

Filter- and Denuder-Based Organic Carbon Correction for Positive Sampling Artifacts

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ABSTRACT

This study describes (1) the impact of positive sampling artifacts caused by not only a filter-based sampling, but also a denuder-based sampling in the determination of particle-phase organic carbon (POC), (2) the effect of sample flow rate on positive artifacts, and (3) an optimum flow rate that provides a minimized negative sampling artifact for the denuder-based sampling method. To achieve the goals of this study, four different sampling media combinations were employed: (1) Quartz filter-alone (Q-alone), (2) quartz filter behind quartz-fiber filter (QBQ), (3) quartz filter and quartz filter behind Teflon filter (Q-QBT), and (4) quartz filter behind carbon-based denuder (Denuder-Q). The measurement of ambient POC was carried out in an urban area. In addition, to determine gas-phase OC (GOC) removal efficiency of the denuder, a Teflon filter and a quartz filter were deployed upstream and downstream of the denuder, respectively with varying sample flow rates: 5, 10, 20, and 30 LPM. It was found that Q-alone sampling configuration showed a higher POC than QBQ, Q-QBT, and Denuder-Q by 12%, 28%, and 23%, respectively at a sample flow rate of 20 LPM due to no correction for positive artifact caused by adsorption of GOC onto the filter. A lower quantity of GOC was collected from the backup quartz filter on QBQ than that from Q-QBT. This was because GOC was not in equilibrium with that adsorbed on the front quartz filter of QBQ during the sampling period. It is observed that the loss of particle number and mass across the denuder increases with decreasing sample flow rate. The contribution of positive artifacts to POC decreased with increasing sample flow rate, showing 29%, 25%, and 22% for 10, 20, and 30 LPM, respectively. The 20 LPM turns out to be the optimum sample flow rate for both filter and denuder-based POC sampling.

Key words: Positive artifact, Organic carbon, Teflon filter, Quartz filter, Adsorption

1. INTRODUCTION

Carbonaceous compounds such as elemental carbon (EC), organic carbon (OC), and a small quantity of carbonate carbon (CC) constitute a significant portion (approximately 10-70% of the PM mass) of the ambient aerosol mass in the various areas (Djajic et al., 2016; Bisht et al., 2015; Casimiro et al., 2011). Carbonaceous compounds play a major role in serious atmospheric pollution such as visibility degradation and climate change, and these compounds have adverse human health effects (Bisht et al., 2015; IPCC, 2007). EC originates from incomplete combustion processes of fossil fuel. On the other hand, OC is directly emitted in particulate matter (primary OC) or formed in the atmosphere from products of gas phase photochemical reactions (secondary OC) (Safai et al., 2014; Salako et al., 2012).

Development of measurement methods for ambient particle-phase organic carbon (POC) has been one of the key research subjects because there have been challenges in selectively collecting POC from the mixture of gas-phase OC (GOC) and POC. In addition, ambient POC is known not only as the first or second largest contributor to the total ambient PM_{2.5} mass (Na et al., 2004; Kim et al., 2000), but also as causes of visibility reduction and climate changes (Kim et al., 2016). Therefore, it is of great importance to accurately collect and quantify POC in order to not only establish reduction strategies of ambient PM_{2.5} mass, but also understand the compositional characteristics of ambient PM_{2.5}. Ambient OC coexists in both gas- and particulate phases.

Quartz fiber filters are the most widely used substrate to collect POC and elemental carbon (EC)

because of their thermal stability, inert surface, and excellent filtering efficiency (Zhu *et al.*, 2012; Olson and Norris, 2005; Na *et al.*, 2004; Kirchstetter *et al.*, 2001). On the other hand, since quartz filters have large enough surface areas, it is prone to adsorb GOC. Consequently, POC sampling without removing GOC from sample air stream results in overestimation of the POC because GOC is quantified as POC (which is referred to as OC positive artifact) or pyrolyzed OC during the analysis. The evaporation of GOC from the collected sample induced an underestimation of OC concentration (that is, negative artifact) (Liu *et al.*, 2014).

Many studies have been investigated to characterize and eliminate OC artifacts to determine POC. POC sampling approaches can be divided generally into two methods according to the time GOC is corrected or removed: (1) denuder-based correction and (2) filter-based correction. Briefly speaking, denuder-based sampling involves removing GOC before it is collected on filters, while filter-based sampling is to correct GOC after it is collected on filters.

Many studies on OC positive artifacts have been conducted with a fixed sample flow rate. 16.7 LPM is the most widely used flow rate because most commonly used $PM_{2.5}$ cyclone is designed operating at that flow rate. Sample flow rate (similar to fact velocity) affects GOC adsorption on quartz filters because a contact time between sample and collection media is one of the most important factors affecting physical adsorption of gas. That is, the longer contact time, the more GOC is adsorbed on the filters. So far little investigations of the effect of sample flow rate on OC artifacts have been done (McDow and Huntzicker, 1990). Therefore, the main purpose of this study is to find out an optimum flow rate that provides a minimized positive sampling artifacts and a maximized OC and EC collection efficiency for the filter- and the

denuder-based correction methods. To achieve the goals of this study, four different sampling media combinations were employed: (1) Quartz filter-alone (Q-alone), (2) quartz filter behind quartz- fiber filter (QBQ), (3) quartz filter and quartz filter behind Teflon filter (Q-QBT), and (4) quartz filter behind carbon-based denuder (D-Q).

2. EXPERIMENTAL METHODS

2.1 Sample Collection

OC and EC were collected by a $PM_{2.5}$ sampler with four separate channels operating on flow controllers and a pump. Each channel is operated on its own flow controller, which enables independent sampling with varying sample flow rates. A $PM_{2.5}$ impactor was designed by the principle made by Biswas and Flagan (1988), which offers a $2.5 \mu m$ cut-point at 40 LPM. This high volumetric flow impactor allows for multi-channel and high flow sampling as opposed to 16.7 LPM cyclone. To evaluate the $PM_{2.5}$ impactor performance, Andersen Reference Ambient Air Sampler (Andersen Instrument, Inc., Smyrna, GA) was employed for concurrent PM sampling. The average differences between the two PM samplers were within 20% for three 24 hour samples. The PM impactor was connected to a manifold with five sampling channels with the channel 5 non-operational, which allows for 8 h sampling per each of the remaining channels. A total of four of each of 8 h ambient samples were collected at a flow rate of 8 LPM for each of the channels.

The sampler configuration of the four channels is presented in Fig. 1 and Table 1. Channel 1 is carbon filter sheet impregnated denuder-based sampling channel (Denuder-Q). Channel 2 and 3 are filter-based sampling channels (Q-alone, QBT, and QBQ in respective order). The channel 4 is employed to evaluate gas-phase

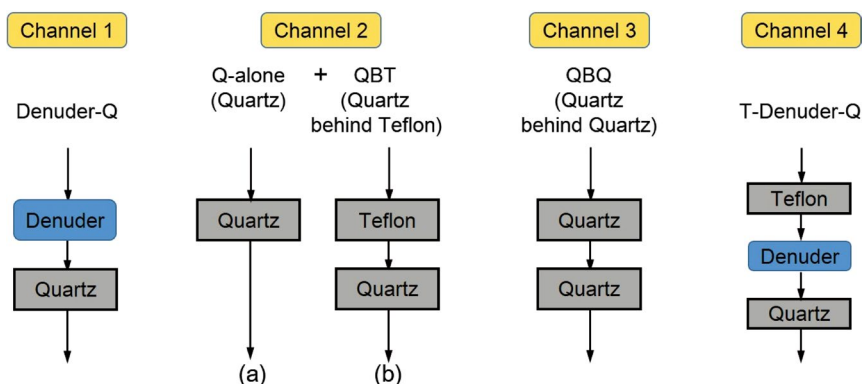


Fig. 1. $PM_{2.5}$ sampler with 4 channels for collection of OC and EC.

Table 1. Configuration of the PM_{2.5} sampler with 4 channels for OC/EC collection.

Channel	1	2		3	4
		(a)	(b)		
Configuration	Denuder-Q	Q-alone	QBT	QBQ	T-Denuder-Q
Front filter	NL	Quartz	Teflon	Quartz	Teflon
Backup filter	Quartz	NL	Quartz	Quartz	Quartz

NL: Not loaded

OC removal efficiency of the denuder and to determine GOC found on dynamic filter blank. The carbon denuder is 28 cm long and 16 cm² of cross section area with a residence time of ~0.30 s at 8 LPM, which ensures a plug flow with a Reynolds number of $Re < 300$ across the denuder. To minimize the interlot variability in adsorption capacity, quartz filters taken from the same lot were used throughout the sampling. Five quartz blank filters were quantified to correct filter blank mass.

2.2 Substrate Preparation

Prior to sampling, the Teflon filters were conditioned in the PM stabilization chamber with the filters exposed to the chamber at $22 \pm 1^\circ\text{C}$ and 9.5 ± 1 relative humidity (RH) for a few days and until the difference between pre-sampling and post-sampling filter mass readings after a few days exposure was maintained within $\pm 10 \mu\text{g}$. Based on previous experiment, fresh Teflon filter needed to be exposed to the weighing chamber for 24 h in order for the moisture adsorbed on the filters to be in equilibrium with that in the weighing chamber. Until the weighed filters are used, they were stored in a petri-dish and in the PM weighing chamber. After sampling and prior to weighing the filters used, they were again exposed to the conditioned PM chamber at the same above specified temperature and humidity as they were exposed for pre-weighing. Quartz fiber filters were pre-cleaned to remove OC/EC present originally in the filters by baking for 24 h at 900°C . Several such baked filters were analyzed as blanks for OC and EC analysis. In all cases, the OC and EC mass found on these filters were below the instrument's detection limit of $0.2 \mu\text{gC}$.

2.3 Sample Analysis

A thermal/optical carbon analyzer (Sunset Laboratory, Forest Grove, OR, USA) was used to quantify OC and EC running on NIOSH (National Institute of Occupational Safety and Health) 5040 Method and modified NIOSH method. For high loading of OC sample on the filter, it was observed that the NIOSH 5040 failed to evolve OC completely within Helium atmosphere region. In other words, vaporized OC was

kept detected until the end of the OC4 fraction. Therefore, the modified NIOSH method with extended analysis time was employed to fully volatilize OC collected on the filter before the end of the He region. The residence times for the NIOSH 5040 are 60 s at 250°C , 500°C , and 650°C , and 90 s at 850°C in the He atmosphere followed by 30 s at 650°C and 750°C , 60 s at 850°C , and 120 s at 900°C in the O₂/He atmosphere. The residence time of modified NIOSH method is longer and varies depending on the amount of OC and EC in filter deposit.

3. RESULTS AND DISCUSSION

Prior to applying the carbon-based denuder for OC/EC collection, the denuder performance was tested with Sunset semi-continuous OC/EC analyzer (Model 4, OR, USA) and a scanning mobility particle sizer (SMPS, Model 3080, TSI Inc., MN) to examine the denuder efficiency and the loss of particle number and particle mass due to the diffusion to the surfaces of the denuder.

3.1 Denuder Efficiency on GOC Removal

To test the denuder efficiency, two channels (channel 1 and 4) were operated simultaneously at a flow rate of 8 LPM for 1 h. The same denuder was installed for each of the two channels. The channel 1 collected ambient POC and denuder-breakthrough GOC. The channel 4 sampled non-removed portion of GOP, which was coupled with a semi-continuous OC/EC analyzer (Sunset Inc., Tigard, OR). The channel 4 was configured with a filter holder loaded with a $2.0 \mu\text{m}$ Teflon filter (Pall Gelman Sciences, Ann Arbor, MI) to remove POC, the carbon-based denuder to remove GOC, and the semi-continuous OC/EC analyzer to collect GOC not removed by the denuder (see Channel #5 in Table 1). The semi-continuous OC/EC analyzer has a dual function capable of sampling and analyzing carbon aerosol on a semi-continuous basis. Once a quartz filter is loaded inside the analyzer, the filter can be repeatedly used for OC/EC sampling and analysis. As soon as the OC/EC collection is done, the analyzer

automatically starts analyzing the OC/EC sample. As a result, filter loaded in the analyzer is automatically cleaned during its analysis. This feature enables to eliminate filter baking, sampled filter handling, storage, and loading to the analyzer for its analysis. Accordingly, the semi-continuous OC/EC analyzer can provide contamination-free filter sampling, eliminating filter blank correction. For the denuder efficiency test, two-layered quartz blank filters were first loaded into the analyzer to better capture GOC and were run with the NIOSH 5040 method to not only remove any possible contaminants present in the blank filters, but also quantify pre-existing OC/EC on the filters. This analysis is repeated until no OC/EC is detected. The GOCs not collected by the denuder were sampled three times from ambient air for 1 h at a face velocity of $\sim 100 \text{ cm s}^{-1}$ which is defined as the volumetric flow rate per OC exposed area. Dynamic filter blank values were defined in this study as the sum of field blank OC and denuder-breakthrough GOC. Dynamic and field blank corrections were made for denuder-based and filter based OC samples in a respective order to correct positive artifacts. The GOC found on the dynamic filter blank behind the denuder was on average $0.28 \pm 0.21 \mu\text{gC cm}^{-2}$, which is approximately 8% of the POC measured on quartz filter placed downstream of the denuder. This shows that the denuder efficiency is sufficient to evaluate positive artifacts found on denuder-based OC/EC sampling methods. The GOC measured on the dynamic filter blank is significantly higher than the average OC (POC + GOC) loading on field blank filters of $0.11 \pm 0.08 \mu\text{gC cm}^{-2}$. Accordingly, the GOC measured on the dynamic blank was subtracted from the POC measured on the quartz filter behind the denuder.

3.2 Denuder Efficiency on POC Removal

Denuder may result in particle loss due to (1) the diffusion to the surface of denuding substrates and (2) POC partitioning into GOC. Accordingly, it is important to examine the effect of denuder on POC prior to using the denuder. For this, indoor ambient air was used as a source of particles to provide better stabilized supply of particles than outdoor ambient air. $\text{PM}_{2.5}$ impactor with a $2.5 \mu\text{m}$ cut-point was used. The denuder effect on the particles losses (i.e., number and

mass) was evaluated with SMPS for the particle size, ranging from 3 nm to 135 nm. Four different sample flow rates were employed: 5, 10, 20, and 30 LPM in order to see how particle residence time affects particle loss inside the denuder. Five data points were obtained for each sample flow rate. The results are depicted in Fig. 2.

It is observed that the loss of particle number and mass across the denuder increases with decreasing sample flow rate. This can be explained by the fact that lower sample flow rate drives more particles to diffuse to the surface of denuder substrates than higher flow rate, resulting in both particle number and mass losses. For the 30 LPM, particle gain is found. This is likely due to flaking of the carbon denuder. The 20 LPM turns out to be the optimum sample flow rate for the denuder-based OC/EC sampling.

3.3 Comparison of Filter-based and Denuder-based POC and Positive Artifacts

Table 2 compares POC and GOC obtained from four different sampling configurations. The Q-alone (bare quartz filter configuration) shows the highest POC, followed by QBQ, Denuder-Q, and Q-QBT. The high-

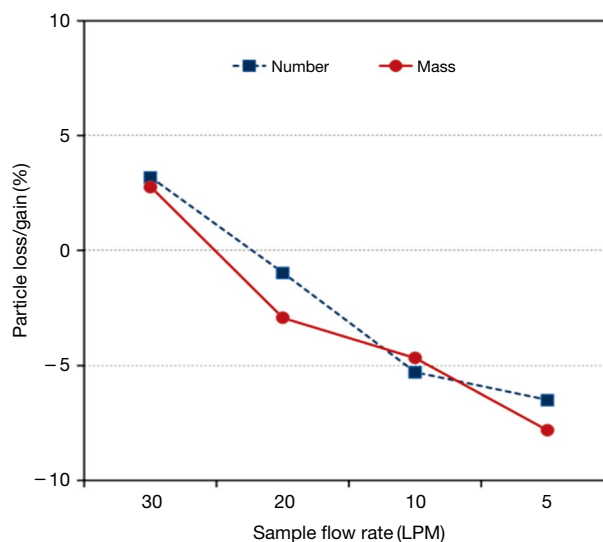


Fig. 2. Particle loss and gain through the carbon-based denuder in terms of sample flow rate.

Table 2. POC and GOC measured from four sampling configurations ($\mu\text{gC cm}^{-2}$).

Channel (No. of samples)	1 (5)	2 (5)	3 (5)	4 (5)
Configuration	Denuder-Q	Q-alone	QBQ	Q-QBT
Front Filter		5.43 ± 1.65	5.43 ± 1.65	5.43 ± 1.65
Backup Filter			0.64 ± 0.19	1.20 ± 0.32
POC Estimated	4.42 ± 1.30	5.43 ± 1.65	4.79 ± 1.47	4.23 ± 1.38

est POC found on the Q-alone was likely due to GOC adsorbed on the filter. Therefore the GOC measured on the bare quartz filter should be corrected to obtain true POC. POC in QBQ configuration was estimated to be approximately 13% and 8% higher than those in Q-QBT and Denuder-Q configuration, respectively. This may be because (1) both the front and backup quartz filters of QBQ are not saturated with GOC and (2) more GOC is present on the front filters than the backup filters. The level of OC (POC + GOC) measured on the backup filters in Q-QBT configuration was higher than the level measured on the backup filter of QBQ. This observation is consistent with previous studies (Cheng *et al.*, 2009; Olson and Norris, 2005; Subramanian *et al.*, 2004; Turpin *et al.*, 2000). This is due to difference in the gas adsorptive capacity between Teflon and quartz fiber filters. In other words, GOC adsorption on Teflon is negligible while the quartz filter has a sufficient surface area to adsorb GOC, causing higher OC in Q-alone configuration than in QBQ and Q-QBT configurations. Denuder-Q configuration produces 4% higher POC than Q-QBT configuration. This may be attributed to the combination of GOC escaping the denuder and GOC breaking through the denuder.

3.4 Effect of Sample Flow Rate on Positive Artifacts

Three sets of the QBQ configuration were employed to examine the effect of sample flow rate on positive artifacts. The OC sampling was performed simultaneously for 8 h at 10, 20, and 30 LPM for each of the three QBQ filter packs. Since the PM_{2.5} impactors used for this study were designed to be operated at 40 LPM, each of the two PM_{2.5} impactors was connected to the common upstream of 10 and 30 LPM filter packs and two 20 LPM filter packs, respectively.

Fig. 3 shows OC measured on front and backup filters, and the ratio (also termed the fraction of positive artifact) of OC measured on backup filter to OC measured on front filter in QBQ configuration in terms of sample flow rate. OC values measured on both front filter and backup filters decreases with increasing sample flow rate. This is consistent with a previous study (McDow and Huntzicker, 1990). As the sampling flow rate increase, the contribution of positive artifacts to POC also decreases. This can be explained by the fact that the longer time GOC contacts quartz filter surface, the more GOC is adsorbed on the filter.

3.5 Validity of the QBQ Configuration on Artifact Correction in Terms of Face Velocity

QBQ configuration has been used as one of the most

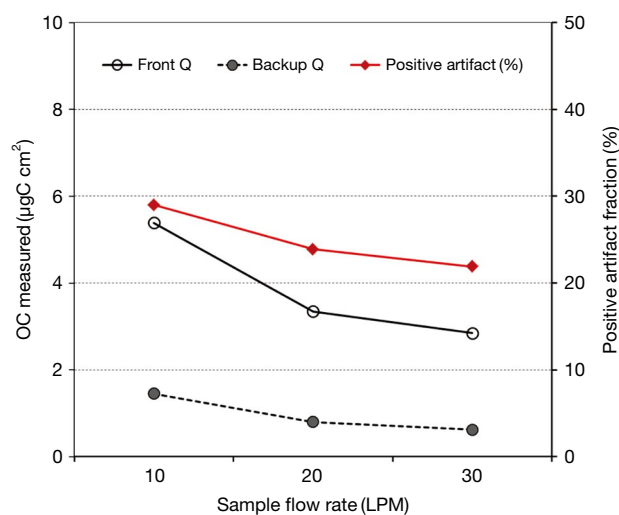


Fig. 3. OC and positive artifacts measured from the QBQ configuration at varying sample flow rates.

common approaches in the determination of positive artifacts involving the use of a backup quartz filter that is placed downstream of the front quartz filter. Since the backup filter is exposed only to particle free air due to the removal of POC by the front filter, OC measured on the backup filter is presumed to originate mainly from the gaseous OC. POC in the QBQ configuration is determined by subtracting gaseous OC measured on the backup filter from the OC collected on the front filter. This positive artifact correction is based on three major assumptions: (1) the amount of gaseous OC collected on the front filter is equal to the gaseous OC collected on the backup filter, (2) adsorption capacity of each quartz filter for gaseous OC is the same, and (3) sampling time is longer than the time for quartz filter to be saturated with gaseous OC. If assumption (1) for the QBQ configuration is valid, subtracting OC collected on the backup filter from that collected on the front filter should be insignificant. In case of invalidity of assumption (1), the correction in the QBQ configuration results in overestimation of particle-phase OC. To evaluate assumption (1), four sets of the QBQ configuration were used with one common Teflon filter pack installed upstream of the inlet of ambient OC samples to remove POC. The OC sampling was performed simultaneously at 5, 10, 20, and 30 LPM for each of the four QBQ filter packs in order to examine the validity of the QBQ configuration sampling along with the effect of face velocity on estimation of gaseous OC adsorbed on the front and backup filters. The samples were collected for 8 h.

Fig. 4 shows a plot of GOC collected on front and backup quartz filters in the QBQ configuration against sample flow rate. Green dots represent positive artifact

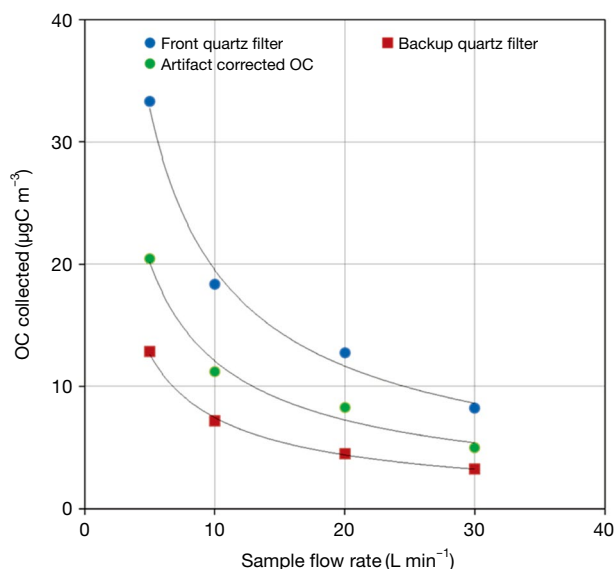


Fig. 4. GOC collected on front and backup quartz filters in the QBQ configuration against sample flow rate.

corrected OC which is calculated by subtracting GOC collected on the backup filter from the GOC collected on the front filter. It is evident from the graph that GOC measured from both front filter and backup filter decreases with increasing sample flow rate, indicating that the faster the face velocity, the less the adsorption of gaseous OC on quartz filter. This suggests that a higher sample flow rate is less prone to positive artifacts in the QBQ configuration. It seems that QBQ configuration tends to overestimate POC.

4. CONCLUSION

Filter-based sampling and denuder-based sampling approaches were employed to examine the impact of positive artifacts on the determination of particle phase organic carbon (POC), the effect of sample flow rate on positive artifacts, and an optimum flow rate that provides a minimized negative sampling artifact for the filter- and denuder-based sampling approaches. It was found that Q-alone sampling configuration showed a higher POC than QBQ, Q-QBT, and Denuder-Q configurations by 12%, 28%, and 23%, respectively at a sample flow rate of 20 LPM due to no correction for positive artifact caused by adsorption of gas-phase OC (GOC) onto the filter. This implies that no correction for the positive artifact results in overestimation of organic carbon fraction in PM mass, which can also affect source apportionment calculation. A lower quantity of GOC was collected from the backup quartz filter on QBQ than that from Q-QBT. This was due to

the difference in the adsorption capacity between quartz filter and Teflon filter and GOC being not in equilibrium with that adsorbed on the front quartz filter of QBQ during the sampling period. The loss of particle number and mass across the denuder increased according to sample flow rate. The contribution of positive artifacts to POC decreased with increasing sample flow rate, showing 29%, 25%, and 22% for 10, 20, and 30 LPM, respectively. The 20 LPM turns out to be the optimum sample flow rate for both filter and denuder-based POC sampling.

DISCLAIMER

The statements and opinions expressed here are solely the authors' and do not represent the official position of the California Air Resources Board. The mention of trade names, products, and organizations does not constitute endorsement.

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