



## Abstract

In this study, we report on the development of a compact lamp-based vacuum ultraviolet (VUV) photoionization mass spectrometer (PIMS; hereafter referred to as VUV-PIMS) in our laboratory; it is composed of a radio frequency-powered VUV lamp, a VUV photoionizer, an ion-immigration region, and a reflection time-of-flight mass spectrometer. By utilizing the novel photoionizer consisting of a photoionization cavity and a VUV light baffle, extremely low background noise was obtained. An ultrasensitive detection limit ( $2\sigma$ ) of 3 pptv was achieved for benzene after an acquisition time of 10 s. To examine its potential for application in real-time sample monitoring, the developed VUV-PIMS was employed for the continuous measurement of urban air for six days in Beijing, China. Strong signals of trace-level volatile organic compounds such as benzene and its alkylated derivatives were observed in the mass spectra. These initial experimental results reveal that the instrument can be used for the online monitoring of trace-level species in the atmosphere.

## 1 Introduction

Volatile organic compounds (VOCs) are an important group of air pollutants: they are active in the formation of photochemical smog and ground-level ozone production; several VOCs present in urban air, such as benzene and its alkylated derivatives, are considered to be carcinogens (Gee and Sollars, 1998; Lee et al., 2005). The average volume fractions of VOCs, in addition to methane, in the atmosphere are generally less than  $1 \times 10^{-9}$  (ppbv) (Elsom, 1996; Schubert et al., 1999). Because of this extremely low VOC concentration, sample enrichment is often necessary prior to their analysis. During sampling or analysis, some active species might change because of oxidation by ozone (Jaouen et al., 1995) or other oxidizing agents, thereby decreasing the representativity of sampling. Meanwhile, conventional detection approaches such as gas chromatography-mass spectrometry and high-performance

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liquid chromatography-mass spectrometry involve a time-consuming chromatographic separation step (Muhlberger et al., 2002). For these reasons, it is imperative to develop real-time online monitoring instruments with high sensitivity for the detection of VOCs.

The proton-transfer-reaction mass spectrometer (PTR-MS) has been demonstrated for the real-time measurement of trace gases with limits of detection (LODs) in the low ppbv to high pptv range (Blake et al., 2004; Trefz et al., 2013). Thus, it is an extremely sensitive instrument. However, it suffers from a limitation in that it is highly sensitive to matrix effects, i.e. the chemical composition of the sampled analyte gas can influence the ionization efficiency of a specific target analyte. On the other hand, laser-based photoionization techniques including resonance-enhanced multiphoton ionization MS (Heger et al., 1999; Muhlberger et al., 2004) and single-photon ionization MS (Muhlberger et al., 2004; Tonokura et al., 2010) have also been applied for the online detection of VOCs at trace levels (ppbv/pptv). However, they suffer from limitations such as the use of expensive, bulky, and sophisticated laser systems to achieve nonlinear optical processes (Muhlberger et al., 2002). Meanwhile, the lamp-based VUV photoionization MS (VUV-PIMS) is another type of an instrument that has been attracting significant attention (Muhlberger et al., 2005a). Muhlberger et al. (2002, 2005a, b, 2007) have developed a series of compact VUV-PIMSs with an electron-beam-pumped rare gas excimer VUV lamp. There is an improvement in the LOD for benzene, toluene, and xylene from ppmv to tens of ppbv. Meanwhile, Hua et al. (2011) have designed a VUV-MS based on a commercial krypton lamp, exhibiting both single-photon ionization (SPI) and chemical ionization capabilities. The LODs observed for benzene, toluene, and xylene are as low as 3, 4, and 6 ppbv, respectively, in the SPI mode. Meanwhile, Kuribayashi et al. (2005) have developed a VUV-PI-ion trap-MS with a microwave-excited VUV lamp and observed an LOD of 10 pptv for trichlorobenzene after an acquisition time of 18 s.

In this paper, we report the design of an ultra-sensitive compact VUV-PIMS, which was employed to measure the LOD of trace-level VOCs such as benzene. An extremely low noise level and excellent detection sensitivity were observed. The instrument de-

sign, calibration results, and urban air measurements will be discussed in the following sections.

## 2 Experimental section

### 2.1 Instrumentation

5 Figure 1a shows the laboratory-built compact VUV-PIMS. Its main components include a radio frequency-powered (RF) VUV lamp, a VUV photoionizer, an ion-immigration region, and a reflection time-of-flight MS (TOF-MS).

10 The VUV lamp, which is constructed in our laboratory, is a krypton lamp with an output of  $\sim 5 \times 10^{14}$  photons  $s^{-1}$  at 123.9 nm. Its structure has been reported in detail by Shu et al. (2013). A plano-convex MgF<sub>2</sub> lens ( $f = 50$  mm) is used as a window for the passage of the VUV light, which is generated by the lamp, into the photoionizer.

15 Figure 1b and c shows the photoionizers A and B, respectively, used in the test, which are made of stainless steel. They are interchangeable in the corresponding position of the VUV-PIMS. Photoionizer A is a straight channel with a length of 30 mm and an internal diameter of 6 mm. On the other hand, photoionizer B is designed with an optical baffle for preventing VUV light from entering the ion-immigration region. The diameter of the VUV light baffle is  $\sim 2$  mm. The exit orifice of photoionizer B is 1.5 mm in diameter. First, the sample is introduced into the photoionizer via a stainless steel tube (1/8 inch outer diameter; OD) using a needle valve. Moreover, the signal intensity is optimized by controlling the sample flow by adjusting the needle valve. The pressure in the photoionizer is not measured directly but indirectly by the pressure of the foreline measured using a convectron gauge.

20 Meanwhile, the ion-immigration region contains seven ion-immigration optics, which focus the ions ejected from the photoionizer into a narrow beam between the back plate and the extraction grid of the reflection MS. To obtain more strong ion beam, the

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excitation tube of the VUV lamp, the photoionizer, and the ion-immigration optics are collimated.

A V-shaped laboratory-built reflection MS was employed, which has a total field-free flight distance of 460 mm, an extraction grid, an ion mirror, and a chevron multichannel plate detector. The relatively short flight distance of 460 mm is selected to make the instrument compact; hence, the highest repetition rates are obtained for the best LODs. The path of the ion flight is orthogonal to the axis of the ion-immigration optics. The generated ions are extracted with a pulsed electric field of 600 V in the extraction grid. The voltage of the acceleration field is 1800 V. The chevron microchannel plates used to detect ions are biased at 2100 V. The signal is amplified using a 100× amplifier (Ortec VT120C) and recorded with a TOF multiscaler (FAST Comtec, P7888). The acquisition time for each mass spectrum is 10 s.

The three-stage differential pumping system is composed of three chambers: a source chamber, differential chamber, and detection chamber. These chambers are pumped by a 300, 200, and 300 Ls<sup>-1</sup> turbo molecular pumps (TMPs), respectively. The first two TMPs are backed by a rotary pump (8.3 Ls<sup>-1</sup>). Meanwhile, the outlet of the third TMP joins the source chamber. Furthermore, during sampling, the pressure of the detection chamber is  $\sim 3 \times 10^{-3}$  Pa.

## 2.2 Sample preparation

Benzene samples of different concentrations were prepared by triple dilutions. First, pure benzene was diluted 4.1-fold with dichloromethane; then 0.1 mL of this diluted solution was injected into a 6.35 L narrow-mouthed bottle filled with synthetic air (80 % N<sub>2</sub> + 20 % O<sub>2</sub>), which was prepared by two mass flow meters using high-purity nitrogen and oxygen; finally, the desired amount of gases in the bottle was extracted using a syringe and added into calibration containers.

Two calibration containers were used in this experiment: a smog chamber of approximately 120 L and a 0.24 L cylinder, respectively. The former is composed of a thin-walled open-head stainless steel drum and a thin Tedlar polyvinyl fluoride film bag,

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observed at an  $m/z$  of 28 ( $\sim 2 \times 10^5$  counts) and 32 ( $\sim 3 \times 10^5$  counts), which correspond to the background ions of  $\text{N}_2^+$  and  $\text{O}_2^+$ , respectively.  $\text{N}_2$  (Ionization Potential = 15.6 eV) and  $\text{O}_2$  (Ionization Potential = 12.1 eV) are not ionized by 10.03 eV VUV photons emitted from the krypton lamp (Shu et al., 2013), but they can be ionized by the bombardment of accelerated light-induced electrons (photoelectron ionization, PEI) (Muhlberger et al., 2001; Gamez et al., 2008). The photoelectrons are generated from metal ion-immigration optics under illumination of VUV light (photoelectric effect) and accelerated by the static electric field of ion-immigration optics. The highest DC voltage biased to the ion-immigration optics is 38 V. Because of the existence of the strong background ions, the baseline level was as high as  $263.6 \pm 15.7$  counts. Furthermore, in the inset of Fig. 2b, a mass peak is observed at an  $m/z$  of 78, corresponds to the molecular ion of benzene. The mass resolution at an  $m/z$  of 78 is  $\sim 200$ . Meanwhile, small mass peaks are observed at  $m/z$  values of 16, 18, 19, and 44. These peaks correspond to  $\text{O}^+$ ,  $\text{H}_2\text{O}^+$ ,  $\text{H}_3\text{O}^+$ , and  $\text{CO}_2^+$ , respectively, which are also generated by PEI. Moreover, other small mass peaks are observed at  $m/z$  values of 29, 34, etc., possibly attributed to the impurities present in synthetic air.

Figure 2c and d shows the mass spectra of 0 and 8.6 ppbv benzene in synthetic air, respectively, recorded using photoionizer B. Compared with the mass spectra in Fig. 2a and b, those in Fig. 2c and d exhibits a decrease in the intensities of  $\text{N}_2^+$  and  $\text{O}_2^+$  mass peaks by 3 orders of magnitude. This decrease is attributed to the fact that the optical baffle of photoionizer B prevents VUV light from entering the ion-immigration region; as a result, the formation of  $\text{N}_2^+$  and  $\text{O}_2^+$  is significantly suppressed. As a result, the baseline level decreases from  $263.6 \pm 15.7$  counts (Fig. 2a) to  $4.1 \pm 1.8$  counts (Fig. 2c), which corresponds to an 8.7-fold improvement in the standard deviation of the noise at an  $m/z$  of 78. On the other hand, the mass signal intensity for benzene observed in Fig. 2d is slightly higher than that observed in Fig. 2b, with the same mass resolution obtained in both cases. Such an enhancement in the photoionization efficiency possibly originates from the reflectance of VUV light by the optical baffle in photoionizer B (Zhu et al., 2014). In addition, as can be seen in Fig. 2d, relatively strong mass peaks are





resonance-enhanced multiphoton ionization MS designed by Heger et al. (1999) and the widely used PTR-MS designed by Trefz et al. (2013).

### 3.3 Atmospheric measurements

To further demonstrate the utility of the developed MS in field measurements, real-time measurements of urban air were conducted for 6 days between 7 and 12 March 2015. Air from outside our laboratory building (Beijing, China) was continuously sampled into the VUV-PIMS. The operating parameters were the same as those employed in the benzene calibration study. Spectra were recorded every 3 min.

Figure 4 shows the VUV-PIMS spectrum recorded at 00:14 (China Standard Time) on 7 March when the intensity of the benzene peak was the maximum during measurement. In the spectrum, a series of strong mass peaks were observed at  $m/z$  values of 19, 37, 55, 73, and 91, corresponding to  $\text{H}_3\text{O}^+$ ,  $(\text{H}_2\text{O})_2\text{H}^+$ ,  $(\text{H}_2\text{O})_3^+$ ,  $(\text{H}_2\text{O})_4\text{H}^+$ , and  $(\text{H}_2\text{O})_5\text{H}^+$ , respectively. Mass peaks were also observed at  $m/z$  values of 43, 47, 61, 69, 78, 92, 106, 120, and 128, corresponding to VOCs. Based on the measurement results of atmospheric VOCs by GC-MS in the same area from 2000 to 2005 (Jiang, 2006), the mass peaks at  $m/z$  values of 78, 92, 106, 120 and 128 can be attributed to benzene, toluene, xylene/ethylbenzene,  $\text{C}_3$ -alkylated benzene derivatives and naphthalene, respectively. In urban environments, benzene, its alkylated derivatives and naphthalene mainly originate from automotive exhaust. They are potential carcinogens and are involved in photochemical reactions (Lee et al., 2005). The mass peaks at  $m/z$  43, 47, 61, and 69 are speculated to be protonated VOCs because of the presence of large amount of  $\text{H}_3\text{O}^+$  observed in the mass spectrum. Atmospheric formic acid, acetic acid, and isoprene may form these ions via proton-transfer reaction (de Gouw and Warneke, 2007). The presence of protonated water and water clusters complicates the mass spectrum. In the future, this instrument will be modified such that these interfering species can be eliminated by decreasing the bias voltage between the VUV lamp and the photoionizer.

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Figure 5a–d shows the variations in the concentrations of benzene, toluene, xylene/ethylbenzene, and C<sub>3</sub>-alkylated benzene derivatives by continuous monitoring by the VUV-PIMS, respectively, and Fig. 5e shows that in the speeds of wind real-time measured by a meteorological station with a distance of ~ 200 m from our laboratory for 6 days. The concentration variations of benzene and its derivatives have a good simultaneity. However, they show a remarkable opposite trend to the wind speeds. These interesting observation results reveal that the wind can remove effectively the airborne pollutants. The concentrations of benzene (*m/z* 78) were quantified by the abovementioned calibration and was found to range between 0.1 and 3.2 ppbv, which is consistent with the reported average level of the benzene concentration in the same area from 2000 to 2005 (4.0 ppbv) (Jiang, 2006).

## 4 Conclusions

A compact lamp-based VUV-PIMS was developed in our laboratory. The photoionizer with an optical baffle significantly reduced the background noise. An ultrasensitive LOD of 3 pptv was achieved for benzene. The PIMS developed herein exhibited better sensitivity for VOCs as compared to that of VUV photoionization MSs reported previously (Kuribayashi et al., 2005; Muhlberger et al., 2005a, b, 2007). A satisfactory linear response ( $R^2 = 0.9968$ ) was obtained. The initial atmospheric measurement demonstrates that the instrument can be used for the real-time monitoring of trace-level VOCs in the atmosphere.

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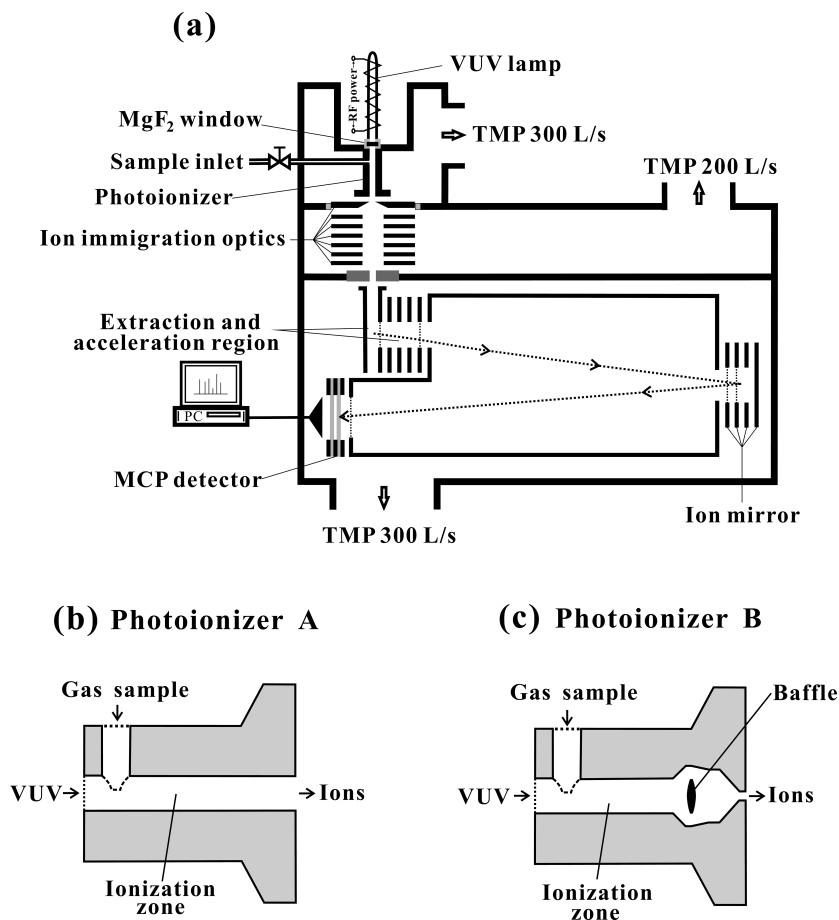
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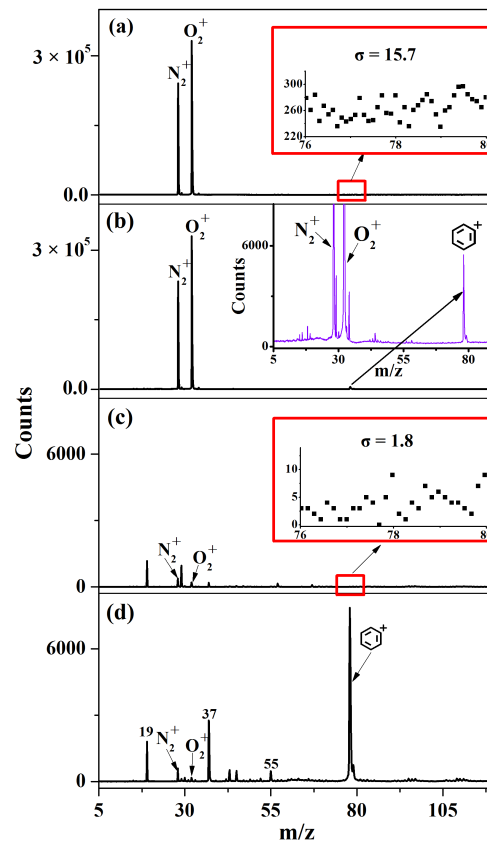


**Figure 1.** Schematic of the VUV-PIMS **(a)**, Photoionizer A **(b)**, and Photoionizer B **(c)**. TMP: turbomolecular pump.

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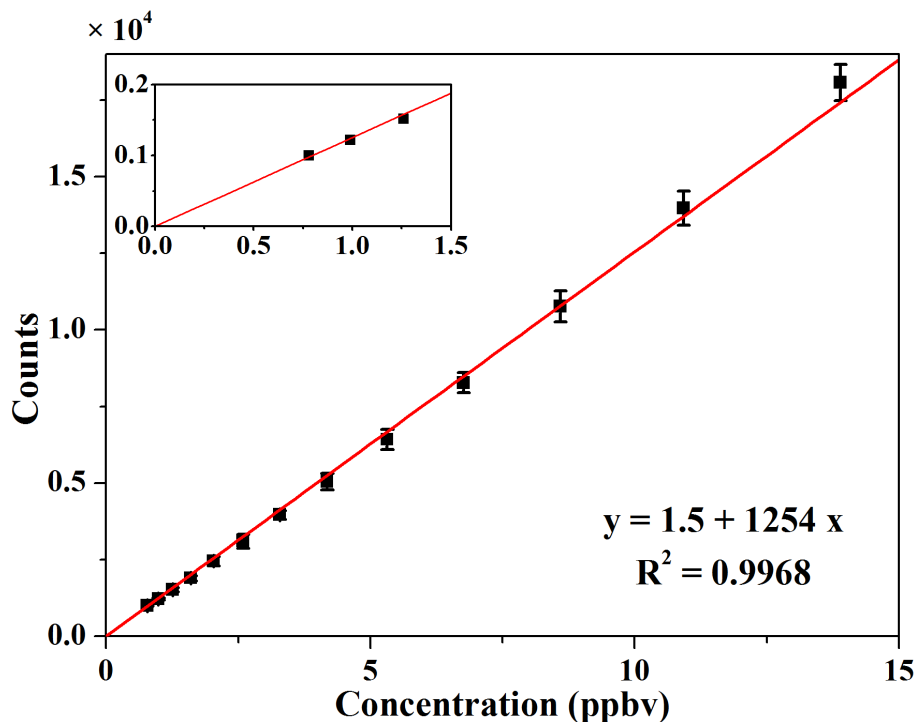


**Figure 2.** Mass spectra of synthetic air (a) and 8.6 ppbv benzene (b) obtained with photoionizer A. Mass spectra of synthetic air (c) and 8.6 ppbv benzene (d) obtained with photoionizer B. The acquisition time for each mass spectrum is 10 s. The insets in (a) and (c) show the magnified baselines of the mass spectra at an  $m/z$  of 78. The inset in (b) shows the magnified benzene mass peak.

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**Figure 3.** Signal intensities vs. concentrations of benzene measured by the VUV-PIMS equipped with photoionizer B. Black squares represent the data points, and the line represents the result from linear fitting.

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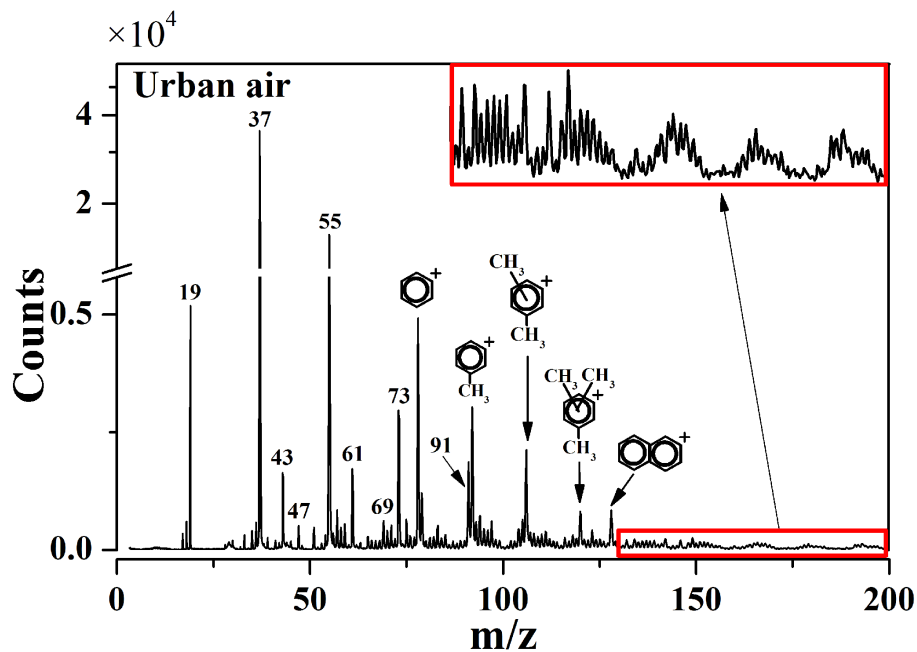
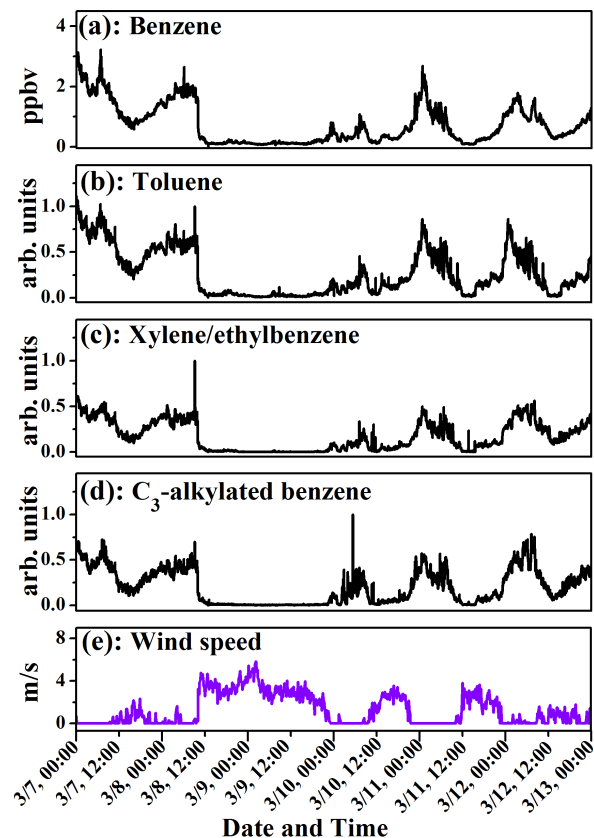


Figure 4. Mass spectrum of outdoor air obtained using the VUV-PIMS at 00:14 on 7 March.

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**Figure 5.** Outdoor measurements for 6 days: concentration variations of benzene (a), toluene (b), xylene/ethylbenzene (c), and C<sub>3</sub>-alkylated benzene derivatives (d) monitored by the VUV-PIMS. The data of wind speeds (e) were obtained from the Beijing Urban Ecosystem Research Station.

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