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Performance evaluation of disposable inhalable aerosol sampler at a copper electrorefinery

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Abstract

The performance of the disposable inhalable aerosol sampler (DIAS), newly developed to be cost-effective and comparable to the inhalable particle sampling convention, was evaluated. Forty-eight sets of copper exposure measurements (23 personal and 25 area) with pairs of the DIAS prototypes and the IOM inhalable samplers were collected at an electrorefinery facility. For the combined data (personal and area), the geometric mean (GM) concentrations of copper were 36.1 μg m^{-3} (range of 0.8 – 337.7 μg m^{-3}) for the DIAS prototype and 26.5 μg m^{-3} (range of 5.1 – 760.8 μg m^{-3}) for the IOM sampler. The GM ratio of exposure measurements (DIAS/IOM) was 1.4 revealing ~ 81% of the ratios greater than the unit ratio. The concordance correlation coefficient tests revealed significant disagreement between the two types of samplers and suggested precision as the source of the disagreement. For the personal, area and combined data, no linear relationships were observed between the DIAS and IOM (all p-values < 0.05). In addition, the estimated average concentrations were always higher for the DIAS compared to the IOM for the combined and area exposure data (p-values < 0.05) and comparable for the personal exposure data (p-value = 0.487). Overall, regardless of the sampling method (i.e., personal and area), the DIAS generated higher exposure measurements compared to the IOM sampler. The present study is limited to one metal component (copper) of the dust at a worksite. As far as we are concerned, this is only the second field evaluation of the DIAS. Thus, it is too early to draw a firm conclusion about the performance of the DIAS vis-à-vis the inhalable convention. Additional field evaluations covering various chemicals and worksites will be necessary. In addition, particle losses on the cap during transportation of collected samples to a laboratory were observed for both

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sampler types. Occupational professionals should be aware of potential errors caused from transferring samples from a field to a laboratory and should be careful not to exclude particles collected on the caps.

Keywords

Disposable inhalable aerosol sampler; copper electrorefinery; IOM sampler; sampling pairs; transportation loss

INTRODUCTION

In the United States, three types of samplers, the IOM, the Button, and 37-mm closed-face cassette (CFC, more widely known as a “total” dust sampler), are frequently used to determine airborne particle concentrations of the inhalable fraction. Among these samplers, the IOM and Button samplers have shown sampling efficiencies comparable to the inhalable particle size-selective sampling convention adopted by the International Standards Organization (ISO, 1995), the European Standardization Organization (CEN, 1993), and the American Conference of Governmental Industrial Hygienist (ACGIH) (ACGIH, 2015). However, these two samplers are expensive and thus constrain occupational professionals to collect minimal numbers of samples from many workers who are potentially exposed to inhalable particles. In contrast, the CFC sampler is considerably cheaper than the other two inhalable samplers, but it is known that this sampler underestimates particles larger than 30 μm in aerodynamic diameter (Kenny et al., 1999; Görner et al., 2010).

In addition, problems originating from particle deposition on the interior walls of sampler have been raised by various researchers (Demage et al. 1990; Ashley and Harper 2013; Harper and Ashley 2013; Andrews et al. 2016; Lee et al., 2014). Sample analysis solely depending on the filter, (i.e. omitting wiping the interior walls and analyzing that dust with the filter catch) might lead to false results (i.e., underestimation of exposures). Thus, for chemical analyses, wiping of the internal wall is recommended as reflected in the National Institute for Occupational Safety and Health (NIOSH) Manual of Analytical Methods (NMAM) 5th Edition, Chapter AE and in other methods of elemental analysis of dusts and aerosols (e.g., NIOSH 7302 and 7303 methods). Weighing of the internal capsule holding the filter in the IOM sampler would prevent the loss of particles deposited on the walls, unlike the other inhalable samplers. Using the similar concept, CFC samplers holding internal capsules, such as Accu-Cap™ internal capsule (SKC, Inc., Eighty Four, PA, USA) and Solu-Sert™ filter capsule (Zefon International, Inc., Ocala, FL, USA), are commercially available, but again this sampler is designed to collect total dust rather than the inhalable fraction

To address the limitations of the aforementioned issues, L’Orange et al. (2016) developed a new, low-cost disposable inhalable aerosol prototype sampler (DIAS). This prototype sampler has features that are similar to both the IOM and CFC samplers. Similar to the IOM, the sampler has a round, 15-mm inlet and an internal capsule to accommodate wall losses. Similar to the CFC, the sampler has fewer components (inlet cover, inlet, capsule and filter and housing). L’Orange et al. tested it in a wind tunnel at a flow rate of 2 L min^{-1} and

confirmed that the sampling efficiency of this new sampler matched with that of the IOM sampler. Stewart et al. (2017) conducted a side-by-side test of the new sampler at two different flow rates (10 L min^{-1} and 2 L min^{-1}) to determine if this sampler can be also used for a task-based sample collection with detectible amount of mass (i.e., increasing sample mass by reducing sampling time). Stewart et al. tested with four different particle sizes of alumina oxide powders (4.9 μm, 9.5 μm, 12.8 μm and 32.7 μm) in a wind tunnel operating at 0.2 m sec^{-1} and reported no significant difference between the samplers' performances. Anthony et al. (2017) compared the performance of the DIAS prototype sampler against the IOM sampler by collecting area exposure measurements in a livestock production facility. They reported that the DIAS generated comparable results for the inhalable dust concentrations and higher inhalable endotoxin concentrations compared to the IOM sampler. Based on the previous laboratory studies (L'Orange et al., 2016; Stewart et al., 2017), it seems that this new disposable inhalable sampler is a promising surrogate for the IOM sampler. However, only one field evaluation based on the comparison of area exposure measurements was performed (Anthony et al.). No performance testing of the DIAS for personal exposure measurements has been conducted to date.

Therefore, the objective of this study is to evaluate the performance of the DIAS by comparing exposure measurements of copper with those collected from the IOM sampler (reference) at an electrorefinery facility. Personal and area exposure samples using side-by-side sampling pairs were collected and analyzed for copper content.

METHODS

Exposure measurements

A field survey was conducted at a copper electrorefinery facility. Tasks involved in this facility were loading/unloading large racks of copper cathodes and anodes from the electrolyte tanks, washing copper cathodes down with hoses, processing finished cathodes, and preparing cathode starter sheets for electrorefining. Sampling took place over the course of four days during the summer; temperatures ranged from 30°C to 47°C during the day in the tankhouse, with an average relative humidity of 64%. The facility was dependent upon natural ventilation (i.e., opening entrances and windows) along with fans, sporadically placed and turned on and off at irregular intervals. Photos were prohibited to protect production trade secrets. Typically, as with most copper refineries, workers in this facility were exposed to airborne metals including copper, arsenic, silver, lead, and selenium.

We collected 48 paired exposure measurements (23 personal and 25 area) using the IOM and the DIAS. Personal exposure measurements were collected from the following job categories: stripper crane man, cleaning stripper, cellar man, scrap operator, scrap washer, crane operator, and supervisor. Area sampling was utilized in aisles between electrorefining tanks and sections in which the acidic electrolyte was heated to a prerequisite temperature and pumped throughout the tankhouse. A mixed-cellulose ester (MCE) filter (37-mm for the prototype and 25-mm for the IOM) with 0.8 μm pore size was mounted in the sampler. In addition, the DIAS has a capsule (made of thin-film polycarbonate) attached to the MCE filter. In previous studies tested this prototype (L'Orange et al., 2016; Stewart et al., 2017; Anthony et al., 2017), the perimeter of a capsule's base was attached to a filter using toluene

(i.e., as glue) so that the capsule and filter could be weighed together. In the present study, we did not glue the capsule with the filter because the capsule material could not be dissolved. Instead, we wiped the internal walls of the sampler to conduct wet chemical analyses. Exposures to airborne particles were collected at 2 L min^{-1} for both sampler types with sampling times ranging from 256 to 529 minutes. We placed samplers randomly on the opposite sides of the worker's torso (i.e., one on the left lapel and one on the right lapel). The sampling pumps were calibrated prior to the sampling and checked after the sampling with a DryCal® DC-Lite device (BIOS International Corporation, Butler, NJ, USA) to ensure the nominal flow rate of 2 L min^{-1} ± 5%. Nine field blank samples for each sampler type were collected.

Sample Analysis

For each IOM sampler, a filter was removed with tweezers and placed in a tube. After removing the filter, we wiped the interior surfaces of the filter holder twice with a quarter-sized clean MCE filter, soaked in isopropyl alcohol, and placed in another tube. Then, the inside of the cap was wiped twice with another quarter-sized MCE filter and placed in a third tube. For each DIAS, the same steps as for the preparation of IOM samples were repeated for separate analyses. These tasks were done by one lab personnel to minimize the variations among different lab personnel for the wiping procedures.

All collected samples were analyzed according to the NIOSH NMAM 7303 method, with some modification. In order to dissolve the MCE filters, a solution of 4 ml concentrated nitric acid and 1 ml 20% hydrogen peroxide was used. After dissolving an MCE filter, water was then added to a total volume of 40 ml and 1 ml of this solution was pipetted into a 15 ml tube to which was added a water solution of 1 % nitric acid and 1% ethanol containing 10 ppb of Yttrium (Spex Claritas standard solution). Calibration solutions were made by diluting a copper standard (Spex Claritas) solution of 1% nitric acid, 1% ethanol, and 10 ppb Yttrium. Calibration solutions included method blanks and copper concentrations ranging from 1 to 100 ppb. All solutions were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS; Perkin Elmer 300D, Waltham, MA, USA). In this study, we initially obtained airborne metal concentrations including copper, arsenic, silver and selenium. However, only copper concentrations were used for further data analyses since the amount of mass determined for the other metals were less than the limit of quantitation (LOQ) for most sample pairs when analyzing the wiped samples from the interior surfaces and/or inside the cap. The LOQs are 158.21 ng for copper. For copper, 288 samples (48 pairs x 2 sampling types x 3 tubes [filter, internal wall wipe and cap wipe] for each sample) were analyzed. Only 3 samples (1 filter and 2 cap wipes) from the DIAS showed analyzed masses less than the LOQ. All field blank samples (including filters and wiped samples from the interior walls and inside the cap) showed masses less than the LOQ and thus, no subtraction of the field blank mass was conducted.

Data Analysis

The analyzed masses from filter, internal wall wipe and cap wipe were combined to calculate total mass concentrations for each sample. Prior to conducting data analyses, all exposure measurements were log-transformed to meet the assumptions of the statistical

tests. We performed data analyses with two data sets, exposure data with LOQ treated by replacing mass below LOQ with LOQ/sqrt(2) and exposure data without LOQ treatment. The results showed no differences of conclusions. Thus, we only reported the results of exposure measurements without LOQ treatment in this paper. In addition, we calculated the proportion of particle mass detected on the cap (determined from wet chemical analysis) and internal walls of each sampler.

For assessing agreement between the DIAS and the IOM, we conducted a concordance correlation coefficient (CCC) test expressing the results as the product of precision and bias coefficients. The precision coefficient represents variation by measuring the distance of each measurement from the best-fit line, while the bias coefficient measures the distance between the best-fit line and the unity line (Lin 1989; Lin et al., 2002; Barnhart et al., 2002; Carrasco and Jover, 2003). Unlike the Bland-Altman test, the results of CCC tests can determine a source of disagreement whether it is from precision or bias. For this test, we used a validated SAS macro provided by Lin et al. (2002). The CCC results were assessed using ± 35% acceptance criterion ($0.878 = [1–0.35^2]$), selected from previous studies to compare different sampler types by Lee et al. (2011) and Lee et al. (2017). Two additional statistical tests were conducted to test 1) if the slope between the DIAS and the IOM sampler is 1 (H_0: slope [β]=1) using Proc Reg procedure and 2) if the average concentration of the DIAS is the same as that of the IOM using Proc Mixed procedure. All statistical analyses were performed with SAS/STAT software version 9.3 (SAS Institute, Cary, NC, USA). A p-value of 0.05 was used for testing the hypotheses.

RESULTS

Comparison of exposure measurements between the DIAS and the IOM samplers

Table 1 shows a summary of exposure measurement data, presenting the combined results of filter samples and wiped samples (interior walls and inside the cap). For the combined data (personal and area), the geometric mean (GM) concentrations were 36.1 μg m^{-3} for the DIAS and 26.5 μg m^{-3} for the IOM sampler. For both sampler types, the average concentrations were considerably higher than the corresponding GM concentrations revealing that the measurement data were positively skewed. The range of exposure measurements was wider for the IOM sampler compared to that for the DIAS. A similar pattern was observed when the exposure measurements were separated by personal and area exposures.

The GM ratio of exposure measurements (DIAS/IOM) was 1.1 for the personal exposures, 1.6 for the area exposures, and 1.4 for the combined personal and area exposures. The average ratio of exposure measurements was not noticeably different compared to the corresponding GM ratio (1.5, 2.1 and 1.8 for the personal, area and combined exposures, respectively). Overall, the variation of area exposure data is similar to that of personal exposure data (coefficient of variation [CV] = 0.7 for the personal and 0.6 for the area exposure data). Figure 1 shows the individual mass concentrations between the pairs of samples for copper. Regardless of the sampling method (personal or area), overall, the DIAS showed higher concentrations compared to the IOM sampler. About 81% of the combined

data (~ 78% of the personal exposures and ~ 84% of the area exposures) showed the concentration ratios (DIAS/IOM) greater than one.

As shown in Table 2, the CCC-total results for the personal, area and combined exposure measurements were lower than 0.878, indicating disagreements between the two types of samplers. Regardless of the sampling method, the CCC-Precision was considerably lower than the corresponding CCC-Bias, suggesting precision (i.e., variation) as the source of disagreement. The CCC-Bias ≥ 0.889 suggests little deviation from the unity line. The results of linear regression analyses to develop slope and test if slope (β) = 1 revealed statistically significant differences of exposure measurements between the two types of samplers: all p-values for the personal, area and combined data were < 0.05 with adjusted R^2 ≤ 0.51. The comparison of exposure measurements between the DIAS prototype and the IOM resulted in no overall statistical difference for the personal exposure data (p-value = 0.487) and statistically significant differences for the combined data (p-value = 0.035) and area exposure data (p-value = 0.031); the estimated average concentrations were always higher for the DIAS prototype compared to the IOM (Table 2).

Loss of particles during the transportation of samples

We obtained the amount of copper mass collected on the cap by analyzing the samples separately to determine the proportion of particle loss due to the transportation of samples to a laboratory. As described in Methods, although we are interested in the proportion of particle mass deposited on the inside of cap, the proportions of particles collected on filter and internal walls were also presented to provide additional information. As shown in Figure 2, the geometric mean percent proportion of mass (cap/total) was similar for both sampler types (6.4% for the DIAS prototype and 7.4% for the IOM sampler). The comparison results of the average proportion are also similar for both sampler types (13.7% for the DIAS prototype and 11.6% for the IOM sampler).

DISCUSSION

Comparison of the exposure measurements between the DIAS and the IOM samplers

The copper exposure measurements for the combined data (personal and area) were 0.8 – 337.7 μg m^{-3} (GM = 36.1 μg m^{-3}) for the DIAS and 5.1 – 760.8 μg m^{-3} (GM = 26.5 μg m^{-3}) for the IOM (Table 1). Regardless of the sampling time ranging from 256 minutes to 529 minutes, none of individual exposure measurements exceeded the occupational exposure limit of 1000 μg m^{-3} by Occupational Safety and Health Administration (OSHA) - Permissible Exposure Limit (PEL), NIOSH-Recommended Exposure Level (REL) and ACGIH-Threshold Limit Value (TLV). For both DIAS and IOM samplers, the GM exposure concentrations were higher for the personal sampling method compared to the area sampling method, leading the difference due to the distance of samplers' location from the emission source.

Overall, the DIAS generated higher exposure data than the IOM sampler. About 81% of sample pairs (39 out of 48 sample pairs) showed the ratios of exposure data (DIAS/IOM) greater than the unity (Figure 1). For the area exposure measurements, the DIAS resulted in

about 1.6 times higher concentrations than the IOM along with the results of disagreement (CCC-Total < 0.878) and statistically significant differences (p-values < 0.05 for the slope test and the comparison of overall average concentrations between two sampler types) (Table 2). Anthony et al. (2017) conducted the performance of the DIAS (running at 10 L min^{-1}) against the IOM (reference, running at 2 L min^{-1}) in a livestock production facility by collecting area exposure measurements; 36 sample pairs of inhalable dust and 44 pairs of inhalable endotoxin were collected. They reported no significant difference of the DIAS compared to the IOM for the comparison of inhalable dust, whereas the DIAS produced higher inhalable endotoxin concentrations compared the IOM. The findings of the present study are inconsistent with the conclusion of inhalable dust but consistent with that of inhalable endotoxin (i.e., overestimation of DIAS) reported by Anthony et al. The sampling flow rate of DIAS utilized in this study was 2 L min^{-1}, not the same as 10 L min^{-1} that Anthony et al. employed. However, because Stewart et al. (2017) reported no difference of mass concentrations of the DIAS between 10 L min^{-1} and 2 L min^{-1}, the difference of sampling flow rates between this study and Anthony et al. might not be a reason for causing the difference of the findings.

For the personal exposure measurements, although the comparison of overall average concentrations between the DIAS and IOM revealed no statistically significant difference (p-value =0.487 > 0.05), the disagreement (CCC-Total = 0.692 < 0.878) and lack of a linear relationship of exposure data between the two sampler types indicates that that the performance of the DIAS is not comparable to that of IOM. The range of exposure measurement ratios (DIAS/IOM) between the personal and area exposure data was similar. Interestingly, the GM value of ratios was higher for the area exposure data compared to that for the personal exposure data (Table 1). It is expected to have a lower GM value of ratios for the area samples compared to the personal samples because the paired samplers located for the area sampling were stationary and thus no influence of exposure concentrations from a worker's behavior. Probably, this high ratio might happen because of abrupt changes in the wind direction from a fan (running on and off, irregular) directing an air stream into the IOM sampler.

As far as the authors are aware, this study is the second field evaluation (after Anthony et al., 2017) using area exposure measurements and the first evaluation using personal exposure measurements for testing the performance of the DIAS. The present study is limited to only one metal component at a workplace with the inhalable exposure range of 5.1 – 760.8 μg m^{-3}using IOM samplers. In addition, there might be some errors arising from the wiping process (i.e., applying inconsistent pressure to wipe internal walls and inside of cap), although the wiping process was conducted by one lab personnel to minimize variations among different personnel. In order to make a firm conclusion about the performance of the DIAS, it will be necessary to characterize the performance of the DIAS in various environments sampling numerous chemical components.

Loss of particles during the transportation of samples

The proportion of copper mass collected on the cap over the total copper mass (i.e., sum of copper masses from filter, internal wall wipes and cap wipes) was similar for both sampler

types (GM [mean] proportions = 6.4% [13.7%] for the DIAS prototype and 7.4% [11.6%] for the IOM), shown in Figure 2. Demange et al. (2002) investigated metal deposits on sampling cassette walls due to transportation disturbances and reported that 1.8% of the sampled mass of barium and 7.9% of the sampled mass of iron were deposited on cassette walls during transportation. On the other hand, Stacey et al. (2013) also checked the weight of respirable dust on 12 filter samples after transporting to Italy and South Africa from the UK but reported no significant differences (the ratio close to 1). Although the methods to determine particle losses due to the transportation were different between the present study and a study by Demange et al., it is obvious that the loss of particles could happen from the transportation of samples. It should be noted that the proportion of particle loss reported in this study is not representative because particle losses from other parts (e.g., from filter to internal walls and/or vice versa), which was not considered here, could happen during the transportation of samples. The findings of this study indicate that occupational professionals should be aware of potential errors caused from transferring samples from a field to a laboratory and be careful not to exclude particles collected on a cap.

CONCLUSION

The Disposable inhalable aerosol prototype sampler, newly developed to overcome the current limitations of inhalable samplers (i.e., developed to be cost-effective and at the same time comparable to the inhalable particle sampling convention), has been evaluated at a copper electrorefinery facility. All personal and area exposure measurements were below the occupational exposure limit of 1000 μgm^{-3} by the OSHA-PEL, NIOSH-REL and ACGIH-TLV. Overall, the disposable inhalable prototype sampler produced higher exposure measurements for copper than the IOM regardless of the sampling method (i.e., personal and area). This is a first field evaluation study to investigate the performance of the new sampler with personal exposure measurements and the second field study with area exposure measurements. It would be too early to make a firm conclusion about the DIAS performance because this study is limited to one metal component at a worksite. Thus, it will be necessary to conduct additional field evaluations covering various chemicals and worksites. In addition, particle loss on the sampler cap during the transportation of collected samples to a laboratory was observed for both DIAS and IOM samplers. Occupational professionals should be cautious when handling of samples by including the particle mass collected on a cap.

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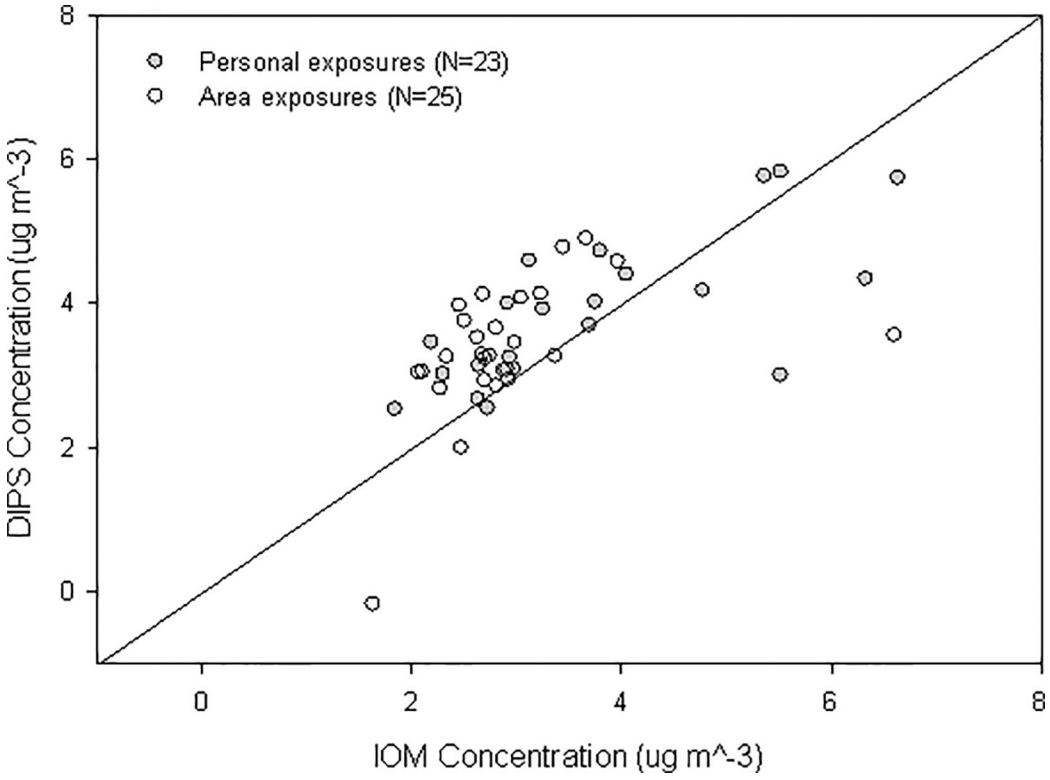

FIGURE 1.

Mass concentrations between the pairs of samples for copper. The diagonal line represents 1:1 relationship.

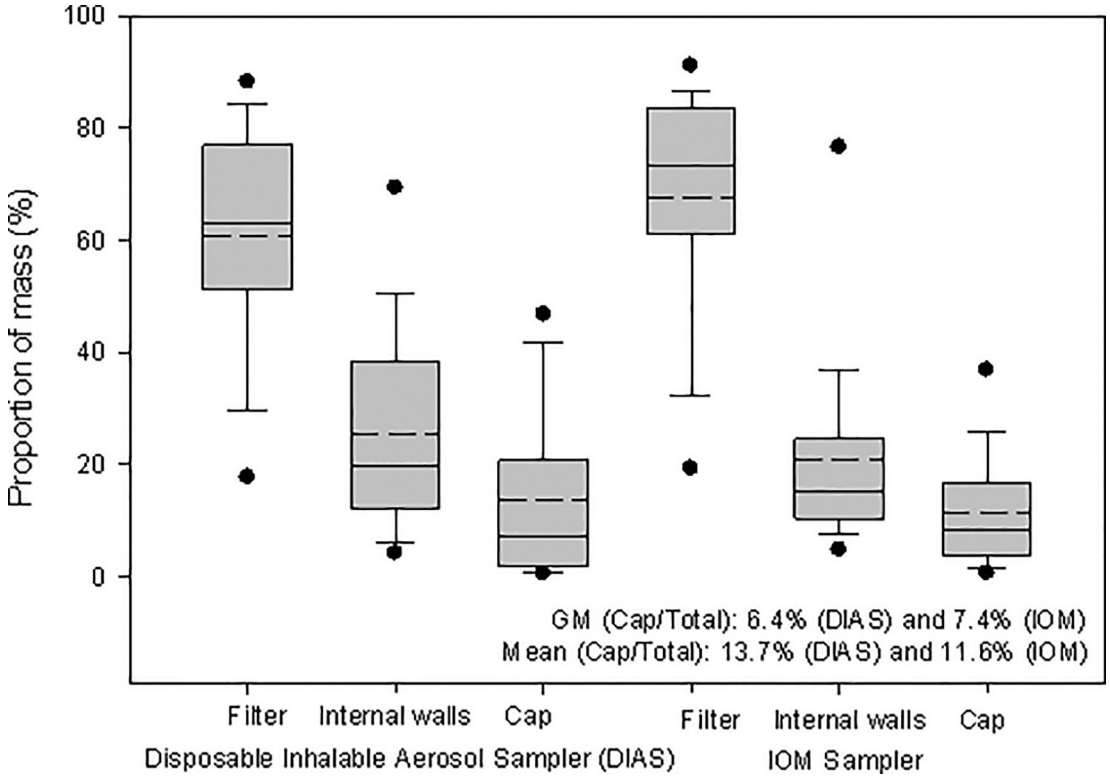

Figure 2.

Percent proportion of mass on the cap over the total mass (i.e., sum of masses from filter, wiped sample from the interior walls, and wiped sample inside the cap). Note that each box plot represents 10^{th}, 25^{th}, 50^{th} (median), 75^{th}, and 90^{th} percentiles and the solid circles indicate the 5^{th} (lower) and 95^{th} (upper) percentiles (dashed line: mean).

Table 1.

Summary of exposure data (Copper; Combined results of filter samples and wiped samples)

Sampling method	N[1]	Sampling time (min)	DIAS prototype (μg m^{-3})		IOM sampler (μg m^{-3})		Ratio of exposures (DIAS/IOM)	
			Range	GM[2] (mean)	Range	GM[2] (mean)	Range	GM[2] (CV[3])
Personal	23	259–516	12.5 – 337.7	45.5 (79.3)	6.3 – 760.8	39.7 (110.5)	0.1 – 4.4	1.1 (0.7)
Area	25	256–529	0.8 – 133.6	29.1 (40.8)	5.1 – 737.4	18.3 (46.8)	0.05 – 4.6	1.6 (0.6)
Combined[4]	48	256–529	0.8 – 337.7	36.1 (59.3)	5.1 – 760.8	26.5 (77.3)	0.05 – 4.6	1.4 (0.6)

(1)N= Number of sample pairs;

(2)GM = Geometric mean;

(3)CV = Coefficient of variation;

(4)Combined = Combined data of personal and area exposure measurements

Table 2.

Summary of statistical analyses between the pair of the samples

Sampling method	N[1]	Concordance correlation coefficient (CCC)			Linear regression			p-value of Proc Mixed (Concentration Estimate, μg m^{-3})[3]
		CCC-Total[2]	CCC-Precision	CCC-Bias	Slope (β)	p-value for β=1	Adjusted R^2	
Personal	23	0.692	0.728	0.951	0.541	0.0005	0.5072	0.487 (D:3.82 ≅ I:3.68)
Area	25	0.395	0.444	0.889	0.476	0.016	0.1620	0.031 (D:3.37 > I:2.91)
Combined[4]	48	0.592	0.624	0.949	0.525	< .0001	0.3756	0.035 (D:3.59 > I:3.27)

(1)N= Number of sample pairs;

(2)CCC-Total = CCC-Precision multiplied by CCC-Bias;

(3)Concentration estimates of DIASs (D) and IOM samplers (I);

(4)Combined = Combined data of personal and area exposure measurements