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Development of an offline Aerosol Mass Spectrometry method for organic aerosol characterization in a globally distributed Surface Particulate Matter Network

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Abstract

Global measurements of organic aerosol (OA) concentrations and chemical composition remain limited and unevenly distributed. While monitoring networks, including the globally distributed Surface Particulate Matter Network (SPARTAN), provide an established framework for measurements, their current methodologies do not fully support comprehensive OA characterization. Aerosol Mass Spectrometry (AMS) is widely used for real-time OA composition measurements, but its cost, complexity, and logistical requirements limit long-term, multi-site online deployment, particularly at the global scale. Here we develop and evaluate an offline AMS methodology to characterize OA in particulate matter collected on Teflon filters routinely used by monitoring networks. Using a commercial ultrasonic nebulizer coupled with a syringe pump, this offline method is highly reproducible, requires small

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extract volumes (2 mL), offers low detection limits (1.7 µg OA and 0.43 µg sulfate per filter), and achieves higher nebulization efficiency than previous methods. We evaluate this offline AMS method by co-located online AMS observations. We find that oxygenated OA is effectively recovered (64 ± 28 %) while the recovery is lower for hydrocarbon-like OA due to its limited water solubility. This approach offers new capability for SPARTAN and is readily adaptable to other monitoring networks. Its application across networks will broaden the spatiotemporal coverage of AMS-based OA measurements and improve methodological and instrumental consistency to support ongoing efforts to build a long-term, globally consistent OA dataset.

1. Introduction

Organic aerosol (OA) constitutes a substantial fraction of atmospheric fine particulate matter ($PM_{2.5}$), contributing approximately 20–90 % of its total mass (Kanakidou et al., 2005; Zhang et al., 2007). OA influences climate through direct aerosol-radiation interactions (Chung et al., 2012) and cloud formation (Sareen et al., 2013), and affects human health due to its high potential to inflict oxidative stress and inflammation by generating reactive oxygenated and nitrogen species (Bates et al., 2019; Tuet et al., 2016). OA originates from both primary and secondary processes. Primary OA (POA) is directly emitted from fossil fuel combustion, biomass burning, and other anthropogenic or natural activities. Secondary OA (SOA) is formed through gas-phase oxidation of anthropogenic and biogenic volatile organic compounds (VOCs) and subsequent condensation or nucleation (Jimenez et al., 2009; Kroll and Seinfeld, 2008). The large number of molecular species and reaction pathways involved leads to extreme chemical complexity, complicating the characterization of OA loadings, spatiotemporal variability, and atmospheric oxidation processes (Kroll et al., 2011; Williams et al., 2007).

Despite advances in instruments capable of detailed chemical speciation, global OA concentrations and chemical composition remain poorly constrained (Sun et al., 2025). Measurements are largely derived from independent field campaigns that vary in

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instrumentation, sampling objectives, and methodologies, and are typically confined to limited time periods. In addition, these measurements are mostly conducted in developed regions in the northern midlatitudes, with relatively sparse representation from the Global South (Jeon et al., 2023; Tsimpidi et al., 2025). Therefore, global OA datasets compiled from collections of unique field campaigns face challenges such as limited comparability and incomplete spatiotemporal coverage, which further inhibit accurate evaluation of global OA simulations in chemical transport and climate models (Pai et al., 2020; Shrivastava et al., 2017). This highlights the need to routinely and consistently measure OA concentrations and composition across long-term monitoring networks of sites, particularly at the global scale.

The Surface Particulate Matter Network (SPARTAN, https://www.spartan-network.org/) collects $PM_{2.5}$ on Teflon filters from globally distributed sites that prioritize undersampled, densely populated regions. This network provides long-term, ground-based $PM_{2.5}$ composition measurements, including black carbon (BC), trace elements, water-soluble ions, and organic carbon (OC), to support the development and evaluation of satellite-based $PM_{2.5}$ estimates (Liu et al., 2024; McNeill et al., 2020; Ren et al., 2025; Snider et al., 2015, 2016; Weagle et al., 2018). Its consistency across globally distributed sites offers the possibility of constructing a comprehensive global OA dataset. Leveraging this potential, however, requires targeted methodological development to enable comprehensive OA characterization within the network.

The Aerodyne High-Resolution Time-of-Flight AMS (HR-ToF-AMS) has been extensively used for real-time measurement of OA with high time resolution and sensitivity in field campaigns (DeCarlo et al., 2006). By coupling thermal vaporization with 70 eV electron ionization (EI), AMS provides quantitative mass spectra of non-refractory aerosol components, including OA, sulfate, nitrate, ammonium, and chloride (Canagaratna et al., 2007). However, its high cost, operational complexity, and logistical requirements limit its feasibility for long-term, simultaneous deployment across multiple sites. This has stimulated the development of offline AMS methodology,

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where atmospheric samples are collected in the field and subsequently atomized for AMS analysis in the laboratory. Previous studies have demonstrated the promise of offline AMS for quantitative OA characterization. Sun et al. (2011) analyzed water extracts of $PM_{2.5}$ in the southeastern US to determine water-soluble organic matter to organic carbon (OM/OC) ratios using AMS, complemented by total organic carbon (TOC) measurements of water-soluble OC. Mihara and Mochida (2011) investigated extraction with multiple solvents (water, methanol, and ethyl acetate) for PM collected in urban Nagoya, Japan, using phthalic acid as an internal standard (IS) for quantification. Daellenbach et al. (2016) systematically assessed water-extract offline AMS and quantified source-related OA recoveries through comparison with co-located online measurements in Paris and Zurich. To extend applicability to lower mass loadings, O'Brien et al. (2019) introduced a small-volume nebulizer for microgram-level analysis with IS-based calibration slopes, while Niedek et al. (2023) developed another micronebulization technique with improved detection limits for PM collected from uncrewed platforms. To further improve OA recoveries, Vasilakopoulou et al. (2023) proposed excluding filtration during water extraction to retain partially soluble and insoluble OA fractions, and Khare et al. (2025) demonstrated improved OC recovery using organic solvents, with high similarity between online and offline mass spectra. Beyond methodological development and evaluation, offline AMS has been directly applied to characterize long-term OA concentrations and infer source properties across sites in Europe (Bozzetti et al., 2016, 2017a, 2017b; Casotto et al., 2022, 2023; Daellenbach et al., 2017; Moschos et al., 2022; Srivastava et al., 2021; Vlachou et al., 2018, 2019), US (Niedek et al., 2025), China (Cui et al., 2024; Ge et al., 2017; Huang et al., 2014; Li et al., 2021; Qiu et al., 2019; Xu et al., 2015; Ye et al., 2017a, 2017b), and India (Bhowmik et al., 2024; Cash et al., 2023).

Despite these developments, offline AMS has not been integrated as a routine measurement within globally distributed monitoring networks. For application within large-scale networks, the offline method is expected to meet several criteria: it should

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efficiently generate particles from small volume extracts given limited filter mass loadings, the setup should be reproducible and easy to maintain for rapid analysis, and the procedures should be compatible with existing protocols within monitoring frameworks. However, existing approaches do not fully satisfy these requirements. For example, the use of customized nebulizers can limit reproducibility, and some configurations require relatively large extract volumes (typically 5–15 mL) or additional operational steps and time to ensure cleaning and stability. In addition, quantification based on internal standard requires rigorous calibration, and the use of externally spiked internal standard (e.g., isotopically labeled ammonium nitrate or ammonium sulfate) may introduce potential interference while increasing sample preparation complexity. Therefore, further development and evaluation are needed to establish a reproducible and operationally streamlined offline AMS method suitable for routine deployment within globally distributed monitoring networks.

In this study, we develop an offline AMS methodology well-suited for additionally characterizing water-soluble OA in SPARTAN Teflon filters while sustaining existing measurements, with broader applicability to other monitoring networks that use similar filter-based sampling. We use a commercial ultrasonic nebulizer for high reproducibility and use sulfate as an internal standard given its routine measurement in monitoring networks. We further evaluate this offline AMS method against co-located online AMS measurements. This development of offline AMS methodology establishes a basis for a comprehensive OA dataset with consistent instrumentation and methodology across globally distributed sites in densely populated regions.

2. Methods

2.1 Chemicals

Ammonium sulfate (AS, American Chemical Society (ACS) grade) and nitric acid (ACS grade) are from Fisher Scientific. Methanol (high-performance liquid chromatography (HPLC) grade), glucose (≥99.5 %, gas chromatography (GC) grade),

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DL-4-Hydroxy-3-methoxymandelic acid (HMMA, ≥98 % HPLC grade), and poly(ethylene glycol) (PEG-400) are from MilliporeSigma. All solutions are prepared using ultrapure water (Milli-Q water, 18.2 MΩ·cm). Prior to use, all glassware is soaked in 1 % nitric acid for 24 hours, rinsed sequentially with methanol and a triple ultrapure water wash, and baked at 500 °C for 5 hours.

2.2 SPARTAN sampling and chemical analyses

SPARTAN is a long-term global network that measures ground-based PM composition at sites distributed across densely populated regions. Detailed descriptions of SPARTAN and its measurements have been described in the literature (Liu et al., 2024; McNeill et al., 2020; Ren et al., 2025; Snider et al., 2015, 2016; Weagle et al., 2018). PM samples are collected on 25 mm Teflon filters (PT25DMCAN-PF03A, Measurement Technology Laboratories, Bloomington, Minnesota, US) assembled in cartridges for use in sampling stations (SS5, AirPhoton, Baltimore, Maryland, US) coupled with a sharp-cut cyclone (SCC1.829, Mesa Laboratories, Lakewood, Colorado, US) typically operated at a target flow rate of 5 L/min for a $PM_{2.5}$ size cut. Each cartridge consists of seven sample filters and one field blank. Sampling duration is site-specific, ranging from 12 to 48 hours per filter to deposit approximately 100–200 µg of collected aerosol mass.

This network routinely provides measurements of PM mass and chemical composition. Filters are equilibrated and weighed before and after sampling under controlled conditions of 21.5 °C and 35 % relative humidity. Chemical analysis begins with non-destructive optical techniques, including Hybrid Integrating Plate/Sphere (HIPS) for BC (White et al., 2016), Fourier Transfer Infra-Red spectroscopy (FT-IR) for OC (Debus et al., 2022; Dillner and Takahama, 2015), and X-Ray Fluorescence (XRF) for elements (Liu et al., 2024). Elements, including sulfur, are quantified using an energy dispersive XRF (Epsilon 4, Malvern PANalytical, Worcestershire, UK). Calibration is performed annually, with monthly checks to confirm instrument stability (Liu et al., 2024). Following these non-destructive analyses, filters undergo a destructive

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extraction in which collected particles are dissolved into solution. Filters are placed aerosol-side up in a 20 mL scintillation vial and covered with 0.2 mL of HPLC-grade methanol followed by 5.8 mL of ultrapure water. The vial is sealed with aluminum foil and a plastic cap and sonicated for 30 minutes at room temperature. Sonication destroys the filter matrix, allowing the collected aerosol to dissolve or become suspended in the extract. The extract is then filtered through a 0.2 μm polypropylene syringe filter. A 4 mL aliquot of the filtered extract is refrigerated for Ion Chromatography (IC) analysis to quantify water-soluble ions. Cations including ammonium, and anions including sulfate, nitrate, and chloride, are measured using two IC systems (Dionex Integrion, Thermo Scientific, Waltham, Massachusetts, US). The cation system uses a methane sulfonic acid eluent generation system with a Dionex CS12A column and a CG12A guard column, while the anion system uses a potassium hydroxide eluent generation system with a Dionex AS18 column and AG18 guard column. Calibrations for both systems are performed at the beginning of each measurement sequence using standards prepared biweekly.

The remaining 2 mL aliquot of the filtered extract is transferred to a glass vial and immediately frozen for subsequent AMS analysis to characterize OA, which is the focus of this study.

2.3 Offline AMS methodology

Our offline AMS methodology consists of a syringe pump (780100I, Thermo Fisher Scientific Inc., Waltham, Massachusetts, US), a commercial ultrasonic nebulizer (U-5000AT+, Cetac Technologies Inc., Omaha, Nebraska, US), a diffusion dryer, a Scanning Mobility Particle Sizer (SMPS, Model 3938, TSI Inc., Shoreview, Minnesota, US), and an HR-ToF-AMS (Aerodyne Research Inc., Billerica, Massachusetts, US). A simplified schematic of the experimental setup is shown in Figure S1.

Filter extracts are delivered by the syringe pump to the ultrasonic nebulizer, where aerosol is generated through oscillation of a piezoelectric transducer operating at 1.4

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MHz (Ohata et al., 2011). Dry, zero air is introduced near the transducer to generate airflow. The downstream tube heater is turned off to minimize potential volatilization loss of organics, and water vapor is subsequently condensed in a tube maintained at 3 °C. The generated particles are then dried using a silica gel diffusion dryer and analyzed by the HR-ToF-AMS and SMPS for chemical composition and size distribution measurements. Figure S2 illustrates the measurement sequence for a SPARTAN cartridge using representative raw AMS data. Field blank filters are analyzed using the same procedure as the sample filters. Each cartridge measurement also includes a laboratory blank with no filter present, with signals used for blank subtraction within the corresponding cartridge. In addition, a 10 mg/L AS solution is analyzed at the beginning and end of each experimental day to verify instrument stability.

The choice of the nebulization system is critical for offline AMS methods. Commonly used commercial nebulizers, such as the TSI nebulizer (Model 3076, TSI Inc., Shoreview, Minnesota, US) (Li et al., 2021; Sun et al., 2011) and the Apex Q nebulizer (Elemental Scientific Inc., Omaha, Nebraska, US) (Khare et al., 2025; Vlachou et al., 2018), typically require relatively large extract volumes, which can be challenging for filters with limited collected mass. Custom nebulization systems (Cash et al., 2023; Niedek et al., 2023; O'Brien et al., 2019) have expanded the capabilities of offline AMS through specialized designs, however, these configurations are not commercially available and may be less accessible for routine implementation. The ultrasonic nebulizer used in this study has been applied in previous studies (Chen et al., 2019; Fang et al., 2015; Xu et al., 2017) and can efficiently nebulize small extract volumes while generating relatively large particles aligned with the AMS transmission efficiency. The system is stable and easy to clean and maintain, with carryover removed by nebulizing ultrapure water between samples within a few minutes (Figure S2), which supports routine analysis of large numbers of samples within monitoring networks.

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The HR-ToF-AMS operating principles, calibration procedures, and analysis protocols have been described extensively elsewhere (Canagaratna et al., 2007; DeCarlo et al., 2006). AMS provides quantitative mass spectra of non-refractory submicron PM (PM_1) components, including OA, sulfate, nitrate, ammonium, and chloride. In this study, the HR-ToF-AMS is operated in V mode (DeCarlo et al., 2006), providing a mass resolution of approximately 2000–3000 m/Δm and a temporal resolution of 1 minute. AMS data are processed using the standard AMS data analysis toolkits Squirrel v1.67A (SeQUential Igor data RetRiEvaL) and Pika v1.27A (Peak Integration by Key Analysis) in the IGOR Pro software package (Wavemetrics Inc., Portland, Oregon, US). Values for the AMS nitrate ionization efficiency (IE) and relative ionization efficiencies (RIEs) for sulfate and ammonium are determined through calibration following standard procedures. A default RIE value of 1.4 is assumed for OA (Xu et al., 2018). The chemical-dependent collection efficiency (CDCE) is applied following Middlebrook et al. (2011).

We perform positive matrix factorization (PMF) analysis for OA factor apportionment. The principles of PMF have been described in detail elsewhere (Paatero and Tapper, 1994; Ulbrich et al., 2009a). Briefly, PMF is a bilinear unmixing receptor model that quantitatively resolves organic mass spectra into a linear combination of static factor profiles and their time-dependent contributions, with each factor attributed to characteristic sources and atmospheric processes. PMF is conducted using the PMF Evaluation Toolkit (PET) v3.08E. Variables with low signal-to-noise (SNR < 0.2) are removed, while "weak" variables (0.2 < SNR < 2) are downweighted by a factor of two (Paatero and Hopke, 2009). Ions including O^+, HO^+, H_2O^+, and CO^+, which are scaled to CO_2^+, are also downweighted to prevent excessive weighting of CO_2^+ (Ulbrich et al., 2009a).

3. Results and Discussion

3.1 Optimization and characterization of nebulization system

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We optimize key parameters of the nebulization system, including carrier gas flow rate and liquid feed rate, to generate particles with high transmission efficiency in the AMS and higher mass concentrations (Figure 1). Increasing the carrier gas flow rate enhances aerosol transport and reduces upstream losses, leading to higher particle mass concentrations. However, at sufficiently higher flow rates, dilution by the carrier gas becomes dominant and reduces particle mass concentrations. Varying the carrier gas flow rate (0.5, 1.0, 1.3, and 1.5 L/min) shows a clear increase followed by a decrease in particle mass concentration, with 1.3 L/min identified as optimal. For the liquid feed rate, higher feed rates increase particle mass concentrations due to increased solute delivery. However, increasing the feed rate also reduces the number of AMS runs per sample of a finite extract volume. We vary the liquid feed rate at 0.10, 0.12, 0.15, and 0.20 mL/min, and select 0.20 mL/min to maximize particle mass concentration while maintaining an adequate AMS sampling period, providing approximately 10 minutes of measurement time from a 2 mL extract.

Under these optimized conditions, we characterize particle size distributions generated from AS solutions at concentrations of 2, 5, 10, and 20 mg/L. These concentrations are selected to represent the range of inorganic ion loadings observed in SPARTAN samples, with 2 mg/L and 20 mg/L corresponding approximately to the 5th and 95th percentiles. Across this range, the regenerated aerosol exhibits a mode electrical mobility diameter (D_m) of approximately 120 to 180 nm (Figure 1c), which aligns well with the AMS transmission efficiency that is nearly 100 % for particles with vacuum aerodynamic diameters (D_{va}) between 70 and 500 nm (Jimenez et al., 2003; Liu et al., 2007).

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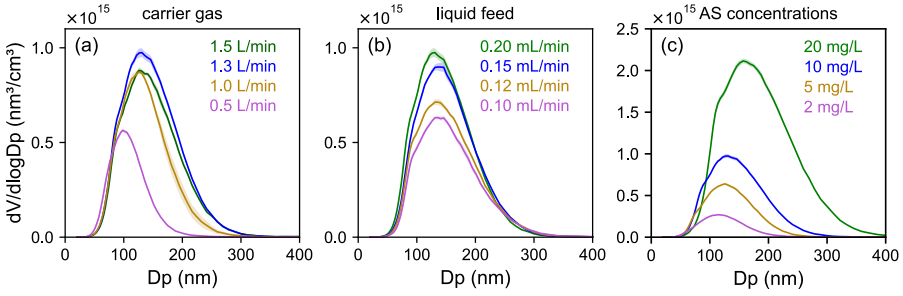

Figure 1. Particle size distributions generated from ammonium sulfate (AS) solutions and measured with a Scanning Mobility Particle Sizer (SMPS) to optimize nebulization system parameters. Panels show the effects of (a) carrier gas flow rate, (b) liquid feed rate, and (c) solution concentrations. Colored lines represent the mean of three runs, with colored shading areas indicating the standard error. The diameter (D_p) here is the electrical mobility diameter (D_m).

3.2 Quantification using internal standard

Nebulization efficiency (NE), defined as the ratio of mass measured by the AMS to the mass of solute nebulized, is a key indicator of offline AMS sensitivity (O'Brien et al., 2019). To quantify NE, 2 mL of binary solutions containing varying proportions of sulfate and organic species are nebulized at a fixed total solute concentration of 40 mg/L. The organic species examined include glucose, HMMA, and PEG-400. The analyte concentrations for each solution are listed in Table S1. For glucose, NE ranges from 0.70 to 1.1 %, while the corresponding sulfate ranges from 0.23 to 0.70 %, with NE varying with solution composition. Results for additional organic species are provided in Figure S3 and Text S1. The NE determined using this offline method is higher than that reported by O'Brien et al. (2019) and comparable to values reported by Niedek et al. (2023) (Table 1). We note that Fang et al. (2015) reported substantially higher efficiencies (47–60 %) using the same nebulizer for XRF analysis of water-soluble elements. This difference arises primarily because the AMS samples only a small fraction of the nebulized air (6.2 % in this study), while Fang et al. (2015) quantified the full nebulized output.

293 Table 1. Summary of offline AMS methods for OA characterization and comparison with this study.

Filter Type	Extraction Solvent	Nebulizer	Required Volume	Particle Mode Diameter[b]	Nebulization Efficiency	Internal Standard	Detection Limit[g]	Bulk recovery[g]	Reference
quartz	Water, methanol, and ethyl acetate	Custom-built	N/A	~ 300 nm[c]	N/A	Phthalic acid	N/A	N/A	Mihara and Mochida (2011)
quartz	Water	Custom-built	5–15 mL	~ 200 nm[d]	3.8 mL/m^3[f]	Sulfate	0.8 µg	64–76 %	Daellenbach et al. (2016)
N/A[a]	N/A[a]	Ultrasonic	N/A	~ 100 nm[d]	N/A	Sulfate	N/A	77–88 %	Xu et al. (2017)
quartz and PTFE	Water and methanol	Custom-built	2–4 µL	~ 150–200 nm[c]	0.02–0.06 %	Isotopically labeled nitrate	N/A	N/A	O'Brien et al. (2019)
PTFE	Methanol-water mixture	Custom-built	10 µL	~ 100–200 nm[d]	0.93–1.2 %	Isotopically labeled sulfate	2.2 ng[h]	N/A	Niedek et al. (2023)
quartz	Water	TSI	N/A	~ 100–200 nm[e]	N/A	N/A	N/A	N/A	Vasilakopoulou et al. (2023)
quartz	Water, methanol, and acetone	Apex Q	N/A	N/A	N/A	Isotopically labeled nitrate and sulfate	3–16 µg[i]	12–85 %[i]	Khare et al. (2025)
PTFE	Methanol-water mixture	Ultrasonic	2 mL	120–180 nm[c]	0.70–1.1 %	Sulfate	1.7 µg	53 ± 25 %	This study

294 Notes:

295 [a] Xu et al. (2017) use a Particle-into-Liquid Sampler (PILS) to convert ambient PM directly into aqueous extracts, rather than collecting particles on filters followed by
296 solvent extraction as in most offline studies; N/A indicates not reported or not applicable.

297 [b] Particle size comparisons across studies should be interpreted with caution due to differences in solvent composition, concentration, and measurement techniques,
298 including AMS, Scanning Mobility Particle Sizer (SMPS), hygroscopicity tandem differential mobility analyzer (HTDMA), and Zetasizer.

299 [c] Electrical mobility diameter.

300 [d] Vacuum aerodynamic diameter.

[e] Hydrodynamic diameter.

[f] The Unit is reported as $mL_{solution}/m^3_{air}$ in the original study, representing the volume of extract solution (mL) consumed per cubic meter of carrier air entering the AMS.

[g] Results are reported for OA unless explicitly stated; detection limits are expressed as mass per filter based on blank measurements unless otherwise noted.

[h] Detection limits are expressed as mass in solution, estimated using a standard binary solution containing sucrose and sulfate, each at a concentration of 0.06 mg/L.

[i] Results reported for OC.

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Variability in NE and the nonlinearity between nebulized and measured mass necessitate the use of an internal standard (IS) to quantify organic concentrations. In offline AMS studies, the IS should be non-refractory, soluble, chemically unreactive, distinguishable from other sample components, internally mixed with organics, and experience consistent and quantifiable nebulization processes (O'Brien et al., 2019). The organic-to-IS ratio determined from AMS signals can then be used to correct for any variations in NE and AMS sensitivity. The IS can be either an inorganic ion originally present in the extract that can be independently quantified, or a species externally spiked into the solution prior to nebulization at a known concentration. IS used in previous studies includes sulfate (Daellenbach et al., 2016; Li et al., 2021), nitrate (Srivastava et al., 2021), externally added isotopically labeled ammonium sulfate or ammonium nitrate (Casotto et al., 2022; Cui et al., 2024; Khare et al., 2025), and externally added phthalic acid (Mihara and Mochida, 2011). In this study, we select sulfate as the internal standard because it is consistently present in samples, water-soluble, nonvolatile, non-refractory, and independently quantified by both AMS and IC within SPARTAN. Limitations of this approach are discussed below.

Figure 2 shows calibration curves for glucose, HMMA, and PEG-400 using sulfate as the internal standard. To cover organic-to-sulfate ratios characteristic of urban background PM, we vary the ratios in solution from 0.18 to 14. We observe strong linearity ($r^2 > 0.996$) for organic-to-sulfate ratios between 0.28 and 2.8 in solution, while nonlinearity appears at higher ratios. This likely reflects increased AMS collection efficiency as organic-coated particles are more likely to adhere upon impact with the vaporizer (Matthew et al., 2008; Xu et al., 2018). Compared with previous offline AMS studies, this work explicitly considers nonlinearity in the calibration relationship. Given the diverse properties of PM across globally distributed SPARTAN sites, extending the organic-to-IS ratio range and accounting for nonlinearity are therefore important for robust OA quantification.

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Within the linear range (Figure 2b), we find slopes of 1.6, 0.89, and 2.2 for glucose, HMMA, and PEG-400, respectively. These slopes reflect the ratio of the AMS RIE of each organic compound to the RIE of sulfate. For complex organic mixtures, the most accurate calibration would ideally use standards comprised of the average chemical composition of ambient OA. However, such representation is challenging given the chemical diversity and spatiotemporal variability of atmospheric OA. For urban background aerosol, an RIE of 1.4 is commonly applied for OA (Canagaratna et al., 2007; Xu et al., 2018), and an RIE of 0.97 for sulfate was determined from the experiment-specific calibration in this study, corresponding to an organic-to-sulfate RIE ratio of approximately 1.4. Given that the glucose-derived slope (1.6) is consistent with this ratio, we select glucose-based calibration to approximate bulk OA in our samples. Accordingly, we apply the nonlinear calibration function derived from glucose in subsequent OA calculations for this offline method, with the associated uncertainty propagated as described in Text S2.

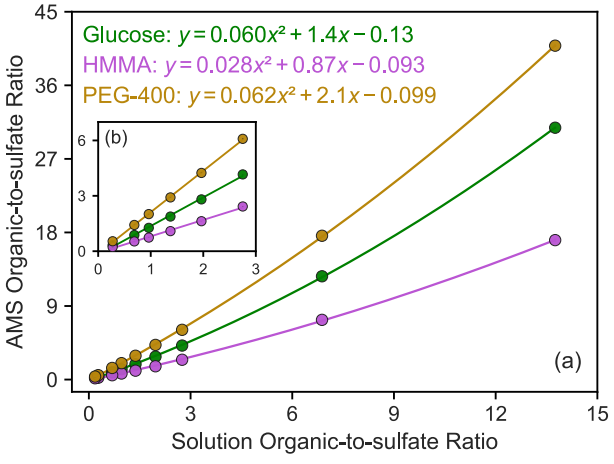

Figure 2. The organic-to-sulfate ratio in known solutions compared to that determined by AMS for three binary solutions (glucose, DL-4-Hydroxy-3-methoxymandelic acid (HMMA), and poly(ethylene glycol) (PEG-400)) across nine sulfate-organic combinations at a total solute concentration of 40 mg/L. The

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main panel (a) shows quadratic fits to all data, with fitted equations annotated for each compound. The inset (b) shows the corresponding linear relationships for six ratios within the solution range of 0.28–2.8. Data are displayed as mean and standard error from 8–12 replicate runs. Standard errors plotted as vertical error bars are on the order of 10^{-2} and imperceptible at the scale of the figure.

Using sulfate as an internal standard, the quantification procedure for the offline AMS method is summarized as follows. Each filter extract (2 mL) generates 8–12 AMS runs (1 minute time resolution) using the optimized nebulization system. For each run *k*, the blank-corrected AMS concentration for species *i* is calculated as:

$$\Delta I_{i,k} = I_{i,k} - \bar{I}_{i,LB} \quad (1)$$

where $I_{i,k}$ is the measured AMS concentration (μg/m^3) and $\bar{I}_{i,LB}$ is the measured mean laboratory blank concentration for the corresponding cartridge (μg/m^3).

For each filter extract, the organic-to-sulfate ratio measured by AMS is calculated as the mean of the ratios across *N* runs:

$$\phi_k = \frac{\Delta I_{OA,k}}{\Delta I_{sulfate,k}} \quad (2)$$

$$\phi_{AMS} = \frac{1}{N}\sum_{k=1}^{N} \phi_k \quad (3)$$

where ϕ_k is the organic-to-sulfate ratio for each run *k* and ϕ_{AMS} is the organic-to-sulfate ratio for each filter extract in AMS.

Assuming an internal mixture with consistent behavior of sulfate and OA during nebulization, ϕ_{AMS} is converted to the organic-to-sulfate ratio in the extract, $\phi_{extract}$, using the inverse nonlinear calibration function $f(\phi_{AMS})$ in Figure 2. The offline OA concentration ($C_{OA,offline}$, μg/m^3) is then calculated as:

$$\phi_{extract} = f(\phi_{AMS}) \quad (4)$$

$$C_{OA,offline} = \phi_{extract} \cdot C_{sulfate,extract} \cdot V_{extract} \cdot \frac{1}{V_{air}} \quad (5)$$

where $C_{sulfate,extract}$ is the sulfate concentration in extract determined by IC (μg/mL), $V_{extract}$ is the extract volume (mL), and V_{air} is the sampled air volume (m^3). Uncertainties

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in these parameters are propagated to estimate the overall uncertainty in the offline OA concentration (Text S2).

The detection limit is defined as three times the standard deviation of blank measurements. For offline AMS, which includes ultrapure water blanks, field blanks, and laboratory blanks (Section 2.3), we calculate detection limits for each blank type and adopt the largest value as a conservative estimate (Table S2). The resulting detection limits are 1.7 μg for OA and 0.43 μg for sulfate, expressed as mass on filters. Approximately 99.8 % of filters collected in SPARTAN exceed the sulfate detection limit, indicating the suitability of this offline method to SPARTAN and its potential for application in other monitoring networks.

3.3 Evaluation with online AMS and other measurements

3.3.1 Online-offline sample collection

To evaluate the offline methodology, we conducted a pilot study by comparing OA concentrations determined by the offline method with co-located online AMS measurements. PM samples were collected from September to November 2025 on the Danforth Campus (38.65° N, 90.31° W) of Washington University in St. Louis, Missouri, US. Sampling was conducted by drawing air through a stainless-steel sampling line from a third-floor rooftop located away from primary aerosol sources to represent an urban background environment. Use of a single sampling line ensures consistent air sampling for both the offline and online configurations in a controlled laboratory environment. Filter sampling was performed using standard SPARTAN stations operating continuously, with 24-hour sampling at flow rates ranging from 6 to 11 L/min. These flows enable the collected filters to primarily capture particles within the effective size range of $PM_{2.5}$ and PM_1, given the PM_1 size cut of the cyclone at 11 L/min and the $PM_{2.5}$ size cut at 5 L/min. In total, six cartridges were sampled, yielding a collection of 42 paired filter samples, along with six paired field blanks. Collected filters were analyzed using the HR-ToF-AMS following the offline methodology

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described in Section 2.3, and 30 paired filter samples exceeded offline detection limits and are retained for further analysis. Concurrently, the same AMS with a standard PM_1 aerodynamic lens was operated alongside filter sampling from the same sampling line to provide continuous online measurements of non-refractory PM_1. Blanks were obtained by periodically inserting a high-efficiency particulate air (HEPA) filter upstream of the cyclone during the campaign. Average online OA concentrations over the duration of each filter sampling period were compared with the corresponding offline filter measurements. Together, this setup enables a direct comparison between offline filter-based measurements and co-located online AMS measurements. Previous online-offline comparisons often have mismatches in instrument configurations (offline AMS and online aerosol chemical speciation monitor (ACSM)), particle size cuts (PM_{10} (aerodynamic diameter <10 μm), $PM_{2.5}$, and PM_1), and sampling time or location, which can introduce additional uncertainty. In contrast, this study uses the same sampling line and the same AMS instrument for both measurements, minimizing potential interferences and providing a robust basis for comparison.

3.3.2 Online-offline comparison

Figure 3 compares bulk OA concentrations derived from the offline AMS method with those measured by the co-located online AMS. Most samples show lower concentrations in offline AMS, with two exceptions that may reflect measurement uncertainty. The OA recovery is 53 ± 25 % (mean ± standard deviation), with a median recovery of 48 % (38 % and 61 % for the first and third quartiles, respectively). For complex mixtures such as ambient OA, recovery depends on the water solubility of its numerous compounds (Daellenbach et al., 2016). Previous offline studies using water extraction have reported a wide range of bulk OA recoveries, from 29 % in the polluted city of Handan and 47 % in Beijing during wintertime in Northern China (Li et al., 2021; Qiu et al., 2019) to 64–76 % for PM collected in Paris and Zurich (Daellenbach et al., 2016). Thus, it reflects both the efficiency of the offline method and intrinsic OA properties that vary with composition and sources.

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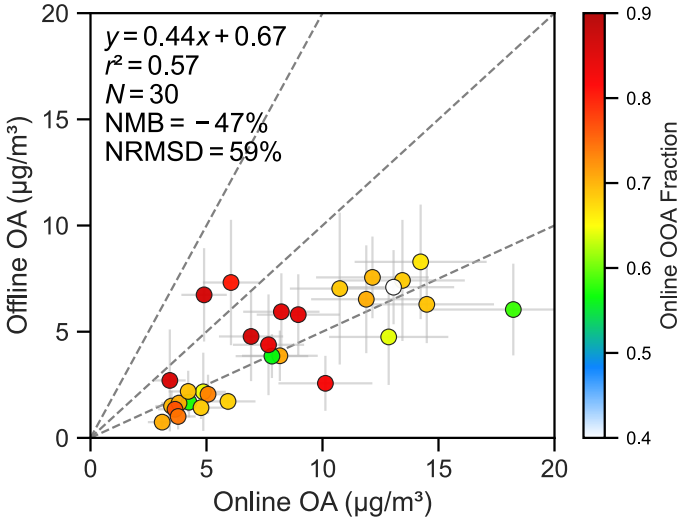

Figure 3. Comparison of online OA mass concentrations directly measured by the AMS with offline OA concentrations derived from filter samples analyzed using the offline method for samples collected in St. Louis. Online OA represents the time-averaged concentration over each corresponding filter sampling interval, with horizontal bars indicating the estimated AMS measurement uncertainty following Bahreini et al. (2009). Offline OA concentrations are calculated using sulfate as the internal standard and the nonlinear glucose calibration function, with vertical bars indicating the propagated uncertainty of offline OA concentrations. Each point presents the mean across 8 to 12 runs. Points are colored by the oxygenated OA (OOA) fraction determined from positive matrix factorization (PMF) of the online measurements. Annotations indicate the ordinary least-squares fit (y), coefficient of determination (r^2), number of comparison points (N), Normalized Mean Bias (NMB), and Normalized Root Mean Square Deviation (NRMSD). Dashed lines indicate reference lines with slopes of 0.5, 1, and 2.

To evaluate how well the offline AMS analysis preserves ambient OA composition, we compare average mass spectra from online and offline measurements (Figure 4). The

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spectra show strong agreement, with an r^2 of 0.85. Similarly, Niedek et al. (2023) found an r^2 of 0.70 for mass spectra collected in north-central Oklahoma, US, and Daellenbach et al. (2016) found an r^2 > 0.97 for mass spectra from Zurich, both comparing offline AMS with online ACSM measurements. Despite the overall agreement, differences are observed in the relative contributions of individual ions, suggesting that certain species may be preferentially recovered during filter collection, extraction, and nebulization. To better understand compositional differences, OA recovery is further examined by grouping ions into representative chemical families following Daellenbach et al. (2016). Fragments are classified into mono-oxygenated ions ($CHO_{z=1}$), poly-oxygenated ions ($CHO_{z>1}$), saturated hydrocarbons (CH_{sat}), and unsaturated hydrocarbons (CH_{unsat}). Recovery varies substantially across ion families (Figure S4). The highest recovery (mean ± standard deviation) is observed for $CHO_{z>1}$ family (80 ± 35 %), dominated by organic acids, followed by $CHO_{z=1}$ family (48 ± 21 %), likely representing alcohols, aldehydes, and ketones. In contrast, hydrocarbon-like families have lower recoveries, with 37 ± 20 % for CH_{unsat} and 25 ± 16 % for CH_{sat}. N-containing hydrocarbon ions (CHN) contribute only a small fraction of total OA and are therefore not explicitly examined.

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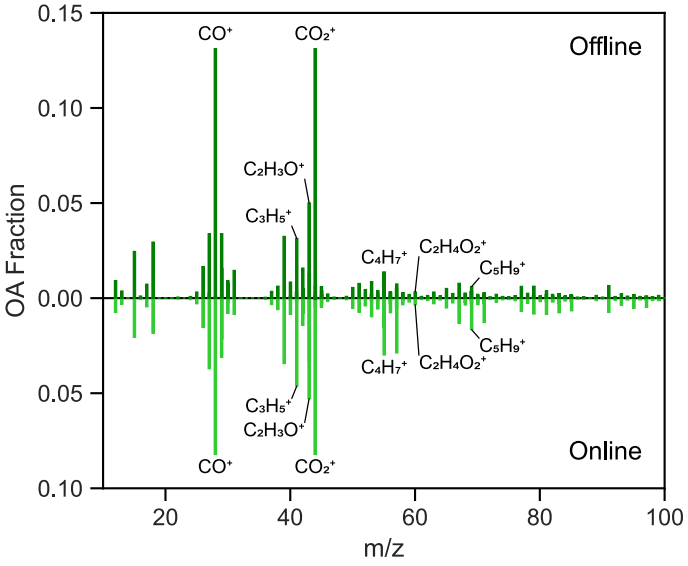

Figure 4. Comparison of average OA mass spectral fractions from online measurements (bottom panel, light green) obtained directly by the AMS and offline measurements (top panel, dark green) analyzed using the offline method for samples collected in St. Louis. Fragments (m/z) commonly considered as source-specific markers are explicitly labeled with their formula.

In addition to individual ions, we evaluate the recovery of OA factors. First, we perform PMF analysis on online measurements and resolve two OA factors: oxygenated OA (OOA) and hydrocarbon-like OA (HOA) (Figure S5). This two-factor solution is selected for its physical interpretability and stable splitting and mixing behavior, providing a broadly applicable framework for future applications (Figure S6). The resulting factor profiles are consistent with established spectral characteristics, with OOA dominated by oxygenated fragments at m/z 44 (CO_2^+) and 43 (mostly $C_2H_3O^+$) and HOA characterized by alkyl fragments ($C_nH_{2n+1}^+$ and $C_nH_{2n-1}^+$) (Cubison et al., 2011; Ng et al., 2010). These online-derived factor profiles are then used as a priori constraints in the offline analysis. Offline factor concentrations are quantified by applying weighted non-negative least squares (NNLS) regression to the offline mass

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spectra using these factor profiles. The coefficients are constrained to be non-negative, and measurement uncertainties are incorporated as weights to minimize the residual between observed and reconstructed signals (Wang and Hopke, 1989). We perform weighted NNLS on offline OA mass spectra for each run, and the mean factor concentration across runs within a filter is used to represent the offline factor contributions for that filter (see details in Text S3).

Figure 5 compares online and offline concentrations for OOA and HOA. OOA exhibits relatively high recovery (mean ± standard deviation) of 64 ± 28 % and a median of 59 % (49 % and 79 % for the first and third quartiles, respectively). In contrast, HOA recovery is substantially lower, with a mean and standard deviation of 9.9 ± 7.1 % and a median of 8.5 % (4.6 % and 14 % for the first and third quartiles, respectively). These results are consistent with the range reported in the literature (Figure 6), with OOA exhibiting relatively high and consistent recovery, while other factors show lower and more variable recovery. For example, Daellenbach et al. (2016) found recoveries of 89 % for OOA and 11 % for HOA for PM collected in Paris and Zurich. Xu et al. (2017) reported that 77 %, 96 %, and 68 % of isoprene-OA, more oxidized OOA (MO-OOA), and less oxidized OOA (LO-OOA), respectively, are water-soluble in Atlanta, while HOA is largely water-insoluble. Li et al. (2021) found recoveries of 49 % for OOA, 37 % for biomass burning OA (BBOA), and 17 % for coal combustion OA (COA) in Handan, northern China. Together, these observations align with the established understanding of chemical characteristics of different OA components, with water solubility decreasing from highly oxygenated species to more hydrocarbon-like species (Kondo et al., 2007; Saxena and Hildemann, 1996).

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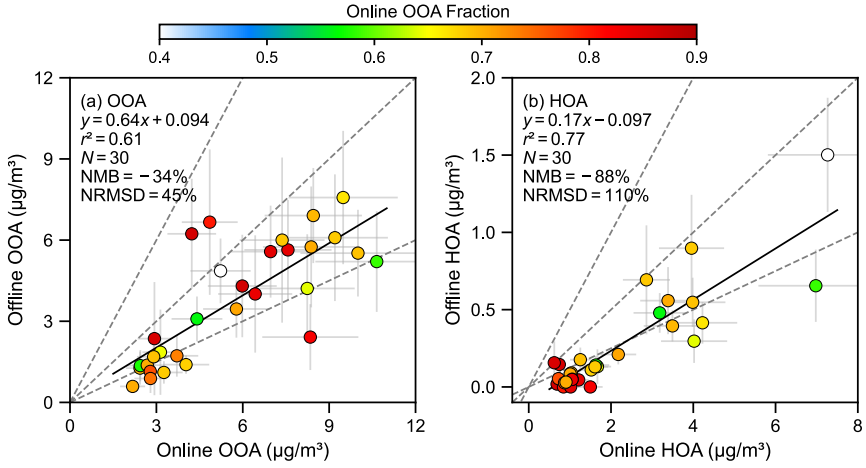

Figure 5. Comparison of online and offline factor contributions for (a) oxygenated OA (OOA) and (b) hydrocarbon-like OA (HOA) for samples collected in St. Louis. Online concentrations are calculated from positive matrix factorization (PMF) factor contributions and time-averaged bulk concentrations over each corresponding filter sampling interval, with horizontal bars indicating the estimated AMS measurement uncertainty. Offline concentrations are calculated using factor contributions derived from weighted non-negative least-squares (NNLS) regression with fixed factor profiles from online PMF, and multiplied by offline bulk OA concentrations determined using the offline method, with vertical bars indicating the propagated uncertainty of offline OA concentrations. Each point presents the mean across 8 to 12 runs. Points are colored by the OOA fraction determined from online PMF. Annotations indicate the ordinary least-squares fit (y), coefficient of determination (r^2), number of comparison points (N), Normalized Mean Bias (NMB), and Normalized Root Mean Square Deviation (NRMSD). Dashed lines indicate reference lines with slopes of (a) 0.5, 1, and 2 and (b) 0.125, 0.25, and 0.5.

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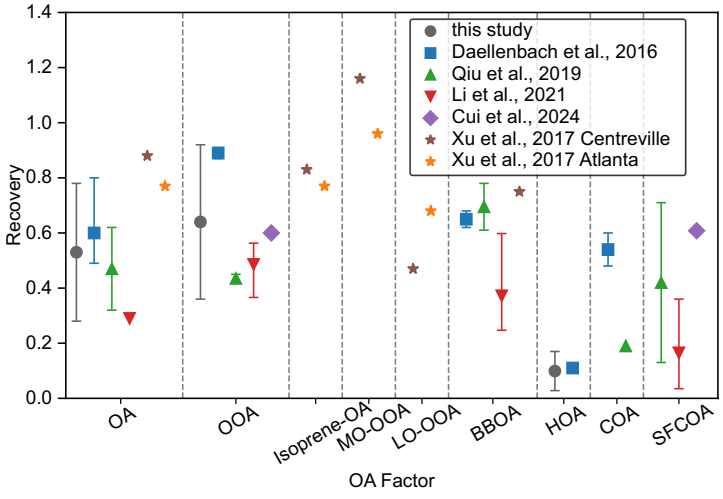

Figure 6. Comparison of OA recovery reported in the literature and in this study across bulk OA and major OA factors, including oxygenated OA (OOA), isoprene-derived OA (isoprene-OA), more-oxidized OOA (MO-OOA), less-oxidized OOA (LO-OOA), biomass burning OA (BBOA), hydrocarbon-like OA (HOA), cooking OA (COA), and solid fuel combustion OA (SFCOA). Symbols indicate different studies. Original values from each study are shown, so direct comparison should be interpreted with caution due to differences in statistical measures across studies. This study is reported as mean ± standard deviation, Daellenbach et al. (2016) as median with first and third quartiles, Qiu et al. (2019) as mean ± standard deviation, Li et al. (2021) as median with the 10th and 90th percentiles, Cui et al. (2024) as mean values, and Xu et al. (2017) as mode. For studies reporting ranges only, the central value is taken as the midpoint of the reported range.

Building on the factor-resolved recovery, we reconstruct OA by weighting each offline-derived factor by its mean recovery and compare it with online OA measurements (Figure 7). This approach aligns with the objectives of this offline method and monitoring networks such as SPARTAN for long-term ambient OA characterization. This comparison shows an r^2 of 0.68 and a Normalized Mean Bias (NMB) of 5.4 %. When evaluated over longer timescales such as at the cartridge level of one week,

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agreement improves further, with r^2 increasing to 0.84 and NMB decreasing to 2.2 % (Figure S7). These results highlight that offline measurements can provide reliable estimates of ambient OA concentrations, particularly over longer periods.

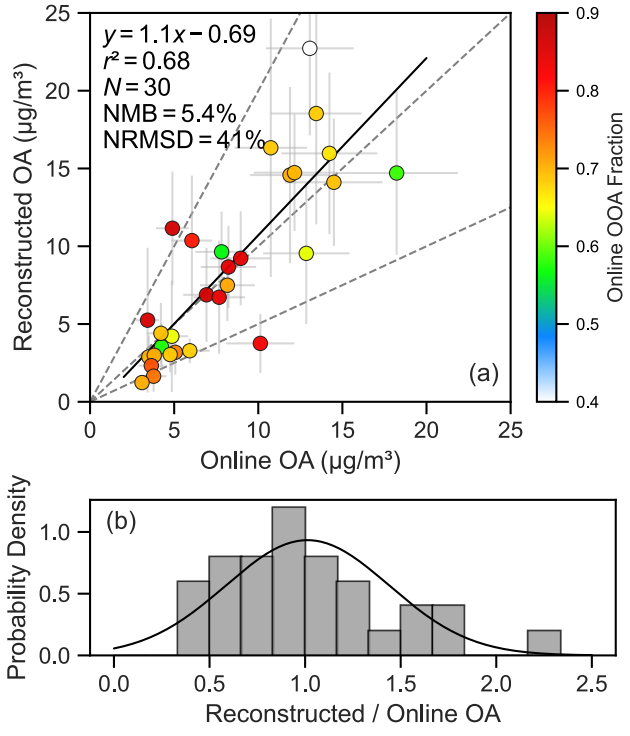

Figure 7. (a) Comparison of reconstructed OA concentrations with online OA concentrations for samples collected in St. Louis. Reconstructed OA concentrations are calculated by dividing each offline-derived factor by its mean recovery. Online OA represents the time-averaged concentration over each corresponding filter sampling interval. Horizontal bars indicate the estimated AMS measurement uncertainty of online OA concentrations, while vertical bars indicate the propagated uncertainty of offline OA concentrations. Each point presents the mean across 8 to 12 runs. Points are colored by the oxygenated OA (OOA) fraction determined from positive matrix factorization (PMF) of the online measurements.

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Annotations indicate the ordinary least-squares fit (y), coefficient of determination (r^2), number of comparison points (N), Normalized Mean Bias (NMB), and Normalized Root Mean Square Deviation (NRMSD). Dashed lines indicate reference lines with slopes of 0.5, 1, and 2. (b) Histogram of the ratio of reconstructed to online OA for the same samples. The fitted curve represents a smoothed probability density function of this ratio.

As an additional evaluation, sulfate, ammonium, and nitrate concentrations derived from the offline AMS are compared with co-located online AMS and complementary offline IC and XRF measurements. Sulfate shows strong agreement across measurement techniques, including online AMS, IC measurements of water extracts, and XRF measurements of filter samples, with r^2 of 0.80 and 0.95 for the respective comparisons (Figure S8a and S8b). Since offline AMS sulfate is intrinsically linked to IC sulfate through its use as the internal standard, the two are treated equivalently in this comparison. The observed agreement suggests a minor contribution from organosulfur compounds in the collected samples and supports the use of sulfate as the internal standard, as further discussed in Section 3.4. Offline AMS-derived ammonium and nitrate are quantified using sulfate as the internal standard, following the same approach as for OA but assuming a unity calibration slope between ammonium-to-sulfate and nitrate-to-sulfate ratios in the extract and in the AMS. We find strong agreement in ammonium concentrations between offline AMS and IC, with a slope of 1.1 and r^2 of 0.93 (Figure S8d), supporting the reliability of the offline quantification. Additional comparisons with other measurements are provided in Text S4.

3.4 Uncertainty in the application to SPARTAN

To support the goal of characterizing ambient OA across SPARTAN using reconstructed OA, we evaluate additional sources of uncertainty beyond those quantified for offline OA in the St. Louis online-offline comparison samples. Specifically, we assess the applicability of sulfate as an internal standard across SPARTAN sites and the variability in OOA and HOA factor profiles and recoveries.

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We use sulfate as the internal standard to leverage its routine measurements in most monitoring networks. Two potential concerns should be noted. First, sulfate concentrations have declined in many regions over time, which may influence its suitability as a reference species (Tsimpidi et al., 2025). Second, part of the AMS sulfate signal may arise from organosulfur compounds rather than inorganic sulfate measured by IC. However, these factors appear to have limited influence in this study. The first concern is mitigated by both IC and AMS providing low detection limits for sulfate, by retaining only samples with sulfate concentrations above the detection limits, and by increasing sulfate in many regions targeted by SPARTAN. The second concern is addressed by comparing water-soluble inorganic sulfate measured by IC with total particulate sulfur measured by XRF to identify and exclude future samples that may contain large contributions from organosulfur compounds. Therefore, errors associated with using sulfate as the internal standard are expected to be small.

We focus on the two most broadly applicable OA factors, OOA and HOA, to provide a simple but robust framework suitable for application across diverse SPARTAN sites. For the St. Louis online-offline comparison samples, offline factor contributions are quantified using weighted NNLS regression with fixed factor profiles derived from co-located online AMS measurements. However, such site-specific, highly accurate factor profiles may not be available at many SPARTAN locations with limited AMS observations. To evaluate this limitation, we use the AMS Spectral Database to identify four additional pairs of deconvoluted OOA and HOA factor profiles (Ulbrich et al., 2009a, 2009b, 2009c), derived from studies conducted in the US (Zhang et al., 2005), Zurich (Lanz et al., 2008), China (Elser et al., 2016), and an average across five sites (Ng et al., 2011). These profiles represent variability in factor spectra and are applied as fixed priors in weighted NNLS regression for the St. Louis offline samples. The resulting four sets of offline OOA and HOA, together with the original set based on co-located measurements, are then extrapolated using recoveries to reconstruct ambient OA. Factor recovery varies with time and location, indicating that recovery estimates

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derived at one site may not be directly applicable to other sites (Li et al., 2021; Vasilakopoulou et al., 2023). We consider this variability in two ways. First, previous studies suggest that OOA recovery is relatively consistent across sites (Figure 6). Second, we define conservative recovery ranges by synthesizing results from this and previous offline AMS studies, yielding 0.36–0.92 for OOA and 0.028–0.17 for HOA. A sensitivity analysis is performed by bootstrapping OOA and HOA recoveries 100 times from these ranges for five sets of factor profiles across 30 samples for reconstruction. Despite variability in factor profiles and the use of broad recovery ranges, the distribution of reconstructed-to-online OA ratios exhibits a mode between 0.8 and 1.2 (Figure S9), supporting the feasibility of future extrapolation of offline OA measurements to characterize ambient OA across SPARTAN sites.

4. Conclusions

This study presents a comprehensive development and evaluation of an offline AMS methodology and demonstrates its applicability to filter-based measurement networks. The commercial nebulization system enables continuous aerosol generation from small sample volumes of 2 mL and is straightforward and reproducible to operate, representing a substantial improvement for large-scale sample processing. Use of sulfate as an internal standard leverages routine sulfate measurements in monitoring networks. Offline AMS results are directly evaluated against co-located online measurements using the same sampling line and instrument. The online-offline comparison shows varying recovery across OA factors, with the highest recovery observed for OOA, likely due to its higher water solubility. Therefore, this offline method provides a robust estimate of oxygenated species, although correction is required when estimating hydrocarbon-like species associated with more primary emissions. Despite source-dependent recovery biases, the offline method reliably represents ambient OA concentrations, supporting its application in monitoring networks and enabling the development of a consistent OA dataset across globally

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distributed sites. Future work will apply this method to the Surface Particulate Matter Network to examine globally distributed variation in OA.

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Data availability

All datasets are available upon request from Lu Xu (xu1@wustl.edu) and Randall V. Martin (rvmartin@wustl.edu)

Author contributions

YR designed the study, conducted the experiments, analyzed the data, and drafted the manuscript. RVM secured funding, co-designed the study, edited the manuscript, and provided guidance throughout the analysis. JHC contributed to AMS instrument maintenance and nebulizer optimization. CRO assisted with SPARTAN operations. BJW contributed to AMS instrument maintenance. RJW assisted with the ultrasonic nebulizer. LX contributed to AMS measurements, co-designed the study, edited the manuscript, and provided guidance throughout the analysis. All authors reviewed and approved the final manuscript.

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