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Evaluation of High Vacuum Pump for Tritium Service

Lucas M. Angelette

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EXECUTIVE SUMMARY

In tritium process systems, vacuum pumps are typically used to evacuate volumes and piping, as well as transfer gas to other parts of the process. This was done using the combination of an all-metal scroll pump with a metal bellows backing pump. The all-metal scroll pump, manufactured by Normetex, has been unavailable since 2012, and efforts continue to find a suitable replacement. The main obstacle is finding a pump that has no oils or polymer components, which degrade when exposed to tritium and introduce corrosive and/or hazardous impurities into the process.

Since turbomolecular pumps are used in tritium processing, it is thought that pumps similar to the turbomolecular pumps would be of interest. A newer model turbomolecular pump with a variable rotation speed has been identified for use in tritium processing. The turbomolecular pump, backed by a Metal Bellows MB-601 pump with the pump heads in series, was characterized for static and gas flow conditions to determine the suction pressure at various discharge pressures or flow rates for various gases.

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LIST OF ABBREVIATIONS

Ar	Argon
BPR	Back Pressure Regulator
CV	Control Volume
D_2	Deuterium
FC	Flow Controller
GCF	Gas Correction Factor
H_2	Hydrogen
He	Helium
HF	Hydrogen Fluoride
IVG	Ion Vacuum Gauge
Kr	Krypton gas
L	Liter, volume
MDP	Molecular Drag Pump
Met-Bel	Metal Bellows
MFC	Mass Flow Controller
N_2	Nitrogen gas
NPT	National Pipe Thread
OTS	Off-The-Shelf
PT	Pressure Transducer
PTFE	Polytetrafluoroethylene, polymer
RD	Rupture Disk
rpm	Revolutions Per Minute
sccm	Standard cubic centimeters per minute, volumetric flow
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TC	Thermocouple
TCVG	Thermocouple Vacuum Gauge
TMP	Turbomolecular Pump

1.0 Introduction

The Normetex® Model 15 all-metal scroll pump, backed by a Senior Aerospace Metal Bellows pump, has been the standard pump used worldwide for tritium processing. However, in 2012, Normetex® halted production of the scroll pumps. Since then, researchers worldwide have been searching for a viable replacement for the Normetex® scroll pumps. There have been studies using a Molecular Drag Pump (MDP) backed by an off-the-shelf (OTS) scroll pump,[1] and an MDP backed by a Met-Bel pump.[2] The OTS scroll pump contains components that are not compatible for tritium service, and the MDP backed by a Met-Bel has a limited operating range due to the narrow overlap of the discharge pressure requirements of the MDP and the suction capabilities of the Met-Bel. Furthermore, the manufacturer of the MDP has decided to discontinue the control unit for the MDP. A manufacturer suggested an alternative model with an adjustable rotational speed, which could fill the role of an MDP or turbomolecular pump (TMP).

1.1 Identification of the Primary Pump

The Normetex® pump that has been in use worldwide has a set of highly desired characteristics for tritium processing: no oil lubricants, no polymer wetted materials, and vacuum levels as low as 0.001 torr at the inlet. A possible alternative is the use of mercury pumps, which are capable of the same pressures and characteristics. However, due to the health hazards associated with mercury, and that mercury vapors will contaminate the system if not properly trapped, mercury pumps are not being considered.

Mechanical pumps appear to be the best replacement option, but the currently available pumps have either greased bearings or polymer seals. OTS scroll pumps have PTFE tip seals, in which HF is present as an off-gas when exposed to tritium.[3] Met-Bel pumps have an all-metal wetted component design similar to the Normetex® , but the Met-Bel pumps can only achieve vacuum levels around 30 to 50 torr at the inlet (with discharge to 1 atm), which is orders of magnitude higher than the Normetex® pumps.

Another pump type that is currently used in tritium service are turbomolecular pumps. They are capable of very high vacuum levels on the inlet but are typically limited to approximately 3 torr maximum discharge. The TMPs also operate at rotational speeds of 90,000 revolutions per minute (rpm), while TPMs with a higher discharge capability like the MDPs operate at lower rotational speeds (27,000 rpm for the MDP). With the discontinuation of the independent controller for the Pfeiffer Vacuum MDP 5011, a recommended alternative was the Pfeiffer Vacuum HiPace 80 turbomolecular pump. The HiPace 80 has a variable speed control, ranging from 45,000 to 90,000 rpm. These are still higher than the MDP operates at, but it is more flexible than the fixed 90,000 rpm. It was of interest to determine if the HiPace 80 would have similar pumping characteristics as that of the MDP 5011.

1.2 Pump Test Scope

The scope of the pump testing includes baseline pump curves with the HiPace 80 backed by a Met-Bel for various gases. The HiPace 80/Met-Bel combination is being tested to determine if it is a comparable replacement to the Normetex/Met-Bel combination for certain applications with the understanding the HiPace 80 has greased bearings. The pump curves of interest are pressure comparisons of the suction and discharge of the HiPace 80 under static and gas flow conditions. The gases of interest include nitrogen (N_2), argon (Ar), krypton (Kr), helium (He), hydrogen (H_2), and deuterium (D_2).

2.0 Experimental Procedure

2.1 Experimental Approach

The system fabricated to test the HiPace 80/Met-Bel combination was constructed to have the capabilities for conducting both the static and gas flow tests. A schematic of the system is shown in Figure 2-1. The HiPace 80 (TMP01 in Figure 2-1) was manufactured by Pfeiffer Vacuum Products. The Met-Bel is a MB-601 manufactured by Senior Aerospace Metal Bellows. The system was built primarily of VCR fittings and welded tubing. The thermocouples (TCs) were held in place with Swagelok compression fittings, and the rupture disks (RD) and MB-601 were NPT/Swagelok unions. Four MKS Baratron Model 690 (10, 100, 1,000, and 10k torr) and one MKS Baratron Model 390 (10k torr) pressure transducers (PTs) were used to monitor the pressures of the system. Four MKS GE50A Mass Flow Controllers (MFCs) were used to control the gas flow. The ranges for the MFCs were, in sccm: 5, 50, and 500, all with H_2 as the reference gas. As a note, the gas correction factor (GCF) of hydrogen, relative to N_2, is 1.01. Control volume CV01, 0.3 L, was used to incrementally dose the system under static conditions, and a second control volume CV02, 1 L, was used to dampen the pressure oscillations caused by the MB-601 as well as create a buffer against over-pressurization of the discharge section. TCs were located along the flow path of each pressure transducer for temperature-related pressure corrections. A cold cathode ion vacuum gauge (IG01) monitored the HiPace 80 inlet to measure the high vacuum levels of the HiPace 80. A thermocouple vacuum gauge (TCVG01) was placed on the HiPace 80 outlet to monitor the vacuum levels during system evacuation and low-pressure static dosing.

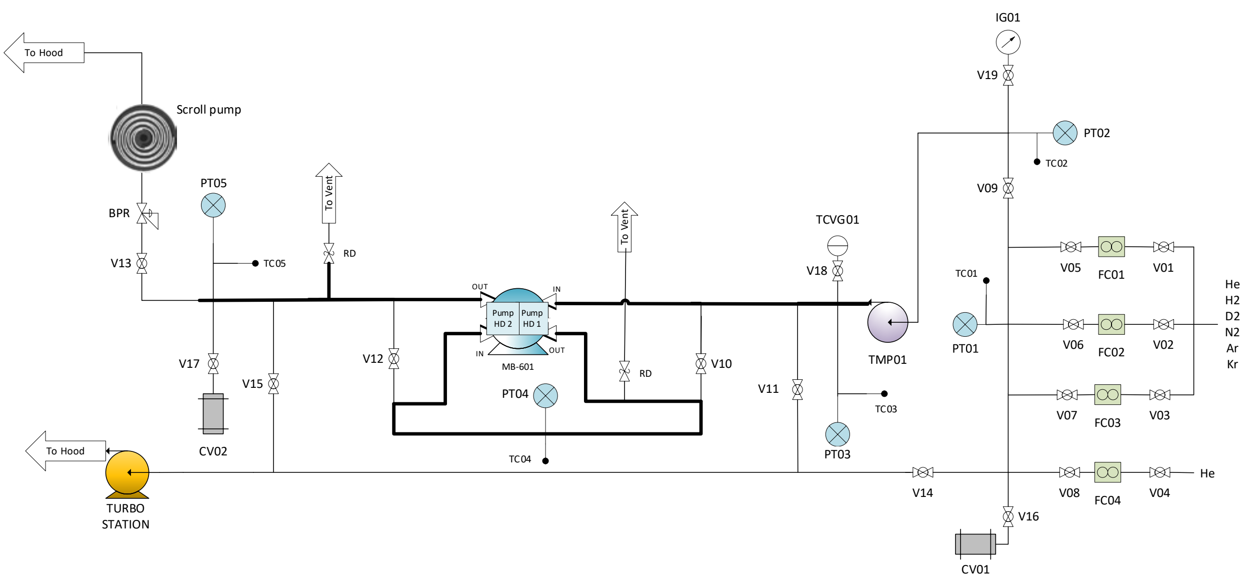

Figure 2-1: Schematic of the pump test system

The PTs, TCs, and MFCs were connected to a LabVIEW Data Acquisition System for data collection, along with supplying the mass flow controller setpoints. The HiPace 80 was tested at three rotational speed setpoints (45,000 rpm, 67,500 rpm, and 90,000 rpm) and three MB-601 discharge pressures (750 torr, 300 Torr, and 150 Torr).

2.2 Static Testing

The static tests were conducted by first closing the system vent valve and evacuating the system using an Adixen Drytel 1025 pumping station. Next, the CV01 was dosed using either the 50 sccm or 500 sccm

MFCs, depending on the dose pressure, with set increments of the target gas and then opened to the HiPace 80 (TMP01). This was repeated until the HiPace 80 discharge pressure was greater than 75 torr, or the pump displayed an error indicating it could not maintain the rotational speed setpoint. The HiPace 80 has a variable gas mode so the pump can perform optimally for different gases. The gas mode was changed according to the manufacturer's directions: gas mode 0 for H_2, D_2, and He; gas mode 1 for N_2; and gas mode 2 for Ar and Kr.

2.3 Flow Testing

The flow tests were conducted by first closing the system vent valve and evacuating the system with using an Adixen Drytel 1025 pumping station. Tests were conducted at three MB-601 discharge pressures, consisting of 750, 300, and 150 torr. For the 750 torr tests, the system was pressurized to approximately 800 torr using the 5 sccm MFC before opening the system vent valve, where the MB-601 discharge was maintained at approximately 750 torr. For the 300 and 150 torr tests, an Edwards Vacuum nXDS-15i scroll pump supplied the sub-ambient vacuum and a Crane Co. BP-3 GO back pressure regulator (BPR) was used to control the system pressure. The 300 and 150 torr tests were pressurized to 350 and 200 torr, respectively, before the vent valve was opened. Flow was then stopped to record the zero-flow pressures. The MFCs were then set at increasing increments up to 400 sccm, with pressure measurements taken before increasing the flow rate. The HiPace 80 was operated in the same gas/gas mode combinations as the static tests.

3.0 Results and Discussion

3.1 Static Testing

Several tests were performed to measure the suction pressure and discharge pressure of the HiPace 80 backed by the MB-601. The flow path for the static testing is shown in Figure 3-1. These tests included incrementally dosing the HiPace 80 and the Met-Bel was discharging to a control volume while the vent valve was shut. This was done using N_2, Ar, Kr, He, H_2, and D_2 gases separately.

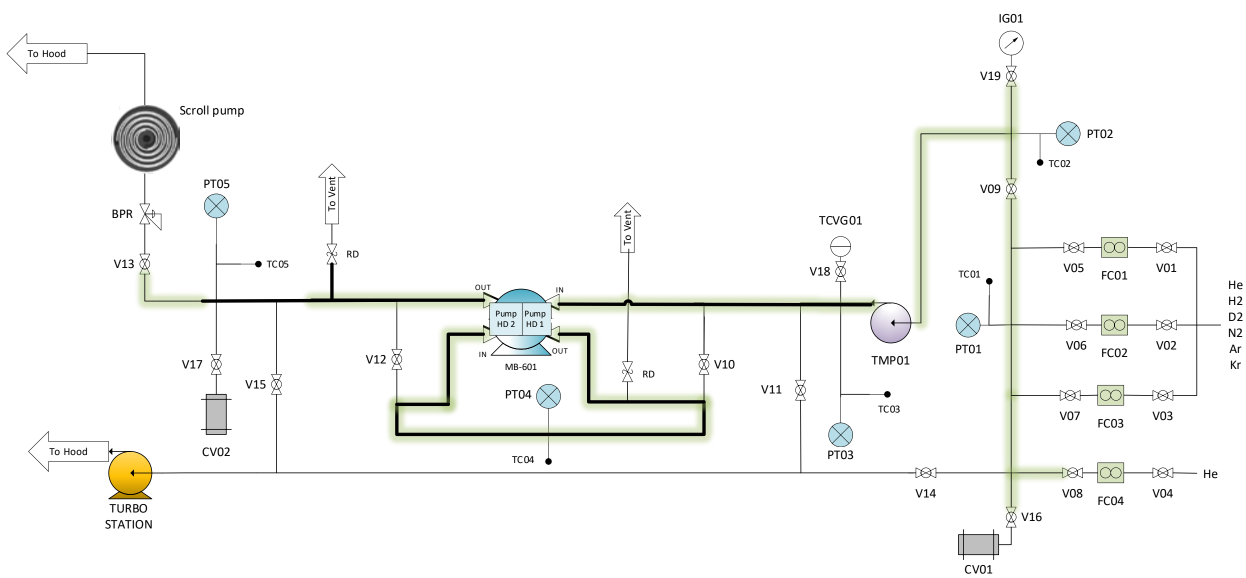

Figure 3-1: Flow path for static testing

The pressure comparison for the HiPace 80 are shown in Figure 3-2, Figure 3-3, and Figure 3-4 for rotational speeds of 45,000 rpm (50%), 67,500 rpm (75%), and 90,000 rpm (100%), respectively. It should be noted that a cold cathode ion vacuum gauge was used for suction pressures below 7.0E-03 torr, denoted as IVG in the figures. Above these values, the measurements were taken using a 100 torr Baratron (capacitance manometer).

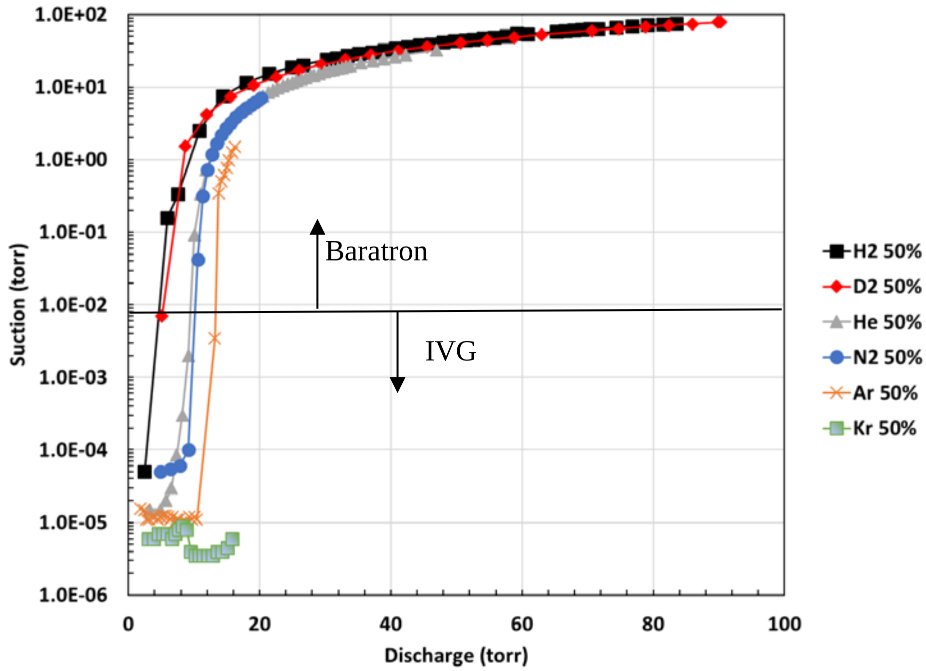

Figure 3-2: HiPace 80 static suction vs discharge pressures at 45,000 rpm

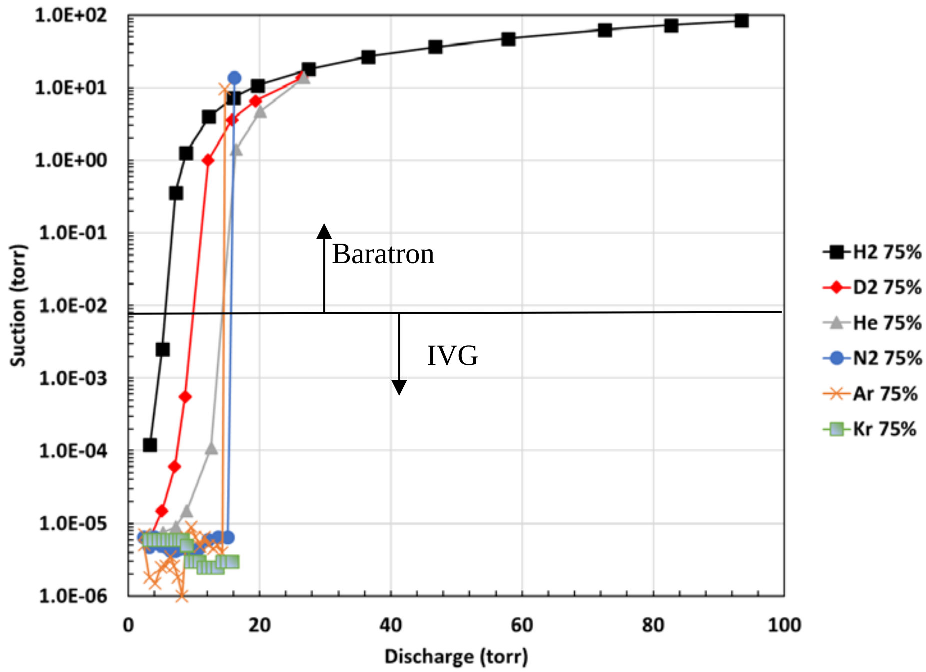

Figure 3-3: HiPace 80 static suction vs discharge pressures at 67,500 rpm

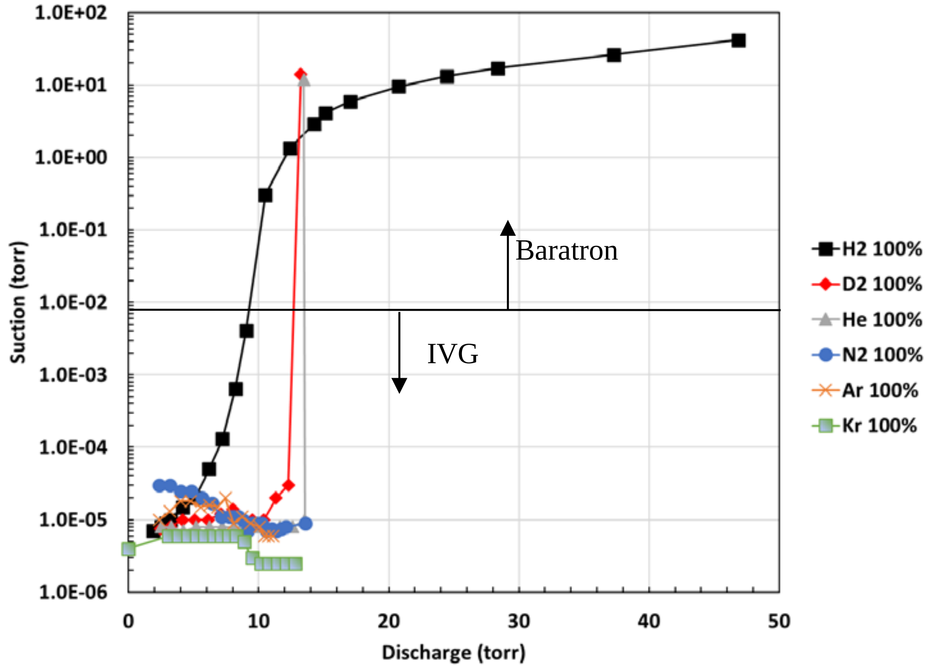

Figure 3-4: HiPace 80 static suction vs discharge pressures at 90,000 rpm

The HiPace 80 pressure comparison indicates that nitrogen, argon, and krypton are able to be discharged at higher pressures compared to H_2, D_2, and He while maintaining suction pressures below 0.01 torr. The TMP discharge pressures at which the HiPace 80 failed to maintain rotational speed is listed in Table 3-1. It should be noted that argon at 45,000 rpm and hydrogen at 45,000 and 67,500 rpm were not run until rotational speed failure. In the case of hydrogen, the rotational speed was able to be maintained beyond the range of the 100 torr Baratron measuring the TMP discharge pressure.

The discharge and suction comparisons raise an interesting trend in the pumping capability of the gases. Hydrogen and deuterium trend similar at 45,000 rpm, but as the pump rotational speed increases, deuterium begins to trend with helium. Nitrogen starts to trend with helium at 45,000 rpm, but at higher rotational speeds it begins to trend with argon. Krypton trends with argon, but the pump is able to maintain a suction pressure below 10^{-5} torr up to the point of rotational speed failure. The trend seems that gases with similar viscosities have similar pump curves at low TMP discharge pressures (<10 torr), but at higher TMP discharge pressures (>20 torr) or higher rotational speeds it shifts to trending with molar mass, shown in Table 3-2. The trend in viscosity can be observed at approximately 8 torr in Figure 3-2, and the trend with molar masses can be observed at approximately 26 torr in Figure 3-3. The different pumping behaviors of the gases at different rotational speeds and HiPace 80 discharge pressures needs to be accounted for in future process designs.

Table 3-1: Static HiPace 80 discharge pressures at failure to maintain rotational speed

Rotational speed (rpm)	Gas	TMP discharge at speed failure (torr)
45,000	H_2	No failure
	D_2	90.3
	He	46.9
	N_2	20.2
	Ar	---
	Kr	15.8
67,500	H_2	No failure
	D_2	26.4
	He	26.7
	N_2	16.1
	Ar	14.7
	Kr	15.8
90,000	H_2	46.9
	D_2	13.2
	He	13.5
	N_2	13.6
	Ar	11.1
	Kr	12.8

Table 3-2: Viscosity and molar mass of test gases

Gas	Viscosity[4] ($x10^7$ poise)	Molar Mass (g)
H_2	920	2.016
He	1950	4.003
D_2	1250	4.024
N_2	1760	28.014
Ar	2200	39.948
Kr	2483	83.798

3.2 Flow Testing

Several tests were performed to determine the operational suction and discharge pressures of the HiPace 80 backed by an MB-601 under gas flow conditions. The flow path of the flow tests is shown in Figure 3-5. These tests included testing the previous gases at increasing MFC set points, in sccm: 1, 2, 3, 4, 5, 10, 20, 30, 40, 50, 100, 200, 300, 400 with H_2 reference. As a note, the GCFs for the six gases are listed in Table

3-3. Flow tests were carried out at three different MB-601 discharge pressures: 150 torr, 300 torr, and 750 torr. To accomplish the flow tests, the system was initially evacuated using the Adixen Drytel 1025 pumping station. The system was then pressurized, with the pumps energized, to 800 torr before opening the vent valve. For the 300 and 150 torr tests, the system was pressurized to 350 and 200 torr, respectively, before the vent valve would be opened. The back pressure regulator was used to maintain the system pressure while the scroll pump sustained the required vacuum.

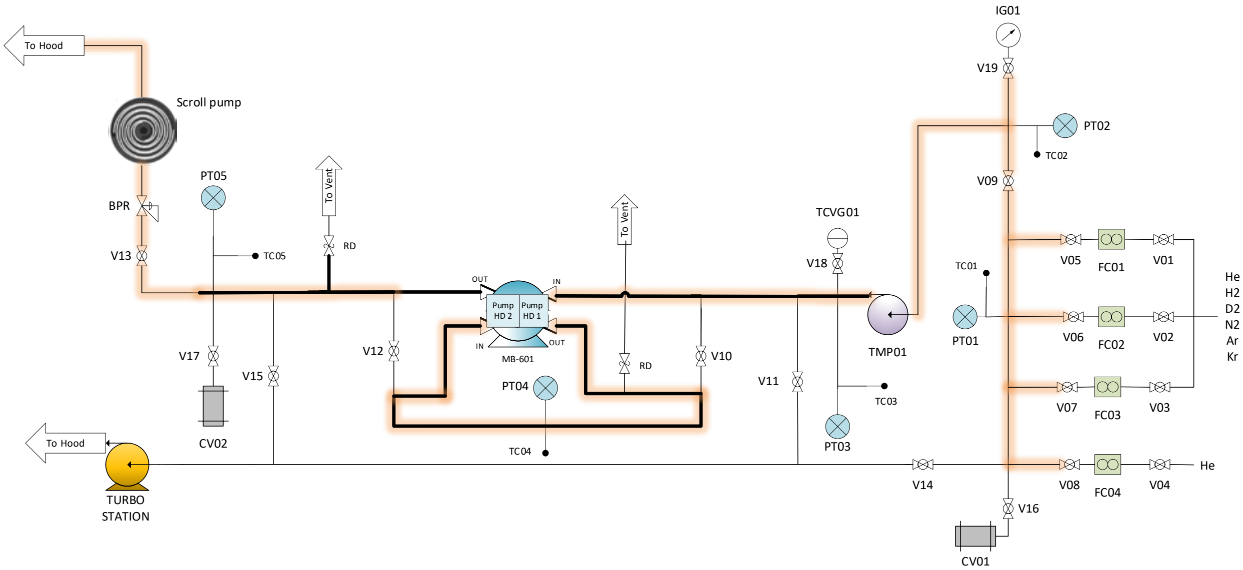

Figure 3-5: Flow path schematic for flow tests

Table 3-3: Gas correction factors

Gas	Gas Correction Factor
H_2	1.01
D_2	1.00
He	1.45
N_2	1.00
Ar	1.39
Kr	1.543

The suction and discharge pressure comparisons under gas flow are shown below for increasing MB-601 discharge pressures: 150 torr (Figure 3-6, Figure 3-7, and Figure 3-8), 300 torr (Figure 3-9 and Figure 3-10), and 750 torr (Figure 3-11). Suction pressure versus flow rate comparisons are also shown below for increasing MB-601 discharge pressures: 150 torr (Figure 3-12, Figure 3-13, and Figure 3-14), 300 torr (Figure 3-15 and Figure 3-16), and 750 torr (Figure 3-17).

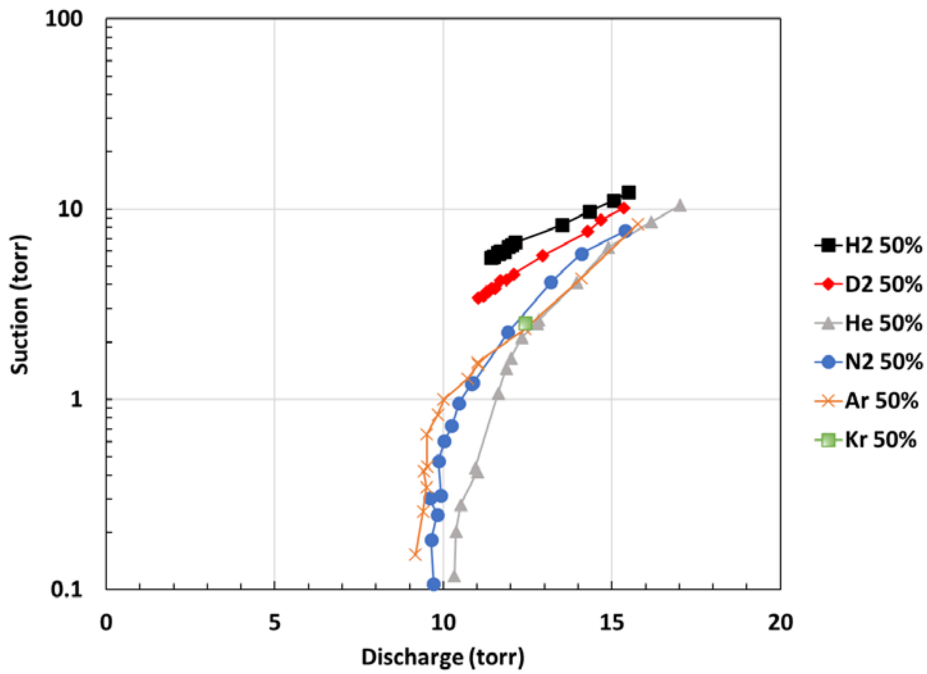

Figure 3-6:HiPace 80 (45,000 rpm) suction vs discharge pressures with gas flow, 150 torr MB-601 discharge

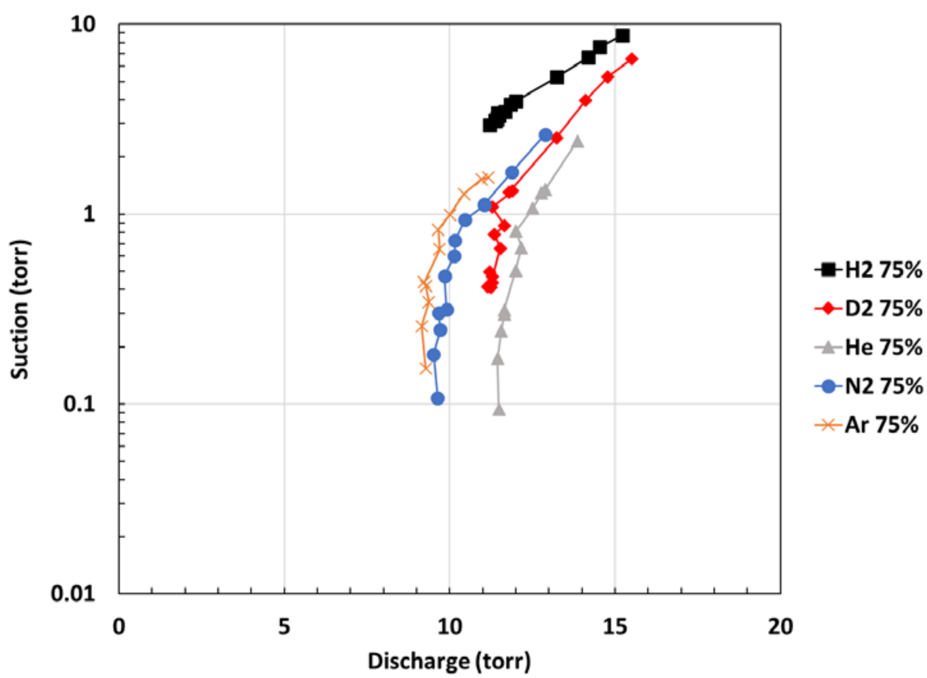

Figure 3-7:HiPace 80 (67,500 rpm) suction vs discharge pressures with gas flow, 150 torr MB-601 discharge

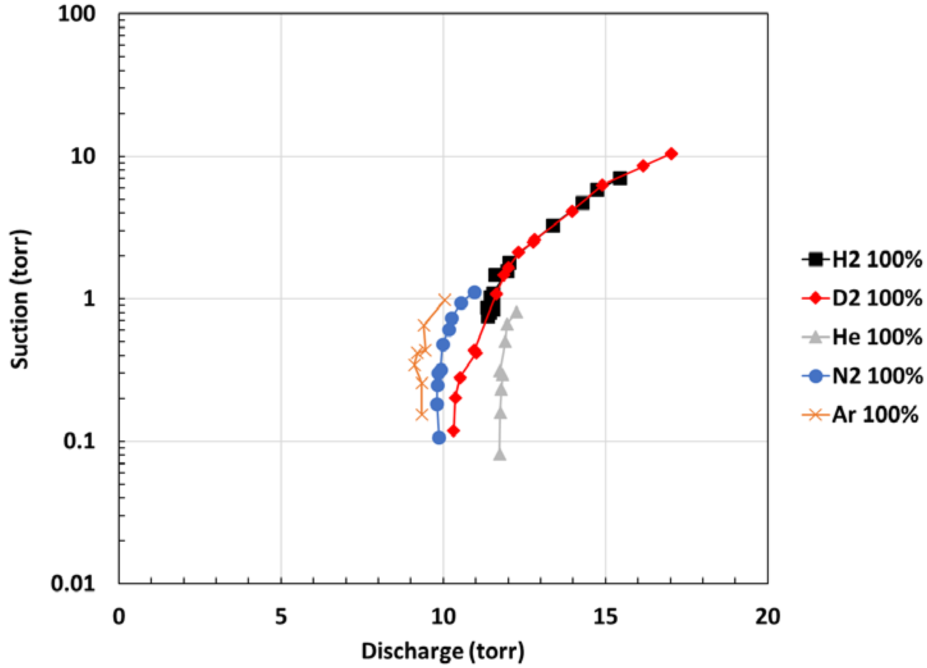

Figure 3-8:HiPace 80 (90,000 rpm) suction vs discharge pressures with gas flow, 150 torr MB-601 discharge

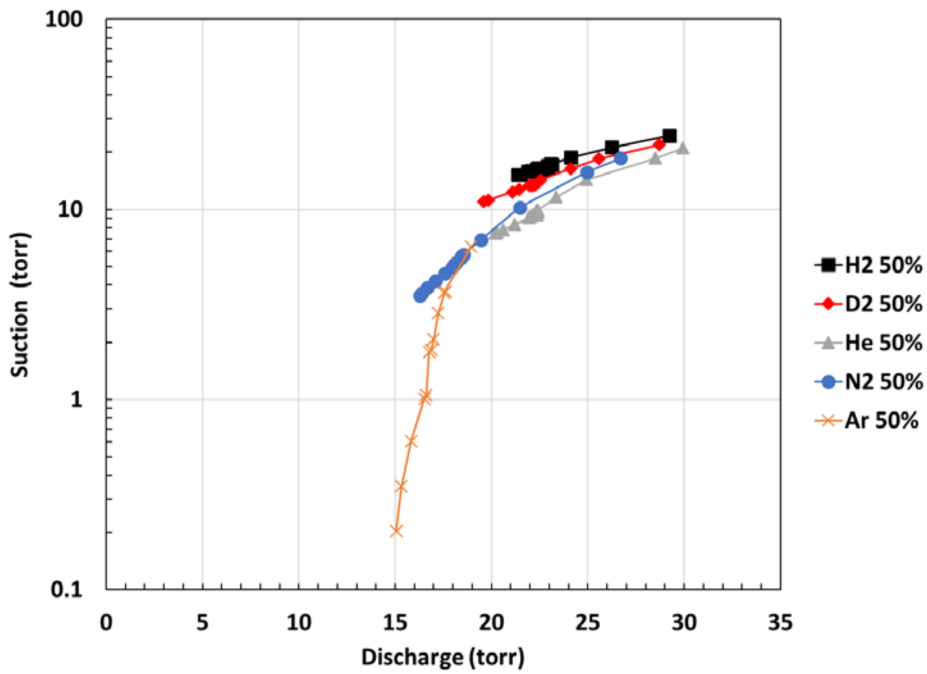

Figure 3-9: HiPace 80 (45,000 rpm) suction vs discharge pressures with gas flow, 300 torr MB-601 discharge

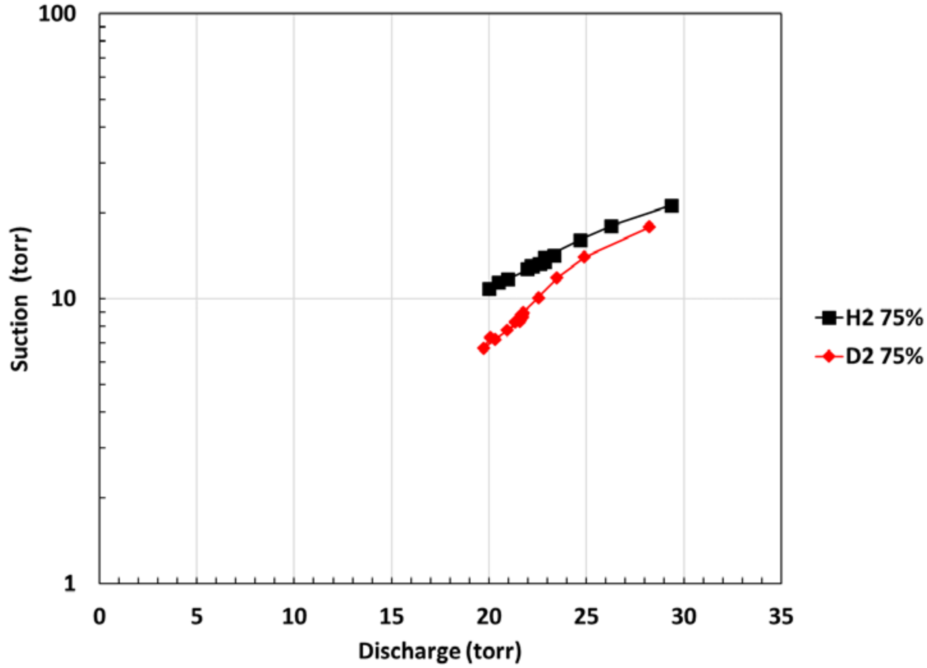

Figure 3-10: HiPace 80 (67,5000 rpm) suction vs discharge pressures with gas flow, 300 torr MB-601 discharge

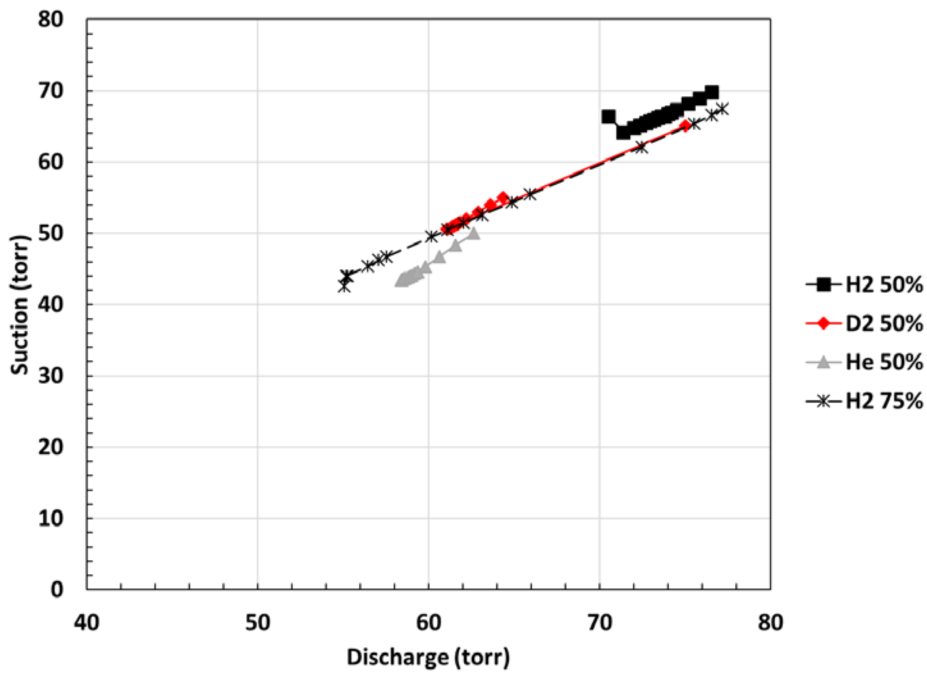

Figure 3-11: HiPace 80 suction vs discharge pressures with gas flow, 750 torr MB-601 discharge

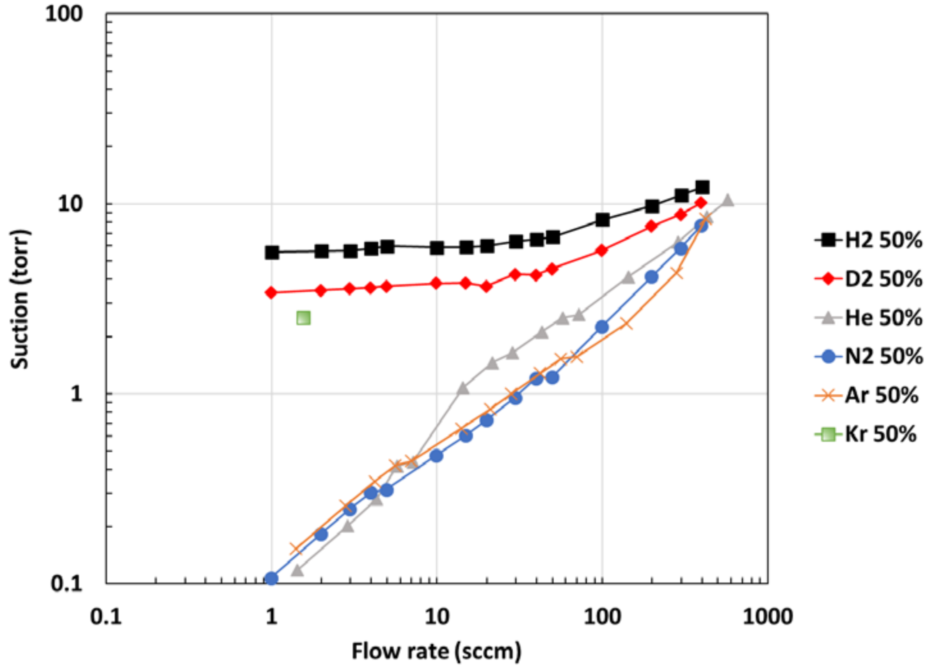

Figure 3-12: HiPace 80 (45,000 rpm) suction pressure vs flow rate, 150 Torr MB-601 discharge

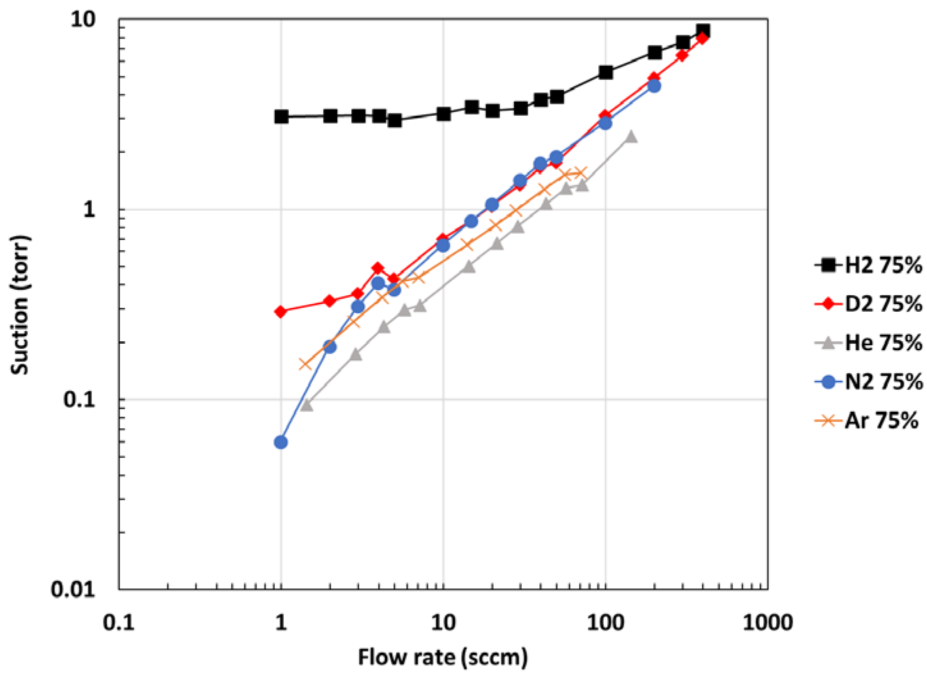

Figure 3-13: HiPace 80 (67,500 rpm) suction pressure vs flow rate, 150 Torr MB-601 discharge

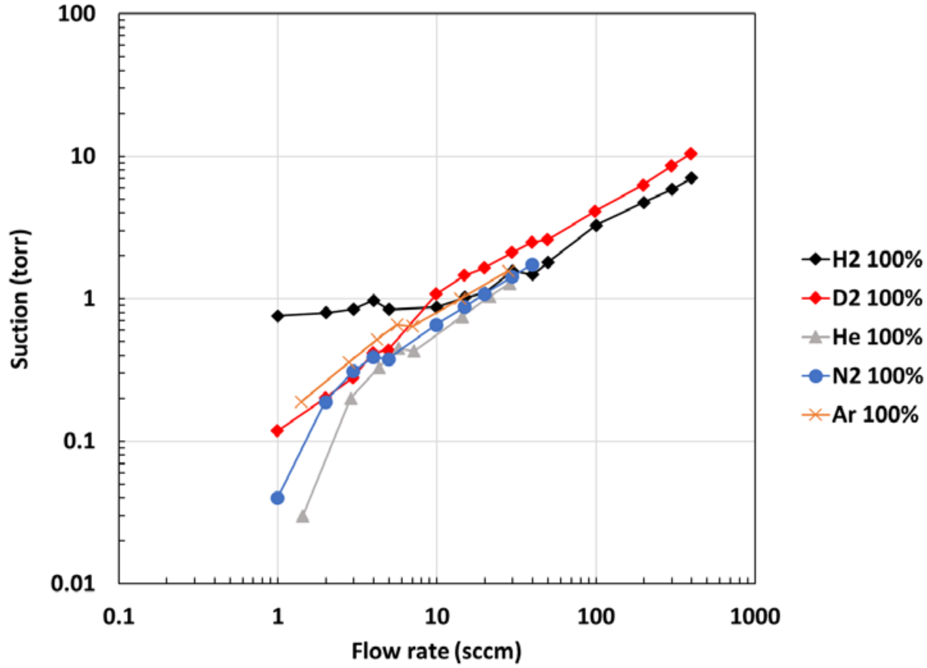

Figure 3-14: HiPace 80 (90,000 rpm) suction pressure vs flow rate, 150 Torr MB-601 discharge

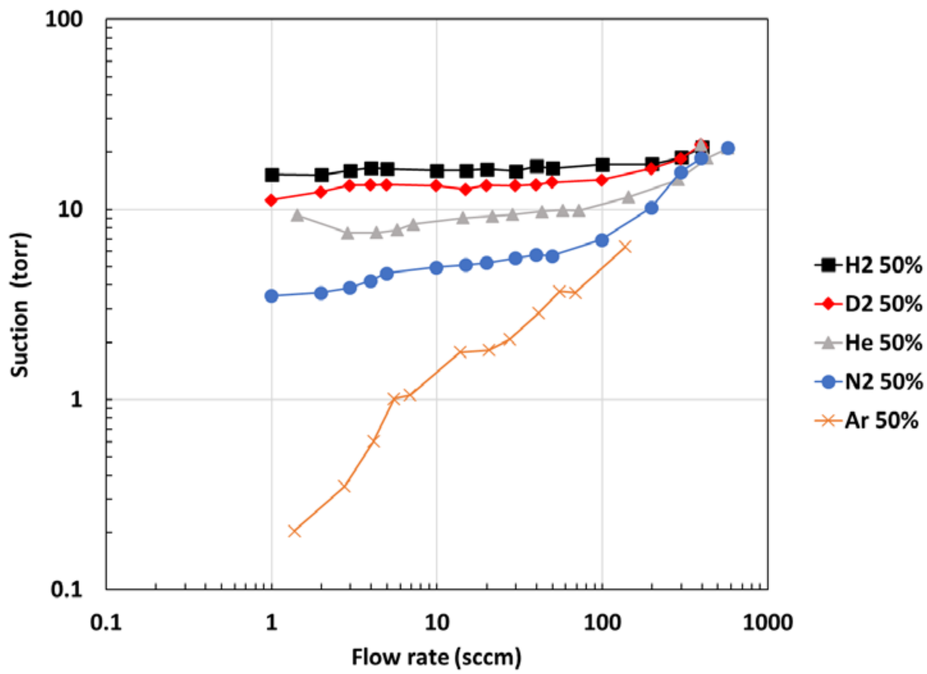

Figure 3-15: HiPace 80 (45,000 rpm) suction pressure vs flow rate, 300 Torr MB-601 discharge

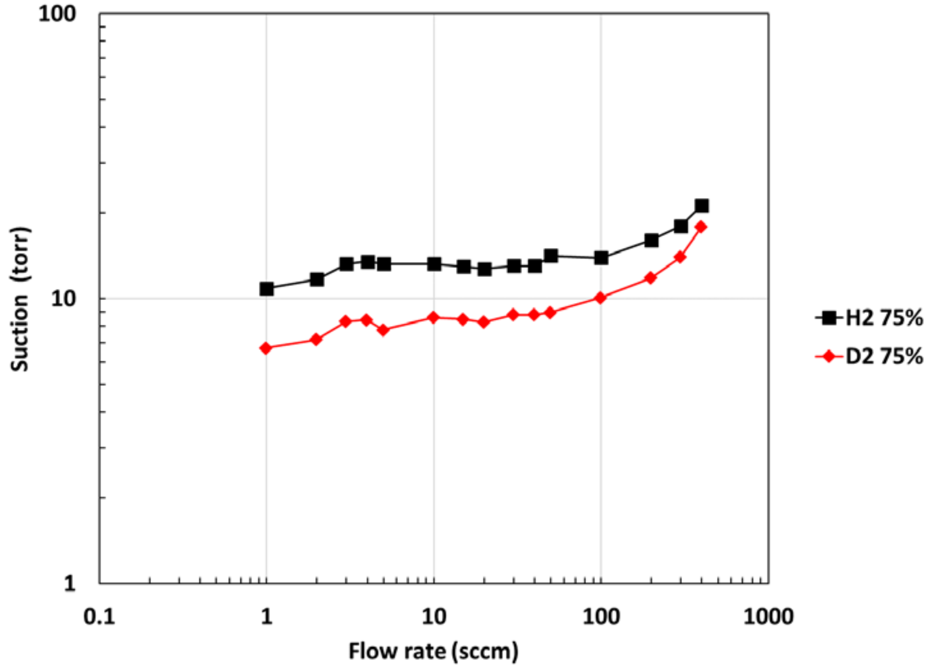

Figure 3-16: HiPace 80 (67,500 rpm) suction pressure vs flow rate, 300 Torr MB-601 discharge

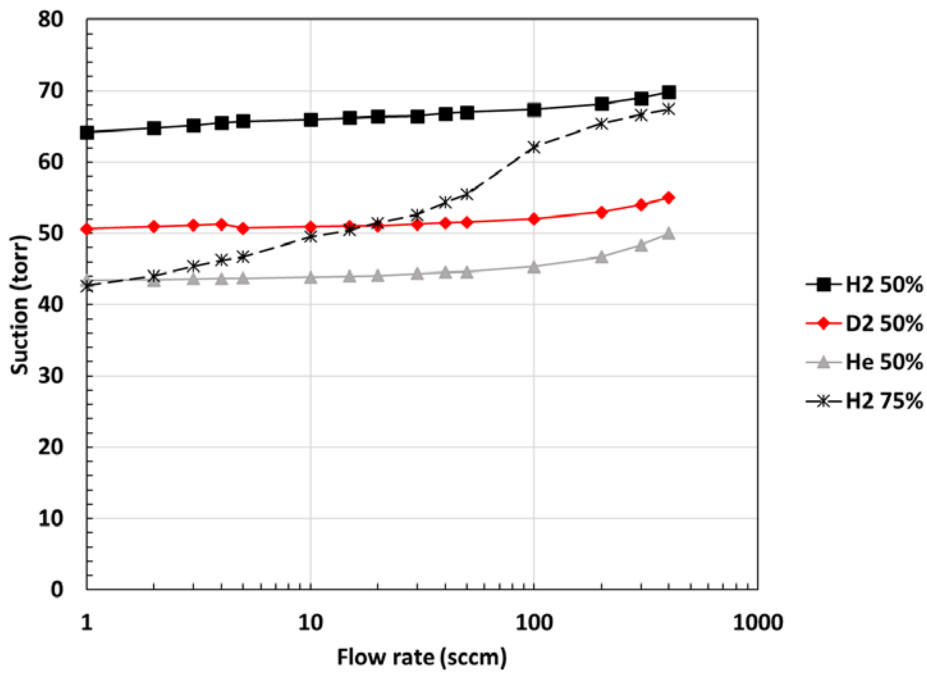

Figure 3-17: HiPace 80 suction pressure vs flow rate, 750 Torr MB-601 discharge. Rotational speeds: 45,000 rpm (50%) and 67,500 rpm (75%)

H_2 and D_2 are consistently at higher suction pressures that the He, N_2, and Ar, having relatively linear and flat behaviors for the HiPace 80 suction pressure compared to the HiPace 80 discharge pressure and gas flow rates, respectively. The HiPace 80 required lower pressures in order for all rotational speed setpoints to be reached without faulting for each gas. As the rotational speeds increase to 67,500 rpm at 150 torr MB-601 discharge pressure, D_2 follows similar pumping behavior as He, N_2, and Ar. H_2 requires speeds up to 90,000 rpm to approach the pumping behavior as the other gases above 10 sccm. For N_2 and Ar, the rotational setpoint was not reached for any speed at 750 torr MB-601 discharge, was reached only at 45,000 rpm at 300 torr, and was reached for all three speeds at 300 torr. The HiPace 80 was only able to maintain rotational speed for krypton at 1 sccm and 150 torr MB-601 discharge. The flow rates at which the HiPace 80 failed to maintain the rotational speed setpoint are detailed in Table 3-4.

Table 3-4: Flow rates HiPace 80 failed to maintain rotational speed

MB-601 discharge (torr)	Rotational speed (rpm)	Gas	Flow rate at speed failure (sccm)
150	45,000	H_2	No Failure
		D_2	No Failure
		He	No Failure
		N_2	No Failure
		Ar	421
		Kr	2
	67,500	H_2	No Failure
		D_2	No Failure
		He	No Failure
		N_2	297
		Ar	140
		Kr	N/A
	90,000	H_2	No Failure
		D_2	No Failure
		He	28.7
		N_2	39.6
		Ar	28.1
		Kr	N/A
300	45,000	H_2	No failure
		D_2	No failure
		He	No failure
		N_2	No failure
		Ar	275
		Kr	N/A
	67,500	H_2	No failure
		D_2	No failure
		He	N/A
		N_2	N/A
		Ar	N/A
		Kr	N/A
300	90,000	H_2	N/A
		D_2	N/A
		He	N/A
		N_2	N/A
		Ar	N/A
		Kr	N/A
750	45,000	H_2	No failure
		D_2	No failure
		He	No failure
		N_2	N/A
		Ar	N/A
		Kr	N/A
	67,500	H_2	No failure
		D_2	N/A
		He	N/A
		N_2	N/A
		Ar	N/A
		Kr	N/A
	90,000	H_2	N/A
		D_2	N/A
		He	N/A
		N_2	N/A
		Ar	N/A
		Kr	N/A

4.0 Conclusions

The results obtained show baseline pump characteristics of the Pfeiffer Vacuum Products HiPace 80 turbomolecular pump and Senior Aerospace Metal Bellows MB-601 pump (heads connected in series) combination. The test conditions were not standard for the HiPace 80, as the discharge pressures during static tests went above the recommended maximum operating pressure of 17 torr, and flow tests were performed for three speeds at flow rates of 1 – 400 sccm (H_2 reference). The HiPace 80 was only able to pump fix gases with all three rotational speeds at low pressures (≤ 150 torr), but was only able to pump H_2, D_2, He, and N_2 for limited flow rates and/or rotational speeds at 750 and 300 torr. The HiPace 80 has a limited ability to pump Kr at even 150 torr, with only 1 sccm being capable before failing to maintain 45,000 rpm, while being unable to pump flowing Kr at higher pressures.

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