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Comparison of Lead and Tin Concentrations in Air at a Solder Manufacturer from the Closed-Face 37-mm Cassette With and Without a Custom Cellulose-Acetate Cassette Insert

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Abstract

A polyvinyl chloride (PVC) cassette insert with PVC filter (ACCU-CAP) in a 37-mm closed-face cassette (CFC) was designed for gravimetric analysis. A customized version of the ACCU-CAP, also to be used in the CFC, was manufactured from an acid-digestible cellulose-acetate cassette insert joined to a mixed cellulose ester (MCE) filter for wet chemical analysis. The aim of this study was to compare metal particle concentrations as sampled by the customized insert (CI) in a CFC sampler with the traditional sampling method using only a MCE filter in the CFC. Thirty-nine personal and 13 area samples were taken using paired filter-based CFC and the CI in CFC samplers at a solder manufacturing plant. The CI was removed from its CFC, and digested and analyzed as a whole. The MCE filter from the typical CFC was removed for analysis and then the interior of the cassette was wiped with Ghost Wipe for a separate analysis. The MCE filter only, Ghost Wipe, and CI were separately dissolved in heated nitric acid for ICP-MS analysis. Overall, the geometric mean concentration of the filter-only (FO) samples was considerably lower than that of the CI samples, by 53% for lead and 32% for tin. However, if the FO analysis was added to the corresponding Ghost Wipe analysis, i.e., filter+interior wipe (FW), the geometric mean concentrations of the FW results were similar to those of the CI results (by 113% for lead and 98% for tin). For both lead and tin the comparison of (log-transformed) metal concentrations between the FW and CI results showed no statistically significant difference (p-value = 0.3009 for lead and 0.800 for tin), while the comparison between the FO and CI results shows statistically significant differences (all p-values < 0.05). In conclusion, incorporating the sampler internal non-filter deposits by wiping or use of an internal filter capsule gave higher results than analyzing only the filter. Close agreement between the two methods of including non-filter deposits is an indication of general equivalency.

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Keywords

aerosol sampling; CFC sampler; internal filter capsule; metals; Solu-CAP; Solu-Sert

INTRODUCTION

Occupational exposure limits of prevailing regulatory and advisory standards are most commonly expressed as mass concentration, that is, the quantity of contaminant collected on a sampling medium in a unit volume of air drawn by a battery-powered personal sampling pump. For aerosol sampling methods the closed-face filter cassette (CFC) equipped with a filter has been widely used in the United States for gravimetric analysis to determine total mass and subsequently for wet chemical analysis to determine the mass of element content. While specific National Institute for Occupational Safety and Health (NIOSH) methods have to date only considered the analysis of the filter of the CFC sampler for both gravimetric and wet chemical analyses, it has long been known that particles entering the cassette can deposit on the internal surfaces of the CFC.

NIOSH noted in Chapter O⁽¹⁾ of the *Manual of Analytical Methods* that these deposits should be assessed as part of the sample, and with the advent of more recent data (summarized in Harper and Demange⁽²⁾) documenting the extent of internal surface deposits NIOSH has published stronger policy guidelines on their inclusion.⁽³⁾ Table 1 of this reference⁽³⁾ provides a summary of the proportion of wall deposits ranging from 5 to 53% for the median and from 12 to 91% for the maximum wall deposits when samples were collected at various workplaces. The Occupational Safety and Health Administration (OSHA) also has noted this issue and requires an inclusion of particles collected from the interior surfaces of the CFC sampler when producing personal sample results for comparison to a permissible exposure limit.^(4,5)

Since the proportion of internal non-filter deposits in a sample is unpredictable, using a correction factor to adjust for them is not possible. Thus researchers and regulatory agencies have suggested various methods to account for wall deposits such as rinsing or wiping techniques,⁽⁴⁻⁶⁾ direct sample extraction from the sampler,^(6,7) and internal sampling capsules.^(8,9) However, there is a concern that wiping and/or washing techniques might not guarantee a complete removal of particles, and it is a tedious manual process that adds to the analytical cost. An internal capsule of aluminum foil incorporating a filter has been used for many years by OSHA for gravimetric analysis of respirable dust, but it is unsuitable for acid digestion and elemental analysis. Polyvinyl chloride (PVC) cassette inserts welded to a PVC filter (ACCU-CAP) have been available for gravimetric analysis for several years, but these are again unsuitable for acid digestion.

Recently, Harper and Ashley^(8,10) performed a round-robin study using a customized version of the ACCUCAP made from an acid-digestible cellulose-acetate cassette insert with MCE filter to demonstrate its utility in wet chemical analysis. They generated samples spiked with lead (aqueous salt solutions, dust and paint) or with aerosolized multi-element salt solutions and distributed them to national and international participants for digestion and analysis by inductively coupled plasma (ICP) with mass spectrometry (MS) or atomic

emission spectrometry (AES). The findings of this round-robin study suggested that the customized inserts could provide a useful method to replace wiping and/or washing techniques to account for the CFC internal non-filter deposits. Since the findings by Harper and Ashley^(8,10) were limited to laboratory-generated samples it was considered worthwhile to perform a field survey to confirm that the mass concentrations determined from the customized insert sampler are equivalent to that of the filter plus wall deposits in field samples. Thus the purpose of this study is to compare measures of metal concentrations between the CFC using the customized insert sampler and the CFC sampler with only a filter (and with wiping of the internal cassette surfaces) by collecting field samples in a metals manufacturing plant with co-located pairs of samplers.

METHODS

Field Survey

The field survey was performed at a solder manufacturing plant where there was recycling of metal products from a wide range of industries and fabricating of new products including high-quality alloys, solders, and pure metal products. Job tasks involved during the sampling were smelting, casting, sieving, and packing of powder, and other miscellaneous tasks including wire extraction, maintenance, and cleaning. Lead and tin alloys were the predominant materials encountered for all job tasks, but small amounts of other metals including silver, copper, cadmium, antimony, zinc, aluminum, bismuth, and iron were also present. In the smelting area, any metal-containing materials such as drosses, ingots, and pastes were recycled to salvage the metals.

The smelter operators were exposed to airborne metals as a result of handling the materials and tapping the furnaces. In the casting area, the operators produced various sizes of alloy bars containing various percentages of lead and tin. The casting operators were exposed to metal fumes during hand ladling and pouring the liquid alloy into molds. After checking the quality of product, the alloy bars were delivered to the powder department. Workers in the powder production area fed the cast bars (both lead-containing and lead-free) into the primary and secondary pots. The molten metals were automatically transferred to a huge spinning chamber to generate size-selected powders. Then, the size-selected powders were collected into a bag in a canister placed under a feeder connected to a spinning chamber. In this area, workers were exposed to metals by handling the casts, tapping feeders, tightening bags, and occasionally changing the spinning disk in the inside of the spinning chambers. Those powder bags in canisters were then delivered to an enclosed area for powder packing. Workers in this room were exposed to metals by pouring powders into a hopper, scooping powders to small size bags, and sealing the bags with nitrogen gas.

Thirty-nine personal and 13 area sample pairs were collected using the typical CFC with filter-only and the CFC with customized inserts. For the personal exposures, the number of samples was 8, 9, 16, and 6 for the smelting operators, casting operators, powder handlers, and others, respectively. For the area exposures, the number of samples was 8 at the smelting area and 5 in the powder department. The customized insert is made up of a 37-mm mixed-cellulose ester (MCE) filter (0.8 μm pore size) on the bottom capped with an acid-digestible cellulose-acetate cassette capsule. The customized inserts were from the same

batch (SKC Inc., Eighty Four, Pa.) as in the previous laboratory studies by Harper and Ashley.^(7,9) This product, incorporating improvements suggested through the cited studies, is now commercially available from SKC, Inc. (Solu-CAP), and a similar product (Solu-Sert) is available from Zefon International (Ocala, Fla.). Airborne metal concentrations were collected at 2 liters per min (LPM) for both sampler types. The sampling times ranged from 3 to 7 hr, but both samples in a pair were collected for the same period. The personal sampling pumps were pre- and post-calibrated with a DryCal DC-Lite device (BIOS International Corporation, Butler, N.J.) to ensure the difference of sampling flow rates within $\pm 5\%$. Five field blank CFC cassettes and customized inserts were collected. Only lead and tin concentrations were determined since the proportions of other metals were minimal.

Sample Analysis

Prior to opening a CFC for analysis, its external surfaces were manually cleaned with Ghost Wipes (Environmental Express, Charleston, S.C.) to minimize the possibility of contamination when it was opened and the filter or CI removed. Each CFC cassette with filter-only was pried open and the filter was removed with tweezers and placed in a 35-mL glass beaker of a diameter slightly larger than 37 mm. After removing the filter, the interior surface of the CFC including the inlet nozzle was wiped twice with a piece of Ghost Wipe, cut to 2.5 cm by 2.5cm size. The two sections of Ghost Wipe were placed in a beaker for a separate analysis from the CFC filter. Each acid-digestible CI was carefully removed and placed in a glass beaker.

Digestion was carried out with 20% nitric acid (volume/volume, aqueous) as follows. Once 10 mL of 20% nitric acid (v/v, Thermo Fisher Scientific, Pittsburgh, Pa.) was added to the beakers, the beakers were placed on a hot plate at room temperature. The hot plate was activated and as the solutions heated (temperature range between 90°C and 95°C) there was some bubbling as the acid reacted with the filters, Ghost Wipes, and inserts, but the solutions were not allowed to reach a full boil. When the solid material in the beaker had completely dissolved (typically, after ~ 45 min), the solution was allowed to remain at elevated temperature until near dryness (i.e., when the fluid in the bottom of the beaker began to retreat to the edges).

Then, after cooling the beakers for ~60 sec, 10 mL of 2% (v/v) nitric acid was added to each. The beakers were further allowed to cool to near room temperature and the contents were then quantitatively transferred with 2% nitric acid to 25 mL glass volumetric flasks. From each volumetric flask a 0.20 mL aliquot was diluted (50:1) with 2% nitric acid in a 15 mL plastic centrifuge tube (SCP Science Catalog #130-010-053, Baie D'Urfé, Quebec, Canada) which had been rinsed with 20% nitric acid prior to use. Yttrium solution (Claritas PPT Grade Standards; Spex CertiPrep, Metuchen, N.J.) was added to bring each solution to 20 ppb yttrium as an internal standard. Lead and tin contents were determined for each sample by ICP-MS. Calibration solutions were made from standard solutions of lead (Claritas PPT Grade Standards; Spex CertiPrep) and tin (Spex CertiPrep) to span the range from 1 to 400 ppb for lead and tin for most runs, though in some cases a narrower range was

covered. Yttrium solution was also added to the calibration solutions as an internal standard at 20 ppb.

The solutions were analyzed by ICP-MS (Perkin Elmer 300D, Waltham, Mass. [running under the NEXion version 1.3 software]) in standard mode using the Pb-206 and Sn-112 isotopes. Calibrations were linear under these conditions and lead and tin concentrations were calculated from a linear fit to the measurements of the calibration solutions. For samples where the measured concentration exceeded the upper limit of the calibration curve, a new sample was prepared at higher dilution with yttrium added as an internal standard at 20 ppb and the sample re-analyzed under otherwise identical conditions. Media blank samples including five customized inserts, six MCE filters, and six Ghost wipes were prepared in the same manner to estimate the limit of detection (LOD) for the ICP-MS measurements. For the purposes of quality control some measurements were repeated to ensure no instrumental drift. During the study, we also analyzed a blind set of spiked Solu-Serts prepared by High Purity Standards (Charleston, S.C.) which included lead and tin. Solu-Serts are similar to the modified ACCU-CAPS used in this study and analysis of these samples supports the quality of our analyses of the modified ACCU-CAPS.

Data analysis

Mass concentrations were calculated from the mass contents determined from the ICP-MS and sampled air volume. Statistical analysis to compare mass concentrations between sampling filters was conducted using SAS/STAT software version 9.2 for Windows (SAS Institute, Cary, N.C.) utilizing the Proc Mixed procedure. All data were log transformed to meet the assumptions of the analysis. Two pairwise comparisons, 1) CFC Filter only (FO) vs. Customized Insert (CI), and 2) CFC Filter plus interior wipe (FW) vs. CI, were made and Cook's Distance was calculated and used in the establishment of outliers. Data points were considered outliers and excluded if they met at least one of three criteria: 1) the collected mass is below the analytical detection limit; 2) the discrepancy of sampling time of each pair is more than 10%; and 3) the sample pair had a Cook's distance score of greater than $4/n$ where n is the number of sample pairs in the analysis, and simultaneously the ratio between the samplers was outside the boundary between $2/3$ and 7 .⁽¹¹⁾ The LODs for the MCE filter-only, Ghost wipe, and customized insert were 0.081 μg , 0.072 μg , and 1.8 μg , respectively, for lead and 0.1 μg , 0.19 μg , and 0.14 μg , respectively, for tin. The High Purity Standards samples were spiked at 10 μg , 25 μg , and 100 μg of lead per sample and 2 μg , 7 μg , and 15 μg of tin per sample and three at each level were analyzed with results (± 2 standard deviation) of 10 $\mu\text{g} \pm 0.52$, 26 $\mu\text{g} \pm 1.5$, 96 $\mu\text{g} \pm 11$ for lead and 2.1 $\mu\text{g} \pm 0.36$, 7.4 $\mu\text{g} \pm 1.1$, 15 $\mu\text{g} \pm 2.8$ for tin.

After removing outliers, pairwise comparisons were reassessed using Proc Mixed and Pearson's correlation coefficients and their corresponding p-values were calculated to determine how well paired samples associated with each other. The mass content of filters, Ghost wipes, and CIs from the field blanks was negligible for lead (median mass $< 2 \mu\text{g}$ for each sample type), with a single value of 3.4 μg /Ghost wipe. The median mass content for tin was 5.4 μg (with a single high value of 8.7 μg /filter), 3.5 μg , and 2.1 μg for field blank filters, Ghost wipes, and CIs, respectively. Since the mass content of field blank samples

was negligible compared to the mass content of samples, it was not subtracted from the mass content of field samples. Data analyses based on job tasks were not performed because the number of sample pairs per job task was not sufficient to represent the specific task.

RESULTS

The number of outliers for the comparison of concentrations between the FO and CI and between the FW and CI was 1 (from personal samples) and 2 (each from personal and area samples) for lead and 3 (2 from personal samples and 1 from area samples) and 5 (4 from personal samples and 1 from area samples) for tin. In this study, only results after removing outliers were reported because the conclusions based on the statistical differences were the same regardless of whether the outliers were included or not. None of the samples showed values less than the LODs.

Table I is a summary of exposure measurements (without outliers) for lead and tin. The exposure ranges were particularly wide due to a few high exposure measurements. Overall, the geometric mean (GM) concentrations were in the order of FW (highest) > CI > FO (lowest) for the lead and CI (highest) > FW > FO (lowest) for tin. Note that the reported values for the CI from FO/CI and FW/CI comparisons were slightly different because outlier tests were separately conducted for each pair comparison. Figure 1 shows box plots of mass concentration ratios between the pairs of samples. For both lead and tin, the average mass concentration ratios for the FO/CI and the FW/CI comparisons were noticeably higher than the median (50th percentile) ratios, indicating presence of a few extreme concentration ratios. As shown in Table I and Figure 1, a comparison of the GM concentrations between the FW and CI indicates they were almost the same (ratio of FW/CI = 1.1 for lead and 1.0 for tin) for both lead and tin, while the GM of the FO data was less than 55% (53% for lead and 32% for tin) of the GM of the CI data. When the measurements were divided into personal and area samples, the ratios of FO/CI and FW/CI were similar to those of the combined data except for the lead area exposures (ratio of lead area exposures: 0.84 (FO/CI) and 1.46 (FW/CI)) (results not shown).

It was observed that the mass deposited on the non-filter surface relative to the total mass (filter plus interior wipe) ranged 4%–77% for lead and 14%–93% for tin. The GM concentration ratios for FO/FW data were not different compared to those for FO/CI, 0.5 for lead and 0.4 for tin (results not shown). As shown in Figure 2, among the CFC samples, 39% of lead measurements and 60% of tin measurements showed more mass on the interior surface of the CFC than on the filter (i.e., the ratio of masses between the filter-only and the interior wipe-only <1).

Figures 3 and 4 show comparisons of log-transformed mass concentrations between the pairs of samples for lead and tin. Note that the log-transformed mass concentrations per job task were shown only for illustration purposes and no conclusions should be drawn from them due to insufficient sample sizes per job task. For the personal exposures the highest GM concentration was observed from the powder handlers in the powder department for both lead and tin. The casting operators' data showed the lowest GM concentration for lead while the data from employees performing other tasks showed the lowest value for tin. The

lead area exposures resulted in higher concentrations than some of the personal exposures (spreading into the whole range of concentrations) while the tin area exposures were shown to be clearly lower than other personal exposures. For the comparison of FO and CI results, the slope was 0.804 (adjusted $R^2 = 0.492$) for lead and 0.766 (adjusted $R^2 = 0.592$) for tin. However, when the masses of internal wipes were included with the filter-only masses, the slope for the mass concentrations between the FW and CI samples was increased to 0.857 (adjusted $R^2 = 0.531$) for lead and 0.921 (adjusted $R^2 = 0.745$) for tin.

As shown in Table II and Figures 3 and 4, all the pairs of samples showed a strong correlation (all correlation coefficients > 0.7) and each pair comparison showed statistically significant correlation (all p-values < 0.0001). For both lead and tin, overall, no statistical difference was observed from the comparison of mass concentrations (log-transformed) between the FW and CI (p-values: 0.3009 for lead and 0.8000 for tin), while the comparison between the FO and CI resulted in statistically significant differences (all p-values < 0.05). For the results showing significant differences, the CI estimates were always higher than the FO estimates (Table II). Also, for each pair comparison, the separation of personal and area exposures did not yield different statistical results (all p-values < 0.05) except for the area exposure of FO/CI (p-value=0.6184) for lead (results not shown).

DISCUSSION

The CFC sampler has been the most widely used device in the United States for aerosol sampling for gravimetric and wet chemical analyses. In the present study, 39% of lead samples and 60% of tin samples showed more masses on the interior walls than on the filter of the CFC sampler, clearly underscoring the importance of internal non-filter deposits to the overall sample. The median mass deposited on the non-filter surface relative to the total mass (i.e., filter plus interior wipe) was 45% for lead and 56% for tin, again confirming that metal concentrations are often severely underestimated if only the filter is analyzed (Figure 1). Overall, the FO concentrations were considerably lower than the CI concentrations (ratio of FO/CI = 0.53 for lead and 0.32 for tin), whereas the results observed from the comparison of the FW and CI (ratio of FW/CI = 1.1 for lead and 1.0 for tin) indicated that FW and CI results were similar.

The statistical analyses revealed significant differences for the comparison of concentrations between the FO and CI for lead and tin (p-values < 0.05) and non-significant differences for the comparison of concentrations between the FW and CI (p-value 0.3009 for lead and 0.8000 for tin). Similar results were observed when the statistical analyses were performed separately for the personal and area exposures (except for the area samples of FO/CI). Harper and Ashley⁽¹⁰⁾ have already shown sufficiently low background levels of other metal elements besides lead and tin from the analysis of customized insert material using ICP-MS and ICP-AES. Thus, although this study was limited to lead and tin exposures, it is expected that the CI sampler can be used for the analysis of other metal elements as well.

The number of samples where the ratio of FW/CI was greater than 7 was 5 for lead and 2 for tin and these were all from personal exposures. The position of the CFC and CI sampler was not fixed (i.e., not always on the left or right side of worker's collar for a sampler type) to

minimize errors due to the position of sampler, so that worker's movements or handedness might be the cause of these differences. Using a multiport sampler might reduce the wide spread of ratios of FW/CI. However, this sampling methodology is limited to only area samples, not personal and particle size-distribution drawn into the multiport sampler which might be different compared to that drawn into the CFC.

CONCLUSION

We demonstrated that the mass concentrations collected with a CFC containing the customized cellulose-acetate insert with MCE filter followed by complete dissolution of the insert with acid did not differ statistically from the combined (filter plus interior wipe) mass concentrations of the CFC sampler with only a filter. Overall, incorporating the sampler internal non-filter deposits by wiping or use of an internal filter capsule gave higher results than when analyzing only the filter. Close agreement of mass concentrations between the customized insert and the filter plus wall deposits is an indication of general equivalency. Another practical benefit of using the customized insert is that analysts can save time and cost by eliminating labor to wipe the internal surface of the CFC samplers. One limitation of using the customized insert sampler is that this type of cellulosic sampler would not be appropriate for gravimetric analysis due to high variability of mass with humidity changes.

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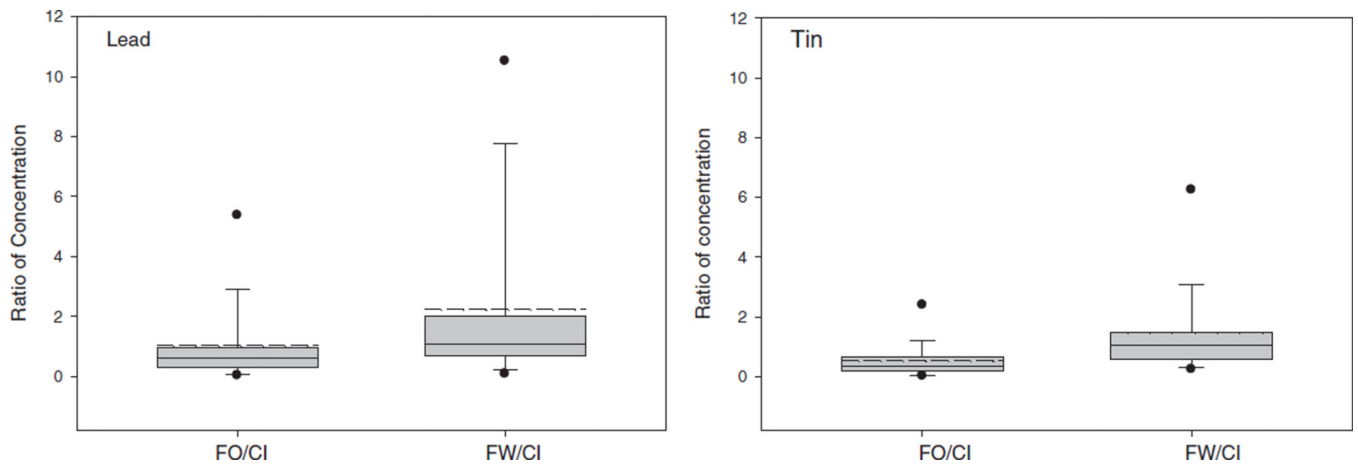
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**FIGURE 1.**

Box plots of mass concentration ratios between the pairs of samples without outliers. 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).

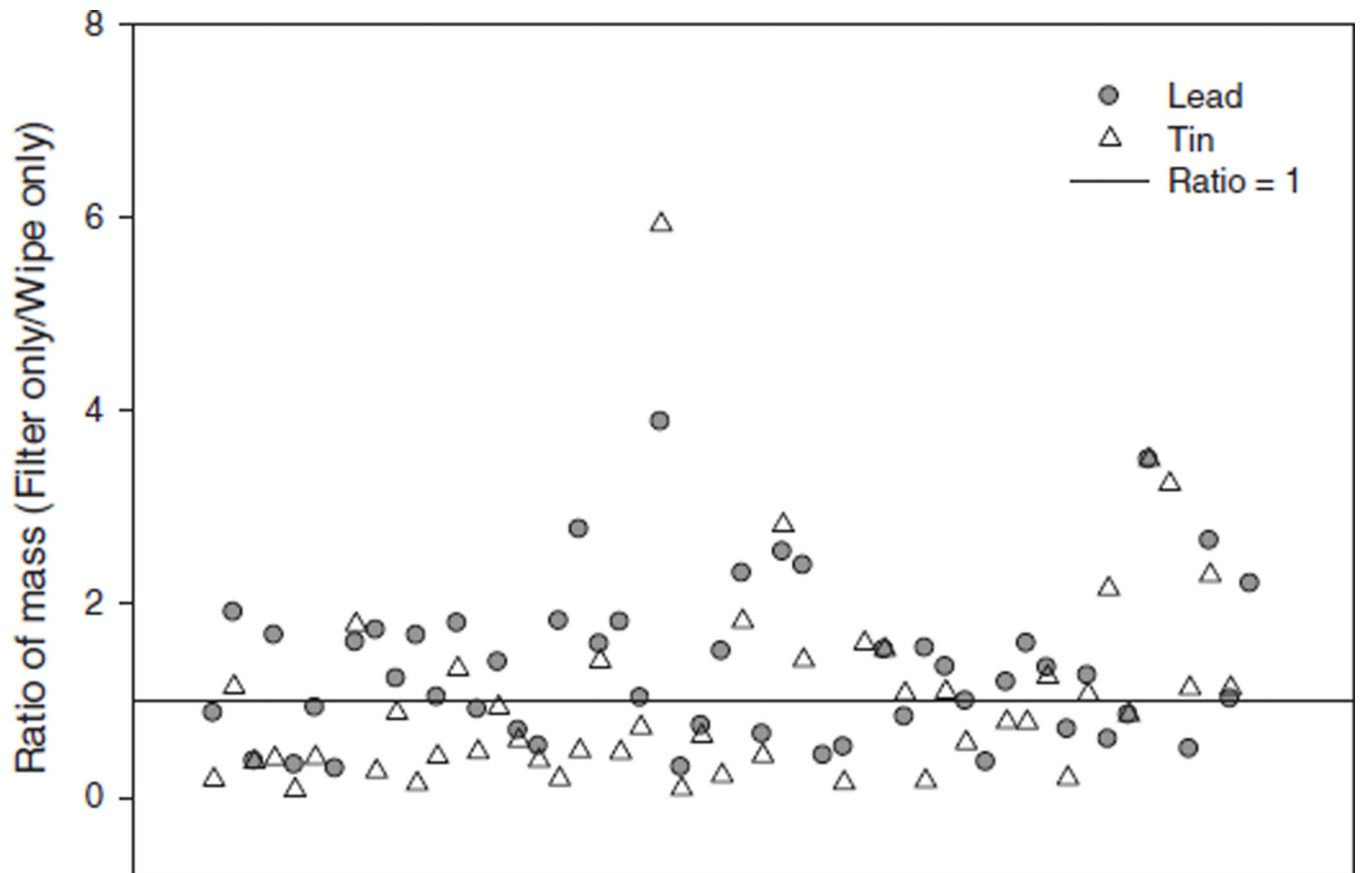


FIGURE 2.

Ratio of masses between the FO and the interior wipe only without outliers (FO/Ghost Wipe). Note that one data point for lead is not shown due to a high ratio (FO/Ghost Wipe = 23.5) but is included in the data analysis.

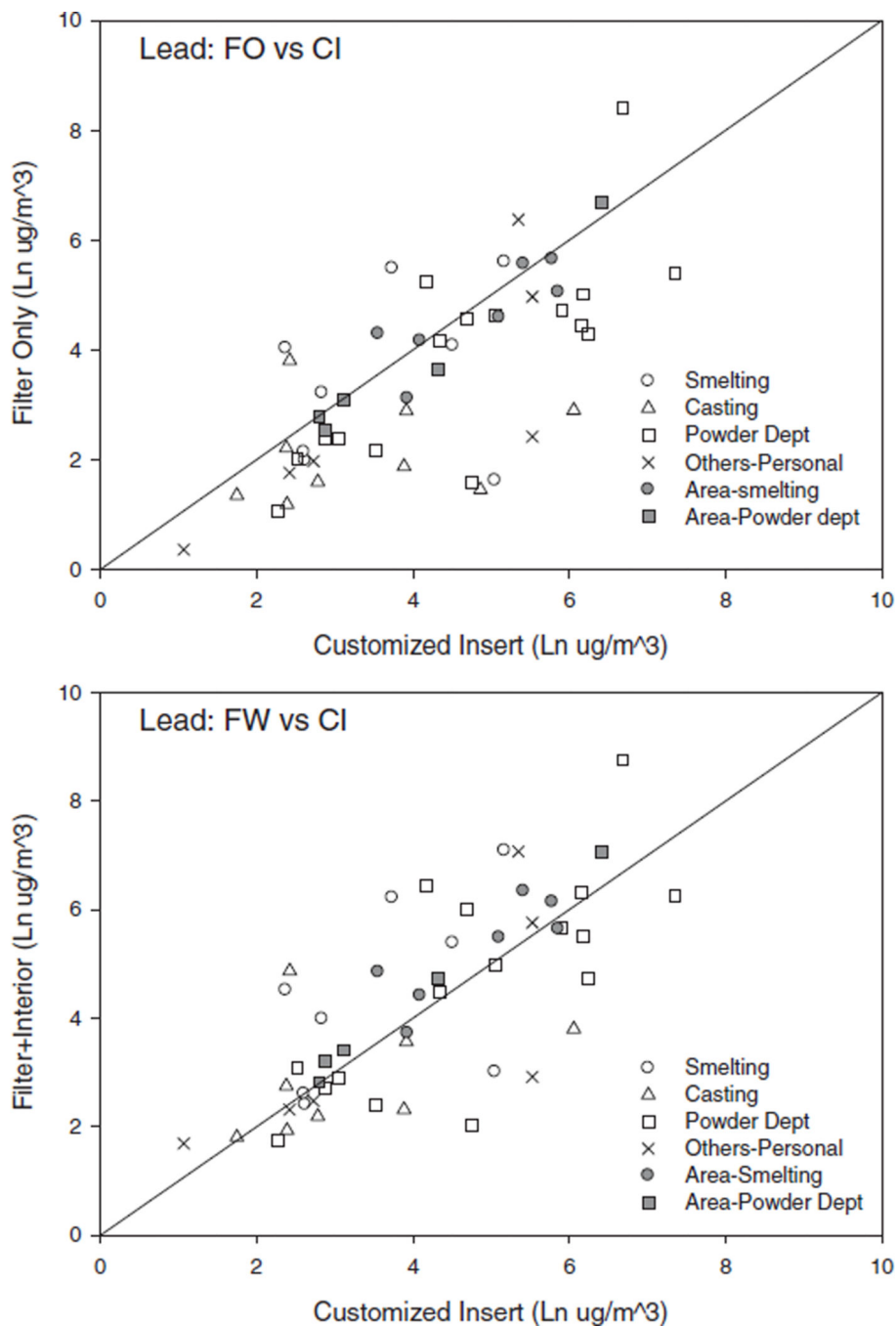


FIGURE 3.

Log-transformed mass concentrations between the pairs of samples for lead. The diagonal line represents 1:1 relationship. ($\text{Ln_FO} = 0.179 + 0.804 \cdot \text{Ln_CI}$ with adjusted $R^2 = 0.492$ and $\text{Ln_FW} = 0.715 + 0.857 \cdot \text{Ln_CI}$ with adjusted $R^2 = 0.531$).

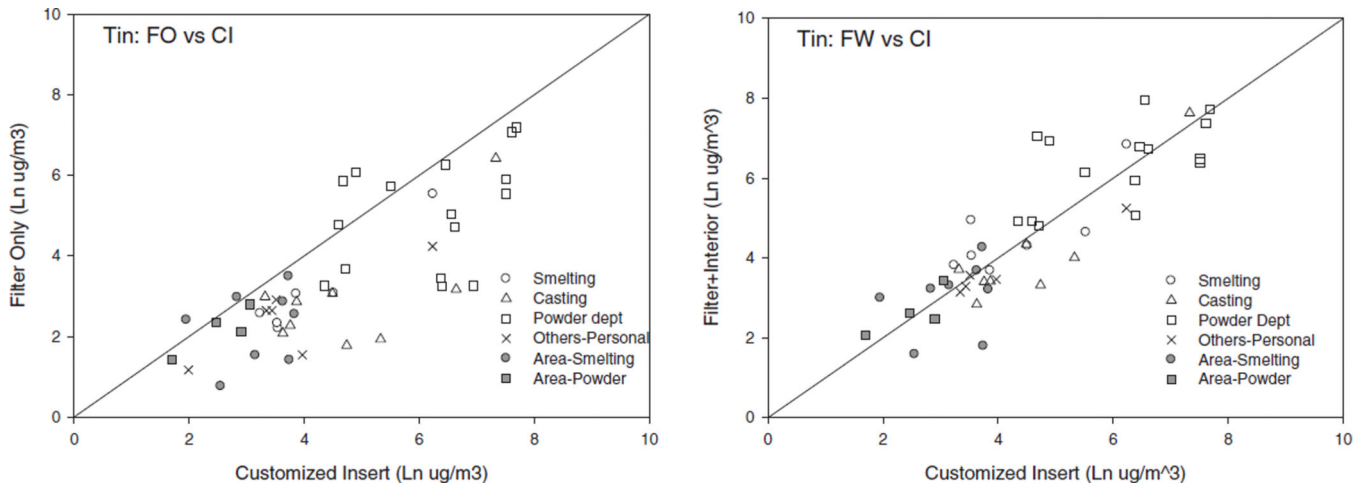


FIGURE 4. Log-transformed mass concentrations between the pairs of samples for tin. The diagonal line represents 1:1 relationship. ($\text{Ln_FO} = -0.0787 + 0.766 * \text{Ln_CI}$ with adjusted $R^2 = 0.592$ and $\text{Ln_FW} = 0.340 + 0.921 * \text{Ln_CI}$ with adjusted $R^2 = 0.745$).

TABLE I

Summary of Exposure Measurements Without Outliers

Sampling Comparison	FO vs. CI ^A			FW vs. CI ^A		
	FO	CI	FW	FO	CI	FW
Lead Combined ^B	Exposure range (µg/m ³) 1.4–4500.0	2.9–1600	5.4–6300	2.9–1600	5.4–6300	2.9–1600
	GM (GSD) ^C 33 (5.5)	63 (4.5)	70 (5.8)	63 (4.5)	70 (5.8)	62 (4.5)
	N ^D	51	50			
Tin Combined ^B	Exposure range (µg/m ³) 2.1–1300	5.5–2200	4.9–2900	5.5–2200	4.9–2900	5.5–2200
	GM (GSD) ^C 31 (5.2)	98 (5.3)	94 (5.4)	98 (5.3)	94 (5.4)	96 (4.9)
	N ^D	49	47			

^A Note that the reported values for the CI from FO/CI and FW/CI comparisons were slightly different because outlier tests were separately conducted for each pair comparison.

^B Combined personal and area exposure measurements.

^C GM (GSD) = Geometric mean (Geometric Standard Deviation).

^D number of sample pairs.

TABLE II

Summary of Statistical Analysis Between Pair of the Samples Without Outliers

Element	Sample pairs	Correlation coefficient (p value) ^A	p value of Proc Mixed (concentration estimates) ^B
Lead	Filter-only(FO)/Customized insert(CI)	0.709 (< 0.0001)	0.0235(CI : 4.24 > FO : 3.77)
	Filter+interior (FW)/Customized insert (CI)	0.735 (< 0.0001)	0.3009(CI : 4.23 \approx FW : 4.44)
Tin	Filter-only(FO)/Customized insert(CI)	0.775 (< 0.0001)	< 0.0001(CI : 4.03 > FO : 3.02)
	Filter+interior (FW)/Customized insert (CI)	0.866 (< 0.0001)	0.8000(CI : 4.04 \approx FW : 4.00)

^A Pearson correlation coefficient with log-transformed concentrations^B log-transformed concentration estimates