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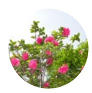

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Novel Linear Ion Trap Mass Analyzer Built with Triangular Electrodes

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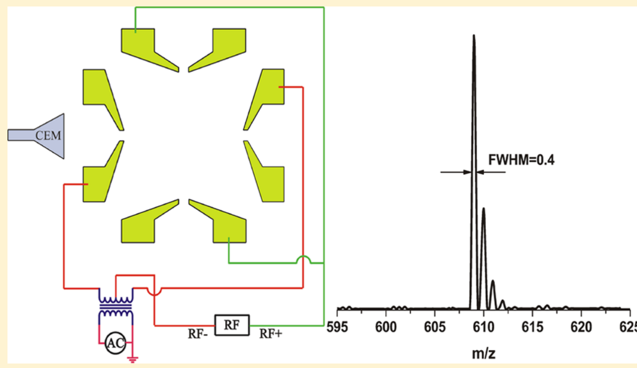
ABSTRACT: A novel linear ion trap mass analyzer built with four triangular electrodes, the triangular-electrode linear ion trap (TeLIT), has been built and its performance has been characterized. The TeLIT has all the properties of a conventional LIT mass analyzer, performing ion trapping, mass analysis, and tandem mass spectrometry functions. The TeLIT was constructed with four identical triangular cross-section shaped electrodes and two planar electrodes. Unlike commercial LITs, which have very well-defined hyperbolic shaped electrodes, the TeLIT electrodes have a much simpler geometric structure and larger mechanical tolerances. The electric field distribution inside the IT region was simulated and there are more quadrupole field components and less higher order fields compared with those in other simplified ITs, such as cylindrical ion trap and rectilinear ion trap; hence, the instrument would potentially offer a relatively high mass resolution. In routine measurement, mass analysis with a resolving power of over 1500 at m/z = 609 Th was obtained. The TeLIT was shown to perform basic mass spectrometer functions such as mass-selected isolation, mass-selected ejection and collision-induced dissociation (CID) of ions comparable to other available LITs. Moreover, given the small size of the TeLIT and its simple structure and good analytical performance, further miniaturization and use as a portable mass spectrometer are envisaged.



Mass spectrometry is a very important analysis technique in modern analytical science, being widely used in many areas on account of its high speed, high specificity, high sensitivity, and high resolving power.[1−7] Conventional mass spectrometers tend to be of relatively large size and weight, consume significant power, and in terms of manufacture and maintenance, are relatively complex. As a consequence, such features have limited the application of mass spectrometers in many key areas. Increasingly there is a demand for analysis techniques to have the capability for in situ analysis, in-line monitoring and even high-throughput analysis.[8−11] All of these practical applications require compact and portable instruments. Among the various types of mass analyzers, the ion trap (IT) mass analyzer has the advantages of simple structure, compact size, tandem mass analysis capability and no excessive need for a low working pressure, so the IT mass analyzer can be an ideal choice for both routine use in the laboratory and for miniaturization purposes.[9,12]

The conventional three-dimensional (3D) Paul trap is composed of a hyperbolic ring electrode and two hyperbolic end-cap electrodes.[12−16] The electrode system of a 3D Paul trap requires rather high manufacturing and assembly accuracy. Also as an IT, 3D ion trap has a relatively low charge capacity and trapping efficiency for achieving a high resolution mass spectrum, and the total ion charge inside the trap must be less than several hundred.[17] Some efforts have been made to overcome these shortcomings. At first, when LITs were developed;[18−21] the sample ions were trapped using a radio frequency (RF) field in the radial direction, while in the direction along the quadrupole axis the trapping field was a constant field created by a DC potential. In this way, as the ion cloud distributes along the quadrupole axis, the trapping efficiency and charge capacity can be greatly improved. Nowadays, commercial LITs normally contain hyperbolic shaped electrodes, but the hyperbolic electrode system still requires high mechanical accuracy in fabrication and assembly. Cylindrical ion traps (CITs) have also been developed.[22−26] Typically these devices contain a barrel-shaped central ring electrode with two flat end-cap electrodes; with this simpler geometry, they are relatively easy to fabricate, but the trapping capacity, trapping efficiency and mass resolving power are low.

To avoid the complexity of the electrode system, as well as the low charge capacity and trapping efficiency, significant efforts have been made in the past decade to simplify the electrode design of the LIT. In 2004, Cooks and co-workers developed a new IT termed the rectilinear ion trap (RIT),[27−31] which takes advantage of the IT capacity of the LIT and the geometrical simplicity of the CIT. The RIT is composed of six rectangular electrodes, so it is very easy to fabricate, with high accuracy being achieved in machining of the rectangular electrodes. With a simple structure and relatively high charge capacity and trapping efficiency, the miniaturized RIT gave

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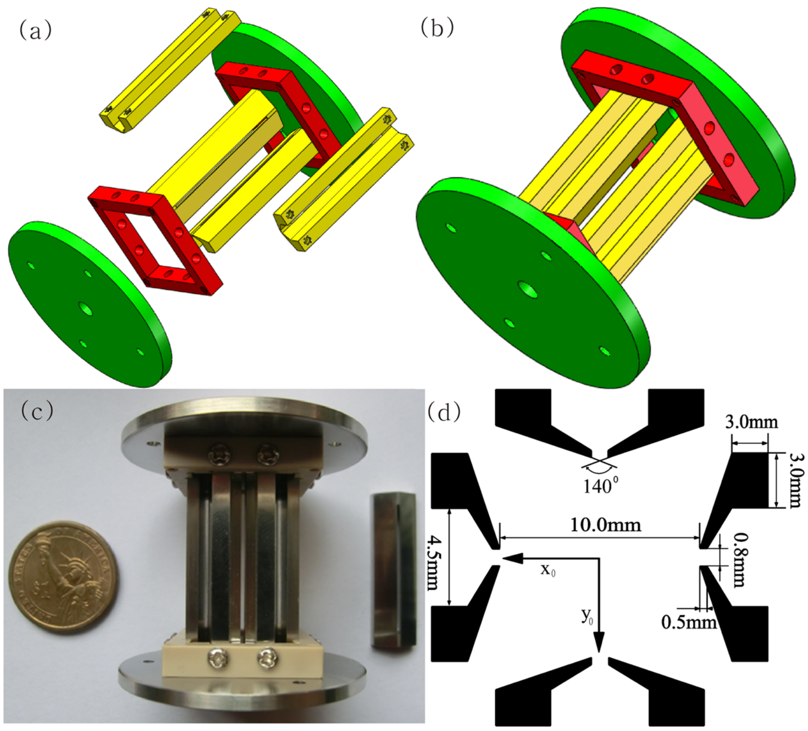

Figure 1. Schematic diagram of the TeLIT. (a). Assembly method for the TeLIT. (b). Structure of a finished TeLIT. (c) Picture of TeLIT and its triangular electrode. (d) Cross section and geometry of the TeLIT.

rather good analytical performance and has featured in hand-held mass spectrometers.[32−35] More recently, ion trap array mass analyzer were proposed.[36−40] For example, the ITA,[36,37] which developed by this group, consists of rectangular electrodes but with a much simpler structure, each analysis channel being equal to an IT composed of four or five electrodes. In all these structures the use of simplified electrodes can lead to aberration of the internal electric field distribution and high-order field components may arise in the IT region in addition to the quadrupole field, and the high-order fields may degrade analytical performance. As is well-known, the properties of ITs depend largely on the electric field distribution. Therefore, the geometric structure of the RIT will affect the electric field distribution and the performance of the IT.[27] To optimize performance of the IT, the printed circuit board (PCB) IT was proposed.[38,41−43] This device is composed of four PCB rectangular electrodes, each PCB plate being fabricated to specially designed patterns and featuring several separate electric strips, the strips being separately insulated from each other, and having applied to them different voltages. Changing the voltage applied to each strip can directly adjust the internal electric field distribution and lead to optimized performance for the PCBIT during the experiment.[41,42] Various other designs of ITs have been proposed, including the Halo IT[44] and the planar quadrupole trap[45] that is aimed to overcome the shortcomings of ITs by changing the geometry of the electrodes. In 1987, Beaty proposed a 3D quadrupole ion trap[46] with cylindrically symmetric triangular electrodes. In the light of his theoretical simulation results, the hyperbolic-shaped electrodes of 3D ion trap could be replaced by triangular-shaped electrodes, and some important features of ion trap could still be achieved. Very recently, a new LIT consisting of four triangular electrodes was proposed by Sudakov and co-workers.[47] According to their theoretical simulation, the field components in this LIT is dominant A_2 with little high order fields A_6, A_{10}, A_{14}, ... with the structure of inscribed radius 5 mm, the simulation results indicated that high resolving power can be obtained at small positive field distortions A_6. The ion motion was simulate using AXSIM software,[48] the buffer gas was helium with a working condition of 0.2 mTorr and 300 K. With a hard sphere collision model, the structure of 140°electrode angle LIT can obtain a mass resolving power higher than 18000 at m/z = 1891.2 Th at the scan rate of 300Th/s, the trapping square wave RF amplitude was 2000 V with a supplementary resonant excitation AC of 1.4 V.

In the present work, a LIT composed of four triangular electrodes was built and characterized experimentally. The electric field distribution was simulated using computer software SIMION 7. Analytical performance was investigated using a homemade electrospray ionization mass spectrometry (ESI-MS) platform. A mass resolution in excess of 1500 at m/z = 609Th was routinely observed, and the mass-selected ion isolation, mass-selected ion ejection and tandem mass analysis functions were all readily realized. The instrument is low cost, of simple design and offers good analytical performance, and has potential for use as a compact, portable IT mass spectrometer.

■ EXPERIMENTAL SECTION

Structure and Fabrication of the TeLIT. Figure 1a−d shows the assembly and the geometric structure of a TeLIT. The triangular electrodes (yellow parts in Figure 1a) were made from 304-stainless steel. Some ceramic parts (red parts) were used as the insulating spacers and as holders for the IT electrodes. The length of the triangular electrodes is 36 mm, and a 30 mm-long ×0.8 mm-wide slit is located in the center of each triangular electrode for ion ejection and detection. Two stainless steel plates (green parts) were mounted at the two ends of the triangular electrodes and the rectangular electrodes,

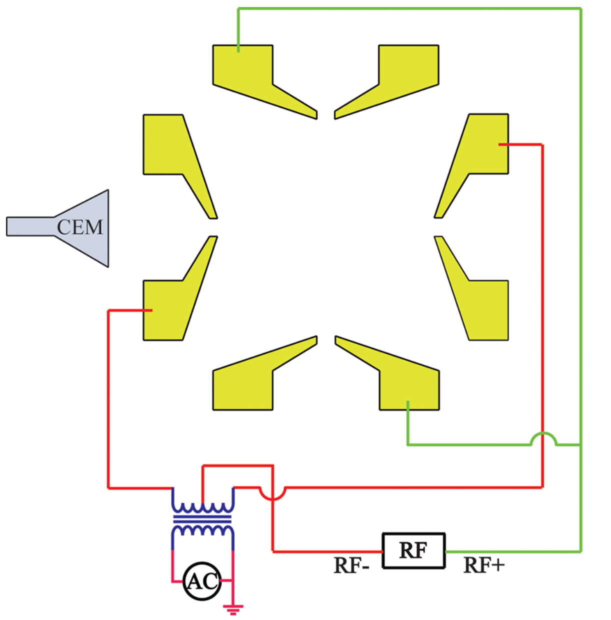

Figure 2. Schematic diagram of the power supply circuit for the TeLIT. CEM stands for Channeltron electron multiplier.

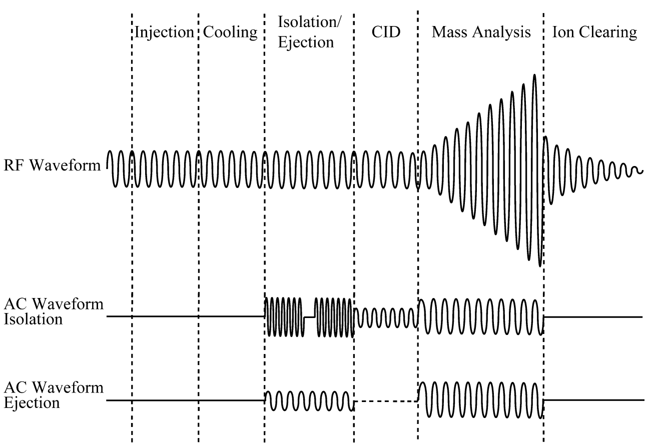

Figure 3. Experimental timing sequence for mass-selected ion isolation and mass-selected ion ejection.

respectively, as end-cap electrodes. A 3 mm-diameter aperture was machined in the center of each end electrode for ion injection. Figure 1c shows the picture of an assembled TeLIT and its triangular electrode, also a one dollar coin was placed aside for comparison. The geometric parameters for the TeLIT are listed in Figure 1d, these parameters were mainly chosen based on Sudakov's and our own simulation results, such as the electrode angel, the inner dimensions, the width of center slit, other parameters were chosen from our former work experiences with RIT,[30] ITA,[37] and PCBIT.[41] The overall mechanical tolerance is around 0.1 mm and this result was tested using optical microscope (DC-3000; Rational Precision Instrument Co., Guangdong, China).

Experimental Setup. A custom-made three-stage differential pumping vacuum system was used in this study, as previously described.[30] Briefly, an electrospray ionization (ESI) source was used to produce the sample ions, and a high voltage of 3800 V was applied typically. The sample solutions were pumped using a syringe pump (55-2222, Harvard Apparatus, Massachusetts, USA) at a flow rate of 1 μL/min and the

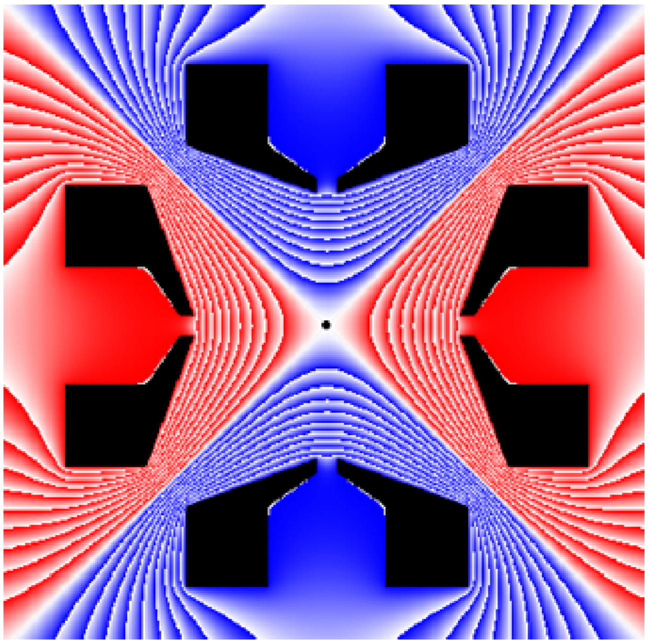

Figure 4. Simulation of electric field distribution at one point in time in the TeLIT.

generated sample ions were directed by an RF-only quadrupole ion guide into the TeLIT through a 3.0 mm diameter aperture on the front end-cap electrode from the axial direction. A TeLIT with inner dimensions of $x_0 \times y_0$ = 5 mm × 5 mm was used, as shown in Figure 1d. Helium gas was used as an ion-cooling buffer gas and to perform the collision induced dissociation (CID) experiments. The helium pressure in the mass analyzer region was 3×10^{-3} Pa for mass analysis processes and 9×10^{-3} Pa for CID experiments, the pressure were directly tested using cold cathode and pirani composite ionization gauge (CPG-600; Tamagawa, Shanghai, China) without applied correction factor. A Channeltron electron multiplier (4220; Photonis, Massachusetts, USA) was used as an ion detector.

The method for connection of the power supply to the TeLIT was quite similar to previous work,[27,30] as shown in Figure 2. The only difference in this study was that all the triangular electrodes were identical and assembled in a symmetrical manner, so there was no difference between the x and y electrodes. A supplementary alternating current (AC) waveform was added to one pair of x or y electrodes for ion excitation. The radio frequency (rf) power supply (rf module 009701, Sciex, Toronto, Canada) with a fixed frequency of 768 kHz[30,36,37] was used to drive the TeLIT. The supplementary alternating current (AC) waveform was superimposed on the rf trapping waveform by a toroidal transformer for resonance ion ejection during the mass scan and dipole excitation in CID.

Timing Sequences. The timing sequences in this study were quite similar to our previous works,[30,36,37] as shown in Figure 3. The experiment concerning the tandem MS capability test involved six stages: ion injection, ion cooling, mass-selective ion isolation, collision-induced dissociation (CID), mass analysis, and ion clear out. During the experiment, the ion injection and ion cooling stages lasted for 12 and 30 ms, respectively, during which, the amplitude and the frequency of the rf waveform signal were fixed at 450 V (zero to peak) and 768 kHz, respectively. For the mass-selected ion isolation stage,

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Table 1. Electric Field Distribution in the TeLIT

fields	A_2	A_4	A_6	A_6/A_2 (%)	A_8	A_{10}	A_{10}/A_2 (%)
components	0.9564	0.0000	0.0069	0.7214	0.0000	0.0029	0.3032

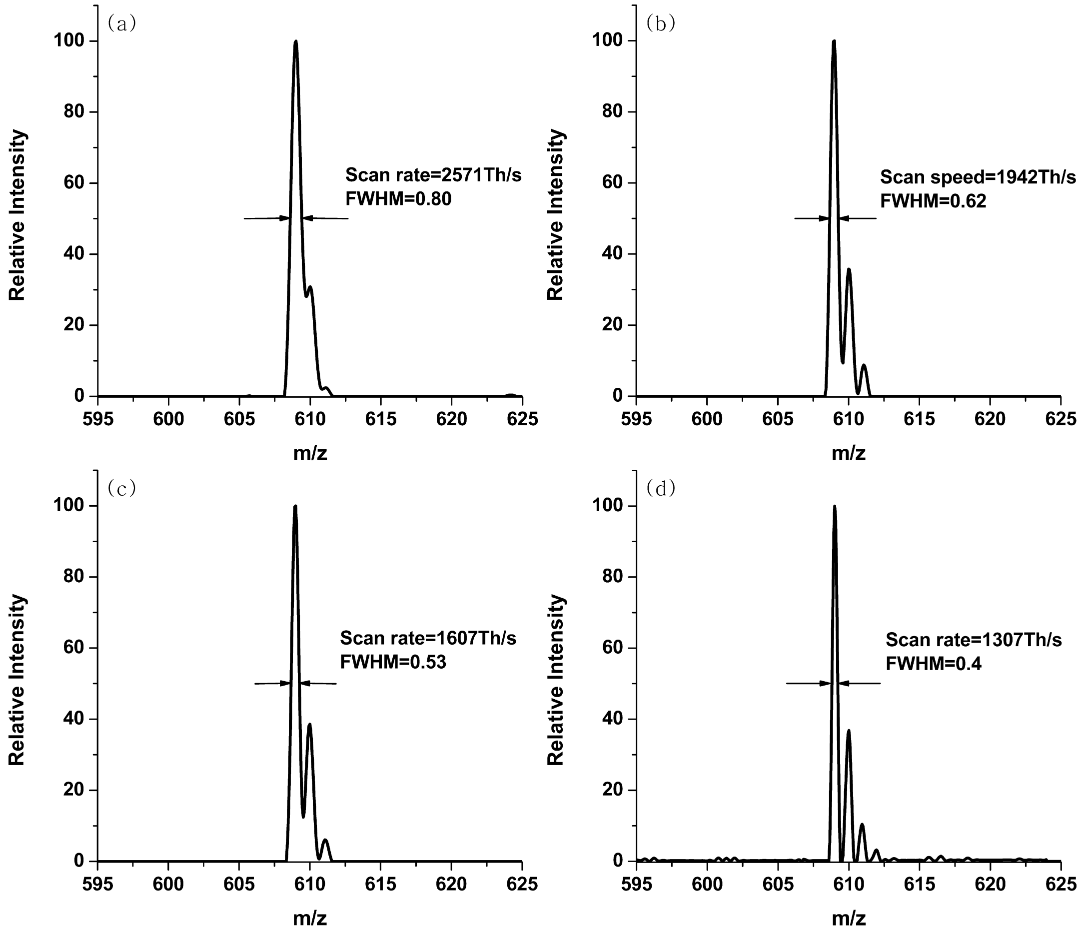

Figure 5. Mass resolution of protonated reserpine molecular ion at different scan rates.

the amplitude and frequency of the rf waveform signal remain unchanged, a notched SWIFT[49,50] signal being added to isolate the precursor ion generated from the sample; this stage lasting for 20 ms. After the isolation period, a resonant AC signal was used to excite the isolated precursor ion for 20 ms. Then, the fragment ions which remained in the trap were mass-analyzed by ramping the voltage of the rf signal during the mass analysis period. After all these stages, the rf waveform signal decreased to 0 V, to clear out all the ions in the TeLIT. The timing sequence for mass-selected ion ejection is similar to the tandem MS capability experiment, as shown in Figure 3, where there are five stages: ion injection, ion cooling, mass-selected ion ejection, mass analysis, and ion clear out. The difference between the two is in the third stage, where for a particular m/z ion there must be a resonant frequency, so in the stage of mass-selected ion ejection, a supplementary AC with a specific resonant frequency value was used to eject the particular m/z ion out of the trap for detection.

Electric Field Simulation. The field distribution inside an ion trap depends on the geometrical structure of its electrodes and their symmetry.[51−54] In this work, we carefully optimized the geometric structure and the symmetry of the TeLIT for achieving good resolving power for both mass scan and mass selective ejection. The electric field distribution inside the IT region of the TeLIT was simulated using Simion 7 and the results are shown in Figure 4 and listed in Table 1. It can be seen that the electric fields in the TeLIT are dominantly quadrupolar fields, although some minor high-order fields also coexist. With such a symmetric geometry, the high-order fields, such as A_6, A_{10}, A_{14} and so on are much smaller in this study compared with those in other simplified ITs, such as the cylindrical ion trap[24] and rectilinear ion trap.[27,30]

■ RESULTS AND DISCUSSIONS

Mass Resolution. In an IT mass analyzer, there are many factors which influence mass resolution, such as the rf scan rate, the DC trapping potential along the z axis, the mass to charge ratio of ion and the buffer gas pressure and so on. For instance, it is well-known that a decrease in the rf scan rate can improve the mass resolution for IT mass analysis.[27,30,36,37,41] The effect of the rf scan rate on the mass resolution was tested in this work and the results are shown in Figure 5. The experiment was operated in conventional rf mode as previously reported[36] and the protonated molecular ion peak of reserpine (m/z = 609) was used to test the mass resolution. During the test, the amplitude of the supplementary ac signal was optimized for each scan rate to achieve the best resolution, although other parameters such as the pressure of buffer gas, resonance

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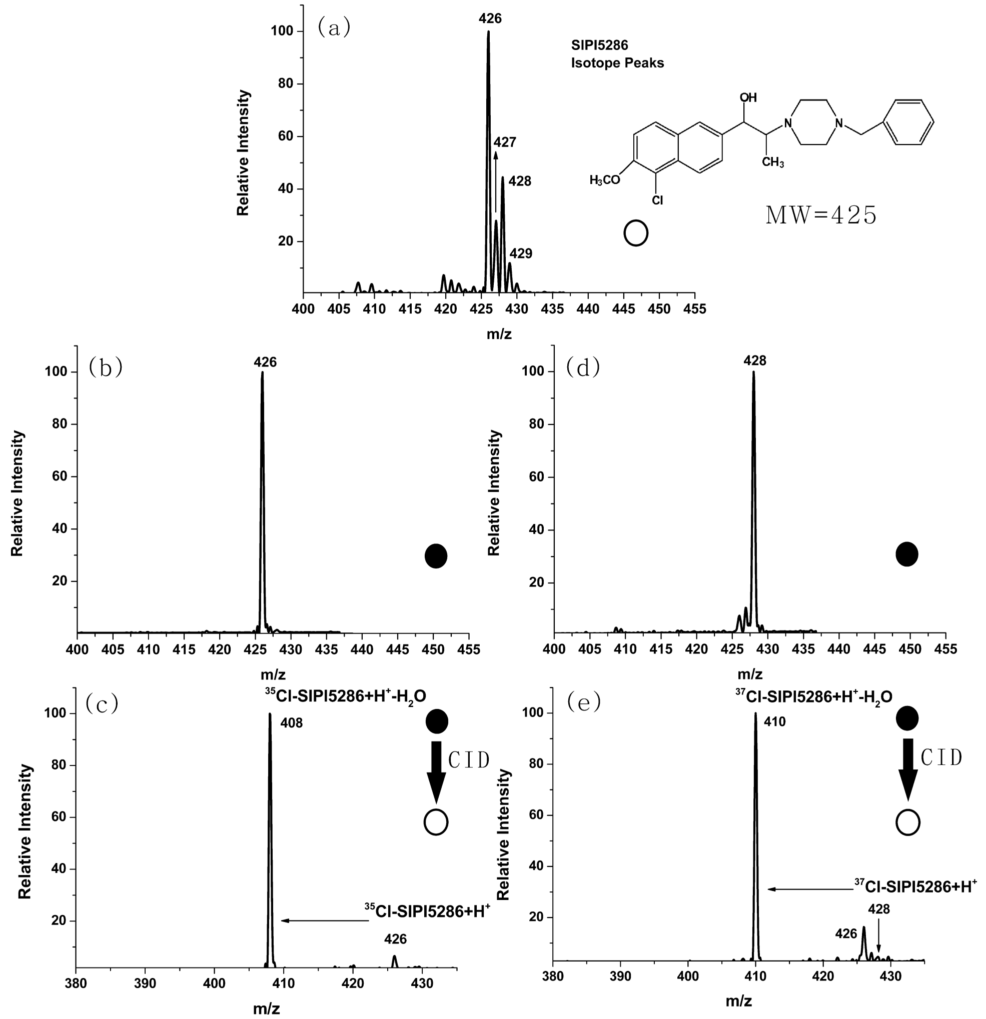

Figure 6. Tandem mass spectrometry of protonated SIPI5286 molecular ion.

ejection conditions, and the axial potential of TeLIT were held constant. As shown in Figure 5, the full width at half-maximum (fwhm) values were calculated for each experiment and results are shown in Figure 4. It can be seen that the mass resolution gradually improved with a decrease in the rf scan rate, and as shown in Figure 4d, a mass resolution of 1500 was attained for a scan rate of 1307 Th/s.

Tandem MS Capability. Tandem mass spectrometric analysis was also tested in this work. In the experiment, the SIPI5286 molecule (MW = 425)[55] was used as the sample and the ESI mass spectrum is shown in Figure 6a; a notched SWIFT was used to isolate the parent protonated molecular ion (m/z = 426). Figure 6b shows the result for mass-selected ion isolation of the peak signal at m/z = 426. During the test, a SWIFT waveform whose frequency varied from 10 to 550 kHz with frequency notches of 204.6−205.5 kHz and an amplitude of 8 V was used. Resonant AC power of frequency 205.1 kHz and an amplitude of 900 mV was used to excite the ion (m/z = 426) and the fragment ion at m/z = 408 was observed with a CID efficiency of 84.8%. The mass spectrum is presented in Figure 6c. In the same way and as shown in Figure 6d and e, the isotope peak at m/z = 428 could also be isolated with frequency notches of 203.6−204.4 kHz and using a resonant AC of 203.7 kHz and 800 mV amplitude; this yielded fragment ions of m/z 410 and the CID efficiency was 96.5%.

Mass-Selected Ion Ejection. In contrast to mass-selected ion isolation, mass-selected ion ejection is a process for ejecting a specific ion, of particular m/z value, out of the trap while the other ions are retained in the trap. For each ion of specific m/z in the IT, there must be a corresponding resonant frequency, thus the ion could be excited and ejected from the IT by using a supplementary AC signal at the specific resonant frequency for the ion. The capability of mass-selected ion ejection for the TeLIT was, therefore, examined. As shown in Figure 7, the ions of m/z 426 were ejected when a supplementary ac waveform with frequency of 204.9 kHz and amplitude of 750 mV was applied for 20 ms. In the same manner, ions of m/z 427, 428, and 429 were ejected when using ac waveforms of frequencies 203.8, 203.2, and 202.3 kHz, respectively.

■ CONCLUSIONS

A LIT built with four triangular shaped electrodes has been constructed and its performance evaluated. The electric field distribution inside the ion trapping region was simulated, and the electric fields in the TeLIT are predominantly quadrupolar fields plus some minor high-order fields. The low multipolar electric fields in the trap could be the reason for the relatively high mass resolving power of the TeLIT, where a mass resolution in excess of 1500 was achieved. This resolving power is higher than that for several simplified LIT designs.[27,36] Mass-

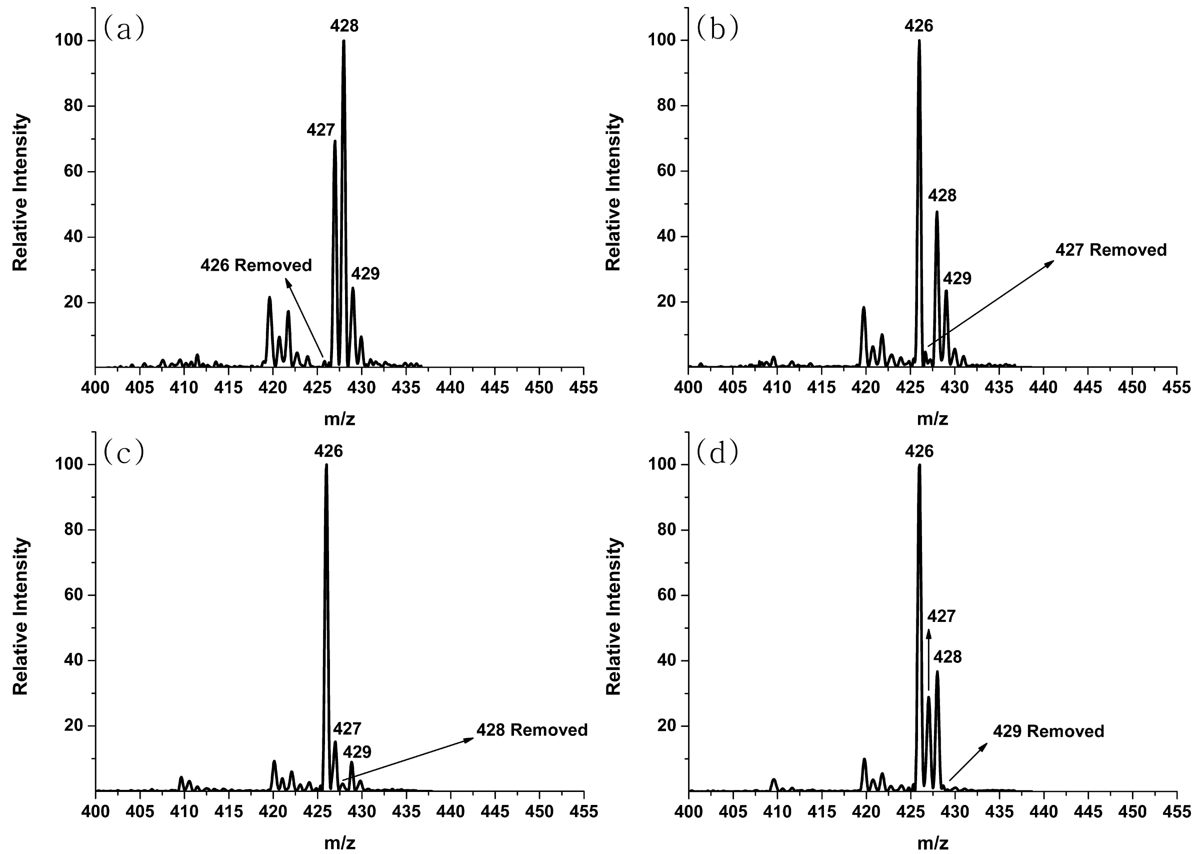

Figure 7. Mass spectrum of protonated SIPI5286 molecular ion for mass-selected ion ejection.

selected ion isolation with at least a mass resolution of 428 was realized. In the case of tandem mass analysis, mass-selected ion ejected was demonstrated for SIPI5286 using ion peaks at m/z = 426, 427, 428, and 429. Further studies on the TeLIT are planned, including studies on geometric structure optimization, mechanical tolerance effects, and the "digital ion trap mode". It is anticipated that further improvements in analytical performance of the TeLIT can be achieved, based on recent reports.[36,56]

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Notes

The authors declare no competing financial interest.

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