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Metal Bellows Valve Reliability Testing

Paul R. Beaumont

September 2019

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PREFACE OR ACKNOWLEDGEMENTS

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EXECUTIVE SUMMARY

Swagelok B® series metal bellows-sealed valves were tested for their cycle performance longevity to evaluate their use in tritium service. The valves selected differed in the size of their pneumatic actuator and the stem tip material. Valves tested had one of three actuator sizes (1C, 3C, or 5C) and one of two stem tip materials: a cobalt-based metal, Stellite™, and a polyimide material, Vespel®.

An automated valve manifold was constructed, allowing for the simultaneous cycling of a total of 10 valves. Automation controls allowed for user inputs, controlling the number of valve cycles per test and the number of tests per experiment. Valves installed on the valve testing manifold to be cycled following a valve frequency test plan. After a defined number cycles, the valves would undergo leak testing using an inline leak detector. The inline helium leak detector provided qualitative detection of any failing or failed valves. If a valve had failed, a leak rate greater than at $1.0x10^{-7}$ std cc He/sec, the valve would be removed from the manifold and quantitative leak rate measurements made. After specific cycling milestones were reached, quantitative valve leak rate measurements were performed to assess the adequacy of the qualitative measurement method.

Valve evaluation began with initial leak tests: valve inlet/outlet seat rates and total body leak rate. The as-received 1C valves, with PTFE stem tips, passed initial leak tests; however, stem tips replacements with either metal or polyimide stem tips essentially created valves which failed subsequent leak tests and were not subject to valve cycling. The 3C actuated valves had been cycled 150,000 times, resulting in 60% of the Stellite™ and Vespel® tipped valves still passing the leak rate criteria. Failure analysis on the other 40% has yet to be conducted. Eight of the 5C valves had reached 150,000 cycles with 80% of the valves passing the leak rate criteria: two Vespel® tipped 5C valves failed leak rate testing after 120,000 cycles.

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
IV	Isolation valve
MSLD	Mass Spectrometer Leak Detector
psi	pounds per square inch – a unit of pressure
psia	Pounds per square inch absolute – a unit of pressure
psig	pounds per square inch gauge – a unit of pressure
PT	Pressure transducer
PTFE	Polytetrafluoroethene (Teflon™)
SRNL	Savannah River National Laboratory
SS	Stainless steel
Std cc	Standard cubic centimeter – a unit of volume
TV	Test valve
UI	User Interface
VP	Vacuum pump

1 Introduction

For tritium processing operations, it is important to use equipment that minimizes components containing polymers. Tritium has the potential to react and degrade polymers resulting in equipment failure. In the case of fluoro-containing compounds such as Teflon™, it also the potential to generate HF (hydrofluoric acid) and TF (tritium fluoride) vapor.[1,2] This vapor will corrode stainless steel, resulting in further damage and potential equipment failure.

Minimization of inert glovebox space and tritium compatibility of valves are two key aspects of this study. The use of the smallest possible size actuator for a cobalt-based metal, Stellite™, tipped valve or polyimide, Vespel®, tipped metal bellows-sealed valves will reduce component size, required glovebox size/volume, and possible valve replacement frequency. However, valve cycle life for pneumatically actuated metal bellows-sealed valves with different size actuators and stem tipe materials has not been determined. Swagelok B series metal bellows-sealed valves[3] have been identified and selected for evaluation with two different stem tips and three different actuator sizes.

1.1 Valve Selection

The Swagelok B-series bellows-sealed valve is available from Swagelok in a variety of sizes and configurations.[3] For this study, 316 stainless steel (SS) body, ¼" butt weld tubes with female 4-VCR fittings, gasketed body-to-bellow seal valves were fitted with normally-closed pneumatically actuators were chosen for testing. Valve working pressures as high as 400 psig (27.5 bar) at 600 °F (315 °C) give this valve flexibility for various process applications (Table 1-1).[3] Three actuator sizes were selected (Table 1-2), referred to henceforth to their size designation of the actuator (Figure 1-1), to investigate the potential sizing options for each stem tip material. Stem tip selection was based on previously determine reports of materials compatible in a tritium process system.[1] Stellite™, a cobalt-chromium alloy, is a hard material with good corrosion resistance, is suitable for use with larger actuators capable of delivering greater amounts of force to form a seal than smaller sized actuators. Vespel® is a softer polyimide polymer material that has acceptable tritium compatibility characteristics which can use a smaller sized actuator to form a seal than a metal tipped valve and reduce the valve footprint inside a confined space (e.g. glovebox). Technical drawings and dimensions of the valves can be found in Appendix A, Appendix B, and Appendix C.

Table 1-1. Working pressures of 316 SS BG Series valves.

Temperature		Working Pressure	
°F	°C	psig	bar
-20 to 100	-28 to 37	1000	68.9
200	93	830	57.1
300	148	660	45.4
400	204	500	34.4
500	260	450	31.00
600	315	400	27.5

Table 1-2. Swagelok B-Series valves selected.

Part #	Actuator Size	Stem Tip Material
SS-4BK-V51-1C	1C	Teflon™
SS-4BG-V51-3C	3C	Stellite™
SS-4BG-V51-VP-3C	3C	Vespel®
SS-4BG-V51-5C	5C	Stellite™
SS-4BG-V51-VP-5C	5C	Vespel®

The Table 1-2 valve with the 1C actuator is supplied from the vendor with PTFE (Teflon™) stem tips which have been deemed incompatible with tritium and required replacement with a more tritium compatible material.[1,4] As a work-around, the Teflon™ stem tips where replaced with Stellite™ and Vespel® tips (Figure 1-2). The tips required separate procurement including new body-to-bellows gaskets (Table 1-3). The larger 3C and 5C valves were available from the vendor with Stellite™ and Vespel® tips.

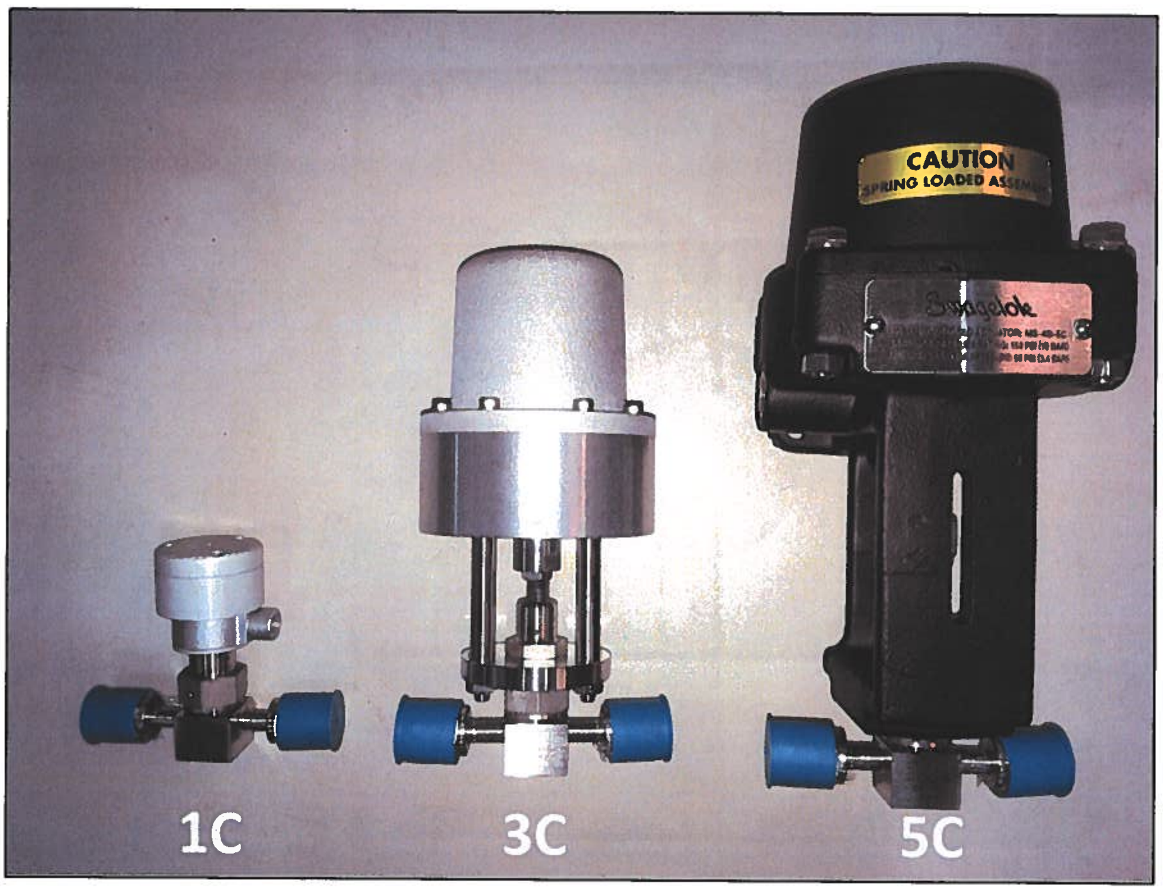

Figure 1-1. Swagelok B-series bellows-sealed valves, noting the three actuator sizes of 1C, 3C, and 5C.

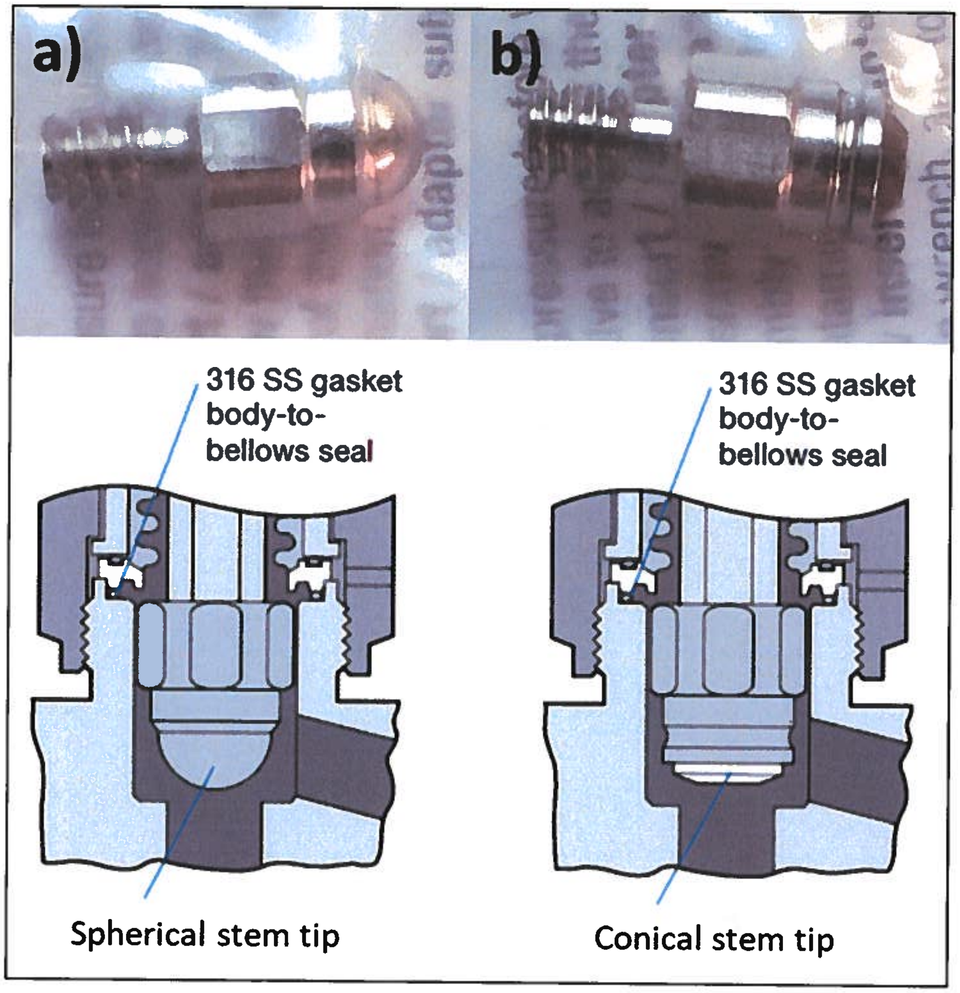

Figure 1-2. Replacement stem tips a) Stellite™ and b) Vespel® with depiction of tip shape and configuration within valve.

Table 1-3. Swagelok part numbers for each stem tip and replacement gasket kit.

Part #	Description
SS-4B-ST-K5	Stellite™ spherical stem tip
SS-4B-VP-K5	Vespel® conical stem tip
SS-4BGO-K5-SV	Gasket kit

1.2 Valve Testing Scope and Manifold Design

To evaluate the longevity of the valves as a function of valve cycle, a valve testing manifold was designed (Figure 1-3) and constructed to automatically cycle the valves for an extended and predetermined number of cycles, testing seating performance of the stem tips, simulating a lifetime of operation. The manifold itself was constructed from stainless steel tubing and various Swagelok compression and VCR fittings. Valve cycle testing followed the ASTM F1373-93, "*Standard Test*

Method for Determination of Cycle Life of Automatic Valves for Gas Distribution System Components."

The valve manifold applied N_2 pressure (40-50 psig) to one side of the valve while pulling vacuum (target 0.0009 psia) on the other side using an Edwards nXDS15iR vacuum pump. Using National Instruments™ LabVIEW automation control software, the system would operate unattended following user defined parameters, all while logging data. Such parameters included valve open and close times, manifold pressures, leak test durations, number of valve cycles per test, and number of complete cycles, discussed in greater detail below.

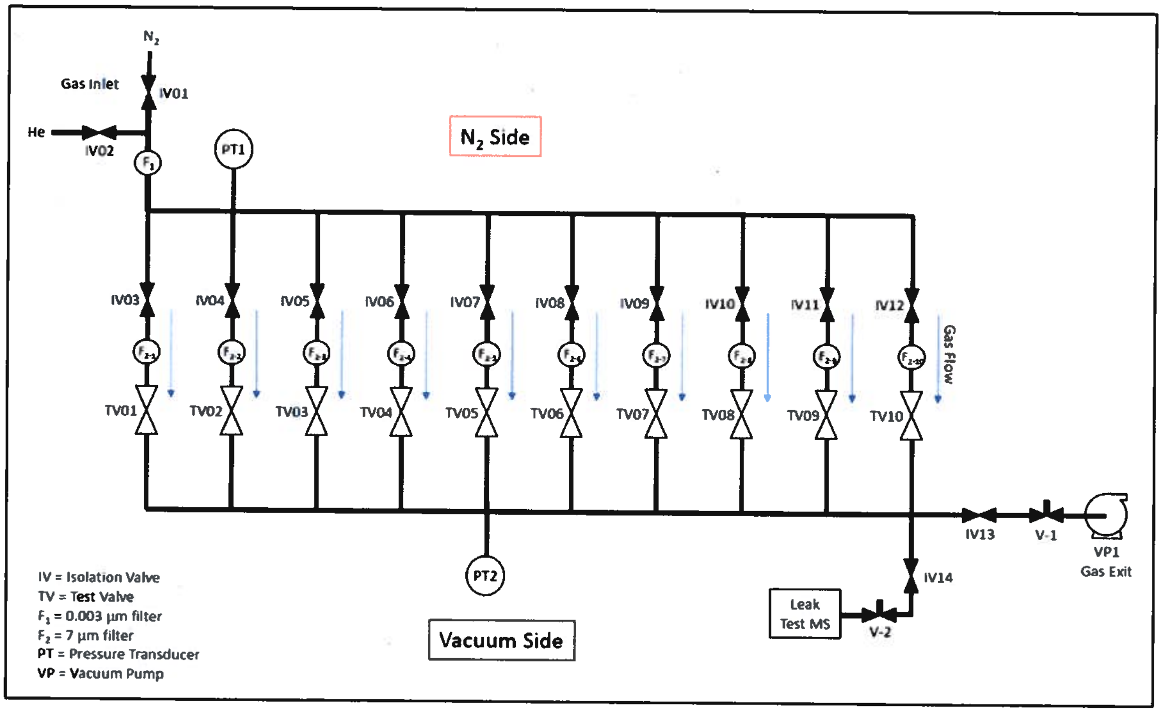

Figure 1-3. Schematic of the valve testing manifold.

After performing a predefined number of cycles, the valves would undergo a qualitative leak test, looking for gross failures. Using an inline Mass Spectrometer Leak Detector (MSLD), each valve was leak tested by pressurizing the inlet side of the valve with He while keeping the outlet under vacuum. Performing this test allowed for the identification of failed or failing individual valves prior to sending all valves for quantitative leak testing. This benefited the project through time and cost savings.

Utilizing a *lessons learned* moment from previous work,[5] 0.003 µm filter (F_1 in Figure 1-3) was installed after the working gas inlets to prevent and foreign debris from entering the system, potentially resulting in particulate contamination and artificial valve failure. Additional protection was applied with the installation of a series of 7 µm filters (F_2 in Figure 1-3) located on the inlet of each test valve (TV).

1.3 Automation

As mentioned previously, the manifold was controlled through LabVIEW automation, guided by programmed valve logic and manually entered test parameters for each experiment. The LabVIEW

User Interface (UI) in Figure 1-4 provide a high level of control and variable flexibility, allowing for adjustments to be made reflecting differing actuator sizes, leak detector sensitivity, etc., as described in the annotated boxes below.

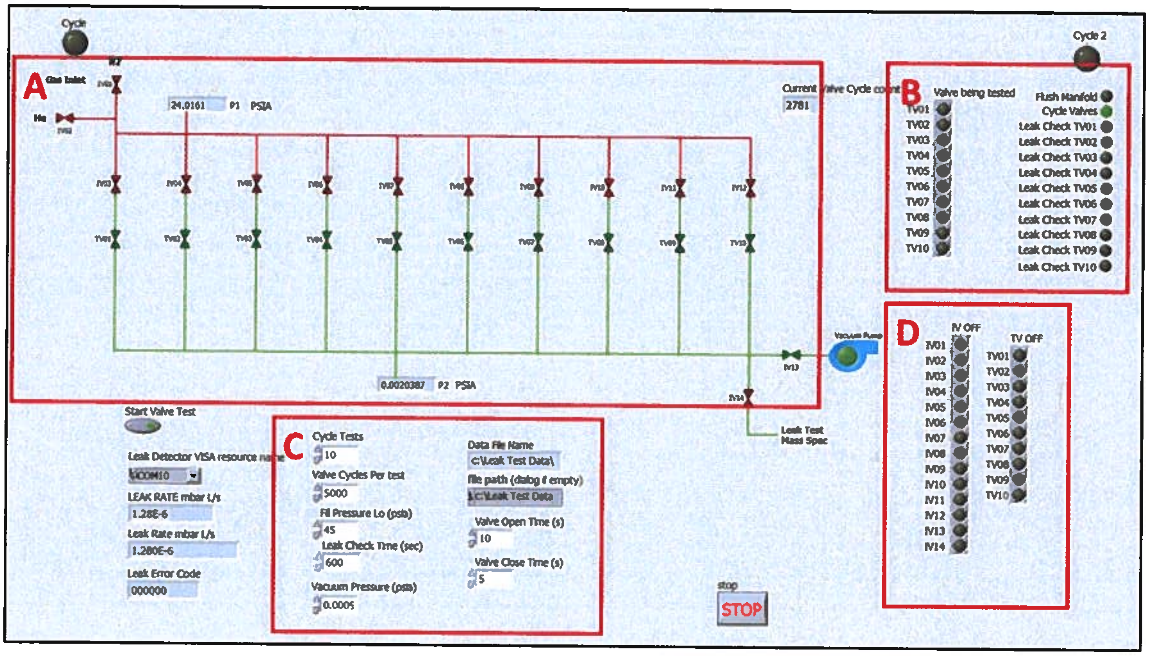

Figure 1-4. Annotated depiction of the LabVIEW UI used in the control and automation of the valve testing manifold.

1.3.1 *Manual Valve Control (A)*

The gas flow and vacuum within the manifold is controlled and regulated by 14 isolation valves (IV, Swagelok, part # SS-BN8VCR8-2C) in addition to the ten TV. Each valve may be manually selected individually to open or close. The red *bowtie* indicates a closed valve while a green *bowtie* indicates an open valve.

1.3.2 *Manifold Status (B)*

During operation, the current status, or procedural step, can be determined quickly and easily using the manifold status indicators.

1.3.3 *Manually Adjustable Variables (C)*

As one of the most important features of the manifold's design, having the ability to manual control and adjust severable variables and testing criteria lends itself to increasing the overall versatility of the program.

1.3.3.1 *Cycle Test*

This valve sets the number of complete cycles per experiment. A complete cycle is defined as the combination of valve cycling followed by the inline leak test. After the number of Cycle Tests has been performed, the program concludes the experiment and stops logging data.

1.3.3.2 *Valve Cycles per Test*

This variable determines the number of valve cycles performed prior to performing the leak test.

1.3.3.3 *Fill Pressure Lo*

The inlet side of the manifold is pressurized with N_2. As the valve cycling progresses, the pressure gradually drops. This variable sets the minimum pressure of the inlet side of the manifold. When the minimum pressure is reached, an IV is automatically opened to repressurize the system, thus maintaining the valve test pressure.

1.3.3.4 *Leak Check Time*

After valve cycling is complete, the individual valves undergo leak testing using the inline leak detector. This variable controls the length of time each valve is leak checked.

1.3.3.5 *Vacuum Pressure*

The outlet side of the manifold is kept under vacuum. After various steps, the testing will not continue until the manifold pressure has been reduced to this manually defined value. To maintain the reduced pressure, an IV is opened to the vacuum pump (VP) until the vacuum pressure valve has been reached. This feature is also used as added protection for the leak detector.

1.3.3.6 *Valve Open Time*

This variable controls the amount time allotted for opening the test valves (TV). As the three actuators tested are differing in size, the larger ones require more time to open as compared to the smaller ones. This allows assurance that the valve will be completely opened before progressing to the next step.

1.3.3.7 *Valve Close Time*

Similar to the *Valve Open Time*, this controls the time allotted for the closing of the valves.

1.3.4 *Valve Control Center (D)*

Over the duration of the evaluation, valves will fail and be removed from the manifold. Once removed, the pneumatic line operating the valve is disabled, preventing the operation of the valve. When a TV is removed, the TV is disabled along with the IV leading to it. This feature has the ability to enable/disable all 14 IV and ten TV.

1.4 Valve Test Plan

The valve testing frequency plan has been summarized in Table 1-4. The valve 1C actuators with metal and polyimide stem tips were assumed to have lower cycle lives than valves with 3C and 5C actuators so initial leak tests were performed more frequently to verify the seating performance of these stem tips. The selected number of cycles performed prior to leak testing may be modified based on valve performance. Inline leak checks were performed at given intervals, as noted by the cycles. If no significant leaks were detected, testing was resumed. Although, if there was a leak detected deemed significant, the valve was removed from the manifold and quantitative leak testing performed. All valves within a group would periodically undergo quantitative leak testing to verify the qualitative inline tests results.

Table 1-4. Valve leak test frequency plan.

Valve Cycles	Leak Test Frequency	
	1C Valves	3C and 5C Valves
Up to 50 cycles:	Leak test every 10 cycles	
Up to 100 cycles:	Leak test every 20 cycles	
Up to 500 cycles:	Leak test every 50 cycles	
Up to 1,000 cycles:	Leak test every 100 cycles	Leak test every 100 cycles
Up to 5,000 cycles:	Leak test every 1,000 cycles	Leak test every 1,000 cycles
After 5,000 cycles:	Leak test 5,000 cycles	
Up to 20,000 cycles:		Leak test every 5,000 cycles
Up to 100,000 cycles:		Leak test every 10,000 cycles

1.5 Leak Detection

As a means of qualitative leak detection, a Pfeiffer Vacuum D-35614 Asslar helium MSLD was installed inline on the manifold, allowing for periodic leak tests to be performed on the valves without the need of uninstalling for quantitative leak testing. This saved time and funding by reducing the scheduled frequency of quantitative leak testing and only performing these tests after significant cycling milestones or noticeable points of failure.

Quantitative valve leak testing was performed after removing the valve from the test manifold. Testing was in accordance with ASME Section V, Article 10, helium (He) seat leak testing (internal leak check past the valve seat or through the closure member when the valve is closed) and body testing (external leak check to evaluate leakage from pressure inside the valve out to the surrounding atmosphere) using a calibrated helium MSLD.

For inline leak testing, the helium pressure was maintained at approximately 65 psig on the inlet of the valve. For quantitative leak detection, the helium pressure was approximately 62 psig for all tests. Valve inlet testing had pressurized He on the valve inlet fitting, with the valve in its normally closed position from the force of the actuator spring, and the MSLD connected to the valve outlet fitting. Valve outlet testing had pressurized He on the valve outlet fitting, with the valve in the normally closed position, and the MSLD connected to the valve inlet fitting. Body leak testing had the valve placed in a bell jar, the actuator pressurized to open the valve, and He supplied to one valve fitting with the other fitting capped. The He leak rate into the bell jar was measured using a MSLD. Valve failure was defined when the leak rate exceeded 1.00×10^{-7} std cc He/sec.

2 Experimental Procedure

All three types of valves underwent as-received quantitative leak testing before valve cycling. Before cycle testing, the 1C valves were also leak tested after the Teflon™ stem tips were replaced with the other stem tip materials. Six valves of each stem tip underwent initial, quantitative leak testing however, only five valves of each stem tip were cycled on the manifold.

Cycling experiments were conducted to test the seating (leak rate) performance of the stem tips over a predetermined number of cycles, simulating a lifetime of operation, as summarized in Table 1-4. Cycling tests were concluded with inline leak detection performed on each valve. After larger cycling milestones were reached, the valves were sent to the HPL for quantitative leak detection analysis as a means to verify that the valves were still operating within the leak rate limits and to verify the qualitative inline leak detection measurements. Valves that failed quantitative leak detection tests were removed from the manifold and the appropriate IV and TV were disabled in LabVIEW.

3 Results and Discussion

3.1 1C Actuated Valves

The 1C actuated valves come from Swagelok with a Teflon™ stem tip installed. The vendor states other stem tips are not a procurement option as the 1C actuator is incapable of applying sufficient force on the stem tip to form a quality seal when using materials harder than Teflon. Testing was conducted with the other stem tip materials anyway to validate the vendor's assertion since Teflon in not a compatible material for use with Tritium.[1,2,4]

The 1C valves with Teflon™ stem tips underwent initial quantitative leak testing to verify the valves were operational as received from the manufacturer with results summarized in Table 3-1. The color coding in the table has green for leak rates passing the leak rate criteria (less than 1.00×10^{-7} std cc per sec), yellow indicating cautionary leak rate results, and red indicating failed or unattainable (too high to be measured on the system) leak rate results. This color coding will be used in subsequent tables to highlight leak test results. The "<" or "---" symbols in the tables represent leak rates higher than the detectable limits of the MSLD.

Table 3-1. Summary of 1C valve leak tests before and after the Teflon™ stem tip replacement.

		Leak Rates (std cc He/sec)				
		Before Teflon Tip Replacement		After Teflon Tip Replacement		
Valve Name	Stem Tip	Inlet Seat	Outlet Seat	Inlet Seat	Outlet Seat	Body
1C01	Stellite	4.10E-08	3.40E-08	<	<	<
1C02	Stellite	3.40E-08	5.90E-08	<	<	<
1C03	Stellite	2.90E-08	4.20E-07	<	<	8.10E-07
1C04	Stellite	3.10E-08	5.80E-08	<	<	5.20E-07
1C05	Stellite	6.40E-09	5.80E-08	<	<	1.70E-05
1C06	Stellite	2.60E-08	5.70E-08	<	<	3.80E-08
1C07	Vespel	6.00E-08	5.50E-08	1.30E-08	3.60E-09	<
1C08	Vespel	4.30E-08	6.00E-08	1.80E-08	1.30E-09	<
1C09	Vespel	4.60E-08	6.90E-08	9.40E-10	1.00E-08	1.60E-07
1C10	Vespel	4.60E-08	4.90E-08	<	<	6.50E-08
1C11	Vespel	3.60E-08	4.70E-08	9.50E-10	1.30E-08	<
1C12	Vespel	4.30E-08	5.10E-08	<	<	1.60E-07

All as-received 1C valves passed the seat leak tests as well as the body leak tests and were deemed acceptable for further study. The Teflon™ stem tips were then replaced with either Stellite™ or Vespel® stem tips followed by quantitative leak testing (Table 3-1), resulting in much less favorable performance.

The 1C Stellite™ stem tip valves did not pass seat leak test criteria and were deemed unacceptable for testing. Despite most of the 1C Vespel® stem tip valves (1C07, 1C08, 1C09, 1C11) having lower seat leak rates than the original Teflon™ stem tips, the body leak rates for most of the valves exceeded the leak rate criteria so none of these valves were cycle tested.

Follow-up investigation into the valve reassembly process discovered the following. The vendor uses two different gaskets to seal B series valve bellows to the valve body. Both gaskets are 316 SS with Teflon™ stem tip valves ("BK" valves) using a Teflon™ coated gasket (the same PTFE Teflon™ as the stem tip material). Valves with Stellite™ or Vespel® stem tips ("BG" valves) utilize a silver plate 316 SS gasket.

The SS-4BGO-K5-SV gasket kit shown in Table 1-3 is supplied with a silver plated gasket. The original Teflon™ coated gaskets were replaced with the (silver plated) gasket from the kit when the valves were reassembled after stem tip replacements. The torque specification for valve reassembly for a silver plated gasket is larger than the torque specification for a Teflon™ coated gasket. It is believed the "4BK" marking on the valve body (indicating a Teflon™ stem tip valve and thus Teflon™ coated gasket) mislead staff members to use the lower torque value when assembling the valves. The data from Table 3-1 and valve test procedures support the errant torque specification conjecture. The explanation for valve 1C10 and valve 1C12 seat leak failures is still unclear.

3.2 3C Actuated Valves

The 3C actuated valves came with the option of being equipped with Vespel® or Stellite™ by the manufacturer. Twelve valves were procured, six of each stem tip, with as-received quantitative leak tests summarized in Table 3-2.

Table 3-2. Summary of 3C valve initial quantitative leak tests.

		Leak Rates (std cc He/sec)		
Valve Name	Stem Tip	Inlet Seat	Outlet Seat	Body
3C-1S	Stellite	1.20E-09	1.20E-09	1.30E-09
3C-2S	Stellite	1.30E-09	1.30E-09	1.20E-09
3C-3S	Stellite	1.20E-09	1.50E-09	1.20E-09
3C-4S	Stellite	1.40E-09	1.40E-09	1.20E-09
3C-5S	Stellite	9.40E-10	2.30E-09	1.30E-09
3C-6S	Stellite	1.20E-09	1.20E-09	1.20E-09
3C-1V	Vespel	1.40E-09	1.40E-09	1.10E-09
3C-2V	Vespel	1.30E-09	1.30E-09	1.20E-09
3C-3V	Vespel	1.40E-09	4.70E-09	1.20E-09
3C-4V	Vespel	1.50E-09	1.50E-09	1.20E-09
3C-5V	Vespel	1.20E-09	5.10E-09	9.60E-10
3C-6V	Vespel	1.20E-09	1.20E-09	1.10E-09

Following the valve leak test frequency plan (Table 1-4), the manifold was programmed to cycle the valves then perform an inline leak test after every 100 cycles until the 1000 cycle milestone was reached. During these initial valve cycles, manifold troubleshooting ran in concert with the cycling, namely with the leak detector. As a result, observed leak rates for the first 3000 cycles were found to be rather high, as observed in Figure 3-1. After additional modifications were made to the valve logic programming, allowing longer leak detection periods for each valve, producing markedly lower reported leak rates.

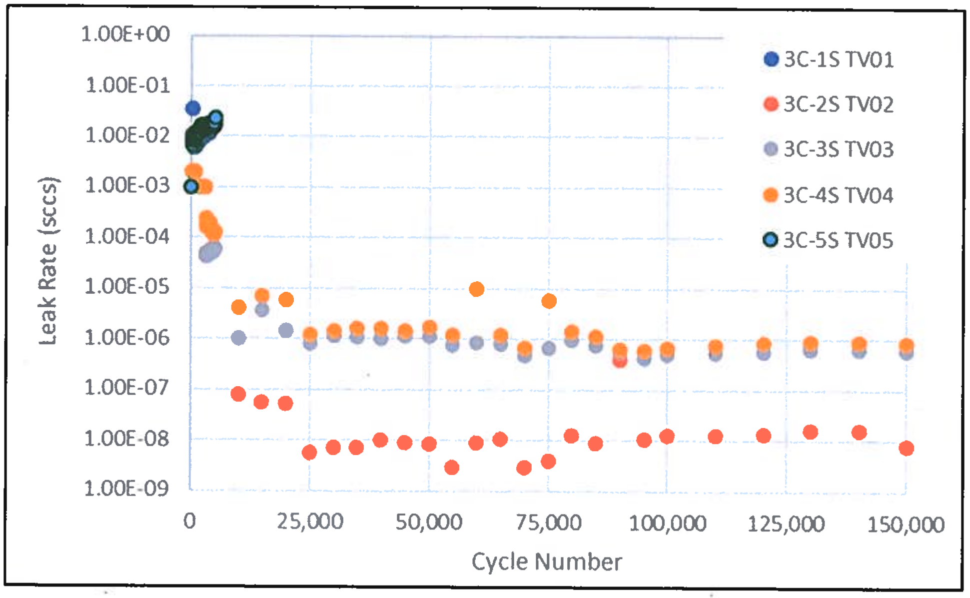

Figure 3-1. Inline leak rates for 3C actuated Stellite™ tip valves.

It should be noted; however, the inline (qualitative) leak rates were always a few orders of magnitude greater than the quantitative leak rates, lending to the acknowledgement that the inline leak detector be used for qualitative purposes only. Furthermore, there was a clear trend observed where the reported leak rates for each subsequent valve tested would be higher than the previous valve tested. This has been attributed to residual He in the system and the leak detector not having adequate time to reestablish a baseline reading between tests, something that can be corrected by increasing the leak check time (see section 1.3.3.4). The Vespel® tipped 3C actuated valves (Figure 3-2) displayed similar leak rate versus cycle number trends as obtained for the Stellite™ tipped valves: a dramatic drop in recorded leak rates after 3,000 cycles and successively higher leak rates for each subsequent valve tested.

After the inline leak detection time was increased, it was observed that valves 3C-1S and 3C-5S were not reaching the leak rate levels observed with the other three valves after 5,000 cycles. These two valves were then removed from the manifold and sent for quantitative leak testing which confirmed valves 3C-1S and 3C-5S had indeed failed body and seat leak tests (Table 3-2). The cause of the valve failures has yet to be determined.

Within the first 5,000 cycles of operation, it was noticed that some of the valves had an oily residue originating from the actuator as well as metal filings below it (Figure 3-3, Figure 3-4, and Figure 3-5). No conclusions were drawn from these observations and the eventual failure of specific valves.

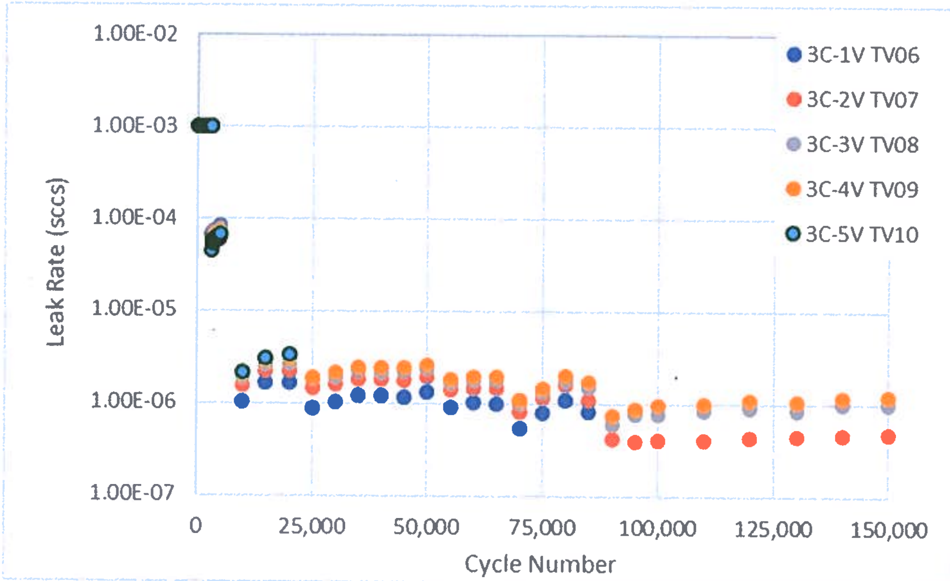

Figure 3-2. Inline leak rates for 3C actuated Vespel® tip valves.

Table 3-3. Quantitative leak test results for the 3C actuated valves at the 5,000, 50,000, and 150,000 cycle milestones.

HPL Tests		5,000 Cycles			50,000 Cycles			150,000 Cycles		
		Leak Rates (std cc He/sec)			Leak Rates (std cc He/sec)			Leak Rates (std cc He/sec)		
Valve Name	Stem Tip	Inlet Seat	Outlet Seat	Body	Inlet Seat	Outlet Seat	Body	Inlet Seat	Outlet Seat	Body
3C-1S	Stellite	---	---	---						
3C-2S	Stellite	Not tested			<1.5E-09	<1.5E-09	2.80E-09	<1.8E-09	<1.8E-09	<1.2E-09
3C-3S	Stellite	Not tested			<9.5E-10	<9.5E-10	<8.9E-10	<1.2E-09	<1.2E-09	<1.1E-09
3C-4S	Stellite	Not tested			<1.3E-09	<1.3E-09	<1.8E-09	<1.1E-09	4.90E-09	<2.1E-09
3C-5S	Stellite	---	---	---						
3C-6S	Stellite	Not tested			Not tested			Not tested		
3C-1V	Vespel	<1.3E-9	9.10E-09	<1.1E-9	<1.3E-9	<1.3E-9	<1.3E-9	Not tested		
3C-2V	Vespel	Not tested			<1.3E-9	<1.3E-9	<1.3E-9	<1.0E-09	2.00E-09	<1.5E-09
3C-3V	Vespel	Not tested			<1.5E-9	<1.5E-9	<1.6E-9	<1.4E-09	<1.4E-09	<1.1E-09
3C-4V	Vespel	Not tested			<1.8E-9	<1.8E-9	<2.0E-9	<1.7E-09	<1.7E-09	---
3C-5V	Vespel	<1.3E-9	<1.3E-9	---						
3C-6V	Vespel	Not tested			Not tested			Not tested		

The first 3C Vespel® tipped valve that failed was 3C-5V when it was noticed the valve periodically would remain closed during cycle testing. The valve failure was a result of mechanical failure of the actuator which would not open the valve. It was believed this event may have started as early as 3,000 cycles; however, it was too intermittent to draw attention and therefore cannot be confirmed.

At the 5,000 cycle milestone, the 3C-5V valve along with another valve believed to be operating successfully, 3C-1V, were removed from the manifold and sent for quantitative leak testing with the results shown in Table 3-3. The results show valve 3C-5V had developed a body leak but, passed the valve inlet and outlet leak tests similar to the results obtained for valve 1C08 shown in Table 3-1.

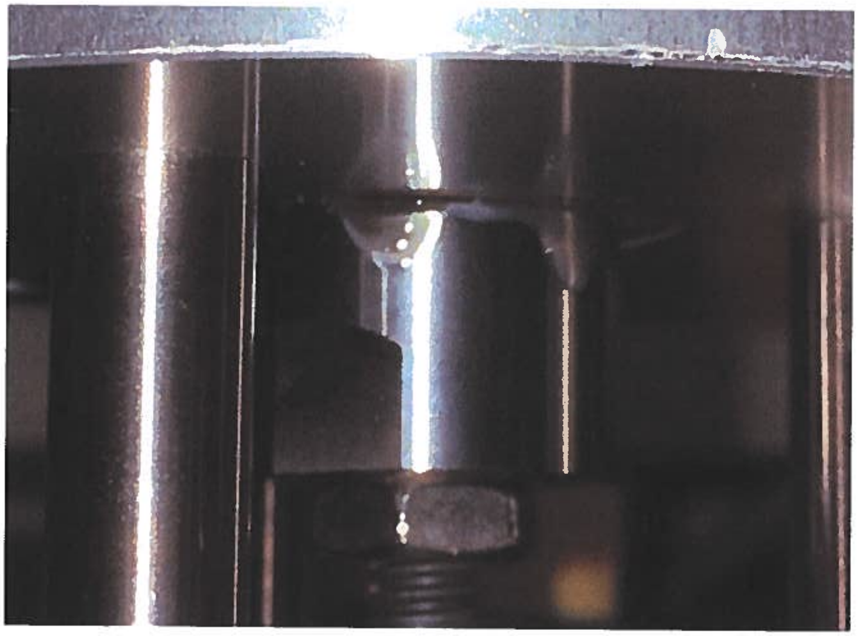

Figure 3-3. Oily residue observed on valve 3C-1V.

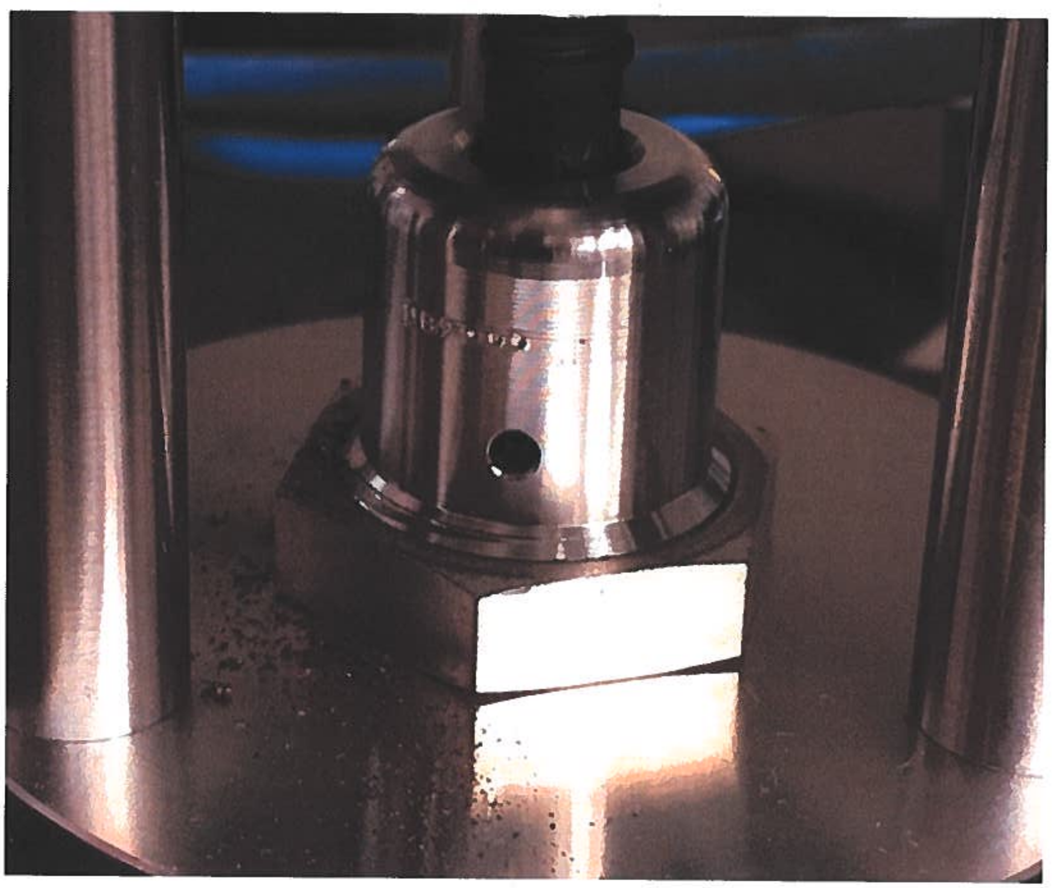

Figure 3-4. Metal filings observed on valve 3C-4S.

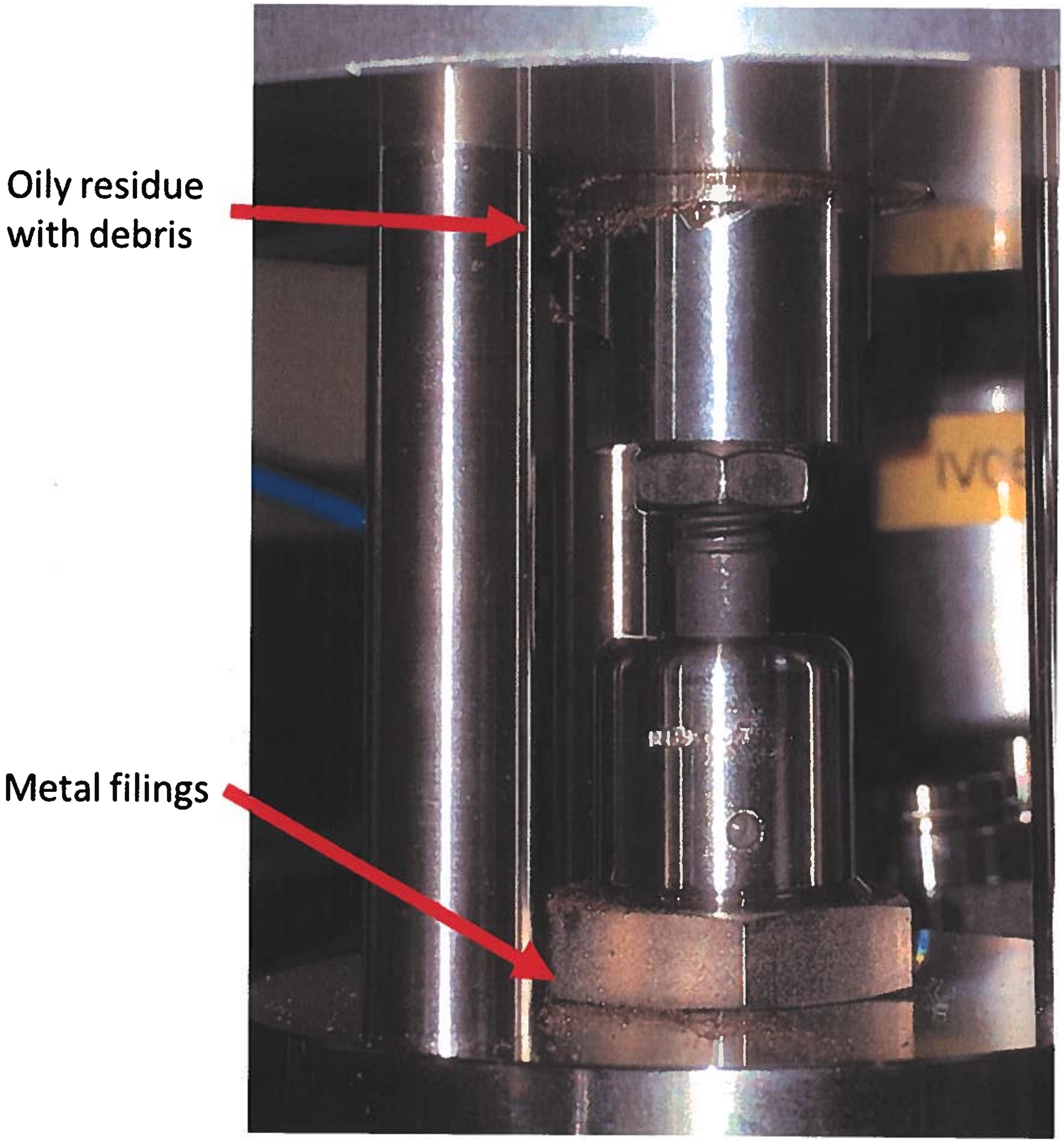

Figure 3-5. Metal filings and oily residue with emulsified debris of valve 3C-4S.

Valve 3C-5V was reinstalled into the test manifold and cycled up to 20,000 cycles (to obtain additional data on stem tip performance) but, was finally removed from testing after it became apparent that the actuator had completely failed. It is not clear if the body leak failure was caused by the actuator which subsequently failed or if the valve body failure subsequently caused the actuator to fail. The exact cause of failure is still to be determined.

The results of quantitative leak testing of valve 3C-1V after 5,000 cycles confirmed the valve was functioning properly. The inline leak testing using the MSLD supported the goal of using the inline leak testing as a screening mechanism to evaluate the need to remove valves from the system for qualitative leak testing.

After 50,000 cycles, the remaining 3C valves where removed from the test manifold and passed quantitative leak testing with the results shown in Table 3-3. Valve 3C-1V had developed a severe body leak after 85,000 cycles. This valve was removed from the manifold and cycling ceased. It was not sent for quantitative leak testing since manifold data showed it would still maintain pressure on the inlet side of the manifold when in the closed position but would actively leak through the body when vacuum was applied to the outlet side of the valve. A failure analysis is to be performed on the valve.

The remaining 3C valves (Stellite™ and Vespel® tipped) were sent for quantitative leak testing for the 150,000 cycle milestone to verify the inline leak detector data quality. As shown in Table 3-3, all of the valves except 3C-4V produced acceptable leak rates after 150,000 cycles. The valve 3C-4V body leak after 150,000 cycles was not observed by the inline leak detector. A post-test failure analysis was performed on the 3C-4V valve to determine the cause of the leak, yielding in no discernable evidences as to the source.

3.3 5C Actuated Valves

Just like the other valves, the 5C actuated valves also underwent and passed initial quantitative leak testing as summarized in Table 3-4. The 5C actuator can apply the largest force to the valve stem tips of the three actuators tested and these results are not surprising.

Table 3-4. Summary of 5C valve initial quantitative leak tests.

		Leak Rates (std cc He/sec)		
Valve Name	Stem Tip	Inlet Seat	Outlet Seat	Body
5C-1S	Stellite	<9.2E-10	<9.2E-10	<1.1E-09
5C-2S	Stellite	<8.6E-10	<8.6E-10	<1.2E-09
5C-3S	Stellite	<1.5E-09	<1.5E-09	<1.2E-09
5C-4S	Stellite	<1.4E-09	<1.4E-09	<1.2E-09
5C-5S	Stellite	<1.7E-09	<1.7E-09	<1.2E-09
5C-6S	Stellite	<1.6E-09	<1.6E-09	<1.2E-09
5C-1V	Vespel	<1.2E-09	<1.2E-09	<1.5E-09
5C-2V	Vespel	<1.1E-09	<1.1E-09	<1.7E-09
5C-3V	Vespel	<1.2E-09	<1.2E-09	<1.2E-09
5C-4V	Vespel	<1.2E-09	<1.2E-09	<1.2E-09
5C-5V	Vespel	<1.3E-09	<1.3E-09	<1.2E-09
5C-6V	Vespel	<1.1E-09	2.40E-09	<1.2E-09

Results of the inline leak detection for the 5C valve tests proved to be more consistent when applying the lessons learned from the 3C leak tests. For the 5C tests, leak detection times were increased to allow for the leak detector to reestablish a more stable baseline measurement; however, further improvements could be made. For example, when testing any particular valve, there was an initial spike in the leak detector readout, which then settles over time, as shown in Figure 3-6.

Allowing an adequate amount of time to settle would provide a leak rate more representative of the true leak rate of the valve as well as bring the baseline to a more uniform reading for each valve tested. This may lead to less of a cascade of leak rates for each valve. As observed, the Stellite™

tipped 5C valves in Figure 3-7 depict the same trend of increasing leak rates for each successive valve tested. As stated previously, this could have been mitigated by increasing the leak detection time for each valve, resulting in observed leak rates for each valve consistent with one another.

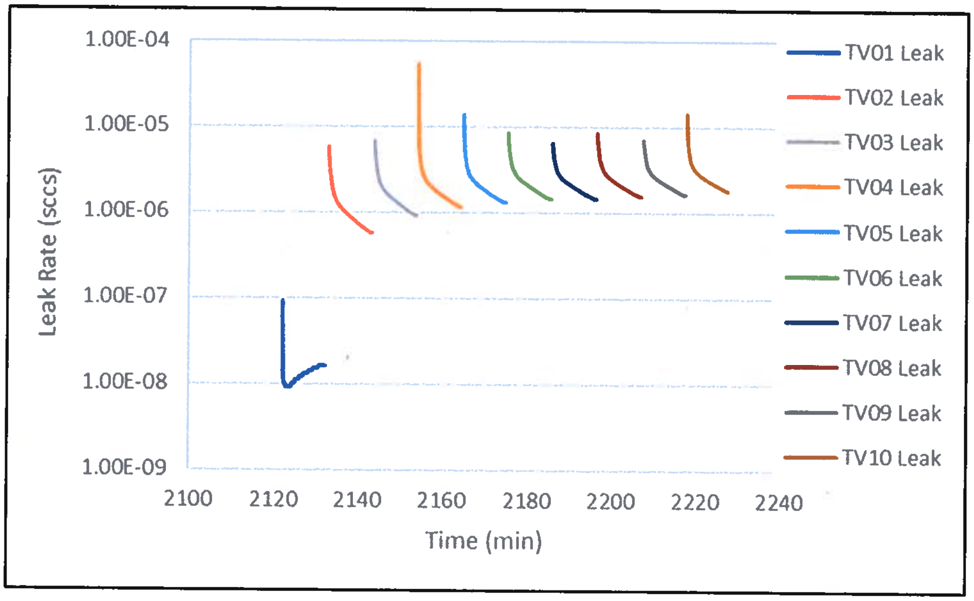

Figure 3-6. Inline leak detection of the 50,000th cycle results of the 5C valves, demonstrating the initial spike in leak rate followed by stabilization.

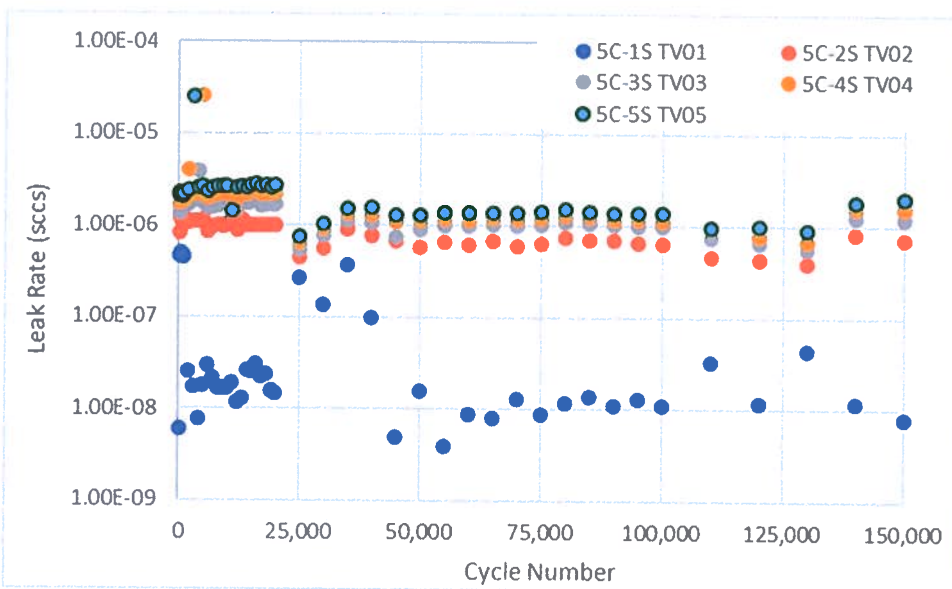

Figure 3-7. 5C actuated valves with Stellite™ stem tips.

Based on the allotted time for leak testing per valve, the baseline reading of the leak rate can be clearly seen at higher levels with the 5C Vespel® tipped valves in Figure 3-8. Although none of the leak tests result in any concern of failure, their leak rates were consistent enough to suggest that longer leak detection periods and/or longer periods between individual valve leak tests would aid in bringing the baseline leak rate to levels more in line with the quantitative MSLD. This claim would require verification for substantiation.

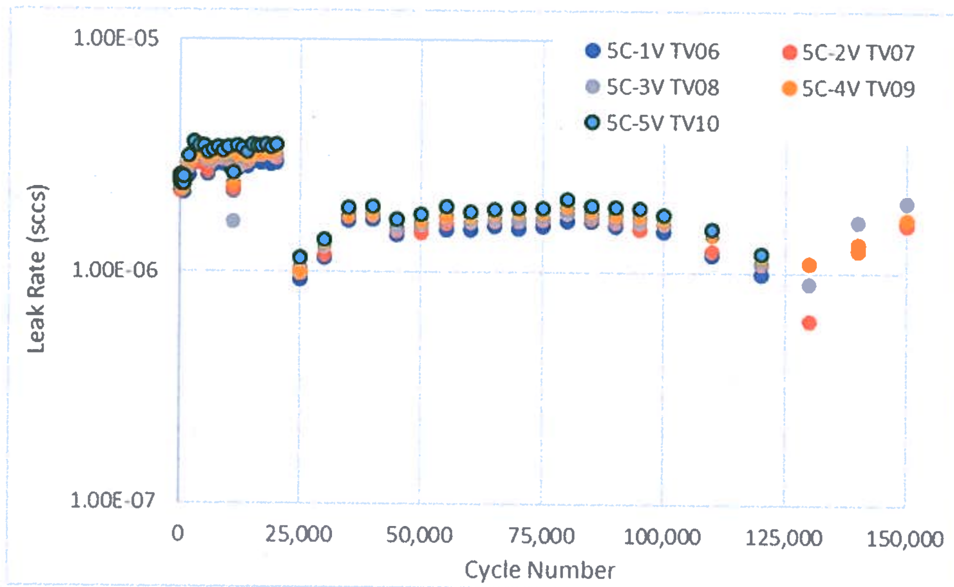

Figure 3-8. 5C actuated valves with Vespel® stem tips.

Performance-wise, the Stellite™ and Vespel® tipped 5C valves all performed as expected up to the 50,000 cycle milestone. The valves were further analyzed using quantitative leak testing to verify the results of the inline leak detector (Table 3-5). After confirmation of each valve's performance after quantitative leak testing, all ten 5C actuated valves were reinstalled on the manifold and cycled to the 100,000 cycle milestone, producing results of unchanged performance, as reported by the inline leak detector (Figure 3-7 and Figure 3-8).

The first incident of failure was observed after 120,000 cycle when two of the Vespel® tipped valves, 5C-1V and 5C-5V, had developed leaks across the body as indicated by attempts to perform inline leak testing. The leak rate through these failed valves was so high the manifold vacuum system could not achieve a low enough pressure to initiate leak testing using the inline MSLD. These two valves were removed from the manifold and the remaining valves cycled to 150,000 cycles without incident, as noted in Figure 3-8.

All ten 5C actuated valves were sent for quantitative leak testing after 150,000th cycles with the results summarized in Table 3-5. The results again supported the use of the inline leak testing as a screening mechanism for valve removal for qualitative leak testing; however, the body leak test results for valve 5C-5V was inconsistent with the inline leak test measurement indicating a failed valve. Another inconsistency was Table 3-5 showed valve 5C-3V as the failed valve instead of 5C-1V which was attributed to as a transcription error, along with the reported body leak value for 5C-5V.

Table 3-5. Quantitative leak test results for the 5C actuated valves at the 50,000 and 150,000 cycle milestones.

HPL Tests		50,000 Cycles			150,000 Cycles		
		Leak Rates (std cc He/sec)			Leak Rates (std cc He/sec)		
Valve Name	Stem Tip	Inlet Seat	Outlet Seat	Body	Inlet Seat	Outlet Seat	Body
5C-1S	Stellite	<1.1E-09	<1.1E-09	<1.3E-09	<2.1E-09	<2.1E-09	<1.8E-09
5C-2S	Stellite	<1.0E-09	<1.0E-09	<1.3E-09	<2.0E-09	<2.0E-09	<2.0E-09
5C-3S	Stellite	<1.1E-09	<1.1E-09	<1.3E-09	<1.6E-09	<1.6E-09	<1.7E-09
5C-4S	Stellite	<1.1E-09	<1.1E-09	<1.2E-09	5.4E-09	4.40E-09	<1.3E-09
5C-5S	Stellite	<3.8E-10	<3.8E-10	<1.2E-09	<1.3E-09	<1.3E-09	1.7E-08
5C-6S	Stellite	Not tested			Not tested		
5C-1V	Vespel	<8.4E-10	3.6E-09	4.0E-08	<2.2E-09	<2.2E-09	<1.9E-09
5C-2V	Vespel	<2.6E-09	<2.6E-09	6.9E-08	<1.5E-09	<1.5E-09	<2.7E-09
5C-3V	Vespel	<2.1E-09	<2.1E-09	<1.4E-09	1.60E-03	2.40E-09	7.3E-05
5C-4V	Vespel	<1.0E-09	9.6E-09	<1.1E-09	6.90E-09	8.5E-09	<1.2E-09
5C-5V	Vespel	<1.1E-09	2.2E-08	<1.5E-09	<1.5E-09	1.7E-03	<1.2E-09
5C-6V	Vespel	Not tested			Not tested		

Post-test failure analysis for valves 5C-3V and 5C-5V were performed to determine the source of the leaks. Using a WILD Heerbrugg M650 microscope, the internal components of the valves were inspected for defects. Upon examination, a crack was observed on the bellows of the 5C-3V valve (Figure 3-9) account for the body leak however, it does not support the inlet seat leak summarized in Table 3-5, based on the direction of the gas flow through the valve (Figure 3-10). When the valve is closed, the seal created should have prevented gas leaking into the valve through the crack during the inlet seat leak test. On the other hand, it could be speculated that with the damaged bellows, the outlet seat leak test would have failed.

Further inspection of valve 5C-3V components revealed a small nick in the seat of the valve (Figure 3-11) however, the stem tip itself (Figure 3-12) was free of any deformations consequential from the nick. Post-test failure analysis results for valve 5C-5V did not produce any evidence as to potential sources of leaking. The body leak test was repeated for valve 5C-5V and verified previous leak test results.

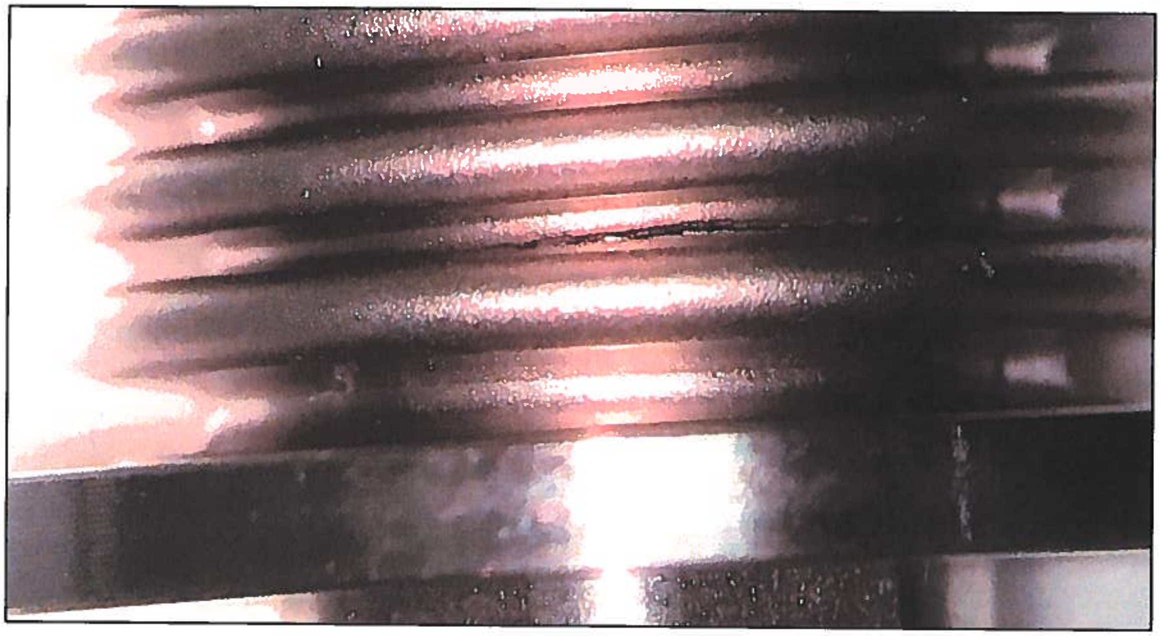

Figure 3-9. Observed crack in the bellows of the 5C-3V valve, magnified at 40x.

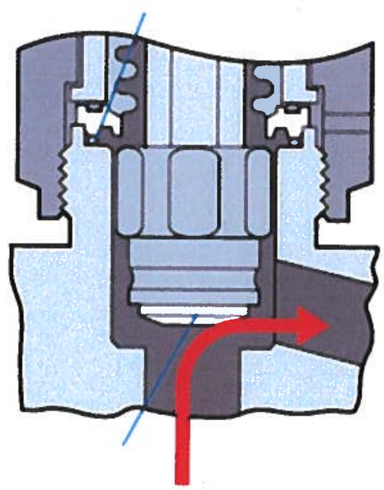

Figure 3-10. Direction of gas flow from inlet to outlet of the BG bellows valve.

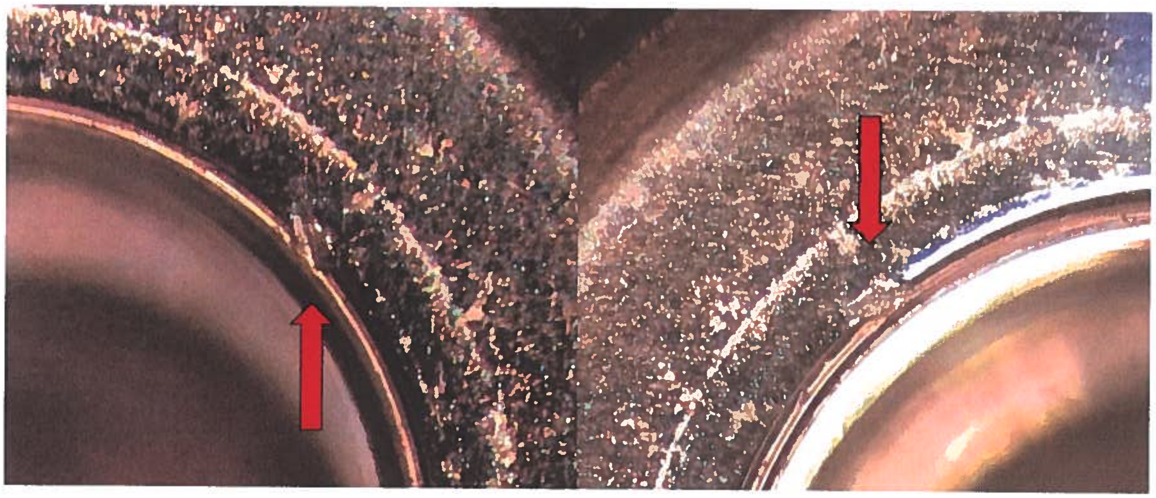

Figure 3-11. 40x magnification of the seat for valve 5C-3V, depicting a small nick, taken from different angles.

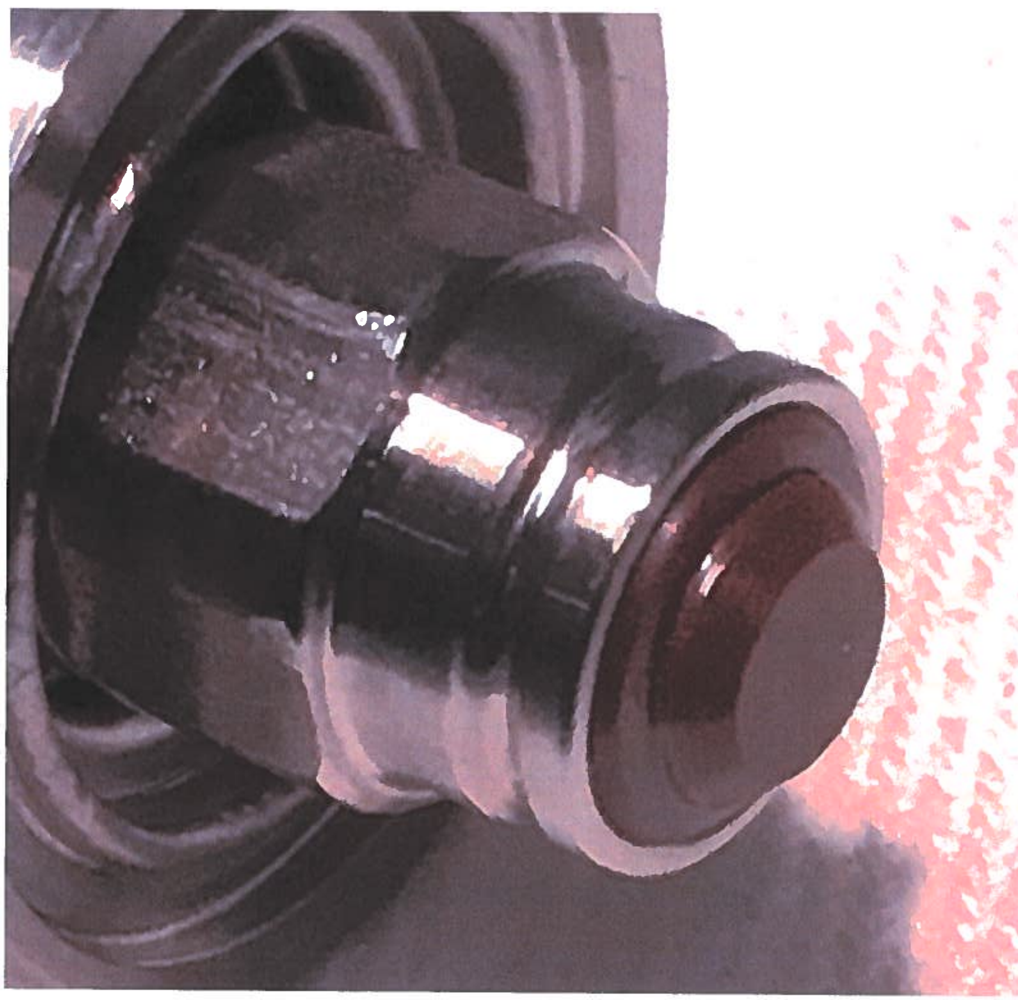

Figure 3-12. Vespel® stem tip of the 5C-3V valve.

4 Conclusions

The failure of the 1C actuated valves was believed to be attributed to improper torqueing during the valve assembly process which did not form the low leak rate seal using the silver plated gasket in the valve body. Successful leak test results for properly assembled valves is unlikely for Stellite™ tipped valves, but possible for Vespel® tipped valves.

All of the 3C actuated valves had acceptable initial leak rate results, but failure of several valves under 5,000 cycles presents a concern. It is uncertain if the valves failed due to the actuators or if the actuators failed due to the valves. Based on the success of the 5C actuator tests, it is speculated the problem is with the 3C actuator. Generation of metallic powder during cycling and an unknown fluid dripping from the actuator from two of the valves creates concerns about use of these valves in an inert glovebox and further discussions with the vendor should generate insight into these observations. For the valves with actuators that did not fail, the 3C actuator does significantly reduce the glovebox footprint needed for the same size (1/4 inch) valve.

Valve testing of the 5C actuated valves demonstrated this valve/actuator combination can easily maintain valve leak spec for a large number of cycles (>100,000 cycles) without failure. This conclusion holds true for either valve stem tip material: Stellite™ or Vespel® with the majority of the valves still passing the leak rate criteria after 150,000 cycles.

5 Recommendations, Path Forward or Future Work

Reworking the 1C Vespel® tipped valves is recommended to determine the cycle life of these valves. The reduction of glovebox footprint with the 1C actuators compared to the 3C actuators is significant and is worth further study. Applying a larger force to the Stellite™ valves than can be applied by the 1C actuator may form an initial sealing surface of the valve which would allow cycle testing with an all metal valve. In some applications, even a valve with a cycle life of several thousands may be of benefit and worth exploring.

Discussions and meetings with the vendor are needed to identify the reason for the presence of liquids and metallic fines generated when testing some of the 3C valves. These materials were present in more than one valve and the extent of this conditions needs examination.

Further study with larger size (1/2 inch) B series valves, as well as study of even larger (3/4 inch, 1 inch) U series valves should be performed to determine if valve cycle life is shortened with larger sealing surface valve. The use of electric actuators, if available for these bellows sealed valves, should also be investigated. Cycle testing with protium in a heated environment is also recommend for further understanding of valve long term performance.

Further modifications to the manifold and valve logic programming could reduce the need to remove valves for quantitative testing which would reduce costs and increase data throughput of such a system. Installation of a more sensitive MSLD, along with calibrated leaks, into the manifold design improve any newly designed/modified valve testing manifold. To complete evaluation of different valves, performance testing in a tritium environment is recommended before complete recommendations can be made towards the use of these valves in tritium service.

6 References

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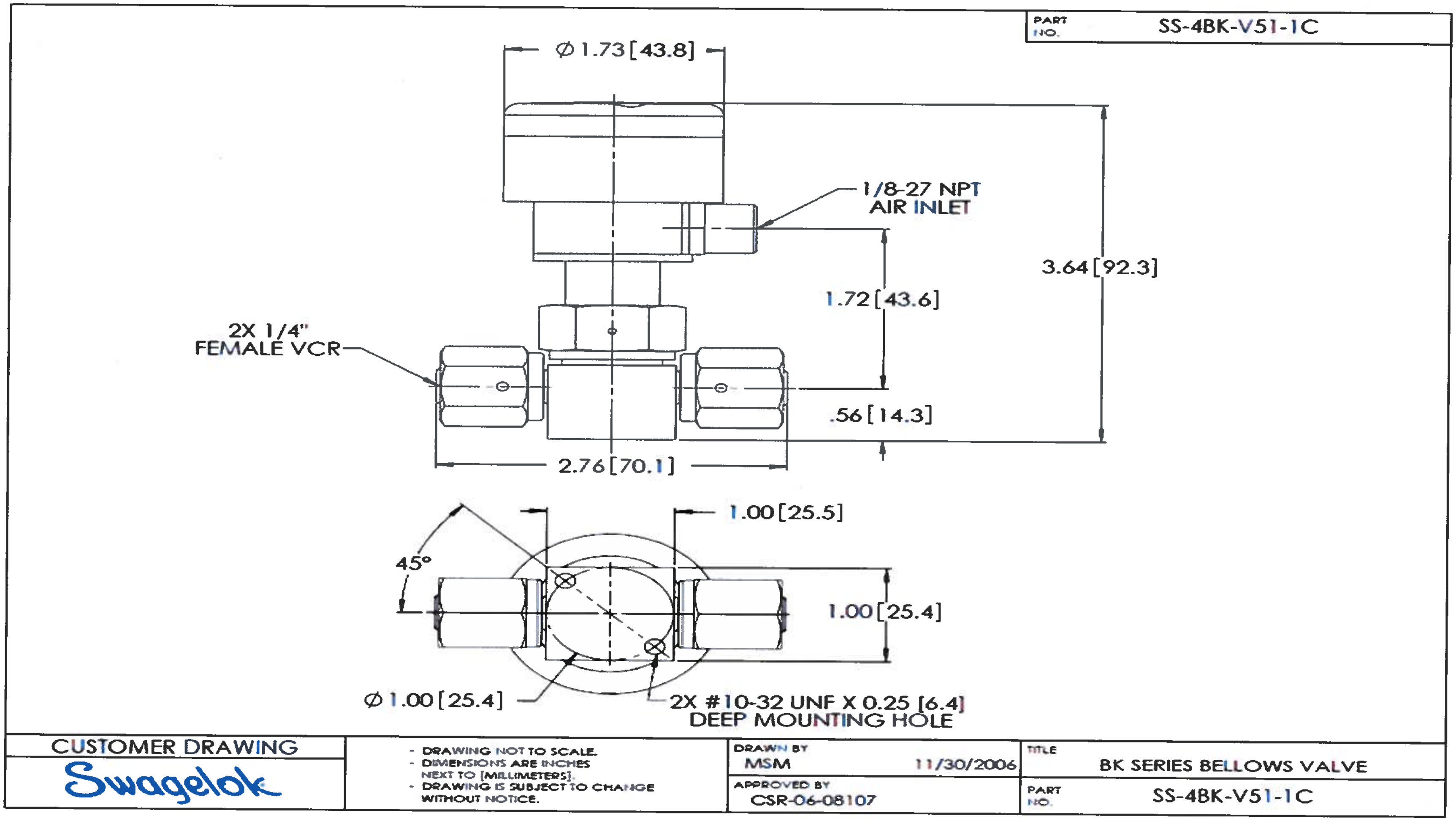
(2) Weaver, B., *Tritium Handling and Safe Storage*, **DOE-STD-1129-2015**, U.S. Department of Energy, Washington, D.C., **2015**.

(3) *"Bellows-Sealed Valves"*, MS-01-22, R13, Swagelok, 2017.

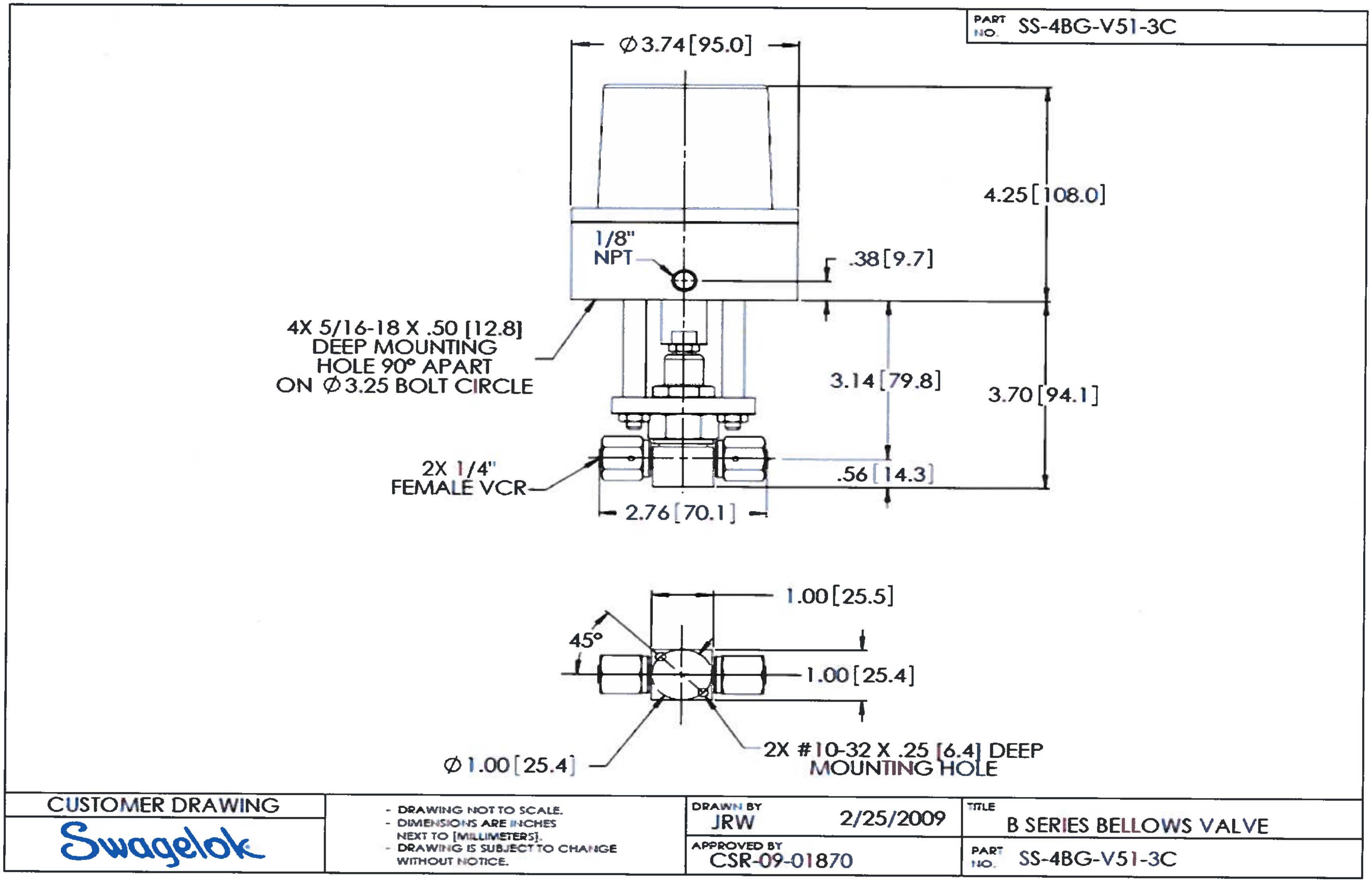
(4) Weaver, W.W., *Guidelines for Valves in Tritium Services*, Office of Nuclear Safety, Washington, D.C., **1994**.

(5) Houk, L.R.; Payton, A.N., *Evaluating All-Metal Valves for Use in a Tritium Environment*, **SRNL-STI-2017-00516, Rev 0**, Savannah River National Laboratory, Aiken, South Carolina, **2017**.

Appendix A Technical drawing of the Swagelok 1C BK Series Bellows Valve



Appendix B Technical drawing of the Swagelok 3C B Series Bellows Valve



Appendix C Technical drawing of the Swagelok 5C B Series Bellows Valve