirculated building water inlet and outlet located along the wall behind the Heat Exchanger. For proper operation, both the inlet and outlet of the recirculated water must be turned on. If the return valve is not open during operation, the Heat Exchanger will not work properly and will become over-pressurized. A pressure switch has been installed so that in the event of excess pressure, water will be redirected to the drain located in the fume hood. A transparent lid has been installed in the top of the heat exchanger so that the user can visually verify proper operation before running the experiment.

##  Furnace

The Furnace houses the Solid Oxide Fuel Cell assembly. It is pressurized to 150 psi during normal operation and the inside temperature can reach up to 1000C. It uses a 6 kW heater powered by a 250V 20A power source and is controlled by the Temperature Controller (see 5.3.1g).

##  Gantry

The Gantry is stationed around the Furnace. It is used to remove the lid for access to components housed inside. A chain is connected to the eye bolt in the center of the lid, and the pulley system is used to move the lid up, down or to the side. The lid weighs 800lb, so caution must be used when moving it. When setting the lid down, the load must be evenly distributed to avoid tipping. Failure to do so can result in personal injury and/or damage to the equipment.

##  Gas Regulators

Gas regulators are located in the gas closets, 310A and 310B. Each regulator is labeled for the type of gas it controls. The arrow on the gas regulator must be pointed toward the bottle that is to be used. When the amount of gas in a cylinder falls to its useful lower limit, open the second gas cylinder turn the regulator switch toward the full bottle, allowing for continuous operation while the empty bottle is replaced.

General gas cylinder safety posters are located inside of the gas closets.

1. Standard Operating Procedures

The following are standard operating procedures (SOPs) for the various proposed experiments and setup, assembly, running and shutdown tests needed to operate the POCET in a safe manner.

## Assembly and Disassembly Procedure

1. **Assembly Procedures**
	1. Disconnect anode heat exchanger gas lines so that it can be moved.
	2. Clean with isopropyl alcohol
		1. Anode/cathode flow plates
		2. Oxidized tray
		3. Anode plate spacer
		4. Heat exchangers
	3. Using a tray as a stencil cut a piece of Ni mesh
	4. Stamp the mesh using the press. Press all sides of the stamp.
	5. Spot weld the mesh to the anode plate around the perimeter and in the center.
	6. Layout all assembly components in the assembly order for Elcogen cell with available thermiculite on 10/9/18:
		1. 1 0.8 mm and 1 0.5mm thermiculte seal for bottom heat exchanger to cathode plate
		2. .5mm small square of thermiculite for middle of gasket
		3. Cathode flow plate
		4. Cathode spacer
		5. Cathode thermiculite seal(s)
		6. Tray
		7. Anode thermiculite seal(s)
		8. Glass seal
		9. Fuel cell
		10. Anode spacer
		11. Anode flow plate with spot welded mesh
		12. 1 0.8 mm and 1 0.5mm thermiculite seal for anode flow plate to top heat exchanger
		13. .5mm small square of thermiculite for middle of gasket
		14. Mica paper for insulating current collector tabs from heat exchangers
	7. Take a picture of the layout for documentation
	8. Orient thermiculite seals in alignment with gas flows
	9. Stamp alignment pin holes into thermiculite seals using tray as a stencil
	10. Insert alignment pins into bottom heat exchanger
	11. Paint a coat of LSM on cathode ridges using sponge applicator.
	12. One by one stack each component in the correct orientation
		1. Place two chromel C wires in contact with the anode and cathode. Attach one wire from each side to the missile wire routing through the furnace jet seal. Route the other two wires through the bottom of the furnace.
	13. Set load distribution plate spacer on top of stack
	14. Set load distribution plate on top of stack
	15. Place bolts on 4 corners of assembly. Do not overtighten, bolts are for alignment purposes only.
	16. Remove alignment pins
	17. Attach anode and cathode heat exchanger gas lines
	18. Place top and bottom ceramic electrical terminal insulators into furnace terminal ports. Hand tighten nuts securing the ceramic insulators into the ports.
	19. Place (+)/(-) copper terminals and bolt to the top of anode and cathode current collecting tabs
	20. Place mica paper between the heat exchangers and current collecting tabs to protect against electrical shorts
	21. Secure terminal cables to bottom of copper terminals
	22. Secure terminal cables to furnace using zip ties. The copper terminals may deform at high temperature with the weight of the cables.
	23. Check furnace for short-circuits or grounding
		1. Checking for short circuits the black lead must touch the anode and the red lead must contact the cathode
	24. Measure resistance between (+)/(-) terminals.
	25. Set ram socket plate on top of distribution plate and coat socket with grease. Ensure that ram socket plate is centered.
	26. Set thermocouples in place
	27. Take a picture of the assembly
	28. Attach furnace lifting bar through hydraulic press support brackets. Center gantry hook directly above the center of the furnace, secure in place using shaft collars. Tighten shaft collars to the lifting bar using ¼” hex wrench.
	29. Before lifting, ensure that the base of the gantry frame is contacting both the Fideris module rack and the cabinets below the fume hood.
	30. Carefully lower lid onto furnace using guide rods
	31. Attach furnace lid and base using four bolts evenly spaced around the furnace base
	32. Snug all blots using 45mm wrench
	33. Remove lifting bar
	34. Clip hoist chain to hydraulic press assembly using the lifting pin
	35. Secure lifting pin to assembly using cotter pins
	36. Move the press as close as possible to the lifting pin to minimize tipping
	37. Use the gantry to lift and move hydraulic press assembly to furnace
	38. Lower press onto top of furnace and align with support brackets
	39. Place pins through rail to lock press into place
	40. Remove hoist and lifting pin from hydraulic press assembly
	41. Clean ram with isopropyl alcohol
	42. Carefully and slowly place ram into furnace
	43. Ensure that the press is directly centered over the ram
	44. Manually pump the hydraulic press until it makes light contact with ram
	45. Slowly Hand pump the hydraulic press until it reaches 10 metric tons.
	46. Slowly depressurize until gage reads 5 metric tons
	47. Attach furnace gas purge lines
	48. Attach furnace wall thermocouple.
	49. Connect the chromel C wires to the cDAQ module
2. **Disassembly Procedures**
	1. Wait for furnace to cool to room temperature
	2. Disassembly is reverse of assembly

Take a picture of the assembly and all relevant components during disassembly

## Start-up, Idle, Park, and Shut-down Procedure

1. **Experiment Startup Procedures**
	1. Check that Hydrogen Detector is active
	2. Check that condensation pan is empty
	3. Check that condensate trap has been pre-filled
	4. Check that De-ionized water reservoir is full
	5. Check furnace for short-circuits or grounding
		1. Checking for short circuits the black lead must touch the anode and the red lead must contact the cathode
	6. Turn on gas exhaust fan
	7. Un-enable air flow and set oxygen and nitrogen flow to 0
	8. Turn Valve handles to Bypass configuration for both anode and cathode
	9. Ensure that individual gasses are NOT enabled in gas handling instruments
	10. In the fuel gas closet, open hydrogen gas bottles.
	11. In load control instrument set load control to current control. Set current to 0.
	12. Enable load control instrument
	13. **Anode reduction:** Based on process in Dillig, M. Thermal Management of Solid Oxide Cell Systems with Integrated Planar Heat Pipes. Modification of the F5 flow has been made.
		1. In the ExperimentGUI monitor under Testing select Flow Control and right click.
		2. Master enable the gas flows using the check box. Following the schedule below set flow rates in the set point box for the specific gas. If gas flows are ramping correctly turn the gas circuit valve handles to Normal configuration for both anode and cathode simultaneously and slowly. Gas flows are set to ramp at .1 slpm/s. Record OCV on experiment plan after each step.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Step | Time | H2 | N2\_A | Air |
| Reduction 1 | 60 | .1 | 2.5 | 1 |
| Reduction 2 | 30 | .1 | 1.5 | 1 |
| Reduction 3 | 30 | .2 | 1 | 1 |
| Conditioning | 30 | .5 | .5 | 1 |

* + 1. If voltage is less than 0.9 V check for electrical shorts, grounding, and gas leaks.
	1. **Cell warm-up**
		1. Step flows to baseline condition. 20% fuel and oxidant utilization for a max current of 20 A with base operating temperature and atmospheric pressure. The total anode flow rate is set to match the cathode with N2. In the Main\_Control monitor set the flow rates:

|  |  |
| --- | --- |
| Anode (slpm) | Cathode (slpm) |
| H2 | N2 | H2O | O2 | N2 |
| 0.7 | 0.7 | 0 | 0.4 | 1.5 |

* + 1. Under the load control instrument, set minimums for current and power to zero, and voltage to 0.6V. Set maximums to 25, 50, and 1.4, respectively.
		2. Select potential control mode. Set operating voltage set point to .85V. Hold constant for 30 minutes.
		3. Verify that performance has not degraded. Record OCV.
1. **Cell Idle Procedures**
	1. Using the Flow Control monitor lower gas flow rates in increments of .1 slpm to the flows in the table below.

|  |  |
| --- | --- |
| Anode (slpm) | Cathode (slpm) |
| H2 | N2 | H2O | O2 | N2 |
| .1 | .4 | 0 | .1 | .4 |

* 1. Leave chamber temperature at base operating temperature
		1. Record time, OCV, and flow rates on experiment plan
	2. Let Idle until ready to continue testing
	3. Verify that performance has not degraded
		1. Record time, OCV, and flow rates on experiment plan
1. **Cell Park Procedures**
	1. Using the Flow Control monitor lower gas flow rates in increments of .1 slpm to the flows in the table below.

|  |  |
| --- | --- |
| Anode (slpm) | Cathode (slpm) |
| H2 | N2 | H2O | O2 | N2 |
| .1 | .2 | 0 | 0 | 0 |

* 1. Leave chamber temperature at base operating temperature
		1. Record time, OCV, and flow rates on experiment plan
	2. Leave parked until ready to continue testing
	3. Verify that performance has not degraded
		1. Record time, OCV, and flow rates on experiment plan
1. **Shut Down Procedures**
	1. Record time and OCV on experiment plan
	2. Turn Valve handles to Bypass configuration for both anode and cathode.
	3. Set target flow rates for all enabled flows to 0
	4. Once flow rates have reached 0 un-enable all flows
	5. Close bottles of: N2, O2, Ar, and H2
	6. Turn off exhaust fan
	7. In the Temperature Monitor set target temperature to 20

Last Update 10/9/18 RH

## Furnace Warm-up and Glass Seal Sintering Procedure

1. **Furnace Warm-up and Glass Seal Sintering Procedures**
	1. Turn on Module rack
	2. Flip up furnace heating breaker
	3. Open FC Power software once all modules are powered on
	4. Place SOFC gas circuit into “Bypass” mode
	5. Master enable GasStick instrument
	6. Open Argon, Nitrogen, and Oxygen bottles, ensure that delivery pressure is at 200 psi.
	7. Turn manual purge control to auto on gas modules
	8. Place cathode gas circuit into “Normal” mode
	9. Ensure the over-temperature sensor is not displaying any failure messages and reads “SAFE” in green letters below the temperature display
	10. Enable
		1. Monitors
			1. ExperimentGUI
		2. Log Sessions
			1. MasterLog1
	11. In Experiment GUI monitor under operations bar select sinter and right click.
		1. Determine operating temperature for the cell. Enter this into dialog box and record on experiment sheet
		2. Sintering\_Kerafol.vbs will now ramp the furnace temperature at 2 C/min while controlling off the top heat exchanger temperature to 500 C. Dwell for 2 hours. Then ramp to 840 C, dwell for 10 hours, then cool to the operating temperature. Simulated air will flow at 1 slpm through this process.

## Pre and Post Experiment Procedures

1. **Experiment Pre-check**
	1. Using the Gas Calculator, determine whether enough gas is present to last until the next gas delivery. Fill out the amount present and estimate the amount of gas needed to run the experiment and when bottles will need to be replaced on the experiment plan sheet.
	2. Fill out and date gas Inventory sheet in gas closet
	3. Make sure that oxidized trays are available
	4. Ensure all gas bottles are free of defects, properly connected, and electrically grounded when appropriate
	5. Determine Anode and Cathode thermiculite seal thicknesses for the cell and interconnects.
		1. Determine space between anode and cathode plate to the sealing tray accounting for:
			1. Anode/cathode plate and flow channel height
			2. Anode/cathode current collector and spacer height
			3. Fuel cell thickness
			4. Glass seal thickness
			5. Tray thickness
		2. Record these on the experiment plan sheet
2. **Experiment Post-check**
	1. All gas cylinders have been closed
	2. All gas flows have been disabled on software before shut down
	3. All gas and liquid supply streams have been shut down
	4. Final pressures on all gas cylinders have been recorded
	5. Cylinders with less than 30 minutes of run time left have been reordered
	6. Water supply to exhaust gas chiller has been shut off
	7. Store experiment data
		1. Create a folder to store data. Ex.: 18.5.30\_RIST SOFC Experiment
			1. Save folder to the Cesilab network device SOFC Test Stand>Test Stand Project Management>Experiments
		2. Save Masterlog1 log session as .xlsx file
			1. Name Ex: MasterLog\_5.30.18
		3. Save VI log session as .xlsx file
			1. Name Ex: VI\_5.30.18
		4. Scan and save Experiment Plan
			1. Name Ex: Experiment Plan\_5.30.18
		5. Save all current operating procedures to file
		6. Save all photos taken during experiment and other relevant information
	8. Write up technical memorandum or full experiment report
		1. Post to slack for comments
		2. Save file to experiment folder. Name Ex: Technical Memorandum\_5.30.18
	9. Store cell and any other relevant material in a plastic bag with date

Last update 5/30/18 RH

## Baseline Pressureless Test Procedure

1. **Determine scan rate for steady state measurements**

Description: Galvanostatic fixed flow rate condition tests. VI\_Monitor script ramps current up to max current, and ramps the current back down in specified increments, and is repeated a specified number of times. The script logs a measurement at the end of a specified time at each set point in log session VI.lgs.

* 1. Open Experiment GUI in monitors tab
	2. Under test select current sweep
	3. Set test parameters
		1. Maximum Current to 20, StepCurrent to .5, DwellTime to 10, and Repeat to 1.
		2. Name Test
	4. Click Start Test
	5. Decrease DwellTime in by 1 second. Click Start Test
	6. Repeat 5 until VI curve shifts upward (steady-state not reached). Return DwellTime in VI script to increment before the VI curve shifts up.
	7. Increase current/load to max power density.
	8. Run an impedance spectroscopy experiment from 50 mHz to 5 MHz
	9. Reduce current/load to 0
1. **Baseline operation characterization**

Description: 50% fuel and oxidant utilization, base operating temperature, atmospheric pressure, for a max current of 43 A.

* 1. Name test
	2. With same test parameters in the current sweep GUI Click Start Test.

|  |  |
| --- | --- |
| Anode (slpm) | Cathode (slpm) |
| H2 | N2 | H2O | O2 | N2 |
| 0.6 | 0.6 | 0 | 0.3 | 1.2 |

After every successful sweep/ data collection we will run an impedance spectroscopy experiment from 100 mHz to 10 MHz with 50 points per decade. The current needs to be set to the maximum power density prior to sweeping. After the sweep set the current back to 0A.

1. **Oxidant partial pressure variation characterization**

Description: Increase cathode flow rate without changing oxygen utilization.

* 1. Measure V-I curve at baseline condition
	2. Increase cathode N2 flow to 1.7 slpm. When voltage reaches steady-state, click Start Test. Changes O2 partial pressure from 20% to 15%.
	3. Decrease cathode N2 flow to .7 slpm. When voltage reaches steady-state, click Start Test. Changes O2 partial pressure from 15% to 30%.
	4. Increase cathode N2 flow to 2.7 slpm. When voltage reaches steady-state, click Start Test. Changes O2 partial pressure from 30% to 10%.
	5. Decrease cathode N2 flow to .2 slpm. When voltage reaches steady-state, click Start Test. Changes O2 partial pressure from 10% to 60%.
	6. Increase cathode N2 flow to 5.7 slpm. When voltage reaches steady-state, click Start Test. Changes O2 partial pressure from 60% to 5%.
	7. Decrease cathode N2 flow to 1.2 slpm to return to baseline conditions. When voltage reaches steady-state, click Start Test.
1. **Fuel partial pressure variation characterization**

Description: Increase anode flow rate without changing H2 utilization by diluting with N2.

* 1. Measure V-I curve at baseline condition
	2. Decrease anode N2 flow to 0.4 slpm. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 50% to 60%.
	3. Decrease anode N2 flow to 0.3 slpm. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 60% to 67%.
	4. Decrease anode N2 flow to 0.2 slpm. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 67% to 75%.
	5. Decrease anode N2 flow to 0.1 slpm. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 75% to 86%.
	6. Decrease anode N2 flow to 0.0 slpm. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 86% to 100%.
	7. Increase anode N2 flow to 0.6 slpm to return to baseline conditions. When voltage reaches steady-state, click Start Test.
1. **Fuel utilization characterization**

Description: Vary nominal fuel utilization from 50%. Observe OCV and voltage stability for any changes. If concentration losses are observed do not increase utilization.

* 1. Measure V-I curve at baseline condition: Click Start Test
	2. Decrease anode H2 flow to 0.5 slpm. When voltage reaches steady-state, click Start Test. Changes H2 nominal utilization from 50% to 60%.
	3. Decrease anode H2 flow to 0.4 slpm. When voltage reaches steady-state, click Start Test. Changes H2 nominal utilization from 60% to 75%.
	4. Decrease anode H2 flow to 0.35 slpm. When voltage reaches steady-state, click Start Test. Changes H2 nominal utilization from 75% to 86%.
	5. Increase anode H2 flow to 0.6 slpm to return to baseline conditions. When voltage reaches steady-state, click Start Test.
1. **Oxidant utilization characterization**

Description: Vary oxidant utilization. Observe OCV and voltage stability for any changes. If concentration losses are observed do not increase utilization.

* 1. Measure V-I curve at baseline condition: Click Start Test
	2. Decrease cathode O2 flow to 0.25 slpm. When voltage reaches steady-state, click Start Test. Changes O2 nominal utilization from 50% to 60%.
	3. Decrease cathode O2 flow to 0.2 slpm. When voltage reaches steady-state, click Start Test. Changes O2 nominal utilization from 60% to 75%.
	4. Decrease cathode O2 flow to 0.18 slpm. When voltage reaches steady-state, click Start Test. Changes O2 nominal utilization from 75% to 83%.
	5. Increase cathode O2 flow to 0.5 slpm. When voltage reaches steady-state, click Start Test. Changes O2 nominal utilization from 83% to 30%.
	6. Decrease cathode O2 flow to 0.3 slpm to return to baseline conditions. When voltage reaches steady-state, click Start Test.
1. **Operating temperature characterization**

Description: At baseline conditions vary temperature from +/- 100 °C in increments of 25 °C. If performance degrades do not increase/decrease temperature further.

* 1. Open Experiment GUI in monitors tab
	2. Under test select temperature sweep
	3. Use the temperature set point in the test parameter section to change the temperature.
	4. Decrease chamber temperature in increments of 25°C at a rate of 2°C/min and run test procedures at each temperature.
	5. Increase chamber temperature in increments of 25 °C at a rate of 2°C/min and run test procedures at each temperature.
	6. Decrease chamber temperature to baseline at a rate of 2°C/min.
	7. Repeat test procedures and verify that performance has not degraded
1. **Fuel humidification characterization**

Description: Increase anode flow rate without changing H2 utilization by diluting with H20.

* 1. Set Cal Omega Humidifier Temp to 200°C
	2. Wait till humidifier heats up
	3. Measure V-I curve at baseline condition: Click Start Test
	4. Decrease anode N2\_A flow to 0.0 slpm. Increase anode H2O flow to 5g/hr. When voltage reaches steady-state, click Start Test. Changes H2 partial pressure from 50% to 85%. And anode humidification to 15%.
	5. Increase anode H2O flow to 10g/hr. When voltage reaches steady-state, click Start Test. Changes anode humidification from 15% to 25%.
	6. Increase anode H2O flow to 20g/hr. When voltage reaches steady-state, click Start Test. Changes anode humidification from 25% to 40%.
	7. Increase anode H2O flow to 30g/hr. When voltage reaches steady-state, click Start Test. Changes anode humidification from 40% to 50%.
	8. Decrease anode H2O flow to 0g/hr. Increase anode N2 flow to 0.6 slpm When voltage reaches steady-state, click Start Test. Returns to nominal
1. Equipment Integrity

The POCET stand was recently conceived and built in the last couple of years and has had only a few proof of concept tests. The rupture disks are new and operational and not in need of replacing. The fideris equipment stand has been inspected in the last couple of years and was operational and in good working order after the visit. The furnace was acquired in the last several years and operated under 2C per min (conservative heating) and is still in good operational condition. Written October 2018.

1. Safety Audit Schedule and Procedures

The POCET stand has many components that need to be inspected on a regular schedule to ensure that the apparatus runs smoothly and operates over its lifetime.

The following items should be inspected daily or prior to each experiments:

1. All laboratory systems should be in working order and observed prior to an experiment including: building electricity, fume hood, exhaust vent, computer software, fideris equipment stand, and furnace.
2. The reusable stainless steel fuel assembly parts should be inspected prior to building a new fuel cell. The should be cleaned, sandblasted, or electroplated as needed.
3. The computer software should be inspected to ensure that it is operational and any previous errors cleared.
4. The fideris equipment stand should be expected to verify all systems are powered and no errors are present.
5. The gas cylinders should be leak tested at the regulator and checked to make sure there is enough gas to perform the desired experiment.

The following items should be inspected once a quarter (3-4 months):

1. Check the furnace internally to see any formation of scales and/or contamination on the inside and remove if needed.
2. Verify all electrical cables to the furnace, fideris equipment stand, and computer are secured and have not loosened with time.
3. Leak test the fuel, oxygen and purge gas lines to verify there are no gas leaks.
4. Have the exhaust hood and fume hood face velocity checked in accordance with its baseline.
5. Download and install any updates to the fideris equipment stand and any Microsoft windows updates.

The following should be inspected once a year:

1. The furnace elements should be inspected for scale and material lose and replaced if needed. Check the overall impedance and report if the number has changed.
2. Inspect the outside of the furnace for oxidation damage or rust to ensure the vessel is rated for the pressurized experiments.
3. Rupture disks should be inspected for wear, holes, or tears and replaced if needed.
4. External Safety Reporting and documentation

## Safety Reporting

If a safety incident or near miss occurs:

1. Call Dr. McLarty if not already informed, call (386) 717-7171
2. When safe, document the timeline of the event with specifics (who, what, when, where, why?) and send the document to Dr. McLarty via e-mail: dustin.mclarty@wsu.edu. Your written document may become part of the record and used to communicate to safety personnel; take your time and do your best to accurately document information.
3. If a lab-wide concern, schedule a meeting for everyone to be debriefed.

Records that are generated and maintained in ETRL 310 include:

* Master Hardcopy Test Plans containing pen-and-ink markups
* Completed checklists for the operational activities performed
* Operation Data Logs
1. Safety Plan Approval

Dustin McLarty with WSU Voiland College of Engineering and Architecture’s (VCEA’s) Clean Energy System’s Integration (CESI) Laboratory contacted WSU Environmental Health and Safety (EHS) to review potential hazards and existing hazard controls before introducing pressurized oxygen to fuel cell testing. Shawn Ringo, EHS assistant director contacted WSU Laboratory Safety Committee members Dr. Jacob Leachman (Hydrogen Properties for Energy Research Laboratory) and Billy Schmuck (Voiland School of Chemical Engineering and Bioengineering) to assist with the review. The CESI laboratory provided the review team drawings depicting the gas delivery and exhaust system and associated controls, a fuel cell cathode, anode and heat exchanger assembly flow diagram, and, an oxygen systems safety review performed by the laboratory. After reviewing the provided information, EHS met with laboratory personnel on-site to discuss controls, procedures and observe the operating systems. The concerns and recommendations on the POCET stand have been taken into consideration and added into this document in the preceding section and implemented into the POCET stand.

1. References

1. Rosales, K. R., Shoffstall, M. S. & Stoltzfus, J. M. Guide for oxygen compatibility assessments on oxygen components and systems. *Eur. Sp. Agency, (Special Publ. ESA SP* (2007).

2. Beeson, H. D., Smith, S. R. & Stewart, W. F. *ASTM Manual 36: Safe Use of Oxygen and Oxygen Systems : Handbook for Design , Operation , and Second Edition*. (2009).

3. Sohal, M. S. & Herring, J. S. Oxygen handling and cooling options in high temperature electrolysis plants. **INL/EXT-08,** (2008).

4. Defense Metals Information Center. *Ignition of Metals in Oxygen*. *Battelle Memorial Institute* (1966).