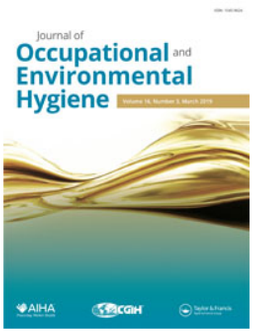

Journal of Occupational and Environmental Hygiene

ISSN: 1545-9624 (Print) 1545-9632 (Online) Journal homepage: https://www.tandfonline.com/loi/uoeh20

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To cite this article: Eun Gyung Lee, Peter J. Grimson, William P. Chisholm, Michael L. Kashon, Xinjian He, Christian L'Orange & John Volckens (2019) Performance evaluation of disposable inhalable aerosol sampler at a copper electrorefinery, Journal of Occupational and Environmental Hygiene, 16:3, 250-257, DOI: 10.1080/15459624.2019.1568444

To link to this article: https://doi.org/10.1080/15459624.2019.1568444

Accepted author version posted online: 14 Jan 2019.
Published online: 22 Feb 2019.

JOURNAL OF OCCUPATIONAL AND ENVIRONMENTAL HYGIENE
2019, VOL. 16, NO. 3, 250–257
https://doi.org/10.1080/15459624.2019.1568444

Performance evaluation of disposable inhalable aerosol sampler at a copper electrorefinery

Eun Gyung Lee[a], Peter J. Grimson[b], William P. Chisholm[a], Michael L. Kashon[c], Xinjian He[b], Christian L'Orange[d], and John Volckens[d]

[a]National Institute for Occupational Safety and Health (NIOSH), Health Effects Laboratory Division, Exposure Assessment Branch, Morgantown, West Virginia; [b]Department of Industrial and Management Systems Engineering, West Virginia University, Morgantown, West Virginia; [c]National Institute for Occupational Safety and Health (NIOSH), Health Effects Laboratory Division, Biostatistics and Epidemiology Branch, Morgantown, West Virginia; [d]Department of Mechanical Engineering, Colorado State University, Fort Collins, Colorado

ABSTRACT

This study evaluates the performance of the disposable inhalable aerosol sampler (DIAS), a new sampler developed to be more cost-effective than the traditional inhalable particle samplers and comparable to the inhalable particle sampling convention. Forty-eight pairs of the DIAS prototype and the IOM sampler were utilized to collect copper exposure measurements (23 personal and 25 area) at an electrorefinery facility. The geometric mean (GM) value of ratios of exposure data (DIAS/IOM) was 1.1, while the GM of ratios (DIAS/IOM) was 1.6 for the area exposure data, revealing 84% of the ratios were greater than one. For both personal and area exposure data, the concordance correlation coefficient tests revealed significant disagreements between the two types of samplers and suggested precision as the source of the disagreement. The estimated mean concentration was higher for the DIAS compared that for the IOM for the area exposure data ($p < 0.05$), while the results were comparable for the personal exposure data ($p = 0.49$). Overall, the DIAS generated higher exposure results compared to the IOM sampler for the area exposures. For the personal exposures, the findings were inconclusive due to inconsistent results of factors aforementioned. This study is limited to one metal component (copper) of the dust at a worksite. To date, this is the first field evaluation using personal exposure data to test the performance of the DIAS and the second evaluation using area exposure data. Thus, it will be necessary to conduct additional field evaluations with various elements to further evaluate the performance of the DIAS. In addition, particle migration to the internal walls of the cap was observed during the transportation of collected samples to a laboratory for both sampler types (6.4% for the DIAS and 7.4% for the IOM). Occupational health and safety 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

Copper electrorefinery; disposable inhalable aerosol sampler; exposure assessment; IOM sampler; sampling pairs; transportation loss

Introduction

In the United States, three types of samplers, the IOM, the Button, and the 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. Both IOM and Button samplers have shown sampling efficiencies comparable to the inhalable particle size-selective sampling convention adopted by the International Standards Organization,[1] the European Standardization Organization,[2] and the American Conference of Governmental Industrial Hygienists (ACGIH®).[3] However, one major barrier to the use of these two samplers is high cost. Therefore, occupational professionals might collect minimal numbers of samples from workplaces that have 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.[4,5]

In addition, problems originating from particle deposition on the interior walls of sampler have been described by various researchers.[6–10] For example, if samples collected with IOM samplers are analyzed by a gravimetric method, no particle losses would be

CONTACT Eun Gyung Lee dtq5@cdc.gov National Institute for Occupational Safety and Health (NIOSH), Health Effects Laboratory Division, Exposure Assessment Branch, 1095 Willowdale Road, Morgantown, WV 26505.

expected because all components constituting the IOM sampler (particles in the filter, particles deposited on the internal wall of cassette and particles migrated to the internal surface of lid during the transport) are measured. However, sample analysis depending solely on the filter (e.g., cyclone samplers for the collection of inhalable fraction) might lead to 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 a similar concept, CFC samplers holding internal capsules, such as the Accu-Cap internal capsule (SKC Inc., Eighty Four, PA) and the Solu-Sert filter capsule (Zefon International Inc., Ocala, FL), are commercially available, but again this sampler is designed to collect dust (i.e., aspirated particles into its inlet) rather than the inhalable fraction.

To address the limitations of the aforementioned issues, L'Orange et al.[11] 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. Like 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 the DIAS matched with that of the IOM sampler. Stewart et al.[12] 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 also be used for a task-based sample collection with detectible amount of mass (i.e., increasing sample mass by increasing the sampled volume). 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.[13] 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,[11,12] 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 has been performed.[13] 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 30–47 °C during the day in the tankhouse, with relative humidity ranging from 30–70%. 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. Typically, as with most copper refineries, workers in this facility were exposed to airborne solid metals including copper, arsenic, silver, lead, and selenium.

We collected 48 paired exposure measurements (23 personal and 25 area) using the IOM made of plastic (SKC Inc., Eighty Four, PA) and the DIAS manufactured by the developer. Personal exposure measurements were collected from the following job descriptions: stripper crane man, cleaning stripper, cellar man, scrap operator, scrap washer, crane operator, and supervisor. Area sampling was conducted 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 DIAS 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 testing this prototype[11–13] 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 256–529 min. The sampling pumps (model AirChek XR5000, SKC Inc.) were calibrated prior to the sampling and checked after the sampling with a DryCal DC-Lite device (BIOS International Corporation, Butler, NJ) to ensure the nominal flow rate of 2 L min^{-1} ± 5%. Prior to sampling, a leak test was performed for both samplers to ensure no leakage. For the personal sampling, we placed the two types of samplers randomly on the opposite sides of the worker's torso (i.e., one on the left lapel and one on the right lapel). For the area sampling, the paired samplers were hanging freely and adjacent each other (distance between two samplers = about 5–10 cm) by ensuring that the inlets of the samplers were facing the same direction. Nine field blank samples for each sampler type were collected.

Sample analysis

For each IOM sampler, the exposed 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 quartered 25 mm clean MCE filter and placed in another tube; prior to wiping, the quartered 25 mm MCE filter was soaked in isopropyl alcohol and the same one was used to wipe twice. Then, the inside of the cap was wiped twice with another quarter-sized 25 mm 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,[14] 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). Yttrium was used as an internal standard. 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 1–100 ppb. All solutions were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS; Perkin Elmer 300D, Waltham, MA). 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 amounts 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 LOQ was 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 three samples (one filter and two 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 measurement results 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[15] $LOQ/\sqrt{2}$ 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 and internal walls of each sampler (determined from ICP-MS).

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.[16–19] Unlike the Bland-Altman test, the results of CCC tests can

Table 1. Summary of exposure data (copper; combined results of filter samples and wiped samples).

Sampling method	N[A]	Sampling time (min)	DIAS prototype (μg m^{-3})		IOM sampler (μg m^{-3})		Ratio of exposures (DIAS/IOM)	
			Range	GM[B] (AM[C])	Range	GM[B] (AM[C])	Range	GM[B] (GSD[D])
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 (2.5)
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 (2.8)

[A]N = Number of sample pairs;
[B]GM = Geometric mean;
[C]AM = Arithmetic mean;
[D]GSD = Geometric standard deviation

determine a source of disagreement whether it is from precision or bias. 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.[20,21] An additional statistical test was conducted to test if the mean concentration of the DIAS is the same as that of the IOM; a p-value of 0.05 was used for testing the hypothesis and estimated mean concentrations were compared using Proc Mixed procedure. All statistical analyses were performed with SAS/STAT software (version 9.3, SAS Institute, Cary, NC). For the CCC test, we used a validated SAS macro provided by Lin et al.[17]

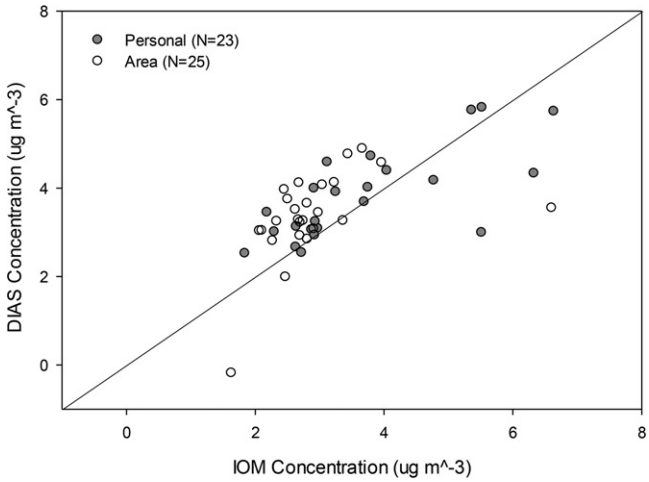

Figure 1. Mass concentrations between the pairs of samples for copper. The diagonal line represents 1:1 relationship.

Results

Comparison of exposure data between the DIAS and the IOM

Table 1 shows a summary of exposure measurement data, presenting the combined results of filter samples and wiped samples. For both personal and area exposure data, the geometric mean (GM) concentrations of the DIAS were higher than those of the IOM sampler. For both sampler types, the arithmetic mean concentrations were considerably higher than the corresponding GM concentrations revealing that the measurement data were positively skewed. The range of exposure data was wider for the IOM sampler compared to that for the DIAS.

The GM value of the ratios of exposure data (DIAS/IOM) was 1.1 for the personal exposures and 1.6 for the area exposures. Overall, the variation of the ratios (DIAS/IOM) for the area exposure data was slightly greater than that for the personal exposure data (geometric standard deviation [GSD] = 2.5 for the personal and 2.8 for the area exposure data) (Table 1). 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 78% of the personal exposure measurements and 84% of the area exposure measurements showed the concentration ratios (DIAS/IOM) greater than one.

As shown in Table 2, the CCC-total results for the personal and area exposure measurement results were lower than 0.878 (± 35% acceptance criterion), 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 area samples showed even lower value of CCC-Precision compared to the personal samples indicating that the variation is even greater than for the area samples. The CCC-Bias $\geq$ 0.889 suggests little deviation from the unity line. The results using Proc Mixed procedure to compare the mean concentration between the DIAS and the IOM showed no overall statistical difference for the personal exposure data (p = 0.487). On the other hand, statistically significant differences between the DIAS and the IOM were observed for the area exposure data (p = 0.031); the estimated mean concentrations were always higher for the DIAS prototype compared to the IOM (Table 2).

Migration of particles during the sample transport

We obtained the amount of copper mass collected on the cap by analyzing the samples separately to

Table 2. Summary of statistical analyses between the pair of the samples.

		Concordance correlation coefficient (CCC)			Proc Mixed	
Sampling method	N[A]	CCC-Total[B]	CCC-Precision	CCC-Bias	p-value	Mean concentration estimate ($\mu g\ m^{-3}$)
Personal	23	0.692	0.728	0.951	0.487	DIAS:3.82 ≅ IOM:3.68
Area	25	0.395	0.444	0.889	0.031	DIAS:3.37 > IOM:2.91

[A]N = Number of sample pairs;
[B]CCC-Total = CCC-Precision multiplied by CCC-Bias

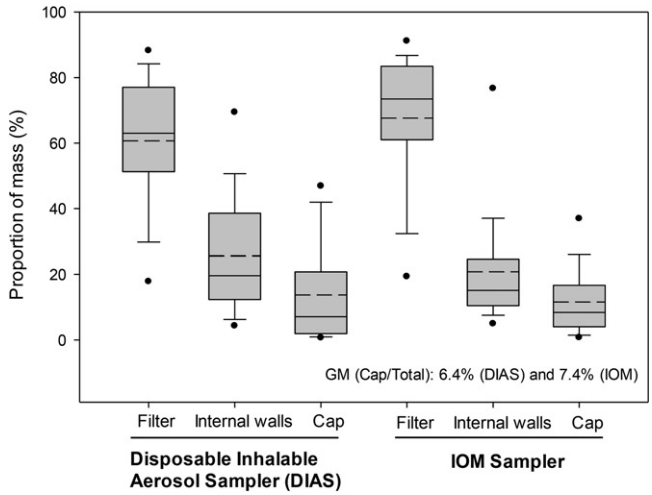

Figure 2. Percent proportion of mass on the filter, internal walls and cap over the total mass (i.e., sum of masses from filter, wiped sample from the interior walls, and wiped sample inside the cap). Note that each box plot represents 10th, 25th, 50th (median), 75th, and 90th percentiles and the solid circles indicate the 5th (lower) and 95th (upper) percentiles (dashed line: mean).

determine the proportion of particles migrated due to the transportation of samples to a laboratory by commercial airfreight (Figure 2). As described in Methods, although we are interested in the proportion of particle mass deposited on the inside of the cap, the proportions of particles collected on filter and internal walls were also presented to provide additional information. 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).

Discussion

Comparison of the exposure data between the DIAS and the IOM

For the personal exposures, copper concentrations ranged from 12.5–337.7 $\mu g\ m^{-3}$ (GM = 45.5 $\mu g\ m^{-3}$) for the DIAS and 6.3–760.8 $\mu g\ m^{-3}$ (GM = 39.7 $\mu g\ m^{-3}$) for the IOM. For the area exposures, the concentrations ranged from 0.8–133.6 $\mu g\ m^{-3}$ (GM = 29.1 $\mu g\ m^{-3}$) for the DIAS and 5.1–737.4 $\mu g\ m^{-3}$ (GM = 18.3 $\mu g\ m^{-3}$) for the IOM (Table 1). Regardless of the sampling time ranging from 256–529 min, none of the individual exposure measurements exceeded the occupational exposure limit of 1000 $\mu g\ m^{-3}$ by the Occupational Safety and Health Administration (OSHA) - Permissible Exposure Limit (PEL),[22] NIOSH-Recommended Exposure Level (REL),[23] and ACGIH-Threshold Limit Value (TLV®).[3] For both DIAS and IOM samplers, the GM exposure concentrations were higher for the personal sampling method compared to the area sampling method, with the difference likely due to the proximity of samplers' location from the emission source.

Overall, the DIAS generated higher exposure data than the IOM sampler. About 78% of sample pairs (18 out of 23 sample pairs) for the personal exposures and 84% (21 out of 25 sample pairs) for the area exposures 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. Statistical test results also showed disagreement of concentrations between the DIAS and the IOM (CCC-Total = 0.395 < acceptance criterion of 0.878) and the comparison of overall mean concentrations between the two sampler types (p-values < 0.05) (Table 2). Anthony et al.[13] 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 endotoxins were collected. They reported no significant difference of the DIAS compared to the IOM for the comparison of inhalable dust analyzed using a gravimetric analysis, whereas the DIAS produced higher inhalable endotoxin concentrations compared the IOM. Anthony et al. reported that one explanation causing a difference for the comparison of endotoxin concentrations might be from the rinsing procedure of the IOM sampler. For the area samples, 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.

Probably, different analytical methods such as weighing method for the dust (Anthony et al.) and ICP-MS method in the present study might be one of reasons causing such a difference. 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.[12] 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 measurement results, although the comparison of overall mean concentrations between the DIAS and IOM revealed no statistically significant difference (p-value = $0.487 > 0.05$), the disagreement (CCC-Total = $0.692 < 0.878$) indicates that that the performance of the DIAS is not comparable to that of IOM. The range of ratios of exposure data (DIAS/IOM) between the personal samples and area samples 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). The paired samplers located for the area sampling were stationary and the distance between one sampler and the other sampler for each pair was closer for the area samples (i.e., adjacent each other) than for the personal samples (i.e., one on the left lapel and the other one on the right lapel). In addition, the inlets of the samplers were facing the same direction during the area sample collection. Thus, it is expected to have a lower GM value of ratios for the area samples compared to the personal samples because there would be no influence from a worker's behavior on exposure concentrations. The root cause of the discrepancies of ratios between the area samples and the personal samples is unclear. Additional co-location study (such as using pairs of the same type of sampler) would be helpful to characterize under what conditions the two types of samplers might agree or differ.

To date, this study is the first field evaluation using personal exposure measurement results to test the performance of the DIAS. In addition, it is the second field evaluation using area exposure data after Anthony et al.[13] 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. L'Orange et al.[11] reported that the sampling efficiencies of DIAS and IOM samplers, tested in a low-velocity wind tunnel, were comparable for the range of particle size from 9.5–89.5 μm. It would be helpful if we had confirmed that the dominant particle sizes at this workplace were within the range of particle sizes that L'Orange et al. tested. Unfortunately, during the field survey, we did not obtain size-selective particle concentrations and thus could not determine the dominant particle sizes at this workplace. Another limitation is that the variation from co-located samples of the same sampler type such as a pair of two DIAS or two IOM samplers was not determined which could have provided additional explanation for the precision. 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, it will be necessary to characterize the performance of the DIAS in various environments sampling numerous chemical components and/or evaluating with different mass concentrations covering various particle sizes.

Migration of particles during the sample transport

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 proportions = 6.4% for the DIAS prototype and 7.4% for the IOM). Although we presented the proportion of mass on the filter and internal walls over the total mass (Figure 2), only the proportion of mass on the cap was valid to determine the migration of particles truly from the sample transportation. For example, if a weighing method was used, it would be possible to measure the collected samples before- and after-transportation. However, the employed analytical method (i.e., ICP-MS) in this study cannot be done at the workplace. Thus, it should be noted that the proportion of particles' migration reported in this study is not representative because particles' migration 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. Demange et al.[24] investigated metal deposits on sampling cassette walls due to transportation disturbances using weighing method 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.[25] 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 particles' migration due to the transportation were different between the present study and a study by Demange et al., it is obvious that the migration of particles could happen from the transportation of samples. This would be the case when collected samples are transported without careful handling of samples such as by commercial airfreight. If analytical chemists do not include the particles deposited on the internal wall of the cap for the wet chemical analysis, it is very likely that the reported concentrations underestimate the true concentrations. The findings of this study indicate that occupational professionals and/or analytical chemists 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 the internal wall of the cap for wet chemical analysis.

Conclusions

The DIAS, newly developed to overcome the current limitations of inhalable samplers, has been evaluated at a copper electrorefinery facility. All personal and area exposure measurement results were below the occupational exposure limit of 1000 μg m^{-3} by the OSHA-PEL, NIOSH-REL, and ACGIH-TLV. Overall, the DIAS generated higher exposure measurement results compared to the IOM sampler for the area exposures. For the personal exposures, the findings were inconclusive due to inconsistent results of the factors considered to test the DIAS performance (e.g., GM value of ratios [DIAS/IOM], CCC test and comparison of mean concentrations using Proc Mixed). This is the 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 is too early to make a firm conclusion about the DIAS's 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, particles' migration to 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 samples by including the particle mass collected on a cap.

Disclaimer

The findings and conclusions in this report are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention. Mention of any company or product does not constitute endorsement by NIOSH/CDC.

Acknowledgments

The authors are sincerely thankful to Dr. Alan S. Echt (NIOSH/Division of Applied Research & Technology) and Mr. Randy Boylstein (NIOH/Respiratory Health Division) for reviewing this manuscript prior to journal submission. The authors also would like to offer special thanks to the employees who participated in our study.

Funding

This project was funded internally by the National Institute for Occupational Safety and Health (Project CAN number: 939ZXEY).

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