

Evaluation of Bias in the Measurement of Condensable Particulate Matter with Method 202

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ABSTRACT

The present study evaluated the positive and negative biases in measurements of condensable particulate matter (CPM) conducted according to U.S. EPA Method 202. To reduce the overestimation of CPM, four factors were investigated: the SO₂ absorption, condensate volume, oxygen content, and residence time. The underestimation was assessed by examining the weight loss related to the electrostatic charge of the evaporation beaker, vapor pressure of the particles, volume of the rinsing solvent, size of the evaporation beaker, and leakage between the filter paper and its holder. Additionally, we constructed a forced ventilation chamber to decrease the drying time of the sampled CPM's organic fraction. The results revealed that our homemade condensate-diverting impinger could neither eliminate nor reduce the artifacts produced by SO₂, as the SO₂ had already oxidized by the time it passed through the Graham condenser and interacted with water. Since the residence time, condensate volume, and oxygen content can all exacerbate the overestimation of CPM, field sampling should be performed as quickly as possible. Furthermore, the evaporation beaker should be electrostatically neutralized prior to its weighing. Nitrogen purging may reduce the mass of the particle fraction possessing high vapor pressure, as these substances are highly volatile. Also, we found that when we used a smaller beaker, less residual mass remained in it after transferring the CPM sample to the weighing tin. We measured a 4% loss in particles due to the gap between the filter paper and the filter paper holder provided by the original manufacturer; therefore, a gasket to minimize leakage is recommended. Finally, the organic fraction required only 1.5–2.5 h of drying time when it was placed in the forced ventilation chamber, and a sample recovery rate of > 98.5% was subsequently achieved. Although artifacts produced by SO₂ are inevitable, the improvements we suggest can enhance the precision of PM_{2.5} measurements.

Keywords: Condensable particulate matter, SO₂ artifact, Method 202, Stack sampling

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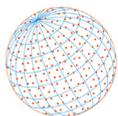
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1 INTRODUCTION

Particulate air pollutants can severely affect the environment, economy, and health (Ramanathan *et al.*, 2007; Apte *et al.*, 2015; Xie *et al.*, 2016). They can travel across large areas and countries, leading to regional or global air pollution (Yang *et al.*, 2008; Karthik *et al.*, 2017). Particulate matter emitted from the stationary sources is one source of artificial particulate pollutants and has a high concentration and complex composition, making it necessary to attain an improved understanding of emission composition (Wang *et al.*, 2016). Particulate matter can be divided into filterable particulate matter (FPM), which exists in the solid or liquid phase in stack flue gas, and condensable particulate matter (CPM), which exists in the gas/vapor phase in stack flue gas. As CPM leaves the stack, it cools down in the atmosphere and condenses to form solid or liquid particulate matter.



Currently, almost all developed countries have established limits for FPM concentration and emission. However, the concentration of CPM is often higher than that of FPM for different stationary sources (Corio and Sherwell, 2000; Miller, 2015; Pei, 2015; Wang *et al.*, 2018; Yang *et al.*, 2018a; Zheng *et al.*, 2018; Yang *et al.*, 2019). For example, the concentrations of CPM are 1.03–2.87 times higher than that of FPM_{2.5} for coal-fired power plants (Yang *et al.*, 2014; Li *et al.*, 2017; Yang *et al.*, 2018b). It is found that CPM is composed mostly of inorganic matter (Corio and Sherwell, 2000) and water-soluble ions such as SO₄²⁻ are main contributors of inorganic component (Qi *et al.*, 2018; Wang *et al.*, 2018; Yang *et al.*, 2018b). Overall, data on the emission of CPM remains limited. Therefore, a thorough investigation of the emission characteristics and concentrations of CPM is urgently required. Stack exhaust temperature is the basis of theoretical definitions of CPM and FPM at the time of sampling, and is the key factor affecting CPM concentration (Yang *et al.*, 2014). The CPM measurement method follows the principle of condensation, reducing the temperature by ice bath cooling or clean ambient air dilution. The cooling sampling method is a direct technique for sampling CPM; the process involves filtering flue gas with filter paper to remove FPM and then cooling the filtered gas to 20–30°C. Once it falls within this temperature range, the gaseous CPM transforms into liquid or solid particles. The impinger and filter paper are then used to capture CPM. Nitrogen is used to purge the sampling system, mainly for removing SO₂ that was likely produced during combustion.

Even though the measurement method for CPM was first promulgated in 1991 and some revisions were made later (U.S. EPA, 2016), its accuracy is often questioned because of high variation. For example, SO₂ often interferes with sampling results, resulting in overestimation (Richards *et al.*, 2005; Wang *et al.*, 2020). The present study involved evaluating the biases and imprecision of the CPM measurement method and proposing measures to improve the overall method performance.

2 METHODS

In this study, an experimental system was set up to simulate CPM sampling and measurement in the laboratory for assessing positive and negative biases (Fig. 1). The testing parameters adopted are shown in Table 1. SO₂ was generated using a standard gas cylinder at a concentration of 100 ppm, which falls in the range of most major stationary sources' SO₂ emission standard. In addition, SO₂ can be easily detected at this concentration. The experimental CPM sampling system used an impinger as specified in Method 202 (Method 202 System; Apex Instruments, Fuquay-Varina, NC, USA), with a sampling flow rate of 10.7 L min⁻¹. In addition, a homemade condensate-diverting (CD) impinger was designed to replace the standard modified Greenburg-Smith (GS) impinger as specified in Method 202 in order to minimize the contact time between SO₂ and water. A funnel was added to the CD impinger to divert the condensate out of the impinger and to reduce the time of contact between SO₂ and the condensate. An SO₂ detector (MultiRAE Lite; Honeywell International Inc., NJ, USA) was calibrated and used to directly measure SO₂ concentration downstream of the GS impinger. The measurements were used to determine the water absorption curve for SO₂. A known volume of water in the range of 100–300 mL was directly added into the GS impinger to simulate the effect of condensate volume on the artifact during sampling. For the evaluation of the CD impinger, water and SO₂ was injected using an injection pump (KDS 200/200P Legacy; KD Scientific Inc., Holliston, MA, USA) into the Graham condenser while simultaneously sampling SO₂. The water injection rate was 1.6 mL min⁻¹, the total water volume injected was 100 mL, and the sampling time was 60 min, which is equivalent to the parameters of a stack with a water content of 21.3%.

Air and high-purity nitrogen were used to assess the effects of purging time and oxygen concentration on the artifact at the time of purging. The wait time between the end of the sampling process and the beginning of nitrogen purging was determined. Two sets of experiments with a total operation time (residence time) of 3 h each were conducted to investigate the effect of wait time, one with a sampling time of 0.5 h and wait time of 2.5 h and the other with a sampling time of 3 h. The Greenburg-Smith impinger was used to evaluate the recovery efficiency of different volumes of hexane. Glycerol and diethylhexyl sebacate (DEHS) were used to simulate organic and inorganic CPM, respectively. An atomizer (Collison Nebulizer;

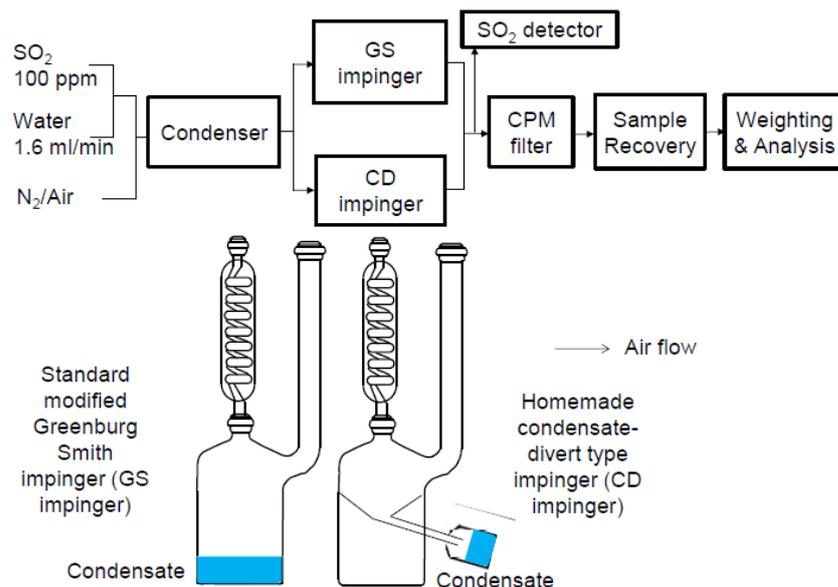
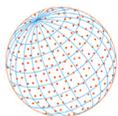


Fig. 1. Schematic of the CPM measurement system.

Table 1. List of operating parameters.

Parameter	Unit	Abbreviation	Range
SO ₂ conc.	ppm	–	100
Adsorbent (water)	mL	–	100–300
Carrier gas	–	–	Air, N ₂
Flow rate	L min ⁻¹	Q _s	10.6
UHP N ₂ purging rate	L min ⁻¹	Q _n	14
Residence time	h	T	0.083–72
Impinger type	–	–	Homemade condensate-diverting (CD) impinger, standard modified Greenburg-Smith (GS) impinger
Forced ventilation chamber purging rate	L min ⁻¹	Q _p	16.1
Distance from outlet to liquid surface	cm	h	2–11

CH Technologies, Westwood, NJ, USA) was used to generate particles of different sizes to determine the correlation between particle size and purging time.

A condensation nuclei counter (P-Trak 8525; TSI Inc., Shoreview, MN, USA) was used to test the CPM filter holder for leakage. The filter paper selected for CPM collection was polytetrafluoroethylene (PTFE) filter paper (GF-3TM; Apex Instruments) recommended by the original manufacturer, with a diameter of 82.3 mm and a testing flow rate of 10 L min⁻¹. The effect of static electricity on weighing results was evaluated using ²⁴¹Am (370 MBq; TSI Inc.) as a neutralizer, and the effects of different beaker sizes (25, 100, 150, and 250 mL) were also investigated. The condition time was improved using a forced ventilation chamber (Fig. 2), with the following dimensions: length of 30 cm, width of 15 cm, and height of 30 cm. The total flow rate at the inlet was 32.2 L min⁻¹. The flow was divided and exited the system from two outlets, each with a flow rate of 16.1 L min⁻¹. The distance from the outlet to the surface of the sample was varied (2–11 cm) to determine the optimal drying time.

3 RESULTS AND DISCUSSION

3.1 Verification of Positive Bias

3.1.1 Absorption and desorption curve of water for SO₂

To detect positive bias in the measurement of CPM due to SO₂, 100 mL of pure water was placed in the GS impinger, and 100 ppm of SO₂ was aerated in water at a flow rate of 10.6 L min⁻¹.

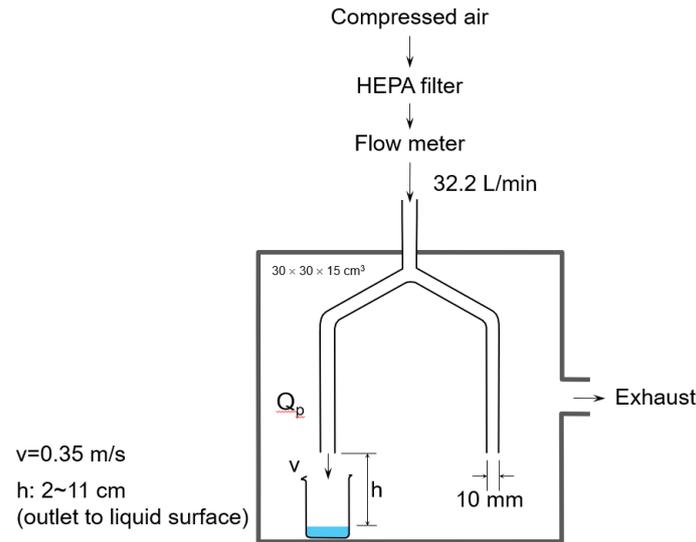
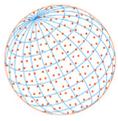


Fig. 2. Schematic of the forced ventilation chamber setup.

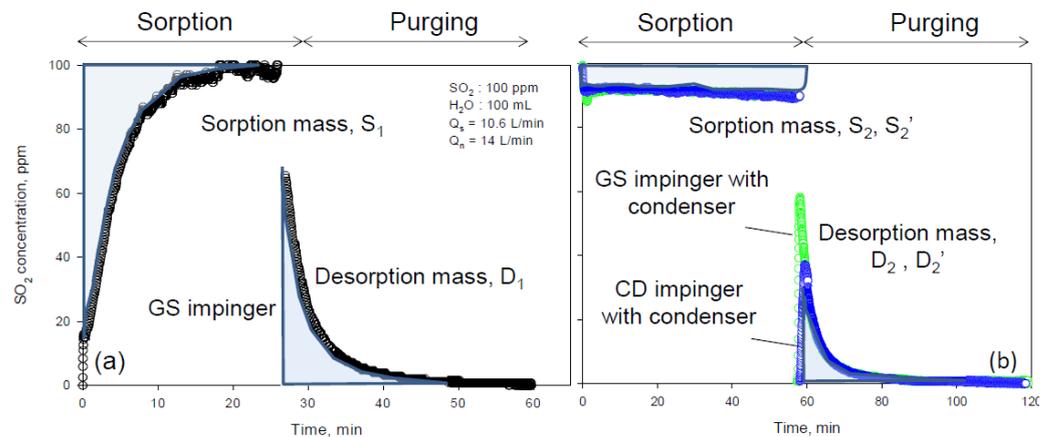
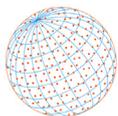


Fig. 3. Sorption and desorption of SO₂ in water.

The results are shown in Fig. 3(a). The absorption curve of water for SO₂ revealed that at the beginning of the experiment, SO₂ could be measured at the outlet of the impinger, and it reached 100% breakthrough in approximately 20 min. The area above the curve is the total amount of SO₂ absorbed by water—approximately 14.9 mg (S_1). Nitrogen purging was directly and immediately applied to the impinger when breakthrough occurred. After 25 min, SO₂ could not be measured. The area under the curve was the total amount of SO₂ that could be purged—approximately 9.82 mg (D_1). It can be inferred that the total amount of SO₂ remaining in the water was 5.08 mg—approximately 33.58%. The forced aeration step used here is different from Method 202 to acquire the greatest amount of possible positive bias resulting from that amount of SO₂ absorbed in the water.

To simulate the contact time between water and SO₂ during field sampling measurement, water and SO₂ were injected into the Graham condenser and impinger at the same time. According to the previous result, the contact time of water and SO₂ is the key point of positive bias. In this experiment, a funnel was placed in the impinger to divert the condensate outside the impinger and thereby reduce the time of contact between water and SO₂ in the flue gas, thereby reducing the amount of artifacts. The results are shown in Fig. 3(b). In terms of the absorption curve, the breakthrough curves in Figs. 3(a) and 3(b) are significantly different because under nonaeration conditions, freshwater was injected continuously into the condenser, thus maintaining contact with SO₂. Therefore, the SO₂ concentration measured at the impinger outlet remained stable, and approximately 10% of the SO₂ could be absorbed. However, the difference



in the mass of SO₂ absorbed in the water between GS impinger (14.99 ± 2.9 mg [S₂]) and CD impinger (15.94 ± 3.56 mg [S₂']) is negligible. Nitrogen purging was performed after the SO₂ concentration reached 100% breakthrough. The mass of SO₂ purged from the water by the GS impinger and CD impinger was 8.34 ± 1.16 mg (D₂) and 9.86 ± 2.19 mg (D₂'), respectively, and the residual SO₂ mass in the water was 5.30 ± 2.61 mg and 6.59 ± 2.59 mg, respectively. The residual percent was 35.29 ± 10.75% and 36.89 ± 11.46%, respectively. The amount of SO₂ artifacts removed through purging also did not differ significantly between the two impingers. A possible reason for this finding is that SO₂ absorption in the water occurred when water was passing through the condenser, even if only for 10 s. Thus, when the gas and liquid were separated by the funnel in the CD impinger, the expected results could not be achieved. Future efforts should be made to reduce the time and area of contact between water and SO₂ to minimize the formation of artifacts.

3.1.2 Effects of sampling time on positive bias

During field sampling, nitrogen purging was not performed immediately after the CPM collection step. The absorption of SO₂ in the water may persist until the completion of other sampling steps. Therefore, the total duration of sampling (aeration) and waiting was the main factor affecting the positive bias caused by SO₂. The residence time was defined as the sum of sampling and wait times. A 3-h experiment was designed to simulate the residence time before the nitrogen purging of field sampling. In one set, aeration lasted for 3 h with no wait time and the SO₂ recovery ratio was 40.2%, whereas in the other set, aeration lasted for 0.5 h with a 2.5-h wait time and the SO₂ recovery ratio was 41.7%; the difference was not significant. Therefore, the factor affecting the SO₂ recovery ratio was the residence time: Neither sampling time nor wait time alone affected the recovery ratio. In general, the shorter the duration of field sampling, the more SO₂ could be removed through nitrogen purging. At the residence times of 5, 27, 32, 150, 566, 2880, and 4320 min, the recovery efficiency of SO₂ was 99.5%, 66.4%, 61.7%, 45.5%, 39.8%, 37.7%, and 38.0%, respectively. The reason for this is that the longer the residence time, the more SO₂ dissolves in the water and forms SO₃²⁻ and SO₄²⁻, leaving less SO₂ to be purged and reducing the amount of time required for nitrogen purging.

3.1.3 Effects of oxygen content and water content on positive bias

The formation of SO₃²⁻ and SO₄²⁻ from SO₂ requires oxygen. Thus, oxygen concentration is also a factor causing CPM positive bias. A fixed time (150 min) was set for two experiments that used air and nitrogen as a carrier gas; the SO₂ recovery ratio was 45.5% and 58.9%, respectively. When air was used for dilution, the oxygen in it allowed SO₂ to easily oxidize and form sulfuric acid, thus increasing the positive bias of the SO₂. In other words, in actual sampling, the higher the oxygen content in the flue gas, the easier it is to overestimate the CPM results.

The stack moisture content and the sampling time affect the moisture content in the impinger. The positive bias caused by 100 ppm of SO₂ at different water quantities (100, 200, and 300 mL) is shown in Fig. 4(a). The amount of SO₂ dissolved, the amount of SO₂ that nitrogen has purged, and the residual amount of SO₂ all increased as the amount of water in the impinger increased, and a linear correlation was observed. Irrespective of the water volume, the ratio of the amount of SO₂ dissolved to the amount of SO₂ that nitrogen has purged was approximately 65%. Thus, the moisture content of the flue gas may lead to overestimation of the sampling results, and when the amount of water increases, although the proportion of SO₂ purged and the amount of residue are almost identical, the mass of the residue is higher than that of the purged SO₂. The more water in the stack, the higher the residual SO₂ mass in the water, and the ratio of overestimation is directly proportional to the amount of water. The results are shown in Fig. 4(b).

3.1.4 Beaker weighing bias

Weighing bias due to static electricity should be avoided. This study explored the effect of electrostatic interaction on beaker weighing for different beaker sizes (Fig. 5). Larger beakers carry more static electricity and are thus more influenced by static electricity during weighing. It may be speculated that frictional charging occurs when the hand touches the beaker and the electric charge of the hand is transferred to the beaker. Larger beakers have more surface area

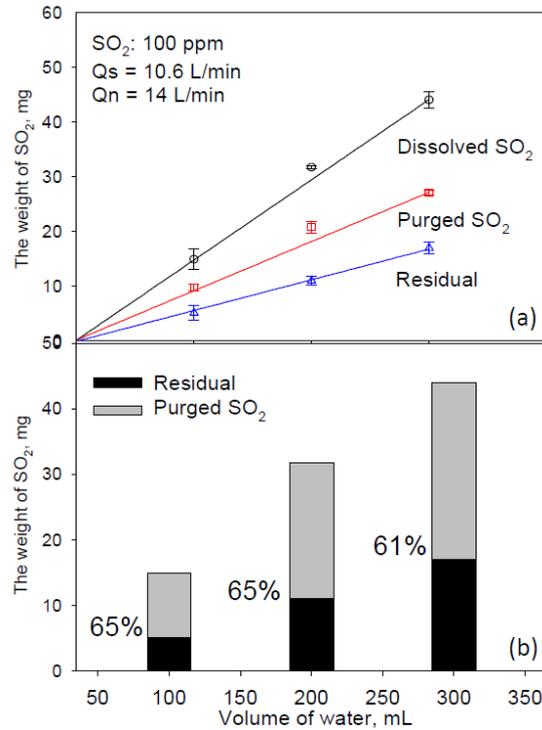
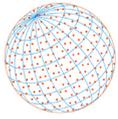


Fig. 4. Effect of water volume on SO₂ recovery and residual amount.

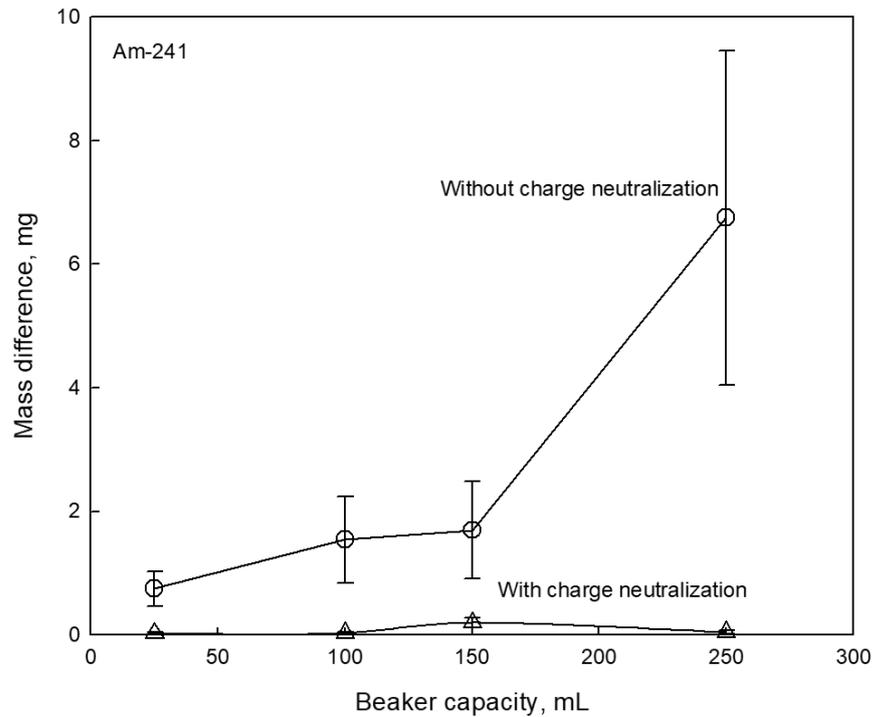
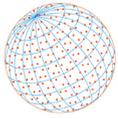


Fig. 5. Effect of electrostatic charge on beaker weighing.

to carry charges. For a 100-mL beaker, the bias caused by nonstatic neutralization can reach 1.8 mg. If the sampling volume is 1 Nm³, then the bias caused by weight concentration is 1.8 mg Nm⁻³. The experiment results indicate that when the weighing container is a poor conductor (e.g., glassware), thorough performance of the electrostatic neutralization step is necessary.



3.2 Verification of Negative Bias

3.2.1 Effect of nitrogen purging on filter paper collection samples

Method 202 involves the use of the nitrogen purging procedure to reduce the positive bias of SO₂ in flue gas, but the CPM collected on the filter paper behind the impinger may cause weight loss during nitrogen purging. In the experiment, glycerol and DEHS were used as CPM with different vapor pressures. The weight loss after nitrogen purging is shown in Fig. 6. The original weights of glycerol and DEHS on the filter paper were 8 and 5.5 mg, respectively. The weight of DEHS did not change after tests with different purging times, but that of glycerol decreased with the increase in purging time. Three particle sizes (1.17, 5.05, and 1178 μm) were employed in this study. Particles with the size of 1178 μm were administered onto the filter paper by using a pipette. The data indicated that the particles with a smaller diameter had larger surfaces and lost weight more easily for the same mass. If nitrogen purging according to this method lasted for at least 1 h, then the percentage of weight loss of the glycerol particles would be between 25% and 50% for particle sizes of 5.05 and 1.17 μm, respectively. However, no significant reduction was observed in the mass of particles with a diameter of 1178 μm because the coverage area on the filter paper was smaller than that for the other two particle sizes (1.17 and 5.05 μm). The volatility of CPM is directly proportional to the vapor pressure at the same temperature. Because glycerol has a saturated vapor pressure (1.68×10^{-4} mmHg at 25°C) that is lower than that of DEHS (10^{-7} mmHg at 25°C), it is the more volatile of the two. Moreover, the weight of substances with a vapor pressure of $< 10^{-7}$ mmHg was unaffected by nitrogen purging. This may be related to the surface area or coverage rate of glycerol on the filter paper.

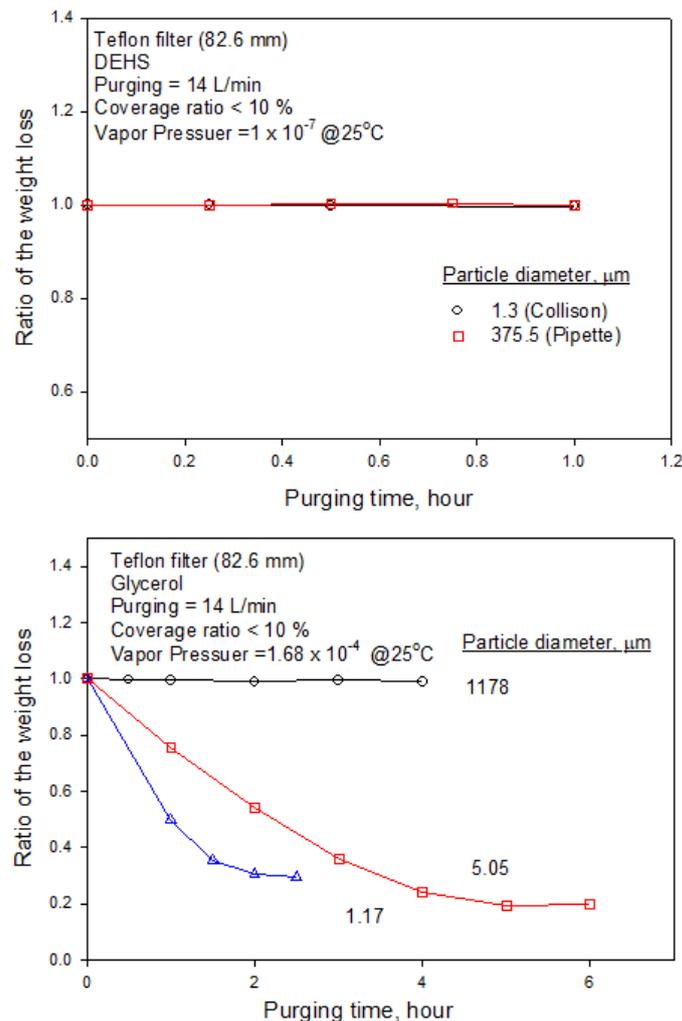
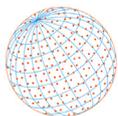


Fig. 6. Effect of purging time on the weight loss for different volumes of glycerol and DEHS.



In the experiment, filter paper was loaded with particles of different weights but the same size to evaluate the effect of mass loading. For glycerol particles with a diameter of 1.17 μm , after 2 h of nitrogen purging with different coverage rates (80%, 50%, 40%, and 10%), the weight loss of glycerol particles was 16%, 30%, 34%, and 65%, respectively. A lower coverage rate resulted in fewer particles on the filter paper. The rate of weight loss of the particles on the filter paper decreased faster with nitrogen purging. The larger the particle numbers, the higher the probability of deposition on the same location; consequently, only the amount of surface CPM decreased with nitrogen purging, and the relative rate of particle weight reduction also decreased. Therefore, the proportion of underestimation caused by nitrogen purging increases at low concentration of CPM. Replacement of the filter paper before nitrogen purging may prevent weight loss.

3.2.2 Effect of solvent volume on recovery efficiency

Rinsing is required for recovery of the CPM adhered to the impinger inner surface. The recovery efficiency of DEHS when different rinse volumes of hexane were used is displayed in Fig. 7. When the DEHS mass in the impinger was 0.45 g, the addition of 35 mL of hexane to the impinger for rinsing achieved 95% recovery efficiency, but the addition of 50 mL of hexane only increased recovery efficiency by an additional 1%, that is, to 96%. Increasing the eluent volume can increase the recovery efficiency, but it also increases the drying time later during the analysis procedure.

After the extraction step in the analysis procedure of Method 202 was performed, the inorganic and organic fractions of the solution were required to be evaporated to approximately 10 mL and then transferred to a 50-mL pre-tared weighing tin. During transfer, the CPM solution may leave some residue in the previous beaker, which would introduce bias in the results. The results are presented in Fig. 8. The residual ratio remaining in the beaker increased as the beaker size and CPM solution volume decreased. For example, with a 150-mL beaker, when the volume of the CPM solution after drying was 5, 10, or 20 mL, the residual amount in the beaker after the solution was transferred to a tin (5.72%, 2.22%, and 0.91%) was progressively smaller because the solute was nonvolatile. Some solutions adhere to the inner surface of the beaker during transfer; hence, the beaker size also plays a vital role. Transferring smaller volumes results in greater underestimation because of the higher residual and transfer volume percentages. Smaller amounts of solution transferred also indicate higher CPM concentrations because the solvent was volatile. Fig. 8 reveals that the residual ratio was the highest when the volume of residue in the beaker was 5 mL, regardless of the beaker capacity, leading to an underestimation of approximately 2–6%. In Method 202, performing the transfer step when the beaker is holding 10 mL of content would lead to a 1–3% underestimation. Therefore, if the total cumulative weight of the extract and the beaker does not exceed the measurable range, then the extract transfer step should not be performed or the mass of the residue in the beaker should be added to the result after transfer to avoid underestimation.

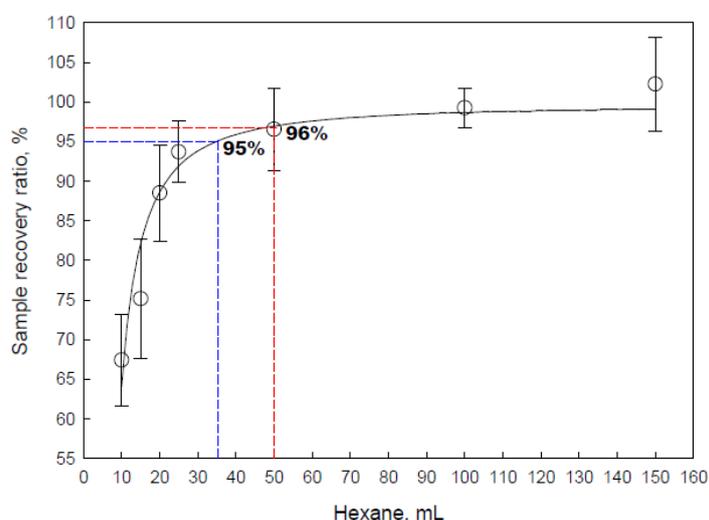


Fig. 7. Effect of the amount of hexane on the sample recovery ratio.

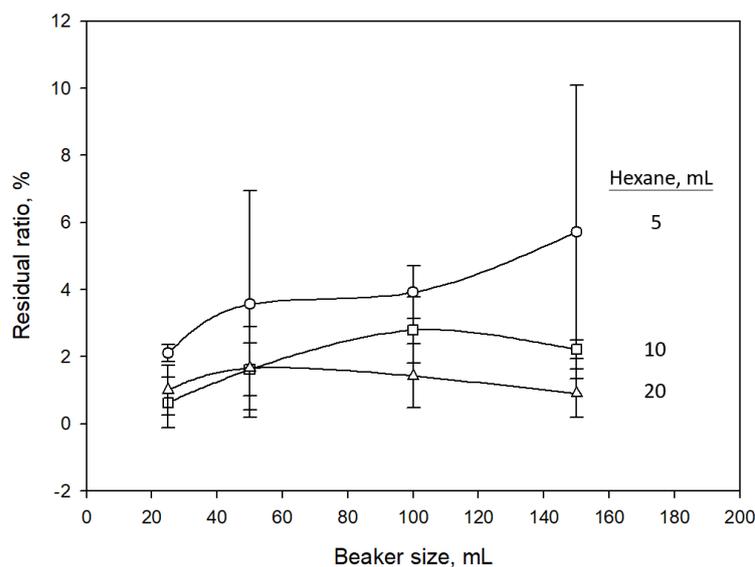
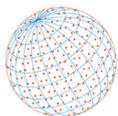


Fig. 8. Effect of the amount of hexane on the residual ratio of beakers with different capacities.

3.2.3 Filter holder leakage

The filter paper behind the impinger was used to capture the CPM escaping from the impinger, but the particles passed through the gap between the filter paper and the filter paper holder, resulting in underestimation of the total mass. The results are shown in Fig. 9. The percentage of particle loss due to this leakage was 4%. After the gap was filled with a gasket, the particle loss was <0.06%. Therefore, attention must be paid to the tightness of the filter paper and the holder in use, and if necessary, an O-ring or gasket must be inserted to increase the air tightness.

3.3 Improvement of Sample Drying Time

The analysis procedure requires evaporation of the organic extract until it is dry at room temperature in a chemical hood, but it takes several days to fully dry, whereas an inorganic extract must be baked on a hot plate or in an oven at 105°C. In this study, a forced ventilation chamber was designed to reduce the drying time of the residue; the results are discussed separately for the organic and inorganic extracts.

3.3.1 Organic extract

DEHS (0.22 g) and hexane (100 mL) as the solute and solvent, respectively, were added to a beaker (4.7 cm in diameter, volume of 100 mL), which was placed in a chemical hood following completion of the procedures for Method 202. Fig. 10 compares the time required for full implementation of the method with that required for using a forced ventilation chamber. It took approximately 100 h to dry the organic extract in the chemical hood. In the forced ventilation chamber, however, when the distance from the air outlet to the liquid surface was 2 cm, it took only 1.5 h for the sample to dry; at a distance of 5 cm, it took only 1.75 h; and at a distance of 11 cm, it took 2.5 h. These results indicate that the distance should be as short as possible to reduce the drying time. However, disturbance of the solution surface should be avoided at short distances. The results of sample drying with a forced ventilation chamber are consistent with those of sample drying with a chemical hood, reaching 98.5% recovery efficiency. Therefore, the organic fraction can be dried using the forced ventilation chamber, thus effectively reducing the drying time.

3.3.2 Inorganic extract

During the experiment on the inorganic extract, 0.22 g of NaCl and 100 mL of water were placed in two beakers that had been weighed. One beaker was placed in an oven at a temperature of 105°C, and the other was placed in a forced ventilation chamber; it took 2 and 5 h, respectively, for the samples to dry. The drying time was not reduced in the ventilation

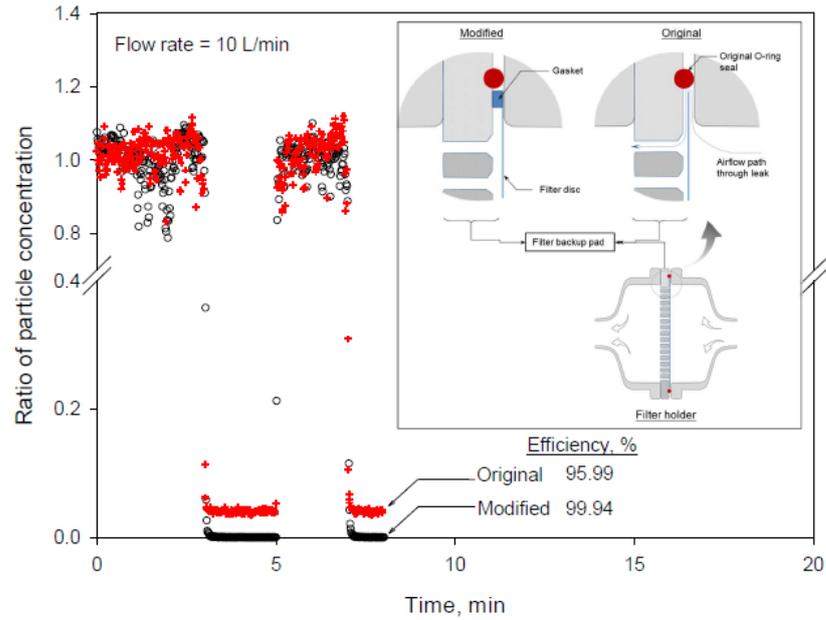
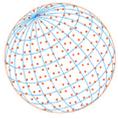


Fig. 9. Effect of O-ring leakage on the recovery efficiency of the CPM filter.

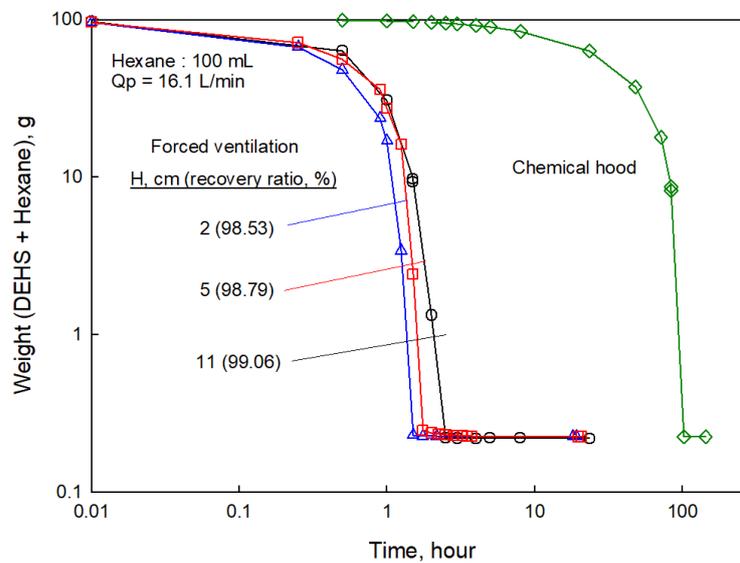


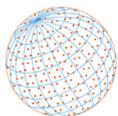
Fig. 10. Effect of conditioning on the weight of the organic sample.

chamber for inorganic samples. Weighing the beaker after drying, whether in an oven or a forced ventilation chamber, led to > 99% recovery of the extract.

In the experiment for improving the conditioning time of the sample, the drying time could be effectively reduced using a forced ventilation chamber because the gas exchange rate could be improved. Nevertheless, water condensation may develop on the beaker's outer surface because of temperature reduction due to evaporation, and it is necessary to remove this condensation before weighing to avoid overestimation of CPM. The drying time for organic samples, but not for inorganic samples, can be reduced using a forced ventilation chamber.

4 CONCLUSIONS AND RECOMMENDATIONS

This study evaluated the sources of positive and negative measurement biases in Method 202. The main findings are as follows:



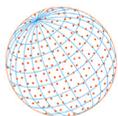
- 1) Artifacts due to the water's absorption of the SO₂, which contribute to positive bias, cannot be avoided as long as condensation occurs.
- 2) Reducing the duration of contact between the SO₂ and the water can decrease the positive bias.
- 3) Replacing the filter paper prior to purging the CPM sample with nitrogen can prevent particle weight loss via evaporation, but it also increases the complexity of the sample and hence the analysis. Lengthening the purging time does not increase the recovery efficiency of the dissolved SO₂ and may cause significant underestimation.
- 4) The gap between the filter paper and its holder can cause CPM loss; thus, an O-ring or gasket should be inserted to enhance the air tightness.
- 5) To reduce the amount of residue (to 0.09%), the aqueous-phase fraction should be evaporated in the beaker till its volume is no more than 20 mL. Alternatively, the residual CPM can be weighed in the beaker both before and after transferring the latter's contents.
- 6) When the weighing container is a poor conductor of electricity (such as glassware), it should be electrostatically neutralized prior to being weighed.
- 7) Using forced ventilation can greatly reduce the drying time of the organic (but not inorganic) fraction, which decreased from 100 to 2.5 h in our experiment.

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