https://doi.org/10.5194/egusphere-2025-2106
Preprint. Discussion started: 29 July 2025

Measurement Report: Collocated speciation and potential mechanisms of gaseous adsorption for integrated filter-based sampling and analysis of water-soluble organic molecular markers in the atmosphere

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https://doi.org/10.5194/egusphere-2025-2106
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Abstract

To better understand the measurement uncertainties and sampling artifacts of particulate water-soluble organic molecular markers (WSOMMs), three quartz filters were stacked and installed in two collocated samplers (Sampler I and II) to simultaneously collect ambient WSOMMs. The paired top filters (Q_f) loaded with $PM_{2.5}$ were analyzed to determine the duplicate-derived uncertainty of particulate WSOMM concentrations. For several WSOMMs (e.g., levoglucosan) specifically associated with aerosol sources, the uncertainty was well below 20%, which was commonly assumed in previous studies for the analysis of particulate WSOMMs. If the WSOMMs detected in the other two filters (Q_b and Q_{bb}) below Q_f were caused by gaseous adsorption, the breakthrough value ($[Q_{bb}]/([Q_b]+[Q_{bb}])$) can be used to estimate the sampling artifact of particulate WSOMMs due to gaseous adsorption on Q_f. To understand the influence of acidic and alkaline conditions on the adsorption of gaseous WSOMMs or their precursors on quartz filters, the bottom filter (Q_{bb}) of Sampler I was treated with $(NH_4)_2SO_4$ or KOH on different sampling days. From the comparison of the measurement results between chemically treated and untreated Q_{bb} samples, it was inferred that $(NH_4)_2SO_4$ can increase the formation of isoprene secondary organic aerosol by reactive uptake of the oxidative intermediates; KOH can promote the adsorption of organic acids through neutralization reactions. Future studies are warranted to develop a suitable method for sampling gaseous WSOMMs using chemically treated adsorbents.

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1. Introduction

As a major component of atmospheric aerosols, water-soluble organic carbon (WSOC) can influence aerosol radiative forcing through absorbing and scattering solar and terrestrial radiation (Malm et al., 1996; Ming et al., 2005) and promoting cloud formation by acting as cloud condensation nuclei and ice-nucleating particles (Novakov and Penner, 1993; Chen et al., 2021). Moreover, the deposition of WSOC provides nutrients for plants and microorganisms on Earth that maintain the balance of the ecosystem (Quinn et al., 2010; Iavorivska et al., 2017; Goll et al., 2023). The heavy metals and toxic organics associated with WSOC also increase the health risks of atmospheric aerosols (Tao and Lin, 2000). WSOC can be released directly by biomass burning (Ding et al., 2013; Du et al., 2014) or can be formed by the atmospheric oxidation of volatile organic precursors and subsequent gas-particle partitioning processes (termed “secondary organic aerosol”, SOA) (Zhang et al., 2007; Kroll and Seinfeld, 2008). Water-soluble organic molecular makers (WSOMMs) are organic compounds with specific origins in the atmosphere and are commonly used to identify the sources of WSOC and particulate matter (PM). In laboratory studies where SOA formation was simulated using a smoke chamber, WSOMMs play a central role in revealing the reaction pathways (Kroll et al., 2006; Ng et al., 2008).

A comprehensive understanding of the physicochemical properties, atmospheric transformation and environmental impacts of WSOC depends largely on its characterization (Noziere et al., 2015). Uncertainty analysis for the quantification of PM components, including WSOC and WSOMMs, is necessary to show the variability of measurement results due to sampling, pretreatment, instrumental analysis, etc (Zhang et al., 2024). The uncertainty data are also needed when the simulation results of atmospheric transport models, e.g. for predicting the spatiotemporal distribution of

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PM components and SOA formation, are evaluated by comparison with measurements (Aleksankina et al., 2019). For PM species with high measurement uncertainty, modeling could aim to obtain a reasonable range instead of a specific value. In addition, the uncertainty data are required for source apportionment using receptor models (Kim and Hopke, 2007). In existing studies, propagation methods (e.g., root sum of squares) have been used to predict the overall uncertainty of the system from different sources of uncertainty (Jaeckels et al., 2007; Dutton et al., 2009b; Feng et al., 2023b). Another method to estimate the uncertainty is to conduct repeated analysis for selected samples (Xie et al., 2017), which only considers the error during chemical analysis. The total uncertainty for the characterization of atmospheric composition is composed of the uncertainties in both sampling and chemical analysis, and can be directly determined by performing collocated sampling. This method has been applied to estimate the concentration uncertainties of bulk PM components (Dutton et al., 2009a; Yang et al., 2021; Xie et al., 2022b), but the duplicate-derived uncertainty for the characterization of WSOMM has rarely been investigated.

The known WSOMMs (e.g., 2-methyltetrols) are mostly semi-volatile organic compounds (SVOCs), in which a mass transfer always takes place between the gas and particle phase (Yatavelli et al., 2014; Xie et al., 2014b). In filter-based sampling of WSOMMs in the particle phase, the adsorption of gaseous WSOMMs on filters ("blow on" effect, positive artifact) leads to an overestimation of particle-phase concentrations (Hart and Pankow, 1994; Mader and Pankow, 2001b; Subramanian et al., 2004). Several studies have used a denuder to eliminate organic gasses in the air stream prior to sampling PM on filters (Eatough et al., 2003; Fan et al., 2004; Subramanian et al., 2004), which creates a large potential for volatilization ("blow off" effect, negative artifact) of particulate organic matter (OM) due to the disruption of the gas-particle equilibrium

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(Subramanian et al., 2004; Watson et al., 2009). The use of a backup quartz filter downstream of the PM-loaded quartz filter or a Teflon filter has been used in many studies to correct for adsorption of gaseous organics, with the target species being mostly bulk organic carbon (OC) (Watson and Chow, 2002; Subramanian et al., 2004, 2009) and non-polar organic compounds (e.g., *n*-alkanes and polycyclic aromatics) (Mader and Pankow, 2001a; Xie et al., 2014a), while sampling artifacts of WSOMMs were less considered.

The existence of gaseous WSOMMs has been reported by integrated gas-particle (G-P) sampling (Limbeck et al., 2005; Bao et al., 2012; Liu et al., 2012; Shen et al., 2018) or online measurements (Williams et al., 2010; Xu et al., 2019; Lv et al., 2022a, 2022b). Polyurethane foam (PUF) was the most commonly used adsorbent for sampling gaseous WSOMMs in offline observations (Xie et al., 2014b; Shen et al., 2020; Lanzafame et al., 2021; Qin et al., 2021). However, the extraction process could be affected by the leaching of the PUF material in methanol, leading to low recoveries (approximately 50%). To prove that methacrylic acid epoxide (MAE) is the key intermediate for the formation of 2-methylglyceric acid (2-MG) from isoprene under high NO_X conditions, Lin et al. (2013b) collected gaseous MAE using an ice-cooled glass bubbler filled with ethyl acetate. Due to the limited flow rate and absorption efficiency, this liquid absorption method was more suitable for qualitative rather than quantitative purposes. The Semi-Volatile Thermal Desorption Aerosol Gas chromatograph (SV-TAG) was developed for hourly measurements of WSOMMs in the gas and particle phase. In the SV-TAG, a parallel thermal desorption cell equipped with passivated high-surface-area stainless steel (SS) fiber filters (F-CTD) was used for sampling (Williams et al., 2010; Zhao et al., 2013a; 2013b; Isaacman et al., 2014, 2016). One F-CTD was used to directly collect WSOMMs in both the gas and particle phases,

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while the other cell was set up to collect only WSOMMs in the particle phase by passing the sample air through an upstream activated carbon denuder. Comparisons between the two cells directly reflected the G-P partitioning of the WSOMMs. However, the resulting particulate fraction (*F*%) was often greater than 100% (Isaacman et al., 2016; Liang et al., 2023), possibly due to the uncertainties associated with the small sampling volume and chemical analysis.

In this study, three quartz filters were stacked and installed in two collocated samplers for sampling WSOMMs. The measurement results of WSOMMs on the top filter were used to estimate the uncertainties of analyzing WSOMMs in the particle phase. The remaining two bare quartz filters in one sampler were analyzed to assess positive sampling artifacts due to adsorption of gaseous WSOMMs or their precursors. To investigate the impacts of acidic and alkaline conditions on the adsorption on quartz filters, the bottom filter of the other sampler was soaked in ammonium sulfate ($(NH_4)_2SO_4$) or potassium hydroxide (KOH) and dried before sampling. The study results unveil the uncertainties in the characterization of WSOMMs in the particle phase, and are beneficial for further studies on sampling and analysis of gaseous WSOMMs.

2. Methods

2.1 Sampling

All filter samples were collected on the rooftop of a six-story building (Binjiang Building) of Nanjing University of Information Science and Technology (NUIST, 32.21°N, 118.71°E). The sampling site is located in a suburb in the western Yangtze River Delta of China (Figure 1a), approximately 20 km north of the city center of Nanjing. The inter-provincial highway G40 and Jiangbei expressway are located about 700 m and 1.5 km northwest and southeast, respectively. The petrochemical industry of Yangzi and the chemical industry of Nanjing (SINOPEC) are located 5 – 10 km

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northeast of the site. The surrounding area consists mainly of residential buildings, road traffic, and parks (e.g., the Longwangshan scenic area).

Three quartz filters (20.3 cm × 12.6 cm, Munktell Filter AB, Sweden) were stacked and placed on each of the two identical samplers (Sampler I and II; Mingye Environmental, Guangzhou, China) to collect ambient air at a flow rate of 300 L min^{-1}. All filters were pre-baked at 550°C for 4 h to remove potential organic contaminants. Twenty-four pairs of collocated samples were collected from August to September 2021 during daytime (08:00 – 19:00 GMT+8, $N = 12$) and nighttime (20:00 – 07:00 the next day, GMT+8, $N = 12$). As shown in Figure 1b, the top filter (Q_f) in each filter was loaded with $PM_{2.5}$, and the subsequent two filters (Q_b and Q_{bb}) were used to evaluate the adsorption of gaseous WSOMMs or their precursors on filters. In Sampler I, Q_{bb} was soaked in 1 M $(NH_4)_2SO_4$ ($N = 12$) or 1 M KOH ($N = 12$) and dried at a temperature of 120°C before sampling, while Q_{bb} in Sampler II was not treated with chemicals. Table S1 shows the sampling date, mean temperature and relative humidity (RH, %), and the type of Q_{bb} treatment ($(NH_4)_2SO_4$, KOH, and no treatment) of Sampler I and II. Field blanks were taken at every 10^{th} sample to correct for possible contamination. All samples and field blanks were sealed and stored at –20°C until analysis.

2.2 Chemical analysis

The method of analysis for WSOMMs in filter samples has been detailly described in our previous work (Qin et al., 2021; Feng et al., 2023a, 2023b). Briefly, one-eighth of each filter sample was spiked with 40 μL of deuterated internal standards (IS, succinic acid-d4, levoglucosan-d7, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d10, and perylene-d12; 10 ng μL^{-1}) and ultrasonically extracted twice for 15 min in a mixture of methanol and dichloromethane (v:v, 1:1). The total extract of each sample was then rotary evaporated and blown to dryness with a gentle stream

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of N_2. 60 μL of derivatization reagent [N, O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) with 1% trimethylchlorosilane (TMCS) and pyridine, 5:1] was added and reacted with the dried extracts at 70°C for 3 hours. Prior to instrumental analysis by gas chromatography (GC, Agilent 7890B, USA)-mass spectrometry (MS, Agilent-5977B, USA), the extract solution was cooled to room temperature and diluted with 340 μL of pure hexane. Quantification of the individual WSOMMs was performed by generating six-point calibration curves and the IS method.

Water-soluble inorganic ions and WSOC in filter samples were extracted with ultrapure water (18.2 MΩ). Cations (NH_4^+, K^+, Ca^{2+} and Mg^{2+}) and anions (SO_4^{2-} and NO_3^-) were determined using Metrohm (930, Switzerland) and Dionex (ICS-3000, USA) ion chromatography (IC), respectively. WSOC was analyzed using a total organic carbon analyzer (TOC-L, Shimadzu, Japan). Bulk OC and elemental carbon (EC) of the filter samples were measured using a thermal-optical carbon analyzer (DRI, 2001A, Atmoslytic, USA) according to the IMPROVE-A protocol. Field blanks were analyzed in the same way as the air samples, and the measurement results of all filter samples were corrected.

2.3 Data analysis

2.3.1 Breakthrough calculation

When Q_b and Q_{bb} were considered as adsorbents for sampling gaseous WSOMMs, the WSOMM concentrations in the Q_b and Q_{bb} samples can be used to calculate the breakthrough (B), which represents the sampling efficiency and is defined as follows:

$$B = \frac{[Q_{bb}]}{[Q_b]+[Q_{bb}]} \times 100\% \quad (1)$$

where $[Q_b]$ and $[Q_{bb}]$ represent the concentrations of each target compound in Q_b and Q_{bb} samples, respectively. A B value of 33% has been commonly used as a threshold for excessive breakthrough, and a B value of close to or higher than 50% indicates

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complete breakthrough (Peters et al., 2000).

2.3.2 Calculation of the particulate fractions of WSOMMs

Assuming that the target WSOMMs measured in the Q_f samples exist in the particle phase, and those detected in the Q_b and Q_{bb} samples are present in the gas phase, the particulate fractions (F%) of the individual WSOMMs can be calculated as follows:

$$F\% = \frac{[Q_f]}{[Q_f]+[Q_b]+[Q_{bb}]} \times 100\% \quad (2)$$

where $[Q_f]$ denotes the concentrations of the target compound in Q_f samples.

2.3.3 Uncertainty assessment

The coefficient of divergence (COD) has often been used as a measure of the similarity of chemical species concentrations between pairs of PM samples (Wilson et al., 2005) and is defined as follows:

$$\mathrm{COD} = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\left(\frac{x_{i1}-x_{i2}}{x_{i1}+x_{i2}}\right)^2} \quad (3)$$

where x_{i1} and x_{i2} in this work are the concentrations of a particular WSOMM in the i^{th} pair of Q_f samples from Sampler I and Sampler II, respectively, and n is the number of sample pairs. Values of COD approaching 0 and 1 indicate identity and complete divergence between pairs of collocated samples.

The standard deviation of paired differences (SD_{diff}) and average relative percent difference (ARPD) were used to quantify the absolute and relative uncertainties of individual WSOMMs based on collocated measurement data (Flanagan et al., 2006; Dutton et al., 2009a; Yang et al., 2021). They were calculated as follows:

$$\mathrm{SD}_{\mathrm{diff}} = \sqrt{\frac{1}{2n}\sum_{i=1}^{n}(x_{i1}-x_{i2})^2} \quad (4)$$

$$\mathrm{ARPD} = \frac{2}{n}\sum_{i=1}^{n}\frac{|x_{i1}-x_{i2}|}{(x_{i1}+x_{i2})} \times 100\% \quad (5)$$

3 Results and discussion

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3.1 Collocated measurements of Q_f samples

3.1.1 Overview of the measurement data

The mean concentrations of WSOMMs and bulk $PM_{2.5}$ components in collocated Q_f samples are summarized in Tables 1 and S2, respectively. Generally, all species showed similar mean concentrations between paired Q_f samples with no significant difference (Student's *t* test, $p = 0.55 – 0.96$). Among the isoprene SOA tracers, the mean concentration of 2-methylglyceric acid (2-MG, 4.48 ± 3.15 ng m^{-3}) was comparable to observations at the same site in summer 2019 (3.62 ± 1.38 ng m^{-3}) and summer 2020 (4.71 ± 1.77 ng m^{-3}) (Feng et al., 2023b). However, the mean concentrations of 2-methyltetrols (2-MTs, 13.1 ± 7.00 ng m^{-3}) and C_5-alkene triols (C_5-ATs, 15.6 ± 14.7 ng m^{-3}) were significantly ($p < 0.01$) lower than in summer 2019 (21.3 ± 18.2 ng m^{-3}, 21.3 ± 26.9 ng m^{-3}) and summer 2020 (27.0 ± 21.6 ng m^{-3}, 36.3 ± 48.0 ng m^{-3}). After the implementation of a series of air pollution control measures in China after 2013 (e.g., the "Air Pollution Prevention and Control Action Plan"), an annual decrease in sulfate concentrations was observed in Nanjing (Xie et al., 2022a). As shown in Table S2, the mean sulfate concentration in this study (5.82 ± 2.07 ng m^{-3}) is lower than in summer 2019 (8.92 ± 3.25 ng m^{-3}) and summer 2020 (7.67 ± 2.92 ng m^{-3}) (Feng et al., 2023b). Since sulfate participates in the reactive uptake of isoprene SOA intermediates to form C_5-ATs, 2-MTs, and hydroxy sulfate esters (Surratt et al., 2007a; 2010), the decrease in sulfate concentrations is a possible reason for the attenuation of isoprene SOA formation (Worton et al., 2013; Lin et al., 2013a; Xu et al., 2015). The concentrations of the primary WSOMMs, including biomass burning tracers, saccharides, and sugar alcohols, in this study had similar mean concentrations as in summer 2019 and summer 2020 (Feng et al., 2023a). This could be due to the weak emissions from biomass burning in summer with little annual variation (Zhang et al., 2008; Li et al., 2020; Fu et

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al., 2023), and sugar polyols mainly originate from biogenic sources during the growing season with minimal influence from human activities (Simoneit et al., 2004; Jia and Fraser, 2011; Kang et al., 2018).

3.1.2 Duplicate-derived uncertainty

Figures 2 and S1 show comparisons of the concentrations of selected typical WSOMMs and other compounds in collocated Q_f samples. The scattering data of all identified WSOMMs fell along the identity line with strong correlations ($r > 0.90$, $p < 0.01$). The COD values of all species were below 0.20, indicating a high similarity between the collocated measurements (Krudysz et al., 2008). Yang et al. (2021) found that the median concentrations of bulk $PM_{2.5}$ components were negatively correlated with the corresponding ARPD values. In this work, such dependence of measurement uncertainties on ambient concentration was not observed for WSOMMs, possibly due to the high sensitivity of GC-MS analysis for derivatized WSOMMs. The SD_{diff} and ARPD values shown in Figures 2 and S1 are the uncertainties for particulate WSOMMs based on direct measurements, which are rarely reported. When using measurement data of particulate WSOMMs for receptor-based source apportionment (e.g., positive matrix factorization), uncertainty data are a required input and are often estimated using a propagation method (Hemann et al., 2009; Dutton et al., 2009a; Aleksankina et al., 2019), where an error fraction of 20% was usually assumed (Zhang et al., 2009). However, the ARPD values of several WSOMMs (e.g., levoglucosan, 2-MTH and mannosan) specifically related to PM sources were close to or even below 10% (Figures 2 and S1), and overestimation of uncertainties may lead to biased source apportionment results (Paatero and Hopke, 2003).

In previous studies, meso-erythritol was often used as a surrogate for the quantification of all isoprene SOA tracers (Ding et al., 2008; Hu et al., 2008; Lin et al.,

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2012; Feng et al., 2023b). Due to differences in molecular structures, MS fragments, and signal intensities, quantification of target compounds using surrogates can be subject to errors. As shown in Figure S2a and c, the quantification results of 2-MG and 2-MEH using authentic standards and meso-erythritol (surrogate) are strongly correlated (r =0.99, p < 0.01). But the mean concentration of 2-MG quantified using the authentic standard was 14.9% higher than that using the surrogate (Figure S2 b). The difference in the quantification of 2-MEH between using the authentic and surrogate standards was not apparent, which was attributed to the similarity of the structure of meso-erythrol and 2-MEH. To obtain more accurate measurement results of WSOMMs, authentic standards or at least surrogates with similar structures should be used for quantification.

3.2 Adsorption of gaseous WSOMMs or their precursors on untreated filters

Owning to the extremely low vapor pressures of the biomass burning tracers, saccharides, and sugar alcohols (Qin et al., 2021), these species were not detected in the Q_b and Q_{bb} samples from Sampler II or showed similar concentrations as the field blanks. Therefore, only the measurement results of isoprene SOA tracers and dicarboxylic acids in Q_b and Q_{bb} samples are presented and discussed. When Q_b and Q_{bb} were considered as adsorbents for sampling gaseous WSOMMs, the mean F% values of isoprene SOA tracers and dicarboxylic acids are well above 50% (Table 2). However, significant amounts of the target species were observed in the Q_b and Q_{bb} samples, indicating that the quartz filter can adsorb semi-volatile WSOMMs in the gas phase or their precursors that undergo heterogeneous reactions at the filter surface. After the sampling air flowed through Q_f and Q_b of Sampler II, the vapor pressures of the target compounds or precursors decreased significantly, resulting in lower concentrations in Q_{bb} samples than in Q_b samples (Table 2).

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Qin et al. (2021) collected particulate and gaseous WSOMMs at the same observation site by passing air samples through stacked Q_f and Q_b and a PUF plug. Similar to this study, Q_f was used to determine the particulate WSOMMs. Assuming that the WSOMMs detected in filters and PUF after Q_f are present in the gas phase, Figure 3 compares the concentrations of isoprene SOA tracers in different sampling matrices of this study and Qin et al. (2021) during the same period (August – September) of the year. In Figure 3a, the mean Q_f and Q_b concentrations of 2-MTs (13.5 ± 7.16 ng m^{-3} and 1.61 ± 1.53 ng m^{-3}; Table 2) and C_5-ATs (16.0 ± 14.7 ng m^{-3}, 0.24 ± 0.13 ng m^{-3}) are lower in this study than in Qin et al. (2021) (2-MTs 20.7 ± 17.6 ng m^{-3}, 3.96 ± 5.41 ng m^{-3}; C_5-ATs 22.0 ± 26.5 ng m^{-3}, 1.18 ± 1.42 ng m^{-3}). However, the Q_{bb} samples in this study had comparable or even higher mean concentrations of 2-MTs (0.75 ± 0.87 ng m^{-3}) and C_5-ATs (0.23 ± 0.29 ng m^{-3}) than the PUF samples (2-MTs 0.99 ± 0.75 ng m^{-3}; C_5-ATs 0.065 ± 0.062 ng m^{-3}; Figure 3c). Figure S3 shows that the *F*% of 2-MTs and C_5-ATs are similar in this study and in Qin et al. (2021) under comparable meteorological conditions, although the sampling year and sampling media are different. Thus, there is no appreciable difference in the gas-particle partitioning results between the use of quartz filters and PUF for sampling isoprene SOA tracers in the gas phase. Considering the higher recoveries in the measurement of isoprene SOA tracers in filter samples (106 ± 1.90%) than in PUF samples (about 50%), which are largely due to the elution of PUF materials, quartz filters can be used instead of PUF for sampling. The SV-TAG method proposes that SS fiber filters are suitable for sampling SVOCs in the gas phase if their surface area is large enough (Zhao et al., 2013b). The specific fiber surface area of quartz filters (~130 cm^2 cm^{-2}) is slightly lower than that of SS fiber filters (~160 cm^2 cm^{-2}) (Mader and Pankow, 2001b; Zhao et al., 2013b), but the diameter of quartz filters (≥90 mm) used for ambient sampling can be much larger.

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Without considering the heterogeneous reactions on the filter surfaces, no excessive breakthrough (B < 33%) was observed for 2-MG and 2-MTs based on the measurement results of the Q_b and Q_{bb} samples from Sampler II, but the B values of C_5-ATs and dicarboxylic acids are close to 50% (complete breakthrough). These results suggest that bare quartz filters are not effective adsorbents for sampling C_5-ATs and dicarboxylic acids in the gas phase. Since adsorption of gaseous WSOMMs on quartz filters is a potential source of artifacts when sampling particulate WSOMMs (Arhami et al., 2006), previous studies have adjusted the particulate concentrations of organic compounds by subtracting the amounts on Q_b samples from those on Q_f samples ($[Q_f]$–$[Q_b]$) (Mader and Pankow, 2000, 2001a, 2001b). In this approach, the amounts of gaseous organic compounds adsorbed in Q_f and Q_b samples are assumed to be equal, and evaporation of the particle phase is neglected. However, Q_b is exposed to lower concentrations of gaseous WSOMMs before Q_f reaches equilibrium with the air sample (Mader and Pankow, 2001b; Watson et al., 2009). Then, the $[Q_f]$–$[Q_b]$ method may lead to an overestimation of particulate concentrations unless the sampling time is long enough (Hart and Pankow, 1994; Subramanian et al., 2004).

In Sampler II of this study, a third bare quartz filter (Q_{bb}) was added after Q_f and Q_b, and the B values given in Table 2 also reflect the relationship between the amounts of gaseous WSOMMs adsorbed on two consecutive quartz filters. As such, it is more appropriate to estimate the amounts of gaseous WSOMMs adsorbed on Q_f ($[Q_f^*]$) by assuming that the B value of Q_f and Q_b ($[Q_b]/([Q_f^*]+[Q_b])$) is identical to that of Q_b and Q_{bb}. In this case, the artifact-corrected particulate concentrations of the WSOMMs can be calculated as $[Q_f]$–$[Q_f^*]$. As Figure 4 shows, the $[Q_f]$, $[Q_f]$–$[Q_b]$, and $[Q_f]$–$[Q_f^*]$ values of all six species have similar time series. However, except for C_5-ATs, the mean $[Q_f]$ and $[Q_f]$–$[Q_b]$ values of 2-MG, 2-MTs, and dicarboxylic acids are 33.8% – 78.1%

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and 11.1% – 40.3% times higher than that of [Q_f]–[Q_f*]. Since the volatilization of particulate WSOMMs in Q_f samples was not known, the values of [Q_f]–[Q_f*] can be regarded as a lower limit for filter-based measurements of particulate WSOMMs.

3.3 Adsorption of gaseous WSOMMs or their precursors on treated filters

The sampling efficiency of gaseous WSOMMs can be improved by treating the sampling medium with chemicals. Bao et al. (2012) collected gaseous organic acids using two tandem annular denuders coated with potassium hydroxide (KOH), and obtained a sampling efficiency up to 98% for short-chain dicarboxylic acids (C_2 – C_6). Kawamura and Kaplan (1987) and Bock et al. (2017) used KOH-impregnated quartz filters to collect motor vehicle emissions, and confirmed that engine exhaust is a source of dicarboxylic acids. In this study, the Q_{bb} on Sampler I was treated with $(NH_4)_2SO_4$ or KOH on different sampling days (Table S1). Table 3 compares the measurement results of the Q_b and $(NH_4)_2SO_4$-treated Q_{bb} samples from Sampler I with those of the collocated samples from Sampler II. The mean concentrations of 2-MTs and C_5-ATs in the treated Q_{bb} samples from Sampler I were 3.34 ± 2.64 ng m^{-3} and 3.92 ± 3.25 ng m^{-3}, respectively, which were 2.83 and 22.1 times higher than those in the untreated Q_{bb} samples from Sampler II. While the collocated Q_b samples had similar mean concentrations of 2-MTs and C_5-ATs.

Referring to the results of the chamber study, 2-MTs and C_5-ATs are formed by the reactive uptake of epoxydiols of isoprene (IEPOX) through the acid-catalyzed ring opening (Surratt et al., 2006, 2010). The coated $(NH_4)_2SO_4$ on Q_{bb} can absorb water vapor and act as an acid to promote the hydrolysis of IEPOX on filters to form 2-MTs and C_5-ATs. In addition, inorganic sulfate on filters can also react with gaseous IEPOX as a nucleophile to form organosulfate esters and oligomeric forms of 2-MTs and C_5-ATs. As shown in Table S2, Q_b and untreated Q_{bb} samples from Sampler II also contain

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a certain amount of inorganic sulfate due to the heterogeneous reactions of SO_2 (Pierson et al., 1980; Cheng et al., 2012), which are favored by the reactive uptake of IEPOX. The concentrations of SO_4^{2-} and NH_4^+ in the Q_b samples from Sampler I (SO_4^{2-} 0.13 ± 0.056 μg m^{-3}; NH_4^+ 0.033 ± 0.026 μg m^{-3}) and II (0.10 ± 0.040 μg m^{-3}, 0.024 ± 0.022 μg m^{-3}) were comparable, indicating that there was no significant transfer of $(NH_4)_2SO_4$ from treated Q_{bb} to Q_b on Sampler I during sampling. This also explains the similar concentrations of 2-MTs and C_5-ATs in Q_b samples between Sampler I and II. During the derivatization process of sample analysis, the organosulfate and oligomeric forms of 2-MTs and C_5-ATs can be converted to their monomeric forms by excess BSTFA (Lin et al., 2013a; Xie et al., 2014b); the conventional GC/EI-MS method also overestimates the concentrations of 2-MTs and C_5-ATs due to the thermal decomposition of less volatile oligomers and organosulfates (Lopez et al., 2016; Cui et al., 2018). Consequently, 2-MTs and C_5-ATs detected in the Q_b and Q_{bb} samples from both Sampler I and II were likely generated by heterogeneous reactions of gaseous IEPOX on quartz filter surfaces rather than by direct adsorption of gaseous molecules.

Unlike 2-MTs and C_5-ATs, 2-MG in $(NH_4)_2SO_4$-treated Q_{bb} samples (0.16 ± 0.12 ng m^{-3}; Table 3) did no show higher mean concentration in comparison to that in untreated Q_{bb} samples (0.24 ± 0.16 ng m^{-3}). 2-MG is formed by the acid-catalyzed ring opening of MAE, an oxidation product of isoprene under high NO_X conditions (Lin et al., 2013b). Surratt et al. (2007b) demonstrated that the formation of 2-MG is almost unaffected by changes in the acidity of the aerosol. Thus, 2-MG is stable in acidic aerosols and an equilibrium between the gas and particle phase could be achieved. The mean concentrations of succinic acid, glutaric acid, and adipic acid in $(NH_4)_2SO_4$-treated Q_{bb} samples were 11.26%, 57.4%, and 74.1% higher, respectively, than those in untreated Q_{bb} samples (Table 3). One possible explanation is that $(NH_4)_2SO_4$ is highly

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hygroscopic and promotes the dissolution of gaseous dicarboxylic acids by moisture absorption (Chen et al., 2021) or facilitates the heterogeneous formation of dicarboxylic acids (Yli et al., 2013; Bikkina et al., 2017).

Table 4 shows that the mean concentrations of 2-MG (1.92 ± 1.38 ng m^{-3}), succinic acid (7.05 ± 5.39 ng m^{-3}), glutaric acid (1.50 ± 1.71 ng m^{-3}), and adipic acid (1.16 ± 1.20 ng m^{-3}) in KOH-treated Q_{bb} samples from Sampler I are up to 13.7 times higher than those in untreated Q_{bb} samples from Sampler II. This can be explained by the formation of low-volatility organic compounds by neutralization reactions of gaseous organic acids on the surface of KOH-treated Q_{bb}. As described in *section* 3.2, the breakthrough in the sampling of gaseous 2-MG (24.1 ± 10.2%) and 2-MTs (28.1 ± 13.1%) is not excessively high when bare quartz filters are used. However, their concentrations in KOH- and $(NH_4)_2SO_4$-treated Q_{bb} samples increased substantially compared to untreated Q_{bb} samples (Tables 3 and 4), indicating that a low *B* value does not guarantee high sampling efficiency of gaseous WSOMMs or their precursors. Owing to the transfer of KOH from treated Q_{bb} to Q_b on Sampler I, the mean concentrations of 2-MG and dicarboxylic acids in Q_b samples from Sampler I are 1.84 – 2.26 times higher than those in Sampler II (Table 4). The reactive uptake of organic acids in Q_b samples from Sampler I during KOH treatment periods also led to increased WSOC and OC concentrations, and the transferred KOH on Q_b accelerated the heterogeneous formation of SO_4^{2-} and NO_3^- (Table S2).

4. Implications and conclusions

In this study, the uncertainties for the concentrations of particulate WSOMMs (5.85% – 19.9%) were determined by direct measurements of collocated Q_f samples. The uncertainties for several compounds (e.g., levoglucosan and 2-MTH) were well below the default value (~20%) commonly used in previous studies. The uncertainty data

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presented in this work are useful for future modeling and field studies on atmospheric transport, transformation, and source apportionment of water-soluble organic aerosols.

When the bare Q_b and Q_{bb} are considered as adsorbents for sampling gas-phase WSOMMs, the *F*% values obtained in this study are comparable to those obtained at the same sampling site using PUF as adsorbent. Based on the breakthrough of gaseous isoprene SOA tracers and dicarboxylic acids calculated from the measurement results of Q_b and Q_{bb} samples, a new method was developed to correct for the adsorption of gaseous organics on PM-loaded filter samples (Q_f), which accounts for the decrease in gas-phase concentrations after the air sample passes through Q_f. The adjusted Q_f measurements could be used as a lower limit for the particulate concentrations of WSOMMs.

By comparing the concentrations of isoprene SOA tracers and dicarboxylic acids between $(NH_4)_2SO_4$-/KOH-treated and untreated Q_{bb} samples, it was inferred that $(NH_4)_2SO_4$ on quartz filters can promote the heterogeneous formation of 2-MTs and C_5-ATs by reactive uptake of IEPOX, and KOH can increase the adsorption of gaseous organic acids on quartz filters by neutralization reactions. Due to the influence of surface reactions, WSOMMs detected in adsorbents associated with SOA sources (e.g., 2-MTs) may not indicate their existence in the gas phase. In further studies, chemically treated adsorbents can be developed for sampling gaseous WSOMMs with specific functional groups.

Data Availability

Data used in the writing of this paper (and its Supplementary Information file) are publicly available on Harvard Dataverse (Feng et al., 2025, https://doi.org/10.7910/DVN/ZD0JQW).

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Author contributions

MX designed the research. WF and XZ managed the sampling work and performed laboratory experiments. WF, XZ, and MX analyzed the data. WF and MX wrote the paper with significant contributions from ZS, GS, HL, and YW.

Competing interests

The contact author has declared that none of the authors has any competing interests.

Acknowledgements

This work was supported by the National Natural Science Foundation of China (NSFC, 42177211).

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Table 1. Mean concentrations of WSOMMs (ng m^{-3}) in Q_f samples from Sampler I and II.

Species	Abbreviation	Sampler I	Sampler II	Means of collocated samples
		Isoprene SOA tracers		
2-methylglyceric acid	2-MG	4.39 ± 3.29 [a]	4.57 ± 3.05	4.48 ± 3.15
2-methylthreitol [b]	2-MTH	3.57 ± 1.83	3.82 ± 1.93	3.69 ± 1.87
2-methylerythritol	2-MEH	9.20 ± 5.13	9.67 ± 5.29	9.43 ± 5.18
2-methyltetrols	2-MTs [c]	12.8 ± 6.91	13.5 ± 7.16	13.1 ± 7.00
cis-2-methyl-1,3,4-trihydroxy-1-butene [b]	cis-MTHB	3.47 ± 3.15	3.70 ± 3.12	3.58 ± 3.12
3-methyl-2,3,4-trihydroxy-1-butene [b]	MTHB	2.14 ± 1.82	2.25 ± 1.85	2.19 ± 1.83
trans-2-methyl-1,3,4-trihydroxy-1-butene [b]	trans-MTHB	10.0 ± 10.5	10.6 ± 10.3	10.3 ± 10.4
C_5-alkene triols	C_5-ATs [d]	15.2 ± 14.9	16.0 ± 14.7	15.6 ± 14.7
		Dicarboxylic acid		
succinic acid		20.8 ± 13.9	22.6 ± 15.4	21.7 ± 14.5
glutaric acid		8.31 ± 5.56	8.63 ± 4.98	8.47 ± 5.20
adipic acid		5.93 ± 3.45	6.59 ± 3.94	6.26 ± 3.60
		Biomass burning tracers		
galactosan		0.36 ± 0.51	0.42 ± 0.63	0.39 ± 0.57
mannosan		1.68 ± 1.04	1.78 ± 1.18	1.73 ± 1.11
levoglucosan		21.5 ± 19.4	22.9 ± 20.2	22.2 ± 19.8
		Saccharides		
fructose		12.5 ± 8.87	13.6 ± 8.99	13.1 ±8.82
glucose		9.29 ± 8.41	10.2 ± 9.04	9.75 ± 8.65
sucrose		28.0 ± 32.8	29.7 ± 33.7	28.9 ± 33.2
lactose		1.61 ± 1.37	1.69 ± 1.42	1.65 ± 1.40
mannose		0.70 ± 0.61	0.79 ± 0.64	0.75 ± 0.62
		Sugar alcohols		
arabitol		5.97 ± 4.66	6.66 ± 4.51	6.31 ± 4.56
pinitol		1.07 ± 0.82	1.15 ± 0.85	1.11 ± 0.84
mannitol		16.9 ± 23.0	18.8 ± 23.4	17.9 ± 23.1
sorbitol		1.00 ± 0.77	1.10 ± 0.72	1.05 ± 0.74
inositol		2.11 ± 1.03	2.25 ± 1.09	2.18 ± 1.04
chiro inositol		0.43 ± 0.41	0.47 ± 0.41	0.45 ± 0.41

[a] Standard deviation; [b] compounds were quantified using meso-erythritol as the surrogate, and other compounds were quantified using authentic standards; [c] sum of 2-MTH and 2-MEH; [d] sum of trans-MTHB, MTHB, and cis-MTHB.

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Table 2. Mean concentrations (ng m^{-3}), *B*, and *F*% of isoprene SOA tracers and dicarboxylic acids based on the measurement results of filter samples from Sampler II.

Species	Q_f	Q_b	Q_{bb}	*B*	*F*%
Isoprene SOA tracers					
2-MG	4.57 ± 3.05	0.85 ± 0.72	0.20 ± 0.13	24.1 ± 10.2	81.7 ± 9.98
2-MTH	3.82 ± 1.93	0.62 ± 0.52	0.16 ± 0.16	21.4 ± 11.9	83.9 ± 9.36
2-MEH	9.66 ± 5.29	1.13 ± 1.16	0.57 ± 0.70	32.2 ± 13.3	86.1 ± 10.1
2-MTs	13.5 ± 7.16	1.74 ± 1.63	0.73 ± 0.86	28.1 ± 13.1	85.5 ± 9.62
cis-MTHB	3.70 ± 3.12	0.053 ± 0.051	0.035 ± 0.025	42.4 ± 13.3	95.9 ± 4.38
MTHB	2.25 ± 1.85	0.064 ± 0.035	0.030 ± 0.016	32.8 ± 8.00	92.7 ± 6.71
trans-MTHB	10.6 ± 10.3	0.16 ± 0.21	0.099 ± 0.074	42.6 ± 13.5	95.2 ± 6.49
C_5-ATs	16.0 ± 14.7	0.29 ± 0.29	0.17 ± 0.11	40.0 ± 11.7	94.9 ± 5.80
Dicarboxylic acids					
succinic acid	22.6 ± 15.4	6.17 ± 3.76	3.66 ± 2.18	39.7 ± 11.2	68.1 ± 8.22
glutaric acid	8.63 ± 4.98	2.36 ± 1.46	1.18 ± 0.38	36.4 ± 12.1	69.4 ± 7.39
adipic acid	6.59 ± 3.94	1.10 ± 0.70	0.68 ± 0.39	40.0 ± 13.8	77.6 ± 6.51

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Table 3. Comparisons of the mean concentrations (ng m^{-3}) of isoprene SOA tracers and dicarboxylic acids in the Q_b and $(NH_4)_2SO_4$-treated Q_{bb} samples from Sampler I and the collocated untreated samples from Sampler II.

Species	Sampler I			Sampler II		
	Q_b	Q_{bb}	Q_{bb}/Q_b	Q_b	Q_{bb}	Q_{bb}/Q_b
Isoprene tracers						
2-MG	0.71 ± 0.73	0.16 ± 0.12	0.38 ± 0.25	1.01 ± 0.84	0.24 ± 0.15	0.36 ± 0.23
2-MTH	0.54 ± 0.54	0.88 ± 0.64	2.40 ± 1.82	0.70 ± 0.61	0.19 ± 0.20	0.30 ± 0.20
2-MEH	1.19 ± 1.29	2.46 ± 2.01	3.00 ± 1.90	1.42 ± 1.44	0.68 ± 0.89	0.49 ± 0.24
2-MTs	1.73 ± 1.78	3.34 ± 2.64	0.59 ± 0.48	2.13 ± 2.00	0.87 ± 1.08	0.42 ± 0.23
cis-MTHB	0.035 ± 0.024	1.05 ± 0.90	30.7 ± 15.1	0.061 ± 0.064	0.036 ± 0.029	0.83 ± 0.54
MTHB	0.046 ± 0.025	0.62 ± 0.64	11.5 ± 7.74	0.067 ± 0.039	0.031 ± 0.018	0.50 ± 0.19
trans-MTHB	0.10 ± 0.080	2.25 ± 1.75	21.9 ± 10.4	0.20 ± 0.27	0.10 ± 0.078	0.81 ± 0.45
C_5-ATs	0.19 ± 0.13	3.92 ± 3.25	20.3 ± 9.87	0.33 ± 0.37	0.17 ± 0.12	0.72 ± 0.38
Dicarboxylic acid						
succinic acid	7.07 ± 3.86	4.67 ± 5.27	0.75 ± 0.68	8.07 ± 4.17	4.18 ± 2.76	0.63 ± 0.26
glutaric acid	2.51 ± 1.55	1.99 ± 1.27	1.06 ± 0.72	2.97 ± 2.04	1.57 ± 1.13	0.67 ± 0.62
adipic acid	1.08 ± 0.59	1.23 ± 1.29	1.41 ± 1.31	1.29 ± 0.74	0.71 ± 0.41	0.64 ± 0.31

https://doi.org/10.5194/egusphere-2025-2106
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Table 4. Comparisons of the mean concentrations (ng m^{-3}) of isoprene SOA tracers and dicarboxylic acids in the Q_b and KOH-treated Q_{bb} samples from Sampler I and the collocated untreated samples from Sampler II.

Species	Sampler I			Sampler II		
	Q_b	Q_{bb}	Q_{bb}/Q_b	Q_b	Q_{bb}	Q_{bb}/Q_b
Isoprene tracers						
2-MG	1.74 ± 1.35	1.92 ± 1.38	1.92 ± 1.84	0.62 ± 0.51	0.14 ± 0.061	0.39 ± 0.32
2-MTH	0.46 ± 0.47	0.15 ± 0.11	0.92 ± 0.95	0.51 ± 0.39	0.13 ± 0.11	0.31 ± 0.24
2-MEH	0.93 ± 1.00	0.38 ± 0.25	0.81 ± 1.00	0.78 ± 0.60	0.43 ± 0.37	0.60 ± 0.49
2-MTs	1.39 ± 1.44	0.47 ± 0.30	0.84 ± 0.94	1.29 ± 0.93	0.56 ± 0.48	0.47 ± 0.37
cis-MTHB	0.068 ± 0.062	0.070 ± 0.090	1.60 ± 2.72	0.038 ± 0.020	0.031 ± 0.021	0.80 ± 0.25
MTHB	0.056 ± 0.037	0.024 ± 0.027	0.92 ± 1.26	0.054 ± 0.032	0.029 ± 0.013	0.75 ± 0.74
trans-MTHB	0.10 ± 0.12	0.033 ± 0.051	0.92 ± 1.12	0.11 ± 0.079	0.091 ± 0.076	0.91 ± 0.37
C_5-ATs	0.22 ± 0.16	0.12 ± 0.15	0.92 ± 1.30	0.21 ± 0.12	0.15 ± 0.11	0.82 ± 0.35
Dicarboxylic acid						
succinic acid	16.0 ± 11.4	7.05 ± 5.39	0.62 ± 0.63	4.90 ± 2.72	3.08 ± 1.18	0.81 ± 0.93
glutaric acid	3.81 ± 3.34	1.50 ± 1.71	0.43 ± 0.39	2.06 ± 1.03	1.14 ± 0.34	0.83 ± 0.93
adipic acid	2.91 ± 5.63	1.16 ± 1.20	0.67 ± 0.57	1.60 ± 2.37	0.71 ± 0.43	1.08 ± 1.62

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Figure 1

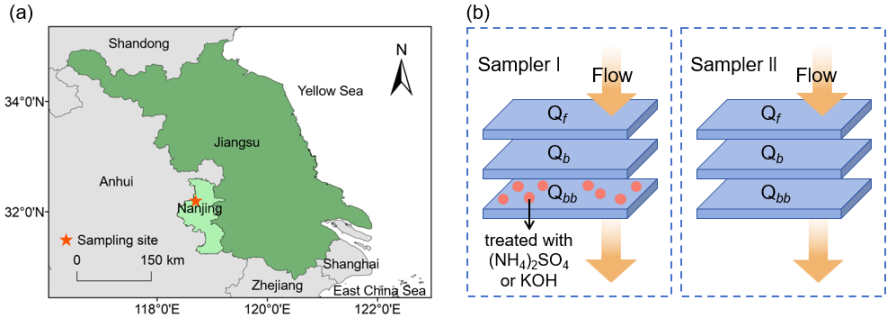

Figure 1. Location of the sampling site (a) and scheme of collocated sampling with three stacked quartz filters (b)

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Figure 2

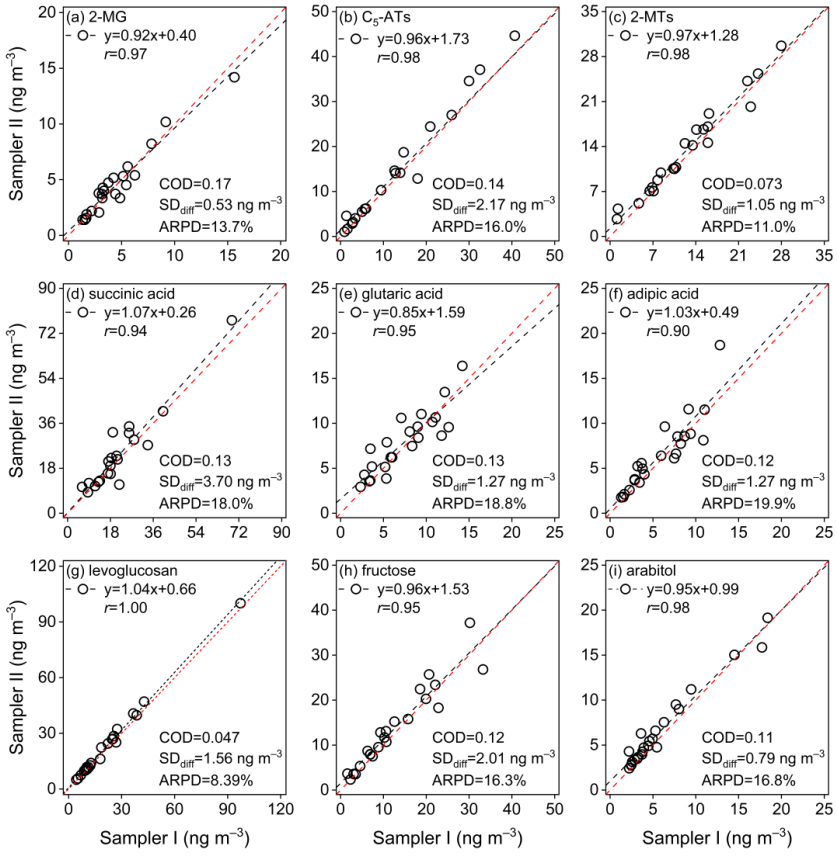

Figure 2. Comparisons of the concentrations of typical WSOMMs in collocated Q_f samples

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Figure 3

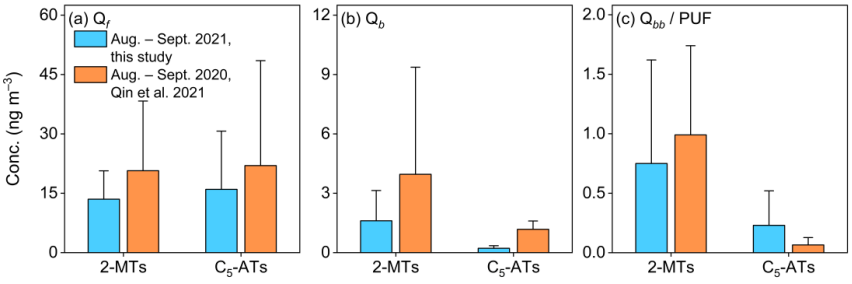

Figure 3. Comparisons of mean concentrations of 2-MTs and C_5-ATs in (a) Q_f, (b) Q_b, and (c) Q_{bb}/PUF samples between this study and Qin et al. (2021).

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Figure 4

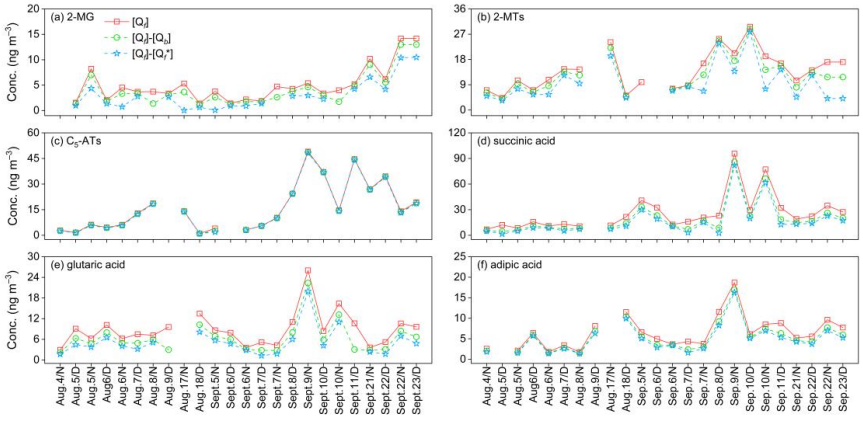

Figure 4. Comparisons of particulate concentrations of isoprene SOA tracers and dicarboxylic acid before and after gaseous adsorption corrections in summer 2021 (N: nighttime; D: day time).