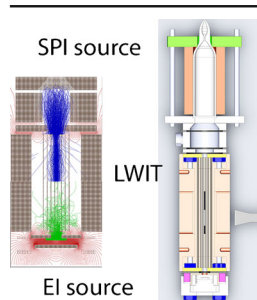


RESEARCH ARTICLE

A Miniaturized Linear Wire Ion Trap with Electron Ionization and Single Photon Ionization Sources

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Abstract. A linear wire ion trap (LWIT) with both electron ionization (EI) and single photon ionization (SPI) sources was built. The SPI was provided by a vacuum ultraviolet (VUV) lamp with the ability to softly ionize organic compounds. The VUV lamp was driven by a pulse amplifier, which was controlled by a pulse generator, to avoid the detection of photons during ion detection. Sample gas was introduced through a leak valve, and the pressure in the system is shown to affect the signal-to-noise ratio and resolving power. Under optimized conditions, the limit of detection (LOD) for benzene was 80 ppbv using SPI, better than the LOD using EI (137 ppbv). System performance was demonstrated by distinguishing compounds in different classes from gasoline.

Keywords: Linear ion trap, Wire electrodes, Vacuum UV ionization, Miniaturized ion trap

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Introduction

Portable mass spectrometry has wide applications, including public safety, environmental protection, and industrial process monitoring [1–8]. Miniaturization of a conventional mass spectrometer includes reducing the size of the mass analyzer and coupling it to one or more appropriate ion sources. Nearly all types of mass analyzers have been miniaturized [1], including a magnetic sector mass analyzer [9], quadrupole mass filters [10, 11], quadrupole ion traps [12–16], a Fourier-transform ion cyclotron resonance mass analyzer [17], and time-of-flight (TOF) mass analyzers [18, 19]. Among miniature mass analyzers, quadrupole ion traps are distinguished because of their compact size, relatively high working pressure and the unique capability to perform multistage tandem mass analysis (MSⁿ). The linear ion trap (LIT) has attracted recent attention because of higher ion trapping efficiency from an external ion source, increased ion storage volume, and good resolving power [20]. In our laboratory we have built a miniaturized linear wire ion trap (LWIT) and found it has additional advantages for portable mass analysis because of high ion transmission rates, low capacitance, and low weight [21].

Ion sources coupled to miniature mass analyzers include electron ionization (EI) [17, 22], discharge ionization [23], electrospray ionization (ESI) [24–26], single photon ionization

(SPI) [27–33], and desorption electrospray ionization (DESI) [34, 35]. Because samples are usually mixtures in a real test environment, the ability to distinguish single compounds from a mixture is very important when no separation devices, such as a gas or liquid chromatograph, are coupled to the ion trap. Single photon ionization (SPI), as a soft ionization technique, has the ability to detect organic compounds in mixtures with good sensitivity [28, 29]. In addition, a vacuum ultraviolet (VUV) lamp is also a suitable photon source for portable mass spectrometry because of its small size, low power consumption, variety of choices for photon energy, and strong photon flux.

VUV lamp ion sources have been coupled to miniature TOF mass spectrometers and proved to be a good combination [27–29, 36–39]. However, the high vacuum and resolution requirements in a TOF analyzer make the further size reductions very difficult in such systems. In a recent study, the combination of a VUV lamp and a commercial ion trap showed good sensitivity [40]. In addition, the MS/MS ability in ion traps can provide structure information and, thus, is very helpful in identifying compounds. However, challenges were encountered when coupling the VUV lamp to the miniature ion trap. First, the photons emitted from the VUV lamp can generate a strong signal on the detector through the photoelectric effect, which increases background noise and even causes the detector to saturate. Therefore, it significantly reduces the lifetime of the detector. Second, to avoid space charge effects inside the ion trap, a control system is needed to control the amount of ions inside the ion trap. A design that simply radiates VUV light into the ion trap is unable to meet this requirement.

Building on our previous work with the LWIT [21], here we demonstrate both SPI and EI ion sources for the miniature LWIT. In the SPI ion source, a VUV lamp is pulsed ON and OFF to control the ionization and avoid spurious photon signals during m/z analysis. The SPI ion source is set outside the ion trap and the transmission rate can be controlled by a potential on the end electrode of the trap. The limit of detection (LOD) for benzene is shown to be 80 ppbv under routine working conditions. Instrument performance is further demonstrated by analysis of compounds present in gasoline.

Experimental

Dual Ion Source Design

Figure 1 shows the configuration of the LWIT with an EI ion source and a VUV lamp ion source. The detailed configuration of the LWIT was introduced in a previous study [21]. In this section, we briefly describe the LWIT. The wire support plates in Figure 1 were made of polyether ethyl ketone (PEEK) and were drilled with 24 holes in each plate. The diameter of each hole is 0.23 mm, and the diameter of each stainless steel wire is 0.2 mm. The tension applied on the wires is provided by eight nuts on four threaded rods. The distance between the two plates is 62 mm. Each plate contains a small end electrode used for axial confinement of the trapped ions. The distances between the Y copper shields and X copper shields are 16.8 mm and

14 mm, respectively. This geometry was designed to increase the ion ejection efficiency in the X direction.

A gated electron gun was set on the right of the LWIT. The electrons generated by the electron gun were pushed into the LWIT under a potential of -60 V. The electron gate alternated between 22 V (allowing electrons through) and -100 V (closed). Neutral sample molecules were ionized inside the LWIT and were confined by two end electrodes, each at 12 V. A magnetic ring was set around the electron gun and used to confine the trajectory of electrons. Previous work shows that the magnetic ring can improve the electron ionization efficiency by up to two orders of magnitude in the photo ionization source [29]. In our setup, simulations show that the magnetic ring can increase the transmission efficiency from 6.8% to 10.6%. In addition, the helix path of the electrons during weak rf field significantly increases their path and, thus, increases the ionization efficiency. The ions are less affected by the magnetic field due to their relatively heavy mass. A study shows that a magnetic field lower than 0.1 T does not significantly perturb the ion's frequency of motion [41]. The magnetic field in our setup (0.07 T close to the ring and 0.015 T in the position 5 mm away) is far below 0.1 T, so the impact of magnetic field on the ions is negligible.

The VUV lamp used to provide soft ionization (PKR 106; Heraeus Noblelight GmbH, Hanau, Germany) was set on the left of the LWIT. The energy of the photons is 10.6 eV (117 nm) with FWHM of about 0.1 eV. A sample inlet and a helium inlet

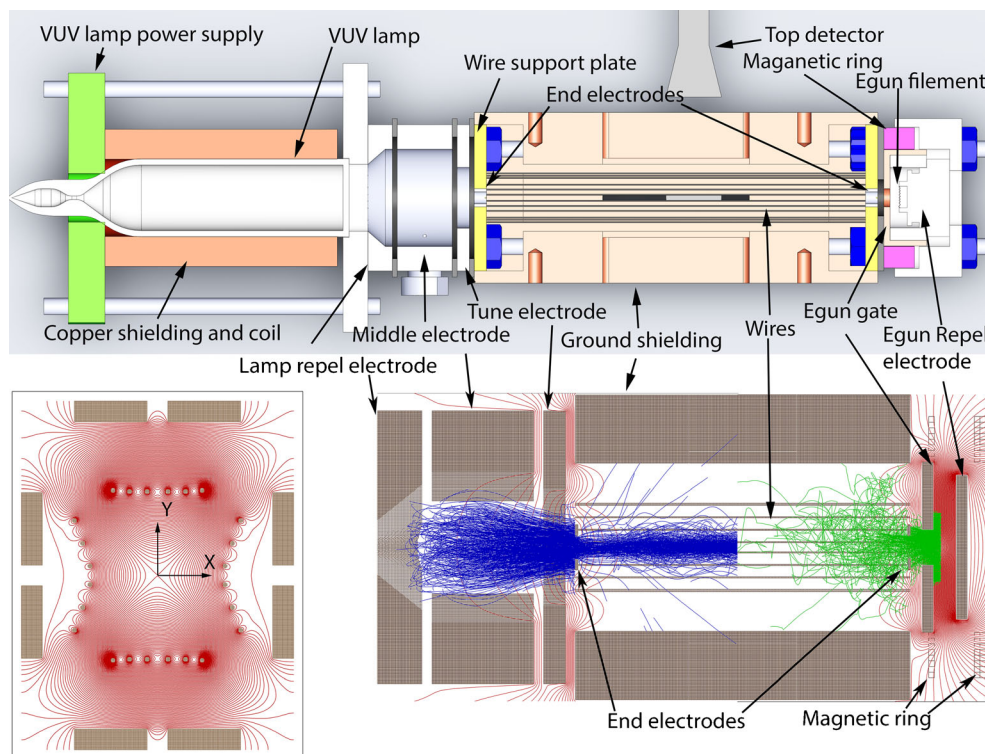


Figure 1. The top figure is the schematic for the VUV lamp and EI ion sources coupled to the LWIT. The bottom left figure is a cross-section view of the electric field in the wire ion trap. The bottom right figure is the model built in SIMION for verifying the configuration. The blue lines in the bottom figure are trajectories of ions generated from the VUV lamp ion source, and the green lines are electrons generated from the EI ion source. The red lines are isopotential, lines

were drilled in the middle electrode on which a DC 20 V potential was applied. A repel electrode at 23 V pushed ions into the ion trap. A tune electrode was used to focus ions into the hole on the wire support plate. The optimized potential on this electrode is around 15 V. The VUV lamp was powered by a 13.5 MHz rf power supply (C210; Heraeus Noblelight GmbH), which needed a 25 V DC potential as input. To avoid the strong noise generated by the VUV light on the detector, a pulse amplifier (Model: GRX-1.5 K-L; Directed Energy Inc., Fort Collins, Colorado, USA), controlled by a pulse generator, served as the power supply for the VUV lamp. VUV lamp ignition takes about 0.02 s, setting a lower bound for the repetition rate on a scan. A copper foil was used to shield the lamp power supply to avoid coupling to the detector signal (not shown).

A model in SIMION 8.1 (Scientific Instrument Services, Inc., Ringoes, NJ, USA) was built for verifying the configuration, as shown in Figure 1. In the study of the electron transmission rate, the time of birth for electrons is set as a unified distribution from 0 to 650 μs (1000 periods of rf power). The rf is 1.53 MHz and the amplitude is 600 $V_{\text{p-p}}$. The birth position of electrons is set as the same position as the hot filament. The magnetic ring is also built into the model and the magnetic field is set according to a measured value from the magnetic ring (0.07 T close to the ring and 0.015 T in the position 5 mm away). Under such conditions, the simulated electron transmission rate through the electron gate is 6.8% without the magnetic ring and 10.6% with the magnetic ring.

The trapping efficiency was also evaluated under the same rf power supply. The birth position of the ions is a cylinder under the VUV lamp with diameter of 8 mm and length of 10 mm with a random distribution. The initial ions are set as $m/z = 100$ and 10,000 ions are used to get precise results. The hard sphere collision model is introduced in the simulation. The buffer gas is set as helium and the pressure is 0.35 Pa, the same as the experimental condition. The electrodes potential is based on the optimized potential in the experiment. Under such conditions, the simulation shows that the trapping rate for ions from the ion source into the trap is estimated to be 6%.

The vacuum was supported by a turbo pumping station (HiCube; Pfeiffer, Nashua, NH, USA) with a pumping speed of 35 L/s for N_2 . The sample gas was leaked into the vacuum through a leak valve (Granville-Phillips Co., Boulder, CO, USA) to reach an indicated pressure of 0.002 Pa. The gas flow rate was estimated to be 0.042 mL/min at atmosphere pressure. After the pressure reached a stable status, helium was introduced as the buffer gas and the pressure of the chamber kept at 0.35 Pa, as measured using a calibrated Bayert-Alpert-type ionization gauge (FRG-720; Agilent, Santa Clara, CA, USA).

Electronics and Time Sequence

A 1.53 MHz rf signal from a power supply (Ardara Quadrupole Power Supply; ArdaraTech, Ardara, PA, USA) was applied to the wires of two opposite sides of the LWIT (wires in the Y direction). A positive DC voltage of up to 12 V was applied to the axial confinement electrodes to provide a DC trapping

potential well for positive ions along the Z axis. The static potential applied on the filament was -60 V. Trapped ions were mass selectively ejected by scanning the rf amplitude at a rate of 3830 Th/s. An AC signal at a frequency of 520 kHz, generated by a waveform generator (DS345; Stanford Research Systems, Sunnyvale, CA, USA) was applied on six X wires on one side to provide the quasi-dipolar field that induced ion ejection at $q = 0.78$ during the rf scan. The ions ejected from the LWIT were detected using an electron multiplier (DeTech XP-2200; Palmer, MA, USA) with estimated gain of $\sim 10^6$. The signal was first amplified using a preamplifier at a gain of 10^7 (Keithley 427 current amplifier; Beaverton, OR, USA), and then acquired using a digital oscilloscope (Teledyne LeCroy Wavesurfer 3024; Chestnut Ridge, NY, USA) at a sampling rate of 500 k samples/s.

Figure 2 shows the trigger sequence for the VUV lamp power supply, electron gun, rf drive waveform, and AC resonant ejection waveform. All these electronics were triggered by pulses from a pulse generator (not shown) (Model: DG535; Stanford Research System, Inc., Stanford, CA, USA). In SPI mode, the VUV lamp was ignited by a positive potential at 22 V. In EI mode, the electron gun gate was turned on by a pulse potential at 22 V and turned off at -100 V. The AC resonant ejection was started at the same time as the rf amplitude began to ramp with peak-to-peak amplitude of 2.2 V.

Materials

Three sample gases were used in this study. Toluene and deuterated toluene (toluene-D8) were used to demonstrate the differences between SPI and EI ion sources. The gas sample was prepared by ejecting 2.85 μL toluene and 2.85 μL toluene-D8 with a 5 μL precise syringe into a 6 L gas bag filled with pure nitrogen, which resulted in a mixture of toluene and toluene-D8 at 115 ppmv. The lower atmospheric pressure (about 86450 Pa) in Provo, UT was considered in the calculation. After 2 h to completely evaporate the liquid samples, a 600 mL gas sample was transferred into a new gas bag with 5.4 L pure nitrogen. In this way, a concentration of 12 ppmv of toluene and toluene-D8 was prepared. A 1.8 ppmv benzene gas sample was prepared in the same way with different values (2.38 μL liquid benzene to generate a concentration of 115 ppmv in a 6 L gas bag and 94 mL sample gas transferred to a 6 L bag filled with 5.9 L nitrogen) and used for evaluating the impact of sample pressure on signal-to-noise ratio and resolution. A gasoline sample gas was produced by evaporating 20 μL gasoline (85-octane regular; Chevron, Provo, UT, USA) into a 6 L gas bag filled with pure nitrogen. This gas sample was used to demonstrate the analytical ability of the SPI ion source.

Results and Discussion

Spectra Comparison

Mass spectra of toluene and toluene-D8 were obtained in both EI and SPI ionization modes and are shown in Figure 3. In SPI

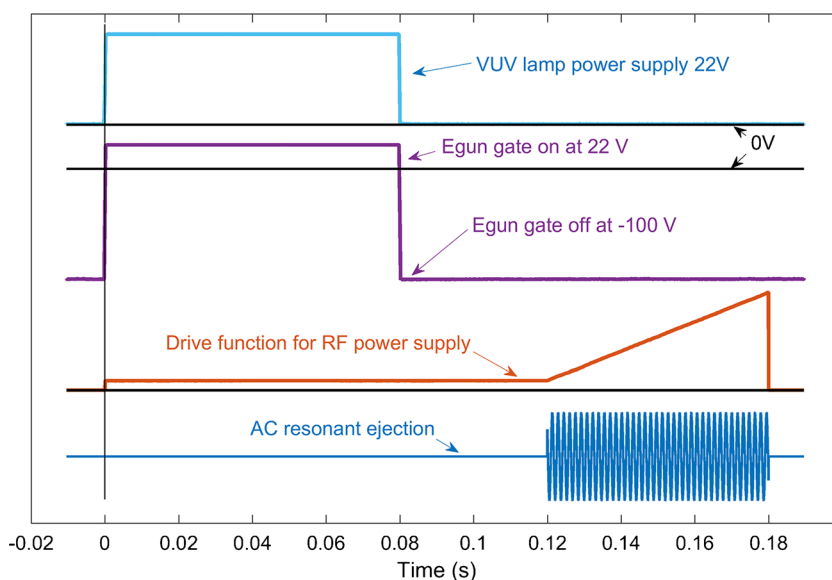


Figure 2. The schematic for the electronic control and sequence during one scan period

mode, the molecular ions at $m/z = 92$ and 100 dominated the mass spectrum and weak fragment ion peaks at $M-1$ and $M-2$ were observed. Other fragment ion peaks were not observed. In the EI mass spectrum, the molecular ions and strong fragment ion peaks were observed. The fragment ion peaks provide structure information for the analytes.

Sensitivity

The sample gas pressure has a significant impact on the resolution and signal-to-noise ratio. Under higher sample gas pressure, more molecules are introduced into the ion trap and, thus, more ions are generated, which increases the detected signal. However, the higher sample gas pressure has a negative impact on the resolution in three ways. First, the collisions between the introduced gas and ions reduce the resolution. Helium was usually

used as the background gas because it can smoothly reduce the energy of ions and cool them to the center of the linear ion trap, whereas the introduced gas molecules are usually heavier than helium and warm up the cooled ions. Second, during the excitation process, the collisions between the high molecular weight molecules and ions significantly deviate the trajectories of ions and, therefore, reduce the resolution. In addition, the resolution is reduced due to space charge effects if the ion intensity inside the trap is too high.

The impact of the sample gas pressure was evaluated using benzene at 1.8 ppmv combined with nitrogen. The sample gas pressure was measured without the helium buffer gas. In the experiment, helium gas was turned on and tuned to keep the working pressure at 0.35 Pa. Figure 4 shows the relationship between the resolving power and signal-to-noise ratio under different sample gas pressure in SPI mode. The signal-to-noise

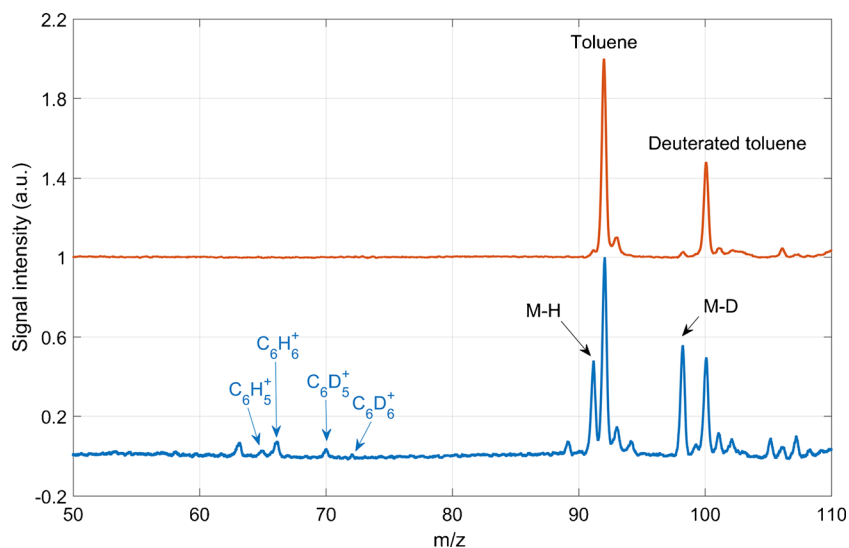


Figure 3. Comparison of mass spectra between VUV lamp and EI ion source. The SPI mass spectrum is shown on the top and EI mass spectrum is shown on the bottom. The mass spectra were an average of 50 scans

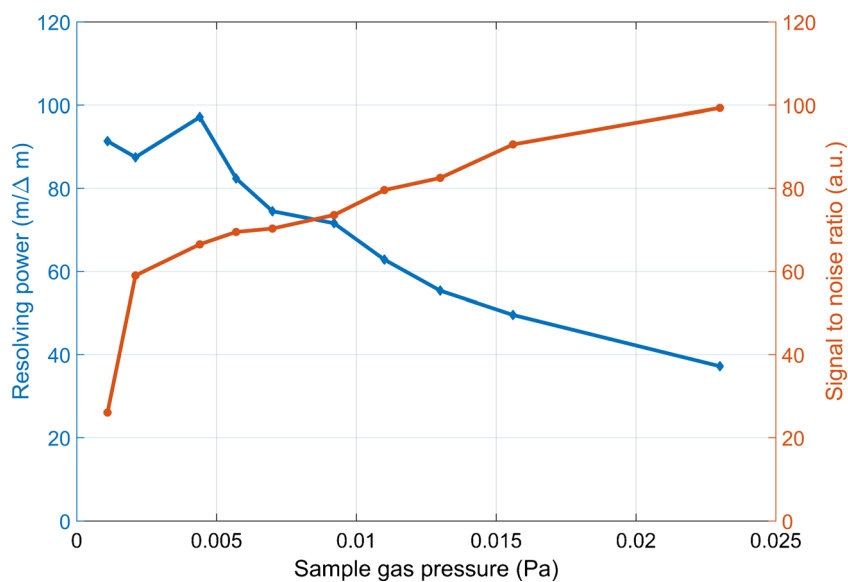


Figure 4. The impact of sample gas pressure on the signal-to-noise ratio and the resolving power. The sample gas is benzene at 1.8 ppmv combined with nitrogen. The sample gas pressure was measured without the helium buffer gas. In the experiment, helium gas was turned on and tuned to keep the working pressure at 0.35 Pa

ratio increases sharply from 0.001 to 0.002 Pa although the reason for this is unclear. After that, the signal-to-noise ratio increases linearly to the sample gas pressure. On the other hand, the resolution reduces with the increased sample gas pressure. To balance the resolution and signal-to-noise ratio, we chose a sample gas pressure of 0.005 Pa as our routine working pressure. Under such a pressure, the signal-to-noise ratio of benzene in SPI is 69, and thus the calculated LOD is 80 ppbv, as extrapolated from the 1.8 ppmv data. This calculation was based on a signal-to-noise ratio of 3:1.

Mass Spectrum of Gasoline

To demonstrate the ability to distinguish organic molecules in SPI mode, we analyzed a sample of gasoline. Although the

gasoline can be well characterized by GC-MS with an EI ion source, this method takes a longer time and is not suitable for in situ applications because of the large and heavy devices required. Figure 5 shows the mass spectrum of gasoline in SPI mode. The spectrum under EI mode (not shown) is very complex with fragments and ions that are difficult to interpret. The SPI mass spectrum clearly shows the molecules in different classes. From the mass spectrum, we can distinguish the hydrocarbons (HCs) with different numbers of carbons, such as C4 HCs, C5 HCs, C6 HCs, and C7 HCs, and the polycyclic aromatic hydrocarbons (PAHs), such as toluene, C2-benzenes, and C3-benzenes. The signal of toluene-D8 is from the background in the system. The signal of toluene may be a combination of the toluene from background and the gasoline sample

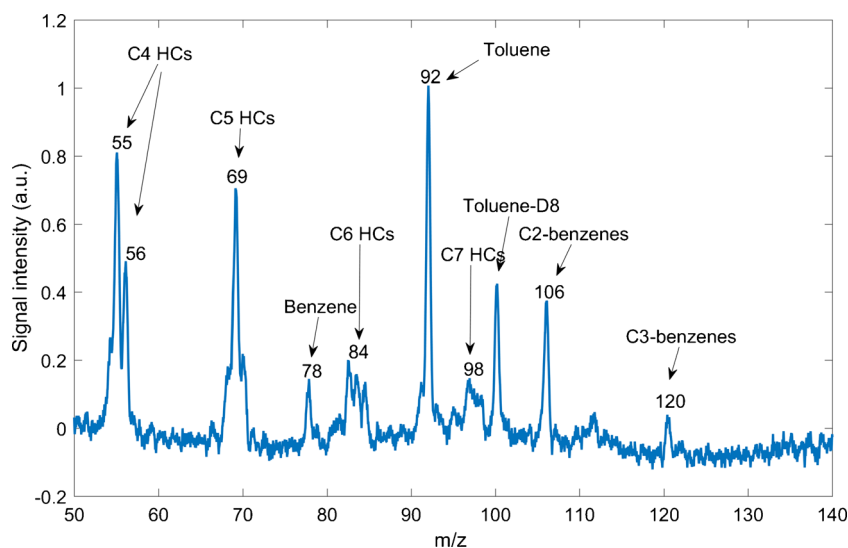


Figure 5. The mass spectrum of gasoline in SPI mode. The mass spectrum was averaged from 500 mass spectra

because its intensity is higher than would be estimated from a purely gasoline sample.

Conclusions

Two ion sources, SPI with soft ionization and EI with hard ionization, were built for the LWIT and tested. The soft ionization in SPI provides information about molecular ions whereas the hard ionization in EI can provide structural information about analytes. These features were demonstrated in the analysis of gasoline, in which compounds from different classes were easily identified. Under optimized conditions, the limit of detection for benzene using SPI was 80 ppbv, and under the routine working conditions, as extrapolated from the 1.8 ppmv data, better than that using EI (137 ppbv). The MSⁿ ability was not available in our current setup, but it could significantly enhance the ability of identifying unknown compounds, especially in SPI mode. The LWIT with dual ion sources is a promising candidate for portable mass spectrometry because of its compact design, good sensitivity, and the ability to detect organic mixtures.

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