



## Article

# Mini 2000: A Robust Miniature Mass Spectrometer with Continuous Atmospheric Pressure Interface

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**Abstract:** A miniature mass spectrometer with continuous atmospheric pressure interface (CAPI) developed previously in our lab has proved to have high stability and rapid analysis speed. With the aim of achieving smaller size, better performance and easier maintenance, in this study, an upgraded miniature mass spectrometer with CAPI was developed, in which all components were optimized and redesigned into a packaged unit. Using a more powerful pumping system, better analytical performances were obtained for this system. The miniature mass spectrometer has the capability to perform tandem mass spectrometry, and could be coupled with ambient ionization sources for analysis of different samples. Good stability (signal relative standard deviation, RSD < 5%), high sensitivity (limit of detection, LOD 10 ng/mL), better than unit mass resolution, and a broad mass range (from 150 Da to 2000 Da) were obtained. Integrated with a tablet computer for system control, the miniature mass spectrometer has dimensions of 38 cm × 23 cm × 34 cm (length × width × height), and is 13 kg in total weight. The whole system is powered by an adapter with a power consumption of 200 watts in total.

**Keywords:** miniature mass spectrometer; continuous atmospheric pressure interface; ion trap; integrated instrument; robustness

## 1. Introduction

Owing to its high sensitivity, high specificity, rapid response, and applicability to a wide range of chemical compounds, mass spectrometry (MS) has become a powerful technique for chemical and biological analysis. By measuring the mass-to-charge ( $m/z$ ) ratio of ions, MS has capability of determining molecular weight and elucidating molecular structure. Traditionally, MS is usually performed in analytical laboratories by lab-based mass spectrometers, which are typically large in scale, time consuming, and complicated in operation for the procedures of sample analysis. However, the increasing demands of in situ analysis in many fields, such as environmental monitoring [1], public security [2,3], space exploration [4–9], and personal health care [10,11], call for the miniaturization of mass spectrometers.

To perform in situ analyses, especially some in harsh environments, a miniature mass spectrometer with portability is also expected to have adequate performances in terms of sensitivity and MS resolution, as well as robustness and repeatability [12,13]. Up until now, different kinds of miniature mass spectrometers have been developed, mainly using an ion trap as the mass analyzer, which is connected with different atmospheric pressure interfaces (APIs), including the membrane inlet (MI) [14–17], discontinuous atmospheric pressure interface (DAPI) [10,18,19], and continuous atmospheric pressure interface (CAPI) [20,21]. Among these APIs, MI was the first one to be adopted in MS systems. On the basis of MI, lots of miniature mass spectrometers have been developed and applied for in situ analysis of volatile samples. Nevertheless, the MI-based miniature mass spectrometers

cannot couple with ionization sources in atmospheric pressure environments, and thereby have no ability to analyze nonvolatile samples. To overcome this problem, DAPI was developed by Purdue University in 2008 [19], which enabled the coupling of ambient ionization sources with miniature mass spectrometers. By introducing ions produced in atmosphere into the vacuum chamber in a pulsed way, miniature mass spectrometers with DAPI could analyze both volatile and nonvolatile samples when coupling with the corresponding ionization sources [22–27]. As a robust ion introduction method with high stability for ion transfer, CAPI has been developed in lab-scale MS systems, but not in mini MS until 2015, when the first CAPI-based miniature mass spectrometer was developed by our group [20]. This miniature mass spectrometer utilizes a differential pumping system, as well as high-pressure ion trap operation. Taking advantage of CAPI, improvement of stability, robustness, and scan speed was achieved in this system. Following the development of mini MS with CAPI, an in-vacuum plasma ionization source [28] and miniature ion funnel [29] were then developed to enhance the analytical performance for in situ applications. With these techniques, for instance, limits of detection (LODs) of 10 ppbv and 50 ng/mL were obtained for both volatile and nonvolatile samples, respectively. Of course, the continuous atmospheric pressure interfaced mini MS can be equipped with two ionization sources in one instrument, an ambient ionization source and an in-vacuum ionization source, to alternately analyze both nonvolatile and volatile samples [30].

Based on mini MS with CAPI and related techniques developed previously, an upgraded instrument was developed in this work. With the aim of achieving smaller size, better performance and easier maintenance, many components of the system, including the vacuum manifold and electronics, were optimized and redesigned into a packaged unit. Configured with a tablet computer for system control, the mini MS has dimensions of 38 cm × 23 cm × 33 cm (length × width × height) and is 13 kg in total weight. The whole system is powered by an adapter with a power consumption of 200 watts in total. For analytical performances, good stability (signal relative standard deviation, RSD < 5%), high sensitivity (limit of detection, LOD 10 ng/mL), better than unit mass resolution, and broad mass range (from 150 Da to 2000 Da) were obtained. In addition, the miniature mass spectrometer is able to couple with different ambient ionization sources for complex sample analysis, together with tandem MS capability.

## 2. Experimental Section

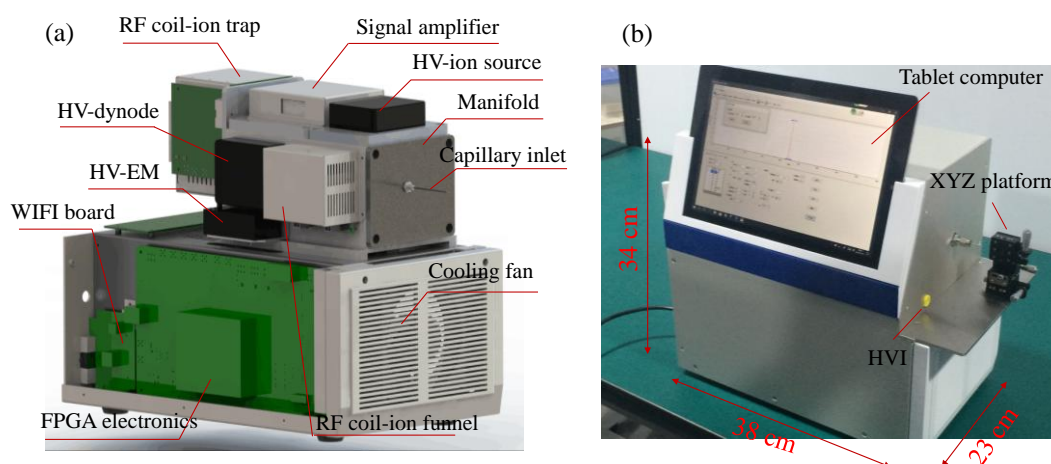
### 2.1. Instrumentation

Based on the miniature mass spectrometer with continuous atmospheric pressure interface developed previously in our lab, the instrument developed in this work also employed a CAPI. The miniature mass spectrometer had a two-stage vacuum chamber with differential vacuum pressures. The first chamber was connected to atmospheric pressure environment with a stainless-steel capillary (i.d. 0.25 mm), and a skimmer was placed between the first and second vacuum chamber. A miniature ion funnel was integrated in the first chamber as reported in detail previously [29]. A hyperbolic linear ion trap with dimensions of 4 × 4 mm (center to electrode distance) was placed in the second vacuum chamber, together with an ion detector, which consisted of an electron multiplier (EM, model 2500, Detector Technology Inc., Palmer, MA, USA) and a dynode assembled in a holder built in-house. To further improve the instrument performance mainly in terms of sensitivity and stability, a Hipace 80 (80 L/s, Pfeiffer Vacuum, Asslar, Germany) was used as the turbo pump, which has a pumping speed ~8 times that of the Hipace 10 used previously. The turbo pump was backed by a mechanical pump (50 L/min, Scroll Tech. Inc., Hangzhou, China). In addition, the length of the capillary was decreased from 20 cm to 10 cm, which brought an improvement in gas flow rate of ~2 times. Although gas flow rate of the current instrument was increased by using a shorter capillary, the background pressure in the ion trap region could be maintained at a lower value (~1 mTorr).

To minimize the dimensions and maximize the robustness, this miniature mass spectrometer was redesigned and integrated into a whole unit. As shown in Figure 1a, the structure of the

instrument could be separated into three parts, which were vacuum manifold with components inside, pumping system, and electronic control system. In the design, the vacuum manifold was pumped by two pumps located below it, and all signals needed were provided by the electronic control system, which included RF coils for ion trap and ion funnel, high voltage modules, control board. RF coils produced RF signals to drive the ion trap and the ion funnel, whose maximum output voltages were around  $2000 V_{0p}$  and  $500 V_{0p}$ , respectively. High DC voltages for EM, dynode and ionization source were provided by high voltage modules. All DC signals were provided and controlled by an electronics board based on a field-programmable gate array (FPGA). This circuit system could also generate AC signal ( $0\sim10 V_{0p}$ ) with stored waveform inverse Fourier transform (SWIFT) function to excite ions stored in the ion trap.

Figure 1b shows the photograph of the packaged miniature mass spectrometer, which has dimensions of  $38\text{ cm} \times 23\text{ cm} \times 34\text{ cm}$  (length  $\times$  width  $\times$  height). An outside high-voltage interface (HVI) was available, providing a high DC voltage ( $0\sim\pm5000\text{ V}$ ) that could be used for ionization sources. Using a cable connected with the HVI, the high voltage could be easily applied to the ionization sources. A tablet computer (X5, TECLAST, Guangzhou, China) was used as the control terminal, which could communicate with instrument control electronics either by a wireless method (wireless fidelity, WIFI) or a cable based on the TCP/IP network protocol. The overall instrument was 13 kg in weight, and power-supplied by an adapter with a power consumption of 200 watts in total.



**Figure 1.** 3D assembly (a); and photograph (b) of the integrated miniature mass spectrometer.

## 2.2. Chemical Samples

MRFA (Met-Arg-Phe-Ala), reserpine, DEET (*N,N*-Diethyl-3-methyl benzoyl amide), rhodamine b, PEG 600 (Polyethylene glycol 600), PEG 1500 (Polyethylene glycol 1500), and caffeine were all purchased from Sigma Aldrich (St. Louis, MO, USA). HPLC grade methanol was purchased from Fischer Chemical (Fair Lawn, NJ, USA), purified water was purchased from Wahaha Inc. (Hangzhou, China). All samples were diluted in methanol–water (1:1 *v/v*).

## 3. Results and Discussion

The goal of designing a miniature mass spectrometer is to expand the applications of MS and make MS more and more suitable for real-time and on-site analysis. The instrument further developed in this study represents an upgrade to the previous generations, and is expected to have better analytical performances, including stability and repeatability, sensitivity, MS resolution, mass range, and the capability to couple with different ambient ionization sources. As an overview, the previous generation of the CAPI mini MS have stability of signal RSD  $< 7\%$ , sensitivity of  $50\text{ ng/mL}$  for LOD, and resolution of better than unit.

### 3.1. Instrument Stability

Stability and robustness are important figure of merits for a MS system, especially for miniature mass spectrometers, which are expected to be used on site rather than in laboratory. Compared with MI and DAPI, a continuous atmospheric pressure interface has been proven to be a more suitable interface for miniature MS, with high stability and robustness. The stable pressure in vacuum chambers was expected to contribute to the stability of the instrument. To test the stability of the instrument developed in this study, MRFA with three different concentrations (1  $\mu\text{g/mL}$ , 5  $\mu\text{g/mL}$ , and 10  $\mu\text{g/mL}$ ) were used as samples. Ionized by a nano-ESI source, the ion currents of MRFA ( $m/z$  524) over 80 scan cycles were recorded as shown in Figure 2. The inset table in Figure 2 lists the relative standard deviations (RSDs) of the ion intensities over 80 scan cycles with respect with different concentrations. The result shows that the ion current is stable with a RSD of  $<5\%$ , which indicates that high stability was achieved by this mini MS.

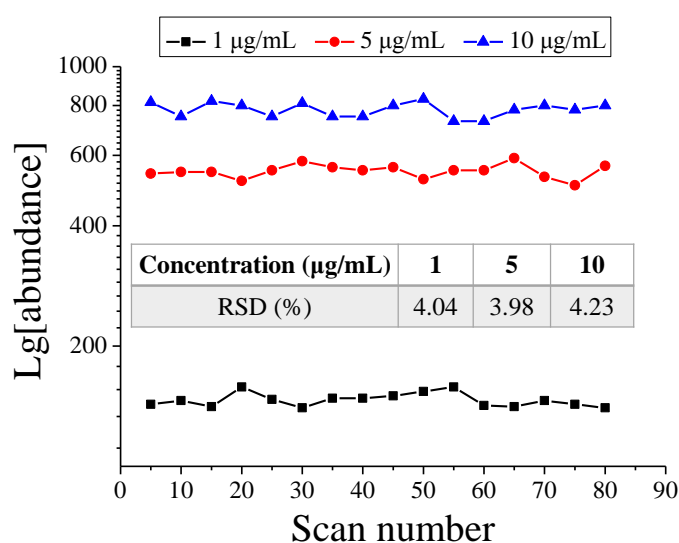
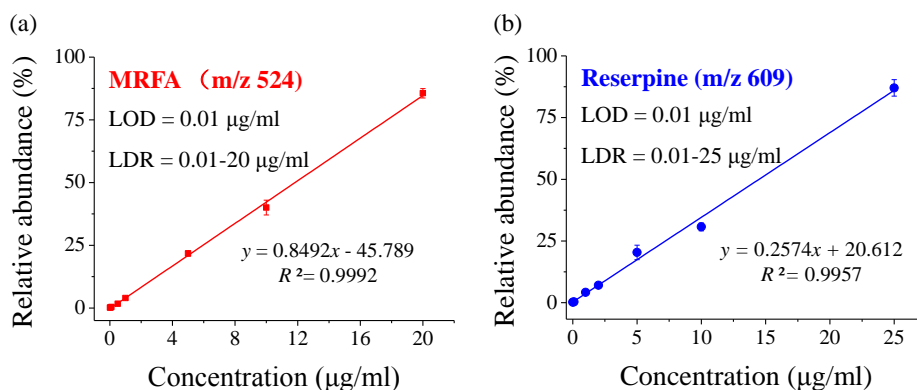


Figure 2. Abundances in log scale of MRFA ( $m/z$  524) over 80 MS scans.

### 3.2. Sensitivity

The sensitivity of the miniature mass spectrometer with a continuous atmospheric pressure interface is determined by many factors, including the gas flow rate through the capillary, and the ion transfer efficiency from the capillary exit to the ion trap. Different from previously developed miniature mass spectrometers with continuous atmospheric pressure interfaces, the instrument in this study used a turbo molecule pump Hipace 80 instead of the Hipace 10, which was able to lower the pressure in the ion trap region, while allowing a reduction in the length of the capillary from the 20 cm used previously to 10 cm, enhancing the gas flow rate through the capillary. In addition, a miniature ion funnel was also integrated into the mini MS system, which has been demonstrated to have capability of increasing the ion transfer efficiency by more than 10 times [29]. Therefore, the mini MS developed in this study was expected to have better performance in terms of sensitivity. To demonstrate the sensitivity of the instrument, experiments were performed using MRFA and reserpine as the testing samples, and nano-ESI as the ionization source. In experiments, the ionization time was kept to a constant value of 100 ms, and tandem MS was employed to isolate precursor ions for quantification. Figure 3 plots the calibration curves of MRFA (Figure 3a) and reserpine (Figure 3b). Limits of detection (LODs) of 0.01  $\mu\text{g/mL}$  were achieved for both samples, and linear dynamic ranges (LDRs) of 0.01–20  $\mu\text{g/mL}$  and 0.01–25  $\mu\text{g/mL}$  were obtained respectively for MRFA and reserpine with good linearities of  $R^2 > 0.99$ .

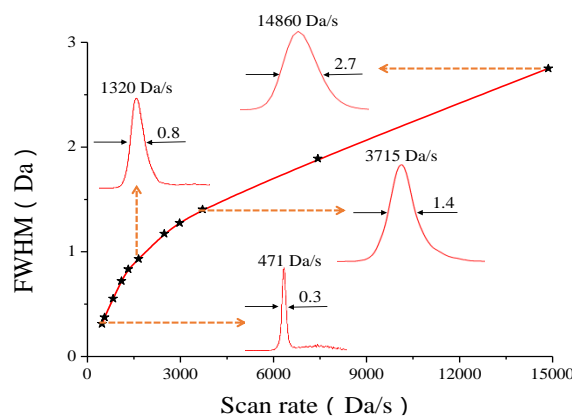


**Figure 3.** Linear dynamic ranges of MRFA (a) and reserpine (b).

### 3.3. MS Resolution

In general, MS resolution of an ion trap could be affected by buffer gas pressure in it. The presence of buffer gas is helpful for cooling ions towards the center of the ion trap, but at the same time, higher buffer gas pressures would broaden mass peaks in a mass spectrum, and cause degradation of MS resolution. To achieve an optimized MS resolution, the buffer gas pressure in an ion trap of a lab-scale mass spectrometer is normally kept below 1 mTorr. However, the pressure in the ion trap region of a miniature mass spectrometer is typically limited by the pumping capacity of the vacuum system. Especially for mini mass spectrometers with a CAPI, the background pressure in the ion trap region is relatively high, which results in reduced MS resolution. For example, a background pressure of 6 mTorr and 3 mTorr were achieved in the first and second generation of CAPI-interfaced miniature mass spectrometers, respectively.

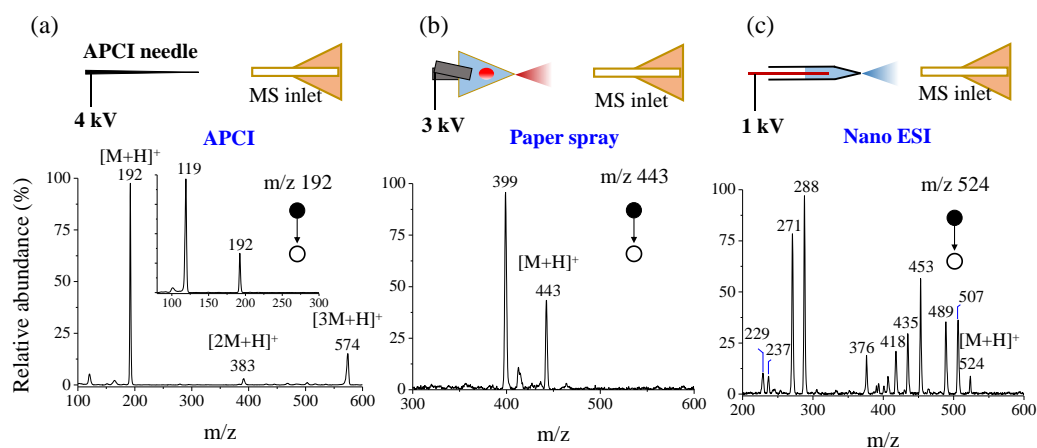
In this study, the buffer gas pressure in the ion trap region was further lowered to around 1 mTorr by using pumps with much higher pumping speeds, which should be beneficial for improving MS resolution. To characterize MS resolution, the full width at half maximum (FWHM) at different scan rates was recorded and plotted in Figure 4. MRFA ( $m/z$  524) ionized by a nano-ESI source was tested. FWHM of ~2.7 Da was obtained at a scan rate of 14,860 Da/s, and better than unit resolution ( $\text{FWHM} < 1$  Da) could be achieved when decreasing the MS scan rate below 1320 Da/s. The result verified that MS resolution could be improved by lowering the MS scan rate. As shown in Figure 4, a FWHM of 0.3 Da was achieved at a scan rate of 471 Da/s. In practice, the MS resolution of a mini MS, especially with high buffer gas pressures, could be optimized by adjusting the scan rate to meet practical requirements in different applications.



**Figure 4.** MS resolution (FWHM) at different scan rates using MRFA as sample ionized by nano-ESI.

### 3.4. Coupling with Different Ambient Ionization Sources

A significant feature of the miniature mass spectrometer with CAPI is the capability of coupling with different ambient ionization sources, which enables the rapid analysis of complex samples on site. To demonstrate this capability, APCI, paper spray and nano-ESI were coupled with the mini MS developed in this study to analyze different samples. In experiments, high voltages of 4 kV, 3 kV, and 1 kV were applied for APCI, paper spray, and nano-ESI, respectively. Figure 5 plots the mass spectra of different samples using the three ionization sources. These mass spectra were recorded at a scan rate of 7500 Da/s. As a volatile sample, DEET was chosen as the analyte for APCI, and DEET monomer ( $m/z$  192), dimer ( $m/z$  383) and tripolymer ( $m/z$  574) were all observed in its mass spectrum (Figure 5a). The inset of Figure 5a shows the tandem mass spectrum of  $m/z$  192. In terms of nonvolatile samples, rhodamine B (10  $\mu\text{g/mL}$ ) and MRFA (10  $\mu\text{g/mL}$ ) were analyzed by paper spray and nano-ESI, respectively. Tandem mass spectra of rhodamine B (Figure 5b) and MRFA (Figure 5c) were obtained. The tandem MS experiments were conducted by first isolating precursor ions using a SWIFT waveform, and then exciting and fragmenting precursor ions with a resonant AC signal.

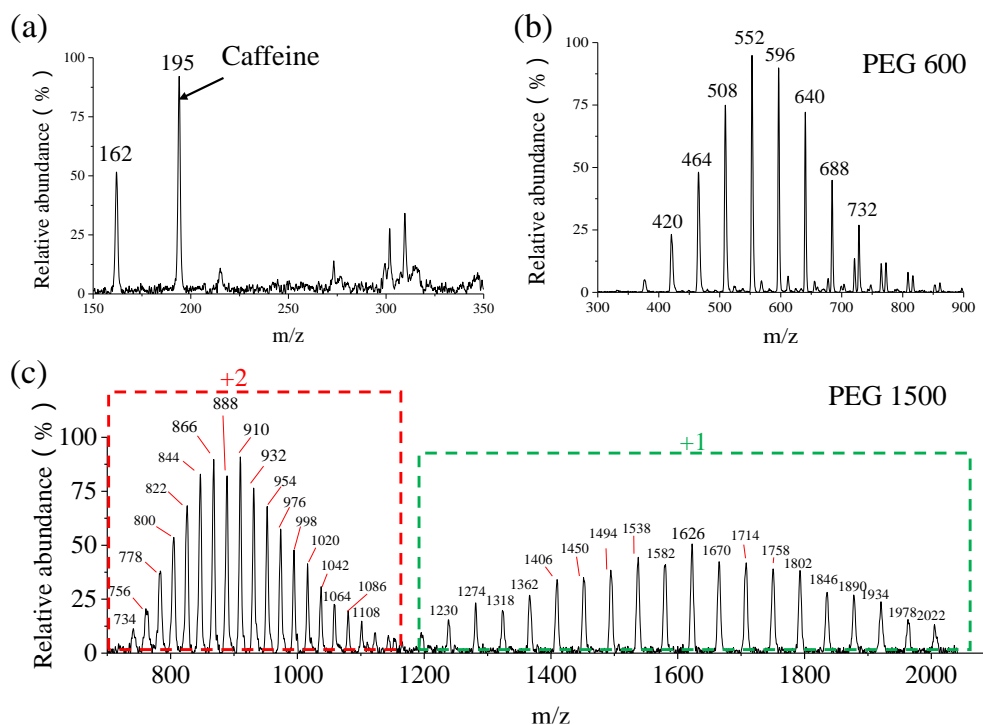


**Figure 5.** Mass spectra of (a) DEET; (b) rhodamine B; and (c) MRFA coupling with different ionization sources of APCI, paper spray and nano-ESI, respectively.

### 3.5. Mass Range

To meet the analytical requirements of different samples, a miniature mass spectrometer should have a mass range that is wide enough to cover ions with various mass over charges as much as possible. The mass range of an ion trap mass spectrometer is determined by many factors, including RF frequency, maximum RF amplitude, and AC frequency for resonance ejection. In this study, RF frequency was optimized at 930 kHz, and a mass range of 150–800 Da was obtained in the typical operation mode (dipolar resonance AC, 310 kHz). Figure 6a shows the mass spectrum of caffeine (10  $\mu\text{g/mL}$ ,  $m/z$  195). Figure 6b plots the mass spectrum of PEG 600 (nano-ESI, 10  $\mu\text{g/mL}$ ), which covers a mass range from 500 Da to 800 Da. To analyze larger molecules, mass range could be extended by lowering the frequency of the dipolar AC excitation signal. As shown in Figure 6c, the mass spectrum of PEG 1500 (nano-ESI, 10  $\mu\text{g/mL}$ ) was obtained when lowering the frequency of excitation AC to 160 kHz, at which the mass range of the instrument could be extended up to 2000 Da. Both singly charged and doubly charged ions were observed in the mass spectrum of PEG 1500.





**Figure 6.** Mass range of the miniature mass spectrometer. Mass spectra of (a) caffeine and (b) PEG 600 recorded at AC ejection frequency of 310 kHz; (c) Mass spectrum of PEG 1500 recorded at AC ejection frequency of 160 kHz.

#### 4. Conclusions

In this study, an upgraded and integrated miniature mass spectrometer with CAPI has been developed. With a power consumption of 200 W, the instrument has dimensions of 38 cm × 23 cm × 33 cm (length × width × height) and a weight of 13 kg in total. As demonstrated using different ionization sources, including APCI, nano-ESI, and paper spray, the mini MS has good robustness and repeatability (RSD < 5%) and high sensitivity (LOD = 10 ng/mL). MS resolution of better than unit could be achieved by adjusting MS scan rate. It has a mass range of 150–800 Da in normal scan mode, and could be extended up to 2000 Da by lowering the AC resonance frequency.

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**Author Contributions:** Wei Xu and Yanbing Zhai conceived and designed the experiments; Xiangzhi Meng performed the experiments; Xiaohua Zhang analyzed the data; Yanbing Zhai wrote the paper.

**Conflicts of Interest:** The authors declare no conflict of interest.

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