Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

We put science to work.™

The manufacturer of the Diffuser Is Power & Energy, same as the Old Vendor.
SRNL - Chose to lie about the Vendor. Why?

Characterization of a Pd-Ag Diffuser from a New Vendor

Gregg A. Morgan

Brittany J. Hodge

38th Tritium Focus Group, Pacific Northwest National Laboratory
May 9-11, 2017

SRNL-STI-2017-00291

Savannah River NUCLEAR SOLUTIONS℠
FLUOR • NEWPORT NEWS NUCLEAR • HONEYWELL

Outline

- **Background and Introduction**
- **Design of Pd-Ag Diffuser**
- **Experimental Design and Set-Up**
- **Results**
  - *Residual Gas Analysis Spectra*
  - *Bleed Flow Rate vs. Total Feed Flow Rate Curves*
  - *Average Shell Pressure vs. Total Feed Flow Rate Curves*
  - *Amount of H_2 in Bleed Stream vs. Total Feed Flow Rate Curves*
- **Summary and Conclusion**
- **Acknowledgments**

Introduction

- **Pd-Ag diffusers in the tritium gas processing system separate hydrogen isotopes from various inert species in the process stream**

- **Given the importance of Pd-Ag diffusers in typical tritium processing facilities, it is imperative to have multiple vendors for key pieces of process equipment**

- **Over the last several years, SRNL has been heavily involved in the identification of potential vendors for the manufacture of tritium compatible Pd-Ag diffusers**

- **Potential Challenges of Finding a Vendor:**
  - **Limited number of domestic vendors**
  - **Pd-Ag diffusers are typically custom components based on flow rates and pressures**
  - **Long Lead times**
  - **Tritium service requirements**

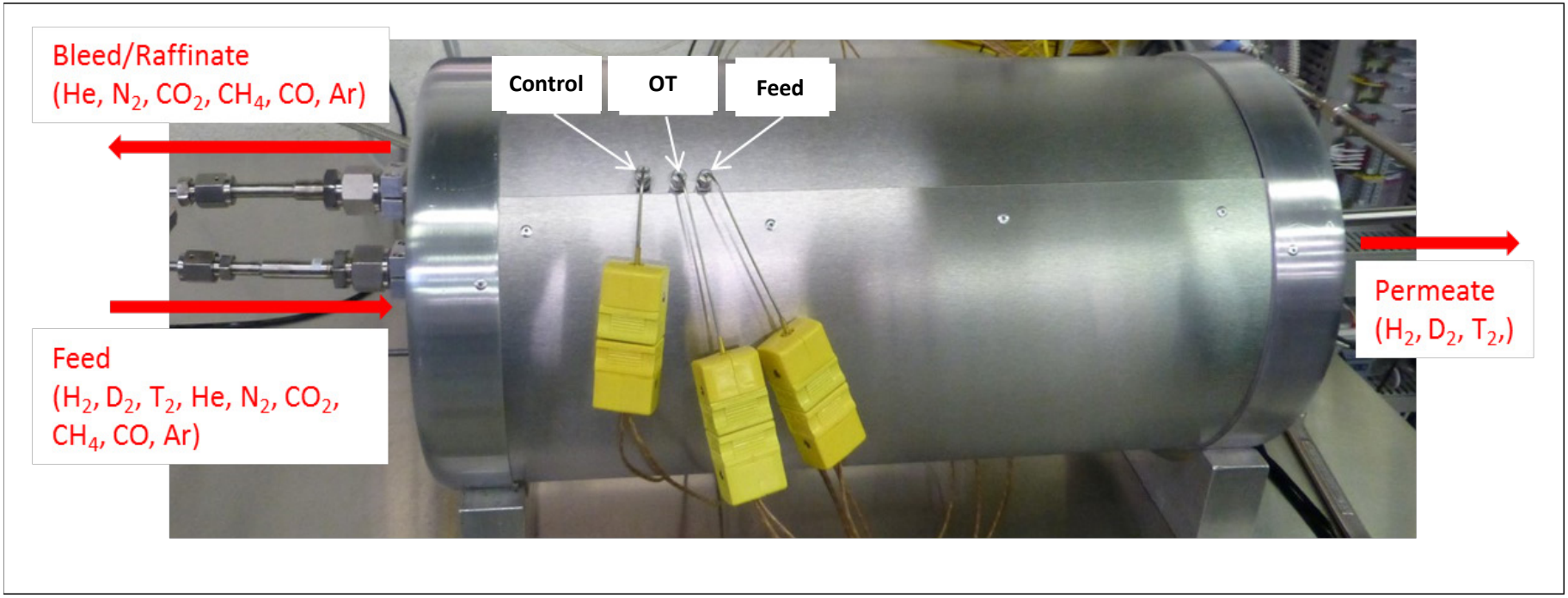
Objective

- **Each diffuser manufacturer uses a different design and each design will most likely give different separation characteristics, even when operated at the same temperature, pressure, and flow composition and rate**

- **It is necessary to characterize the diffuser over a range of operating conditions to determine the optimal operating conditions**

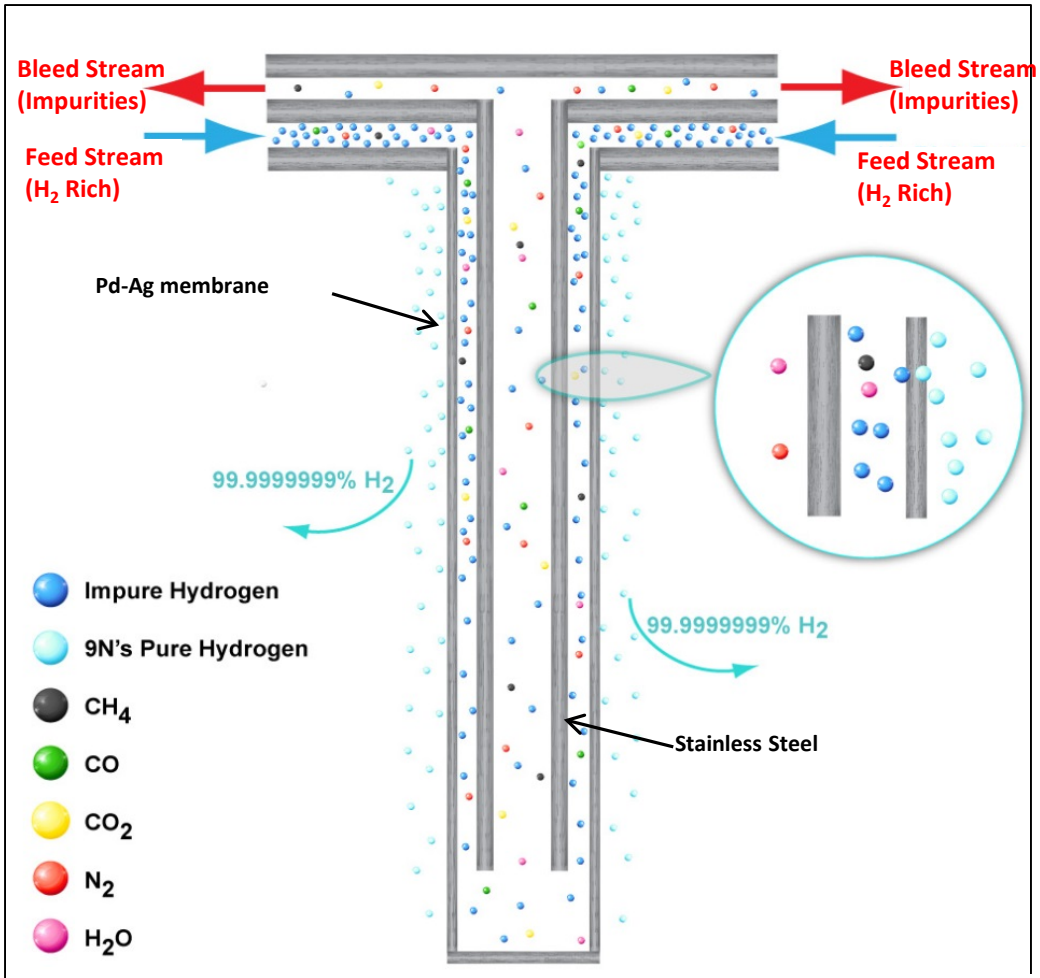
- **The purpose of this test is to determine the hydrogen removal efficiency of Pd-Ag diffusers procured from a new vendor.**

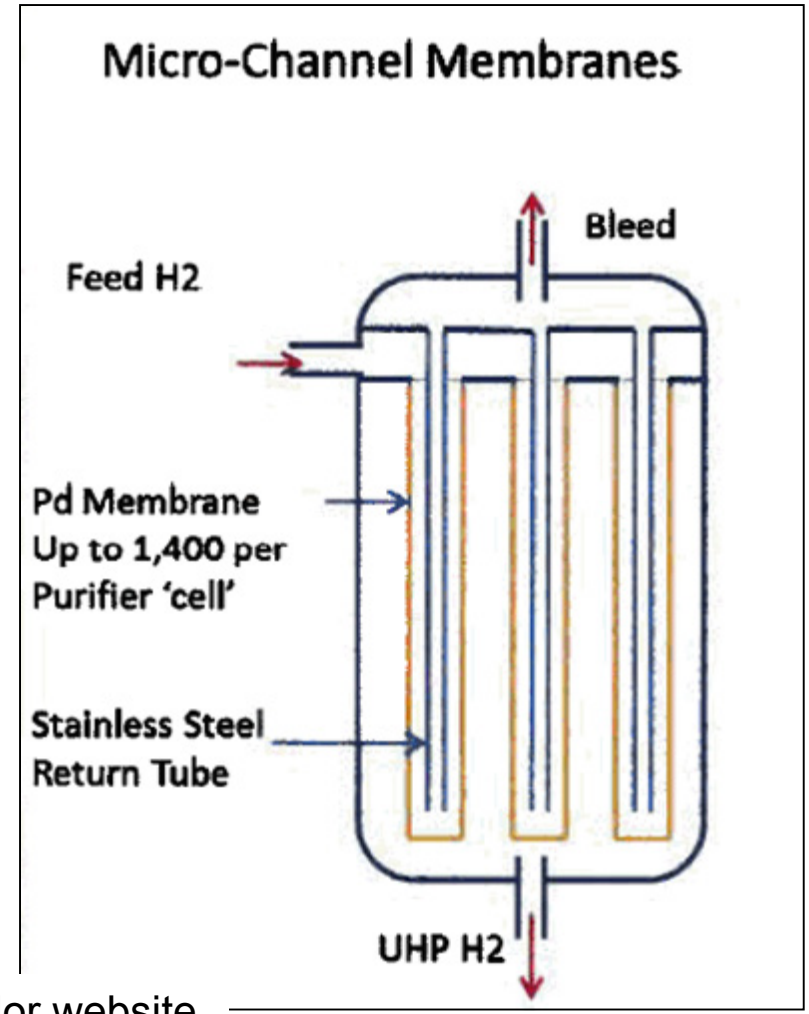
Background and Introduction



The H_2 in the feed gas permeates through the wall of the Pd-Ag tubing into the lower pressure cavity of the diffuser shell and is known as the permeate stream. The non-hydrogen gases of the feed stream continue through the tubes and discharge to the bleed or raffinate stream. This type of diffuser is known as an "inside-out".

Design of New Pd-Ag Diffuser

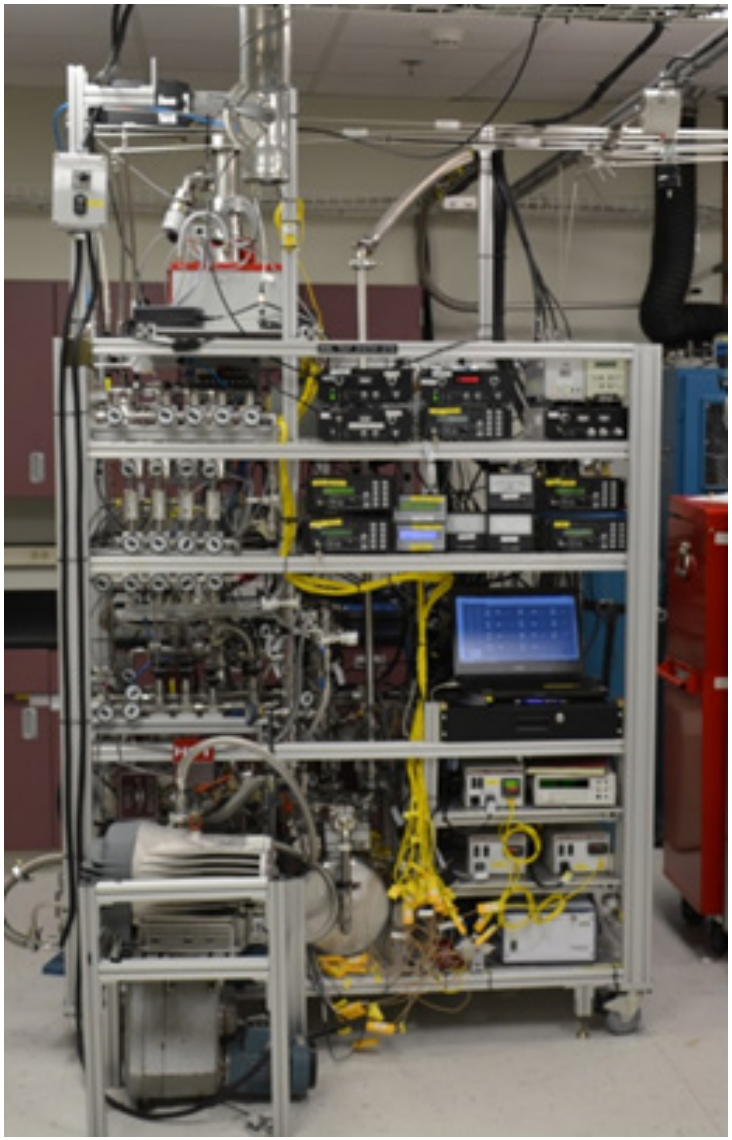




Images taken from vendor website

The Pd-Ag diffuser uses a micro-channel membrane technology. This particular Pd-Ag diffuser contains greater than 200 in^2 of Pd-Ag surface area.

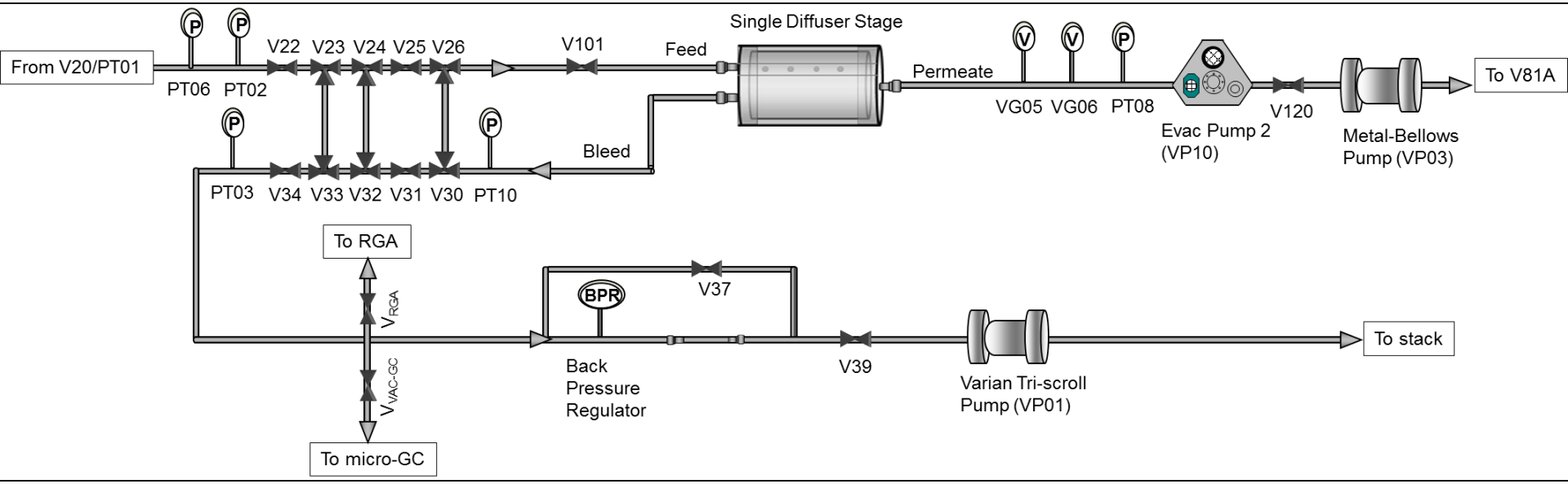
Experimental Manifold



20 SLPM test system

- Mass flow controllers up to 20 SLPM
- Mass flow meters to measure the permeate
- Pressure transducers
- Back pressure regulators
- Dycor Dymaxion DM200M Residual gas Analyzer (RGA)
- Inficon 3000 micro gas chromatograph
  - Dual molecular sieve columns
  - helium and argon carrier gas

System Schematic with Pd-Ag Diffuser

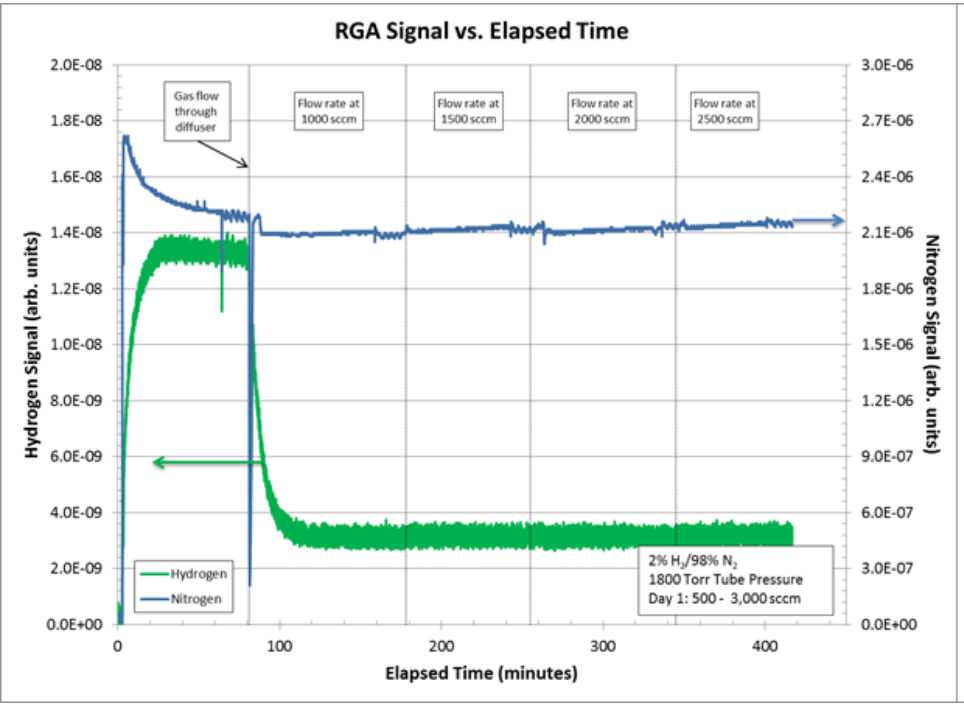


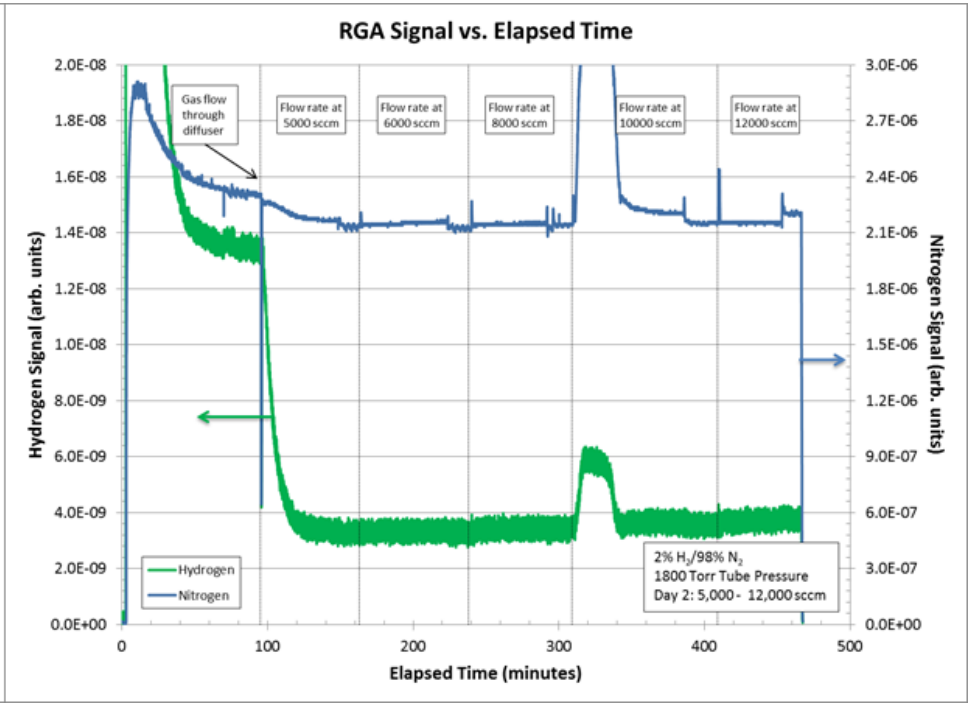
The permeate stage of the Pd-Ag diffuser is pumped by a Normetex Model 15 scroll pump , backed by a Metal Bellows 602 with the two heads connected in series.

Experimental Test Parameters

- Gas Compositions from 2% H_2 to 96% H_2
  - 2%, 25%, 50%, 75%, 96% H_2
  - Balance of nitrogen or helium
- Flow Rates from 500 sccm to 14000 sccm
- Internal Tube Pressures of 800 torr and 1800 torr
  - Manually maintained using a manual back pressure regulator
  - Able to maintain pressure to set pressure ± 10 torr
  - Adjustment of pressure at each flow rate
- Bleed gas analyzed by RGA and μ-gas chromatography
- Permeate flow rate measured by flow meters

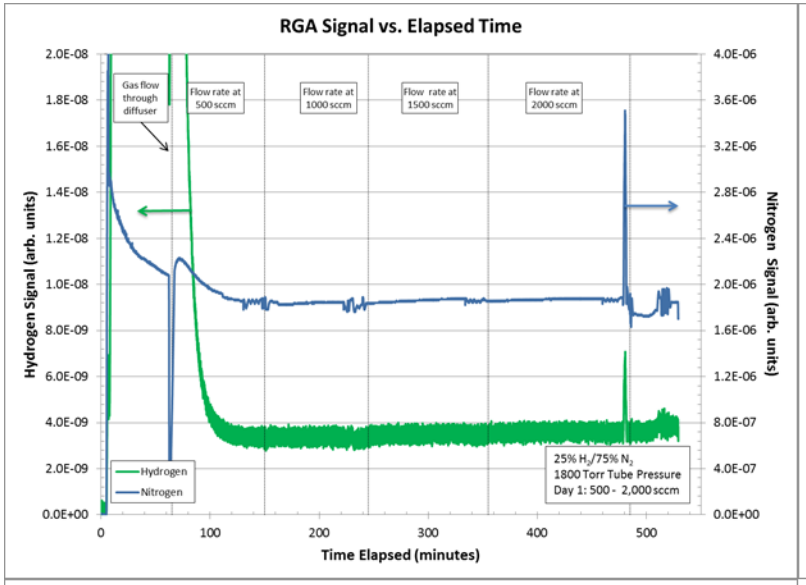
RGA Spectra for 2% H_2 - 98% N_2

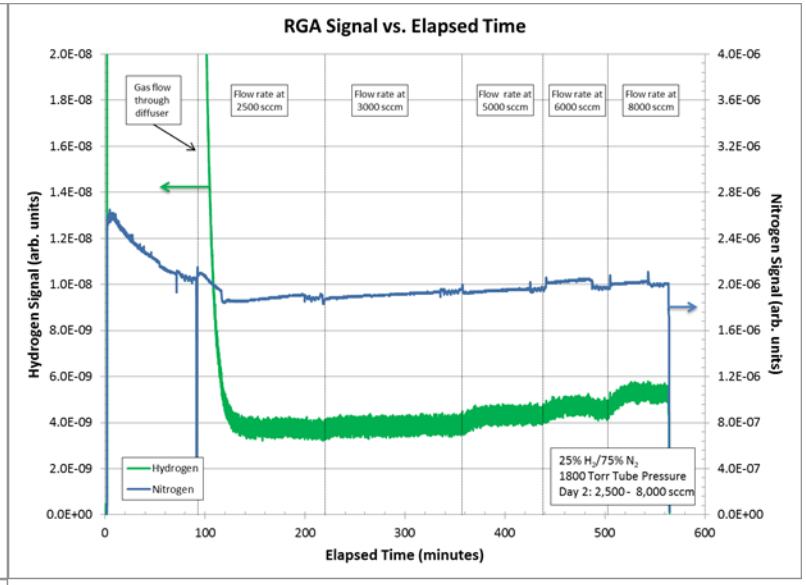


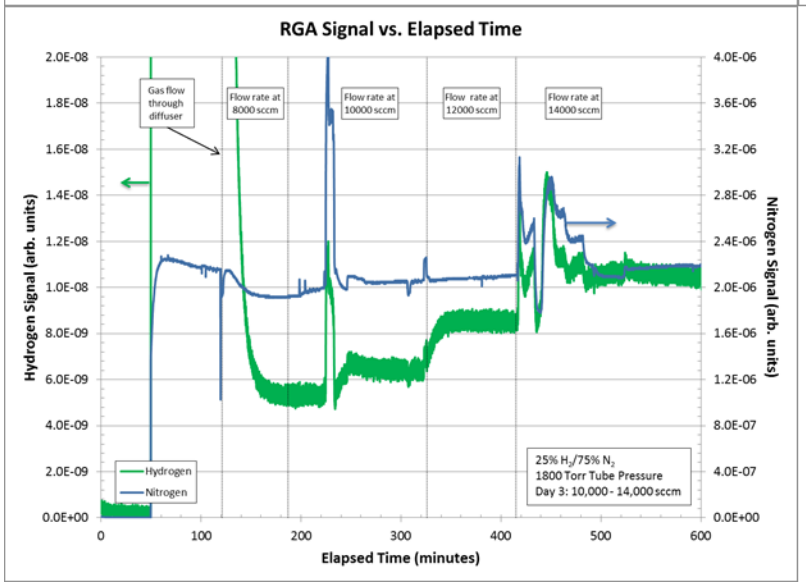


The RGA spectra above indicate that there is a small amount (barely detectable) of hydrogen present in the bleed stream. As the flow rate increases there is a very slight increase in the hydrogen signal in the RGA.

RGA Spectra for 25% H_2 - 75% N_2

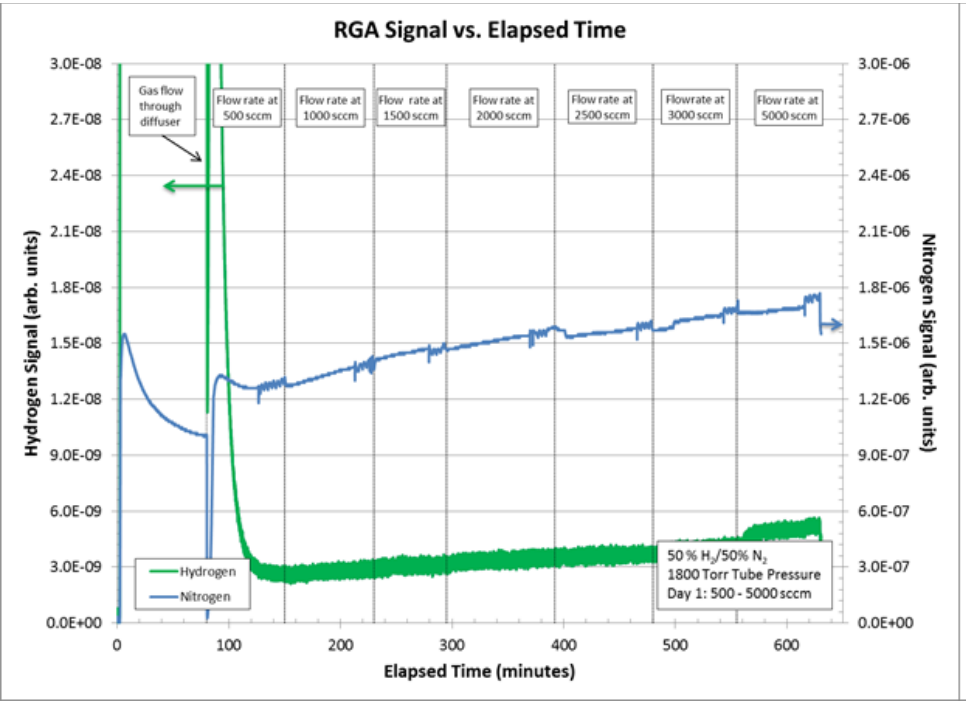


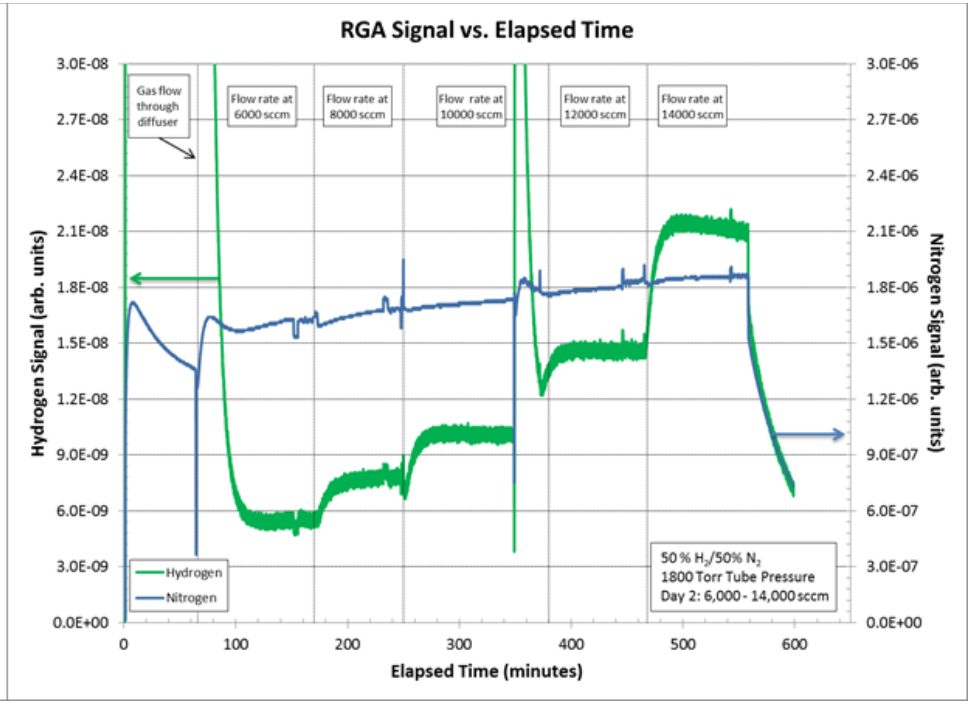




The top left figure indicates that the hydrogen in the bleed is barely detectable at flow rates up to 2000 sccm. As the flow rate is increased a corresponding step-wise increase in the hydrogen signal is observed in the other two figures.

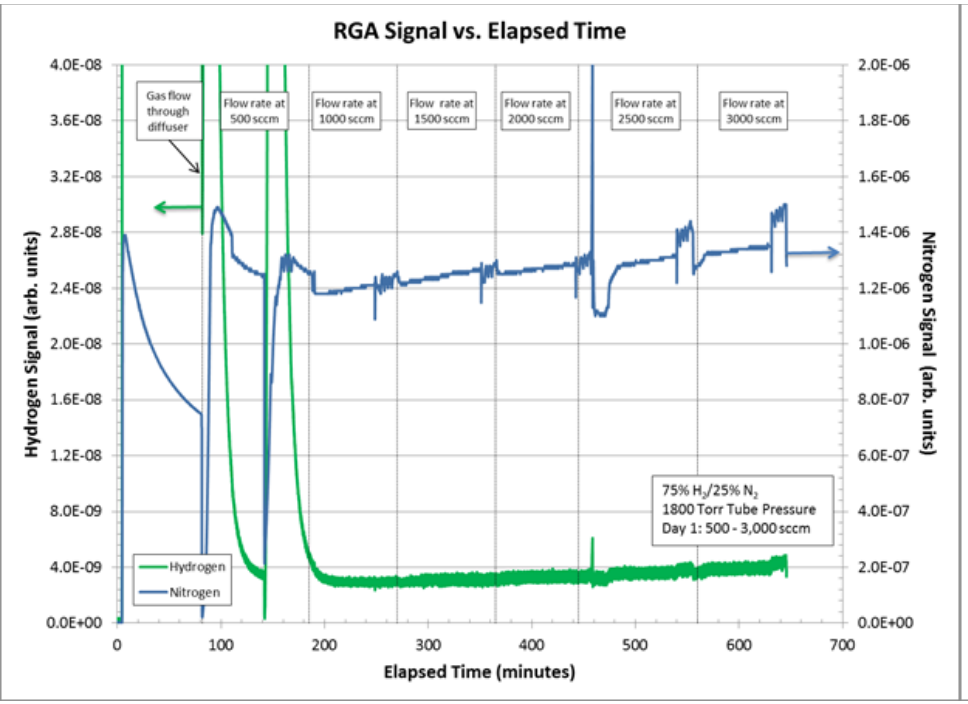
RGA Spectra for 50% H_2 - 50% N_2

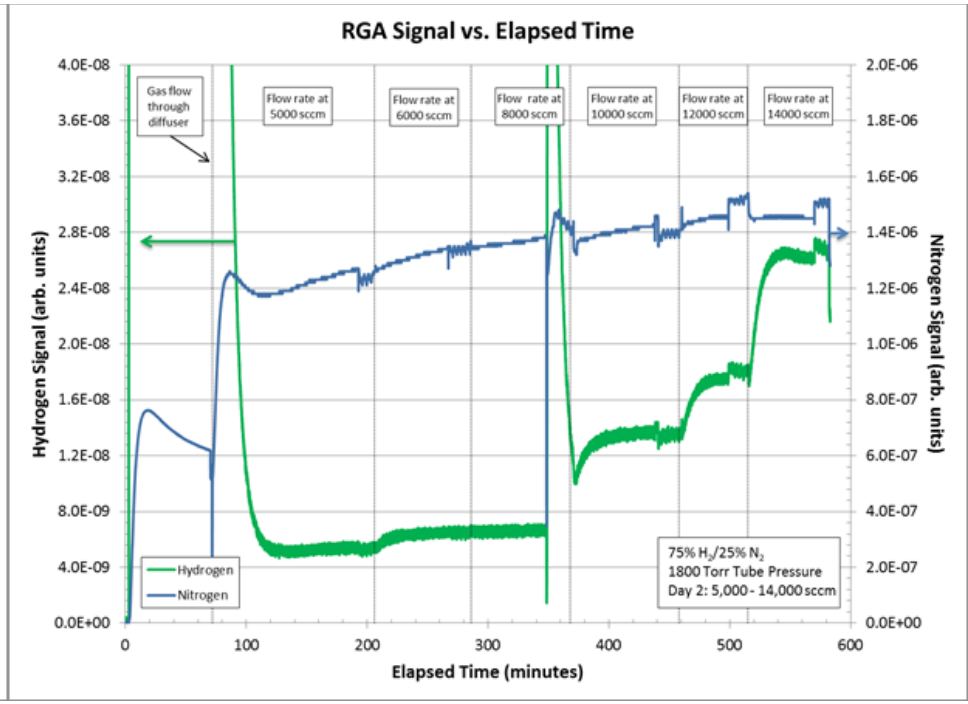




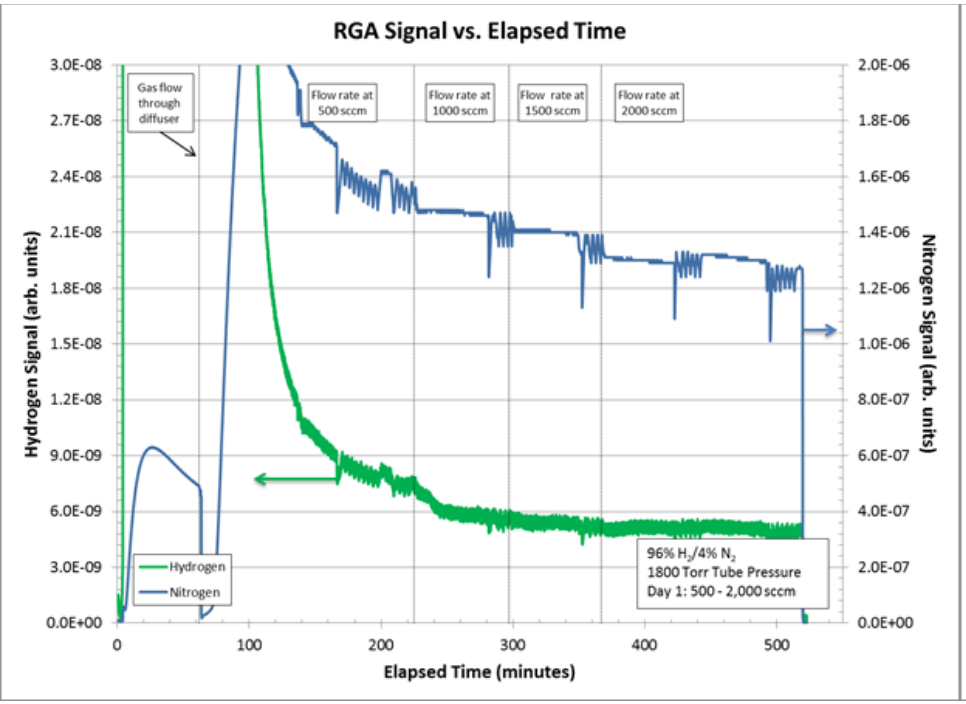
As the amount of hydrogen increases in the feed stream, the amount of hydrogen in the bleed stream increases as well. This is indicated by the slightly steeper slope and larger steps at each increase in the flow rate.

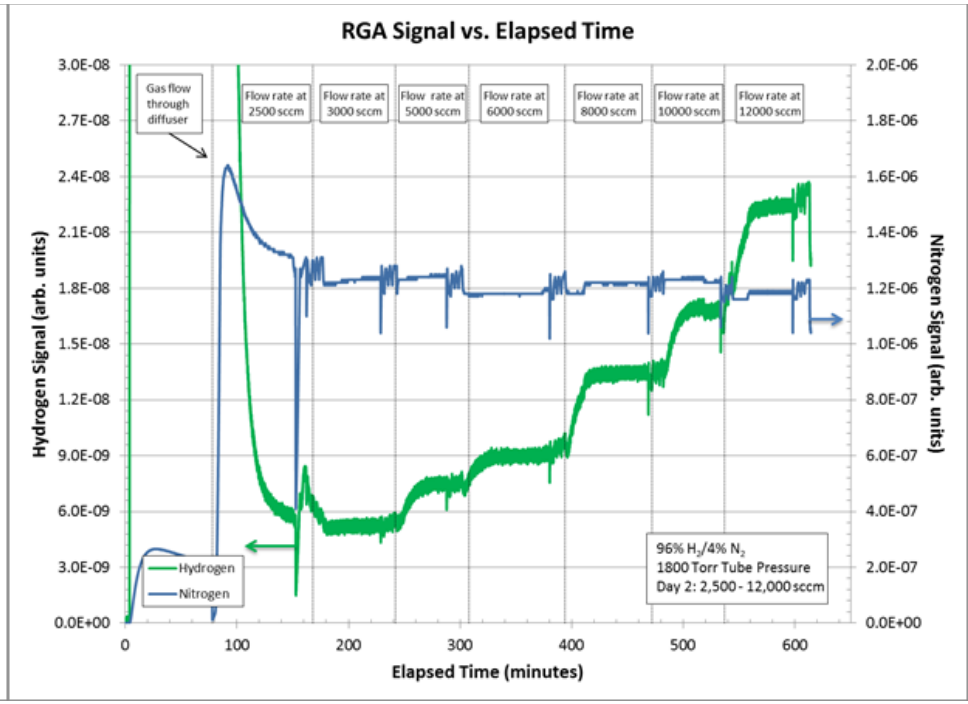
RGA Spectra for 75% H_2 - 25% N_2



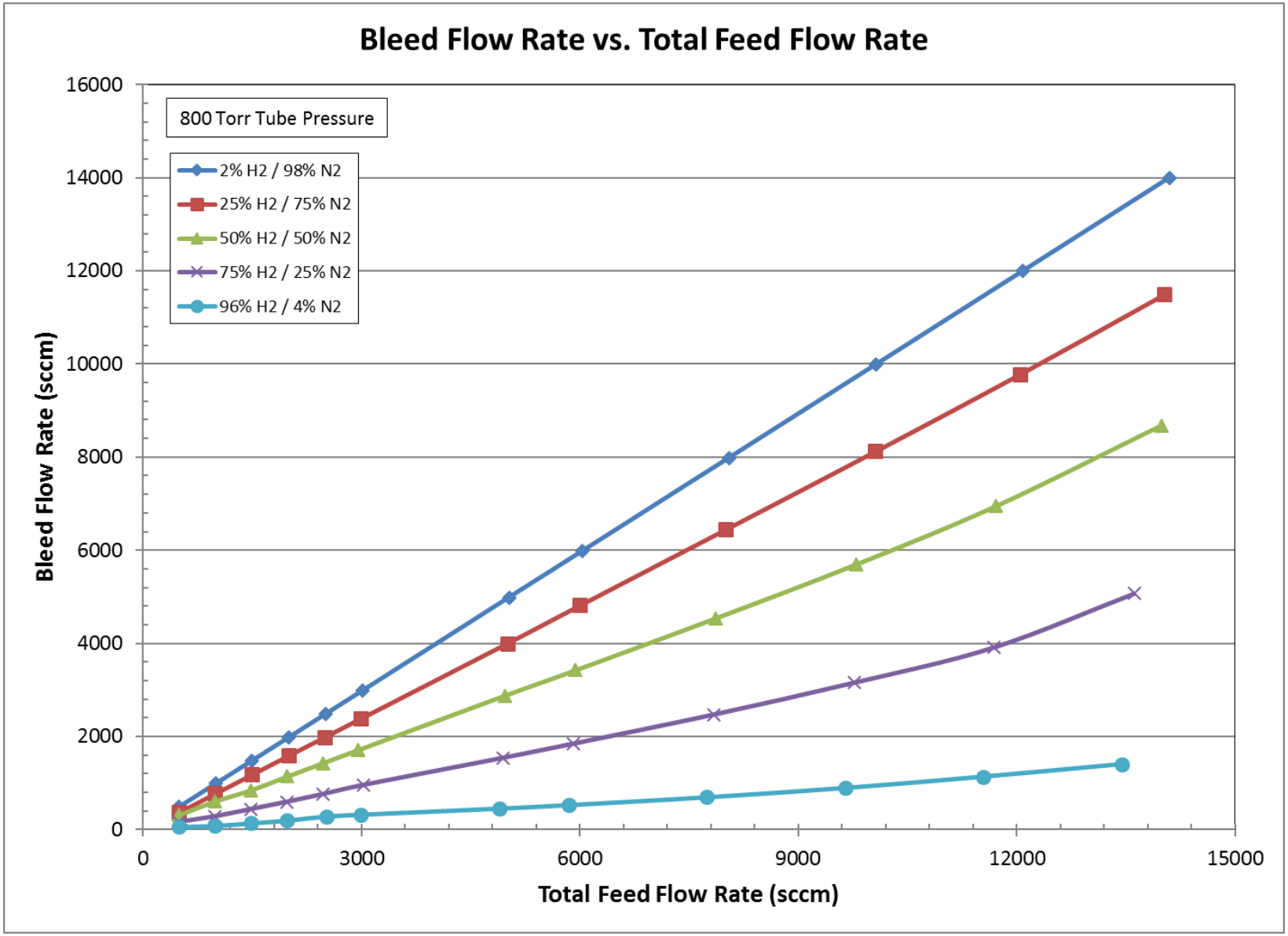


RGA Spectra for 96% H_2 - 4% N_2

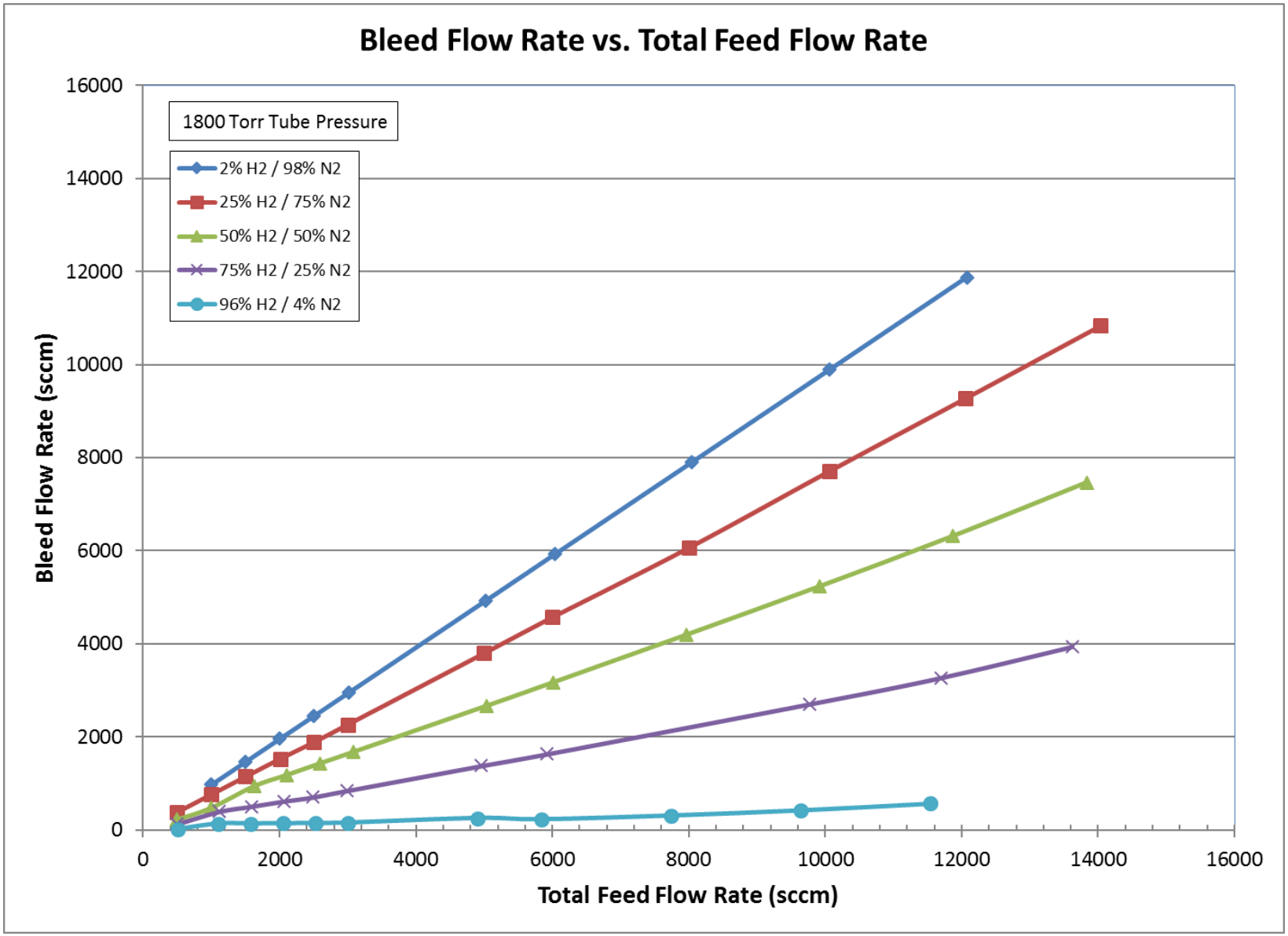




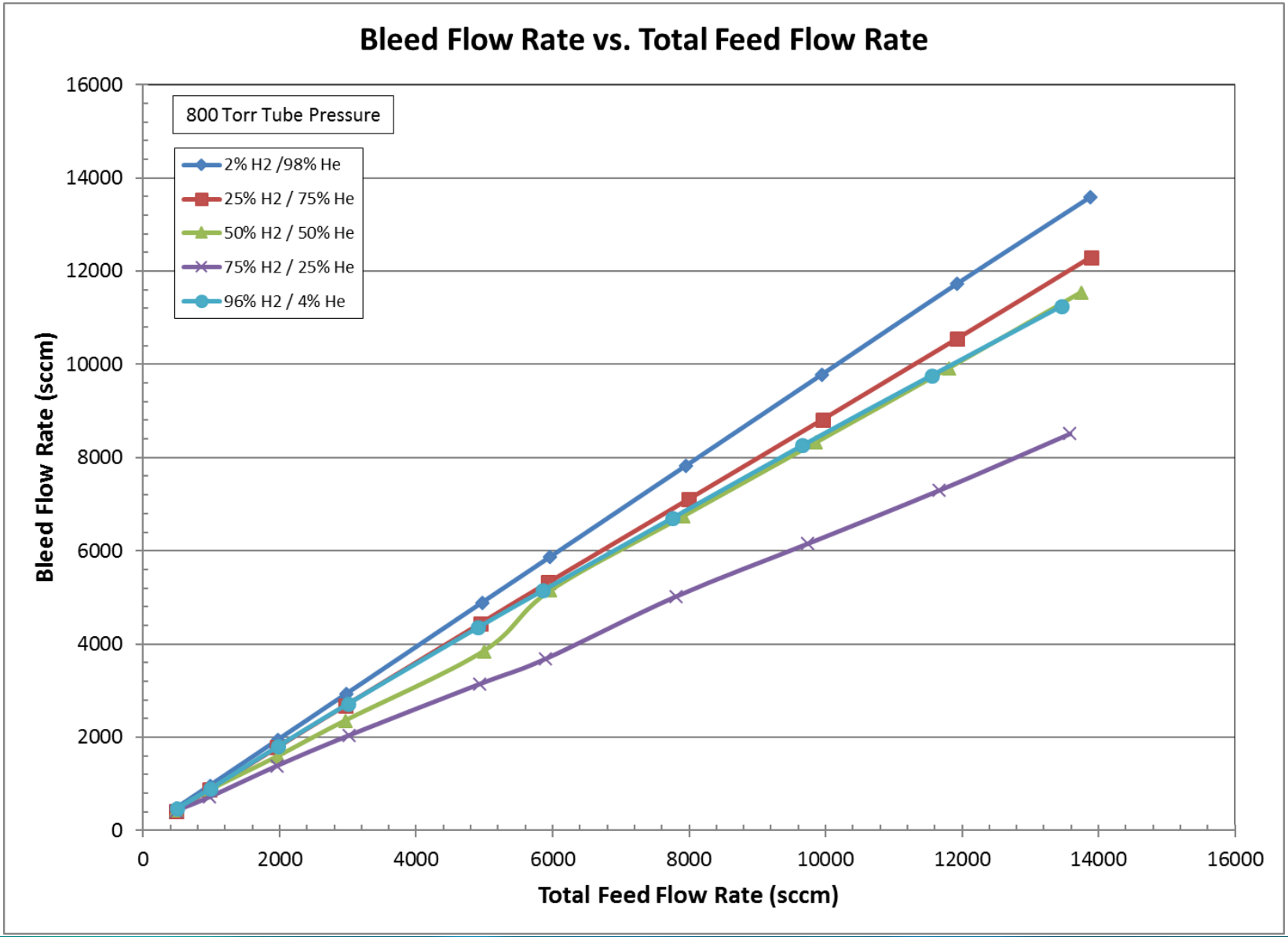
Bleed Flow Rate vs. Total Feed Flow Rate for H_2 in N_2



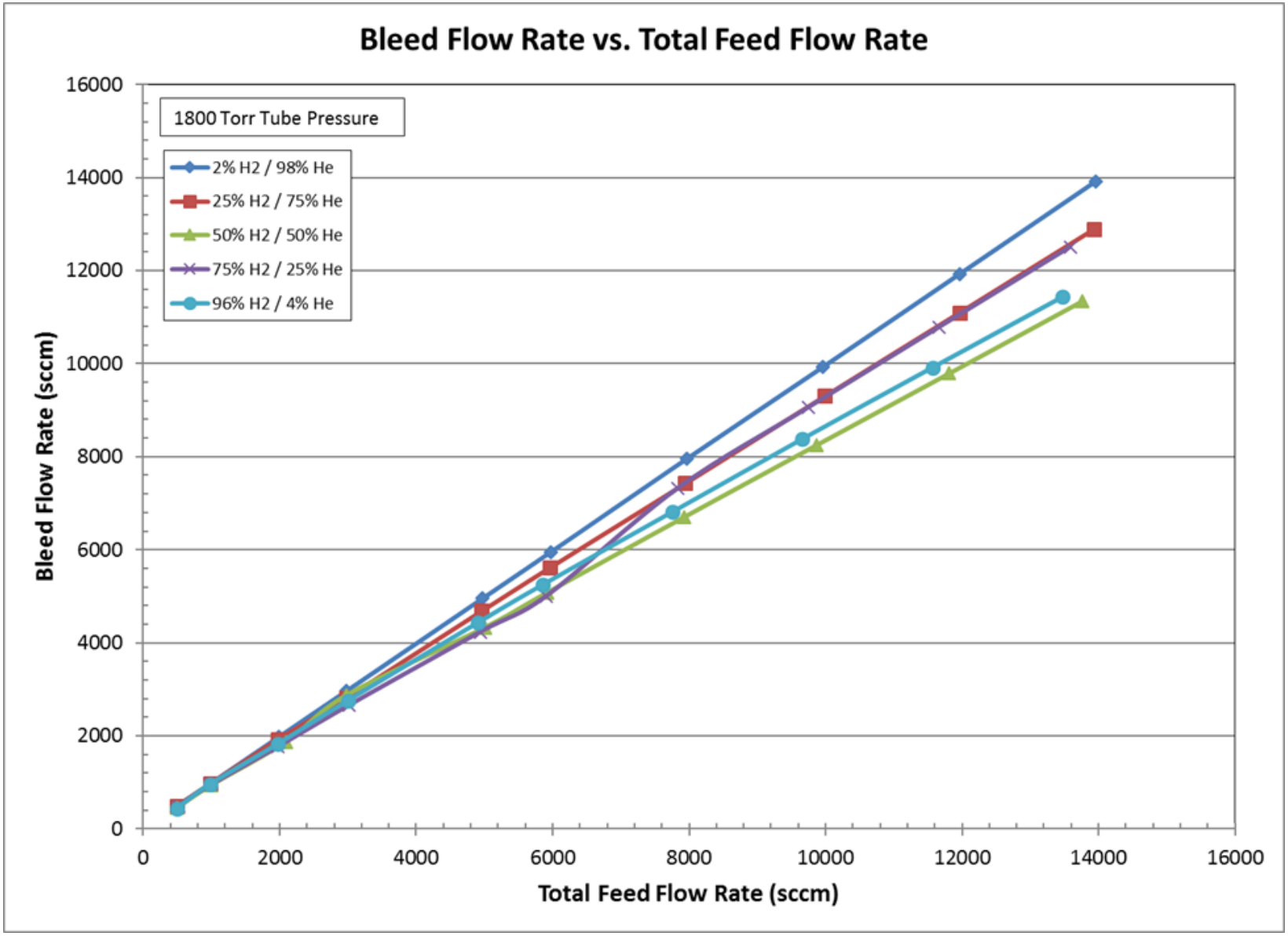
Bleed Flow Rate vs. Total Feed Flow Rate for H_2 in N_2



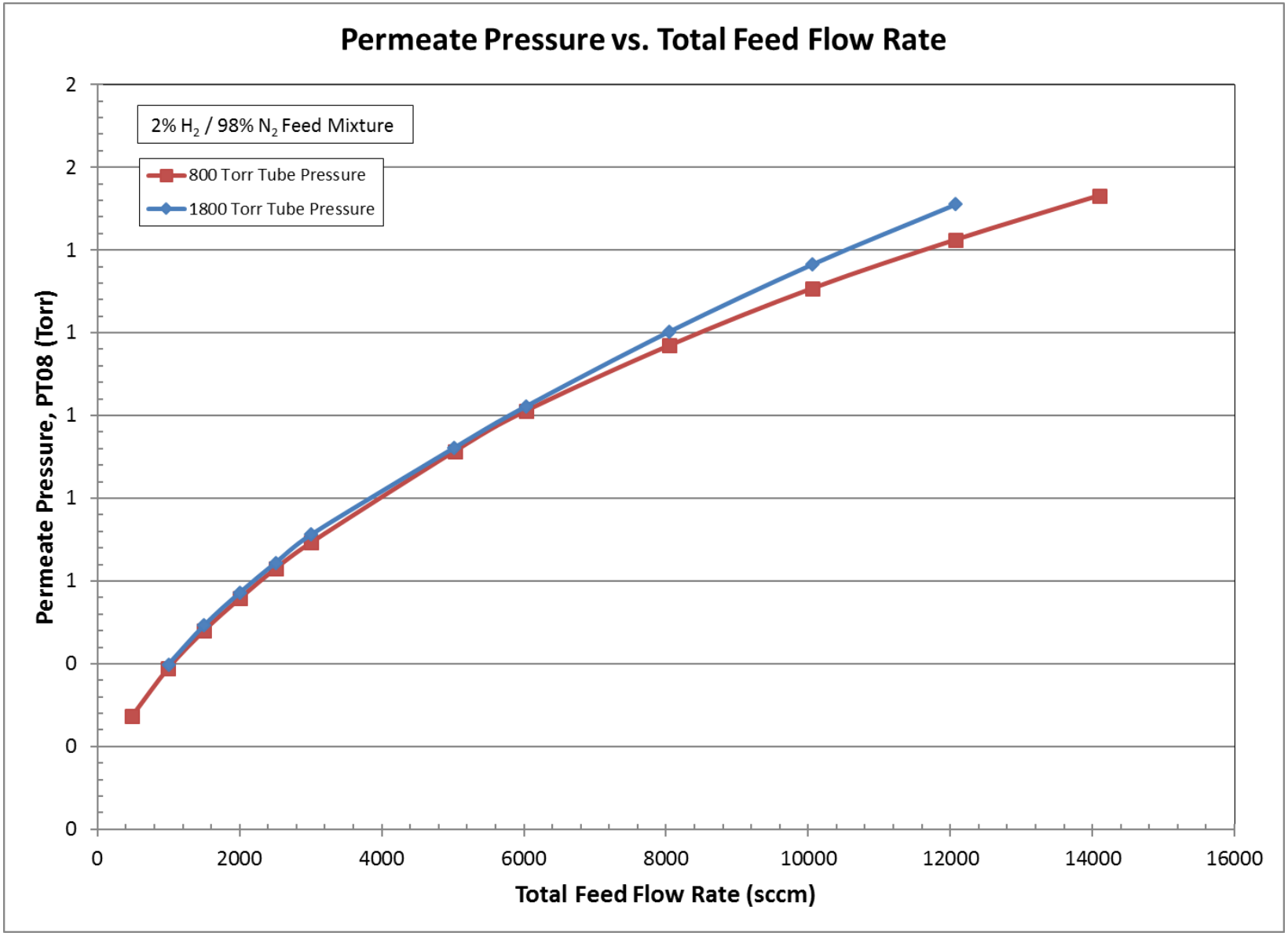
Bleed Flow Rate vs. Total Feed Flow Rate for H_2 in He



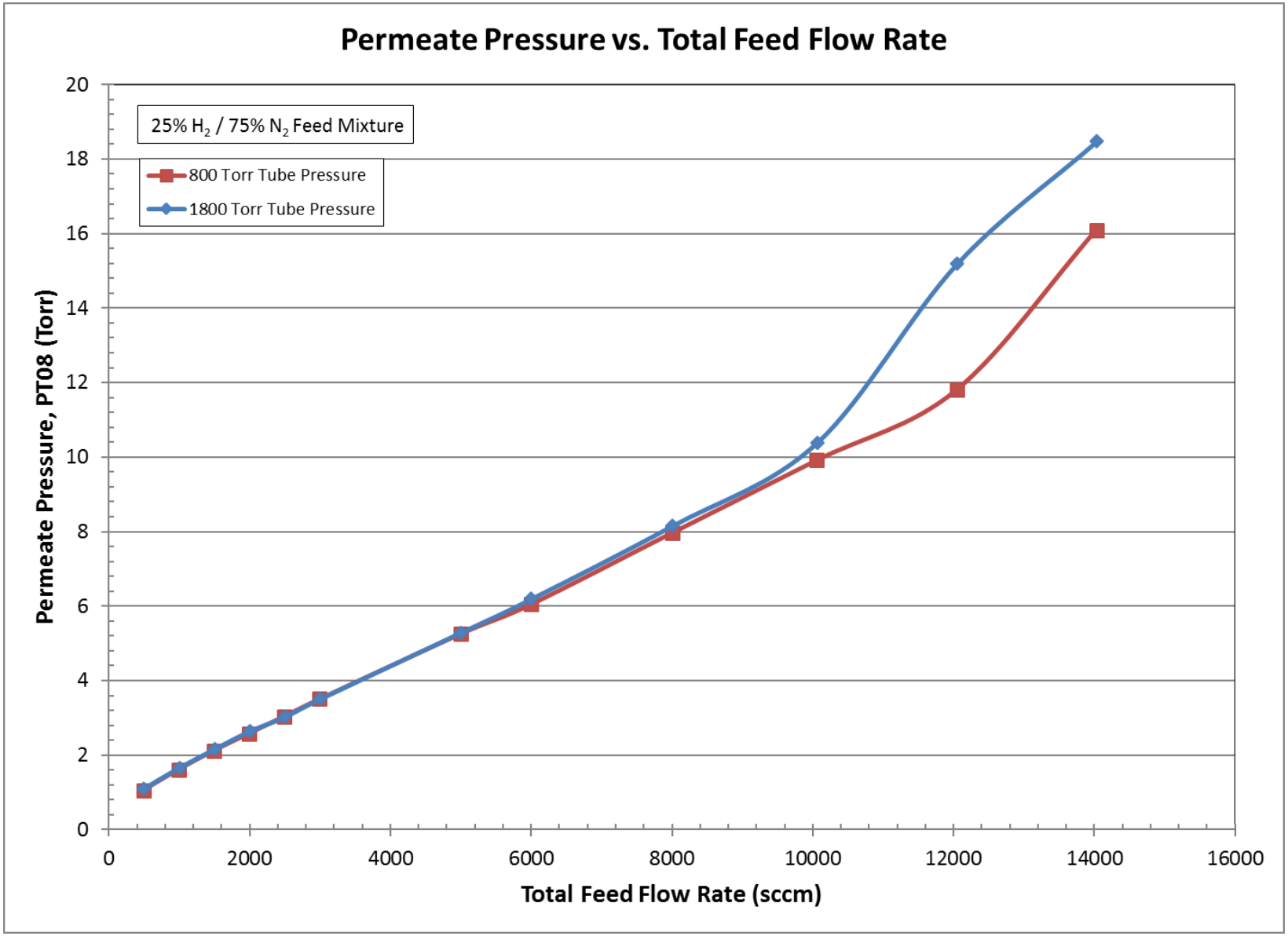
Bleed Flow Rate vs. Total Feed Flow Rate for H_2 in He



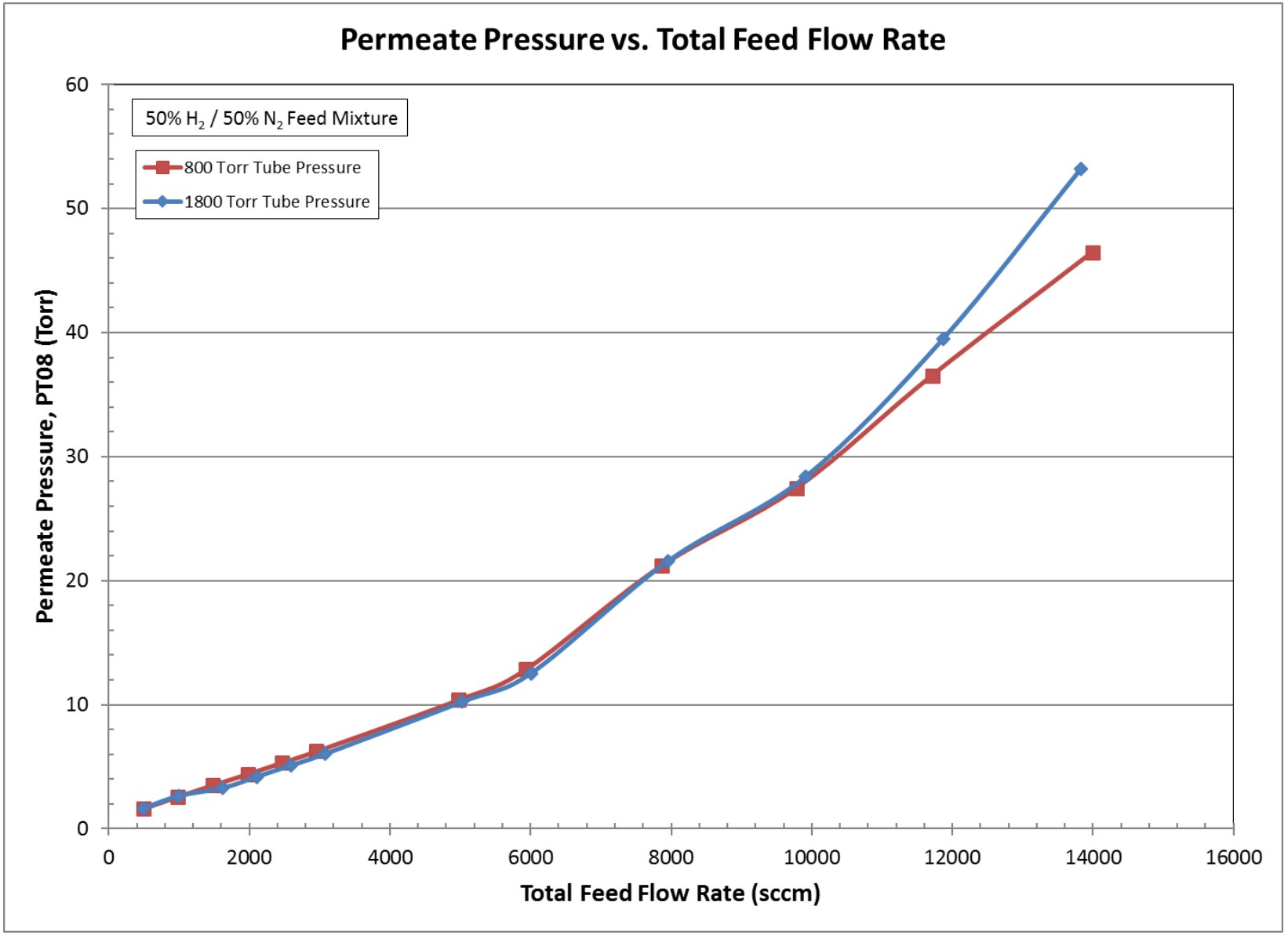
Permeate Pressure vs. Total Feed Flow Rate for 2% H_2 in N_2



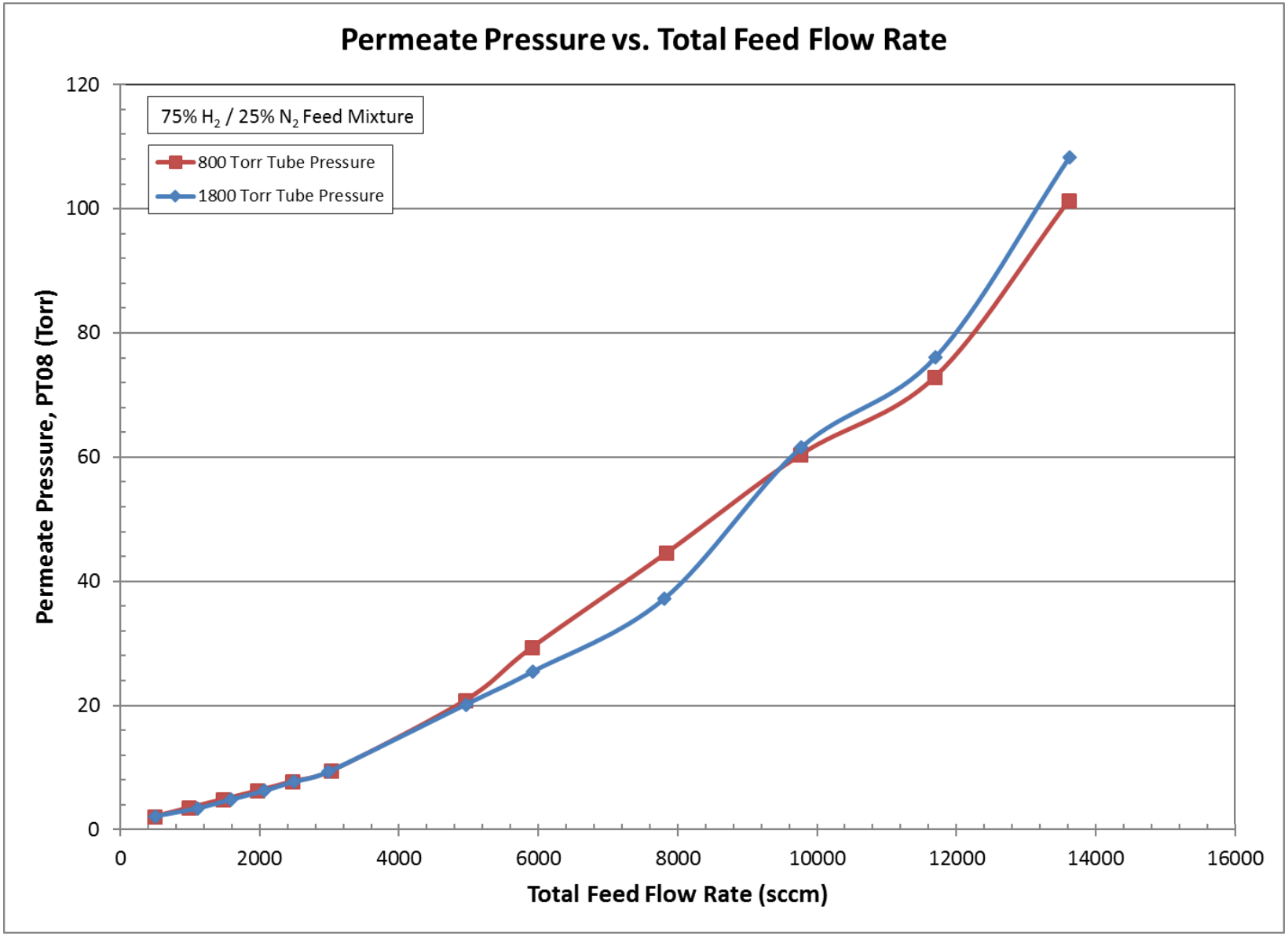
Permeate Pressure vs. Total Feed Flow Rate for 25% H_2 in N_2



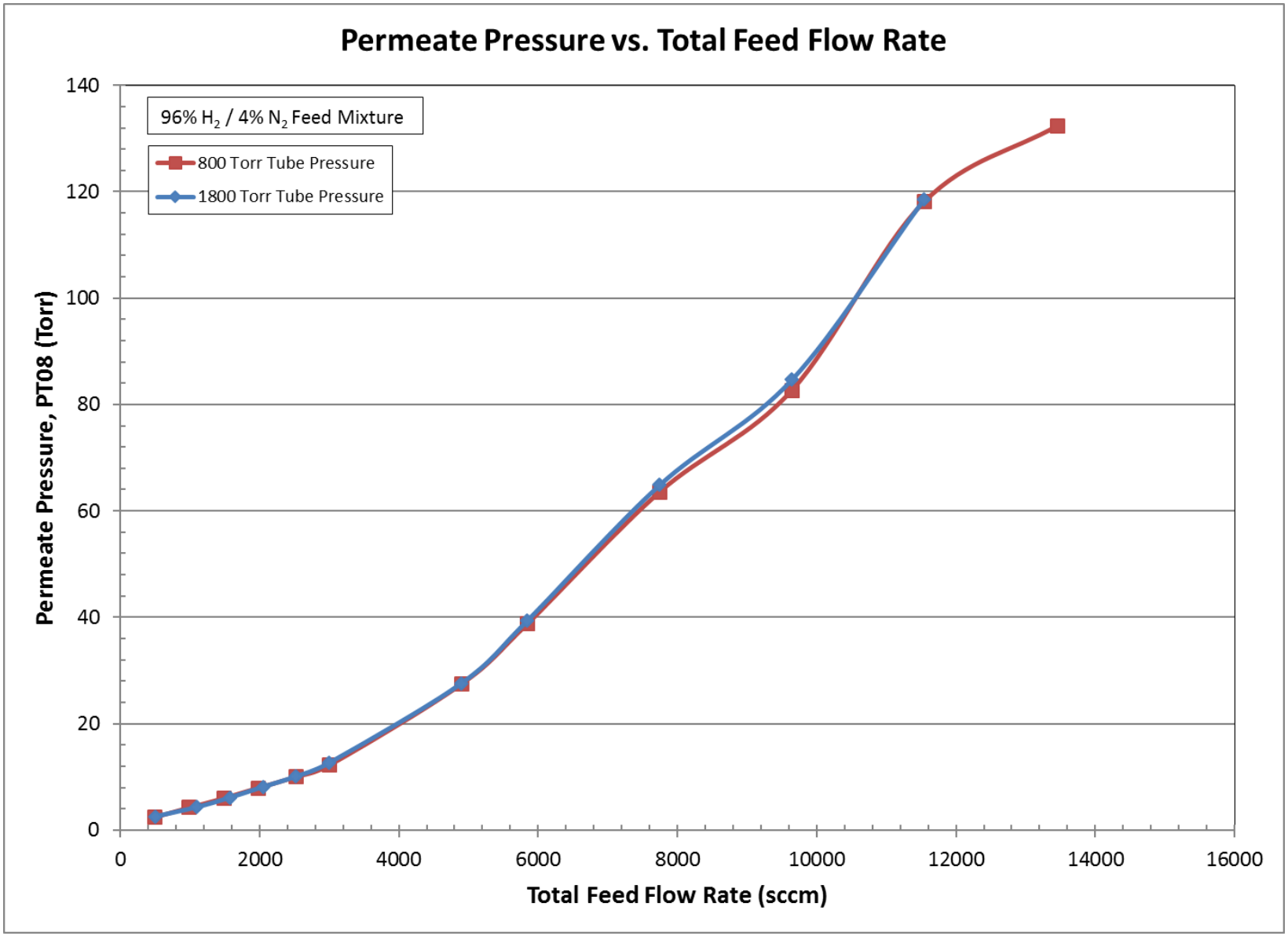
Permeate Pressure vs. Total Feed Flow Rate for 50% H_2 in N_2



Permeate Pressure vs. Total Feed Flow Rate for 75% H_2 in N_2



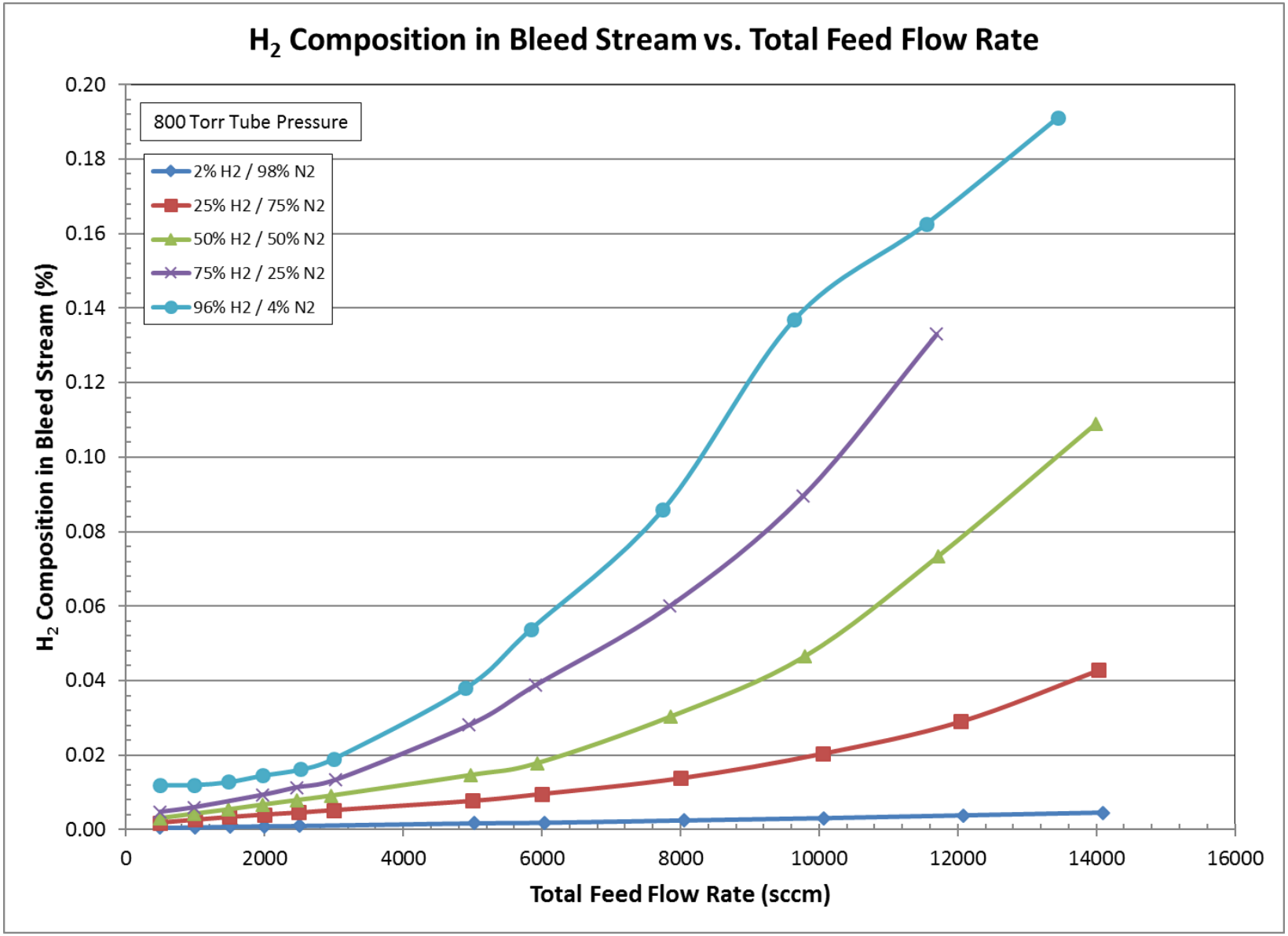
Permeate Pressure vs. Total Feed Flow Rate for 96% H_2 in N_2



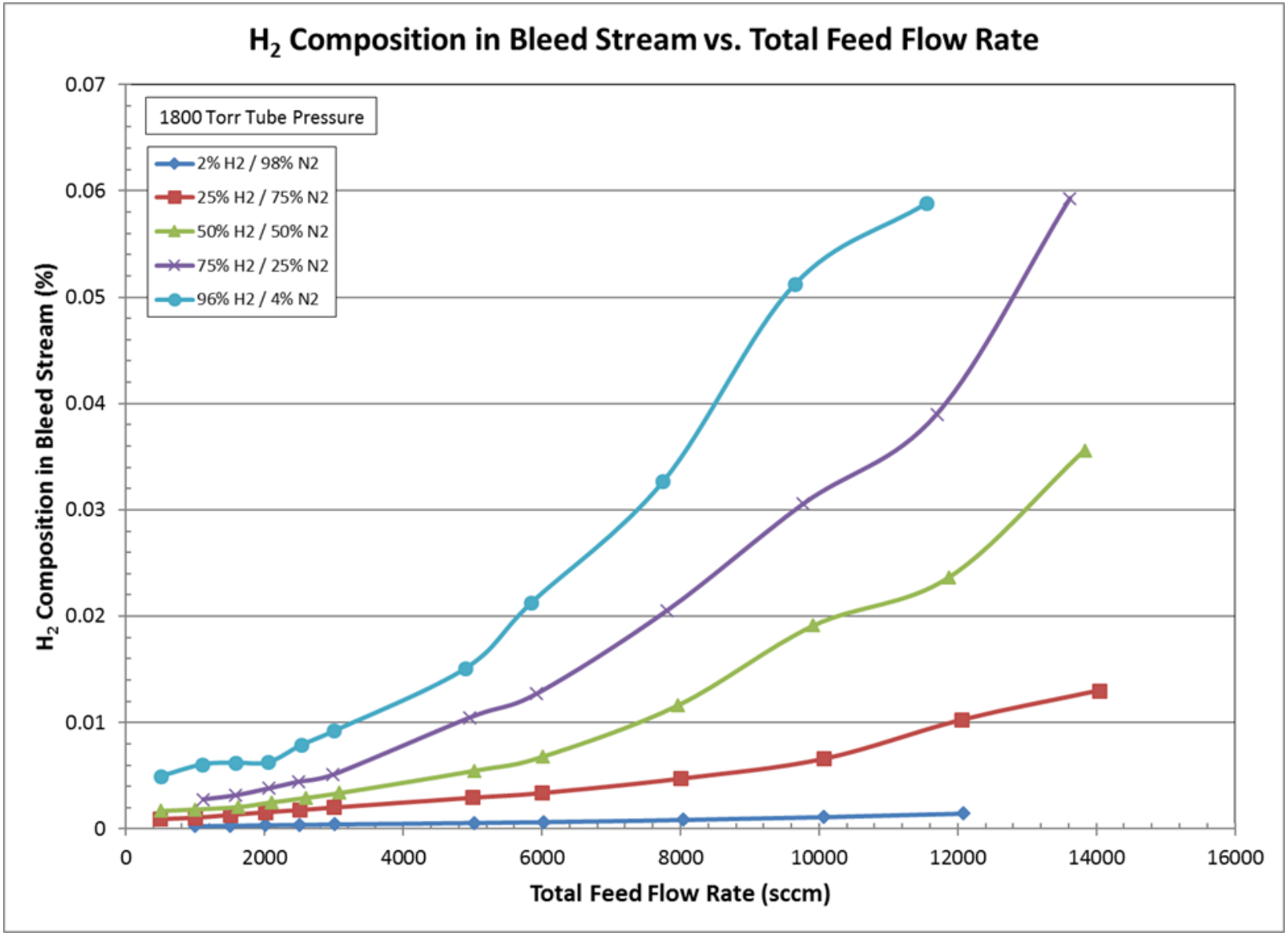
General Conclusions regarding Permeate Pressure

- **Permeate pressure increases as the amount of H_2 in the feed increases and as the flow rate increases**
  - **This is a function of the more H_2 available in the Pd-Ag tubes, the more H_2 that will permeate through the membrane**
- **In addition the pumping speed of the Normetex may not be adequate to effectively remove all of the hydrogen that is permeating through the membrane**
  - **At an inlet pressure of 6 torr, the Normetex pumping speed is ~1300 sccm and at an inlet pressure of 20 torr, the pumping speed is only 2200 sccm.**
- **Due to the fact that H_2 flow rate (and permeate rate) exceeds the pumping rate for most of the tests, the pumping capacity may impede additional H_2 permeation through the membrane**
- **Permeate flow rate measured by flow meters**
  - **The permeate flow rate and the feed flow rate can be used to calculate the bleed flow rate**

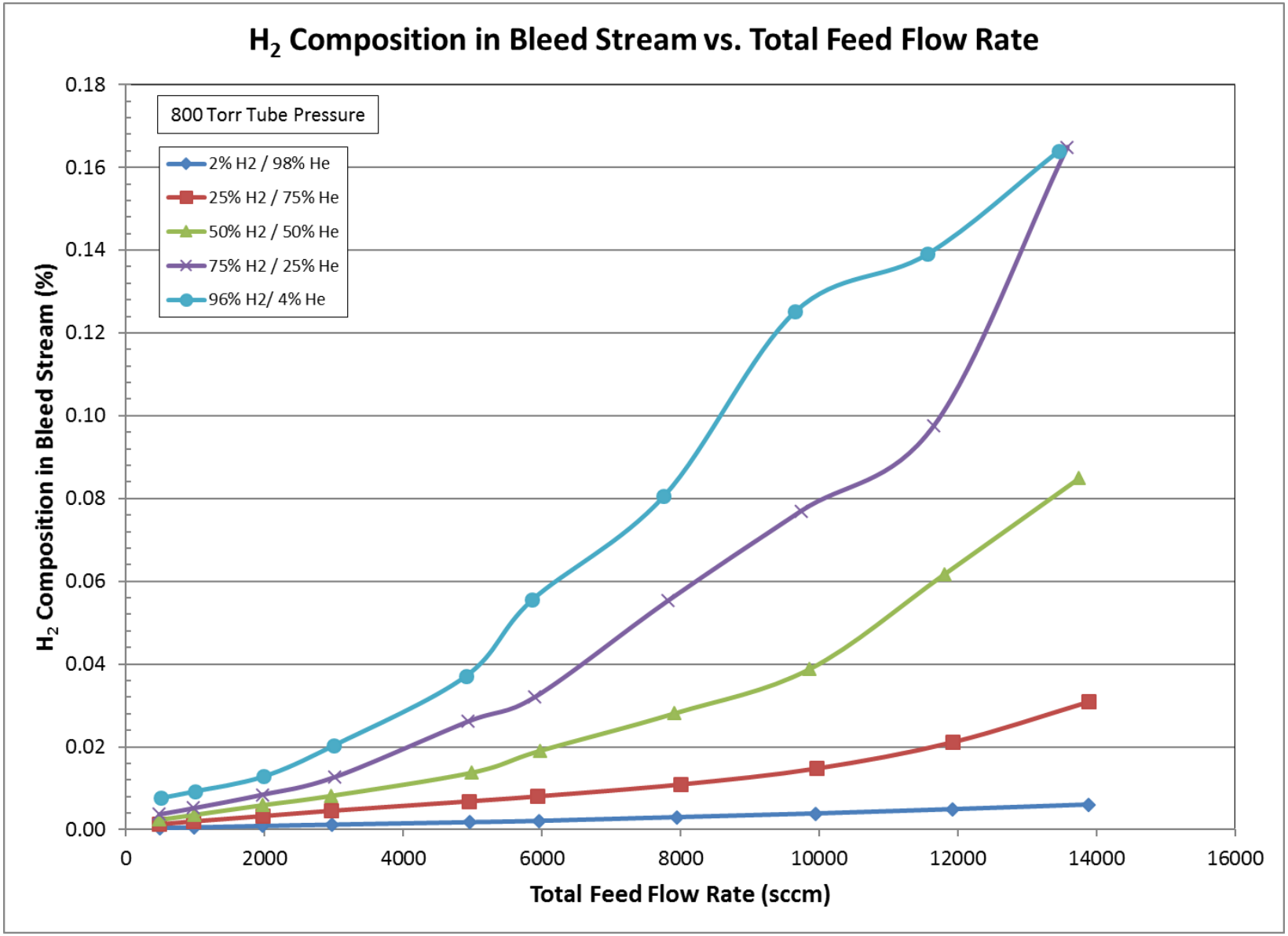
Amount of H_2 in the Bleed for H_2 in N_2



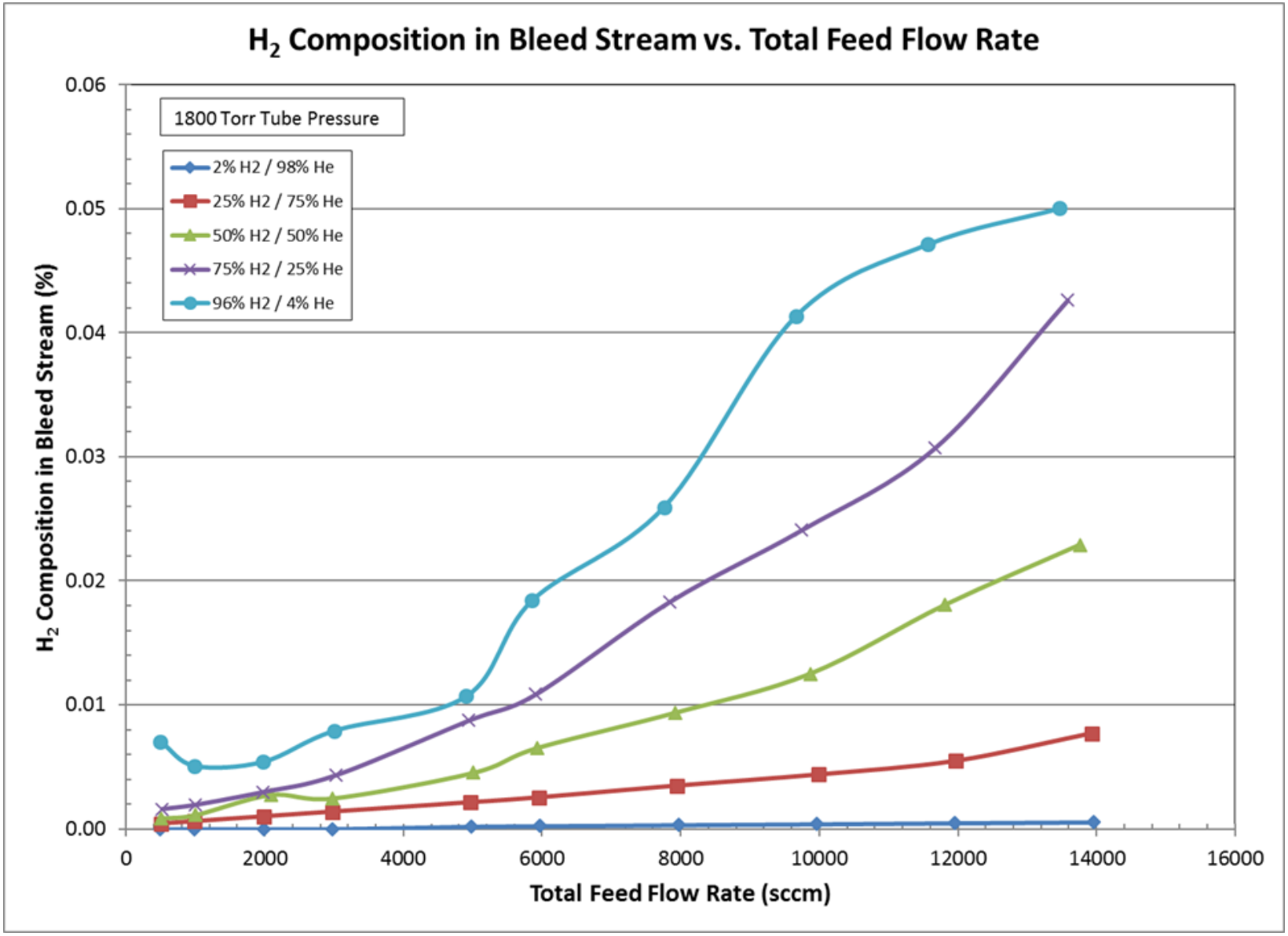
Amount of H_2 in the Bleed for H_2 in N_2



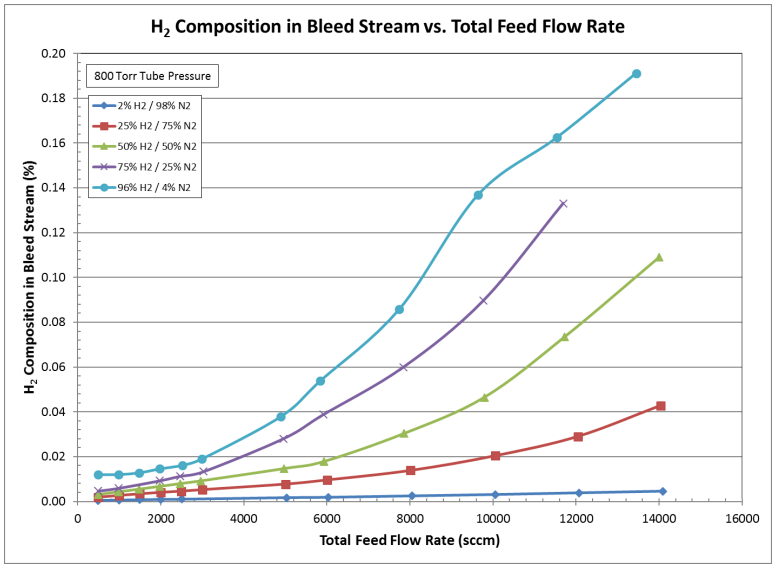
Amount of H_2 in the Bleed for H_2 in He

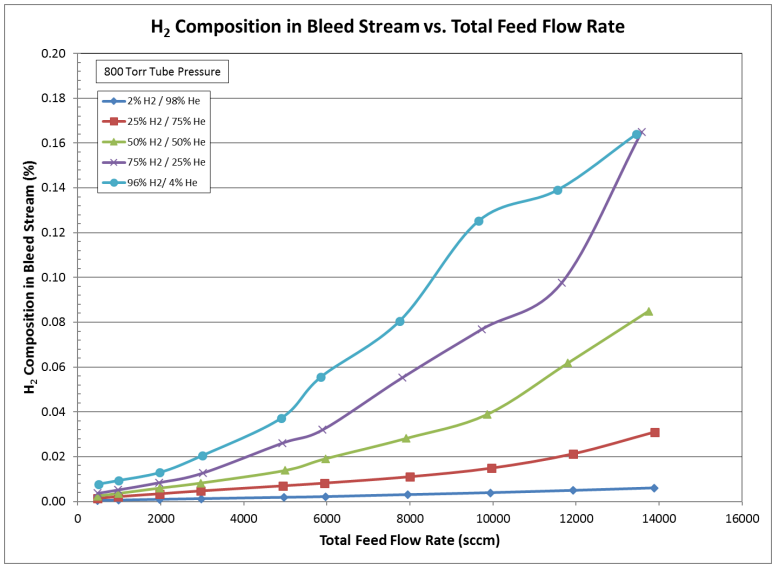


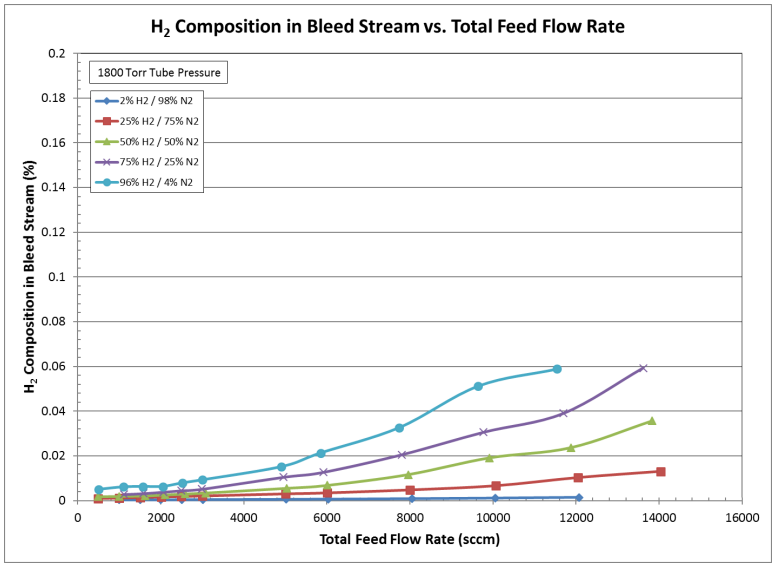
Amount of H_2 in the Bleed for H_2 in He

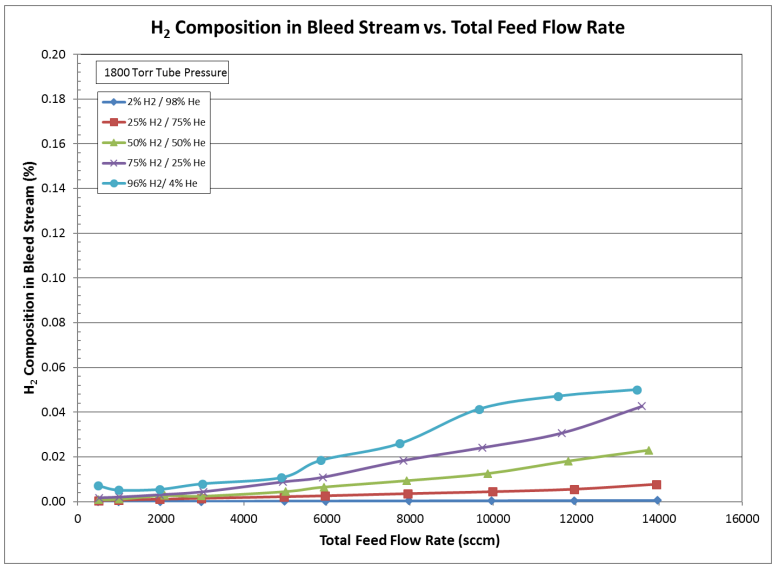


Summary of Amount of H_2 in the Bleed for H_2









Summary and Conclusions

- Characterization tests on a new Pd-Ag diffuser design have been completed to gain an understanding into how the permeation of H_2 is affected by varying experimental test conditions

- Pd-Ag diffuser was characterized at various flow rates, gas compositions, and internal tube pressures
  - Gas compositions ranging from 2% H_2 – 98% H_2 with the balance of N_2 or He
  - Tube Pressures at 800 and 1800 torr
  - Flow rates from 500 – 14,000 sccm

- At low H_2 concentrations the Pd-Ag removes nearly all of the hydrogen for all flow rates tested
  - For a 2% H_2 gas mixture, the amount of H_2 in the bleed is less than 0.01% for the flow rates tested

- As the amount of H_2 increases in the feed, the amount of H_2 in the bleed increases

- In general the amount of H_2 in the bleed stream is 3-4 times less when the internal tube pressure is 1800 torr compared to 800 torr

Recommendations and Future Work

- One option to increase the hydrogen removal efficiency from the feed stream would be to increase the pumping capacity of the permeate stream.
  - By increasing the pressure differential across the membrane more H_2 will be able to permeate through the Pd-Ag membrane
  - Additional testing is planned with alternate pumps on the permeate side of the Pd-Ag diffuser

- **Testing has been completed to determine the hydrogen removal efficiency of several Pd-Ag diffusers installed in series**

Acknowledgements

- **The authors acknowledge the contributions of**
  - *Anita Poore*
  - *Benton Randall*
  - *Henry Sessions*
  - *Kipplin Neikirk*

- **The authors gratefully acknowledge the financial support of the Savannah River Tritium Facilities**


Thank you for your attention!!!
Questions?