A Minimum Thickness Gate Valve with Integrated Ion Optics for Mass Spectrometry

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A minimum thickness gate valve design for mass spectrometry is described in detail. The ion optics required to transmit ions from the source to the ICR cell are integrated into the design to minimize fringe field effects on the ions as they travel through the gate valve. The total thickness of the complete gate valve assembly is 1.03 in. (26.2 mm) with a maximum fringe field distance of 0.065 in. (1.7 mm). The gate valve is able to maintain a vacuum of $<10^{-10}$ mbar at the ICR cell when the source is vented to atmosphere and the estimated ion transfer efficiency is >95%. (J Am Soc Mass Spectrom 2005, 16, 441–445) © 2004 American Society for Mass Spectrometry

The installation of a gate valve between an external ion source and the high vacuum region of a mass spectrometer facilitates the cleaning and maintenance of the source while maintaining high vacuum conditions in the remainder of the instrument. Many gate valves are commercially available and a number of gate valve designs have been presented in the literature [1–7]. Many of these gate valves were not designed for mass spectrometers and, therefore, do not lend themselves to high ion transmission efficiencies. Marshall et al. have implemented a system where the source vacuum system is moved forward so that the ion optics are inserted through a commercial gate valve once it is opened. This design reduces the separation distance between the optical elements to either side of the gate valve, improving ion transfer efficiency [8,9]. This approach provides reproducible placement of all ion optics and simplicity of design, yet it increases the number of moving parts in the system which increases the possibility of mechanical failures.

A minimum thickness gate valve has been previously reported by Stolow [5], in which a combination linear/rotary motion feedthrough was used to move the blade into position and then to compress the o-ring to form a seal. This design did not allow for the blade of the gate valve to be electrically isolated and required the use of a combined motion feedthrough. A second minimum thickness gate valve design was presented by Teodoro and Moutinho, where a linear motion feedthrough was used to move a blade into and out of position [7]. In this design, an o-ring was affixed to the blade and moved across the opening machined into a conflat flange, introducing the possibility of increased wear on the o-ring. A feature unique to their gate valve design is the ability to differentially pump the high pressure side of the gate valve before the valve was opened to vacuum. Again, this design did not allow for the electrical isolation of the gate valve blade. Neither of the two minimum thickness designs discussed was used for mass spectrometry.

In this paper, we present a minimum thickness gate valve design that integrates ion transfer optics. Minimizing the thickness of the valve limits the fringe field region experienced by ions exiting an ion source and entering a long transmission hexapole to the ICR cell. A transmission efficiency of >95% has been routinely observed. The gate valve maintains a vacuum of $<10^{-10}$ mbar in the mass spectrometer when the source is open to atmosphere.

Gate Valve Design

A drawing of the assembled gate valve with integrated hexapole ion guides is shown in Figure 1a. The stainless-steel neck with a mini-conflat flange welded to the top of the assembly is the attachment point for a commercially available linear motion feedthrough. Figure 1b is a cross-sectional view of the assembled gate valve with the dimensions between the hexapole ion-guides and the gate valve blade noted. The hexapole ion-guides used in this instrument have an internal diameter of 0.375 in. (9.53 mm) and the distance separating the exit of the first hexapole from the entrance of the second hexapole is 0.205 in. (5.21 mm). The width of the gate valve blade is 0.1 in (2.54 mm) so that the total width of the fringe field region is reduced to 0.105 in. (2.67 mm). The gate valve blade is electrically isolated

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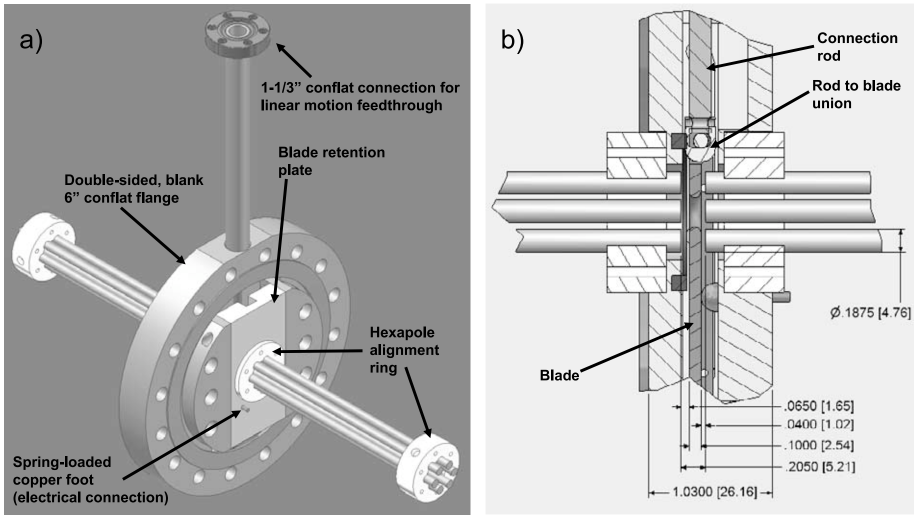

Figure 1. (**a**) The minimum thickness gate valve with integrated hexapole ion-guides. (**b**) A cross-sectional view of the gate valve showing the total dimensions and distances separating the two hexapole ion-guides (inches [mm]).

so that it can be used as an electrostatic ion lens to improve transfer efficiency from one ion-guide to the next. This distance represents a fraction of the hexapole internal diameter and prevents any significant decrease in the transfer efficiency of the ion-guides across the thickness of the gate valve.

Materials

A 6-in., double-sided, blank conflat flange (P/N 140025), mini-conflat flange (P/N 110003) and linear actuator (P/N 660004) were purchased from MDC Vacuum Products Corporation (Hayward, CA). The Buna-N, double seal o-ring (1.3125 in. o.d., 1.0625 in. i.d.) used to form the seal between the blade and the body of the gate valve was purchased from McMaster-Carr (P/N 90025K348; Dayton, NJ). Nylon rollers (0.1875 in. o.d., 0.25 in. long) used to guide the gate valve blade were purchased from Small Parts Inc. (RSN-04/02-25; Miami Lakes, FL). The hexapole alignment rings were machined from cylindrical Macor stock material. The center portion of the gate valve blade retaining plate was machined from Delrin. All other parts were machined from non-magnetic no. 304-stainless steel. While nylon and Delrin are not generally considered UHV compatible materials, the reader will note that these materials are used on the front region of the gate valve, which in the system used is an HV region with a base pressure of ~1e-6 mbar. If important, these can be substituted with Vespel, which also has higher temperature stability and will allow UHV bake-outs.

Blade Design

The blade of the gate valve is machined from a piece of type 304-stainless steel (Figure 2a). The two long sides and the top of the blade are 0.200 in. (5.08 mm) thick and the blade is tapped at the four corners to accept 0.25 in. (6.35 mm) long 2-56 machine screws. The remainder of the blade is 0.100 in. (2.54 mm) thick and allows for minimum hexapole to hexapole spacing. At the "top" of the blade, a notch is cut and a 0.130 in. (3.30 mm) clearance hole is drilled through the top of the blade to accept a 0.125 in. (3.18 mm) diameter alumina rod, which is used to connect the gate valve blade to the linear actuator. Alumina is used because of its strength and insulating properties. The blade of the gate valve is electrically isolated from the total gate valve assembly. This is accomplished by carefully insulating the blade from each of the connecting parts. The sides of the blade are prevented from contacting the stainless steel tracks by strips of Teflon which are held in place by the four 2-56 screws. These screws attach the nylon wheels that guide the blade along the track machined into the stainless steel plates that hold the assembly together (Figure 2b).

A 0.250 in. (6.35 mm) diameter hole is machined into the blade to allow transmission of ions through the gate valve. The diameter of this orifice can be chosen to act as a conduction limit or to match the inner diameter of the multipole ion-guides placed to either side of the gate valve. The opening in the gate valve blade is reproducibly positioned in the center of the hexapole ion-guides by a series of tracks machined into the blade retention plate (Figure 2b).

Blade Retention Plate

The blade of the gate valve and the ion optics on the high-pressure side of the gate valve are attached to the retention plate, which is machined in three parts (Figure 1a). The center part of the assembly is machined from Delrin and has a recess cut in the center to align

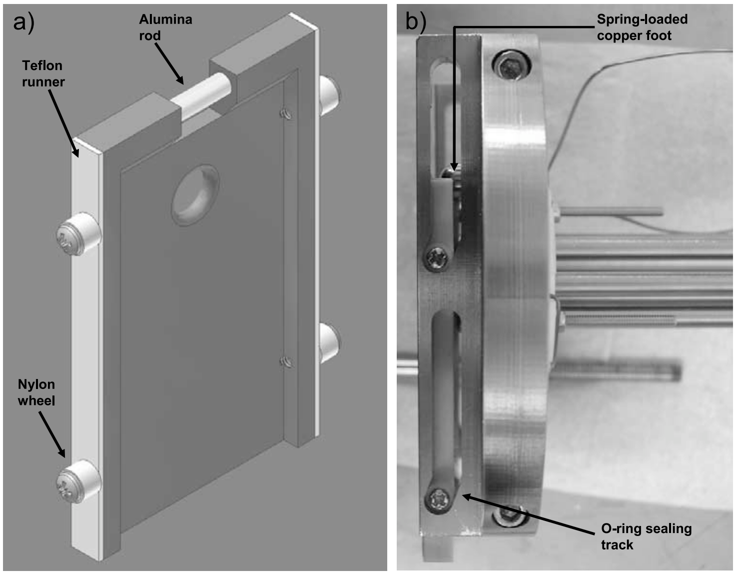

Figure 2. (**a**) The gate valve blade is shown with insulating nylon wheels and Teflon runners. The opening in the gate valve blade is 0.250 in. (6.35 mm) in diameter. (**b**) A photograph of the assembled retention plate and blade showing the placement of the blade wheels in the tracks machined into the stainless steel plates. The spring-loaded copper foot can also be seen through the left-hand track.

the ceramic hexapole alignment ring. An electrical connection to the gate valve blade is made by a spring-loaded copper foot that is housed inside the Delrin plate. This foot maintains contact with the blade while it is moved between the open or closed positions, allowing for a DC voltage to be applied to the blade. DC voltages up to ±250 V have been used without electrostatic breakdown.

The remaining two pieces of the retention plate assembly are machined from type 304-stainless steel and must be machined to tolerances of ±0.001 in. (0.03 mm) in order to guarantee proper alignment of the blade and to form a good vacuum seal (Figure 2b). These two pieces allow reliable positioning of the gate valve blade assembly relative to the opening that is machined in the body of the conflat flange. The blade is reproducibly placed in the open or closed position by a pair of tracks that are cut into the stainless steel plates. The track system not only reproducibly positions the blade, but it also controls the pressure that is applied to the o-ring to form the vacuum-tight seal. Pressure on the o-ring is released prior to the blade moving into the open position in order to minimize wear on the o-ring, which had been noted in previous gate valve designs.

Ion-Guide Alignment

The main body of the gate valve is machined from a standard, blank, 6 in. (152 mm) double-sided conflat flange. The main transmission hexapole leading to the ICR cell is aligned with the orifice of the gate valve by inserting the ceramic hexapole mounting ring into a 1.36 in. (34.5 mm) diameter by 0.150 in. (3.81 mm) deep recess machined into the ultra-high vacuum (UHV) side of the conflat flange. A corresponding 0.40 in. (10.2 mm) deep recess is machined into a piece of Delrin on the opposite side of the gate valve assembly. A ceramic hexapole mounting ring for a short transmission hexapole is mounted directly to the face of the gate valve ensuring that the ion-guides are both perfectly aligned with the gate valve orifice and that the gate valve is aligned properly with the ion source.

Experimental

Materials

Kwik-Fil borosilicate glass capillaries, HPLC-grade methanol and formic acid were obtained from VWR International (Bristol, CT). Bovine ubiquitin was purchased from Sigma-Aldrich (St. Louis, MO). All water was filtered using a Milli-Q Gradient A10 filtration system from Millipore (Billerica, MA).

Methods

A solution of 1 pmol/μL bovine ubiquitin was made in 1:1 (vol/vol) methanol to water, 1% formic acid solution and was sprayed using a home-built nanospray source. All nanospray tips were made from Kwik-Fil borosilicate glass capillaries and pulled in-house using a Sutter Instrument Company (Novato, CA) Model P-97 Flaming/Brown micropipette puller. The orifice of each nanospray tip was ~1 μm in diameter. All experiments were conducted on an electrospray ionization (ESI) Fourier-transform mass spectrometer (FTMS) built in collaboration with MDS Sciex (Toronto, ON, Canada). The design

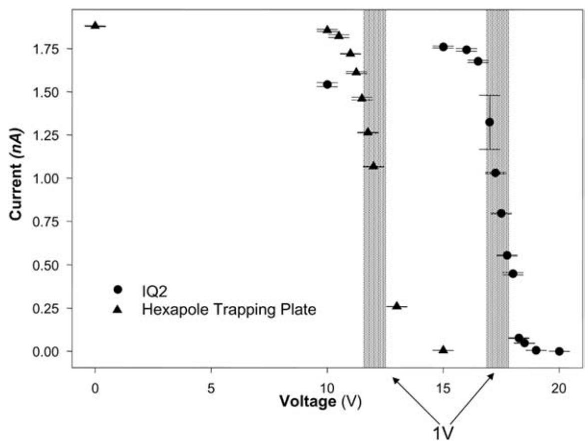

Figure 3. Plot of total ion current versus applied DC potential at IQ2 and the hexapole trapping plate. Error bars are ±one standard deviation (N = 3).

and operation of this instrument are discussed in detail elsewhere [10].

A retarding grid study was performed in order to determine the effect of the gate valve and the integrated ion optics on the kinetic energy distribution of ions being transferred to the ICR cell. A DC offset potential of 0–20 V was applied to the orifice at the entrance to the last quadrupole of the ESI source (IQ2), and to the hexapole trapping plate at the entrance to the ICR cell. Total transmitted ion current was measured on a grid at the end of the ICR cell opposite the long transmission hexapole, using a Keithley 485 Autoranging Picoammeter (Keithley Instruments Co.; Cleveland, OH). A total of three current measurements were taken at each applied DC offset, and the average and error bars (one σ) are plotted in Figure 3.

Results and Discussion

Figure 3 is a plot of the total transmitted ion current versus the DC offset applied to either IQ2 or the hexapole trapping plate which acts as a retarding grid for ion current before and after the gate valve respectively. A total kinetic energy distribution of approximately 1.5 ± 0.1 eV is observed both before (IQ2) and after (hexapole trapping plate) the gate valve, indicating that the gate valve has minimal impact on the distribution of the kinetic energy of the ions reaching the ICR cell. In order to determine the width of the kinetic energy distribution, the derivative of the raw current data was calculated and plotted in a spreadsheet and the FWHM was extracted from the plot.

The maximum observed current in Figure 3 is 1.8 nA, indicating efficient transfer of ions from the source to the ICR cell; however, the total anticipated current for a 1 pmol/μL solution of bovine ubiquitin is approximately 250 pA, based on a flow rate of 30 nL/min and an average 10+ charge state. Clearly, not all of the current is derived from ubiquitin ions. While all electrospray ion sources generate some solvent clusters, commonly known as "chemical noise", the lack of a desolvation capillary in the current design increases the abundance of these complexes. This discrepancy between the current that is measured at the ICR cell and the current that should be possible from the ESI source is attributed to these clusters. This same effect has been noticed before with the ion funnel measurements [11].

In order to characterize ion transfer efficiency across the gate valve, it was important to record the total ion current on the hexapoles both before and after the gate valve. In these current measurement experiments, one set of hexapole rods was positively biased by 5 V in order to force the ions into the other rod and improve collection efficiency. The other set of rods was attached to a picoammeter. Typical values are 1.75 nA for Hex 1 (before the valve) and 1.8 nA for Hex 2 (after the valve). Note that the total ion current observed on Hex 2 was higher by ~3% than that measured on Hex 1. While it would appear to be impossible for the current after the gate valve to be greater than the current before the gate valve, it is important to note that this method of measuring the current is not ideal for determining exact transfer efficiency, but represents the best method for estimating transfer efficiency without venting the instrument to install additional collector plates. It was impossible to use the blade of the gate valve itself as a collector due to low level capacitive coupling of the RF (measured on an oscilloscope to be ~5 mV_{rms}) from Hex 1 to the blade which was sufficient to saturate the picoammeter. The apparent 103% transfer efficiency, therefore, is an anomaly caused by the short ~6 in. long hexapole (Hex 1), in which some ions are not collected on the rods. Thus, although the total ion transfer efficiency clearly approaches 100%, the accuracy of the measurements does not allow this assertion to be made as there is a 3% error in the measurement. However, estimating the gate valve transmission efficiency at >95% under these conditions is reasonable.

Conclusions

We have shown that a minimum thickness gate valve can be machined into a commercially available vacuum component and has high transmission efficiencies (>95%). This gate valve design has been observed to hold a vacuum pressure of $<2.0 \times 10^{-10}$ mbar while the source was vented to atmosphere. We have also shown that the installation of this gate valve has no apparent impact on the kinetic energy distribution of the ions transmitted from the source to the ICR cell for detection. This design can be easily scaled to fit conflat flanges larger that the 6-in. flange presented here and can be installed into any vacuum system with a minimal redesign of the transfer ion optics.

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Supplemental Information

Complete drawings of this gate valve design will be available on-line at http://www.bumc.bu.edu/ftms or by contacting the authors at poconnor@bu.edu.

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