HYPER SAFETY PLAN

CRYO-CATALYSIS HYDROGEN EXPERIMENT FACILITY (CHEF)

HYPER-SP-CHEF-004

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1. Scope and Contents

This safety plan is prepared for the redesign of the Cryo-catalysis Experiment Facility (CHEF) to allow higher test cycle rates and increased test pressure for the optimization of the Heisenberg Vortex Tube (HVT) under DOE "Optimizing the Heisenberg Vortex Tube for Hydrogen Cooling" project. This document is an extension of the previously approved safety plan by the Department of Energy (DOE) Hydrogen Safety Panel.

The safety plan was developed following the Department of Energy "Safety Planning for Hydrogen Fuel Cell Projects" document available on-line at: http://energy.gov/sites/prod/files/2014/03/f10/safety_guidance.pdf

This document will guide the safe conduct of all work.

Contents

2. ORGANIZATIONAL SAFETY INFORMATION

2.1. STATEMENT OF PURPOSE:

The current goal of this experiment is to perform a bench-top scale test of a catalyzed vortex tube at cryogenic temperatures and with hydrogen. Our main objective is to validate our predictive models for cryogenic hydrogen vortex tube performance. This document ensures the experimental aspect of these objectives is completed with minimized safety concerns.

2.2. DESCRIPTION OF WORK

The bench-top scale test will be performed in the Engineering Teaching/Research Laboratory (ETRL) Building room 221 in the Cryo-catalysis Hydrogen Experiment Facility (CHEF).

The total amount of stored hydrogen between ETRL 219 (ventilated gas room) and 221 will be less than 985 scf and includes safety devices beyond the minimum required by the relevant safety codes listed in the next section.

Operating states of the experiment consist of Work-In-Progress, Liquefaction, Test, Warm-up, Emergency-abort, and Power-loss states:

- Work-In-Progress state: Consists of a lock-out tag-out of hydrogen storage where the experiment is first purged and then opened to safely work on per NFPA 2 code.
- Liquefaction state: Consists of the acquisition of liquid hydrogen via cryocooler operation from ETRL 219 GH2 storage area and is completed via Section 3.2 with redundant safety systems as detailed in Section 5 of this document.
- Test state: Consists of the reproduction of in-field boil-off conditions which is processed through the vortex tube and finally vented to the ETRL 221 dedicated hydrogen vent line which discharges directly to the exterior of the building in accordance to CGA G-5.5 code.
- Warm-up state: Consists of the controlled warm-up of the cryogenically chilled components to room-temperature with hydrogen storage in a lock-out tag-out state following Section 3.4 of this document.
- Emergency abort state: Red button is pushed automatically engaging a dedicated relief of liquefaction volume and isolation of storage.
- Power-loss state: Emergency electrical feedline to ETRL 221 has lost power. Fuel-cell back up power will autonomously maintain hydrogen detection equipment, vacuum systems, actuated valves, and computer system while safing the system through consumption of stored hydrogen.

All hydrogen will be processed with a two-stage pressure relief system (with redundancy) and will be connected to the dedicated ETRL 221 hydrogen vent line at all times per codes listed below.

2.3. WSU Laboratory Safety Policies and Procedures:

All experiments at WSU are advised to have a safety plan within a printed notebook next to the experiment. This notebook is in addition to the laboratory safety and chemical hygiene plan on the laboratory website and HYPERDRIVE. It is recommended that this safety plan be revised whenever extensive modifications occur to the experiment and at least annually.

2.3.1. Washington Code

WSU must adhere to Washington State codes and laws that cover hydrogen technologies. The specific codes concerning the Cryo-catalysis Hydrogen Experiment Facility include WAC 296-24-31505 Liquid Hydrogen Systems, and WAC 296-24-31503 Gaseous Hydrogen Systems. These codes make frequent reference to NFPA 55 Compressed Gases and Cryogenic Fluids which frequently references CGA 5.5-2014 the Hydrogen Vent Systems design code. However, the WAC codes have not been updated with the most recent and specific NFPA 2-2016 Hydrogen Technologies code.

WSU Environmental Health and Safety provides guidance on the development of safety plans. WSU Environmental Health & Safety (EH&S) maintains an on-line laboratory safety manual at: https://ehs.wsu.edu/labsafety/LabSafetyManual.html. A list of 1-page helpful fact-sheets is available at: https://ehs.wsu.edu/training/EHS-Factsheets.html. Both are also available on the laboratory website. Shawn Ringo is currently the head person in this area and has been involved in this project. Safety evaluations and audits are conducted annually by the Experimental and Laboratory Safety Committee within the School.

All experiments at WSU are advised to have a safety plan within a printed notebook next to the experiment. This notebook is in addition to the laboratory emergency and chemical hygiene plan on the laboratory website and the HYPER lab internal intranet. It is recommended that this safety plan be revised whenever extensive modifications occur to the experiment and at least annually.

2.4. HYPER Laboratory Experience

Dr. Jacob Leachman is the HYPER laboratory Director. Dr. Leachman developed specific expertise for hydrogen research by creating the current equations of state for hydrogen property estimation during a Master's Thesis at the University of Idaho from 2005-2007 and was trained for cryogenic hydrogen experiments during a Doctoral Dissertation at the University of Wisconsin-Madison from 2007-2010. He has directed the HYPER laboratory since founding in 2010. The HYPER laboratory has operated cryogenic hydrogen experiments since inception. WSU became a founding member of the AIChE Center for Hydrogen Safety in spring of 2019.

Carl Bunge is a 4th year graduate student and has been involved/leading the operation of CHEF since 2016. Carl was involved with the original DOE Safety Panel review of CHEF in 2016 and designed the pressure relief systems per CGA S-1.1 code. Carl has operated CHEF for over 7500 hours of cryocooler operation and the successful liquefaction and test of over 150L of liquid hydrogen to date.

HYPER Lab facilities include a testing area in room 221 of the Engineering Teaching and Research Laboratory (ETRL). This approximately 750 square foot space contains four cryostats capable of liquefying hydrogen with the maximum capacity of any system being 6.7L. Below is a rendering of the ETRL 221 space and adjacent gas closet in 219.

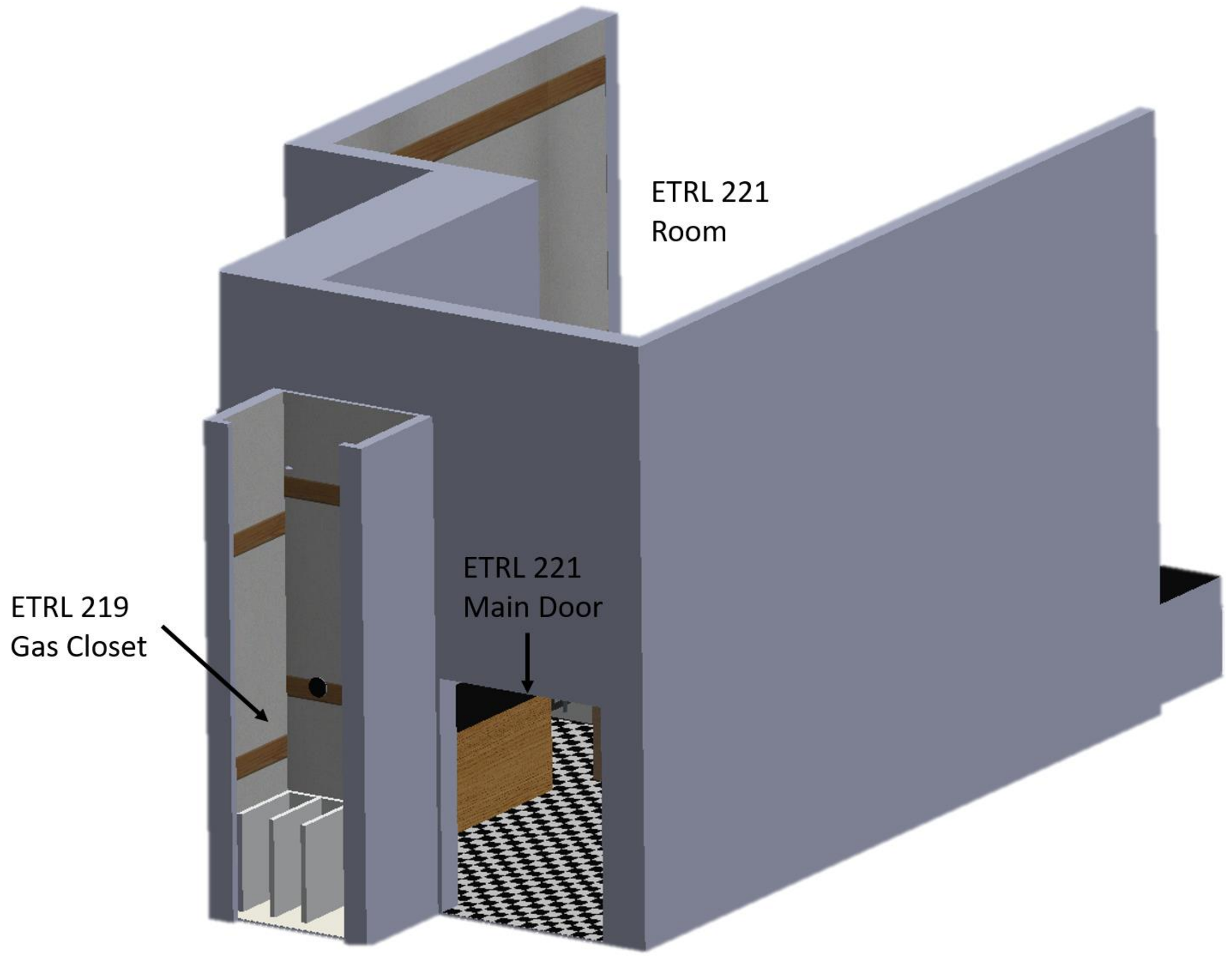


Figure 1: ETRL 221 and 219 renderings without ceiling and north wall with windows.

The Cryo-catalysis Hydrogen Experiment Facility (CHEF) recently surpassed 12,000 hours in operation, over which 350L of hydrogen has been liquefied. No accidents have occurred in the HYPER Lab. A near miss event occurred in 2016 due to a campus wide power outage. Additional safety measures have since been put into place to reduce the possibility of a similar event in the future. More information on the near miss event is available here: https://hydrogen.wsu.edu/2016/08/15/our-near-miss-hydrogen-vent-in-etrl-221/. The near miss prompted installation of a dedicated hydrogen vent stack that exhausts to the roof of ETRL. The Department of Energy (DOE) Hydrogen Safety Panel has reviewed several HYPER Lab Safety Plans and project proposals, and provided recommendations that have informed HYPER Lab operating practices and procedures. The HYPER Lab will fill the capacity of ETRL 221 in the spring of 2020 with installation of a fourth cryostat. Figure 2 represents the location of CHEF within the ETRL 221 space. It is located in the northeast corner of the lab.

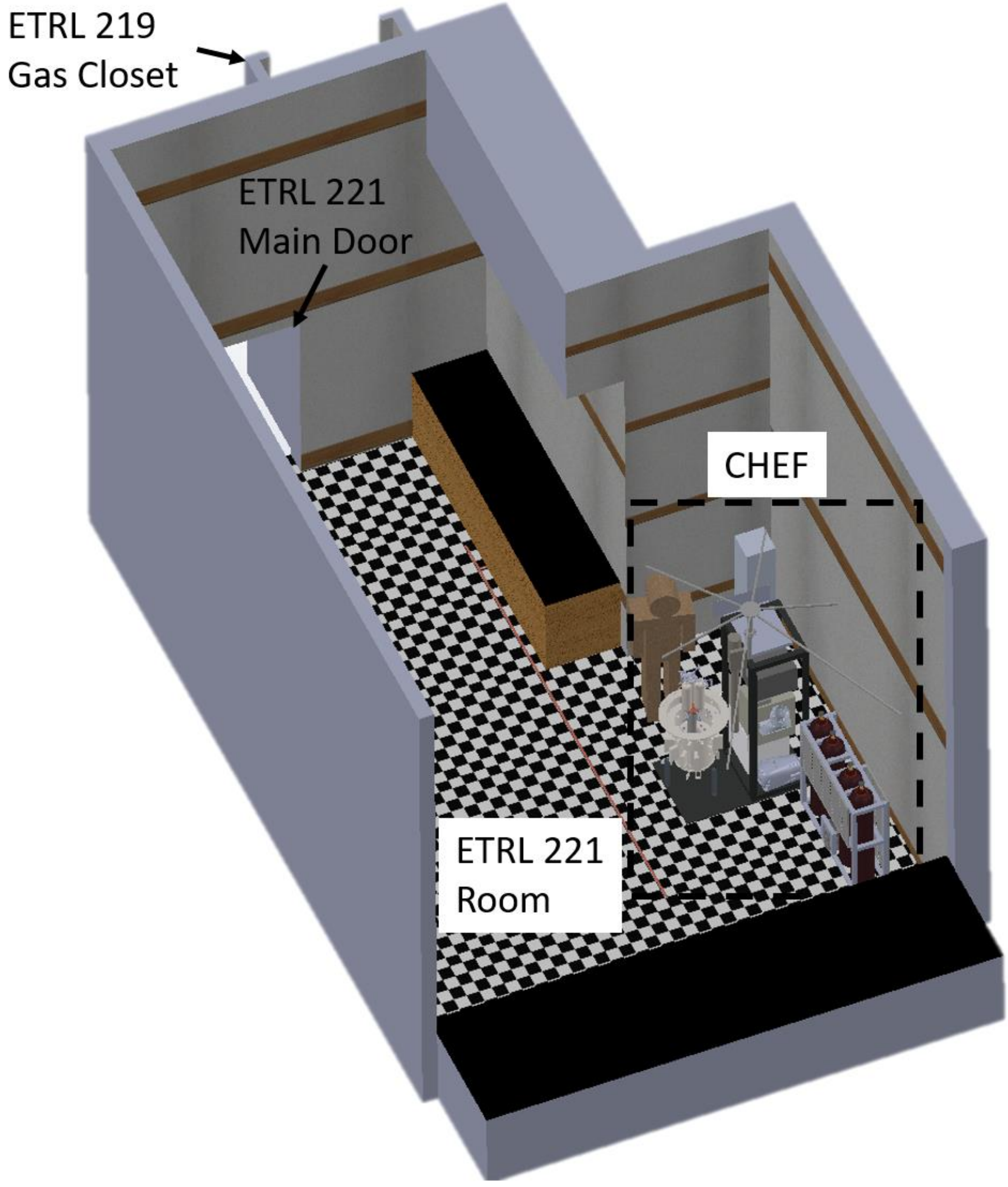


Figure 2: Rendering of the inside of ETRL 221 with CHEF located at the northeast corner of the room.


Figure 3 (Left): ETRL 221 Laboratory and testing space. Figure 4 (Right): TFRB 113 build space.

HYPER Lab design, planning, and construction work occurs in the Thermal Fluids Research Building (TFRB). 5500 sq. ft of space in this building is dedicated to the work done by the HYPER Lab, including a high-bay workshop and associated design suite. However, TFRB is a building without fire suppression equipment and hence is not suitable, nor feasibly modified, to allow for hydrogen testing.

2.5. History of CHEF:

The Cryocatalysis Hydrogen Experiment Facility (CHEF) was originally developed in 2012 to investigate the performance of cryogenic catalysts for Parahydrogen-orthohydrogen conversion. The experiment is currently able to liquefy or 6.7L (1.7 gallons) of hydrogen. The first version of the experiment was constructed of brass NPT pipe fittings and operated for ~6 months and approximately 10 cycles. The experiment boiled-off parahydrogen to flow through a catalyst bed and over a calibrated hot-wire para-ortho composition cell. Ron Bliesner received his Master of Science (MS) degree in mechanical engineering in 2012 for developing CHEF.

In the fall of 2014 the experiment was refurbished with primarily welded stainless steel plumbing within the cryostat and two additional catalyst beds were added with a change to a vertical orientation. This change allowed characterization of scrim-deposited Ruthenium-Iron-Oxides for para-orthohydrogen conversion. This experiment functioned for ~15 cycles until February of 2016 when it was transformed into the current configuration. Brandt Pedrow received a Master of Science (MS) degree in 2016 for the refurbishment and catalyst blanket tests.

In 2016 CHEF was adapted to testing a cryogenic vortex tube through the Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Fuel Cell Technologies Office project for "Improved Hydrogen Liquefaction through Heisenberg Vortex Separation of para and orthohydrogen". Multiple types of vortex tubes have been tested. Catalytic material for para-orthohydrogen conversion is deposited on the inner wall and compared to a non-catalyzed version. After a near-miss and DOE Hydrogen Safety Panel review, several improvements to the operating procedure, building capability, and general safety plans, including this safety plan document have been made.

After extended tracking of cold thermal expansion differences (cold leaks), the experiment was able to seal at cryogenic temperatures and allow interesting results sub – 50K for applications related to boil-off reduction on in-space cryogenic tanks for NASA by Carl Bunge then funded under a NASA Space Technology Research Fellowship (NSTRF16) "Heisenberg Vortex for light-weight Liquid Hydrogen Refrigeration".

The current project is funded by the Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the Fuel Cell Technologies Office project for "Optimizing the Heisenberg Vortex Tube for Hydrogen Cooling" focused on optimizing the HVT for on-ground applications related to reducing boil-off through the implementation of a TVS system, a pump subcooler, and a supercritical expander with Plug Power. This requires the most recent update of the experimental facility to increase test cycle time, reduce number of internal connections, and allow supercritical pressure operation.

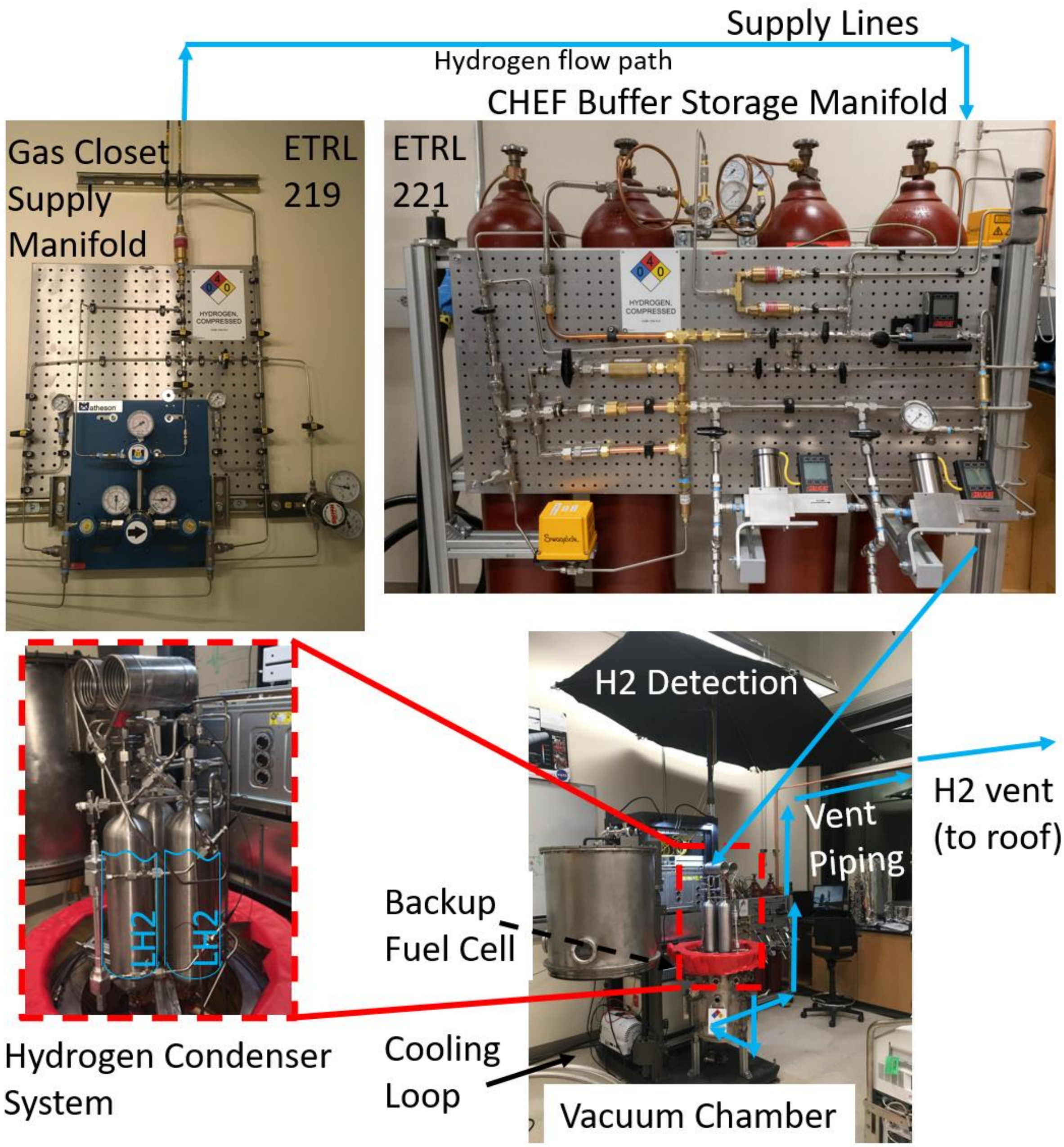
In total CHEF has successfully liquefied approximately 350L of LH2 over 8 years with review by the DOE Hydrogen Safety panel in 2016.

3. IDENTIFICATION OF SAFETY VULNERABILITIES

The identification of primary and secondary failures through a Hazard and Operability (HAZOP) is provided in section 3.3 and a Failure Modes and Effects Analysis (FMEA) is in section 3.2. The block diagram below details how hydrogen is processed through the CHEF system.

3.1. PROCESSED HYDROGEN BLOCK SYSTEM DIAGRAM

All hydrogen to be processed through the Cryo-catalysis Hydrogen Experiment Facility is delivered in 197 cu ft compressed gas K-bottles stored in the ETRL gas closet. This system is housed in the Engineering Teaching Research Laboratory rooms 219 and 221 as seen below.



Hydrogen starts from the gas closet supply manifold in the NFPA 2 compliant vented gas closet. It then flows though a dedicated hydrogen supply line into room 221 and to the CHEF buffer storage manifold through a flow limiting orifice to prevent lower flammability levels in ETRL 221. Once a liquefaction cycle is under way, the low pressure (140 psi max) gaseous hydrogen flows into the hydrogen condenser system within the vacuum chamber. It is here where the liquid hydrogen accumulates. Once the condenser tanks are at a desired fill level, heaters are activated which initiates boil off through a catalytic process. The outlet of the now processed hydrogen exits the vacuum chamber and proceeds to the vent piping. The vent piping lead to the H2 vent system which routes the now near atmospheric pressure and temperature hydrogen to the roof of ETRL. The H2 detection and purge system monitors hydrogen levels with a flame retardant hood with a hydrogen detector. The backup fuel cell maintains emergency systems during a power outage.

3.2. Hazard and Operability (HAZOP) Study

Per recommendation of the H2 Safety Panel for the 7/30/2019 safety plan version, a HAZOP is recommended in addition to the above FMEA in section 2:

Per the Department of Energy "Safety Planning Guidance for Hydrogen and Fuel Cell Projects" document a HAZOP involves the following:

Method	Description	References
HAZOP Hazard and Operability Analysis	Systematically evaluates the impact of deviations using project information. Method was developed to identify both hazards and operability problems at chemical process plants.	An extensive description and worked example of the HAZOP procedure can be found in *Guidelines for Hazard Evaluation Procedures*, *Second Edition with Worked Examples*, Center for Chemical Process Safety, American Institute of Chemical Engineers, 1992.

The following HAZOP analysis is from example from Guidelines for Hazard Evaluation Procedures, Third Edition with Worked Examples, Center for Chemical Process Safety, American Institute of Chemical Engineers, 2008.

3.2.1. Deviation-by-deviation (DBD) HAZOP:

HAZOP studies are designed for interdisciplinary teams which allow creativity on a team basis to allow more adverse situations to be identified and corrected. It is designed to be implemented when the P&ID is in final stages or complete for review and there is still opportunity to change without major cost impacts. Therefore, the following study will consist of an in-depth view of the operating procedures and P&ID of all sub-systems nodes captured in a blended library-based and knowledge-based approach. A deviation-by-deviation HAZOP table will be used to document the sections below.

3.2.1.1. Process Type

The CHEF operates in a batch-type process to accumulate liquid hydrogen in 6.7L which is then boiled off for testing purposes.

3.2.2. Library of Relevant Deviations:

As part of the library-based approach it is common to incorporate more realistic deviation guide-words as the possible choices. These modified deviations are adapted to HYPER Lab specific deviations and seen below. For example "cryogenic" temperature instead of "low" temperature.

The following table identifies the relevant process sections and how they are related to the various deviations.

HAZOP library of relevant deviations for process section types

Deviation	CHEF process section type						
	Vacuum Chamber	Supply lines	Vent piping	Fuel cell backup	Hydrogen Condenser system	H2 Detection/ purge*	Cooling loop
High flow		SL1	VP1				
Low/no flow				FC1	HC1		CL1
High level					HC2		CL2
High pressure	VC1	SL2		FC2	HC3		
Low (near vacuum) pressure	VC2	SL3		FC3	HC4		
High temperature					HC5		CL3
Cryogenic temperature			VP2				
High concentration (H_2 + air)						DP1	
Reverse/misdirected flow	VC3	SL4	VP3	FC4			
Leak	VC4	SL5	VP4		HC6		CL4
Rupture	VC5	SL6	VP5		HC7		CL5
No Power				FC5		DP2	CL6

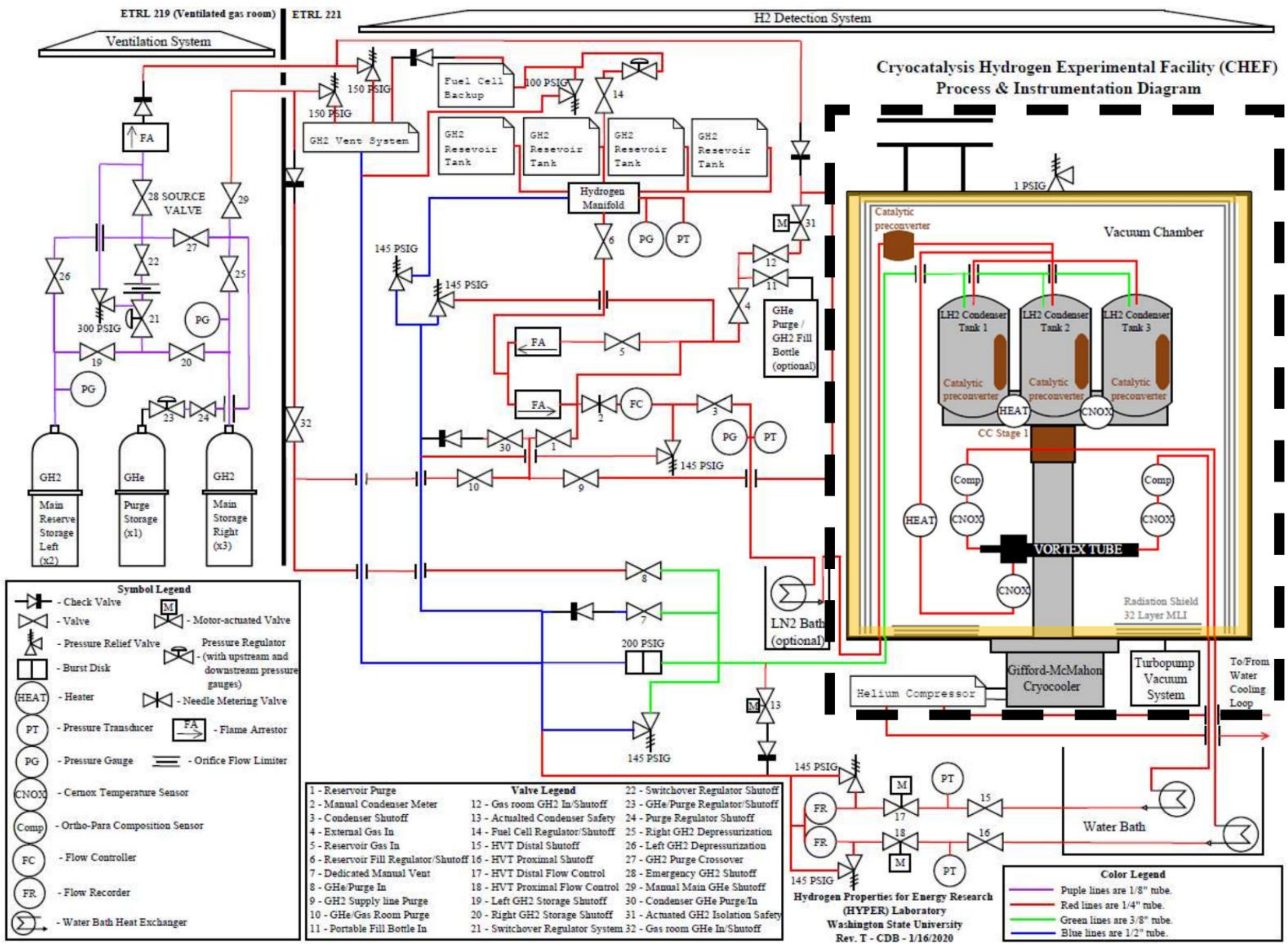
* The H2 detection and downstream purge system deviations are considered in the vent piping section.

A HAZOP consisting of the combination of the library-approach and deviation-by-deviation strategy will be used to address personnel and hardware hazards.

3.2.3. TABULAR HAZOP STUDY:

The tabular HAZOP here details the responses of each of the above deviations. Each node is highlighted in yellow and out lines with a dashed box.

3.2.3.1. VACUUM CHAMBER

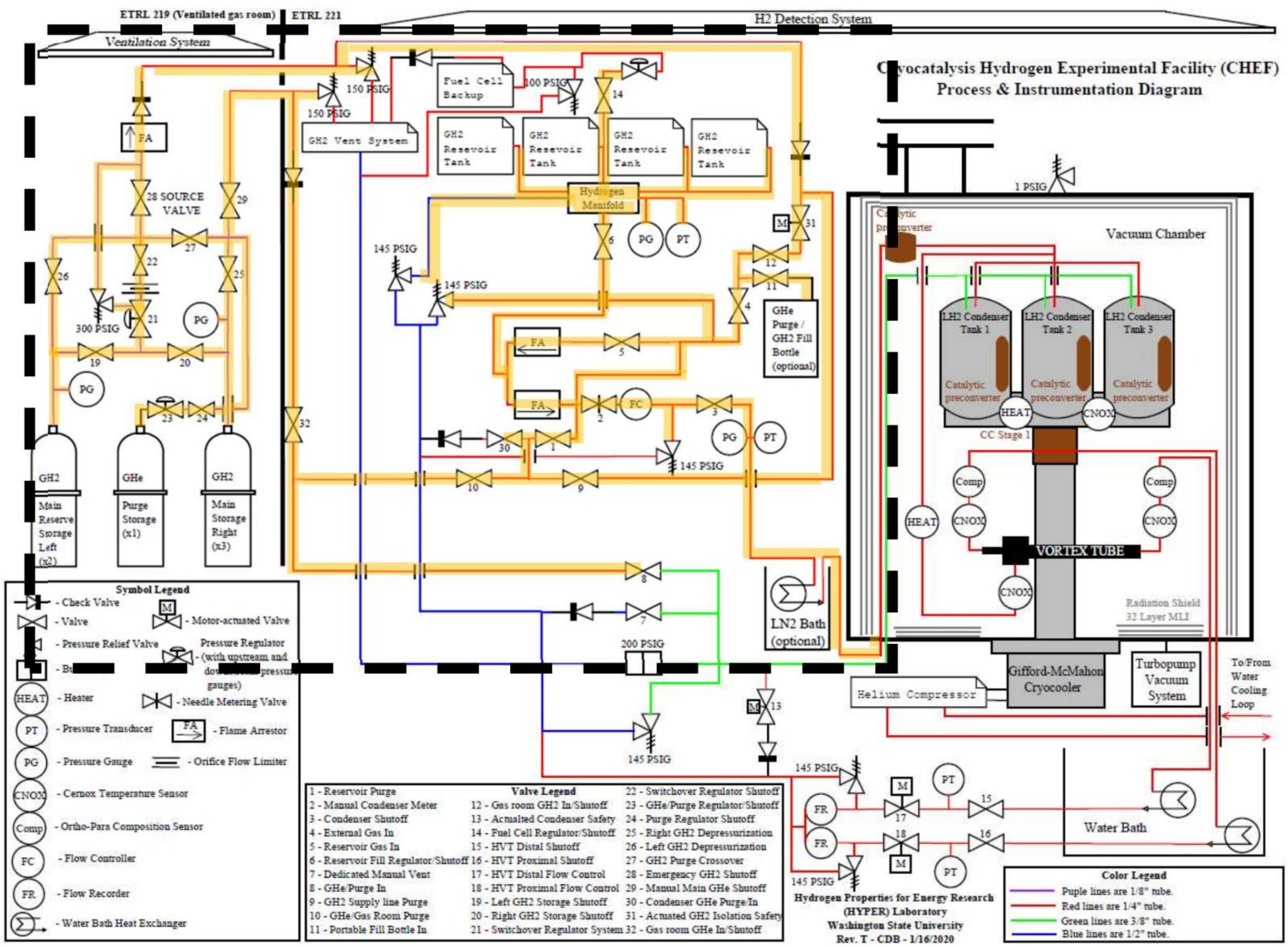


Team: WSU HVT Team			P&ID No.: Rev. Q Study approach: Deviation by deviation		
Item	**Deviation**	**Causes**	**Consequences**	**Safeguards**	**Actions**
VC - Vacuum Chamber. Intent – Maintain convection-less environment to limit heat transfer to cryogenic components corresponding to a pressure level of 1e-5 and 1e-9 torr					
VC1	High pressure reading (>1e-5 torr)	Faulty vacuum gauge	Inability to reach cryogenic temperatures	Operator monitor of cooldown period	Annual inspection/ cleaning of vacuum gauge

		Leak into vacuum chamber from external interfaces	Freezing of water moisture from air on cryogenic (cold) components including within MLI shield		Annual inspection of vacuum chamber seals via leak check procedure 4.2.1.
		Leak into vacuum chamber from internal components	Large turbomolecular pump load (overheating) resulting in VC4.		Helium leak check (per section 4.2.1) before each test
VC2	Low pressure (Ultra High Vacuum = <1E-9 torr)	Vaporization of organic or polymeric components	Degradation of room-temperature O-ring seals	Routine inspection of O-ring seals	Consider inspection of cryocooler O-ring seal in major experimental updates
VC3	Reverse/mis-directed flow	Incorrect attachment of turbo backing roughing pump	Release of positive pressure through chamber pressure relief valve	Always inspect vacuum inlet/outlet labels	Maintain vacuum pump inlet/outlet labels on all vacuum pumps
VC4	Leak	Non-functional seal (piping or chamber)	Excessive convection in vacuum chamber resulting in atmospheric water vapor condensing on components and boil-off of cryogen	Conduct a chamber / piping leak check per section 4.2.1	Discontinue test and complete warm up procedure in section 4.4.
			Result in VC1 – High pressure reading	Conduct a chamber / piping leak check per section 4.2.1	Complete a hard leak check annually per maintenance schedule
VC5	Rupture	Transient internal plumbing rupture	Rapid increase in internal pressure resulting in VC3 and potential unplanned rapid disassembly	Ensure vacuum chamber containment bonnet is	Inspect bonnet annually for fatigue from

		attached for every test	attaching and detaching
	Degradation of turbomolecular pump Vertical lift of the vacuum chamber to release internal pressure into Kevlar/Nomex containment bonnet directed upwards towards H2 detection sensor	Test turbopump in separate test to ensure balance and bearing health	Execute emergency abort per section 4.5.4

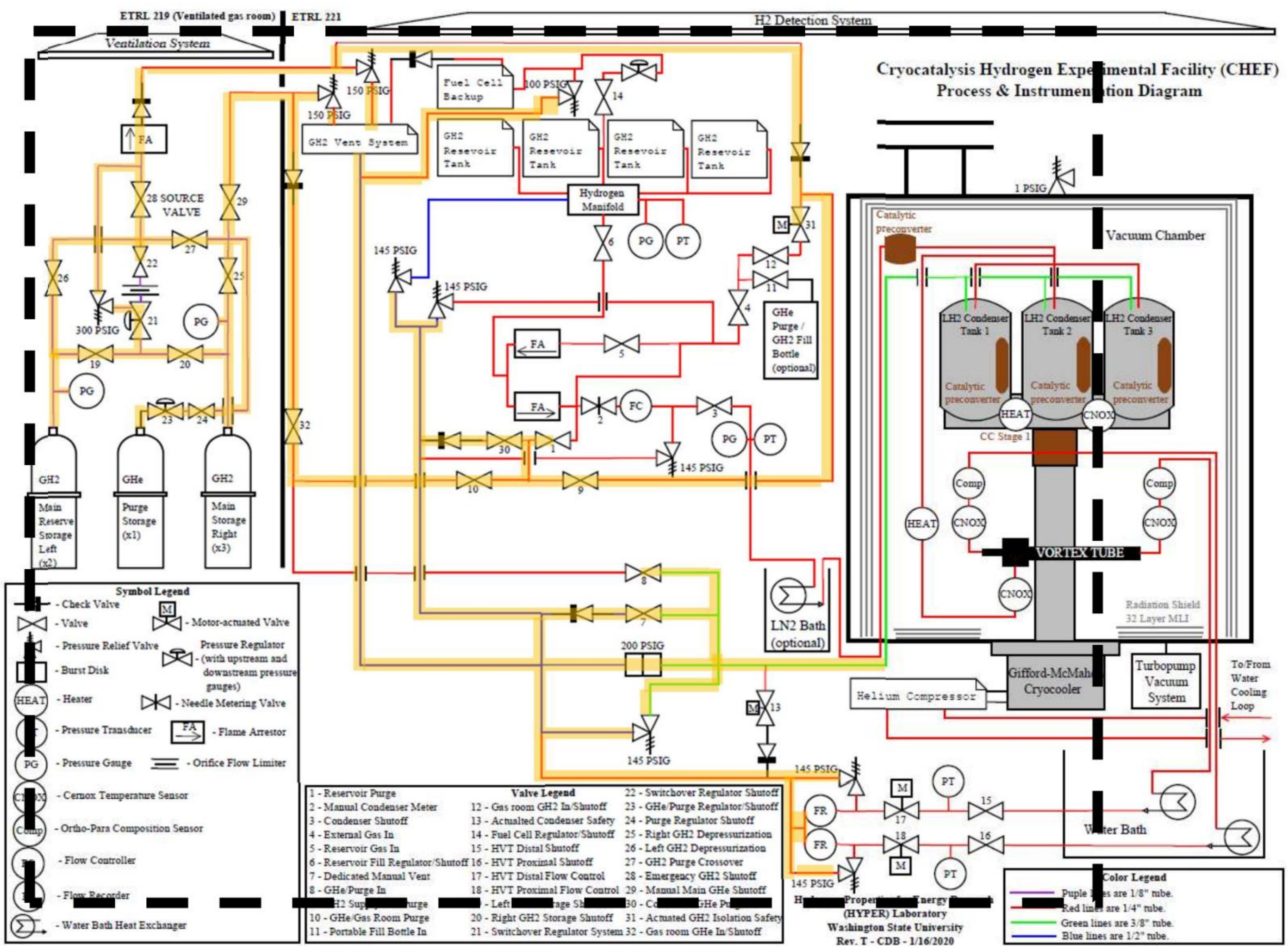
3.2.3.2. GH2 AND GHE SUPPLY LINES



Team: WSU HVT Team			P&ID No.: Rev. Q Study approach: Deviation by deviation		
Item	Deviation	Causes	Consequences	Safeguards	Actions
SL – Supply Lines.					
Intent – Deliver low pressure (<145 psig) hydrogen to the Cryo-catalysis Hydrogen Experiment Facility in ETRL 221 from hydrogen rated ETRL 219 bottle closet.					
SL1	High flow (>0.45 g/s)	Failed orifice flow limiter in combination with failed switchover gas regulator	Activation of dedicated supply line pressure relief (145 psig) and discharge of ETRL 219 closet H2 supply into dedicated hydrogen vent system	Solid-state, welded in-line orifice plate limiter	Automated hydrogen detection and purge system will passivate system of hydrogen
SL2	High pressure (>145 psig)	Pressure regulator run away	Activation of dedicated pressure relief	10 year max service life of regulator	Shut valve **[28]** source valve in gas closet
		Process intent (test to 150 psig pressure relief valve)	Activation of dedicated pressure relief	Training for acceptable pressure relief testing	Follow standard pressure relief guidelines
SL3	Low (near vacuum) pressure	Complete depletion of supply and reserve GH2 supply	Depending on level of vacuum, the system might pull atmospheric air into process plumbing	User checking reserve and supply banks in gas closet on a regular basis	Regular check of supply and swapping bottles per section 4.2.6.1
SL4	Reverse/mis directed flow	Increased process pressure beyond that of the supply lines in combination with check valve leak	Back flow of process into supply storage	Hardware testing of check valves per maintenance log	Depending on the back filling contaminant, purge system per section 4.2.4
SL5	Leak	Leak in supply lines	Increase in hydrogen detection sensor	Fully welded construction of supply lines	Annual helium leak check of

			Audible anomaly	with secure wall fixturing	both He and H2 supply
SL6	Rupture	Over pressurization (>5100 psi, 351 bar)	Rapid dispersion of hydrogen into ETRL 219 bottle closet or ETRL 221	H2 detection sensor to passivate all hydrogen out the dedicated building vent line Do not connect 350 bar+ bottles to supply system (124 bar bottles preferred)	Follow emergency abort procedures in section 4.5.4 or allow system to auto passivate through hydrogen detection and purge system

3.2.3.3. VENT PIPING

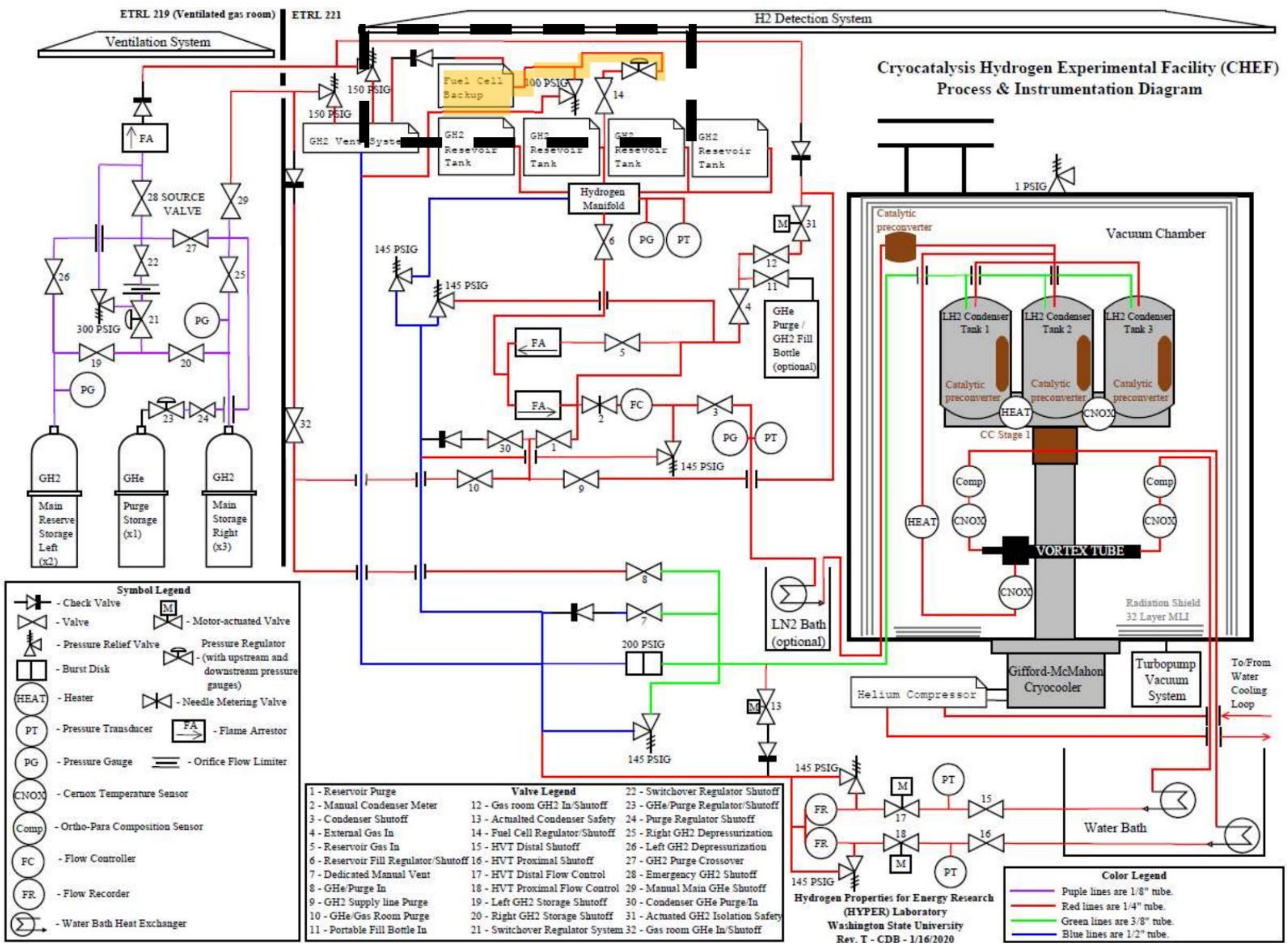


Team: WSU HVT Team | P&ID No.: Rev. Q | Study approach: Deviation by deviation

Item	Deviation	Causes	Consequences	Safeguards	Actions
VP – Vent Piping. Intent – Allow safe venting of processed hydrogen to dedicated ETRL hydrogen vent system.					
VP1	High pressure reading (>1800 psi)	Failed pressure regulator Active helium and/or hydrogen vent occurring out dedicated building vent line	Waste of GHe or GH2 out the vent line	1st and 2nd stage pressure relief to dedicated building vent system	Operational review of helium pressurized liquid (supercritical) hydrogen tests Pressure reduction to below 1800 psi
VP2	Cryogenic temps (<123 K)	Vent of boiled off liquid hydrogen to the dedicated vent line	Thermal contraction of brass NPT connections with pipe tape	Line hangars with the ability to allow thermal contraction	Helium leak check of vent piping on an annual basis or after cryogenic fluid introduction event
VP3	Reverse/mis-directed flow	Decrease of process pressure below that of atmospheric pressure in combination with a failed check valve	Introduction of atmospheric (and/or other vented fluids from other experiments) into experimental process plumbing	Always check valves per maintenance procedure Communicate with other experiments in lab to develop testing times	Check non-return (check) valves per experimental maintenance log Lab testing coordination sheet
VP4	Leak	Leak in vent lines	Increase in hydrogen detection sensor measurement	Always complete helium leak	Execution of section 4.5.2

			Audible anomaly	check before each test Minimum annual helium leak check of vent piping per section 4.2.1	
VP5	Rupture	Over pressurization	Rapid dispersion of hydrogen into ETRL 221 Reach a maximum H2 vol % in air of ~2%	H2 detection sensor to passivate all hydrogen out the dedicated H2 vent line Dissipation via building vent system with 1st and 2nd stage (burst disk) at ~2000 psi	Execute emergency abort protocol per section 4.5.4 Follow annual leak check procedures

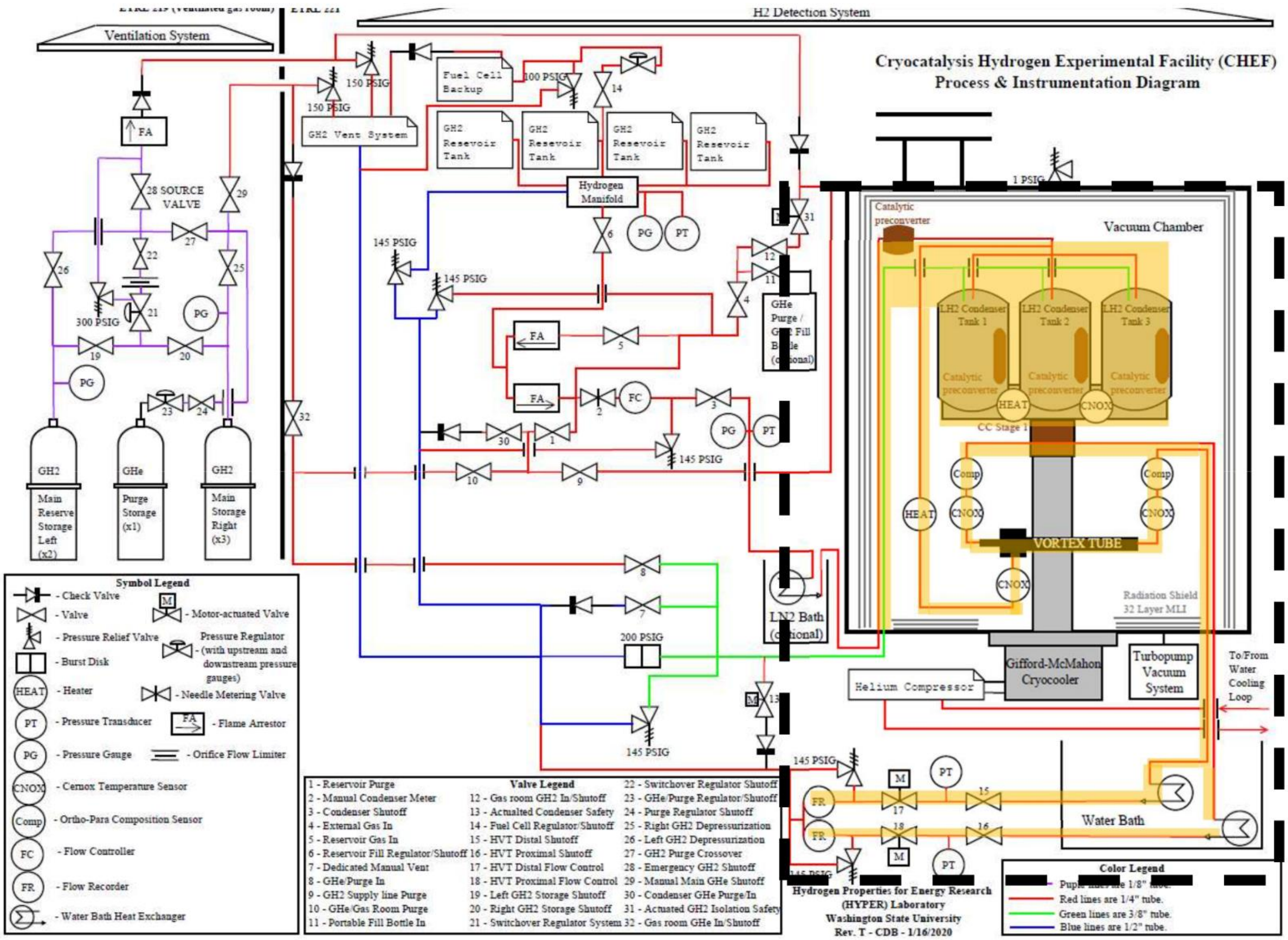
3.2.3.4. Fuel cell backup



Team: WSU HVT Team			P&ID No.: Rev. Q Study approach: Deviation by deviation		
Item	**Deviation**	**Causes**	**Consequences**	**Safeguards**	**Actions**
FC – Fuel Cell Backup.					
Intent – Allow vacuum and auxiliary safety systems to continue operating while system warms.					
FC1	Low/no flow	Valve to let hydrogen into fuel cell is shut off to hydrogen buffer storage	Inability to back up vacuum and emergency systems beyond 8 minutes in the event of a power outage	Operational inclusion of fuel cell standby procedure	Include fuel cell standby and shutdown procedures at the start of all relevant procedures

FC2	High pressure (>100 psig)	Regulator run away and/or set beyond 100 psig	Activation of dedicated pressure relief at 100 psi	Operational warnings (100 psig max)	Reduce pressure to within 100 psig
FC3	Low pressure (<25 psig)	Depletion of buffer storage hydrogen supply in the event of a power outage	Shutdown of hydrogen fuel cell	Designed shut off of fuel cell Systems will shut off as normal	None or increase regulator set point
FC4	Reversed/ misdirected flow	Procedural deviation from shutdown procedures	Introduction of GHe into fuel cell Introduction of non-purged H2 into FC	Follow procedural shutdown and isolation in the event of full system purge Default system purge of Reli-On FC	Inclusion of fuel cell reminders in the purge procedures
FC5	No power	Depletion of buffer storage hydrogen supply in the event of a power outage	Shutdown of hydrogen fuel cell	Designed shut off of fuel cell Systems will deactivate per nominal designed shutdown in the eventual loss of power from backup system	Isolation and lockout tagout of hydrogen supply to FC and troubleshooting per Reli-On recommendations

3.2.3.5. HYDROGEN CONDENSER SYSTEM

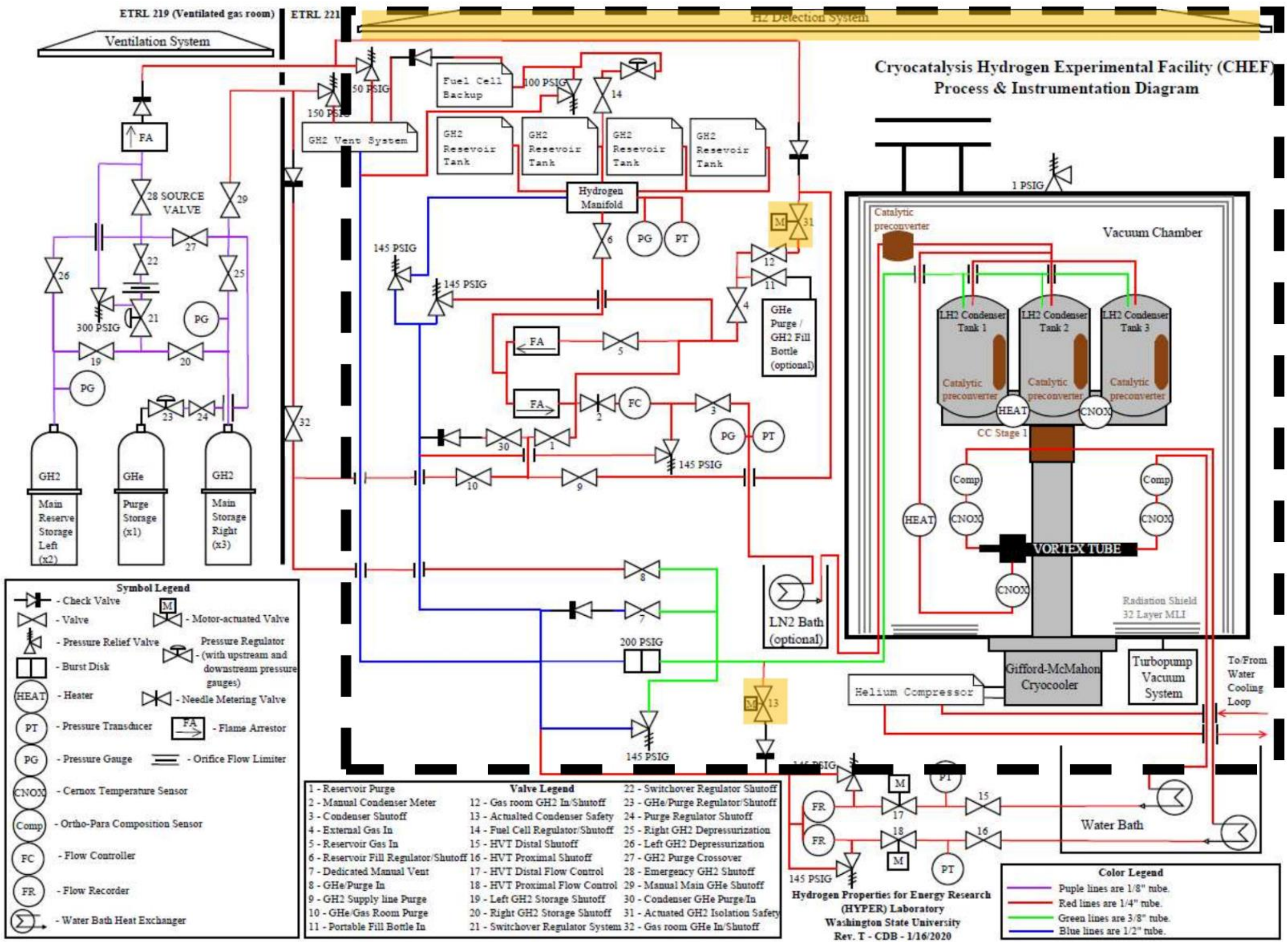


Team: WSU HVT Team			P&ID No.: Rev. Q Study approach: Deviation by deviation		
Item	Deviation	Causes	Consequences	Safeguards	Actions
HC – Hydrogen Condenser system. Intent – Allow collection of liquid hydrogen and delivery of various ortho-parahydrogen percentages, temperature, pressures, and flow conditions to catalytic processes of interest.					
HC1	Low/no flow (H2 into condenser)	Full condenser tanks in thermal equilibrium	None, ready to complete a test.	Flow tracking and control via Alicat flow controller	None
		Ice or contaminant	Little or no communication between internal tank	Always order ultra-high purity	Follow warm up procedure 4.4

		blockage in fill line	temperature and indicated pressure upstream of blockage. Little to no flow out of flow controller Alicat after line is vented down.	hydrogen (99.999%)	
		Gas room supply depletion	See section 4.2.6.1	Check backup supply bank pressure	If needed replace depleted tanks per section 4.2.6.1
HC2	High (LH2) level	Software restart resulting in loss of flow level tracking	Faster cool down with initial liquid extraction (for gaseous test)	Always maintain a consistent computer update cycle (in between runs)	Always maintain a consistent computer update cycle (in between runs)
HC3	High pressure reading (>1800 psig, 124 bar)	Faulty pressure transducer gauge (electronic)	Inability to read actual process pressure via LabVIEW	Redundant pressure gauges (Bourdon gauge)	Review of wiring to pressure transducer
		Over pressurization of process fluid	Activation of 1800 psi pressure relief valve	Activation of dedicated burst disk at (~2000 psig)	Review of process heating procedure
HC4	Low (near vacuum) pressure	HC6 that results in a small leak into an operational vacuum environment	Overload and potential thermal shutdown of turbo Warming of condenser tanks due to loss in chamber vacuum and activation of pressure relief	Always complete a leak check per section 4.2.1	The system will passivate via pressure relief
		Blockage in condenser which causes small tube volume upstream to	Inability to condense more hydrogen. Potential triggering of pressure relief due to VC4	Always procure 99.999% ultra high purity hydrogen	Follow warm up procedure 4.4

		condense and create a low pressure reading on the pressure transducer			
HC5	High temp (>415K)	Heaters set on manual mode without upper temperature limit	Nearing the melting point of indium. Leak check Raman cells	Always warm up experiment with PID temperature limits	See warm up procedure 4.4
HC6	Leak	See VC4	See VC4	See VC4	See VC4
HC7	Rupture	See VC5	See VC5	See VC5	See VC5

3.2.3.6. H_2 DETECTION / PURGE SYSTEM

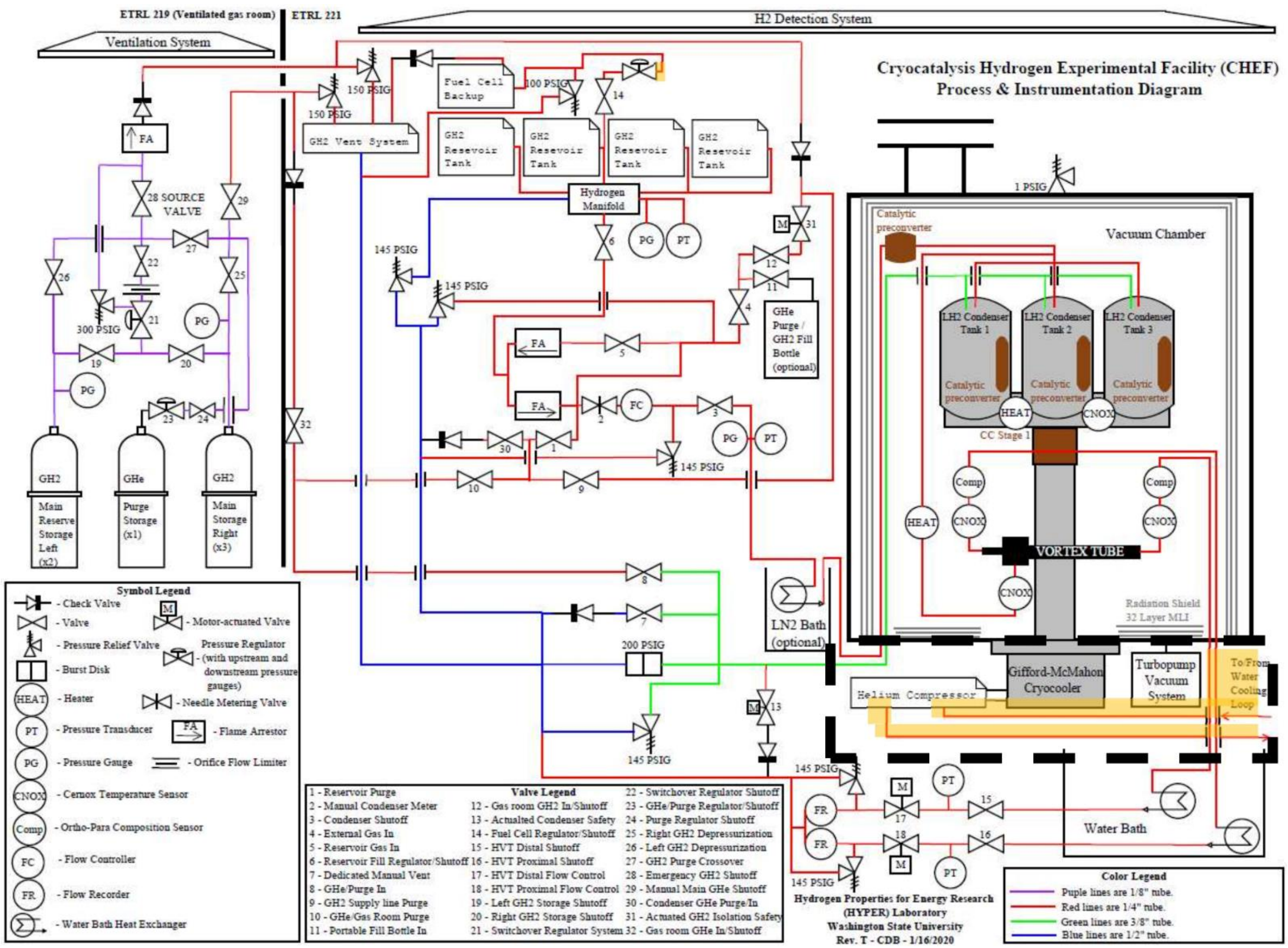


Team: WSU HVT Team | P&ID No.: Rev. Q
Study approach: Deviation by deviation

Item	Deviation	Causes	Consequences	Safeguards	Actions
DP – Detection and Purge system. Intent – Detect any hydrogen vented to the room and activate auto purge system is concentration is above 60% LFL (2.4% H2 absolute)					
DP1	High Concentration (>60% LFL)	External plumbing leak	Activation of emergency valve system (supply shuts and vent opens)	Follow leak check procedure 4.2.1 before each experimental cooldown	Follow procedure 4.5.2 if occupying the room, otherwise system will

					passivate hydrogen
		Sensor calibration gas too close (or above) LFL	Triggering of LFL alarm if not in calibration mode	Always procure <60% LFL calibration gases	Discontinue calibration and procure calibration gases within specification (<60% LFL)
DP2	No (electrical) Power	Backup power system shuts down nominally after providing power	Supply and vent valves will remain in their anomaly state to maintain separation from supply and will continue to vent any liquid boil-off due to vacuum loss	None	None
		Fuel cell backup fails to ramp to sufficient power within buffer battery storage holdover time	If the backup battery buffer is able to maintain inverter for ~3 seconds, then the emergency valves will actuate and passivate. If not, the first stage pressure relief will passivate the system	Always maintain proper backup battery buffer charge via remote operation of fuel cell during runs	Follow section 4.2.6 to enable fuel cell remote mode when starting a run

3.2.3.7. Cooling Loop



Team: WSU HVT Team — P&ID No.: Rev. Q
Study approach: Deviation by deviation

Item	Deviation	Causes	Consequences	Safeguards	Actions
CL – Cooling Loop.					
Intent – Provide chilled water to the cryocooler room-temperature compressor.					
CL1	Low/no flow	Water pump overheat	Water pump will shutoff causing cryocooler compressors to gradually warmup and activate thermal temperature shutoff causing experiment to lose cooling and warm. All systems shall be	Ensure cooling loop is free from debris that could jam pump	Avoid use of galvanized parts in cooling loop and always use DI water

			designed for this deviation and be able to passivate the hydrogen		
		Blockage in cooling line and/or filters	Minimal flow to pump causing abnormal operating conditions	Change filters per maintenance schedule	Avoid use of galvanized parts in cooling loop and always use DI water
CL2	High (water) level	Overfilling of buffer tank volume with DI water	Slip hazard on adjacent flooring	Never fill beyond maximum water buffer storage	Use caution when refilling water buffer storage tank
CL3	High temp (>80°F)	Building cooling water supply not turned on and/or CL1 deviation	Cryocooler compressors will gradually warmup and activate thermal temperature shutoff causing experiment to lose cooling and warm. All systems shall be designed for this deviation and be able to passivate the hydrogen	Always follow procedure 4.2.6 to ensure building cooling loop is active	Always make sure building cooling loop is on per section 4.2.6
CL4	Leak	Loose fitting and/or hose rupture	Slip hazard on adjacent flooring and depletion of cooling loop buffer water storage	Visually inspect cooling loop tubing per maintenance schedule	Tighten/replace fitting and/or fix hose
CL5	Rupture	Improper sizing of hose and/or fittings to MAWP of pump	Slip hazard on adjacent flooring	Size components correctly during modification	None
CL6	No Power	Building power outage	Cryocooler compressors will gradually warmup and activate thermal temperature shutoff causing experiment to lose cooling and warm. All systems shall be designed for this deviation and be able to passivate the hydrogen	None	All systems shall be designed for this deviation and be able to passivate the boiled off cryogen

3.3. Failure Modes and Effects Analysis (FMEA)

The following FMEA will continue to allow the safety of personnel, equipment, and environment. A three-dimensional process is applied through the safety failure/hazards matrix:

1) Detection -ability of a safety vulnerability,

2) Frequency that the safety vulnerability occurs, and

3) Severity/consequence of a safety failure.

Each of these dimensions are rigorously defined and categorized in the paragraphs below before the Safety Failure/Hazards Matrix.

3.3.1. Detection

Detection concerns the ability to Identify a Safety Vulnerability (ISV). For example, hydrogen is an odorless, colorless, tasteless gas and may be present in a room. Without a calibrated hydrogen sensor, it may not be possible to ISV. The following detection categories and criteria will be applied:

Detection	Rating	Criteria
Extremely Remote	**9**	Very remote chance that the control will PREVENT or DETECT the failure mode, effect or cause. *Process example: Control is achieved with indirect of random checks only.*
Remote	**7**	Low chance that the control will PREVENT or DETECT the failure mode, effect or cause. *Process example: Control is achieved with visual or double visual inspection only.*
Moderate/ Occasional	**5**	Moderate chance that the control will PREVENT or DETECT the failure mode, effect or cause. *Process example: Control is achieved with control charting (SPC) or is based on gauging the parts after the parts have left the station (100% go/no go gauging, variables gauging).*
Probable	**3**	High chance that the control will PREVENT or DETECT the failure mode, effect or cause. *Process example: Error detection in subsequent operations (can not accept discrepant part), gauging of set up or first piece check (set up causes only), error detection in station.*
Almost Certain	**1**	Almost certain that the control will PREVENT the failure mode or cause. *Example: Discrepant parts cannot be made because item has been error proofed by progress/product design.*

3.3.2. Frequency

Frequency concerns how often the safety vulnerability occurs. For example, it is safer to store a hydrogen bottle in the gas cabinet than in the laboratory, however frequently bringing the bottle in to charge an experiment increases the risk of a regulator failure. The following frequency categories and criteria will be applied:

Frequency	Rating	Criteria
Almost Certain	**9**	Expected to occur one or more times during a project lifecycle; or likely to occur many times in the lifecycle of the system.
Probable	**7**	Expected to occur between once a year and once every few years; or will occur several times in the lifecycle of the system.
Moderate/ Occasional	**5**	Expected to occur several times during the lifetime of the facility (30 years); or likely to occur sometime in the lifecycle of the system.
Remote	**3**	Expected to occur no more than once during the lifetime of the facility (30 years); or unlikely, but possible to occur in the lifecycle of the system.
Extremely remote	**1**	Not expected to occur during the lifetime of the facility (30 years); or so unlikely it can be assumed not to occur in the lifecycle of the system.

3.3.3. Severity

Severity concerns how critical the severity of the failure/safety vulnerability is. For example, a hydrogen ignition can be as small as an audible pop, or as big as the entire wall of the building blowing off.

Severity	Rating	Criteria
Catastrophic	**9**	Could result in multiple critical incidents to individuals and buildings.
Critical	**7**	Could result in one or more of the following: death; permanent total disability; irreversible significant environmental impact; monetary loss equal to or exceeding $10 million; or loss of the system.
Substantial	**5**	Could result in one or more of the following: permanent partial disability; injuries or occupational illness that may result in hospitalization; reversible significant environmental impact; monetary loss equal to or exceeding $1 million but less than $10 million; or major system damage.

Marginal	**3**	Could result in one or more of the following: injury or occupational illness resulting in one or more lost work day(s); reversible moderate environmental impact; or monetary loss equal to or exceeding $100K but less than $1 million; or minor system damage requiring repair.
Negligible	**1**	Could result in one or more of the following: injury or occupational illness not resulting in a lost work day; minimal environmental impact; monetary loss less than $100K; or minor system damage not requiring repair.

3.3.4. Risk Assessment and Reduction

These dimensions and scoring categories are then related. For example, frequency and severity combine in the following risk matrix:

		Frequency					Key (SxF)	
		Almost Certain	**Probable**	**Moderate Occasional**	**Remote**	**Extremely Remote**	**Risk**	**Value**
Severity	**Catastrophic**	High	High	High	Moderate	Moderate	**High**	>50
	Critical	High	High	Moderate	Moderate	Low	**Moderate**	16 to 50
	Substantial	High	Moderate	Moderate	Low	Low	**Low**	7 to 15
	Marginal	Moderate	Moderate	Low	Low	Routine	**Routine**	<7
	Negligible	Moderate	Low	Low	Routine	Routine		

The detection dimension adds an additional layer of complexity to the overall assessment of risk. The three dimensions can be multiplied together (detection x frequency x severity) to achieve an overall risk score. However, a risk level approaching 729 is clearly unacceptable for any project. Therefore, the above frequency vs. severity matrix should be used to determine the minimum level of detection abilities in place for a given safety vulnerability. For example, a "high risk" safety vulnerability **must** have "Almost Certain" detection and control methods, but a "moderate" safety vulnerability may be able to properly manage risk using only "Moderate/Occasional" detection and control systems. Better detection and control systems are always desired, but may not always be possible or feasible. Thus, the risk assessments below only multiply the severity and frequency of detection dimensions.

The following failure modes and assessment matrix will document the system, ISV, assess risk, detection and mitigation strategy, and failure strategy within a single efficient form.

The following sub-systems are defined for CHEF in a failure type method separate from the above HAZOP:

Vacuum: includes the vacuum pumps, gauges, vacuum vessel, and leak detector.

Cryocooler: includes the cryocooler, compressor, cooling water pump and lines.

Fluid Piping: includes all hydrogen handling containers, valves, and lines.

Electrical: includes the computer, controllers, and any powered components.

Laboratory: includes all lab-wide systems such as building power and alarms.

Mitigation, detection, and failure strategies should either be self-explanatory or defined in detail in the operation procedures portion of this report and simply referred to by number in the matrix.

The matrix is given on the following pages.

System	Safety Vulnerability	S	F	Risk	D	Mitigation Strategy:	Detection Strategy:	Failure Strategy:
Piping	Blockage in a line	5	3	Low	5	Always order high purity gasses. Always purge lines at least two full times according to 4.2.4. Keep positive pressure at all times.	Pressure not relieving in cryostat or liquefier pressure reading below ambient (indicative of a frozen nitrogen/oxygen at inlets of liquefier tanks)	Depends on location of blockage. Use heaters to determine location of blockage. If appropriate, vent hydrogen/helium in the lines through Alicat flow meters and/or dedicated vent line, and/or fill line to determine the location of the blockage. Purge the system with inert gas. Warm up the system slowly with warm up procedure.
Piping	Damage to fill port threads	1	7	Low	1	Follow proper compression and VCR piping procedures	Visually inspect threads before each fill	In the event that the connection is not tight, immediately stop filling and replace the damaged fitting(s).
Piping	Vacuum level increases slightly upon refill due to small leak in the piping	3	9	Mod	1	Follow proper piping techniques and utilize orbital TIG welder where possible. Tune needle valve on liquefier to mitigate sudden thermal and pressure shocks.	Monitor turbomolecular pump vacuum gauge before and after each fill.	Determine the order of magnitude of the leak and determine if the slight increase in vacuum is due to thermal phenomena. If confirmed, follow warm up procedure and conduct thorough leak check to identify and fix problem areas.
Piping	Line breach	5	5	Mod	1	Follow proper piping techniques and utilize orbital TIG welder where possible.	Monitor manual line pressure gauge while filling.	Immediately stop the fill process by shutting off reservoir tank. Hy-Alerta hydrogen sensor will activate the emergency abort if breach

						Pressure relief valve on chamber will relieve pressure along with vacuum pumps.	Calibrate Hy-Alerta sensor at the manufacture's recommended interval	is external to the vacuum chamber. Once chamber and line pressures have equilibrated to a steady state vacuum level follow warm up procedure.
Piping	Over pressurization - Lines	5	3	Low	5	Pressure relief on all lines and pressure vessels. Use of gas shroud to direct sub flammability limit gases upwards towards detection sensor and away from personnel.	Manual and electric pressure gauges. Visual and audible.	Pressure relief should relieve pressure in the lines and vent to relief line. Immediately stop the flow of gas that caused the issue and fix the problem. Follow the warm up procedures if needed.
Piping	Gas Regulator Failure	5	5	Mod	7	Replace regulators every 10 years. Only use H2 compatible regulators labeled for the use. Do not modify regulators without approval. Always use a pressure relief. Avoid resonance while filling and reduce flow if necessary to exit resonance regime.	Visual, always inspect a regulator before use.	If safe, close bottle valve immediately. Otherwise, the entirety of the bottle will vent out the vent line via the 145 psig pressure relief (or 100 psig if fuel cell regulator). If hydrogen is venting into lab, pull the fire alarm. Else, gas detection system will enable auto shutdown of the gas room supply.
Lab	Building power goes out	3	9	Mod	1	Practice power-outage procedure annually	Visual, text notification, email from fuel cell inverter	Follow power-outage procedure. The fuel cell backup unit will provide power for safety systems and vacuum systems.
Lab	Building requires evacuation	1	5	Routine	1	Practice evacuation procedure annually	Visual, audible	Follow emergency abort procedure

Lab	Building HVAC system stops working	1	3	Low	3	Evacuate	Audible alarm from fume hood.	If power is still on, maintain operation of the vacuum system. Follow emergency abort procedure.
Lab/Vacuum	Line breach while HVAC system is not working	5	3	Low	3	Follow proper line construction techniques and cryogenic testing procedures.	Audible and visual from line pressure readout on manual gauge.	Follow emergency abort procedure. If occurs in off hours Hy-Alerta will trigger the EMERGENCY STOP to passivate hydrogen through outside vent line.
Electric	CHEF computer crashes	1	7	Routine	1	Keep computer updated, only work on your computer with approved programs.	Visual	Restart the computer. Use instrument loggers or manual data loggers to complete run. Else follow warmup procedure.
Electric	Temperature sensor disconnection	1	7	Routine	1	Follow proper temperature sensor build procedure, maintain clean wire provision, ensure secure connections	Visual from Lakeshore	Follow warm up procedure to fix the sensor if the sensor is critical to the experiment run.
Electric	Flow control needle valve malfunctions	3	1	Routine	7	Follow instructions to reconnect with the needle valves	Visual from flowrate measurements	Pressure relief valves will prevent the Alicat flowmeters from going beyond their MAWP.
Electric	Flow control of fill flow control is unresponsive	3	1	Routine	5	Follow instructions to reboot LabView. Restart flow measurement, watch for consumption rate to drop.	Visual, flowrate measurement	A full open flow controller will allow liquefaction to proceed normal until tank full capacity is reached. A closed flow controller will result in lower liquefaction rate.

Electric	Electrical short within cryostat	5	3	Low	3	Ensure all electrical leads are insulated and maintain nominal connections	Visual from Lakeshore, circuit breaker trips on heaters	Follow the warm up procedure to gain access to electrical connections and address the issue.
Electric	Electrical shock or sparking	3	7	Mod	7	Never store system in an energized state. Inspect wires annually for fraying. Check resistances before every run. Use only UL certified wiring.	Visual, resistance check	Disconnect wires, fix, and replace. Ensure the pinout document is updated after any wiring change.
Electric	Fuel Cell back-up fails to maintain battery charge	3	3	Low	3	Ensure fuel cell stacks are continually hydrated. Ensure batteries are replaced every 3 years. Check 48VDC-120VAC converter for nominal operation.	Visual, remote monitor system	Since the backflow prevention N.O. valve on the turbo will open (to spin down the turbo more quickly more so than 3-4 hours and allow minimal time in the resonance areas), air may solidify on the inside of the vacuum chamber. Shutoff cryocooler and vent remaining hydrogen.
Vacuum	Over pressurization – Vacuum chamber	3	1	Low	5	Vacuum chamber pressure relief, labeling of vacuum pump ports.	Vacuum chamber manual gauge, vacuum gauge, pressure relief.	Keep vacuums on to remove gas. Engage emergency abort procedure.
Vacuum	Vacuum pump(s) shut off / break	2	5	Low	1	Follow pump maintenance schedule, change pump oil, turn on at right vacuum levels.	Vacuum gauge	If vacuum holds, use emergency abort procedure. If not, see Sudden loss of vacuum.
Vacuum	Chamber breach	5	1	Routine	1	Inspect flange bolts, welds, and seals annually.	Visual, leak detector	Depending on if cryogen present, if not abort test, if yes, emergency abort procedure.

Vacuum	Main vacuum gauge stops	1	7	Routine	1	Never shock load the vacuum gauge with gas. Routinely calibrate the gauge once every 5 years.	Visual	Engage warm-up procedure and replace gauge.
Cryocooler	Cooling loop pump stops	1	7	Low	3	Inspect water filter monthly. Replace every 2 months.	Audible, cryocooler interrupt	Check electrical power, follow water filter replacement procedure, restart pump.
Cryocooler	Cryocooler stops	5	3	Low	3	Check that helium charge is within operating limits, check cooling loop water, clean HEX.	Helium pressure gauge, cooling loop water maintenance schedule	Restart cooling loop and cryocooler with power trip circuit reset, if the previous attempts are not successful follow emergency abort procedure.
Cryocooler	MLI shield becomes sharp	1	5	Routine	1	Always wear appropriate protective safety equipment.	Visual inspection of inner and outer shield.	Maintain fresh gloves on hands at all times and follow the procedure of building a new sharp-less MLI shield.
Cryocooler	Burst cryocooler sleeve	3	1	Routine	1	Never mechanically load the cryocooler cold finger.	Visual inspection, pressure in compressor	Replace cryocooler and system that caused it to load.

4. OPERATING PROCEDURES

There are many operations that must be performed to operate CHEF successfully. The procedures for the most routine operations are provided here: 4.2 Liquefaction Procedure, 4.3 Vortex Tube Data Run Procedure:, 4.4 Warm-Up Procedure:, and 4.5 Emergency Procedure:. Following each valve name is a bolded number in brackets (**[#]**), each valve on the pegboard is labeled with both a name and this number for clarity and ease of operation.

4.1. NOTES TO THE OPERATOR

Here are some insights from past operations:

- Resonating check valves, allow an audible confirmation of gas flow. This is another confidence mechanism which manifests itself at flowrates during the purge procedure. However, this can cause unnecessary ware on these components especially those built into the flame arrestors on the hydrogen regulators during fills. In certain operating regimes, a buzzing sound at low pressure differentials may be heard. Reduce the flowrate by untwisting the regulator main valve and slowly increasing again to find the most optimal flowrate outside of resonance.
- Be sure to follow the full purge procedure.

4.2. LIQUEFACTION PROCEDURE

4.2.1. LEAK CHECK PROCEDURE

- ☐ Use mass spectrometer with helium to check for leaks in system, following ASTM E499/E499M – 11 Test Method A:
  - ☐ With fittings properly torqued and installed with copper VCR fittings, pressurize liquefier tank to the leak check pressure of ~60 psia.
  - ☐ Close the helium bottle valve once the system is at check-pressure. This ensures that there is minimal residual helium leak into the room which can produce a rising background leak rate.
  - ☐ Sniff all fittings, welds, and solder joints with mass spectrometer by passing the sniffer probe over likely leak points. Start at the bottom of the assembly and work your way up, holding the probe on or not more than 1 mm from the surface. Do not move the probe faster than 20 mm/s.
    - ▪ Be sure to make a pass over the external pegboard, reservoir tank manifold and external heat exchanger fittings.
  - ☐ Continue sniffing in an orderly procedure from bottom to top. Mark any leaks so they can be remedied. Be aware that helium will rise, so a leak above a previously found leak may not actually exist. It is also important to be aware of the airflow in the room, as helium can be blown around the experiment and produce small "leaks" that don't exist via wafting helium clouds.
    - ☐ Conduct a cold nitrogen leak check if there is a cold leak in the system discovered by first an initial cooldown. See the following procedure for hints: https://hydrogen.wsu.edu/2017/05/27/cryo-cycling-in-place-styrofoam-cups-and-silly-putty-to-the-rescue/

- ☐ If any leaks are identified, take corrective action once system is depressurized and restart this procedure.
- ☐ Leave positive pressure helium or hydrogen (60-80 psia) in system before cooldown.
- ☐ Replace cap on inlet line after removing helium gas line.

4.2.1.1. Soapy Bottle Leak Check

- ☐ The main bottle connection to K-bottles are prone to leaks along with the main bottle valve.
- ☐ Follow the given schematic for classifying the leak:

4.2.2. Experimental Checkout

- ☐ Follow section 4.2.1 until a successful leak check is completed.
- ☐ Verify all electrical connections are nominal.
- ☐ Complete pinout checks on both D-sub connectors located on the exterior of CHEF with ohmmeter and designated CHEF breakout board.
- ☐ Ensure all electrical routes are clear from the inner stage MLI shield tie down points.
- ☐ Ensure gloves are worn to prevent oils from accumulating on the MLI material.
- ☐ Place MLI shield over experiment carefully.
- ☐ Affix MLI shield by latching the three points of contact.
- ☐ Ensure all electrical connections are working properly by checking the Lakeshore readouts, it is also acceptable to repeat the pinout check above.

4.2.3. Establish Vacuum

- ☐ Ensure the rubber seal on the bottom of the upper chamber is in nominal condition.
- ☐ If dry, place a very small amount of vacuum grease on the sealing surface.
- ☐ Clean the metal seal surface with acetone and methanol. Inspect the corresponding metal surface to make sure there are no large blemishes that would compromise the vacuum seal.
- ☐ Lower vacuum shell via controller hydraulic lift until the lid is fully in contact with the metal surface.
- ☐ Close the vent valve on top of the vacuum shell.
- ☐ Start dry scroll pump.
- ☐ The dry pump is to be used to pull down initial vacuum and will run during the entire test.
- ☐ Ensure turbomolecular pump is off and the (normally open) turbo vent valve is closed using Agilent T-plus software on the CHEF computer.
- ☐ Watch for falling system pressure as well as overall vacuum level to look for new leaks.
- ☐ When vacuum level reaches 10^{-1} Torr and still shows consistent trends, start turbomolecular pump.
- ☐ Watch vacuum level to ensure it reaches 10^{-5} Torr range (the nominal vacuum range of CHEF without cryocooler operating).

4.2.4. System Purge

It is essential to purge the internal piping of the system before turning on the cryocooler for any test/calibration. This prevents air from solidifying in the experiment and deviations as outlined in VC or HC sections of the HAZOP.

4.2.4.1. Supply line purge

- ☐ Note: Update this procedure when additional experiments are connected to the supply lines. As of Spring 2020 only CHEF is utilizing the helium and hydrogen supply.
- ☐ Utilizing the helium purge line via the gas room (ETRL 219), connect the inert regulator flex line if not already done in the gas room. Assure the regulator adjustment is in the fully backed-out "off" position.
    - NOTE: Ensure a snug tight fit but do not over tighten the CGA 580 fitting with 1-1/8" wrench
- ☐ Ensure valves [19], [20], [22], **[23], [24], [25], [26], [27]** and **[29]** are in the closed position before opening the helium bottle main valve.
- ☐ Check on the gas manifold on CHEF that valve **[8]** and **[10]** are closed.
- ☐ Ensure GH2 supply line purge [9] is closed along with reservoir purge [1] and manual main vent [30].
- ☐ Ensure valves **[15]** and **[16]** are also closed located at the inlet of the flow meters.
- ☐ Next, we are going to purge the air out of the helium supply line with helium.
    - Back in the gas closet, ensure the helium regulator twist valve **[23]** is shut at the main valve (unscrewed "backed-out" is closed) and valve **[24]** attached to the regulator is also closed.
    - Open the main helium bottle valve approximately 1 rotation.
    - With the **[24]** on the regulator closed, begin to screw in the regulator valve **[23]** until ~60 psig is showing on the bottle regulator.
    - Slowly open **[24]** on the regulator to avoid a pressure impulse by watching gauge on regulator.
        - Allow pressure to equalize to this purge pressure as the helium line is filled to 60 psi.
        - Double check to make sure **[8]** and **[10]** valves on CHEF are closed.
- ☐ First purge the helium line tubing leading to CHEF (via a sweep purge method):
    - Slowly open **[29]** to allow inert to travel to CHEF.
    - Once pressure equalizes, move to CHEF pegboard and ensure valve **[1], [8], [9], [10] [30]** are closed.
    - Open valve **[10]** then slowly open valve **[30]** for approximately 15 seconds to complete the sweep purge.
    - Close valves **[10]** then close **[30].**
    - Close valve **[29]** in the gas closet.
- ☐ Next purge the hydrogen line tubing leading to CHEF with helium gas (via a sweep method):
    - With **[29], [28]**, **[27], [26], [25]**, **[22], [20] and [19]** closed, (keep [24] open), ensure valves **[9]**, **[11]**, and **[12]** are also closed on CHEF.
        - Also, reminder - keep valve **[11]** closed and capped off during this process at the CHEF pegboard.
    - Slowly open **[27]** to allow helium to travel to the short section on the hydrogen piping side.
    - Ensure all valves on top of hydrogen cylinders are <u>closed</u>
    - Connect and tighten CGA 350 fittings to ***all*** H2 bottles
    - With **[19]** and **[20]** closed, open **[26]** to purge reservoir storage slowly and validate pressure on pressure gauge.
    - With **[27]** still open, open valve **[25]** to introduce pressure to the right cylinder bank connections.

- NOTE: Listen for cap pressurization with click noises

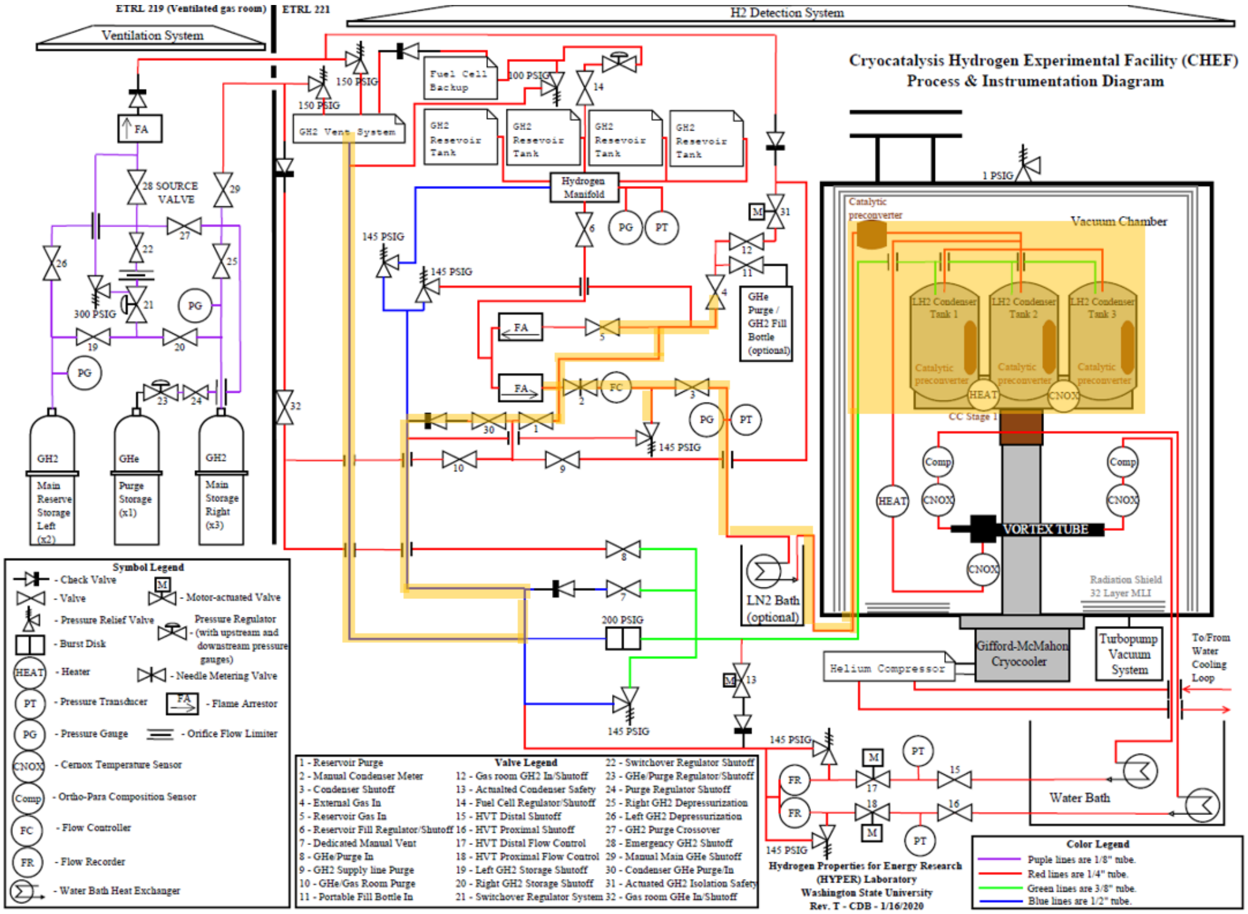
- Now shut **[24]** and we are going to do the first cycle purge in the next steps.
- Ensure valves **[9]**, **[11]**, **[12]** on CHEF are closed.
- Open valve **[28]** slowly then walk into ETRL 221 and ensure **[1], [10], [30]** are also closed.
- Open valve **[9]**, then open valve **[30]** to vent down the first cycle purge.
- Close valve **[30]**, then walk back to the 219 bottle closet.
- Open valve **[24]** gently and allow pressure to equalize.
- Close valve **[24]**.
- Walk back to CHEF and open valve **[30]** to vent down the next cycle purge.
- Close valve **[30]**, then walk back to the 219 bottle closet.
- Open valve **[24]** gently and allow pressure to equalize.
- Close valve **[24]**.
- Walk back to CHEF and open valve **[30]** to vent down the last cycle purge.
- This time, leave valve **[30]** open and walk back to the 219 closet.
- We are now going to do a sweep purge through the switchPro regulator.
- Close valve **[27]** and **[26]** as the left bank is now helium purged.
- With **[19], [20], [21],** and **[22]** closed (no flow), elevate the pressure at **[23]** to ~70 psig.
- Ensure switchover manifold arrow is pointing to the right and open [24] to allow pressure on right side tubing.
- Gently open valve **[20]** to allow pressure to enter inlet of switchover manifold.
- Begin turning up the switchover manifold regulator **[21]** to approximately 60 psig.
- Again making sure **[26]** and **[27]** are closed, gently open **[22]** to gently to begin flow out the already opened valve **[30]** on CHEF.
- Allow flow to sweep for approximately 15 seconds.
- Once 15 seconds has elapsed, shut valve **[25]** then [22] to depressurize the switchPro regulator.
- Also close [20].
- Dial valve **[23]** (helium regulator) back from ~70 psig to ~60 psig while opening [27] and venting through CHEF in the room. Close [27] once 60psig has been achieved.
- Ensure **[26]** is still closed.
- Walk into 221 and close valve **[30]**. This will leave the hydrogen vent line at +1 psig from the check valve after [30].
- This leaves us with purged 60 psig helium on the right side between valves **[29] [27]** and **[25].**
- We can now purge the helium out with hydrogen on the hydrogen supply side of the pegboard in the 219 bottle closet.
- With **[19]** and **[26]** closed, begin VERY slowly opening the main bottle valves to allow hydrogen into the left side of the manifold. If any audible leak arrised, close immediately and wait for pressure to dissipate. Verify the pressure in the tanks with the associated pressure gauge.
- With **[20]** and **[25]** also closed, open the main bottle valves on the right bank of hydrogen cylinders. Verify pressure with associated pressure gauge.
- Ensure the switchover arrow is pointing to the right (primary bank)
- Unscrew switchover regulator **[21]** such that it is closed. Ensure **[22]** is closed.
- Open valve **[20]** slowly to allow the ~1800 psig hydrogen to fill the inlet of the **[21]**.
- Open valve **[19]** to activate the reserve bank.

- With **[22]** closed, begin to increase pressure to ~60 psig via [21].
- With **[26], [27], [25]** and **[30]** closed and valve **[9]** open, proceed to slowly open **[22]**.
- Walk into 221 and open valve **[30]** for approximately 15 seconds to sweep purge the helium from the hydrogen supply line.
- Close valve **[30]** to stop the flow, then close **[9]**, then open and close [30] again to vent down remaining purge tree volume.
  - This provides a purged hydrogen stream at valve **[12]** (and **[9]**)
- Double check that valve **[10]** is shut and now we can re-open **[29]** with **[23]** at approximately 60 psig.
  - This leaves us with purged helium and hydrogen supply lines at approximately 60 psig each.
  - This a great pressure for purging the experiment and can later be increased during/for liquefaction on the hydrogen supply side.

☐ Next, we are going to purge the air in the condenser system with helium. This would be a great time to do a leak check per 4.2.1. If the leak check is acceptable, proceed to this section:

4.2.4.2. Condenser system purge

- There are multiple gas exits in CHEF. We must purge all of these routes twice to ensure < 0.25% air in the system. We will evacuate each route as described below.
  - Note: install the needle valves that are anticipated for the test before this purge is completed.
  - Evacuation #1:

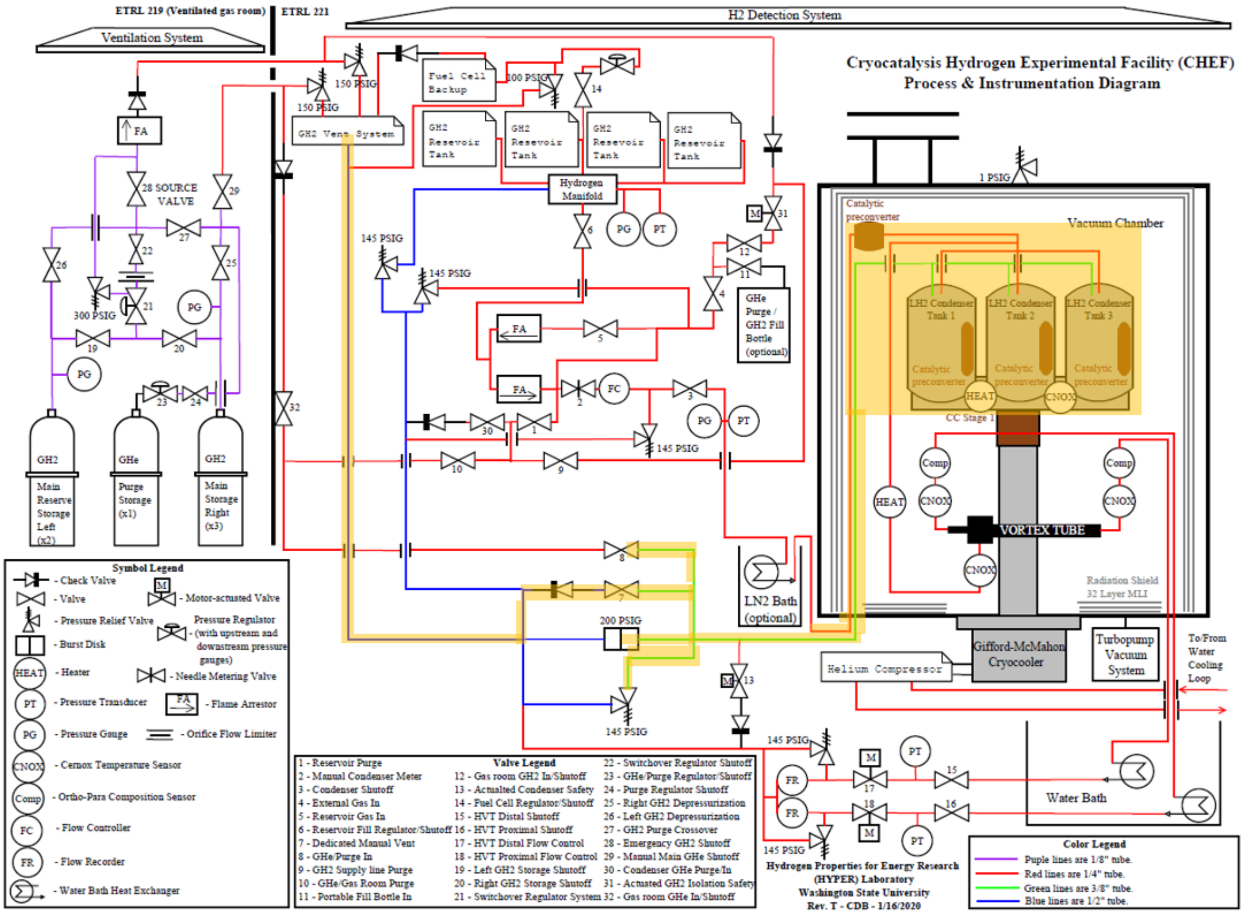


- ☐ Ensure valves **[10]**, **[30]**, **[1]**, **[7]** and **[8]** are still closed. And that both **[15]** and **[16]** are closed.
- ☐ Ensure purge line is at purge pressure (~60 psig) by checking 4.2.4.1 procedure.
- ☐ Now ensure valves **[2]**, **[3]**, **[4]**, and **[5]** are closed.
- ☐ Open valve **[10]**, then **[1]**, then **[3]** at a slow pace to avoid pressure shocks to the system.
- ☐ Adjust meter valve **[2]** to gradually bring up pressure in the condenser system.
- ☐ Allow pressure to equalize in condenser tanks to the purge pressure.
- ☐ Next, close **[10]**, and gently open **[30]** to relieve pressure in the tanks to the vent line.

    - NOTE: You may hear a chatter from the check valve downstream from this valve. This is normal.

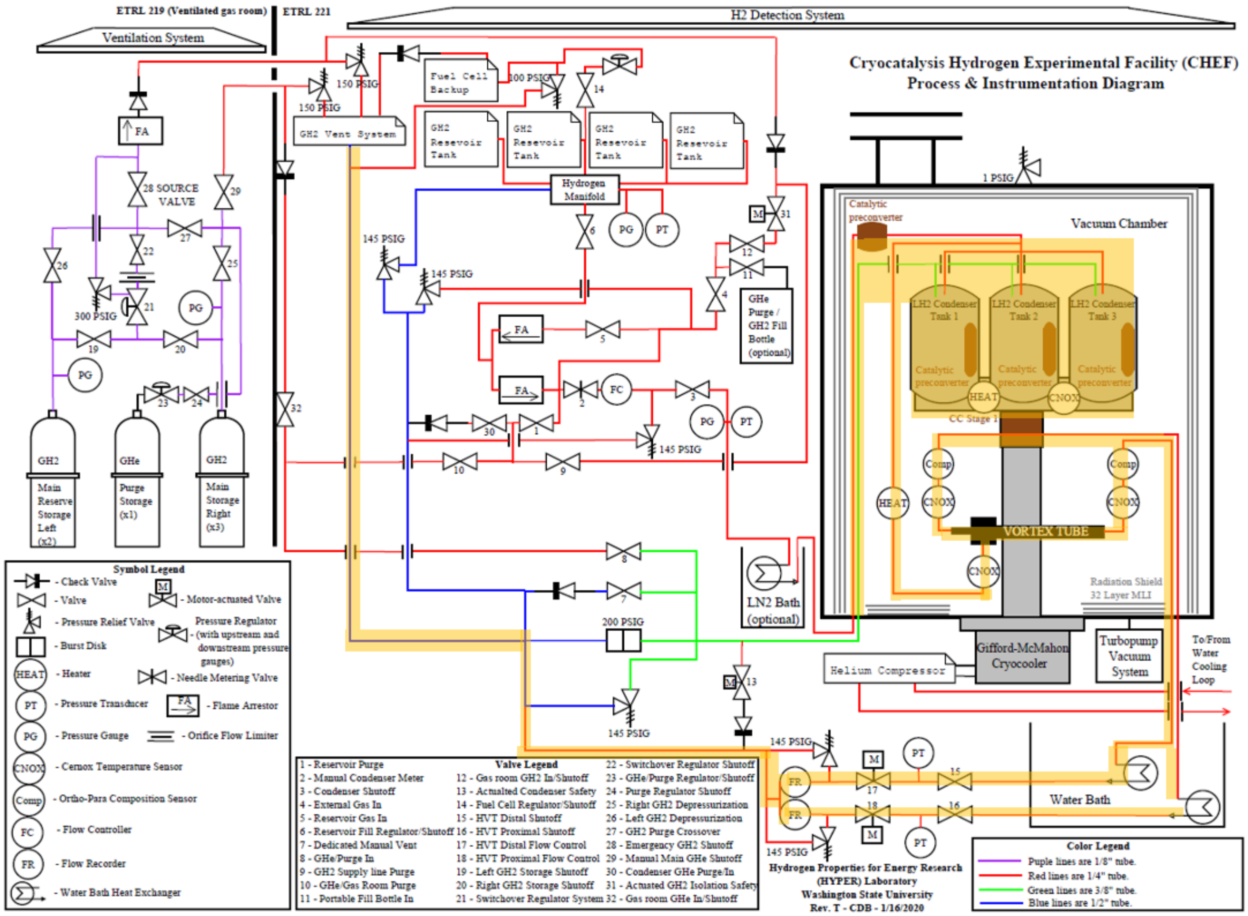
  - After the audible sound of gas whooshing past the valve ceases, close purge valve **[30]**.
  - Repeat the previous purge another time by opening (and once equalized) closing **[10]**, then vent the condenser by keeping **[10]** closed and opening **[30]**.

    - Evacuation #2:



- ☐ With valve **[10], [3]** (and **[15], [16]**) closed, now open valve **[8]** slowly to equalize pressure in the condenser tanks.
- ☐ Close valve **[8]**, Open valve **[7]** to relieve pressure in the tanks to the vent line.
    - NOTE: You may hear a chatter from the check valve downstream from this valve. This is normal.
  - After the audible sound of gas whooshing past the valve ceases, close purge valve **[7]**.
  - Repeat the previous purge another time by opening (and once equalized) closing **[8]**, then vent the condenser by keeping **[8]** closed and opening **[7]**.

    - Evacuation #3:



- ☐ Lastly, to purge the downstream tubing of the vortex tube, fill the tanks once again with helium to the desired purge pressure via the dedicated supply line by ensuring **[3]** is closed, leaving **[10]** closed, and opening **[8]** slowly.
  - While pressure equalizes, open the LabVIEW program and ensure that the program is reading correct flowrates (0 SLPM) and the actuated valves **[17]** and **[18]** are shut
  - Open **[15]** and **[16]** ball valves slowly.
  - When ready to begin purge, set **[8]** to allow 5 SLPM helium flow through each vortex tube outlet lines and increasing alicat setpoints.
    - NOTE: Depending on whether this is a purge for a calibration (no extended time needed beyond one minute) and a catalyzed run (potentially longer purge to allow for catalyst activation) the purge time will vary. Once complete, the ball valves can be closed then a "0" (then enter to send the command) can be placed in the LabVIEW program to relieve the pressure between the ball valves and needle valves.

  - Once the desired purge time has been met, valve **[8]** can be closed.
  - The upstream ball valves **[15]** and **[16]** can be closed.
- ☐ Refill the tanks with helium to obtain a pressure of approximately 60-80psia in the tanks with the hydrogen reservoir closed off to avoid sub-atmospheric pressure conditions in the tanks during cooldown.

- ☐ With **[8]**, **[1], [9]** and **[10]** closed, open valve **[30]** to depressurize small helium section.
- ☐ Then close valve **[30]** to relieve the pressure down to the 1 psi level that the check valve holds.

4.2.4.3. Optional Reservoir Purge

(Completed only post impurity event)

- ☐ First step is to complete a leak check (section 4.2.1) of the fittings on the pegboard (and internal to CHEF) to ensure no air will be pulled into the system upon pulling vacuum.
    - Note: k-bottle tank connections need to be checked also.
- ☐ To start the purge procedure, check that the reservoir is isolated (valve **[6]** closed).
- ☐ Next, prepare to vent the reservoir to the vent system by closing valves **[3, 4, 5]**.
- ☐ Open valve **[6]**, then **[1]** and **[31]** to start venting the remaining reservoir gas to the vent line.
- ☐ Once at atmospheric pressure, close **[6]**, **[1],** and **[12]**.
- ☐ With valve **[4]** and **[12]** still closed, attach a roughing pump (dry pump) to valve **[11]** and configure the helium line to have 60-80psia via opening (and purging the line per above) via the **[29]** valve.
    - Note: A KF to compression adapter is needed to connect to the inlet of vacuum pump and a union may be needed to connect the outlet vacuum pump hose to the vent system.
- ☐ Once this is complete, ensure valves **[30]**, **[3]**, **[4]**, **[5]** are shut,
- ☐ Once a confident connection is made at the inlet of the vacuum pump to compression fitting ball valve **[11]**, and the helium line has purge pressure backing the **[10]** valve, turn on the vacuum pump and open valve **[11]** to evacuate air out of the line leading to valve **[4]** and **[12]**.
- ☐ During this initial pump down upstream of valve **[4]**, ensure valves **[3, 1, 6, 5]** are closed.
- ☐ Once initial pump down of lines upstream of **[4]** is complete CAREFULLY crack valve **[4]** to begin letting the atmospheric gas in the lines into the vacuum pump.
- ☐ Allow at least 1 minute of pump down of the intermediary lines after full communication (valve **[4]** is full open) is achieved.
    - Note: Per CGA S1.1 Pressure Relief Device Standards (part I), section 6.8.1.8 it is a part of the pressure cycle test to pull vacuum on the relief device. Just as long as no more than 10 cycles/minute are performed. The same is true for the burst disk (though it is not needed in this procedure).
- ☐ Once the intermediary lines are pumped down, CAREFULLY open valve **[6]** to allow the pump down of the reservoir tanks.
    - Also, if the internals of CHEF (condenser tanks) also need a vacuum purge, then valve **[3]** can be slowly opened after valve **[6]** has achieved full communication with the condenser tanks.
- ☐ Allow sufficient time for the pump down to occur of the reservoir tanks (and CHEF).
    - Note this may take several minutes depending on the size of the roughing/dry pump.

- ☐ Once the LabVIEW pressure readout of the reservoir has indicated a steady state vacuum and the audible sound of the roughing pump is similar to when the intermediary lines were fully pumped down, close valve **[11]**.
- ☐ With valve **[11]** closed, open valve **[10]** to slowly begin introducing helium into the reservoir tanks (and CHEF, if needed via **[3]**).
- ☐ Once a 60 – 70 psia helium level has been attained then shut valve **[10]** and purge the volume in the reservoir tanks (and CHEF, if needed) to atmosphere via valve **[1]** and **[30]**. Close **[29]** when finished.
- ☐ Once at atmospheric pressure, close valve **[1]** and then SLOWLY open valve **[11]** to begin pulling vacuum again.
    - Note: do not introduce the vacuum pump with sudden atmospheric gas. Use audible cues to meter the valve.
- ☐ Wait until this has fully pumped down.
- ☐ Once pumped down, close valve **[4]** and valve **[11]**.
- ☐ Reintroduce helium opening **[29]** and pressurize the reservoir tanks to 60 – 70 psia again (this is the start of purge #2) with valve [10]
- ☐ Once a 60 – 70 psia helium level has been attained then shut valve **[10]** and purge the volume in the reservoir tanks to atmosphere via valve **[1]** and **[30]**.
- ☐ Close valve **[4]** and valve **[11]**.
    - Note: if the CHEF purge is also included, then <u>close valve</u> **[3]** at this time to maintain slightly above atmospheric helium in the CHEF tanks.
- ☐ Purge the short hydrogen line connection by introducing a small amount of hydrogen (20 – 30 psia) by opening valve **[12]** with **[4]** still closed.
- ☐ With valve **[4]** still closed, close **[28]**.
- ☐ Gently use valve **[11]** to pull vacuum on the hydrogen delivery lines to purge the hydrogen through the vacuum pump by slowly opening **[12]**.
- ☐ Close valve **[11]** and complete another line purge by pressurizing the hydrogen supply line to 20 – 30 psi with valve **[4]** still closed.
- ☐ Ensure **[4]** and **[28]** are closed, then with **[12]** still open, slowly open **[11]** to start pulling vacuum on hydrogen supply line.
- ☐ Once the hydrogen supply line upstream of **[4]** is once again pumped down by the vacuum pump, ensure valves **[1,3,5]** are closed.
- ☐ SLOWLY introduce vacuum on the reservoir tanks to pump all the helium out (if it is not desired) by opening **[4]**. Then open **[6]** to pull vacuum to get all helium out of reservoir tanks.
- ☐ Once pumped down, close valve **[4]** and **[11]** and open valve **[12]** to pressurize the line to approximately 20psia hydrogen.
- ☐ Now there should be a vacuum in the intermediary lines (downstream of valve **[4]**) and hydrogen on the other (20psia, upstream).
- ☐ With valves **[1]** and **[3]** still closed and **[6]** open, open valve **[4]** to introduce hydrogen into the intermediary lines.
- ☐ Open valve **[5]** slowly to begin filling the reservoir with hydrogen. Increase the regulator pressure to the amount desired in the tank for the first run.
- ☐ Once the pressure is attained, close valve **[12]**.

- ☐ Close valve **[6]** and vent intermediary lines of pressurized hydrogen by opening valve **[1]** and **[30]**.
- ☐ Close valve **[4]**, turn off the roughing pump and then reintroduce pressure to the vacuum pump by opening valve **[11]**.
- ☐ Disconnect purge roughing pump outlet from vent system.
- ☐ Determine if a helium environment or a hydrogen environment is needed during the cooldown.
  - For a catalyzed tube test, to ensure maximum activation, leave helium in the system during initial cooldown.
  - For a non-catalyzed test, and if there is absolutely no helium allowed in the system during the liquefaction, it is wise to use hydrogen as the cooldown gas. Otherwise, the hydrogen can be filled directly into the non-condensable helium environment.

4.2.5. Initial Hydrogen Introduction

☐ If the system is leak tight via section 4.2.1 and investigation of the vacuum level is acceptable for room temperatures, begin hydrogen fill process.
  - Note: this can be done at room temperature or when cold depending on catalyst requirements.

- ☐ When working with hydrogen, wear flame resistant lab coat and face shield. These are the blue lab coats hanging on the laboratory coat hook and shields in the basket above the sink.
- ☐ Ensure hydrogen bottles are connected and source valve is open (replace if below/at 180 psi, see section 4.2.6.1)
- ☐ First, purge the hydrogen line (of helium) that leads to CHEF from the gas room (see 4.2.4.1 for supply line purge details)
- ☐ Assuming the condenser system is currently purged with helium, either remove the helium from the condenser tanks via **[7]** or leave it in the system depending on requirements.
- ☐ Ensure valve **[11], [4], [5], [2]** and **[1]** are closed
- ☐ Open **[12]** to allow equilibration.
- ☐ Close **[12]**
- ☐ Ensure **[10] [9] [30]** are closed.
- ☐ Open **[1]** then **[30]** and allow first cycle purge to clear.
- ☐ Close **[30]**
- ☐ Repeat the last 5 steps two more times
- ☐ Ensure the buffer storage is purged per 4.2.4.3. Open **[5]**, then **[6]** to gain communication with the buffer storage (i.e. start filling the buffer storage with hydrogen)
  - NOTE: The check valves in the system will prevent any backflow of contaminants to the bottle closet in the event of a operational error.
- ☐ Next initiate the auto tracking fill feature in LabVIEW via the 20 SLPM Alicat.
- ☐ Then open **[3]** and begin to crack open **[2]** to allow a 20psi/minute pressure ramp rate to desired liquefaction pressure.
- ☐ Monitor vacuum level for leaks.

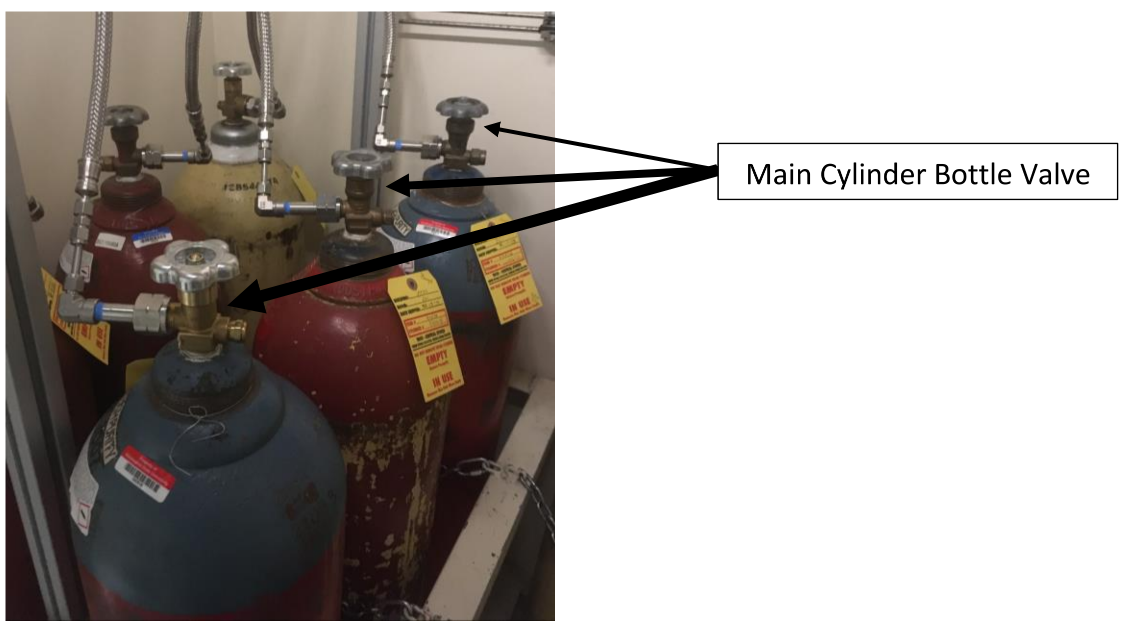
4.2.6. Hydrogen Liquefaction

- ☐ Be sure to check the indicating pressure gauges on both banks of the storage volume before liquefaction start. If below 550 psig on both banks then calculate appropriate time for replacement to avoid unnecessary increase in liquefaction time. See Section 3.2.6.1 for the bottle replacement procedure.

- ☐ Once an acceptable chamber vacuum level has been reached, and the initial hydrogen introduction completed, the cooldown process can begin.
- ☐ Start by ensuring the cooling loop is circulating and chilled. Then turn on the cryocooler.
- ☐ The backup power fuel cell unit can be enabled in "Remote" mode at this time.
- ☐ Once this has begun, the liquefaction pressure can be increased to 140 psig via the gas room valve **[21].**
- ☐ Overfilling is not an issue as the ambient heat leak will drive the system to an equilibrium fill level without the worry of trapping liquid volumes without pressure relief.

4.2.6.1. HYDROGEN BOTTLE REPLACEMENT

- ☐ Be sure to check the indicating pressure gauges on both banks of the bottle closet storage volume.
- ☐ Once 180 psig has been reached on the main bank (right bank), the system will automatically switch over to the reserve side (left bank). The following procedure outlines the process to replace the main bank while liquefying. Ensure others experiments are also ready to be shut from the bottle closet.
    - o If the reserve bank needs to be replaced follow this same procedure.
- ☐ Close main bottle valves on all the cylinders about to be disconnected (As shown below)



- ☐ For the main bank of cylinders (right side), ensure valves **[8], [10], [19], [20], [27], [26], [25], [22], [23]**, **[24]**, and **[29]** are all closed. The 4 bottle buffer storage locally at CHEF will enable the full shutoff of CHEF from the bottle storage during this replacement to avoid sub-atmospheric pressures in the condenser.

- ☐ Ensure the purge line is in a purged state per Section 4.2.4. If not, follow the procedure to purge the purge line per Section 4.2.4.
- ☐ With valve **[1]** closed, open valve **[10]** and **[30]**.
- ☐ To relieve/equilize residual pressure, open valve **[25]** to mix the small hydrogen volume with helium. Then open [29] to relieve pressure through chef vent.
- ☐ Visually confirm from the 219 bank pressure gauge that the pressure from the bank has relieved.
- ☐ Close **[10]** and **[30].**
- ☐ Then close **[25]** and **[29].**
- ☐ Ensure all main bottle valves of depleted cylinders are closed.
- ☐ Disconnect the CGA 350 fittings from the depleted bottles with wrench on front of bottle manifold.
- ☐ Replace depleted bottles with new bottles.
- ☐ With main bottle valves closed on the fresh bottles, carefully secure the CGA 350 fittings with 1-1/8" wrench
- ☐ Next we are going to purge the lines of the atmospheric air entrained from the main valves of the bottles to **[20]** and **[25]**.
- ☐ With **[26]** (and **[27], [29], [23]** and **[24]** still) closed, ensure the helium bottle is at a purge pressure of ~60 psig on the gauge of valve **[23].**
- ☐ Ensure that at least ~80 psig is in the helium bottle. If not, depressurize and replace.
- ☐ Assuming over 80 psig is in the helium tank, with **[24]** closed, begin to dial up the pressure to ~60 psig via **[23]**.
- ☐ Ensure **[24], [25], [26], [27], [29], [8],** and **[10]** are closed.
- ☐ Open **[24]**
- ☐ Open **[25]** to pressurize the intermediary plumbing in the gas room.
- ☐ Open **[29]** to pressurize helium line into ETRL 221
- ☐ Close **[24]** after equalization
- ☐ Ensure **[1]** is closed, then open **[10]** and **[30]** to relieve 1st purge. Keep [30] open.
- ☐ Close [29].
- ☐ Open **[24]** to allow equalization.
- ☐ Close [24].
- ☐ Open [29] to depressurize.
- ☐ Repeat last 4 steps as many times to achieve level of purity needed.
- ☐ Close **[30]** and **[10]**
- ☐ Open [24] to repressurize helium line.

- ☐ Now close **[25]** (with **[20]** still closed) and open main bottle valves of newly installed cylinders.
- ☐ Inspect pressure gauge to confirm bottle opening
- ☐ Very gently, open valve **[20]** to allow switchover to switch back to main storage.
- ☐ Once purged, reintroduce the hydrogen to the liquefaction process stream by opening **[22]** and **[28].**

4.3. Vortex Tube Data Run Procedure:

4.3.1. Gaseous hydrogen test:

This procedure describes how to run a test of gaseous phase hydrogen through the vortex tube. The liquid hydrogen tanks should be close to full and the vacuum level of the chamber should be nominal.

NOTE: Before the beginning of this test, review the goal operation state points.

4.3.1.1. Part A: Setup and Pre-Check:

Here we will check all major functions of the system before we run the test.

- ☐ Turn on TEC coolers for all spectrometers about 1.5 hours before a test. Ensure that the spectrometers are setup for correct scan intervals and functioning properly (via lights on side of case)
- ☐ Turn on both Kepco vapor and tank power supplies.
- ☐ Close **[3]** shutting off the reservoir buffer storage.
- ☐ On the Lakeshores, set the desired inlet temperature for the boil-off and temperature for the preheater and the tanks.
- ☐ Open Laser Quantum laser control and verify com ports. R1 – com4, R2 – com6, R3 – com5.
- ☐ Turn on Laser power toggles. Connect each via the RmoteApp Laser Control. Verify via temperatures that they are connected.
- ☐ Turn on Swd SpectraWiz and verify connection to the three spectrometers. Perform as many “Save dark spectrum/Zero dark” scans until a clean baseline is achieved. Note: This is without the lasers active.
- ☐ Check warmup heat exchanger water level to ensure DI water is covering the coils.
- ☐ Slowly ramp lasers to 520mW in 100 mW increments.
- ☐ Read over the entire procedure before continuing.
- ☐ Turn on data collection on the LabVIEW program via the “Test Data” toggle.

4.3.1.2. Part B: Cooldown and data collection:

Here we will start cooling down the system and taking data for the test. Once it is started, we need to collect these points efficiently, so ensure you have read and understand the entire procedure before continuing. It is highly recommended to review this testing procedure before every test. Note that at any point if the condenser temperature suddenly rises, indicating the last of the LH2 has boiled off, end the experiment by moving to Part C.

- ☐ Open **[15]**.
- ☐ Set hot end mass flow control to 5 SLPM.

- ☐ Open **[16]**.
- ☐ Set cold end mass flow control to 5 SLPM.
- ☐ Verify both Alicats are measuring the correct amount of flow out of the experiment.
- ☐ Allow a VT chill down time to conserve LH2.
- ☐ Begin to ramp boil-off heater based on desired state point.
- ☐ Manually increase the mass flow meters in the LabVIEW dashboard. Do this smoothly.
- ☐ Wait 10 minutes or until VT Inlet temperature sensor is close to the desired setpoint (ex: 40K).
- ☐ While waiting, ramp Raman lasers to desired power.
- ☐ Turn on the pre-heater PID controls, set to LOW/MED/HIGH.
- ☐ Set hot end MFC to get desired flow fraction / Pressure drop.
- ☐ Ensure boil-off heater is obtaining desired state point pressure.
- ☐ Monitor vortex tube temperature, wait for steady state conditions.
- ☐ Continue taking data through steady state (i.e. leave the data collection running).
- ☐ If there is enough liquid in the tank and another flow-fraction test is desired, adjust to new flow fraction (~ 0.4 or next parameter to be tested next).
- ☐ Wait for steady state.
- ☐ The data recording will record steady state valves automatically.
- ☐ Repeat as necessary or if there is time for another cold flow fraction point.
- ☐ Read 3.3.3 for preparation of what to do once the LH2 runs out during the test.

4.3.1.3. Part C: Test Ending:

- ☐ Immediately unpower the boil-off and preheater
- ☐ Turn laser power off and turn off spectrometer TEC.
- ☐ Begin ramping down the flow to 0 off both ends.
- ☐ If another test is to be had, then flow 4.2.6 hydrogen liquefaction. If a warm up is needed them follow 4.4.
- ☐ Close **[15]** and **[16]**.

4.3.2. Liquid hydrogen test:

This procedure describes how to run a test of liquid phase hydrogen through the vortex tube. It mirrors 3.3.1 through 3.3.3 however, gaseous helium is used via valve [8] to provide pressurization. Ensure that the long fill tubes are installed in the condenser tanks for liquid collection and thus a liquid test.

4.3.2.1. Part A: Setup and Pre-Check:

Here we will check all major functions of the system before we run the test.

- ☐ Turn on data collection on the LabVIEW program.
- ☐ Close **[3]** shutting off the reservoir buffer storage.
- ☐ Purge the helium purge line in accordance to 3.2.4 if not already done so.
- ☐ Ensure **[10]** is closed and a low pressure (2-3 psig) is available at the inlet of **[8]** via **[23]** gauges.
- ☐ Read over the entire procedure before continuing.

4.3.2.2. Part B: Cooldown and data collection:

Here we will start cooling down the system and taking data for the test. Once it is started, we need to collect these points efficiently, so ensure you have read and understand the entire procedure before continuing. It is highly recommended to review this testing procedure before every test. Note that at any point if the condenser temperature suddenly rises, indicating the last of the LH2 has been evacuated, end the experiment by moving to Part C.

- ☐ Open **[15]**.
- ☐ Set hot end mass flow control to 5 SLPM.
- ☐ Open **[16]**.
- ☐ Set cold end mass flow control to 5 SLPM.
- ☐ Verify both Alicats are measuring the correct amount of flow out of the experiment.
- ☐ Begin to slowly introduce helium through **[8]** and increase pressure until a 5 SLPM flow is had and the liquid hydrogen begins to chill the inlet line and vortex tube.
- ☐ Ensure Raman data is being collected.
- ☐ Allow a VT chill down time to conserve LH2.
- ☐ Begin to ramp helium pressure via **[23]** to desired state point.
- ☐ Manually increase the mass flow meters in the LabVIEW dashboard. Do this smoothly.
- ☐ Wait 10 minutes or until VT Inlet temperature sensor is close to the desired setpoint (ex: 40K).
- ☐ While waiting, ramp Raman lasers to desired power.
- ☐ Set hot end MFC to get desired flow fraction / Pressure drop.
- ☐ Monitor vortex tube temperature, wait for steady state conditions.
- ☐ Continue taking data through steady state (i.e. leave the data collection running).
- ☐ If there is enough liquid in the tank and another flow-fraction test is desired, adjust to new flow fraction (~ 0.4 or next parameter to be tested next).
- ☐ Wait for steady state.
- ☐ The data recording will record steady state valves automatically.
- ☐ Repeat as necessary or if there is time for another cold flow fraction point.
- ☐ Read 3.3.3 for preparation of what to do once the LH2 runs out during the test.

4.3.2.3. Part C: Test Ending:

- ☐ Once tank temperature increases rapidly from warm helium flow, shut off valve **[24].**
- ☐ Turn laser power off.
- ☐ Begin ramping down the flow to 0 off both ends.
- ☐ If another test is to be had, then flow 3.2.6 hydrogen liquefaction. If a warm up is needed them follow 3.4
- ☐ Close the **[15]** and **[16]**.

4.4. Warm-Up Procedure:

- ☐ Open condenser vent **[7]** and once pressure is below 140psig.
- ☐ Leave **[7]** open until liquid is gone (if any).
- ☐ Manually shut off the cryocooler.
- ☐ If no one else has a cryocooler on, you can turn off the water cooling loop and the building water supply.
- ☐ Reminder: Do not shut off the cooling water if another cryostat is running.

- ☐ Vaporize remaining liquid hydrogen by ramping condenser heater until temperature increase of 0.5 K is observed on the temperature monitor at the liquefier tanks.
- ☐ Note: If there is a cold leak in the system: Use the warm up period to diagnose the location of the cold leak. If the vacuum level improves right as the tank warms up then it is most likely located near the tank. If the vacuum level improves further out in a warm up then its likely the leak originated in the tubing.
- ☐ Allow everything to warm up to room temperature.
- ☐ Monitor the temperature as CHEF warms up, and turn off the heater if temperatures rise above 300K.
- ☐ Keep **[12]** and **[6]** off and lock out tag out to prevent accidental introduction of hydrogen in to the system.
- ☐ Purge the intermediary pressure by ensuring **[10]** is closed and opening **[1]** and **[30].**
- ☐ Open **[3]** while **[1]** and **[30]** are open to purge remaining hydrogen in the condenser tanks.
- ☐ Ensure **[10]** is closed and complete a helium line purge if not already done so.
- ☐ Close valve **[30],** open valve **[1]** slowly to begin letting in helium to the intermediary manifold area by opening **[10].**
- ☐ Open **[2]** and **[3]** to allow a purge pressure to build in the condenser tanks.
- ☐ Shut **[3],** purge contents out **[7].**
- ☐ Do the last 3 box steps (one more time) for a total of 2 purges.
- ☐ When lowest temperature reading is above 273 Kelvin, you may shut off the turbo but leave the roughing pump running.
- ☐ Once the turbo has spooled to 0 rpm, turn off the roughing pump. At this point, use extreme caution to gently open the vacuum relief valve on the top of the vacuum chamber. Monitor the digital readout to maintain at very slow increase in vacuum level up to the 3e-03 vacuum level. Any vacuum above this is measured through a sensitive Pirani gauge. Once the vacuum is in the single torr range the vacuum release can be increased. Achieve audible air flowing into the experiment. Leave this valve open until ready to run again in order to allow trapped helium in the shell to escape.
- ☐ Leave CHEF's vacuum chamber closed when not in use to keep it clean and prevent accidental damage.
- ☐ Place the red protective ring around the metal sealing surface to prevent unnecessary wear.

4.5. EMERGENCY PROCEDURE:

4.5.1. IN CASE OF POWER OUTAGE:

The system is completely autonomous and will passivate itself without any intervention. The fuel cell backup system will maintain power for up to 2 hours when the closet supply is shut off in the event of an emergency abort, H2 sensor alarm, or power outage. This will provide power to the Hanbay electronic actuators, Alicat flow meters, Lakeshore temperature controllers, data acquisition board, CHEF computer, vacuum dry scroll pump, turbomolecular pump, monitor screen, and pressure transducers. Preserve the liquefaction progress by following these recommendations:

Do not activate the EMERGENCY STOP unless you perceive a risk to equipment and personnel. In most cases, an EMERGENCY STOP is not necessary unless it is believed that hydrogen is leaking into the room.

The primary pressure relief system will activate at 145 psig while liquefying to maintain pressure regardless of the state of the fuel cell backup.

All the hydrogen will vent out the roof vent line.

If you believe hydrogen is leaking into the room, or you believe any personnel are in immediate danger, follow the emergency abort procedures below.

4.5.2. In Case of Hydrogen Leak into the Room:

Follow the emergency abort procedures below by pressing the EMERGENCY STOP button. Evacuate all personnel immediately and pull the fire alarm.

4.5.3. In Case of Hydrogen Leak into the Vacuum Chamber

In the event that hydrogen leaks into the vacuum chamber, it should be immediately visible by an increase in vacuum chamber pressure. In most cases, this leak will be small enough that the vacuum will suck any hydrogen out and vent into the hood system. The system can be safed by venting all hydrogen and purging with helium, then following the standard warm up procedure. If vacuum pressure rises above 5×10^{-2} Torr, you believe hydrogen is leaking into the room, or you believe any personnel are in immediate danger, follow the emergency abort procedures below.

4.5.4. **EMERGENCY ABORT:**

Press the red illuminated EMERGENCY STOP button located near the Lakeshore temperature monitors.

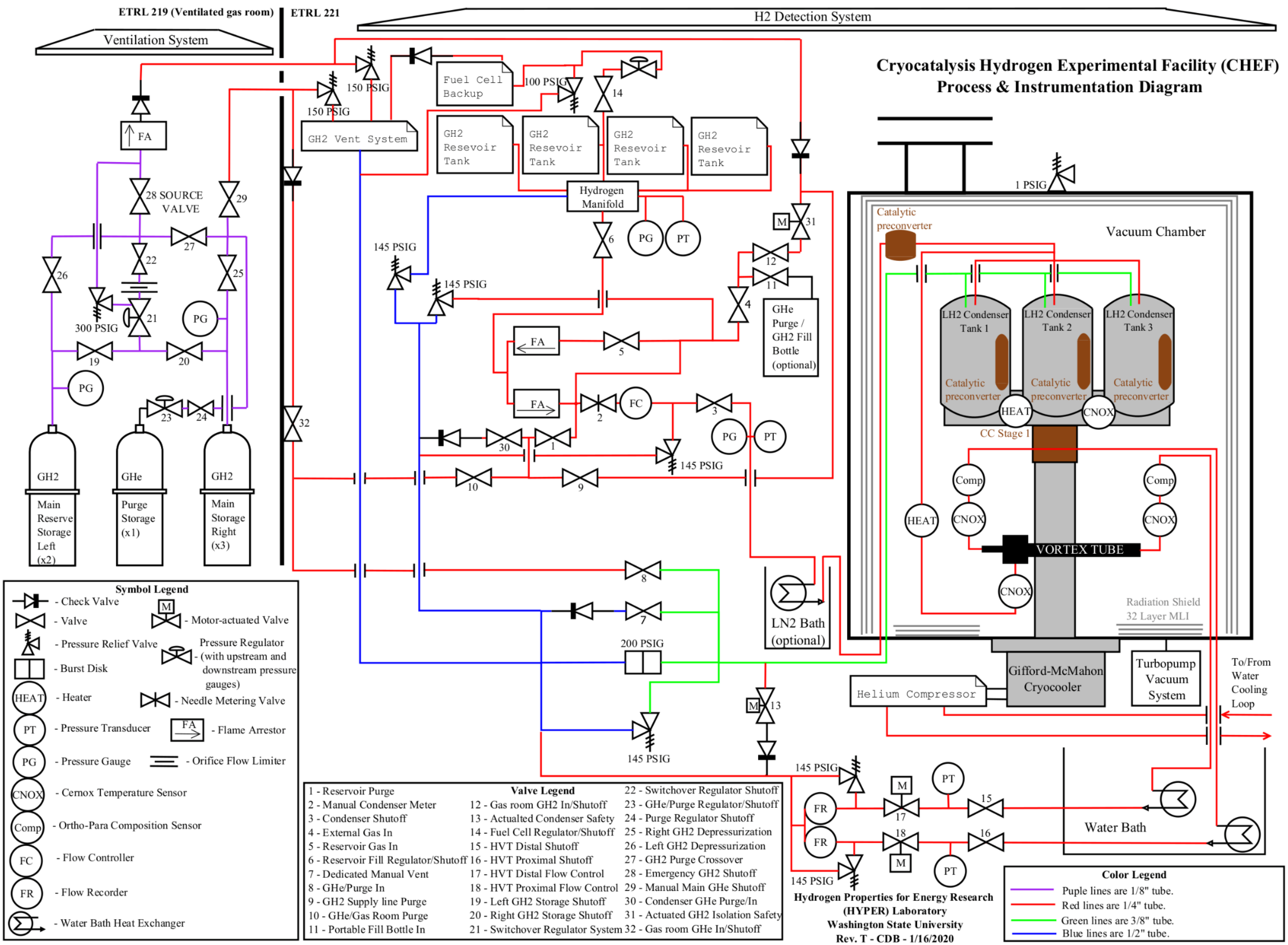
This will vent all hydrogen in the system via autonomous actuation of [13] and isolate the storage volume via [31] to prevent any additional storage losses.

5. Project Safety Documentation:

The documentation for this project is maintained in the HYPER Lab controlled ITAR compliant file storage system (HYPERDRIVE). All project personnel have access to this folder where the most recent safety plan (and archived safety plans) is kept. Process and Instrumentation Diagram, maximum material inventory, pressure, temperature, flowrate, electrical, materials construction, pressure relief, and ventilation aspects are included in this section.

5.1. Process and Instrumentation Diagram (P&ID)

Please see the following page for the most current P&ID diagram.


ETRL 219 (Ventilated gas room)
ETRL 221
Ventilation System
H2 Detection System
Cryocatalysis Hydrogen Experimental Facility (CHEF)
Process & Instrumentation Diagram
Fuel Cell Backup
GH2 Vent System
GH2 Resevoir Tank
GH2 Resevoir Tank
GH2 Resevoir Tank
GH2 Resevoir Tank
Hydrogen Manifold
150 PSIG
150 PSIG
100 PSIG
145 PSIG
145 PSIG
145 PSIG
300 PSIG
200 PSIG
1 PSIG
28 SOURCE VALVE
GHe Purge / GH2 Fill Bottle (optional)
GH2 Main Reserve Storage Left (x2)
GHe Purge Storage (x1)
GH2 Main Storage Right (x3)
Vacuum Chamber
Catalytic preconverter
LH2 Condenser Tank 1
LH2 Condenser Tank 2
LH2 Condenser Tank 3
CC Stage 1
VORTEX TUBE
Radiation Shield 32 Layer MLI
Gifford-McMahon Cryocooler
Turbopump Vacuum System
Helium Compressor
To/From Water Cooling Loop
LN2 Bath (optional)
Water Bath
Symbol Legend
- Check Valve
- Valve
- Motor-actuated Valve
- Pressure Relief Valve
- Pressure Regulator (with upstream and downstream pressure gauges)
- Burst Disk
- Heater
- Needle Metering Valve
- Pressure Transducer
- Flame Arrestor
- Pressure Gauge
- Orifice Flow Limiter
- Cernox Temperature Sensor
- Ortho-Para Composition Sensor
- Flow Controller
- Flow Recorder
- Water Bath Heat Exchanger
Valve Legend
1 - Reservoir Purge
2 - Manual Condenser Meter
3 - Condenser Shutoff
4 - External Gas In
5 - Reservoir Gas In
6 - Reservoir Fill Regulator/Shutoff
7 - Dedicated Manual Vent
8 - GHe/Purge In
9 - GH2 Supply line Purge
10 - GHe/Gas Room Purge
11 - Portable Fill Bottle In
12 - Gas room GH2 In/Shutoff
13 - Actuated Condenser Safety
14 - Fuel Cell Regulator/Shutoff
15 - HVT Distal Shutoff
16 - HVT Proximal Shutoff
17 - HVT Distal Flow Control
18 - HVT Proximal Flow Control
19 - Left GH2 Storage Shutoff
20 - Right GH2 Storage Shutoff
21 - Switchover Regulator System
22 - Switchover Regulator Shutoff
23 - GHe/Purge Regulator/Shutoff
24 - Purge Regulator Shutoff
25 - Right GH2 Depressurization
26 - Left GH2 Depressurization
27 - GH2 Purge Crossover
28 - Emergency GH2 Shutoff
29 - Manual Main GHe Shutoff
30 - Condenser GHe Purge/In
31 - Actuated GH2 Isolation Safety
32 - Gas room GHe In/Shutoff
Hydrogen Properties for Energy Research (HYPER) Laboratory
Washington State University
Rev. T - CDB - 1/16/2020
Color Legend
Puple lines are 1/8" tube.
Red lines are 1/4" tube.
Green lines are 3/8" tube.
Blue lines are 1/2" tube.

5.2. Process Chemistry

The process chemistry in CHEF will consist of Gaseous and Liquefied Hydrogen as well as Gaseous Helium.

5.3. Maximum intended inventory of chemicals

The maximum amount of hydrogen inventory stored at one time in the gas storage room is 985 ft^3 GH_2 qualifying the amount as non-bulk GH2 storage per NFPA 3.3.161. This is also under the Maximum Allowable Quantity (MAQ) threshold for requiring special provisions for an unsprinklered gas room of 2000 ft^3 GH_2 per NFPA 2 Table 6.4.1.1.

5.4. Safe system operating limits

- The Maximum Allowable Working Pressure (MAWP) during liquefaction is 145 psig (10 bar) due to Alicat flow meters/controllers. The MAWP during tests is 1800 psig (124 bar) due to maintaining a factor of safety of 3 on the condenser tanks and availability of high accuracy absolute pressure transducers wetted with 316L stainless steel.
- The maximum temperature of the system is dictated by the indium sealing material melting point at 430K and the minimum is dictated by the cryocooler operating range of <14K.
- The maximum flowrate possible in CHEF is 594 SLPM (1259 SCFH) hydrogen if no restrictions other than the 0.004 Cv needle valve is fully open in the mass flow actuated valves. In testing conditions, this maximum flow rate would rarely be over 296 SLPM (628 SCFH) combined between the two outlets.

  Minimum flowrate is determined by the speed of the manual adjustment of ball and/or needle valves. Therefore, this could be as low as 0.02 SLPM.
- Concentrations in CHEF will either be above the Upper Flammability Limit (75%) while liquefying. Or below the Lower Flammability Limit (4%) while under maintenance. Helium purge gas will ensure the transition between these two states occurs without contamination.

See Failure Modes and Effects Analysis and HAZOP for an evaluation of the consequences of the deviations from these limits.

5.5. Materials of construction

The CHEF is constructed of primarily steel. The following details ensure the compatibility of the system with cryogenic temperatures, elevated pressures, and reduction of the effects of hydrogen embrittlement.

Non-hydrogen exposed materials

- Vacuum chamber: Stainless Steel 304

- Server rack: Powder-coated steel

Hydrogen exposed materials (low pressure < 145 psi)

- Interior components:
    - Swagelok ASME SA479/ASTM A276/ASME A182/SEMI F20-0305 Stainless Steel 316L/316LV VCR and weld fitting components
    - Swagelok Copper VCR gaskets
    - Edmund Optics UV-Fused Silica lens
    - Indium sealing material
    - Cryo-rated PTFE ball valve seals

Hydrogen exposed materials (elevated pressure < 1800 psi, during test)

- Liquefaction tanks: Stainless Steel 316L
    - Luxfer DOT approved 316L Sample cylinder with Swagelok certified welds
    - Raman composition cells: High-grade UV fused silica / Indium / Copper gasket / Stainless Steel 316L
    - Vortex Tube (v1.0): 6061 Aluminum / Indium seals / 316L Stainless Steel / Ruthenium catalyst
- Temperature probes: 316L Stainless Steel

Note, the use of 316 Stainless Steel is advantageous due to its resistance to hydrogen embrittlement (improves at cryogenic temperatures). The ductility of SS316L also maintains due to the avoidance of a glass transition phase change. There is no epoxy used in the system and all seals are either welded or indium press fit.

5.6. Electrical classification

The electrical classification of CHEF is per NFPA 55 Table 2-2.1.1 all electrical in a location where up to 2500 scf is located in a separate room is classified as class III. ETRL 219 gas room contains no electronic equipment. Electrical wiring and equipment shall be in accordance with NFPA 70 in ETRL 221 and follow electrical grounding per WAC 296-24-31505 Liquid Hydrogen Systems, and WAC 296-24-31503 Gaseous Hydrogen Systems.

5.7. Pressure relief system design and design basis

The CHEF pressure relief system features a dual stage, redundant relief scheme to ensure a build-up of pressure beyond the MAWP is not possible. The system is designed to the following standards:

- CGA S-1.1 – Pressure Relief Device Standards – Part I – Cylinders for Compressed Gases
- CGA S-1.3 – Pressure Relief Device Standards – Part III – Stationary Storage Containers for Compressed Gases

5.7.1. Primary Pressure Relief Stage Design

To verify that the relief device(s) sizing was adequate we consulted a print copy of the CGA S-1.3 standard. This code contained "commodity-based requirements" in section 4.3 which included "Liquefied compressed gases, refrigerated fluids and refrigerated (cryogenic) fluids" in 4.3.3. throughout the following calculations we assumed a worst case scenario in which the inside vacuum area of CHEF is at atmospheric pressure and hydrogen gas has filled the internal vacuum volume. Fire is not a concern for this design. Therefore, the parameters in which the valve(s) flow rate in the primary stage are specified in 4.3.3.1 of the standard:

"The minimum total flow capacity of the primary system of pressure relief valves for operational emergency conditions, except fire, shall be calculated using the applicable formula in 5.2.2 for a flow rating pressure not to exceed 110 percent and 116 percent of the MAWP of the container, respectively, for single and multiple primary pressure relief valves."

Therefore, the formula in 5.2.2 was used to determine:

> "5.2.2 - The total minimum required flow capacity for the pressure control valve(s) or primary pressure relief device(s) on insulated containers for liquefied compresses gases, refrigerated fluids, and refrigerated (cryogenic) fluids shall be calculated by the formula:"

$$Q_a = \frac{0.383*(C1-T_{5.1.3})}{(C2-T_{5.1.3})} * F * G_i * U * A$$

Where F = Correction factor specified in 5.1.4:

$$F_{5.1.4} = \sqrt{\left(\frac{Z\_i_{5.1.4}*T\_i_{5.1.4}}{Z_{5.1.3}*T_{5.1.3}}\right)} = 7.814$$

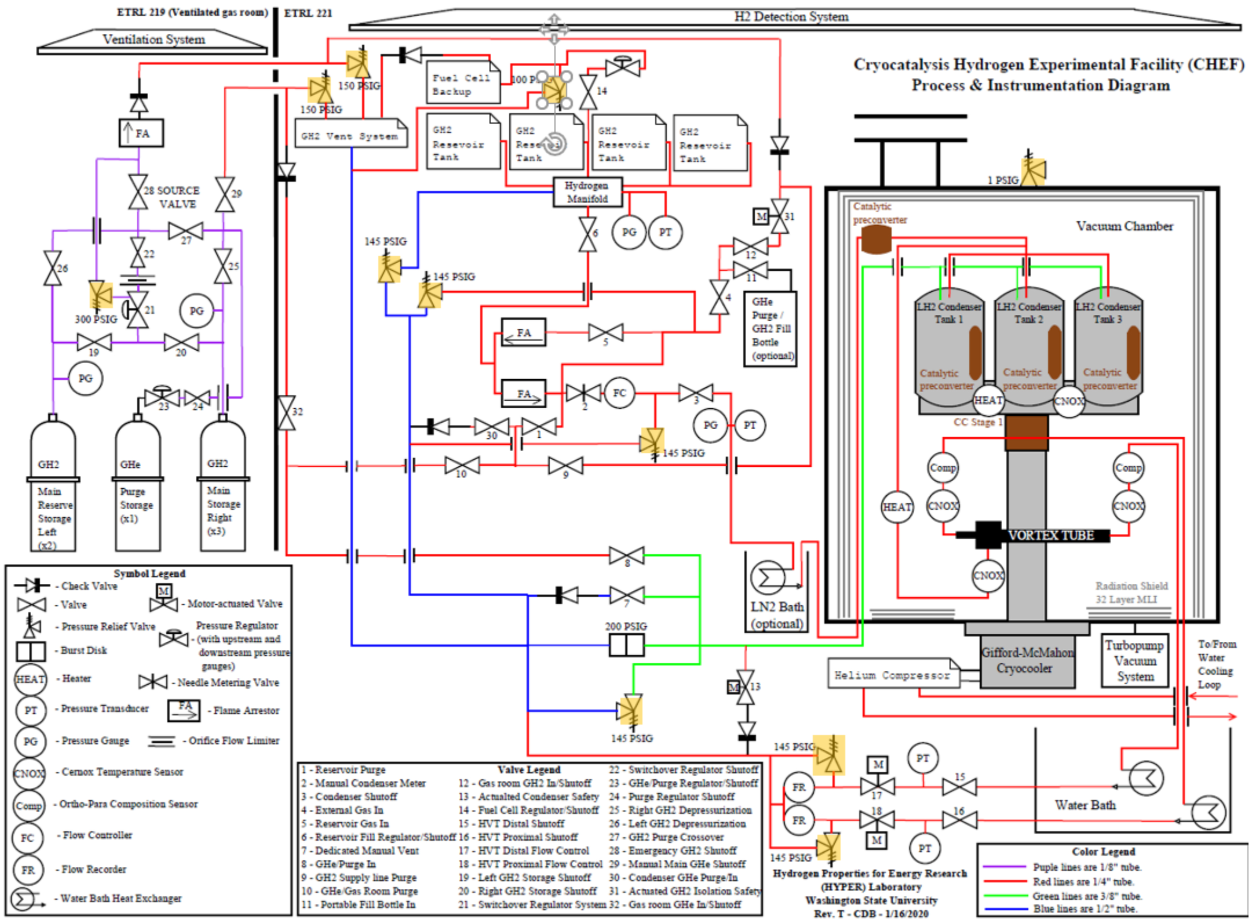
This correction factor accounts for the maximum compressibility differences at the venting conditions assuming worst case scenario (the hydrogen has warmed up to the external container conditions (293K and atmospheric pressure) to the condition for this same flow rating pressure of 145psi, but where the ratio of the specific volume to the specific heat input (at a constant pressure) multiplied by the specific volume. This occurs at 33.5K and 145psi flow rating pressure which results in a $Z_{5.1.3}$ of 0.2706 compared to the compared to that of the room temperature hydrogen which $Z_{5.1.4}$ = 1.0075.

G_I = The Gas Factor constant for hydrogen as calculated through the example problem in the code on pages 26 – 31. The example followed mirrors the metric unit example on the bottom of page 30. This value equates to U = Overall Heat Transfer coefficient in kJ/(hr*m^2 *C) assuming worst case scenario with no vacuum, hydrogen gas filled insulation space at room temperature. This is a very extreme case which in assumes steady state from the beginning of a power outage. As we saw in out anomaly with CHEF, the vacuum slowly decreased and the temperature of the metal components within the experiment also take a significant amount of time to warm. The estimated steady state overall heat transfer within CHEF during the anomaly to be 3.701 W/m^2K. While a sudden loss of vacuum is also possible we estimated through a flat plate EES estimate of an atmospheric pressure, hydrogen gas laden on the 22K liquefier exterior area. This estimate provided a heat transfer over 4-times higher at 16.31 W/m^2K.

A = Area of the outside of CHEF's liquefier: 0.2782 m^2

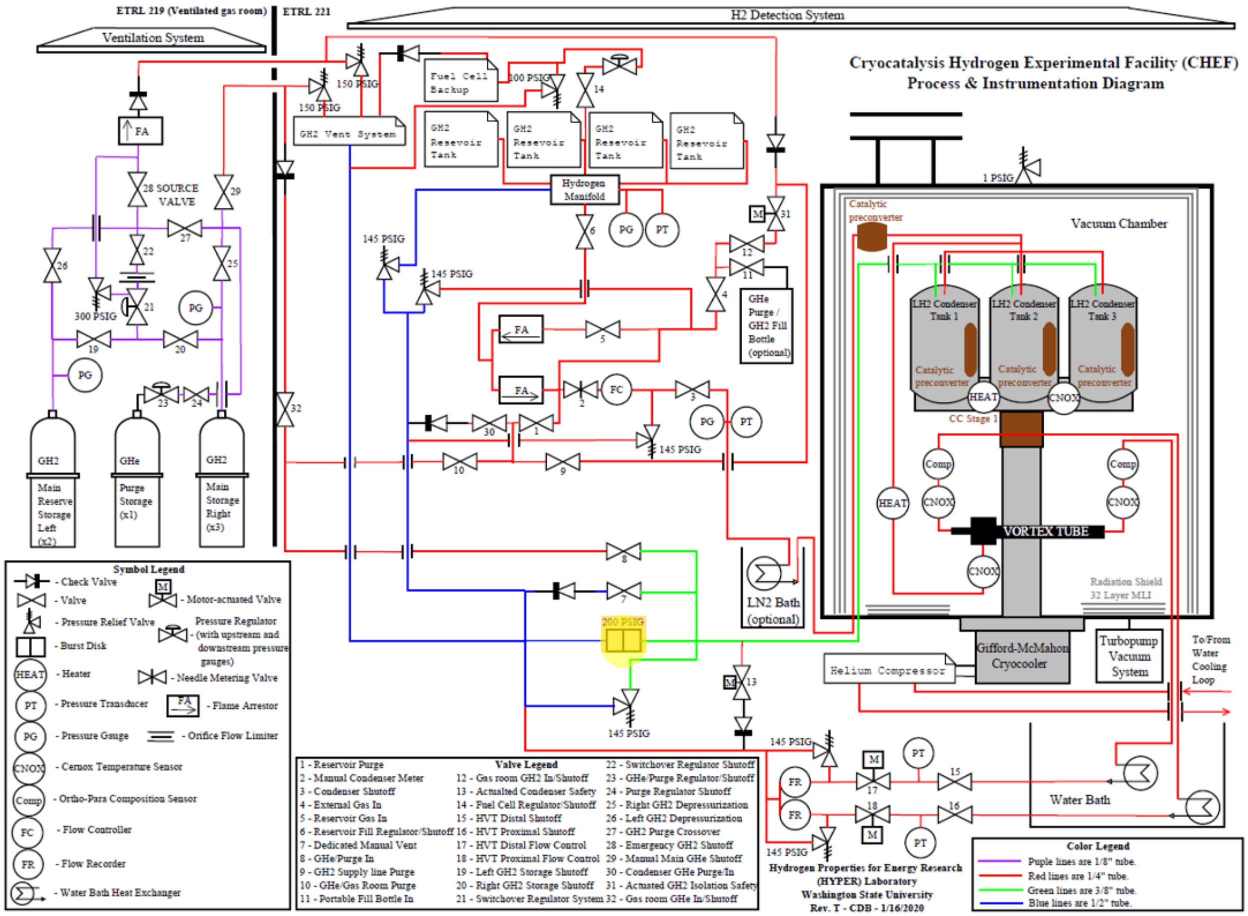
The result of these calculations was a required flow capacity of 19.73 cubic feet per minute (cfm) of air at 60 degrees F between all primary pressure relief valves.

In our system we have 4 primary relief valves to account for the liquefier shutoff valve to the liquefier. One attached to the reservoir vapor space and the other 3 in communication with the liquefier vapor space. See the example P&ID diagram below for yellow highlighted areas:



5.7.2. Secondary Pressure Relief Stage Design

The secondary stage of CHEF's pressure reliefs are aimed to act as an additional safety redundancy. The burst (rupture) disks are used in combination with the pressure relief valves to provide a last resort if the pressure in CHEF increases to 1990 psi. This value is 110% of the MAWP of the test configured system and does not require testing every 5 years. This system is highly unlikely to be utilized as the primary stage has three pressure reliefs which have a real factor of safety of 20 over a chocked flow analysis. The burst disk is manufactured by BS&B (316 Stainless) and has extensive material information included. The burst disk will be able to flow the 40.1 cfm air equivalent highlighted in yellow in the P&ID below.



5.8. Ventilation system design

The CHEF environment and non-bulk GH2 gas room storage area is ventilated at a rate compliant with NFPA 2 code section 6.17.1. The ETRL 221 room is ventilated at a rate of 487 scfm over a floor area of 490 ft^2. This narrowly eliminates the ETRL 221 space from being qualified as a gas room from a ventilation standpoint which is 1 scf/minute/ft^2 but does not affect the system architecture anyways. The dedicated ETRL 219 gas room storage is ventilated at a rate of 70.6 scfm over a floor area of 14.1 ft^2 which results in a ventilation factor of safety of 5 and is compliant with NFPA 2 Section 6.17.1.

5.9. Design codes and standards employed

The design codes and standards for the CHEF is driven primarily from the following:

- NFPA 2 Hydrogen Technologies Code – 2016: Chapters 16, 1-4, 6-8, and 12
- NFPA 55 Standard for Storage, Use, and Handling of Compressed and Liquefied Gases in Portable Cylinders
- NFPA 70 National Electric Code
- NFPA 110 and 111 Standard for Emergency and Standby Power Systems

- NFPA 79 Electrical Standard for Industrial Machinery
- CGA S-1.1 Pressure Relief Device Standards Part I – Cylinders for Compressed Gases
- CGA S-1.3 Pressure Relief Device Standards – Part 3 – Stationary Storage Containers for Compressed Gases
- CGA G-5.5 Hydrogen Vent Systems
- WAC 296-62-09005 Nonionizing radiation
- ANSI Z136.1 American National Standard for Safe Use of Lasers

5.10. MATERIAL AND ENERGY BALANCES

At maximum liquid hydrogen capacity (6.7 L), CHEF contains approximately 15.8 kWh of energy at 2.36 kWh/L. This is enough energy to move an average passenger car 46.5 miles (assuming 100% efficiency). Therefore, this safety plan will ensure this energy is dissipated in a safe manner via our direct-to-atmosphere hydrogen vent stack in ETRL 221 in the event of an emergency.

5.11. SAFETY SYSTEMS

The CHEF consists of a multilayer safety approach that minimizes risk of component and personnel risk. The following analysis will break down the specific safety layers and the code which backs up the rationale:

- Given ETRL 221 is part of the Hydrogen Properties for Energy Research (laboratory) it qualifies as a "laboratory" per NFPA 2 Section 3.3.129.
  - This qualifies this work to be subject to Chapter 16 of the NFPA 2 code "Laboratory Operations"
    - Per NFPA 2 Section 16.1.1.1 the total GH2 is greater than 75 scf (985 scf), and liquefied quantities can exceed 3.8L (6.75L).
  - The ETRL 221 space is constructed to meet the fire, ventilation, operations, and apparatus requirements as outlined in Chapter 16.
    - Per NFPA 16.1.1.3 chapters 4, 6 through 8 are applied to the design of the CHEF.
- Chapter 7: Gaseous Hydrogen
- Quantity of GH2 stored in ETRL 219 gas room:
  - Per NFPA 2 section 7.2.2.2.1 indoor gaseous hydrogen in control areas less than the maximum allowable quantity shown in Table 6.4.1.1 shall be located per provisions of Table 7.3.2.2.1.
    - Table 7.3.2.2.1 allows the 985 scf to be stored in a gas room given compliance with section 6.4.

- Per NFPA 2 section 6.4.1.5.1.2 all piping shall be in compliance of section 7.1.15.1
    - All piping systems are installed in accordance with applicable parts of ASME B31.3, Sections 704.1.2.3, 704.1.2.4, and 7041.2.5 of the ICC International Fuel gas Code (IFGC) per NFPA 2 Section 7.1.15.1.
        - Before operation all piping will be inspected and pressure tested in accordance with ASME B31.12 and ICC IFGC Section 705 per NFPA 7.1.15.1.1.
    - Back flow prevention is accounted for via the check valve attached within the gas room on the hydrogen outlet per NFPA 2 Section 7.1.15.1.5.
    - The total maximum amount of hydrogen that could be stored via the gas room bottle manifold is 985 scf. This amount of hydrogen amounts to 2.4 kg and if this were to release into the 8820 ft^3 (250 m^3) ETRL 221 suddenly then a total hydrogen percentage of 12.57% would be had. This is 314% beyond that of the Lower Flammability Limit of 4%.
        - To eliminate this risk, a orifice flow limiter is installed directly at the storage bottles in the ETRL 219 gas room to limit the possible flow into the room. With a 0.01” diameter flow limiter, the maximum flowrate into ETRL 221 is 0.4497 g/s or 12.46 cfm at 2200 psi bottle pressure.
            - If the ventilation shut off in the ETRL 221 room and there was a regulator failure on the gas room manifold, then it would vent out the dedicated pressure relief to the atmosphere on top of the ETRL 221 building. There is also a redundant pressure relief on the system while in liquefaction mode.
            - However, if the dedicated (and redundant) pressure relief failed and there was a large leak in the feed line, then it would take 28 minutes for the quantity to surpass the Lower Flammability Limit of 353 scf of hydrogen in the ETRL 221 space. This is the 5^{th} layer of failure and the likelihood of this is extremely small.
        - If the maximum allowable quantity is reached the automatic isolation valve will be triggered via the GH2 detection system per Section 6.12 of the NFPA 2 code.
        - This flow limiter does not interfere with liquefaction rates as this will be at most 0.15 cfm with the current system.
    - The piping will be cleaned and purged according to NFPA 2 Section 6.21 in the conditions in Section 6.21.1.1 are met.
    - General system design and installation are in accordance with NFPA 2 Sections 7.1.2 and 7.1.3.
    - Piping systems are to be labeled in accordance to NFPA 2 Section 7.1.6.4.1.

   - The Acopian and Lakeshore temperature controller comply with NFPA 2 Section 7.1.9.1.5.1 "Electrical heating devices shall be in accordance with NFPA 70.
   - The dedicated vent stack in ETRL 221 which vents to the exterior of the building is built in accordance to NFPA 2 Section 7.1.17 GH2 Venting Systems which is in accordance with CGA G-5.5 Hydrogen Vent Systems.
- Given the maximum amount that can stored in the gas room is below 1000 scf it does not qualify for a Hydrogen Equipment Enclosure (HEE) per NFPA 2 section 7.1.23. An HEE is defined as:

  > "A prefabricated area designed to protect hydrogen equipment that is confined by at least 3 walls, not routinely occupied, and has a total area less than 450 ft^2 (41.8 m^2)." - NFPA 2 section 3.3.145

   - The ETRL 219 gas room is 14.1 ft^2 but again does not have the ability to store more than the 1000 scf threshold, and does not contain generating or processing equipment, therefore the system does not apply to section 7.1.23 of the NFPA 2 code.
- Per section 7.1.24.1.1 and 7.1.24.2 Manual emergency shutoffs will be labeled and located at the exit of the supply ([28] on the P&ID diagram) and the point of use at the CHEF gas manifold ([12] on the P&ID diagram).
   - Note that there is no need to include an emergency valve at the entrance of the piping to the ETRL 221 space:
      - "Emergency shutoffs shall be located at the point of use and at the tank, cylinder, or bulk source, and at the point where the system piping enters the building" – NFPA 2 Section 7.1.24.2
      - Since the ETRL 221 room in not another building, there is no emergency shutoff needed at the point where the tubing enters ETRL 221. The 18' ceilings also provide physical barriers to enabling this type of shutoff. The use of one continuous line also minimizes opportunities for leaks in the delivery system.
- Per 7.1.25.3 Section of the NFPA 2 code, number (3) states "The requirements of 7.1.25 shall not be required for the following: (3) Where the source of the gas is not in excess of the quantity threshold indicated in Table 6.4.1.1"
   - Therefore, there is no need for an emergency isolation valve given the source supply (985 scf) is within the limit per NFPA 2 Section 7.2.2.2.1.
   - However, valve [31] in the P&ID diagram acts as an automated isolation valve in the event of being put in the emergency state.
- Maintenance of the system is currently compliant with NFPA 2 Section 7.1.28 and will kept this way through the redesign process.

- There are no incompatible materials requiring set back distances per NFPA Section 7.2.1.1.
- Operating Instructions as outlined in section 3 of this plan shall satisfy NFPA 2 Section 7.1.28.
- Both the ETRL 219 gas room manifold and the CHEF buffer storage tank manifold will be permanently placarded according to NFPA 2 code section 7.1.6.5.2.
- The security of the areas where hydrogen systems are maintained prevent unauthorized access or accidental dislodgement of the bottles per NFPA 2 code section 7.1.7.1 and 7.1.7.2.
- Chapter 8: Liquefied Hydrogen
    - This update to CHEF includes previously reviewed and approved design philosophy including the detailed first and second stage pressure relief design shown in section 5.7.
    - Some improvements include the following given the lower liquefaction pressure (145 psig) compared to the upper limit on test pressure (1800 psig):
        - This strategy was demonstrated via a on-off ball valve on the P&ID diagram as the controlling valve per NFPA 2 Section 8.1.4.7.2.2. However, now the pressure relief system will be swapped when going from low pressure (145 psig) to high pressure (1800 psig). Therefore there is no chance for operator error in the pressure relief system.
    - To satisfy NFPA 2 Section 8.1.4.6.1 a removable gas deflector will surround the rim of vacuum chamber lid CHEF to prevent any gas from impinging on personnel in the event of a rapid decompression.
    - All piping adheres to ANSI/ASME B31.3 Process Piping and B31.12 Hydrogen Piping and Pipelines to satisfy NFPA 2 Section 8.2.3.1.1.4.
- Chapter 12: Hydrogen Fuel Cell Power Systems
    - The incorporation of the Reli-On Independence-1000 stationary fuel cell which functions as backup power and safing in the event of a power outage is to comply with the following:
        - Per NFPA 2 Section 12.3.1.1.1 Pre-engineered fuel cell systems and matched modular components shall be designed and tested to the meet the intent of ANSI CSA FC.1 American National Standard for Fuel Cell Power Systems.
- The Raman cells in CHEF consist of embedded lasers which need to comply with WSU's laser policy.
    - Per ANSI Z136.1 necessitate Class 3B laser classification when being worked on. This includes appropriate eyewear and clothing. However, in the embedded state a lower class laser rating can be assigned.

- The lasers are equipped with a removable master key switch where the laser cannot be operated without the key per ANSI Z136.1.
- Warning sign(s) will be posted where appropriate per ANSI Z136.1.

5.12. Safety review documentation

The 2016 (12-12-2016) CHEF Safety Plan was approved via email. The following approval form is to be used for the current review:

Project Safety Plan Approval Form

DOE Award Number: 1874-1759

Project Title: Optimizing the Heisenberg Vortex Tube for Hydrogen Cooling

Organization: Washington State University

Safety Plan submitted by: C D Bunge and J W Leachman

The attached safety plan is being submitted to the U.S. Department of Energy in compliance with the Fuel Cell Technologies Program requirement under the terms of the above-referenced award. The completed approvals noted below are consistent with organization's policy for such submittals.

Project safety plan prepared by: C D Bunge / J W Leachman

Carl Bunge

Graduate Research Student

School of Mechanical and Materials Engineering

Project safety plan reviewed by:

Project safety plan approved by:

5.13. Operating procedures

See section 4 Operating Procedures for full operating procedures.

5.14. Material Safety Data Sheets (MSDS)

The Safety Data Sheets for CHEF consist of Hydrogen and Helium, and are located in Appendix A.

5.15. References

Codes and standards listed in section 5.9: “Design codes and standards employed”

6. Management of Change Procedures:

In order to make changes to CHEF, 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 the Safety Failure/Hazards matrix and the operational procedures section of this report to make sure no new hazards have been created and that operational procedures remain current. This section also contains information on the maintenance of equipment along with dates and estimated performance.

6.1. Management of Change Process:

1. A need to change CHEF 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:
    a. relevant engineering standards,
    b. necessary sizing calculations,
    c. details of implementation of the change, and
    d. 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 CHEF folder on HYPERDRIVE or on the CHEF website for future reference. 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.

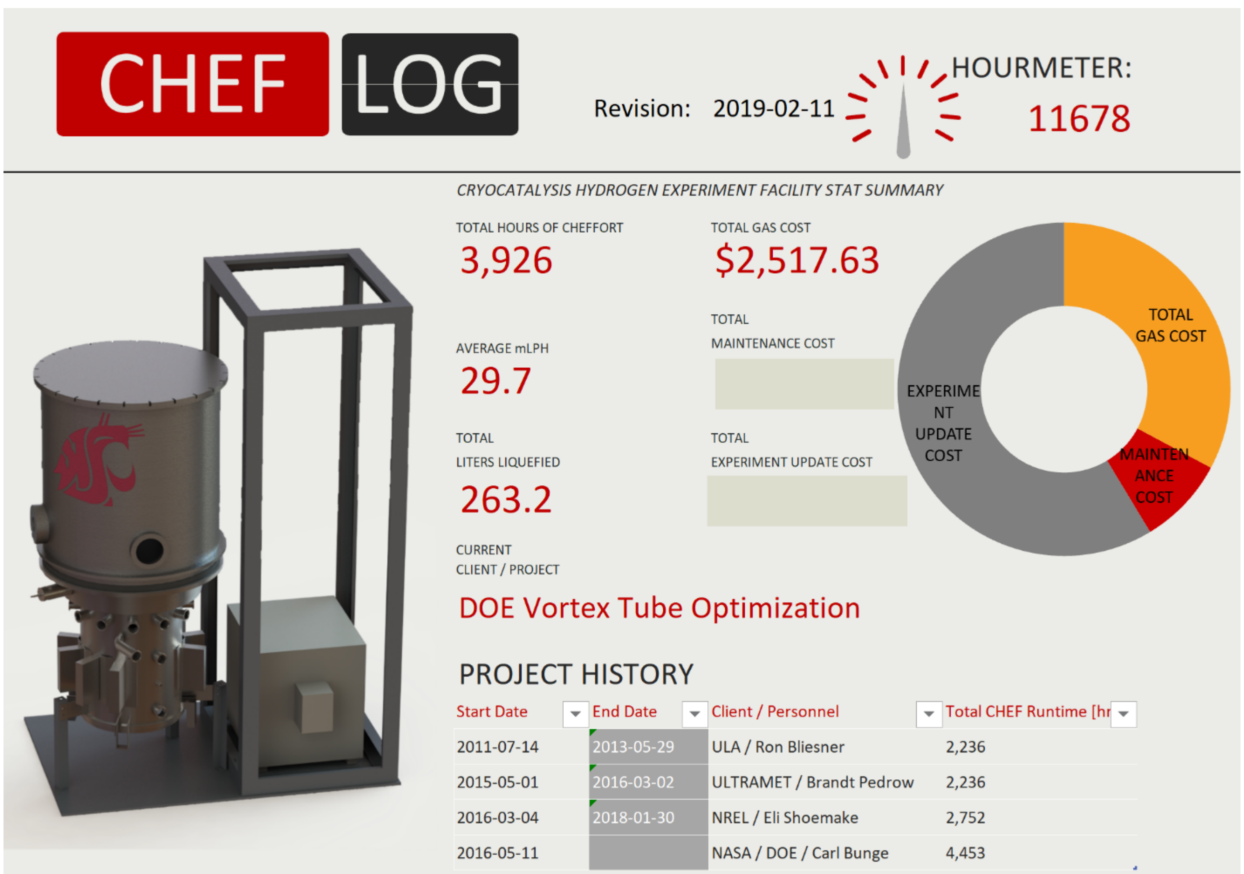
6.2. Management of New Primary Operators:

1. Have the new operator read the history and documentation on the CHEF folder of HYPERDRIVE. They should familiarize themselves with what has been accomplished with CHEF in the past, and how the experiment is currently set up.
2. Give the new primary operator this document so they can familiarize themselves with the scope of the experiment, potential safety issues with its operation, current operating procedures, and required maintenance issues.

3. Establish a multi-week mentor program to train the new primary CHEF operator on emergency, leak check, liquefying, experimentation, and warming up scenarios and procedures. Have the mentee show the existing primary operator all steps for each scenario.

6.3. Maintenance and Repair Schedule:

The maintenance and repair schedule will be kept in the CHEF work-log as a sub-tab in the Excel file labeled 'CHEF Log_rev[X]' in the CHEF folder of the HYPERDRIVE. This workbook keeps track of the CHEF experiment including the history projects and total time elapsed on individual components. This workbook also tracks the total funds spent on maintenance parts, hydrogen gas, and experimental updates. A required maintenance schedule in the document tracks required periodic maintenance. This excel file adds visibility to the CHEF experiment and allows for a predictive model to diagnose and prevent issues caused by thermal cycling.



7. Safety plan Communication:

The FCTO Safety Guidance for Fuel Cell projects recommends five layers of Safety Plan Communication: 1) Training, 2) Safety Reviews, 3) Safety Events and Lessons Learned, 4) Emergency Response, and 5) Self-Audits. Each of these are detailed specific to the HYPER lab below.

7.1. Training

Two levels of training are applied at the general lab level (concerning hydrogen and cryogenic safety) and the experiment level (training specific to operating CHEF). The HYPER lab has a community commitment that each seasoned lab member (graduate student or undergrad with more than one year of experience) develop a hands-on safety demonstration/exercise prior to departing the lab. These safety demonstrations/exercises are stored in a designated tote/bin with a procedure manual and uploaded to the HYPER lab website. Each new student to the lab is required to complete the training before operating an experiment. The training includes completion of the hands-on demos as well as an oral exam of hydrogen and cryogenic safety to be administered by the seasoned lab members. The training will occur within the first two weeks of the semester that a new student starts in the lab with the intent to operate the experiments. A document of the training discussing oral and hands-on exam performance will be logged.

CHEF specific training must also be completed prior to operating the experiment. The potential new operator(s) will be paired with a mentor(s) that have been trained on the experiment. The first step of the CHEF Mentor Training Program is for the new operator to research the procedures and history of CHEF and ask questions on anything which is unclear. An observation step will be initiated next where the mentee observes the mentor performing the various operations/procedures with the opportunity to ask questions. The new operator will be asked to demonstrate proficiency with this document via oral examination and hands-on demonstration of the operating procedures and only then will the training be considered complete. This marks the completion of the CHEF Mentor training program and they are allowed to operate CHEF. Ensure this document is updated with progress as milestones are completed. See the log below for documentation of when the Research Procedure (R), Observation Procedure (O), and Demonstration Procedure (D) was achieved for each member.

7.1.1. Training Log

Name	Years Operated	Mentor	Start Procedure			Fill Procedure			Stop Procedure		
			R	O	D	R	O	D	R	O	D
Ron Bliesner	2011	First Operator	×	×	×	×	×	×	×	×	×
Patrick Adam	2013	Ron	×	×	×	×	×	×	×	×	×
Brandt Pedrow	2015 - 2016	Ron's Thesis	×	×	×	×	×	×	×	×	×
Elijah Shoemake	2015 - 2017	Brandt	15-05-01	15-12-09	15-12-20	15-05-01	15-12-09	15-12-20	15-05-01	15-12-09	15-12-20
Carl Bunge	2016 - 2020	Brandt / Elijah	16-02-12	16-03-21	16-03-30	16-02-12	16-03-21	16-03-30	16-02-12	16-03-21	16-03-30
Jacob Leachman	2016 - 2019	Elijah / Carl				16-12-23	16-12-23	16-12-23			
Kevin Cavender	2017	Carl / Elijah	17-01-10			17-01-10	17-01-10	17-01-10	17-01-10		

Note: Ron, Patrick, Brandt completed training before these measures were recorded.

7.2. Safety Events and Lessons Learned

In the event of an unplanned safety event, such as the power outage on August 2nd 2016 that created this plan, the following steps are to be taken:

1) If an emergency and potentially catastrophic risks are present, press the EMERGENCY STOP button, evacuate the lab, pull the fire alarm, leave the building and work with appropriate safety personnel.
2) If the experiment is safe to abort, complete procedures for safing the system and ensure the system is in a stable "safed", i.e. no stored hydrogen, cryogen, or powered systems mode.
3) Call Dr. Leachman if not already informed, call his cell 208-816-0288 (if in a meeting he may not pick up, accompany your call with a text of the issue and he'll know he needs to leave the meeting).
4) When safe, document the timeline of the event with specifics (who, what, when, where, why?) and send the document to Dr. Leachman via e-mail: Jacob.leachman@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 ensure accuracy.
5) If a lab-wide concern, schedule a meeting for everyone to be debriefed.
6) Your safety incident report will be logged in this document (if CHEF specific), the HYPER-drive under the Safety folder, on the lab website under the "safety" header, and potentially on the H2 best practices website.

7.3. Emergency Response

It is always best to be honest and responsive with emergency personnel, even if you are at fault. An emergency is no time for filtered responses. Follow the safety event procedure above.

Copies of this document must be kept in the front of the CHEF Safety and Procedures manual located next to the experiment on the desk. This area should be kept clean always. A Plumbing and Instrumentation Diagram (P&ID) mirroring the physical layout of the plumbing manifold should be displayed on the manifold board.

7.4. Self-Audits

The significance of a hydrogen ignition or an autogenous cryogen pressurization will be demonstrated with the lab's hands-on trainings. This should create sufficient motivation for self-audit and review of these policies and procedures prior to conducting a test. These audits should be conducted with Dr. Leachman or seasoned lab personnel when necessary.

8. INCIDENT REPORTS AND EVENT RESPONSE

8.1. POWER OUTAGE AND HYDROGEN LEAK - AUGUST 2ND, 2016

On August 2, 2016, CHEF had a nearly full tank of liquid hydrogen when a power outage resulted in boil off of the system's stored liquid. This boil off caused over-pressurization of the experiment, eventually leading to venting of hydrogen to the laboratory workspace.

8.1.1. EVENT TIMELINE

09:00	A bird flew into a sub-station, causing a power outage for the entire WSU-Pullman campus. Campus-wide backup diesel generators began restoring power to campus sectors in a predetermined order based on need. A test of a hydrogen vortex tube in the Cryocatalysis Hydrogen Experiment Facility (CHEF) using 4.25 liters of liquid hydrogen had been planned to start at about this time.
09:20	The principal investigator (PI) was informed of the power outage, including all CHE and the fume hood ventilation. He was told that a mechanical pressure gauge on the experiment read 90 psi. Instructions were given to manually vent the experiment if the pressure rose to 120 psi.
09:35	Power was restored and the ancillary support systems for CHEF were turned back on. The cryocooler had warmed to the extent that the thermal wave was moving towards the stored liquid hydrogen, and it could not be mitigated in time to prevent rapid boiling.
09:40	The PI arrived at the lab. The CHE pressure had now risen to 120 psi, and a manual vent of the hydrogen through the fume hood was initiated. The pressure continued to build in CHE as the temperature had risen above 30 K and the liquid was rapidly boiling while transitioning to a supercritical fluid. (Note: CHE has three proportional, pressure relief valves that are designed to vent through the lab fume hood. All three valves are adjustable and had been tested within the last 8 months to open at 150 psi, 160 psi, and 165 psi. The 150-psi valve failed to open. When this occurred, the manual vent was increased. The 165-psi valve was the first to open; however, a small liquid vent port on the side of the 165-psi valve was jetting a hydrogen stream towards electrical equipment.)
10:05	The project team was discussing ducting the venting hydrogen stream away from electronics when they heard a "pop" but did not know what it was. In retrospect, the team thought it was likely the 160-psi pressure relief valve opening. However, a small ignition could be not be ruled out. The project team could not determine the amount of hydrogen entering the room (versus venting through the fume hood), so the lab was evacuated and the fire alarm pull station was activated along with a 911 call to explain the situation.
10:30	The Pullman Fire Department was on site and briefed about the situation, hazards of hydrogen, and maximum quantities that may have been released into the room. A real-time readout of the experiment dashboard from a cell phone revealed that the experiment had

	vented the remaining hydrogen and was depressurized. As a precautionary measure, firefighters allowed the hydrogen time to diffuse before entering the building.
11:00	Fire fighters entered the building with a thermal imaging camera, hand-held hydrogen detector, and CO_2 extinguisher. The maximum hydrogen level detected in the lab was 1% of the lower flammability limit.
12:00	The project team was permitted back into the lab to ensure the area was safe.
12:30	The all-clear was given, allowing everyone back into the building. The experiment was put in a safe configuration with inert gases. The PI met with the college communications team to explain the event should there be a media inquiry.

The project team led a discussion and a tour of CHEF on August 11, 2016 for the DOE Hydrogen Safety Panel. Members of the project team were cooperative and transparent in discussing the event and configuration of the equipment. Based on the discussions, tour of the lab, and a review of the timeline, opportunities for improvement to ensure the safety of personnel and equipment were identified. These recommendations were considered by the project team and a response was developed for each recommendation.

8.1.2. Response to DOE H2-SP Recommendations

In response to the event on August 2nd 2016, the Hydrogen Safety Panel (Nick Barilo and David Farese) conducted a site visit, analysis, and provided the following recommendations for change to CHEF. Each recommendation is addressed item-by-item below.

1. Based on the event timeline, it appears that personnel remained in the laboratory while hydrogen was leaking in the room. This approach places people at risk and should be avoided.

Recommendation #1: Establish procedures and educate lab personnel to ensure that everyone evacuates the facility quickly when an uncontrolled hydrogen leak occurs. (IMMEDIATE NEED)

Change: This problem has been addressed in two ways:

1) We now have an EMERGENCY STOP button that allows lab members to hit the button and walk out of the lab with no concerns.
2) A change to lab procedures (emergency abort procedure) and safety training that requires people to execute the Emergency Abort procedure using the new EMERGENCY STOP button.

2. The equipment and project activities lacked a U.S. Department of Energy safety plan, comprehensive risk analysis, and process safety review. A thorough assessment of the potential hazards associated with an activity is essential to protect people and facilities, and to ensure that proper safeguards are in place. While a simplified failure modes analysis was performed, a hazard and operability study (HAZOP) would be a better tool for understanding the risks associated with these activities.

Recommendation #2: Perform a HAZOP for the project using a qualified team. It may be beneficial to identify a process safety resource to assist with, document, and help implement recommendations of the HAZOP. (IMMEDIATE NEED)

Change: This document.

3. Piping and instrumentation diagrams (P&IDs) for this activity could be improved with more detail, such as lines sizes and set points. P&IDs are foundational to the maintenance and modification of the process that they graphically represent. At the design stage, the diagram also provides the basis for developing system control schemes in the HAZOP.

Recommendation #3: Add detail on line sizes and set points to the P&IDs. Also consider including:
• Control and shutdown schemes
• Safety and regulatory requirements
• Basic startup and operational information
(IMMEDIATE NEED)

Change: P&ID has been updated in conformance with CGI and other international standards. The management of change section above ensures all components are present and include line sizes and types and all set points for PRDs.

4. The primary cause of the incident was a combination of 1) an electrical failure causing a triple mode failure and 2) design flaws in the pressure relief system. Failure of the electrical supply resulted in:
a. Failure of the cryocooler.
b. Failure of vacuum due to the active vacuum pump system. Vacuum increased to 11 torr, which is ineffective for multilayer insulation.
c. Failure of process exhaust due to loss of the vent hood ventilation when the power failed. This likely led to flammable gas concentrations within the hood.

Recommendation #4: Consider using fail-safe solenoid valves or other automatic controls to vent the equipment on loss of vacuum. Alternatively, identify other methods to maintain the system vacuum in the event of a power loss. (IMMEDIATE NEED)
Comment #1: It may be beneficial to evaluate the operational need for backup power for the CHE (batteries, generators, etc.). However, there are likely less expensive methods than backup power to ensure safety of personnel and equipment.

Change: A CyberPower 1500 VA backup power system will sustain ancillary power for 62 minutes (the campus backup power systems takes ~40 minutes). This backup supply runs:

A) The Swagelok solenoid valve that can be actuated by the hydrogen sensors, or the emergency abort switch, to vent the system.
B) The LakeShore temperature controllers and pressure transducers.
C) The CHEF computer and Data Acquisition Board
D) ALICAT flow control meters.

If the pressure within CHEF rises to 145 PSI, the PRVs will automatically vent the system through the vent line. Should all three primary stage PRVs fail to open, a secondary stage burst disk will blow into the vent system at 200 psi. An Emergency Abort switch also allows the system to be vented via a fail-safe solenoid valve that can also be actuated if a hydrogen leak is detected by the H2-Scan hydrogen sensor.

5. A number of issues were identified with CHE system design:

a. CHE vent lines were piped to a local fume hood. It is likely that the fume hood's exhaust flow is inadequate to prevent a flammable concentration of hydrogen should a pressure relief device operate. Additionally, the exhaust was not available during the building's power outage (a condition when it would be needed since the CHE is currently configured to boil off during a power outage).

Recommendation #5: Consider routing all vent lines to a location outside the building. The vent termination should not discharge near openable windows or air intakes for mechanical equipment. (IMMEDIATE NEED)

Change: A 1-1.5" vent line was designed by an independent contracting firm (Coffman Engineering in Spokane WA), then constructed to run through the ceiling to the peak in the roof at the top of the building. This line conforms to CGI vent standards for cryogenic hydrogen and will be directly connected (without valving) at all times.

b. The CHE incorporates redundant piping for the process vent, a positive feature that reflects a good design. However, no calculations were available to determine potential vent flows needed with loss of cooling/vacuum, and project personnel were not aware of guidance to perform these calculations, such as those found in CGA S-1.3, "Pressure Relief Device Standards-Part 3-Stationary Storage Containers for Compressed Gases." This document addresses loss of vacuum insulation, fire, and other relevant hazards.

Recommendation #6: Calculate the relief demand to verify relief device sizing using CGA S-1.3. (IMMEDIATE NEED)

Change: Complete redesign of the pressure relief design has been completed and can be seen in the CHEF documentation.

Recommendation #7: Design the system using industry standard practice for inlet line pressure drop of no more than 3% (e.g., apply ASME or API standards). (IMMEDIATE NEED)

Change: After reviewing the applicable standards we found the ASME standards to be the most conservative and followed those in the re-design of the system, no more than 3% pressure drop on an inlet, no more than 10% pressure drop on an outlet. The calculations were performed in EES and verified by Coffman Engineering.

c. Inlet lines are likely too small based on expected relief flows and were fabricated in a manner that allowed the line cross sectional area to be reduced in diameter (bent with too tight a radius and/or kinked).

Recommendation #8: The project should either ensure that the reduced area of the inlet lines is adequate or replace the bent lines. (IMMEDIATE NEED)

Change: Vent lines were increased to 3/8" tubing size, if bent the reduced area will be verified before installation.

d. CHE relief devices were low-quality units and did not have the reliability needed to protect vessels.

e. Standard industry practice for cryogenic vessels would be to have relief valve and rupture disc for greater reliability.

Recommendation #9: Consider higher reliability valves and adding a rupture disk. (IMMEDIATE NEED)

Change: We purchased 145 psi Generant Pressure Relief Valves and a BS&B 200 psi burst disc.

f. Relief devices could be manually reset with no documentation program to verify/record the set-point.

g. The Rego relief device had an unknown vent location that vented within the lab during the incident.

Recommendation #10: Change the style of the relief valve to ensure gas is captured the vent piping. (IMMEDIATE NEED)

Change: The new relief valves do not have 2-phase relief ports that are unvented. The new relief valves are pre-set. However, the management of change procedures in this document account for the issues.

h. Three relief valves were in the CHE design, but one was located on the downstream side of a purposely throttled hand valve. Since this valve would be isolated/restricted under normal conditions, it would not be considered an effective layer of protection in a HAZOP.

i. Vent lines from relief devices were insufficiently sized. The lines should be sized for no more than a 10% pressure drop.

Recommendation #11: Design the vent lines using standard industry practices (e.g., ASME, API, CGA standards). (IMMEDIATE NEED)

Change: See above.

j. The vent lines were constructed of plastic tubing. Such material is not recommended due to its poor performance in fire conditions. In a fire, this tubing could fail and result in venting of hydrogen to the laboratory.

Recommendation #12: The vent lines should be replaced with a metallic material, preferably stainless steel. (IMMEDIATE NEED)

Change: Coffman Engineering in Spokane is experienced with designing this type of system and selected copper vent lines (1” and 1.5”) due to non-sparking characteristics.

k. The piping and manifold were unsupported and susceptible to damage from surrounding equipment or personnel.

Recommendation #13: Properly support the manifold. (IMMEDIATE NEED)

Change: An entirely new gas manifold system was designed and installed on a bosch-tubing frame that is secured with positionable base mounts.

l. The hydrogen inlet line was equipped with a ball valve and the inlet connection was not capped when not in use. Inadvertent bumping of the valve could lead to liquid hydrogen ejection from the pressurized vessel.

Recommendation #14: Install a check valve on the inlet and cap the line when it is not in use. (IMMEDIATE NEED)

Change: We have installed multiple check valves and flame arrestors now installed in the gas manifold. Use of gas caps will minimize inflow.

m. The hydrogen inlet valve is not arranged to allow purging of the system.
Comment #2: It may be beneficial for the project to install a valve to permit purging of the inlet line. Such an arrangement will prevent a small amount of air from being introduced into CHE on each fill, which could subsequently freeze and build up in the apparatus.

Change: This valve has been installed to minimize the effects of regulator failure. The vacuum line can be connected to allow purging of the system.

n. The manual vent valve was not equipped with a check valve. When the manual valve was opened, there was a concern about pulling air back into apparatus.
Comment #3: Installing a low cracking pressure check valve on the outlet side of the manual valve will help prevent the introduction of air into the CHE.

Change: Check valves and flame arrestors are now installed.

o. As currently configured, there is potential for substantial leakage of hydrogen into the cryostat from a leaking fitting or broken line. The cryostat has a relief line (which is good), but it vents within the building. Additionally, it is anticipated that most venting will likely pass through the vacuum pump if it is running.

Recommendation #15: The project team should evaluate what would happen if a significant quantity of hydrogen is released within the cryostat, including effects on the vacuum pump (both running and non-operational) and cryostat relief device. (NOTE: The CHE is different from most cryogenic dewars, which have welded construction on internal piping, significantly reducing the likelihood of internal leakage.) This evaluation should include verification that sufficient relief capacity exists to prevent failure of the cryostat, and identify modifications that are needed. (IMMEDIATE NEED)

Response: We have installed an additional vacuum port in the top of the vacuum chamber with a connection for a Hy-Alerta 600 solid-state hydrogen sensor to detect and vent if hydrogen has accumulated in the top of the chamber. During operation the chamber is under vacuum and hydrogen cannot accumulate in flammable quantities within the chamber. If the vacuum system shuts down prematurely, and a leak appears within the vacuum vessel, a pressure relief on the chamber itself can open to the vent line. This occurs before atmospheric air is allowed to enter into the vacuum chamber and form a combustible mixture.

Change: We have ordered an overpressure valve from Generant to relieve pressure. It has been sized according to the CGA S1.3. We have also refurbished the vacuum pump.

p. It may be possible that some of the vacuum degradation was from leaking seals on the cryostat. This incident may have been an inadvertent test that demonstrated it was insufficient.

Recommendation #16: Verify that the clamping force of the cryostat at cryogenic temperature is sufficient to maintain hydrogen containment. (LONG-TERM NEED)

Change: We have ordered an overpressure valve from Generant to relieve pressure. It has been sized according to the CGA S1.3

q. As currently configured, it appears that leakage from the CHE into the cryostat could occur during an incident, resulting in a flammable atmosphere in the cryostat. Potential consequences from such an event would be a hydrogen release to the room or ignition within the cryostat.

Recommendation #17: Consider adding a hydrogen detector within the cryostat to enable researchers to detect hydrogen prior to manually lifting the top. (NEAR-TERM NEED)

Response: From the design of the experiment there will never be a scenario where hydrogen has positive pressure in the vacuum chamber. The vacuum chamber itself prevents any accumulation of hydrogen in the system by completely evacuating any leakage. In the event that a line discharges and the power is off, the overpressure valve is vented through the building vent line. Once power is restored, the vacuum evacuates any excess hydrogen. This occurs before atmospheric air is allowed to enter into the vacuum chamber and eliminate vacuum.

Change: We have implemented an Emergency Abort System (EAS) which is able to automatically vent the hydrogen contained in the system in the event that our H2scan Hy-Alerta sensor located above the experiment senses a hydrogen level over the LFL. After calling h2scan, their testing protocol only includes pressure conditions slightly below atmospheric conditions. Therefore, they do not recommend the use of the sensor in high vacuum scenarios.

Recommendation #18: Verify that electrical equipment in the cryostat will not be an ignition source. (NEAR-TERM NEED)

Response: All electrical equipment will be verified to follow good practices in cryostat design to ensure that they will not be an ignition source. All electrical resistance heater controllers have circuit breaker or fused protection to prevent any possible sparking.

r. The project team has repurposed a propane tank for use with hydrogen. The tank has a thinner wall and is intended for outdoor use only (as stated on its label). Thin-walled tanks do not respond well in an exposure fire. Additionally, the tank is located near potential cryo release points, potentially exposing it to cryogenic temperatures.

Recommendation #19: Consider replacing the tank with one designed for indoor hydrogen service, and located to prevent potential liquid hydrogen impingement. (NEAR-TERM NEED)

Change: New hydrogen k-sized compressed gas bottles are purchased to replace the propane tank to provide a better factor of safety in our hydrogen storage reservoir.

s. The hydrogen fill regulator is not equipped with a relief valve.

Recommendation #20: Consider adding a relief valve on the inlet fill line with a captured vent to protect against regulator failure. (IMMEDIATE NEED)

Change: Lab procedures are updated to include a pressure relief and flame arrestor on all regulators. A relief/purge valve was added on CHEF to account for regulator failure.

t. A vacuum pump is installed on hydrogen piping with only a ball valve for closure. Inadvertent bumping or opening of the ball valve could release gaseous or liquid hydrogen into the vacuum pump at pressure.

Recommendation #21: Evaluate the impact from introducing hydrogen into the vacuum pump at pressure. Consider disconnecting the vacuum pump during testing. (IMMEDIATE NEED)

Response: We re-evaluated industry documents indicating it was acceptable to pump hydrogen through rotary vane vacuum pumps: https://www.lesker.com/leskertech/archives/0g11m3h/05-025_leskertech_v4i2.pdf The specific Leybold roughing pump we have is designed specifically for pumping volatile compounds. Combustion is unlikely until outlet of roughing pump as H2 only added under vacuum. Outlet plumbed to fume hood, always on with vacuum pump. Hydrogen would fill pipe and extinguish flame. The roughing pump cannot pump enough hydrogen to create a flammable mixture in the fume hood.

u. As configured, there is no definitive way to know how much liquid hydrogen is in the CHE, and it is unclear what the impacts would be if it is overfilled.

Recommendation #22: The project team should determine if overfilling the CHE with liquid hydrogen is an issue and/or consider a means to determine the system's contents. (NEARTERM NEED)

Response: Overfilling the liquid hydrogen tanks is not possible - when the tanks are full, they will simply reach thermal equilibrium with the heat leaks going into the system. This is because we cryo-condense the hydrogen into the tank in order to fill them. Once the tank is full, there simply is not cold surface sufficient to continue condensing hydrogen. This is not a concern.

v. CHE relief devices are set to activate at 160-180 psig, but a comment was made by the project team that flow controllers downstream are rated to 125 psig.

Recommendation #23: Evaluate the pressure rating of the entire system and adjust relief device set points accordingly. (IMMEDIATE NEED)

Change: The pressure relief system has been designed to CGA S-1.3 and API standards with both primary and secondary stages. The Alicat flow controllers are rated to 145 psi instead of the 125 psi originally stated. The new pressure relief design takes this limit into account.

w. The CHE equipment is located outside of a ventilated enclosure in the laboratory (see NFPA 2-16.3.2.1.2.5). Also, no hydrogen detection is provided in the lab area.

Recommendation #24: Relocate the CHE equipment into a ventilated enclosure. (LONGTERM NEED)

Change: No plans to do this are currently in progress. In the event this becomes feasible with the above gimbal design, we will reconsider. A gimbaled vent enclosure design has been conceptualized and can be implemented if need in the future with use of the central vent duct in the lab.

Recommendation #25: Provide hydrogen detectors in the laboratory until the CHE is relocated to a ventilated enclosure. Upon activation, the detectors should provide audible/visual notification and control a safe shutdown of the equipment (when a maximum of 60% of the lower flammability limit is detected). (NEAR-TERM NEED)

Change: We have implemented an Emergency Abort System (EAS) which can automatically vent the hydrogen contained in the system upon the engagement of an emergency button and/or in the event our H2scan Hy-Alerta 600b sensor senses a hydrogen level at 60% of LFL.

x. The cylinder storage locker is not ventilated.

Recommendation #26: Add exhaust ventilation to the storage locker or consider another area for storage of gas cylinders. (IMMEDIATE NEED)

Change: We have confirmed that the gas storage locker is ventilated and meets the storage requirements for storage of up to 1000 cu. ft. of hydrogen gas per WA State codes AND NFPA 2-2016. We are installing a hood to direct any gas directly into the vent location approximately ¾ of the way up the height of the storage closet. The addition of a sprinkler will allow a maximum storage of 4000 cu. ft. of hydrogen, though we will never top 1000 cu. ft.

6. It does not appear that researchers are wearing flash fire protective coveralls or lab coats when working with or around hydrogen.

Recommendation #28: The project team should consider using flash fire personal protective equipment when working with or around hydrogen. (IMMEDIATE NEED)

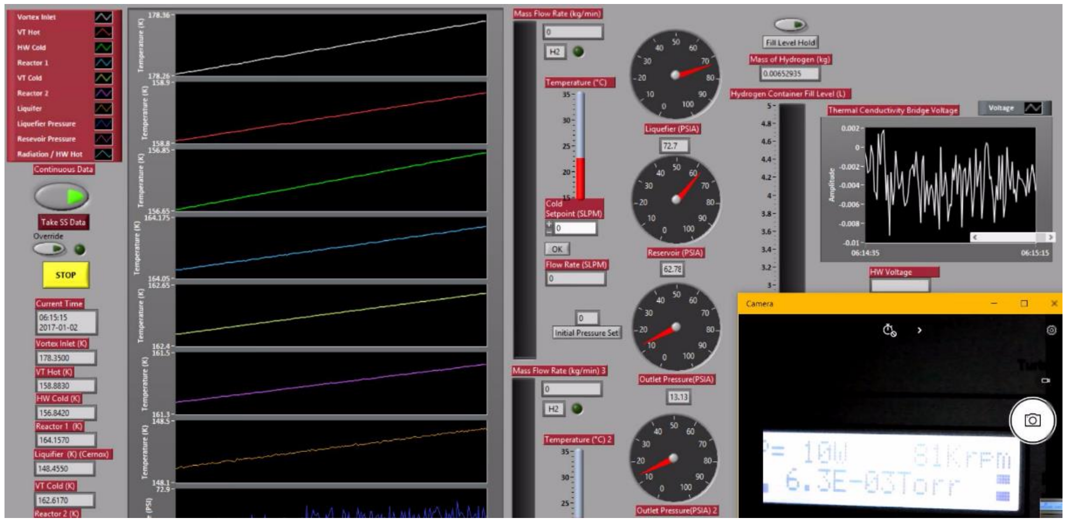
Change: A pair of fire resistant lab coats has been purchased to protect against the possibility of fires. Face shields are now also available in the lab.

8.2. Water Cooling Loop Failure and Hydrogen Vent - January 2nd, 2017

On January 2, 2017, a power failure to the laboratory water cooling loop caused cryocooler overheating and shutdown. This eventually led to an over-pressurization event, and venting of the stored hydrogen.

8.2.1. Event Timeline

00:01	CHEF is running and liquefying hydrogen. Approximately 4 liters of hydrogen have already been liquefied. A test is anticipated to be run later in the day.
~02:00	The laboratory water pump is powered by a surge protector strip. A plastic switch on this strip breaks, and the switch is pushed up by its spring, breaking electrical contact. The water pump stops running the water cooling loop.
02:10	The cryocooler cooling CHEF overheats and shuts itself off to avoid damage. The condenser tanks immediately begin warming up from the heat leak into CHEF.
04:07	The pressure builds to 145 psi and the pressure relief system opens as designed. Pressure climbs to a maximum of 164 psi in a 7-minute vent. After relieving pressure, the pressure relief system closes again. At some time during the overpressure, a leak opens in the vortex tube.
06:18	Carl Bunge wakes up and checks in on CHEF. The liquefier is at 148 K and everything in CHEF is warming up. Vacuum level is 6.3 e-3 Torr.



A screenshot of the LabVIEW interface when Carl logged in.

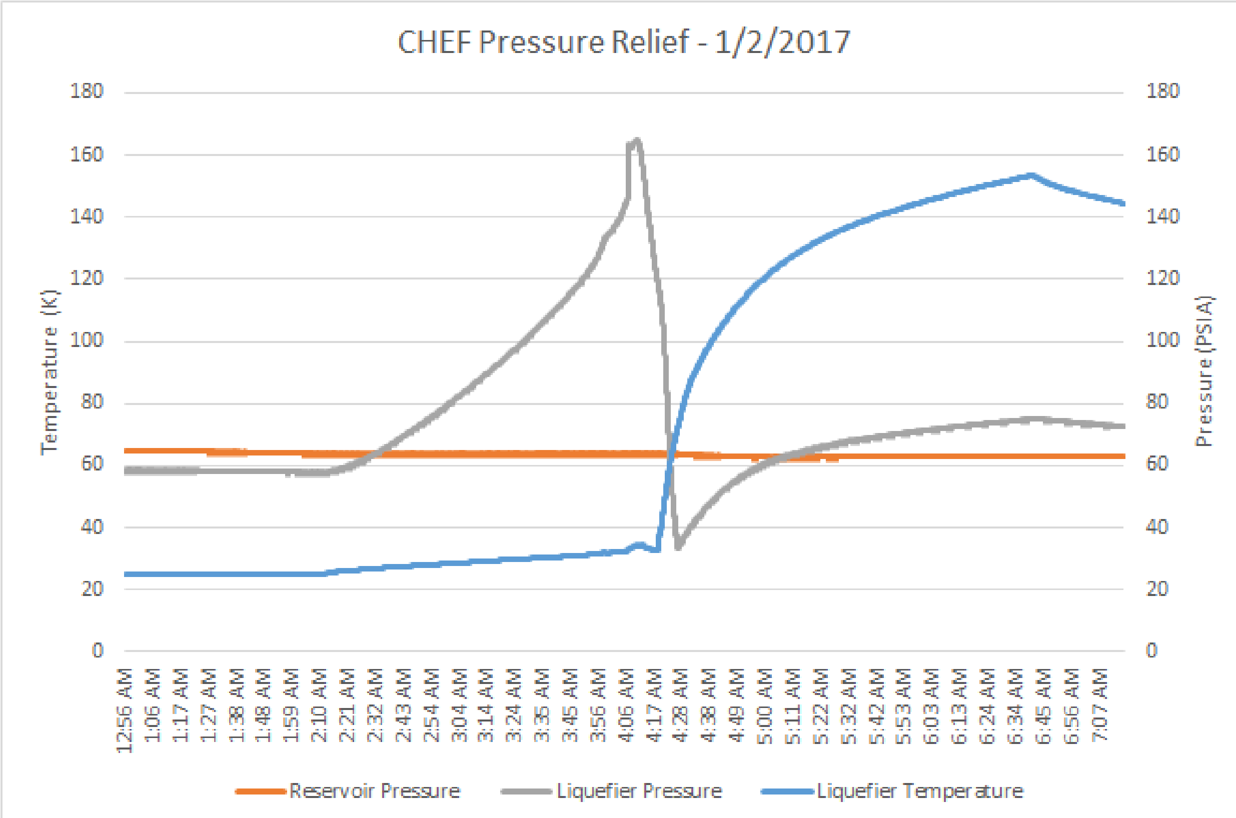
Carl gets into the lab and discovers the switch failure. He documents the cause on Slack and restarts the cooling water loop and the cryocooler. The vacuum level and temperature begins to drop again.

06:43

The broken switch, as documented by Carl. Notice the broken plastic on the switch and remaining in the switch housing.

10:00 The decision is made by Carl and Jake to warm up CHEF all the way to check out the systems. The cryocooler is shut off and warmed up.

The data collection system was running throughout the event, a graph of significant temperatures and pressures throughout the event is given below:



8.2.2. Response to the Event

Following the event, a walkthrough of the entire hydrogen vent line took place with Shawn Ringo to try to spot any potential leak locations that could have resulted from the large quantity of vented hydrogen. It was determined that the cast iron caps on the vent line should no longer be used due to hydrogen embrittlement, so these were replaced with polymer fittings. No new issues with the venting system were found.

Initial analysis proposed that the cause of failure of the power supply was due to a dirty filter in the water cooling loop. Upon later examination of the failed switch, it seems unlikely that this was a contributing factor, however dirty water was likely a contribution to decreased cryocooler performance for this test run. To fix this issue the following steps were introduced:

- Remaining galvanized fittings were removed and replaced with brass versions.
- A second water filter holder and filter was added with Y shutoff valves to allow for filter changes mid-cycle, if needed. This will also allow higher flowrates through the filters and longer times between filter changes.
- The entire water cooling loop system was flushed with CLR solution twice for at least 10 minutes each time. This helped ensure rust was removed from the system.
- Updates to procedures and maintenance logs ensured the filters would be changed at least monthly when in use.

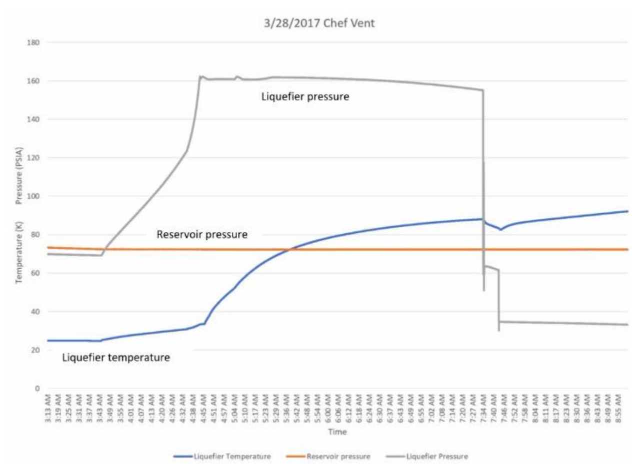
The water pump was on the surge protector primarily to ensure the water pump was placed on a separate circuit from running vacuum pumps to avoid tripping a circuit breaker. A closer independent circuit was found and the pump is plugged directly into the outlet. If necessary to move the water pump back to its original circuit, a longer electrical cord will be installed on the water pump so a direct connection is possible.

Finally, by examining the detailed log of pressures and temperatures during the event (as shown at the end of section 7.2.1), we could confirm that the pressure relief system worked as intended, and stayed under the max flow rating pressure factor (116% = 168psia). This gives good evidence that the pressure relief system was properly designed and worked as intended.

8.3. Unexpected Heating and Overpressure Events

8.3.1. March 28th, 2017

The first unexpected heating and overpressure event occurred at about 03:40 on March 28. A sudden temperature rise had occurred overnight, boiling off the ~1.1 L of liquid hydrogen and over pressurizing the system. Upon Eli entering the lab at approximately 07:30, the liquefier temperature was as high as 87K and the vacuum level in the cryostat was at ~2e-2 Torr. Eli shut off the cryocooler, which was still running, and vented pressure from the liquefier tanks. Upon venting pressure, the vacuum quickly recovered to the e-4 Torr range.



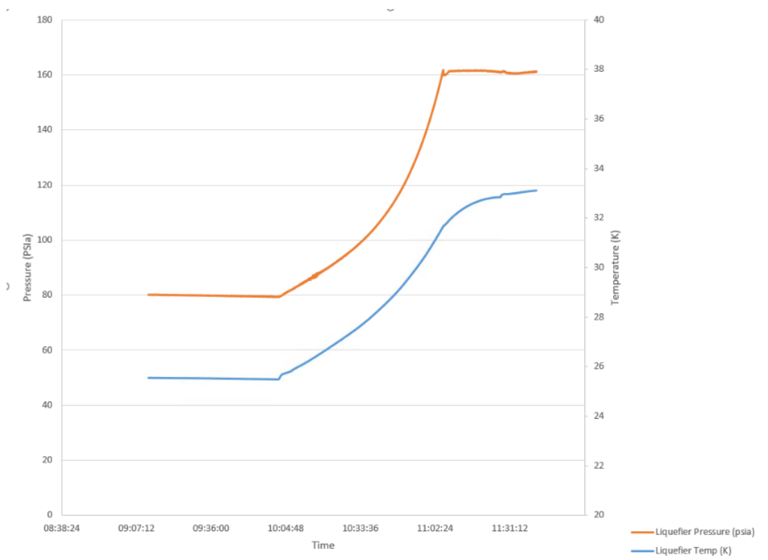
Graph of temperature and pressures of the event.

It was believed to be a leak opening in CHEF that caused the issue, as the failure happened not too far into the liquefaction cycle and the cryocooler seemed to be running well the entire time. After opening CHEF, one of the pass-through Swagelok connections was found to be leaking. The connection was repaired and on a second test another leak was found in the hot hotwire epoxy joint. This hotwire was also repaired. CHEF was put back into service

8.3.2. April 7th, 2017

Slightly more than halfway through the next test's liquefaction, another thermal event occurred at 10:01:40 on April 7. Carl was in the room at the time, but did not notice the event as it was occurring. He left about 10:30 and the pressure relief vented shortly thereafter, around 10:40.

When Carl got back and Eli arrived around 11:40, they noticed unusually high pressure on the gauge, then confirmed with the pressure transducer and temperature readings to be correct. They vented the hydrogen and once again the vacuum level dropped from e-2 Torr range down to the e-4 Torr range at around 70 psi in the liquefier. Further reduction in pressure again reduced the vacuum level. At low pressure and 2.8e-5 Torr the second stage of the cryocooler bottomed out at 21.89 K.



Temperature and pressure of the second event

The second event was very like the first, such that the discovered leaks were no longer thought to be the problem in the system. Once again, the cryocooler remained on and appeared to be working shortly after the event. In this case, nearly 2.7L had been liquefied and the ninth liquefaction cycle was being attempted before the issue occurred. It was determined that the cryocooler may be an issue, and steps were taken to contact SHI Cryogenics of America to ascertain if the cryocooler could be causing the issues.

9. CHANGELOG

9.1. 2016-11-29 – DOE HYDROGEN SAFETY REVIEW PANEL UPDATE

During a routine test in July 2016, a power outage occurred within 10 minutes of a planned test, with maximum liquid hydrogen fill. The details are provided elsewhere: https://hydrogen.wsu.edu/2016/08/15/our-near-miss-hydrogen-vent-in-etrl-221/ After meeting with Nick Barilo of the Department of Energy's Hydrogen Safety Review Panel, several changes to the procedures in this document were recommended. Upon these recommendations, the following changes were made:

- Removal of the term HAZOP from this document, as it was agreed that this document serves as more of a Failure Mode and Effects Analysis (FMEA) and Safety Plan than a proper HAZOP.
- Addition of a document changelog to note changes to the document and when they occur.
- Revisions throughout the document to improve clarity in sections where intent was unclear. This included changing wording to increase clarity, as well as ensuring that terms used across the document were consistent.
- Valves are numbered to match numbers added to CHEF. This should make valve identification easier.
- Explicit directions to perform several steps were added to the procedures to ensure they don't get forgotten or missed. (Capping inlet line, keeping vacuum valve open, wearing PPE, etc.)
- An emergency safety procedure to follow when hydrogen is suspected in the vacuum chamber was added.
- Maximum roughing pump flow rate for hydrogen venting into the hood was calculated, and a maximum roughing pump size was listed for hydrogen operations.

9.2. 2016-12-01 – SAFETY FAILURE / HAZARDS MATRIX ADDITIONS

The following additions were made to the Safety Failure / Hazards Matrix:

- A section was added detailing the possibility of a failure of the Uninterruptible Power Supply (UPS) system with power still on.
- The process of checking the resistances of all electrical connections before each test was added to improve the mitigation and detection strategies.

9.3. 2016-12-05 – Added CHEF training specifics and log

Updated the Management of Change section with CHEF specific training information and the details of the CHEF Mentor Training Program. Added log with who has been trained on the experiment.

9.4. 2016-12-12 – Refined CHEF purge and fill procedure, Updated Formatting

Small changes to the CHEF purge and fill procedure were made to increase clarity. Several actions were made more explicit in the instructions and valve opening / closing instructions were reordered to make the procedures easier to follow and safer.

Formatting was changed to use the latest formatting from the Design Documentation document. This adds additional features including more available subheading levels, numbering sections and a nicer Table of Contents. This also ensures consistency across the CHEF documentation.

9.5. 2016-12-17 – Procedure Updates

Procedures were updated with missing instructions. A new section was added before the procedures to note insights learned operating the experiment that don't fit into the procedure instructions.

9.6. 2016-12-28 – Fill Procedure Update

Fill procedures were updated with more details.

9.7. 2017-01-04 – Cooling Loop Update

Updated cooling loop check to ensure that the water filter maintenance schedule is followed.

9.8. 2017-01-05 – Fill Procedure Update

New fill procedures from the 28th were reviewed and updated with any necessary changes during a walkthrough with the experiment.

9.9. 2017-01-06 – Cooldown Procedure Update

Added step to ensure continuous data collection during cool down to track cryocooler performance.

9.10. 2017-01-09 – Section 7 update

Following a new unexpected event, section 7 was updated to provide more documentation.

9.10.1. Change of Section 8 to Incident Reports and Event Response

Section 8 was updated to be a general section for recording reports of emergency or unexpected events relating to the experiment. The details on the August 2 power failure and hydrogen leak were moved to a new subsection, section 8.1. The response to the DOE HSP recommendations was specifically moved to section 8.1.2.

9.10.2. Addition of Section 8.1.1, August 2 Event Timeline

Under the new section 8.1 detailing the August 2, 2016 event, a new section 8.1.1 was added with the event timeline. This timeline recorded the events that occurred during the incident, as recorded by DOE HSP review, and indicated the timeline of events used to formulate recommendations. This timeline was compiled from the official lab statement on the event and input from WSU and Avista personnel as well as the project team.

9.10.3. Addition of Section 8.2, January 2 Event Details

A new section 8.2, including subsections on the event timeline and response was added to detail the loss of power to a water cooling loop pump on January 2, 2017.

9.11. 2017-01-13 – Training Log Update

The training log was updated to be in its own section to allow for landscape orientation. This gives more room for full dates of completed training without requiring splitting the table or moving to a tiny font size. Training information was added for Kevin Cavender and Jacob Leachman.

9.12. 2017-01-26 – Added Vortex tube run procedure

Added vortex tube run procedure section 4.3 for completeness. A separate run procedure exists on hyper drive, yet this allows an all in one procedure document.

9.13. 2017-04-10 – Added Unexpected Heating and Overpressure Events

Slight updates to the document to bring it in line with newly created templates for other experiments were completed. Section 8.3 was added detailing the circumstances up until the present of unexpected heating and overpressure issues with the latest runs of CHEF.

9.14. 2017-05-27 – Document Updates

9.14.1. Updated Leak Checking Instructions

Updates were made to provide more information on how to properly use the mass spectrometer to leak check the experiment in Section 4.2.1.

9.14.2. Updated Section References

Sections were updated with hyperlinked cross-references so they will both auto update with the document and also link to the section for quick jumping to the referenced section.

9.15. 2018-02-09 – Updated Procedure for Higher Pressure Ratios

New test conditions for higher pressure ratio measurements were recorded after completing Test 28.

9.16. 2018-07-23 – Updated Procedure added hotwire calibration procedure

Updated existing procedure for modular reference to vacuum establishment, leak checking, ect. separately. Added normal-hotwire calibration to the procedure.

9.17. 2018-07-30 – Updated purge procedure

Updated existing purge procedure with figures to demonstrate purge routes to ensure no contamination within the experiment.

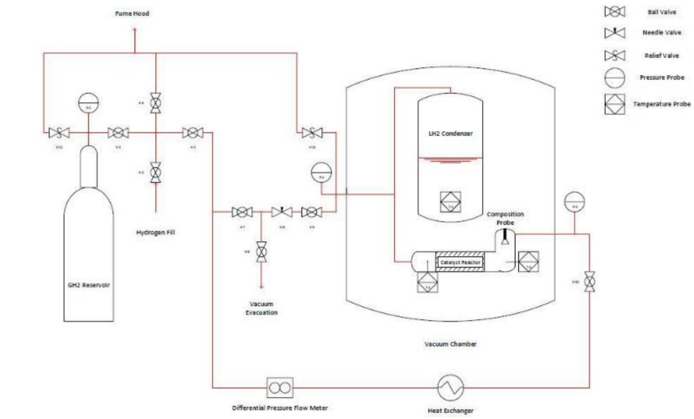
9.18. 2019-03-27 – Updated operator operation years

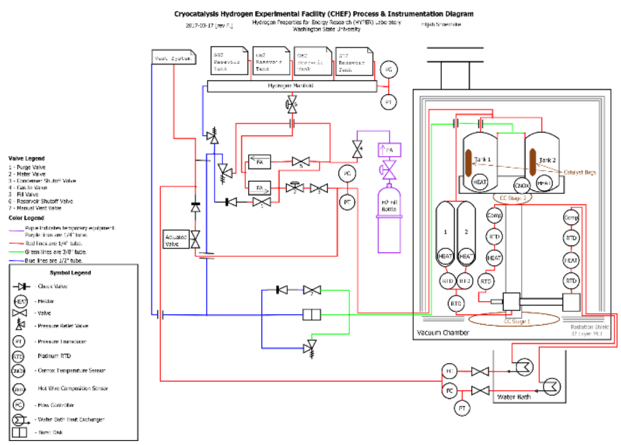
Updated Jake and Carl's operating years from 2016-2017 to 2016 to 2019.

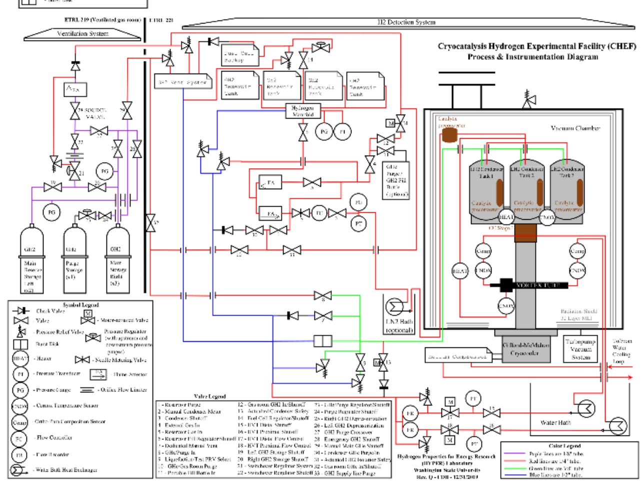
9.19. 2019-04-18 – Major revision for CHEF upgrade to higher pressure test operation and autonomous fills

- Updated scope to include "Optimization of the Heisenberg Vortex Tube for Hydrogen Cooling" goals.
- Changed title of section 2 from "Background Information" to "Organizational Safety Information" to align with DOE safety plan guidance document.
- Changed title of section 3 from "Safety Failure/Hazards Matrix" to "Project Safety" to align with DOE safety plan guidance document format.
- Changed title of section 2.6 from "Risk Assessment" to "Risk Assessment and Reduction" to align more closely with DOE safety plan guidance document format.
- Added section 4 "Equipment and Mechanical Integrity" to align more closely with DOE safety plan guidance document format.
- Added section 5 "Project Safety Documentation" to align more closely with DOE safety plan guidance document format.
- Updated CHEF background to include most recent history.
- Added gas shroud to "piping – line over pressurization" FMEA.
- Added calibration of Hy-Alerta sensor to mitigate the "piping – line breach" FMEA.
- Swapped the UPS battery failure with Fuel Cell backup not kicking on hypothetical situation in the FMEA.
- Updated P&ID:

- 2013 – 2015 (top):
- 2016 – 2019 (middle)
- 2019 – 20XX (bottom)







9.20. 2019-05-13 – UPDATED P&ID

Removed the flow recorder for the fuel cell, updated feedlines and bottle closet to 1/8" lines, added solenoid type valve on [31].

9.21. 2019-12-09 – FORMAT UPDATE

The chemical hygiene plan may be found on HYPERDRIVE (not laboratory website)

The Organizational Safety Information has been updated

Appendix Z has been added along with lab standard format header, footer and title page

Cross-referenced sections

Title page reflects NASA technical document layout (LAB-TYPE-EXPERIMENT-REVISION) (HYPER-SafteyPlan-CHEF-004)

Update 2.3.1 to NFPA 2020

Update HYPER Lab Experience – section 2.4

Chapter 3 is now called Identification of Safety Vulnerabilities (ISV)

Do a cross reference to all sections given the section moving

9.22. 2019-12-31 – P&ID AND HAZOP UPDATE

The P&ID reflects the most up to date CHEF pegboard layout to reflect more closely the physical layout of the system. There are dedicated supply line purge routes instead of activating the always active pressure relief system to achieve purge status.

The HAZOP is complete to have cross-referenced procedures.

9.23. 2019-1-15 – P&ID AND PROCEDURE UPDATE

The P&ID reflects a safer version of pressure relief on the primary stage. If the previous valve 9 is/was left in the high pressure select mode after a test (operator error) then there is a chance to exceed the 145 psig rating of the flow tracking alicat when the manual metering valve is closed or in the metering mode. This results in the elimination of the previous valve 9 and reassigned valve 9 to the gaseous hydrogen supply line purge. There is now a dedicate 145 psig pressure relief before the alicat.

9.24. 2019-1-16 – P&ID AND PROCEDURE UPDATE

The P&ID reflects the inclusion of pressure relief setpoints on the P&ID. There is also two check valves on the supply lines to be able to ensure no back filling can occur to potentially contaminate the supply cylinders.

APPENDICES

APPENDIX A: SUPPLEMENTARY DOCUMENTS

A.1 SAFETY DATA SHEET - HYDROGEN

ADVANCED SPECIALTY GASES®

Safety Data Sheet
Hydrogen

www.advancedspecialtygases.com

Section 1: Product and Company Identification

Advanced Specialty Gases
135 Catron Dr. Reno, NV 89512
775-356-5500

Product Code: Hydrogen

Section 2: Hazards Identification

Danger

Hazard Classification:
Flammable (Category 1)
Gases Under Pressure

Hazard Statements:
Contains gas under pressure; may explode if heated
Extremely flammable gas

Precautionary Statements
Prevention:
Keep away from heat/sparks/open flames/hot surfaces. - No smoking.

Response:
Eliminate all ignition sources if safe to do so.
Leaking gas fire: Do not extinguish, unless leak can be stopped safely.

Storage:
Protect from sunlight.
Store in well-ventilated place.

Section 3: Composition/Information on Ingredients

CAS #
1333-74-0

Chemical Substance	Chemical Family	Trade Names
HYDROGEN	inorganic, gas	HYDROGEN GAS; HYDROGEN COMPRESSED; HYDROGEN (H2); DIHYDROGEN; UN 1049; H2

Section 4: First Aid Measures

Skin Contact	Eye Contact	Ingestion	Inhalation	Note to Physicians
Wash exposed skin with soap and water.	Flush eyes with plenty of water.	If a large amount is swallowed, get medical attention.	If adverse effects occur, remove to uncontaminated area. Give artificial respiration if not breathing. If breathing is difficult, oxygen should be administered by qualified personnel. Get immediate medical attention.	For inhalation, consider oxygen.

Section 5: Fire Fighting Measures

Suitable Extinguishing Media	Products of Combustion	Protection of Firefighters
Carbon dioxide, regular dry chemical Large fires: Flood with fine water spray.	None known	▪ Any self-contained breathing apparatus with a full facepiece. ▪ Any self-contained breathing apparatus with a full facepiece.

Section 6: Accidental Release Measures

Personal Precautions	Environmental Precautions	Methods for Containment
Keep unnecessary people away, isolate hazard area and deny entry. Do not touch spilled material. Ventilate closed spaces before entering.	Avoid heat, flames, sparks and other sources of ignition.	Reduce vapors with water spray. Remove sources of ignition.

Methods for Cleanup	Other Information
Stop leak if possible without personal risk.	None

Section 7: Handling and Storage

Handling	Storage
Store and handle in accordance with all current regulations and standards. Grounding and bonding required. Subject to storage regulations: U.S. OSHA 29 CFR 1910.101.	Keep separated from incompatible substances.

Section 8: Exposure Controls/Personal Protection

Exposure Guidelines
HYDROGEN: ACGIH (simple asphyxiant)

Engineering Controls

Handle only in fully enclosed systems.

Eye Protection	Skin Protection	Respiratory Protection
Eye protection not required, but recommended.	Protective clothing is not required.	Any self-contained breathing apparatus with a full facepiece.

General Hygiene considerations

- Avoid breathing vapor or mist
- Avoid contact with eyes and skin
- Wash thoroughly after handling and before eating or drinking

Section 9: Physical and Chemical Properties

Physical State	Appearance	Color	Change in Appearance	Physical Form	Odor	Taste
Gas	Colorless	Colorless	N/A	Gas	Odorless	Tasteless

Flash Point	Flammability	Partition Coefficient	Autoignition Temperature	Upper Explosive Limits	Lower Explosive Limits
Flammable gas (burns at all ambient temperatures)	Not available	Not available	752 F (400 C)	0.75	0.04

Boiling Point	Freezing Point	Vapor Pressure	Vapor Density	Specific Gravity	Water Solubility	pH	Odor Threshold	Evaporation Rate	Viscosity
-423 F (-253 C)	-434 F (-259 C)	760 mmHg @ -253 C	0.07 (Air=1)	Not applicable	1.82% @ 20 C	Not applicable	Not available	Not applicable	0.008957 cP @ 26.8 C

Molecular Weight	Molecular Formula	Density	Weight per Gallon	Volatility by Volume	Volatility	Solvent Solubility
2	H2	0.08987 g/L @ 0 C	Not available	Not available	Not applicable	Soluble: Not available

Section 10: Stability and Reactivity

Stability	Conditions to Avoid	Incompatible Materials
Stable at normal temperatures and pressure.	Stable at normal temperatures and pressure.	Metals, oxidizing materials, metal oxides, combustible materials, halogens, metal salts, halo carbons, nitrogen triflouride, oxygen diflouride, magnesium and calcium carbonate, sodium, potassium

Hazardous Decomposition Products	Possibility of Hazardous Reactions
Miscellaneous decomposition products	Will not polymerize.

Section 11: Toxicology Information

Acute Effects

Oral LD50	Dermal LD50	Inhalation
Not available	Not available	Nausea, vomiting, difficulty breathing, irregular heartbeat, headache, fatigue, dizziness, disorientation, mood swings, tingling sensation, loss of coordination, convulsions, unconsciousness, coma

Eye Irritation	Skin Irritation	Sensitization
Not irritating	Not irritating	Difficulty breathing

Chronic Effects

Carcinogenicity	Mutagenicity	Reproductive Effects	Developmental Effects
Not available	Not available	Not available	No data

Section 12: Ecological Information

Fate and Transport

Eco toxicity	Persistence / Degradability	Bioaccumulation / Accumulation	Mobility in Environment
Fish toxicity: Not available	Not available	Not available	Not available

Invertibrate toxicity: Not available Algal toxicity: Not available Phyto toxicity: Not available Other toxicity: Not available			

Section 13: Disposal Considerations

Dispose in accordance with all applicable regulations. Subject to disposal regulations: U.S. EPA 40 CFR 262. Hazardous Waste Number(s): D001.

Section 14: Transportation Information

U.S. DOT 49 CFR 172.101

Proper Shipping Name	ID Number	Hazard Class or Division	Packing Group	Labeling Requirements	Passenger Aircraft or Railcar Quantity Limitations	Cargo Aircraft Only Quantity Limitations	Additional Shipping Description
Hydrogen, compressed	UN1049	2.1	Not applicable	2.1	Forbidden	150 kg	None

Canadian Transportation of Dangerous Goods

Shipping Name	UN Number	Class	Packing Group / Risk Group
Hydrogen, compressed	UN1049	2.1	Not applicable

Section 15: Regulatory Information

U.S. Regulations

CERCLA Sections	SARA 355.30	SARA 355.40
Not regulated.	Not regulated.	Not regulated.

SARA 370.21

Acute	Chronic	Fire	Reactive	Sudden Release
Yes	No	Yes	No	Yes

SARA 372.65

Not regulated.

OSHA Process Safety

Not regulated.

State Regulations

CA Proposition 65
Not regulated.

Canadian Regulations

WHMIS Classification
A, B1.

National Inventory Status

US Inventory (TSCA)	TSCA 12b Export Notification	Canada Inventory (DSL/NDSL)
Listed on inventory.	Not listed.	Listed on inventory.

Section 16: Other Information

NFPA Rating
HEALTH=0 FIRE=4 REACTIVITY=0

0 = minimal hazard, 1 = slight hazard, 2 = moderate hazard, 3 = severe hazard, 4 = extreme hazard

A.2 SAFETY DATA SHEET - HELIUM

ADVANCED SPECIALTY GASES®

Safety Data Sheet
Helium

www.advancedspecialtygases.com

Section 1: Product and Company Identification

Advanced Specialty Gases
135 Catron Dr. Reno, NV 89512
775-356-5500

Product Code: Helium

Section 2: Hazards Identification

Warning

Hazard Classification:
Gases Under Pressure

Hazard Statements:
Contains gas under pressure; may explode if heated

Precautionary Statements

Storage:
Protect from sunlight.
Store in well-ventilated place.

Section 3: Composition/Information on Ingredients

CAS #
7440-59-7

Chemical Substance	Chemical Family	Trade Names

Chemical Substance	Chemical Family	Trade Names
HELIUM	inorganic, gas	HELIUM GAS; HELIUM COMPRESSED; HELIUM-4; ATOMIC HELIUM; UN 1046; He

Section 4: First Aid Measures

Skin Contact	Eye Contact	Ingestion	Inhalation	Note to Physicians
Wash exposed skin with soap and water.	Flush eyes with plenty of water.	If a large amount is swallowed, get medical attention.	If adverse effects occur, remove to uncontaminated area. Give artificial respiration if not breathing. If breathing is difficult, oxygen should be administered by qualified personnel. Get immediate medical attention.	For inhalation, consider oxygen.

Section 5: Fire Fighting Measures

Suitable Extinguishing Media	Products of Combustion	Protection of Firefighters
Non-flammable. Use suitable extinguishing media for surrounding fire.	Non-flammable	▪ Non-flammable ▪ Non-flammable

Section 6: Accidental Release Measures

Personal Precautions	Environmental Precautions	Methods for Containment
Keep unnecessary people away, isolate hazard area and deny entry. Stay upwind and keep out of low areas.	Avoid soil, waterways, drains and sewers	Stop leak if possible without personal risk.

Methods for Cleanup	Other Information
Stop leak, evacuate area. Contact emergency personnel.	None

Section 7: Handling and Storage

Handling	Storage
Store and handle in accordance with all current regulations and standards. Subject to storage regulations: U.S. OSHA 29 CFR 1910.101.	Keep separated from incompatible substances.

Section 8: Exposure Controls/Personal Protection

Exposure Guidelines
HELIUM: ACGIH (simple asphyxiant)

Engineering Controls

Handle only in fully enclosed systems.

Eye Protection	Skin Protection	Respiratory Protection
Eye protection not required, but recommended.	Protective clothing is not required.	Non-flammable

General Hygiene considerations

- Avoid breathing vapor or mist
- Avoid contact with eyes and skin
- Wash thoroughly after handling and before eating or drinking

Section 9: Physical and Chemical Properties

Physical State	Appearance	Color	Change in Appearance	Physical Form	Odor	Taste

Physical State	Appearance	Color	Change in Appearance	Physical Form	Odor	Taste
Gas	Colorless	Colorless	N/A	Gas	Odorless	Tasteless

Flash Point	Flammability	Partition Coefficient	Autoignition Temperature	Upper Explosive Limits	Lower Explosive Limits
Not flammable	Not available	Not available	Nonflammable	Nonflammable	Nonflammable

Boiling Point	Freezing Point	Vapor Pressure	Vapor Density	Specific Gravity	Water Solubility	pH	Odor Threshold	Evaporation Rate	Viscosity
-452 F (-269 C)	-458 F (-272 C) @ 26 atm	1719 mmHg @ -268 C	0.138 (Air=1)	Not applicable	0.94% @ 0 C	Not applicable	Not available	Not applicable	0.02012 cP @ 26.8 C

Molecular Weight	Molecular Formula	Density	Weight per Gallon	Volatility by Volume	Volatility	Solvent Solubility
4.0026	He	0.1785 g/L @ 0 C	Not available	100%	Not applicable	Insoluble: Not available

Section 10: Stability and Reactivity

Stability	Conditions to Avoid	Incompatible Materials
Stable at normal temperatures and pressure.	Stable at normal temperatures and pressure.	No data available.

Hazardous Decomposition Products	Possibility of Hazardous Reactions
Miscellaneous decomposition products	Will not polymerize.

Section 11: Toxicology Information

Acute Effects

Oral LD50	Dermal LD50	Inhalation
Not available	Not available	Nausea, vomiting, difficulty breathing, irregular heartbeat, headache, fatigue, dizziness, disorientation, emotional disturbances, tingling sensation, loss of coordination, suffocation, convulsions, unconsciousness, coma

Eye Irritation	Skin Irritation	Sensitization
Liquid: frostbite, blurred vision	Liquid: frostbite	Difficulty breathing

Chronic Effects

Carcinogenicity	Mutagenicity	Reproductive Effects	Developmental Effects
Not available	Not available	Not available	No data

Section 12: Ecological Information

Fate and Transport

Eco toxicity	Persistence / Degradability	Bioaccumulation / Accumulation	Mobility in Environment
Fish toxicity: Not available **Invertibrate toxicity: Not available** **Algal toxicity: Not available** **Phyto toxicity: Not available** **Other toxicity: Not available**	Not available	Not available	Not available

Section 13: Disposal Considerations

Dispose in accordance with all applicable regulations.

Section 14: Transportation Information

U.S. DOT 49 CFR 172.101

Proper Shipping Name	ID Number	Hazard Class or Division	Packing Group	Labeling Requirements	Passenger Aircraft or Railcar Quantity Limitations	Cargo Aircraft Only Quantity Limitations	Additional Shipping Description
Helium, compressed	UN1046	2.2	Not applicable	2.2	75 kg or L	150 kg	N/A

Canadian Transportation of Dangerous Goods

Shipping Name	UN Number	Class	Packing Group / Risk Group
Helium, compressed	UN1046	2.2	Not applicable

Section 15: Regulatory Information

U.S. Regulations

CERCLA Sections	SARA 355.30	SARA 355.40
Not regulated.	Not regulated.	Not regulated.

SARA 370.21

Acute	Chronic	Fire	Reactive	Sudden Release
Yes	No	No	No	Yes

SARA 372.65

Not regulated.

OSHA Process Safety

Not regulated.

State Regulations

CA Proposition 65
Not regulated.

Canadian Regulations

WHMIS Classification
A

National Inventory Status

US Inventory (TSCA)	TSCA 12b Export Notification	Canada Inventory (DSL/NDSL)
Listed on inventory.	Not listed.	Not determined.

Section 16: Other Information

NFPA Rating
HEALTH=0 FIRE=0 REACTIVITY=0

0 = minimal hazard, 1 = slight hazard, 2 = moderate hazard, 3 = severe hazard, 4 = extreme hazard

APPENDIX Z: RECORD OF ACCEPTANCE

Z.1 STATEMENT OF ACCEPTANCE

By signing the record sheet below corresponding to the year of acceptance, I certify that I have reviewed this document with all relevant stakeholders and that it meets HYPER Lab standards of Safety and Professionalism. To the best of my knowledge, this document is an accurate representation of the system as it currently exists and the procedures in place to ensure the system is operated safely. This system and these procedures have been developed to the best of the HYPER Lab's ability to meet all applicable codes in the Code of Federal Regulations, the Washington Administrative Codes, and WSU Policies and Procedures; as well as industry standards and best practices. As Lab Director, or other such authority as having been designated by the Lab Director, I accept this document to be up to date and meeting all requirements of the HYPER Lab.

Z.2 RECORD

Signed as of the date listed below the signature line.

X ______________________________

Date: / /2020. Reviewed and accepted in 2020.

X ______________________________

Date: / /2021. Reviewed and accepted in 2021.

X ______________________________

Date: / /2022. Reviewed and accepted in 2022.

X ______________________________

Date: / /2023. Reviewed and accepted in 2023.