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Comparison of a wipe method with and without a rinse to recover wall losses in closed face 37-mm cassettes used for sampling lead dust particulates

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Abstract

Closed-face 37-millimeter (mm) polystyrene cassettes are often used for exposure monitoring of metal particulates. Several methods have been proposed to account for the wall loss in air sampling cassettes, including rinsing, wiping, within-cassette dissolution, and an internal capsule fused to the filter that could be digested with the filter. Until internal capsules replace filters, other methods for assessing wall losses may be considered. To determine if rinsing and wiping or wiping alone is adequate to determine wall losses on cassettes, we collected 54 full-shift area air samples at a battery recycling facility. We collected six replicate samples at three locations within the facility for 3 consecutive days. The wall losses of three replicate cassettes from each day-location were analyzed following a rinse and two consecutive wipes. The wall losses of the other three replicates from each day-location were analyzed following two consecutive wipes only. Mixed-cellulose ester membrane filter, rinse, and wipes were analyzed separately following NIOSH Method 7303. We found an average of 29% (range: 8%–54%) recovered lead from the cassette walls for all samples. We also found that rinsing prior to wiping the interior cassette walls did not substantially improve recovery of wall losses compared to wiping alone. A rinse plus one wipe recovered on average 23% (range: 13%–33%) of the lead, while one wipe alone recovered on average 21% (range: 16%–22%). Similarly we determined that a second wipe did not provide substantial additional recovery of lead (average: 4%, range: 0.4%–19%) compared to the first wipe disregarding the rinse (average: 18%, range: 4%–39%). We concluded that when an internal capsule is not used, wall losses of lead dust in air sampling cassettes can be adequately recovered by wiping the internal wall surfaces of the cassette with a single wipe.

Introduction

Closed-face 37-mm polystyrene cassettes are often used for exposure monitoring of metal aerosols. However, analysis of only the aerosols captured on the filter underestimates the true concentration of airborne particles because sampled aerosols also accumulate on internal walls of the cassette sampler (Demange et al. 1990; Puskar et al. 1991; Demange et al. 2002; Dobson et al. 2005; Harper and Demange 2007; Harper and Ashley 2013). Collected particles will adhere to the internal surfaces of the closed faced cassettes during sample collection, transport, and handling (Baron 1998). Several methods have been

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proposed to account for this wall loss, including rinsing (Ashley et al. 2001), wiping (Hendricks et al. 2009), within-cassette dissolution (Fabriès 1985; Harper and Ashley 2012), and an internal capsule fused to the filter that could be digested with the filter (Harper and Ashley 2013).

Hendricks et al. (2009) found that rinsing alone did not adequately remove metal particulates from the internal cassette walls and suggested that wiping these internal walls was a preferable method to account for wall losses in closed-face air sample cassettes. Inadequacy with rinsing was also reported by Ashley et al. (2001). Hendricks et al. (2009) performed two experiments to explore methods of accounting for wall losses. The first experiment rinsed sample cassettes with 10% nitric acid and then wiped the internal walls of the sample cassettes with a filter moistened with deionized-water. The rinse was analyzed with the sample filter, and the wipe was analyzed separately. For the second experiment, Hendricks et al. (2009) wiped the internal wall surface of the sample cassette twice with a filter moistened with deionized water; the first wipe and sample filter were analyzed together, and the second wipe was analyzed separately. Hendricks et al. (2009) did not analyze the cassette sample filters separately from the rinse in the first experiment or the first wipe in the second experiment. Therefore, comparing the relative amount of analyte in the filters, rinse, or wipe was not possible.

Some methods to account for wall losses have been incorporated into validated sampling and analytical methods. For example, the ASTM International (ASTM 2008; 2010), the International Organization for Standardization (ISO) (ISO 2011), and the Occupational Safety and Health Administration (OSHA) ID 121 (OSHA, 2014) have adopted the use of internal cassette wipes into their analytical procedures. Ashley and Harper (2014) have recommended accounting for wall losses on 30 analytical methods, including NIOSH Method 7303. The most accurate measure of occupational exposures should include all aerosol particles entering workplace air samplers for gravimetric analysis and analytes such as metals and metalloids (Ashley and Harper 2013, 2014).

This article describes the results from a lead monitoring field study using two approaches to determine the recovery of wall losses in closed-face 37-mm polystyrene air sampling cassettes. One approach uses a rinse before two wipes, and the other approach uses only two wipes. The objectives were to 1) determine relative lead recovery in a rinse followed by two wipes compared to the sample filter and assess if a rinse was necessary to accurately account for wall losses in the cassette and 2) determine the relative lead recovery in the first and second wipe compared to the sample filter, and assess if a second wipe was necessary to accurately account for wall losses.

Method

We collected 54 full-shift area air samples (average sampling time of 460 minutes) at a secondary lead smelting and processing facility that recycled lead-acid vehicle batteries. The sampling train included closed-face 37-mm polystyrene cassette samplers with 0.8-micrometer pore-size, mixed-cellulose ester membrane (preloaded filter cassette, SKC part no. 225-3-01) attached with Tygon® tubing to SKC AirChek 2000 pumps operating at a nominal air flow rate of 2 liters per minute. Pumps were calibrated before and after sampling with a BIOS DryCal® DC-Lite-H air flow calibrator. We attached the sample media to tripods or to a building support beam at a height of 3 to 6 feet above floor level. We collected six replicate samples at three locations within the battery recycling facility for 3 consecutive days (Table I). The first sample location was the end of a conveyor where workers unloaded the batteries from pallets (Figure 1). The conveyor carried the batteries to an unventilated shredder that separated plastic from lead battery components. The second location (Figure 2) was between two rotating furnaces. Employees used large front-end loaders to transfer the lead battery components to the furnaces to melt. Molten lead from each furnace was transferred to kettles (Figure 3) for further refining. The third sampling location was between two of these kettles.

Three of the six cassettes collected at each location each day were randomly assigned into a “no rinse” group, with the remaining three assigned into a “rinse” group (Table I). The three cassettes in the no rinse group were wiped with two filters moistened with deionized water, consecutively. Each wipe was analyzed and results reported separately. The three cassettes in the rinse group were first rinsed with deionized water followed by wiping the interior cassette surfaces with two deionized water moistened wipes, consecutively. Wipes were collected using a 0.8-micrometer mixed-cellulose ester membrane filter, the same type used for the sample filter. Sample filters, rinses, and wipes were analyzed separately for lead by inductively coupled plasma-atomic emission spectroscopy following NIOSH Method 7303 (NIOSH 2014). At least two field blanks were collected each day and included in the analysis. No blank correction was necessary.

Data Analysis

Because replicates were collected at the same time, the total mass of lead recovered was the main variable considered in the analysis. We calculated percent recovery by first calculating the total mass of lead recovered on the filter and wall losses recovered by wipes and rinse (if performed) [Equation 1]. We then compared the measured mass for each separate analysis, “*i*”, of the filter, rinse, wipe 1, and wipe 2 to the total mass recovered for that sample [Equation 2]. Recovery in this manuscript refers to the total amount of lead that was recovered from what was present inside the samplers.

$$Total\_\ mass = mass_{filter} + mass_{rinse} + mass_{wipe1} + mass_{wipe2} \quad \text{Equation 1}$$

$$\mathrm{Recov}\,ery_i = \left(\frac{mass_i}{Total\_\ mass}\right)(100) \quad \text{Equation 2}$$

We used SAS Institute Inc. version 9.3 software to compare the lead recovered from the no rinse and rinse groups. We compared the means and medians of the total mass of the two groups using t-tests and the Wilcoxon Scores (rank sums) tests. Using the same tests, we compared the means of the first wipe (in the no rinse group) to the rinse and first wipe (in the rinse group).

We also compared the cumulative percent recovery of lead on the first and second wipes by examining the frequency of the wipes having greater than 5 and 10 cumulative percent recovery.

Results

Table II presents the total mass and average concentration of lead in air per triplicate samples by location of the samplers. Detailed data are included in supplemental Tables I–III. The highest geometric mean concentration by location, across the 3 days of sampling (Table II), occurred in the battery breaker (150 micrograms per cubic meter, $\mu g/m^3$), followed by the furnace (120 $\mu g/m^3$), and kettle (110 $\mu g/m^3$). The highest geometric mean concentration throughout the facility by averaging all samples per day was 180 $\mu g/m^3$ on day 3, followed by 120 $\mu g/m^3$ on day 1, and 89 $\mu g/m^3$ on day 2. (Geometric means per day are not shown in Table II.) The lead recovered from the walls of the cassettes accounted for an average of 29% (range: 8%–54%) of the total lead in the samplers. In a majority of our samples (41 out of 52), more than 20% of the total lead in the cassette was on the interior cassette walls.

Comparing the means and medians of the total lead mass collected by no rinse and rinse samples showed that the no rinse group had a mean of 125 micrograms (µg), and the rinse group had a mean of 123 µg. Neither the t-test (*P* value 0.88) nor the Wilcoxon scores rank sum test (*P* value 0.94) saw a statistically significant difference between the two groups. We also compared the means of the wipe 1 (no rinse group) versus rinse plus wipe 1 (rinse group). Neither the t-test (*P* value 0.66) nor the Wilcoxon scores rank sum test (*P* value 0.78) saw a statistically significant difference between the two groups. The mean percentages of the lead wall losses for the no rinse and rinse groups were very similar (Figure 4).

On average, 74% (range: 67%–81%) of the total lead was recovered from the filter from the no rinse group. The first wipe (with no rinse) contained, on average, 21% (range: 16%–22%) of the total lead recovered. The second wipe recovered, on average, 5.3% (range: 2.7%–7.8%) of the total lead recovered. Together, the filter and first wipe recovered, on average, 95% (range: 81%–99.6%) of the total lead. Figure 4 shows the mean percent of lead recovered on the filter, wipe 1, and 2 of the no rinse group.

On average, 74% (range: 63%–84%) of the total lead was recovered from the filter from the rinse group. The rinse recovered 9.8% (range: 5.4%–16%) of the total lead, but the combination of a rinse and wipe contained 23% (range: 13%–33%) of the total lead. The second wipe recovered, on average, 3.2% (range: 2.5%–3.7%) of the total lead. Figure 4 shows the mean percent of lead recovered on the filter, rinse, wipe 1, and wipe 2 of the rinse group.

Most of the lead recovered from the walls of the cassettes was found on the first wipe (Figure 4 and supplemental Tables I–III). For all samples, disregarding the rinse, the average percentage of lead recovered on the first wipe was 18% (range: 4.3%–39%) of the total lead, compared to 4.4% (range: 0.4%–19%) of the total recovered on the second wipe. Approximately 75% of the first wipes had greater than 10% cumulative recovery, and 96%

had over 5% cumulative recovery, including those that were first rinsed. Only 4% of the second wipes had greater than 10% cumulative recovery and 39% had greater than 5% cumulative recovery.

Discussion

In this study, the average percentage of total lead recovered on the filter of the air sampling cassettes was 74% (range: 46%–92%), with recovery increasing to 95%–97% with the addition of a rinse followed by a single wipe or wiping alone without a rinse. Unlike Hendrix (2009), in which 27% of samples did not have metal analyte on the walls of the cassette, all of our samples had quantifiable amounts of lead on the internal cassette walls, similar to that described by Harper et al. (2006). Our results corroborate that laboratory analysis of only the filter portion of the cassette sample underestimates actual airborne lead concentration. On average, the lead we recovered from the walls of the cassettes accounted for 29% of the total lead in the samplers, which agrees with previous work (Harper and Demange 2007; Lee et al. 2014). For a small number of our samples, airborne lead concentration found by incorporating wall losses in laboratory analysis was nearly double that found by analyzing only the cassette filter. Analyzing only the filter may lead to erroneous interpretation of airborne monitoring results and could affect selection of appropriate control measures or decisions about which employees should be included in medical monitoring programs. Further, erroneous interpretation of airborne monitoring results (i.e., not including wall losses) can result in challenges when pooling data from different studies to understand the relationship between employee exposures and biomonitoring data - which has being used historically to guide occupational exposure limits.

Our first objective was to assess if a rinse before wiping was needed to assess wall losses adequately. We found that rinsing with deionized water alone is not adequate to remove lead from the internal cassette walls. However, rinsing followed by a single wipe or just wiping alone without a rinse recovered, on average, approximately 85% of the amount of lead on the internal sampling cassette walls. We found the difference in total lead recovered from a rinse and one wipe (22.8%) versus a wipe only (21.0%) was minimal. Therefore, recovering lead from the internal cassette wall using a wipe alone may be preferred over the combination of a rinse and a wipe because it is less labor intensive and yields similar results.

Our second objective was to confirm if a second wipe was needed to assess wall losses adequately. We found that a second wipe did not add sufficient recovery (average of 4%) and its use, therefore, was not likely to outweigh the extra cost and analysis time required for a second wipe.

Had these area samples been personal samples, the airborne lead concentrations in all but two of the samples would have been above the OSHA permissible exposure limit (PEL) of 50 μg/m^3, and the majority of our samples were more than double the OSHA PEL. It is unclear how wall losses would affect samples in this particular workplace if most concentrations were below the OSHA PEL or OSHA action level of 30 μg/m^3. However, there is a risk of underestimating actual exposures if only the filter is analyzed. Particles containing lead have shown no qualitative difference in the size distribution when aerosols deposit on the filter or on the internal surfaces of the cassette in controlled laboratory (Lee et al. 2009) and field (Chisholm et al. 2012) experiments.

Our analysis was limited to recovering airborne lead dust from internal walls of polystyrene cassettes. The results may be different for other metal or non-metal particulate. Differences in type of filter and air sampling flow rate may affect behavior of particles in the airstream within the cassette sampler and analyte losses on cassette walls. Furthermore, the cassette and filter materials and their interaction with the analyte may also affect the amount of analyte deposited on the cassette walls. For example, highly charged particles may have greater attraction to certain plastic cassette walls than particles with lesser charges.

While our sample size is small, we feel it is representative of results that are likely to be obtained in the field. The sampling strategy was designed to take into account expected natural variability among the sample replicates as well as variability across sampling days and sampler location. Multiport samplers offer superior uniformity of aerosol collection among a particular batch of samples (Ashley et al. 2009). However, our approach reflects replicate samples taken during a typical field sample collection. Future studies could compare the approach described here along with the use of internal capsules in a multiport sampler.

Conclusion

We concluded that wall losses for lead aerosols can be adequately recovered by wiping the interior wall of the cassette with a single wipe. We found that rinsing of the interior cassette walls before wiping produced comparable recovery to wiping alone. We also found that a second wipe provided no substantial additional recovery of lead compared to the first wipe. Therefore, we do not recommend the use of a rinse or a second wipe to recover wall losses for metals like lead. NIOSH researchers recommend the use of an internal capsule as the ideal way to account for wall losses in a non-gravimetric sample (Harper and Ashley 2013; Lee et al. 2014). However, when an internal capsule is not used, wall losses can be accounted for by wiping the interior surfaces of the cassette with one wipe, thus providing a more accurate characterization of total airborne lead exposure.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Figure 1.
Area air sampling location 1 – sampling pumps were located by the battery breaker conveyor

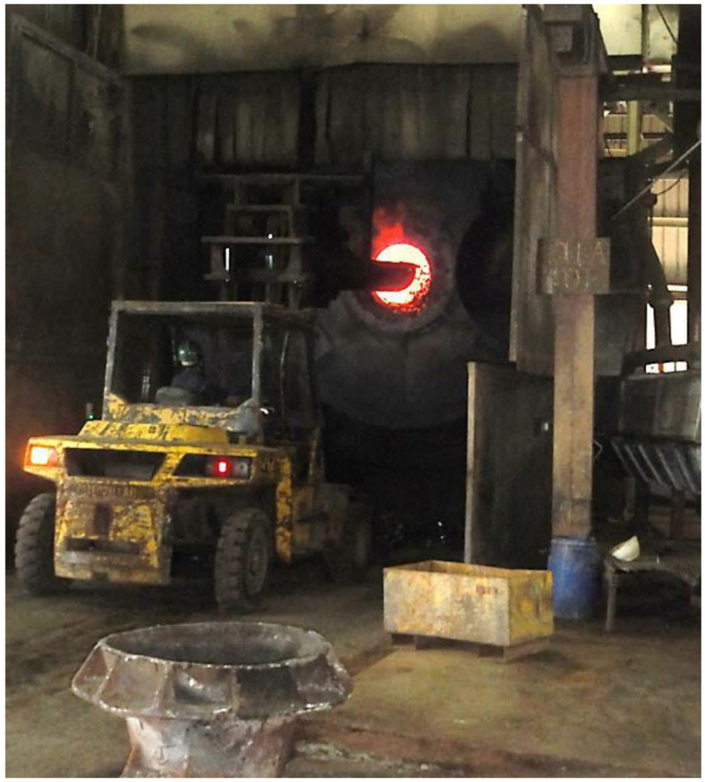

Figure 2.
Area air sampling location 2 – sampling pumps were located between two furnaces; the photo shows one of these furnaces being loaded.

Figure 3.
Area air sampling location 3 – sampling pumps were located between two kettles

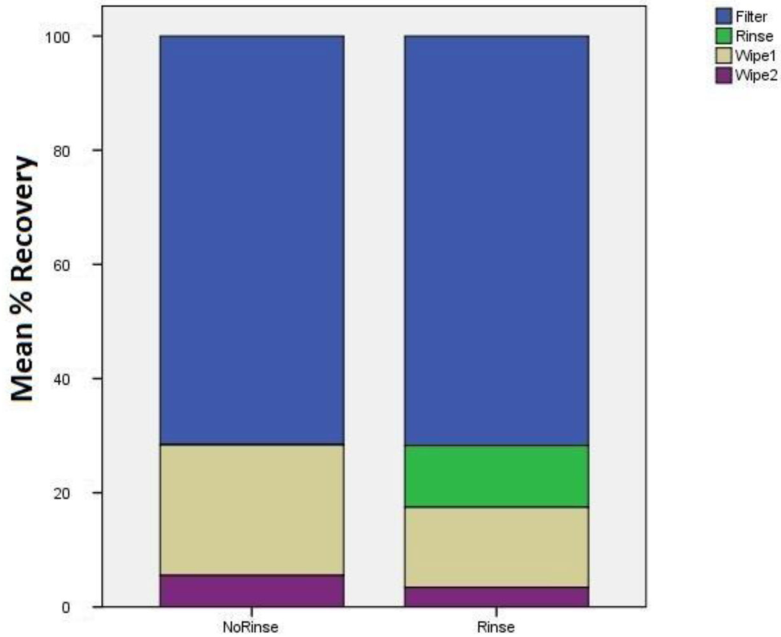

Figure 4.
Average percent lead recoveries for samples with no rinse versus samples with a rinse

Table I

Number of samples and type of analysis performed

			Number of samples	
Location	**Location name**	**Description**	**No rinse filter, wipe 1, wipe 2**	**Rinse filter, rinse, wipe 1, wipe 2**
1	Battery breaker	Near in-feed of battery breaker conveyor	3 replicates × 3 days = 9	3 replicates × 3 days = 9
2	Furnace	Between two furnaces	3 replicates × 3 days = 9	3 replicates × 3 days = 9
3	Kettle	Between two kettles	3 replicates × 3 days = 9	3 replicates × 3 days = 9

Table II

Average total lead mass collected in the sampler and air concentration for the triplicate samples

No rinse or rinse	Day	Average total lead mass (µg)	Average time (minutes)	Average volume (Liters)	Average concentration* ($\mu g/m^3$)
Location 1 – battery breaker					
No rinse	1	102	432	857	120
	2	105	459	914	110
	3	204	442	882	230
Rinse	1	91.2	432	863	110
	2	133	540	1088	120
	3	221	442	877	250
				Location 1 geometric mean	110
Location 2 – furnace					
No rinse	1	65.3	434	859	76
	2	90.2	502	1004	90
	3	203	428	853	240
Rinse	1	69.3	411	816	85
	3	101	502	1000	100
	2	219	428	857	260
				Location 2 geometric mean	150
Location 3 – kettle					
No rinse	1	186	385	762	250
	2	78.1	509	1016	77
	3	92.1	432	870	110
Rinse	1	131	436	864	150
	3	61.1	509	1022	60
	2	78.7	432	873	90
				Location 3 geometric mean	120

* Average concentration rounded to two significant digits