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# **Evaluation of Alternative Vacuum Pumps** for Tritium Service

Lucas M. Angelette September 2019 SRNL-STI-2019-00534, Revision 0

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# Development of Alternative Vacuum Pumps for Tritium Service

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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. The pumps would need to operate at lower rotational speeds to handle higher pressures and flow rates, which is best suited for Molecular Drag Pumps (MDP). To determine this, the vacuum pump characteristics must be determined for the MDP. This report details pump characteristics using an MDP backed by a Metal Bellows (Met-Bel) pump under static and flow conditions for various gases.

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# LIST OF ABBREVIATIONS

Ar	Argon		
CV	Control Volume		
$D_2$	Deuterium		
FC	Flow Controller		
GCF	Gas Correction Factor		
H <sub>2</sub>	Hydrogen		
Не	Helium		
HF	Hydrogen Fluoride		
IVG	Ion Vacuum Gauge		
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		
RD	Rupture Disk		
sccm	Standard cubic centimeters per minute, volumetric flow		
SRNL	Savannah River National Laboratory		
SRS	Savannah River Site		
TC	Thermocouple		
TCVG	Thermocouple Vacuum Gauge		

#### **1.0 Introduction**

The Normetex<sup>®</sup> 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<sup>®</sup> halted production of the scroll pumps. Since then, researchers worldwide have been searching for a viable replacement for the Normetex<sup>®</sup> scroll pumps. There have been studies using just metal bellows (Met-Bel) pumps in series<sup>1</sup> and a Molecular Drag Pump (MDP) backed by an off-the-shelf (OTS) scroll pump.<sup>2</sup> The Met-Bel pumps in series cannot achieve the vacuum levels of the Normetex<sup>®</sup> pumps, and the MDP backed by an OTS scroll pump contains components that are not compatible for tritium service. This led to identifying the MDP backed by a Met-Bel as a possible alternative to the Normetex<sup>®</sup> pumps, but more information was needed on how the pumps performed while in series. This report details the pumping characteristics of the MDP with the Met-Bel backing pump under static and flow conditions for select gases.

#### 1.1 Identification of the Primary Pump

The Normetex<sup>®</sup> pump that has been in use worldwide has a set of highly desired characteristics for tritium processing: no oil lubricants, no polymer wetted-materials, vacuum levels as low as 0.001 torr at the inlet, and discharge pressures up to 250 torr.<sup>3</sup> 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.<sup>4</sup> Met-Bel pumps have an all-metal wetted component design similar to the Normetex<sup>®</sup>, 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<sup>®</sup> 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. Since the Met-Bel pumps are limited to around 30 torr at the inlet, use of turbomolecular pumps will not be sufficient if backed by the Met-Bel pump. However, a similar pump to the turbomolecular is the MDP. Using a different design, the MDP is rated for a continuous discharge pressure of 30 torr and a max continuous inlet pressure of 7.5 torr, both for nitrogen. Also, the MDP has an operational rotation speed of 27,000 rpm, while the turbomolecular pump has a rotation speed of 90,000 rpm. Data from the manufacturer is limited, as the data used a backing pump that was capable of <1 torr ultimate vacuum. It is of interest to determine the pumping characteristics of the MDP with a backing pump that has an ultimate vacuum greater than 10 torr.

#### 1.2 Pump Test Scope

The scope of the pump testing includes baseline pump curves with the MDP backed by a Met-Bel for various gases. The MDP/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 MDP has greased bearings. The pump curves of interest are pressure comparisons of the suction and discharge of the MDP under static and gas flow conditions. The gases of interest include nitrogen  $(N_2)$ , argon (Ar), helium (He), hydrogen  $(H_2)$ , and deuterium  $(D_2)$ .

#### **2.0 Experimental Procedure**

#### 2.1 Experimental Approach

The system fabricated to test the MDP/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 MDP is an MDP Model 5011 manufactured by Pfeiffer Vacuum Products, formerly Adixen Alcatel. The Met-Bel is a MB-601 manufactured by Senior Aerospace Metal Bellows, with the heads piped in series. 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 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 is 1.01. One control volume, 0.3 L, was used to incrementally dose the system under static conditions, and a second control volume, 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 monitored the MDP inlet to measure the high vacuum levels of the MDP. A thermocouple vacuum gauge (TCVG) was placed on the MDP outlet to monitor the vacuum levels during system evacuation and lowpressure 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.

#### 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 with an AMD4 diaphragm pump. 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 MDP. This was repeated until the MDP suction pressure was 6 torr or the MDP was unable to maintain full rotational speed of 27,000 rpm.

#### 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 with an AMD4 diaphragm pump. Next, the system was pressurized to approximately 800 torr using the 5 sccm MFC before opening the system vent valve, where the discharge was maintained at approximately 750 torr. 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.

#### 3.0 Results and Discussion

#### 3.1 Static Testing

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



Figure 3-1: Flow path for static testing

The pressure comparison for the MDP and the MB-601 are shown in Figure 3-2 and Figure 3-3, respectively. It should be noted that a cold cathode ion gauge was used for suction pressures below 7.0E-03 torr. Above these values, the measurements were taken using a Baratron (capacitance manometer), so a slight discontinuity appears in the figures. As the suction pressure plateaus (5.5E-05 for N<sub>2</sub> and Ar, 3.5E-05 for He), the MDP is reaching the vacuum limits for that particular gas in the current test system. The MDP is rated for a vacuum level of 1.0E-06, but that is with backing pumps that maintain lower MDP discharge pressures than the MB-601.

The MDP pressure comparison indicates that nitrogen and argon are able to be discharged at higher pressures (>40 torr) while maintaining suction pressures below 0.01 torr. However, it should be noted that at an argon discharge pressure of 56 torr, the MDP was not able to maintain full rotational speed of 27,000



rpm. The MDP was not able to maintain the suction pressures lower than 1.0E-02 torr, above a discharge of 12 torr for hydrogen, 20 torr for deuterium, 33 torr for helium, 42 torr for nitrogen, and 52 torr for argon.

Figure 3-2: MDP static suction vs discharge pressures.



Figure 3-3: MB-601 static suction vs discharge pressures.

The discharge and suction comparisons raise an interesting trend in the pumping capability of the gases. For suction pressures less than 0.01 torr the discharge pressures capable for a given suction pressure are aligned closer to the distribution of molar masses for the gases, except for helium and deuterium with similar molar mass, as shown in Table 3-1. Helium and deuterium, despite having similar molar masses, have drastically different pumping behaviors that are more in line with the differences in viscosity. Helium and nitrogen have similar viscosities at 20°C, and the suction pressures are very close for discharge pressures above 45 torr. The different pumping behaviors of the gases need to be accounted for in future process designs.

Gas	Viscosity <sup>5</sup> (x10 <sup>7</sup> poise)	Molar Mass (g)
H <sub>2</sub>	920	2.016
He	1950	4.003
D2	1250	4.024
N <sub>2</sub>	1760	28.014
Ar	2200	39.948

Table 3-1: Viscosity and molar mass of test gases

#### 3.2 Flow Testing

Several tests were performed to determine the operational suction and discharge pressures of the MDP backed by a MB-601 under gas flow conditions. The flow path of the flow tests is shown in Figure 3-4. These tests included testing the previous gases, except deuterium, at increasing flow rates, in sccm: 1, 2, 3, 4, 5, 10, 20, 30, 40, 50, 100, 200, 300, 400 with  $H_2$  reference. Deuterium was measured at flow rated up to

50 sccm. To accomplish the flow tests, the system initially evacuated using the OTS MDP pumping station. The system was then pressurized, with the pumps energized, to 800 torr before opening the vent valve. The MB-601 discharge pressure was nominally 754 torr during the testing.



Figure 3-4: Flow path schematic for flow tests

The suction and discharge pressure comparisons under gas flow are shown in Figure 3-5, and the suction pressure/flow rate comparison is shown in Figure 3-6. Deuterium and helium are able to continue pumping at discharge pressures up to 64 torr, hydrogen up to 78 torr, and nitrogen up to 50 torr, but argon is only able to be pumped up to 44 torr discharge pressure. At an argon flow rate of 200 sccm flow with hydrogen reference (275 sccm with GCF), the MDP is unable to maintain the 27,000 rpm rotational speed. No additional testing was performed for argon above 200 sccm (275 sccm with GCF). This indicates that with atmospheric pressure at the discharge of the MB-601, the MDP could maintain the 27,000 rpm rotational speed with 400 sccm of gas for nitrogen (396 sccm with GCF) and hydrogen, and 400 sccm for helium (575 sccm with GCF). Deuterium was only measured to 50 sccm (49.5 sccm with GCF), and at that point the MDP was able to maintain 27,000 rpm.

The limits of the MDP/Met-Bel combination appear to be the ultimate vacuum the Met-Bel can achieve. If the Met-Bel could achieve a suction pressure below 40 torr, the MDP would be able to achieve much higher vacuum levels at the inlet. In the current configuration, the MDP is limited by the MB-601 compression ratio when discharging to near atmospheric pressure.



Figure 3-5: MDP suction vs discharge pressures with gas flow



Figure 3-6: MDP suction pressure vs flow rate with GCF applied

#### 4.0 Conclusions

The results obtained show baseline pump characteristics of the Pfeiffer Vacuum Products MDP 5011 and Senior Aerospace Metal Bellows MB-601 pump combination. The test conditions were not standard for the MDP, as the discharge pressures of the MDP during static tests went above the recommended maximum operating pressure of 30 torr, and all flow tests were performed above the 30 torr manufacturer recommended threshold. The MDP was able to function at full rotational speeds for all the tests except with argon when the discharge pressure of the MDP was above 55 torr for a static system or a flow rate of 200 sccm (275 sccm with GCF).

#### 5.0 Recommendations, Path Forward or Future Work

The discharge pressure of the MDP is dependent on the suction capabilities of the MB-601. Additional testing to determine the system performance at pressures higher and lower than atmospheric would expand the baseline data needed for process design. Also, the MDP manufacturer, Pfeiffer Vacuum, has indicated that the control unit for the MDP 5011 will no longer be sold individually, other than in a pumping station. A comparable alternative was recommended as the Pfeiffer Vacuum HiPace 80 Hybrid Turbopump, which has an adjustable operating rotational speed between 45,000 - 90,000 rpm. Additional testing is recommended to determine the baseline characteristics of the HiPace 80.

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