RESEARCH ARTICLE

Characterization of the Ion Beam Focusing in a Mass Spectrometer Using an IonCCD™ Detector

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Abstract

A position sensitive pixel-based detector array, referred to as the IonCCD, has been employed to characterize the ion optics and ion beam focusing in a custom built mass spectrometer designed for soft and reactive landing of mass-selected ions onto surfaces. The IonCCD was placed at several stages along the path of the ion beam to determine the focusing capabilities of the various ion optics, which include an electrodynamic ion funnel, two radiofrequency (rf)-only collision quadrupoles, a mass resolving quadrupole, a quadrupole bender, and two einzel lens assemblies. The focusing capabilities of the rf-only collision quadrupoles and einzel lenses are demonstrated by large decreases in the diameter of the ion beam. In contrast, the mass resolving quadrupole is shown to significantly defocus the mass-selected ion beam resulting in an expansion of the measured ion beam diameter. Combined with SIMION simulations, we demonstrate that the IonCCD can identify minor errors in the alignment of charged-particle optics that result in erratic trajectories and significant deflections of the ion beam. This information may be used to facilitate the design, assembly, and maintenance of custom-built mass spectrometry instrumentation.

Key words: IonCCD, Pixel-based detector, Position-sensitive detection, Ion optics, Ion focusing, Soft landing

Introduction

The design, assembly, maintenance, and utilization of custom-built mass spectrometry instrumentation is dependent on the ability to route an intense, highly focused beam of ions through a desired trajectory using both electrostatic and electrodynamic potentials applied to charged-particle optics [1]. Over the years, a variety of tools and techniques have been developed to determine the optimum geometry and placement of charged-particle optics and mass spectrometer components in a vacuum chamber to obtain maximum ion transmission. For instance, the commercially available SIMION software program may be used to test and validate entire mass-spectrometer designs as well as determine appropriate

potentials prior to any actual construction. However, with many common ionization sources, ions are typically transferred from ambient pressure through many stages of differential pumping where the pressures may range from several Torr all the way to 10^{-6} to 10^{-10} Torr. While SIMION may be employed routinely to simulate ion trajectories at low pressures where there is an absence of collisions, simulations at higher-pressures are slow and tedious, rely on coarse assumptions related to energy transfer in collisions, and do not accurately account for high-pressure gas flow. Moreover, SIMION simulations assume a perfect alignment of the ion optics which is, of course, impossible to accomplish in reality.

Due to the reasons detailed above, once the assembly process starts, the experimentalist generally employs a couple of standard laboratory techniques. A fairly common practice for aligning ion optics employing straight through geometries is to direct the output of a low-powered laser,

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which produces highly collimated light, into the entrance of the vacuum chamber (usually through a skimmer or conductance limit) and through to the detector or final desired destination of the ion beam. Once this line-of-sight is established, the remaining components, which are typically supported on adjustable mounts, may be inserted into the vacuum chamber such that they do not obstruct the transmission of the laser beam through the instrument. A typical method for determining the appropriate potentials to apply to each ion optic is to attach an electrometer or picoammeter to an ion optic of interest and to optimize the total ion current collected on that optic by varying the voltages applied to the preceding components. While these techniques are effective for routing a portion of the ions from the ionization source to the final destination, they do not provide insight into how the various charged-particle optics in a custom-built mass spectrometer shape, steer, and focus the ion beam. Moreover, other than visually inspecting components for ion deposition (burn) following a long period of operation, it is often impossible to identify minor alignment errors, which may result in the ion beam exhibiting non-ideal trajectories. Consequently, the ability to visualize the ion beam at various stages in a mass spectrometer during assembly and maintenance procedures offers a significant advantage to the experimentalist.

Over the years, a variety of different approaches have been developed for the imaging of ion beams in vacuum. In particular, the need to detect relativistic charged particles in high-energy physics experiments lead to the development of the monolithic active pixel sensor (MAPS) [2] and the complementary metal oxide semiconductor (CMOS) active pixel sensor [3]. Detectors capable of imaging ion beams, better known as position sensitive detectors (PSDs), have also been used in scan-free sector field type mass spectrometry instruments. Examples include photographic plates [4], variable position collectors attached to electrometers [5], the electro-optical ion detector (EOID) [6], the δ -doped charge-coupled device (CCD) [7], the faraday-strip array detector [8], and the channel electron multiplier array [9]. PSDs based on microchannel plates (MCPs) [10, 11] and MCPs combined with phosphor screens (electronic imaging detector, EID) [12, 13] have also found widespread application. Indeed, beam imaging systems based on this technology are currently available from commercial suppliers [14–16]. However, a principal disadvantage of the use of phosphor screens is their relatively low sensitivity, which necessitates the use of MCPs to convert, with a large gain, the incoming ion beam to electrons through secondary emission. The high voltages that must be applied to the plates of the MCP (1–3 kV), in turn, require that the device be operated at vacuum conditions of around 10^{-6} Torr. This complicates the use of these devices and prevents their application at pressures where collisions between ions and gas molecules may influence the trajectories and focusing of ions beams. Furthermore,

their use is typically limited to the profiling of ion beams that are in a line of sight of viewport flanges. The cumbersome electrical feedthroughs associated with these devices also reduce their utility in instrument design and diagnostic applications.

Herein we describe the use of a position-sensitive pixel-based detector array (IonCCD) for direct charged-particle detection to characterize the ion optics and ion beam focusing in a custom built mass spectrometer designed for mass-selected ion deposition. The capabilities of various components, including an electrodynamic ion funnel, rf-only collision quadrupoles, a mass resolving quadrupole, a quadrupole bender, and einzel lens assemblies to steer and shape the ion beam are examined. It is shown that the collision quadrupoles and einzel lenses can produce tightly collimated and focused ion beams, respectively, with narrow diameters, while the mass-resolving quadrupole produces a wide diameter, unfocused beam of mass-selected ions. Combined with SIMION simulations, we demonstrate that the IonCCD can identify minor errors in the alignment of charged-particle optics that result in erratic trajectories and significant deflections of the ion beam. Moreover, because this detector may operate at pressures from ambient to ultra-high vacuum, we were able to characterize each of the ion optics through all of the various stages of pressures and differential pumping in a mass spectrometer. In addition, because of its compact size and shape, we had no difficulty installing and operating the detector within the limited spatial confines of the vacuum chamber.

Experimental

Mass Spectrometer

Characterization of the ion beam focusing was performed within a recently constructed custom-built ion deposition instrument described in detail elsewhere [17] and shown schematically in Figure 1. Briefly, doubly charged ruthenium *tris*(bipyridine) cations $\text{Ru}(\text{bpy})_3^{2+}$ were generated by electrospray ionization (ESI) at ambient pressure. An electrodynamic ion funnel [18] was used to transfer the ions into vacuum, a collision quadrupole was employed to axially compress the ion beam, and a mass-resolving quadrupole was utilized to select one ionic species from the full distribution of ions generated by ESI. The tri-filter mass resolving quadrupole (Extrel CMS, Pittsburgh, PA) has 19 mm diameter rods and is powered by a 300 W, 880 kHz, QC-150 power supply. An einzel lens was used to focus the mass-selected ion beam into a collision quadrupole (10^{-3} Torr Ar), which served to axially compress the ion beam a second time. A quadrupole bender was employed to turn the ion beam 90° and a series of two einzel lenses was utilized to focus the beam on a deposition surface. The doubly charged $\text{Ru}(\text{bpy})_3^{2+}$ cations result from dissolution and dissociation of *tris*(2,2'-bipyridyl)dichlororuthenium(II)

Figure 1. Schematic illustration of the ion deposition instrument with points of ion beam profiling marked in red numbers. Pressures: ion funnel region (~ 1 Torr), collision quadrupole region (1×10^{-1} Torr), mass-selection and focusing region (2×10^{-4} Torr), deposition region (1×10^{-6} Torr)

hexahydrate in methanol solution and were selected for the ion beam profiling studies due to their relatively high electrospray ion currents of 500–800 pA measured at the deposition surface. The IonCCD detector was mounted inside the vacuum chamber directly behind the components marked with a red number in Figure 1. Therefore, the ion beam was profiled directly following: (1) the 1.5 mm internal diameter conductance limit, which constitutes the final plate of the electrodynamic ion funnel; (2) the 2.0 mm internal diameter conductance limit following the first collision quadrupole; (3) the mass resolving quadrupole, which has an inscribed diameter of 16.6 mm; (4) the 14 mm internal diameter einzel lens following the mass-selecting quadrupole; (5) the 2 mm internal diameter conductance limit following the second collision quadrupole; (6) the quadrupole bender; and (7) the two 14 mm internal diameter einzel lenses following the quadrupole bender.

IonCCD

The IonCCD used for characterization of the ion beam focusing was purchased from OI Analytical (Pelham, AL). This device, which is described in detail elsewhere [19], consists of a pixel-based detector array (24 μm pitch) that incorporates a modified light-sensitive charge-coupled device (CCD) that was engineered for direct charged-particle detection by replacing the photosensitive part of the CCD pixel with a metal-oxide-semiconductor (MOS) capacitor for ion detection. The detection efficiency was measured to be about 100 ions per IonCCD count (or digital number, dN) per pixel with a noise floor of about nine counts at sub-second integration time and linear dynamic range over 10^3 . In terms of ion beam currents, the IonCCD has a limit of detection (LOD) of 5 fA/pixel/100 ms. At high beam currents (nA) the IonCCD can run at very short integration times (down to 83 μs) to avoid signal saturation. The detector was mounted in the vacuum chamber using custom-built aluminum brackets that were screwed into the floor of the chamber. All of the ion beam profiles presented in this article were obtained with the detector

housing grounded to the vacuum chamber. Though not applied in this work, the IonCCD can be floated at ± 3 kV, which covers most instrument bias values, hence achieving true mesh-free profiling. The detector signal was transferred through the chamber wall using a custom NW40 cable feedthrough purchased from OI Analytical. Data acquisition was performed using the IonCCD software.

SIMION Simulations

Ion trajectories through the einzel lens and quadrupole bender were simulated using the commercial software package SIMION 8.0 (Scientific Instrument Services, Inc. Ringoes, NJ, USA). A single ion mass of 570 with a charge of 2 was selected to simulate the $\text{Ru}(\text{bpy})_3^{2+}$ $m/z=285$ ions used experimentally. The ions were given a filled cone direction distribution with a 0.5 degree half angle and a Gaussian distribution of kinetic energies centered at 1 eV.

Materials

Tris(2,2'-bipyridyl)dichlororuthenium(II) hexahydrate was purchased from Sigma-Aldrich. The powder was dissolved in methanol (Sigma-Aldrich) to create a stock solution with a concentration of 1×10^{-3} M. The stock solution was diluted by a factor of either 10 or 100 in methanol to obtain optimum electrospray ion currents.

Results and Discussion

Figure 1 presents a schematic illustration of the custom-built ion deposition instrument that was characterized using the IonCCD detector. The detector was mounted directly behind the components marked with a red number. In this fashion, a one-dimensional ion beam profile was obtained following each of the charged particle optics in the instrument. Figure 2a presents the profile of the ion beam that was obtained directly after the 1.5 mm internal diameter conductance limit which constitutes the final plate of the

Figure 2. Ion beam profile obtained directly after (a) the electrodynamic ion funnel (integration time=100 ms) and (b) the first conductance limit (integration time=25 ms). The colors correspond to different potentials applied to the CL.

electrodynamic ion funnel. The beam profile has a full width at half maximum (FWHM) of 1.7 mm, which is slightly larger (+0.2 mm) than the diameter of the conductance limit (1.5 mm). This may be explained as the result of some minor broadening of the beam diameter in the short (~4 mm) field free region between the final plate of the ion funnel (potential ~ +30 V) and the detector surface. The shape and diameter of the ion beam were found to be almost completely independent of the potential applied to the rear plate of the ion funnel. The top of the profile exhibits a “hairy” looking jagged shape, which is attributed to the impact of charged droplets on the detector surface. Indeed, when the flow rate of the syringe pump was increased, this jagged structure became much more pronounced, which is consistent with the production of a larger number of charged droplets at the ESI source. In a similar fashion, reduction of the flow rate decreased this jagged structure in the ion beam profile but also resulted in an undesirable decrease in total ion current. The ion beam profile presented in Figure 2a demonstrates that the IonCCD may be operated at relatively high pressures of around 1 Torr and provides information that would not be obtainable using the MCP phosphor screen imaging devices described in the introduction.

The ion beam profile obtained directly after the 2.0 mm internal diameter conductance limit (CL) following the first RF only collision quadrupole is presented in

Figure 2b. In this case, the profile was measured with a potential of +5, +10, +20, and +34 V applied to the conductance limit to examine the influence of the extraction field in the collision quadrupole on the shape of the ion beam. The potential on the conductance limit that gave optimum ion transmission was determined to be +34 V which closely matches the direct current (DC) pole bias (+33 V) applied to the rods of the collision quadrupole. At this potential the IonCCD measured the ion profile shown in blue which has a FWHM of 1.4 mm that is slightly smaller (−0.6 mm) than the internal diameter of the conductance limit (2.0 mm). Through the use of the Ion CCD, therefore, we demonstrate that the collision quadrupole has strong axial focusing capabilities and is capable of compressing the ion beam to a diameter smaller than that of the conductance limit, thereby ensuring efficient transfer of ions from one region of differential pumping to another. Furthermore, as the voltage on the conductance limit is lowered and the potential difference between the collision quadrupole and the conductance limit increases, the ion beam defocuses as can be seen clearly by the increase in the width of the beam profile.

Figure 3a presents the ion beam profile obtained following the mass resolving quadrupole (RQ). This quadrupole has a significantly larger inscribed diameter (~16.6 mm) than a standard quadrupole with 9.5 mm diameter rods. The larger inscribed diameter provides a wider acceptance angle for ions exiting the first collision quadrupole but also results in significant broadening of the mass-selected ion beam at the exit of the resolving quadrupole. The mass-resolving quadrupole was set to provide a stability region centered at $m/z=285$ that enabled the best transmission of mass-selected $\text{Ru}(\text{bpy})_3^{2+}$ ions. Maximum transmission of mass-selected $\text{Ru}(\text{bpy})_3^{2+}$ $m/z=285$ was obtained with a DC bias of around +20 V applied to the quadrupole rods and +35 V on the quadrupole exit lens. These potentials produced the ion beam profile shown in red, which has a FWHM of 2.8 mm. Reduction of the exit lens potential to 0 V resulted in a significant decrease in overall ion transmission through the quadrupole as shown by the black line in Figure 3a. In addition, by adjusting the amplitude of the rf pre/post filters, it was possible to almost completely suppress the ion transmission through the quadrupole and to split the beam profile from one peak into two peaks as shown by the blue and purple curves in Figure 3a, respectively. These results indicate that non-Gaussian ion beam profiles may be produced through improper selection of pre/post filter amplitudes in mass resolving quadrupoles. This is consistent with previous investigations, which found that fringing fields present at the exit of a quadrupole ion guide result in ion beam divergence that may be reduced by lowering the strength of the rf field at the exit [20]. Indeed, it has been shown that when quadrupole rods are placed in close proximity to a metal conductance limit, the rf field at the quadrupole exit is rapidly quenched, which results in a radial excitation of the ions. Using the IonCCD it is possible to easily visualize these effects. Moreover, the beam

Figure 3. Ion beam profiles obtained directly after (a) the resolving quadrupole (integration time=50 ms, the colors correspond to different potentials applied to the quadrupole exit lens, the quadrupole pole bias, and rf pre- and post-filters) and (b) after the einzel lens following the resolving quadrupole (integration time=50 ms, the colors correspond to different potentials applied to the center element of the einzel lens)

profiles in Figure 3a indicate that for efficient transmission of ions through the next conductance limit (3 mm internal diameter) into the second collision quadrupole, it is necessary to refocus the beam.

Figure 3b presents the ion beam profiles obtained following the einzel lens mounted at the exit of the mass-resolving quadrupole. By adjusting the potential applied to the central element of the einzel lens one can observe, in real time, the focusing capability of this common charged-particle optic. A potential of +20 V applied to the central element with -50 V applied to the entrance and exit optics of the einzel lens was found to give the best ion transmission and the ion beam profile shown in red with a FWHM of 1.8 mm. Potentials lower and higher than +20 V resulted in defocusing of the ion beam at the IonCCD detector as shown by the remaining curves in Figure 3b.

Figure 4a presents the ion beam profiles that were obtained directly after the 2 mm internal diameter conductance limit (CL2) following the second collision quadrupole (CQ). Of the various settings applied to the conductance limit, it was found that a potential of -300 V yielded the best ion transmission. Such a large negative potential is required

Figure 4. Ion beam profiles obtained directly after (a) the second conductance limit (integration time=50 ms, the colors correspond to different potentials applied to the CL) and (b) after the exit lens of the quadrupole bender (integration time=50 ms, the colors correspond to different potentials applied to the center element of the einzel lens before the quadrupole bender)

to extract the ions from the high-pressure region of the second collision quadrupole which was DC biased at around +15 V. At a CL potential of -300 V the beam profile exhibited the peak shown in red with a FWHM of 1.7 mm, which is slightly smaller (-0.3 mm) than the internal diameter of the conductance limit (2.0 mm). Higher potentials applied to CL2 resulted in weak, unfocused beam profiles. Therefore, as was the case for the first collision quadrupole, the second collision quadrupole also efficiently compresses the ion beam to a smaller diameter than the conductance limit, thereby ensuring efficient transfer of ions from one region of differential pumping to another. In addition, it should be noted that the second collision quadrupole takes a relatively defocused ion beam (2.8 mm) from the mass resolving quadrupole and converts it into the tightly focused (1.7 mm), highly collimated beam that is necessary to achieve an efficient 90° deflection at the following quadrupole bender.

The ion beam profiles obtained after the exit lens of the quadrupole bender are provided in Figure 4b. After optimizing the potentials applied to the rods and lenses of the quadrupole bender to achieve maximum ion trans-

mission, it was found that the potential applied to the central element of the einzel lens (L22) before the quadrupole bender exhibits a strong steering effect on the ion beam. One can see that as this potential is lowered from 0 V to -50 V the ion beam is steered from the left side of the IonCCD to the right side. A potential of -20 V provides the best ion transmission and the tightly focused ion beam shown in black with a FWHM of 0.5 mm. To better understand this steering effect of the einzel lens, ion trajectories through the einzel lens and quadrupole bender were simulated using SIMION 8.0. Figure 5 shows the ion trajectories for $\text{Ru}(\text{bpy})_3^{2+}$ ions with potentials of 0, -20 , and -50 V applied to the central element of the einzel lens. In both the experiment and simulation the entrance and exit optics of the einzel lens were biased at -100 V. The simulations reveal that this steering effect is the result of a slight misalignment of the position of the einzel lens and quadrupole bender with respect to the ions exiting the second collision quadrupole. This SIMION result was confirmed by careful visual inspection of the positioning of the charged-particle optics in the vacuum chamber. The misalignment results in the ions entering the einzel lens slightly off-center. Consequently, at the potentials of 0 and -20 V the einzel lens steers the ion beam across the entrance of the quadrupole

bender which, in turn, results in the ion beam moving from left to right across the exit of the bender, as observed experimentally by the IonCCD detector. These findings, made possible by the unique capabilities of the IonCCD detector, demonstrate that small errors in the alignment of charged-particle optics with respect to each other may result in an ion beam exhibiting non-ideal behavior that would be very difficult to identify through conventional alignment and ion focusing techniques.

The final ion beam profile, obtained at the location of the deposition substrate in the soft-landing instrument, is presented in Figure 6a. This profile demonstrates the focusing capabilities of the two einzel lenses that follow the quadrupole bender. At the potentials that provide the best ion transmission the beam profile has a FWHM of 0.3 mm. Our findings, therefore, show how an intense beam of ions may be generated by electrospray ionization, transferred into vacuum using an electrodynamic ion funnel, axially focused by a collision quadrupole, mass-selected, collimated again by a second collision quadrupole, turned 90° by a quadrupole bender, and tightly focused onto a deposition surface by a series of einzel lenses. The collimating and focusing effect of the collision quadrupoles and einzel lenses as well as the defocusing effect of the resolving quadrupole can be seen

Figure 5. SIMION simulation of ion trajectories for $\text{Ru}(\text{bpy})_3^{2+}$ exiting the second collision quadrupole with a potential of (a) 0 V, (b) -20 V, and (c) -50 V applied to the central element of the einzel lens. Note the migration of the beam position exiting the quadrupole bender as a function of the potential applied to the einzel lens

Figure 6. (a) Ion beam profile obtained at the deposition platform (integration time = 50 ms) and (b) plot of the ion beam diameters (FWHM) at various stages in the deposition instrument. Note the focusing effect of the collision quadrupoles and einzel lenses and the defocusing that occurs in the resolving quadrupole

clearly in Figure 6b, which presents a plot of the ion beam diameter (FWHM) at the various stages in the soft landing instrument.

Conclusions

In summary, we demonstrate the application of a position sensitive detector, the IonCCD, for characterization of the ion beam focusing in a custom-built mass spectrometer used for soft- and reactive landing of mass-selected ions onto surfaces. The detector can provide detailed information to the experimentalist for the common tasks of aligning charged-particle optics and mass spectrometry components in a vacuum chamber and determining the appropriate potentials to apply for maximum ion transmission from ionization source to final destination (detector, surface, etc.) Moreover, the detector provides insights into how small errors in the alignment of ion optics may result in non-ideal trajectories and unforeseen effects from applied potentials. Furthermore, due to its sensitivity and the relatively short integration times (25–100 ms) that are necessary to obtain ion beam profiles, the detector should enable time-dependant patterns to be visualized. Combined with existing techniques and tools, such as SIMION, this work demonstrates how the IonCCD may potentially be used to facilitate the design, assembly, maintenance, and utilization of custom-built mass spectrometry instrumentation.

The findings presented above also suggest that a two-dimensional (2D) IonCCD design would enable more complete ion beam diagnostics, especially in non-cylindrical symmetry conditions. Such a detector may be mounted on the multiple gate valves that typically separate various stages of differential pumping in a custom instrument, thereby allowing a systematic optimization of the current and shape of an ion beam along the entire beam line. This is particularly relevant as most defocusing and loss of ion beam current occurs in the grounded regions of gate valves where it is impossible to mount charged-particle optics. In addition, though not addressed in this study, due its ability to operate at high-pressure and its sensitivity to charge rather than kinetic energy, the IonCCD may potentially be employed to study the residual highly charged micro-droplets that are produced by ESI sources. This information may be used to aid the design of future ESI sources with increased ion yield.

Acknowledgments

The authors acknowledge support for this research by a grant from the Chemical Sciences Division, Office of Basic Energy Sciences of the U.S. Department of Energy (DOE), and the Laboratory Directed Research and Development Program at the Pacific Northwest National Laboratory

(PNNL). This work was performed at the W. R. Wiley Environmental Molecular Sciences Laboratory (EMSL), a national scientific user facility sponsored by the U.S. DOE of Biological and Environmental Research and located at PNNL. PNNL is operated by Battelle for the U.S. DOE.

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