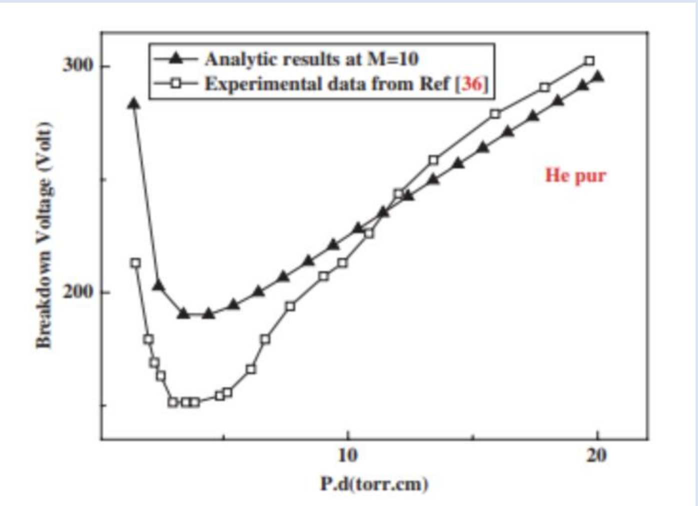
Hydrogen Sensor via Plasma Techniques Development

Sarah Feldman[a], Kenneth Engeling, PhD.[b], Carolina Franco, PhD.[b], Nicholas Spangler[c], and Jessica Schwend[b]

Motivation

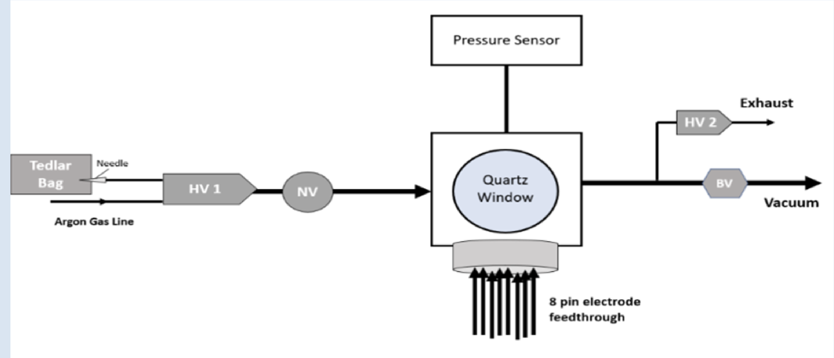
- Liquid hydrogen (LH2) is frequently used as rocket fuel for propulsion systems → consistent venting typically required for storage + high risk of leaking through the small pores formed in welds and joints
- Requires H2 sensors with quick response, highy sensitivity and selective detection are of great importance for processing launch vehicles utilizing LH2/O2 fuel-oxidizer propellant systems
- This work aims to produce an anaerobic (oxygen-less) sensor for the detection and quantification of hydrogen gas at varying concentrations and pressures for use in launch facility infrastructure with potential further employment via hydrogen plasma technology.
- There is no direct correlation to the concentration of hydrogen and intensity across a pressure range → The resulting plasma is dependent on pressure and therefore emission intensity, all detectable via spectroscopy



Paschen curve for the theoretical and measured breakdown of a helium gas discharge. [3, 4]

Optical Emission Spectroscopy Testing

- Used a 1.33 inch conflat flange 6 way cube with a window, an 8-pin feedthrough for plasma, a Granville convection-enhanced Pirani vacuum gauge pressure sensor, and Swagelok feedthroughs for gas input/output, eventually switched to VCR
- The cube connected to a roughing pump and a turbomolecular pump, achieving overall 10 mTorr/minute leak rate → still caused air interference due to sensitivity



Schematic of experimental Setup

Image of the custom fit fiber optic mount that covered the 1.33" conflat flange

Discharge from plasma source inside conflat flange 6-way cube

- Gas mixtures were made using a series of serial dilutions with gas syringes and tedlar sampling bags
- Early tests used hydrogen in a helium (He) background, starting with 2% H2 in He from a pre-mixed k-Bottle → He determined not to be a good background gas due to interfering spectrum with hydrogen, moved to argon (Ar) background as a result

H_2 Concentration	Dilution Mixture	
50,000 ppm	Obtained from K-Bottle	
25,000 ppm	700 ml 50,000 ppm H_2 in Ar	700 ml Ar
10,000 ppm	500 ml 50,000 ppm H_2 in Ar	2000 ml Ar
1000 ppm	400 ml 25,000 ppm H_2 in Ar	600 ml Ar
500 ppm	200 ml 1000 ppm H_2 in Ar	1800 ml Ar
50 ppm	9 ml 500 ppm H_2 in Ar	900 ml Ar

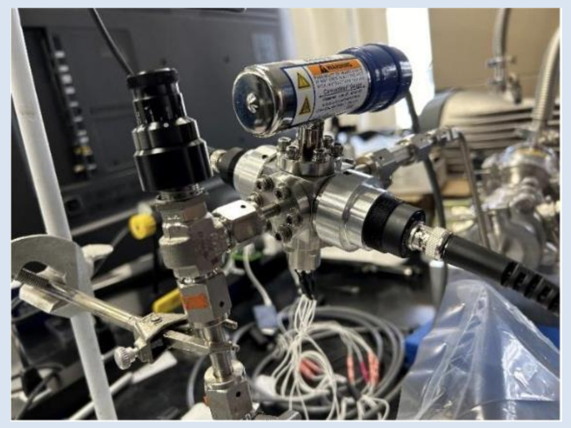
Serial dilution method for hydrogen concentrations

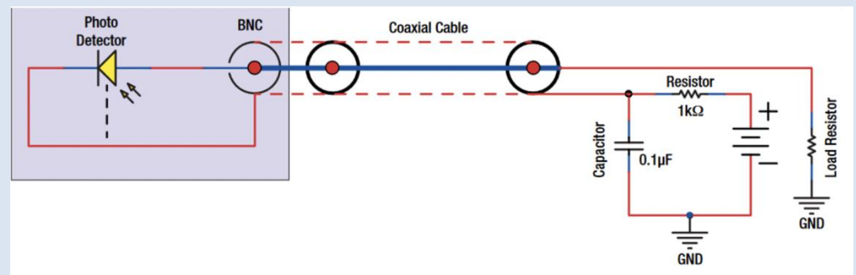
Gas syringe and tedlar bag used for gas dilutions

- Plasma source was switched from standard high voltage AC to a commercially available, small footprint, rechargeable, battery powered plasma lighter
- Tested with a Stellarnet Black Comet OES Spectrometer and UV-VIS fiber optic with a 184-847 nm range, data obtained using Stellar Net provided software SpectraWiz

Photodiode Testing

- Identical test setup to OES system with second window
- ThorLabs bandpass filter with a center wavelength at 488 nm and FWHM of 10 nm and an Edmund Optics bandpass filter with a center wavelength at 656 nm and a FWHM of 10 nm
- Prototype circuit board would apply a reverse bias to the diode and carry the data to an HDO4034A 350 MHz oscilloscope for visualization and processing

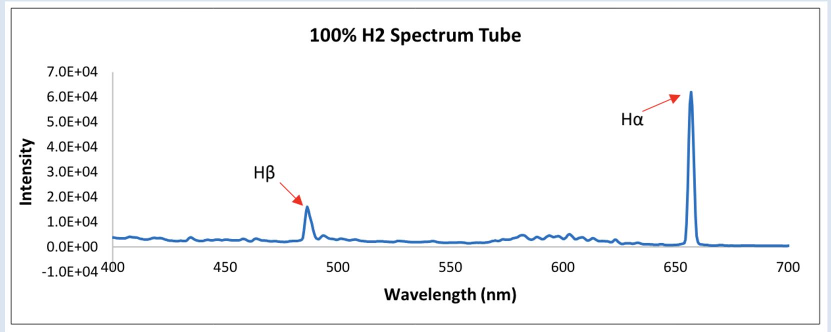




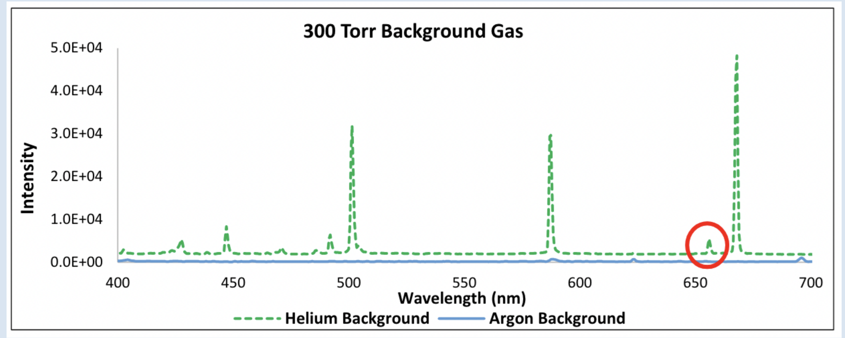
Above: Custom photodiode-feedthroughs
Left: Circuit diagram to control photodiodes

Results & Discussion

- Hydrogen spectra has 2 predominant emission lines in UV-VIS (Balmer series): 656 nm (Hα) and 486 nm (Hβ) → ratio used to account for variabilities in plasma brightness
- 100% hydrogen yielded visible peaks and Hα/Hβ=4.31, above LOD and LOQ
- LOD and LOQ (limit of detection/quantification) were determined using 10 argon blanks, average taken; LOD is + 3σ and LOQ is +10σ

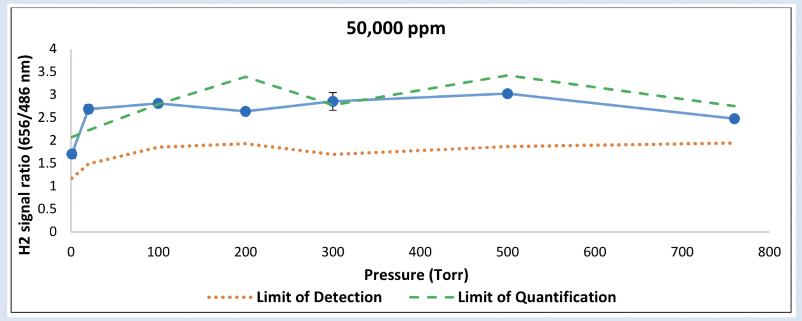


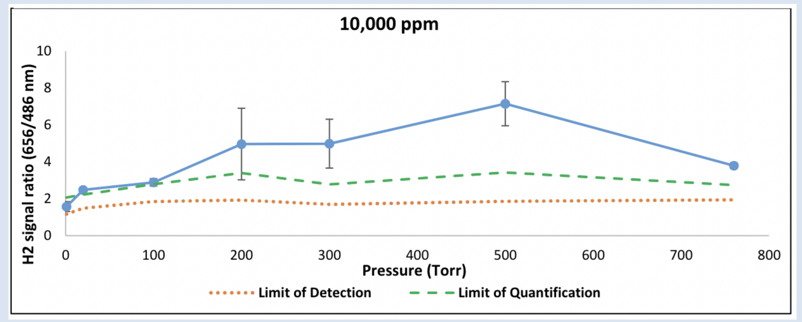
100% hydrogen vapor plasma optical emission spectrum taken from the commercial spectrum tube discharge

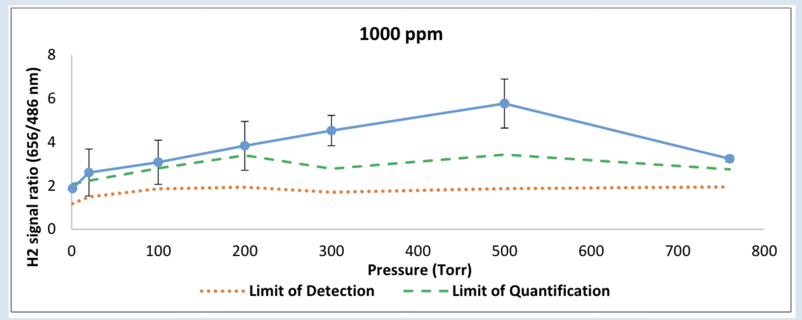


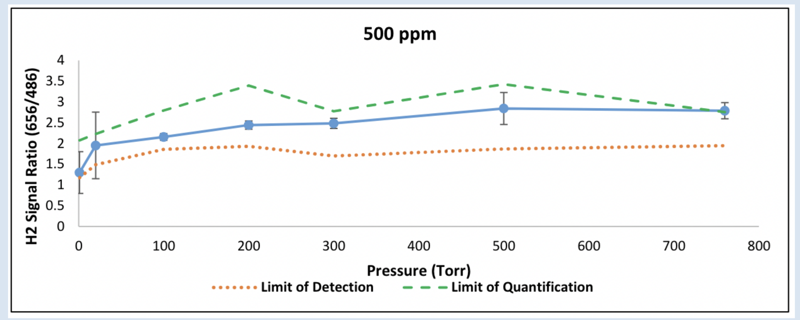
Helium vs. argon background spectrum at 300 Torr

- From 50,000 ppm to 50 ppm, the hydrogen signal ratio was above the limit of detection of the setup; exceptions at 1 Torr and 20 Torr
- Magnitude of H2 signal ratio was found not to correspond to the concentration of hydrogen, rather just serve as a qualitative indicator of hydrogen presence

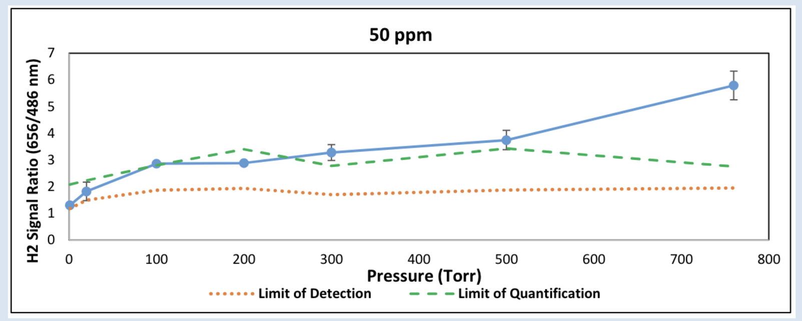








Hydrogen signal ratio over various pressures and H2 concentrations (in ppm) using OES. Error is shown as ± standard deviation



Conclusions & Future Work

Key Performance Parameters (KPPs)	Threshold	Goal
KPP 1: Required detection limits	SLS Requirements (<100 ppm O_2 environment and 0.1-10% H_2 range)	< 10 ppm O_2 and 0.01-10% H_2 range
KPP 2: Anaerobic operation	Effective operation and detection in anaerobic conditions	
KPP1 Results	Success	Success
KPP2 Results	Successful operation and detection in anaerobic conditions	

Key Performance Parameters determined during the proposal stage of the project

- Tests with mixtures of 70 torr H2 or 10% down to 0.07 torr H2 or 0.01% was successfully achieved
- Brought from TRL 2 to TRL 3
- Future work would include a more appropriate power source to generate an intense enough plasma for successful analysis of the atomic Boltzmann plot method and the two-line ratio method
- Would also need to investigate positive pressure environments and produce a functional prototype for field testing

References

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(a) University of Michigan (sarfeldm@umich.edu)
(b) NASA Kennedy Space Center Applied Chemistry Laboratory (kenneth.engeling@nasa.gov, carolina.franco@nasa.gov, jessica.schwend@nasa.gov)
(c) NASA Kennedy Space Center Applied Physics Laboratory (nicholas.b.spangler@nasa.gov)

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