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# ASSESSING PARTICULATE MATTER WALL LOSS WITH CONDUCTIVE VS. NON-CONDUCTIVE CASSETTES USED FOR AIRBORNE MINERAL DUST SAMPLING

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# ASSESSING PARTICULATE MATTER WALL LOSS WITH CONDUCTIVE VS. NON-CONDUCTIVE CASSETTES USED FOR AIRBORNE MINERAL DUST SAMPLING

by

**Curtis Carroll** 

A thesis submitted in partial fulfillment of the requirements for the degree of

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### Abstract

Occupational exposure assessments for aerosols are commonly conducted by drawing air through a filter media, which is housed in cassette. Previous studies assessing metal and coal dust aerosol concentrations have shown a significant amount of particulate matter adhering to the inner cassette wall, thus underestimating the mass concentration reported from filter media. A common control strategy to minimize wall loss is to perform sampling with cassettes comprised of conductive material. The objective of this study was to assess the potential for cassette wall loss associated with mineral dust air sampling techniques employing conductive vs. nonconductive cassettes. Area total and respirable dust sampling was conducted in a primary rock crusher of a copper mine. In addition to analyzing the filter media, the interior of the sampling cassette was wiped with a PVC filter post sampling to quantify potential wall loss. Results revealed a significant decrease (P<0.05) in percent wall loss mass with conductive vs. nonconductive cassettes when sampling for respirable dust. A correlation was apparent (P=0.048) between wall loss mass and filter mass for conductive cassettes when using respirable dust sampling methods. However, no correlation was apparent between wall loss mass and filter mass for non-conductive cassettes when using respirable dust sampling methods. No correlation was apparent between wall loss vs. filter mass for non-conductive and conductive cassettes when applying total dust sampling. These results suggest that substantial (mass %) sample loss may occur when sampling for respirable mineral dust with non-conductive cassettes, and this wall loss may be mitigated with the use of a conductive sampling cassette.

Keywords: Air Sampling, 37 mm Conductive Cassette, Wall Loss, Electrostatic Effects

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## **Table of Contents**

ABSTRACT		
ACKNOWLEDG	EMENTS	
LIST OF TABLES	j	V
LIST OF FIGURE	۶	VI
1. INTR	ODUCTION	1
2. BACK	<ground< th=""><th></th></ground<>	
2.1	Particulate Matter and Health Effects	
2.2	Particulate Matter Occupational Exposure Limits	
2.3	The Evolution of PM Sampling Techniques	5
	2.3.1 Respirable Dust Sampling Techniques	6
	2.3.2 Total Dust Sampling Techniques	6
2.4	Previous Studies Evaluating Wall Loss	7
2.5	Recovery Strategies for Wall Loss	
3. Овје	CTIVE	
4. Met	HODS	
4.1	Statistical Analysis	
5. Resu	JLTS	
6. Disc	USSION	
6.1 Sti	udy Limitations	
7. Con	CLUSION	
8. Refe	RENCES	
Appendix	A	

# List of Tables

Table I: Particulate matter size-selective occupational exposure limits and 50% cut point	ıts4
Table II: Summary of the sampling schedule	20
Table III: Summary Results of Particulate Mass Collected per Cassette Type/Method	26
Table IV: Wall Loss, Filter, and Combined Mass Concentration	40
Table V: Wall Loss, Filter, and Combined Masses	.41

# List of Figures

Figure 1. Illustration of paired sampling methodology employed for each sampling trial21
Figure 2. A comparison of mean wall loss percentage differences between conductive vs. non
conductive cassettes when sampling for respirable dust
Figure 3. A comparison of mean wall loss percentage differences between conductive and nor
conductive cassettes when sampling for total dust
Figure 4: Respirable wall loss mass vs. filter mass
Figure 5: Total wall loss mass vs. filter mass

### 1. Introduction

Along with anticipating and recognizing potential hazardous exposures in the workplace, a critical component of industrial hygiene is to adequately evaluate exposures and implement control strategies if deemed necessary. One exposure of interest is particulate matter (PM). Evaluation of PM exposures typically involves air sampling techniques where air containing aerosol is drawn through filter media and then analyzed. One such way to analyze the filter is by gravimetric analysis. This consists of weighing the filter pre and post sampling to determine the aerosol's gravimetric mass. This mass, along with the volume of airflow, is then used to derive a mass/volume concentration which may then be compared to applicable occupational exposure limits (OELs). An OEL is a general term for the maximum acceptable concentration of a hazardous substance to which a worker can be exposed for a duration of time. Specific OELs are set by governmental or other organizations and are either enforced by legislation or recommended as best practices to protect worker health (Anna, 2011, p. 57).

Workplace aerosol sampling methods commonly specify the use of 37 mm closed-face cassettes when collecting workplace air samples (Ashley & Harper, 2013). These cassettes, which are commonly comprised of polycarbonate, polytetrafluoroethylene, polyvinyl chloride (PVC), and polystyrene materials, retain high charge levels and, as a result, attract PM to the walls of the cassette (Baron, 2003). This interception of PM on the cassette wall surface is referred to as "wall loss" (Ashley & Harper, 2013; Harper & Demange, 2007; Baron, 2003). Studies involving metal aerosols and coal dust have revealed that wall loss reduces the amount of particulate matter detected on the filter media (Baron & Deye, 1990; Ashley & Harper, 2013; Ceballos, King, Beaucham, & Brueck, 2015). Therefore, wall loss, if not accounted for, may

result in an under estimation of worker exposures (Witschger, Grinshpun, Fauvel, & Basso, 2004).

To minimize wall loss, some sampling methods recommend the use of conductive sampling cassettes (Ashley & Harper, 2013). Conductive sampling cassettes are comprised of carbon filled polypropylene and are designed to minimize wall loss by evenly distributing charge across the cassette surface, thus reducing the potential for PM deposition on the cassette walls (Baron, 2003). The evenly distributed charge of conductive cassettes minimizes the potential for particle trajectory and results in a more symmetrical particle deposition, thus enhancing filter capture efficiency and reducing wall deposition (Baron & Deye, 1990).

Addressing the impact of potential wall loss associated with non-conductive and conductive cassettes used for dust sampling may contribute to the science of aerosol collection and analysis, with outcomes potentially influencing industrial hygiene exposure assessments and procedures in numerous industries ranging from mining to construction.

Additionally, information gained from this study may be beneficial to the design of the Fourier Transform Infrared (FTIR) direct-reading instrument, which is used to monitor respirable silica, allowing for an end-of-shift estimate of respirable crystalline silica in the field without subsequent laboratory analysis. The Office of Mine Safety and Health Research, Pittsburgh, a division of National Institute for Occupational Safety and Health (NIOSH), has initiated a program to evaluate the application of a portable, direct-reading FTIR analyzer in conjunction with a direct-on-filter approach for field use to monitor exposure to respirable crystalline silica (Cauda, 2016; Hart et al, 2018). This FTIR design incorporates the use of conductive 37mm filter cassettes. Data obtained through this research may validate the use of the conductive cassettes with this FTIR instrument.

#### 2. Background

#### 2.1 Particulate Matter and Health Effects

There are various forms of PM in occupational and ambient environments. Dusts are a form of PM that are typically generated from crushing, grinding or other physical processing of materials such as rock, ore, metal, and coal (Anna, 2011, p. 332). Health effects can vary depending on the type of dust, duration, and frequency of exposure. In addition, factors affecting the toxicity of dusts include the size, shape, elemental composition, surface properties of the dust, and the concentration inhaled (Klaassen, Casarett, & Doull, 2018). Health effects caused from inhalation of dust is generally termed pneumoconiosis which simply means "dusty lung". A wide variety of diseases can result from inhalation of dust, such as but not limited to asbestosis, silicosis, coal pneumoconiosis, and stannosis (Anna, 2011, p. 90). For example, respirable silica dust exposure is a causal factor for silicosis. Dust, such as carbon or iron, can cause mild damage to the lung without scarring.

Numerous human airway models have been used to characterize particulate matter deposition in different regions of the respiratory tract. The deposition of particles in the respiratory tract depends on factors, such as geometry, flow that changes with time, cycles in direction, particle deposition mechanisms, and the aerodynamic diameter of the particle (Hinds, 1999). Aerodynamic diameter (AED) is a common way of describing the size of a particle in terms of how the particle travels in air and standardizes irregularly shaped particles based on their shape and density (Hinds, 1999).

The site of respiratory deposition of particles often co-relates with specific health effects. As a result, many OELs involve size-selective particle sampling techniques, in order to provide effective worker protection. Occupational exposure limits for particulate matter typically include particle size-selective criteria which can range from 100 microns (µm) to less than 1 micron in aerodynamic diameter (Hinds, 1999).

#### 2.2 Particulate Matter Occupational Exposure Limits

There are numerous regulatory and best practice OELs designed to minimize PM exposures in various industries. The Mine Safety and Health Administration (MSHA) and the Occupational Safety and Health Administration (OSHA) have PM permissible exposure limits, which include both respirable and total size-selective criteria. Whereas the American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit values (TLV) include criteria for inhalable, thoracic, and respirable size-selective criteria (as illustrated in table 1). Size-selective sampling techniques are used in order to meet these criteria. The collection efficiency of particle size-selective sampling techniques is commonly denoted by the 50% cut point. This means that at the specified cut point, 50 % of the particles at this size will be collected. Particles smaller than the cut point will be collected with an efficiency greater than 50%, whereas particles larger than the cut point will be collected with an efficiency less than 50% (Hinds, 1999, p. 125).

Agencies	Technique	50% Cut Point			
	Inhalable	100 µm			
ACGIH	Thoracic	10 µm			
	Respirable	4 µm			
	Total Dust	*30 µm			
υδπα	Respirable Dust	4 µm			
MOTIA	Total Dust	30 µm			
мэна	Respirable Dust	4 µm			
		1 2000)			

Table I: Particulate matter size-selective occupational exposure limits and 50% cut points

\*Estimate (Hinds, 2009)

Size-selective OELs used by ACGIH include three techniques: inhalable, thoracic, and respirable. The inhalable fraction includes particles that may be deposited anywhere within the

respiratory tract (ACGIH, 2019, p. 79). The thoracic fraction includes particles that may deposit within the lung airway and the gas-exchange region (ACGIH, 2019, p. 79). The respirable fraction includes particles that are small enough to reach the gas exchange region (ACGIH, 2019, p. 79). In order to capture the inhalable fraction of PM for exposure assessment purposes, an inhalable monitor, such as an Institute of Occupational Medicine (IOM) sampling device, is used (Hinds, 1999, p. 249). While MSHA and OSHA both refer to samples collected with a 37 mm closed-face cassette as total dust, total dust sampling techniques may underestimate the inhalable fraction of dust by a factor of 2.5 for larger particles such as mineral dust (Hinds, 1999).

#### 2.3 The Evolution of PM Sampling Techniques

Sampling for particulate matter has evolved over the years. Initial PM sampling was performed with the use of impingers which utilized a specially designed bubble tube to draw dust-laden air through a liquid medium, most commonly water. At the conclusion of sampling, the liquid was poured onto a counting surface and particles were counted through a microscope. This was the most common technique used for determining aerosol concentrations in the 1950's and throughout the 1960's (Harper & Demange, 2007). The impinger was eventually replaced with a filter collection technique which allowed for an efficient means of collecting aerodynamically fine particles.

The 37 mm closed-face cassette (CFC) was created to house the filter media and allow air to be drawn through. Along with the evolution of filter media, the composition of the sampling cassette evolved from metal to plastic Tenite (Harper & Demange, 2007). Tenite material was later found to kill many organisms during sampling and was later replaced by polystyrene material which was more suitable for microbiological applications (Harper & Demange, 2007). The polystyrene sampler eventually became known as the "cassette" and consisted mainly of a 37 mm diameter filter. The cassette was being used in industry between 1970 and 1975 and eventually replaced the impinger by 1980 (Harper & Demange, 2007). Along with the evolution of the polystyrene filter cassette method, particle size-selective techniques were developed in order to make samplers more representative of the human air way.

#### 2.3.1 Respirable Dust Sampling Techniques

Respirable dust sampling is typically accomplished with a sampling pump, tubing, filter media, a 3-piece filter cassette, and a particle size-selective device such as a cyclone. The cyclone separates the respirable fraction of particles based on aerodynamic diameter and consists of 3 pieces: inside, base, and apex. The inside is configured as a cone. The base of the cone is located towards the top having short cylindrical sections containing a feed inlet, a fixed vortex finder, and an overflow orifice. The apex is pointed down having a cap at the bottom that acts as a grit pot for oversized particles. The cyclone operates with two separate flows, one within the other. Centrifugal acceleration forces the heavier particles to the outside spiral, next to the wall of the cylinder. The lighter particles rotate in the same direction but upwards, exiting through the vortex finder and overflow orifice to eventually be captured by the filter. The larger particles exit through the apex and are captured by the grit pot (Mineral Dust-Gravimetric Method, 2006).

#### 2.3.2 Total Dust Sampling Techniques

Total dust sampling consists of an air pump, tubing, filter media, and a 2-piece filter cassette. The filter media is placed inside the cassette. The tubing is connected between the air pump and one end of the cassette. The pump draws particulate-containing air through the cassettes opposite end. The particulate matter is captured on the filter media as the air passes through the cassette. After sampling, the filter media is then removed from the cassette and analyzed.

#### 2.4 Previous Studies Evaluating Wall Loss

When comparing sampling of particulate matter with applicable OELs, it was originally assumed that PM entering the sampling cassette was collected on the filter media and no other component of the sampling cassette was analyzed (Harper & Demange, 2007). However, as early as 1990, studies revealed that PM deposited on the walls of cassettes could comprise a significant portion of total PM that entered the cassette.

A study by Demange, Gendre, Herve-Bazin, Carton, & Peltier (1990) compared two different methods for analyzing aerosol sampler filters and wall-particle depositions. The first method analyzed only particles collected on the filter. The second used a chemical digestive method, which is performed directly inside the cassette. The analysis was performed in two steps: gravimetric determination of total dust sampling by weighing the filter alone, then chemically digesting the same filter along with contents from the cassette's inner walls. Fiftynine measurements were conducted using an open filter configuration while another 286 measurements were conducted suing a closed filter configuration. Results that showed losses can vary from 0 to 100%. Particle deposition on cassette walls can come from losses during transit or during sampling. Overall, Demange et al. (1990) concluded that particles depositing on the cassette walls are sometimes ignored, are clearly part of the sample fraction, and if not used can result in underestimation of exposure, compared with analyzing only the filter.

Particle size distributions of laboratory generated lead aerosols ranging in size from 5 μm through 20 μm were evaluated for IOM and CFC samplers (Lee, Chisholm, Slaven, Harper, 2009). The wall and filter deposits from each sampler were quantified by scanning electron

microscopy and characterized separately. Mann Whitney statistical analysis revealed airborne lead particles sized 20 µm or less had no quantitative difference in size distributions of particulate deposits on the filter or on the walls of both samplers (Ashley & Harper, 2013). A similar lead-dust field study was performed to evaluate and compare mass-weighted size distributions of aerosols captured on the filter and internal walls of the CFC and IOM samplers in environments where larger particles may be present (Chisholm, Lee, Slaven, Nelson, Harper, 2012). This study revealed that particle size distribution of material on the walls of the paired (conductive) IOM and (non-conductive) CFC area samplers was indistinguishable. However, the study noted few particles greater than 20 µm were observed, suggesting that larger particles may be rare. The study concluded that the CFC and IOM filter and wall deposit results are very similar when only particles of less than 20 µm are present (Ashley & Harper, 2013). While conductive sampling cassettes minimize wall deposits due to static attractions, these wall losses are not eliminated with conductive cassettes (Baron, 2003; Ashley & Harper, 2013).

A study conducted by Puskar, Harkins, Moomey, & Hecker, (2010) measured pharmaceutical dust wall loss on 37 mm closed-face cassettes. The study's first objective was to determine if a negative sampling bias occurs during pharmaceutical monitoring due to internal cassette wall losses. Fifteen pairs of cassettes were collected concurrently as area samples over an 8-hour time period in a production area. Each cassette was filled with an extraction solvent, removed, and then analyzed. The study concluded a negative bias error finding 62% of sampled dust to be on the inside surface of the cassette tops. Only 22% of the dust was found on the filters and 16% was found on the inside of the cassette's bottom. Electrostatic effects appeared to be the reason for particle deposition. The humidity of air sampled may have a significant impact in the magnitude of attraction. Particle deposition on cassette walls was found to be variable, affecting 2-100% of the dust collecting on 37 mm cassettes. Baron (2003) references studies finding 22% of particles on the filter and 65% on the cassette. In one study it was found that wall deposits could be eliminated by incorporating corrective measures such as the use of conductive cassettes, reducing wall loss by up to 30%. These losses seem to be caused by a combination of electrostatic charge, inertial, gravitational, and diffusion mechanisms.

A study by Demange, Gorner, Elcabache, & Wrobel (2002) compared wall losses between the 37 mm CFC and IOM sampler and were conducted in three different plants containing lead, bronze and iron. Each cassette type was hung side by side during sampling. After sampling, the filters were removed and gravimetrically weighed. After weighing, acid was used to digest the filter and added to the cassettes solubilizing its wall deposits. The solution was then analyzed using an inductively coupled plasma. The filter gravimetric results from the IOM showed a higher collection efficiency than that of the 37 mm cassette. However, the collection efficiency between both samplers was nearly identical if wall losses from the 37 mm cassettes were included (Demange, et al., 2002). The study notes most aerosol samplers have wall loss deposits but not all recovery is feasible.

A chamber study evaluating wall loss mass between different types of respirable particle size-selective samplers when sampling for quartz laden coal dust was conducted by Charm, Kashon, & Harper (2014), using three piece polystyrene (non-conductive) and static dissipative polypropylene (conductive) cassettes in combination with four different types of cyclones. Each post weighed filter was placed inside a porcelain crucible and ashed using a muffle furnace. The inner wall of each cassette was wiped visually clean using wetted PVC filters and then ashed using a porcelain crucible. The ashed sample was redeposited on an acrylic copolymer filter. The quartz mass was quantified by using a Fourier-Transform Infrared spectroscopy (FTIR) and the Omnic software package. The results found the mean percent wall loss of 17.9% for the 37 mm polystyrene cassette and a mean wall loss of 3.73% for the 37 mm static dissipative cassette (Charm et al, 2014). The test results revealed that the mass fractions from the inner cassette surfaces were significantly different (p<0.05) between conductive and non-conductive cassettes (Charm et al, 2014). The study suggested that the interior surface deposits on non-conductive cassettes with particle size-selective cyclones can be a substantial part of the sample, and the authors recommended that interior surface deposits be included in any analysis for most accurate exposure assessment (Charm et al, 2014). Furthermore, samples taken with the conductive cassettes interior walls (Charm et al, 2014). The authors concluded that internal surface deposits in conductive cassettes used with cyclones are negligible, and only the filter needs to be analyzed (Charm et al, 2014). However, particle deposition on inner cassette surfaces vary with particle size and this recommendation does not extend to systems sampling larger particles (Charm et al, 2014).

A major deposition mechanism proposed to explain wall loss is particle charge and electrostatic attraction. Aerosol particles generated in the workplace have high charge levels. After suspending in air for a couple of hours, particles usually achieve minimum charge due to interacting with naturally occurring ions. Freshly fractured aerosols have been demonstrated to be charged. When these freshly generated particles are sampled with highly charged samplers, particle trajectory may be modified to an extent that particles are insufficiently sampled. Electrostatically induced particle motion does not occur when the charge on both particle and sampler are zero. When both particle and sampler are highly charged, particle acceleration is much greater than that of gravity, inertia, diffusion, or other mechanisms (Baron, 2003). Samplers made from certain non-conductive plastic materials, such as polyvinyl chloride and polystyrene, readily retain high charge levels and exhibit particle losses to cassette walls. Whereas conductive samplers can have lower losses when sampling charged particles by adequately distributing its charge across its surface. Not only the cassette but the type of filter used during sampling can introduce unwanted electrostatic effects. Filters made of nonconductive material retain less water weight but can retain high charge levels resulting in nonuniform particle deposition and can repulse particles from the filters surface. It's important to note more weight stable materials tend to be more statically charged resulting in increased particle repulsion and deposit non-uniformly (Baron, 2003).

The earliest research evaluating particle charge, collection efficiency and sampling uniformity was performed for asbestos fibers in various workplaces using a variety of sampling cassettes and it concluded that the effects of electrostatic charge were dependent on particle charge, sampler charge, sampler conductivity, and sampling flow rate as well as direction (Baron & Deye, 1990). Nonconductive cassettes distribute their electrostatic charge unevenly resulting in a non-uniform distribution of particles on the filter during sampling (Baron & Deye, 1990). This uneven distribution of charge can generate complex electrostatic fields, depending on the location, magnitude, and polarity of its charge (Baron & Deye, 1990). Conductive cassettes distribute charge evenly across their surface, resulting in a more symmetric distribution of particulate on the filter during sampling (Baron & Deye, 1990). Electrostatic charge can affect a particle's trajectory, thus its final point of deposition (Baron & Deye, 1990). Electrostatic effects can be influenced by both humidity and a sampler's air flow rate. It is postulated as humidity decreases below 15%, a strong increase in electrostatic charge occurs. Furthermore, increasing the sampling flow rate increases filter efficiency due to the decreased time particles have to interact with the static field generated by the cassette (Baron & Deye, 1990).

As asked by Harper & Demange (2007), "what exactly is the sample," and "should wall deposits be included?" are both fundamental questions to consider when sampling for aerosols. An article written by Brisson & Archuleta (2009) answers these questions and provides direction on how to further address these questions. While aerosol sampling procedures remain similar, techniques and equipment have evolved over time. It wasn't until the use of the closed-face cassette, which was developed over a half century ago, that the concept for wall loss became introduced. When compared to the open-face cassette, the design of the CFC made the filter less likely to be damaged during sampling, and this feature contributed to their popularity. At the time of its introduction, the CFC was not compared to any performance standard since one did not exist. However, newer data suggests that the sampling efficiency of the CFC is much lower for sampling particles greater than 10µm AED (Brisson & Archuleta, 2009). Since the CFC sampling efficiency decreases as particle size increases, it can be inferred that particles on the cassette walls are within the range of 10 to 100µm AED (Harper & Demange, 2007). It wasn't until the 1990s that an inhalation convention become agreed upon. The convention was based on human inhalation efficiency measurements and raised the inhalability limit to particles up to 100µm AED. The CFC and other inhalable samplers have been evaluated against the inhalable convention. The IOM sampler has been tested against, and found to match, this convention most closely. The IOM method also recognized internal wall deposits and recommended that they be accounted for in the analysis. When compared to newer size-selective sampling devices, the sampling efficiency of the CFC gained attention and, with it, concerns that particulates not

captured by the filter may not be representative of workplace exposure (Brisson & Archuleta, 2009).

In the development of the IOM sampling device for the inhalable fraction of PM, the U.K. Institute of Occupational Medicine recognized the significance for including wall deposits into the analysis, as earlier studies found 25-44% of total sampled material adhered to the IOM sampler walls (Harper & Demange, 2007). The deposits adhering to the walls were also size-selective having a greater percentage of wall loss as the particle size increased (Harper & Demange, 2007). The authors concluded that the amount of total sample found on the walls of the IOM is likely to be significant in all industries (Harper & Demange, 2007). The dominant mechanisms contributing to the loss of particles were electrostatic attraction for smaller particles and gravitational settling or inertial impaction for larger particles (Harper & Demange, 2007).

#### 2.5 Recovery Strategies for Wall Loss

It is becoming commonly accepted that all particles entering the sampler be considered part of the sample and analyzed accordingly (Hendricks, Stone, & Lilliquist, 2009). This includes not only particles that are deposited on the filter, but particles that may adhere to the cassette walls. Various methods have been developed to account for and recover such losses. As noted in the previous section, one strategy developed to minimize wall loss is the use of conductive cassettes. Additional control methods include 1) thoroughly washing the internal cassette surface, 2) performing the sample extraction directly in the cassette, 3) wiping the internal cassette surface, and 4) use of internal capsules or cartridges (Ashley & Harper, 2013).

Wall- loss recovery strategies of rising with deionized water and wiping with a deionized moistened wipe were evaluated for routine metal analysis in closed-faced 37 mm samples collected by OSHA compliance officers (Hendricks et al.). Prior to wiping, the interior walls of

the cassette were rinsed using 10% nitric acid. The samples from the first study were analyzed for 13 different elements by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). Out of 185 samples, 126 samples showed metal analyte on both the filter and cassette walls (Hendricks et al, 2009). The second study analyzed the same samples but for lead, cadmium, and antimony. The analysis was performed using an Atomic Absorption Spectroscopy. The analysis gave a total of 65 results. Out of that total, 31 samples showed metal analyte on both the filter and sampler walls using a single wipe and 9 samples showed metal analyte on the second wipe (Hendricks et al, 2009). OSHA suggests all material entering the cassette should be considered part of the sample for metals. The study concluded that significant portions will be deposited on the inner cassette walls, and recovery is fairly easily resolved through appropriate wall loss control strategies.

Ceballos et al (2015) conducted a lead monitoring study and compared two different approaches for recovering wall loss on closed-face 37 mm polystyrene cassettes. Air samples from three different locations within a lead smelting and processing facility were collected following identical sampling methodologies. After sampling, the cassettes were assigned to one of two groups: no rinse and rinse. The cassettes assigned to the "no rinse" group were each wiped with two filters moistened with deionized water, consecutively. The cassettes assigned to the "rinse" group were first rinsed with deionized water and then wiped with two filters moistened with deionized water, consecutively. All filters and wipes were analyzed separately by ICP-AES. The primary variable during analysis was the total mass of lead recovered. The study compared the means and medians of total mass between the two groups using t-tests and Wilcoxon Scores tests. The study also compared the percent of lead recovered on the first and second wipes. Overall, this study revealed on average that 29% of the total lead recovered was from the walls of the non-conductive 37 mm cassette (Ceballos et al, 2015). The results comparing the means of total lead mass collected by no rinse and rinse groups found no statistically significant difference between the two groups (Ceballos et al, 2015). However, the difference in total lead recovered between the first wipe and second wipe was significant (Ceballos et al, 2015). The first wipe recovered an average of 21% of total lead while the second wipe recovered an average of 5.3% total lead (Ceballos et al, 2015). The average total lead recovered between the filter and first wipe totaled 95% (Ceballos et al, 2015). The study concluded that using the control strategy of a single wipe adequately recovered lead wall losses (Ceballos et al, 2015).

An inter-laboratory study (Harper & Ashley, 2012) sampling for lead used digestible capsules placed inside closed-face 37 mm plastic cassettes. Each cassette consisted of a cellulose acetate internal capsule and 0.8-micron pore size MCE filter. Each sample was spiked with a specific, known concentration of lead and submitted to various volunteer laboratories for analysis. The laboratories prepared the samples by acid digestion and analyzed using atomic spectrometry procedures. The results from laboratory analysis and known lead concentrations were compared to each other. The comparison showed quantitative lead recovery within 100%  $\pm$  10% for majority of samples using capsules (Harper & Ashley, 2012). The study concluded the use of cellulosic internal capsules attached to MCE are precise, offering solution for recovering wall deposits (Harper & Ashley, 2012).

The National Institute of Occupational Safety and Health has recently added new gravimetric methods, 0501 and 5100, as alternative techniques for ensuring non-filter deposits of airborne particles are included in the gravimetric analysis (NIOSH, 2016). Analytical method 0501 is specific to sampling for total dust, also called nuisance dust or particles not otherwise

regulated. Both methods employ the use of internal capsules, which meld the filter to the cassette housing, eliminating the potential for particulate matter adhering to the inner cassette walls. The entire capsule, including wall deposits, is weighed during sample preparation. Internal capsules are also used for the IOM inhalable sampler in the mining industry, when sampling for coal dust, and as previously mentioned, with the 37 mm cassette. The accuracy of the sample can be improved using internal cartridges (Baron, 2003).

OSHA has also made changes to its analytical methods to reduce wall losses. OSHA analytical methods for metals specifically require interior cassette wiping (Hendricks, 2009).

### 3. Objective

While various methods have been demonstrated for minimizing particulate matter wall loss, to date there is limited information available to quantify the potential wall loss associated with mineral dust sampling. The research objective of this study was to characterize wall loss associated with conductive vs. non-conductive sampling cassettes used for total and respirable mineral dust field sampling in a surface metal/nonmetal mine.

The research objective was addressed through the following research hypotheses.

Null Hypothesis 1: The mean percent wall loss from conductive cassettes applied in respirable dust sampling will be greater than or equal to the mean percent wall loss from non-conductive cassettes.

Research Hypothesis 1: The mean percent wall loss from conductive cassettes applied in respirable dust sampling will be lower than the mean percent wall loss from non-conductive cassettes.

Null Hypothesis 2: The mean percent wall loss from conductive cassettes applied in total dust sampling will be greater than or equal to the mean percent wall loss from non-conductive cassettes.

Research Hypothesis 2: The mean percent wall loss from conductive cassettes applied in total dust sampling will be lower than the mean percent wall loss quantified from non-conductive cassettes.

Null Hypothesis 3a: There will not be a correlation between wall loss mass and filter mass in conductive cassettes applied in respirable dust sampling.

Research Hypothesis 3a: There will be a correlation between wall loss mass and filter mass in conductive cassettes applied in respirable dust sampling.

Null Hypothesis 3b: There will not be a correlation between wall loss mass and filter mass in non-conductive cassettes applied in respirable dust sampling.

Research Hypothesis 3b: There will be a correlation between wall loss mass and filter mass in non-conductive cassettes applied in respirable dust sampling.

Null Hypothesis 3c: There will not be a correlation between wall loss mass and filter mass in conductive cassettes applied in total dust sampling.

Research Hypothesis 3c: There will be a correlation between wall loss mass and filter mass in conductive cassettes applied in total dust sampling.

Null Hypothesis 3d: There will not be a correlation between wall loss mass and filter mass in non-conductive cassettes applied in total dust sampling.

Research Hypothesis 3d: There will be a correlation between wall loss mass and filter mass in non-conductive cassettes applied in total dust sampling.

#### 4. Methods

Sixty-four area air samples were collected in a primary rock crusher plant at a surface copper mine in the northwestern United States. A location having relatively high PM concentrations was chosen with assistance from mine management. Total and respirable dust samples were collected per NIOSH manual of analytical methods 500 and 600, respectively (NIOSH, 2016). The sampling duration was approximately 420 minutes for all samples. Sampling trains were equipped with either a conductive (static dissipative polypropylene) Zeflon 37MM-3-CF or non-conductive (polystyrene) Zeflon 37MMH-2 cassette. Sampling was conducted during four separate days when the rock crusher was in operation as illustrated in Table 2. During sampling days 1 and 2, 32 respirable dust samples were collected. Of the 32 respirable dust samples, 16 samples utilized 3-piece, conductive cassettes, the other 16 samples utilized 3-piece, non-conductive cassettes. During days 3 and 4, 32 total dust samples were collected. Of those 32 total dust samples, 16 samples utilized 2-piece, conductive cassettes, the other 16 samples utilized 2-piece, non-conductive cassettes. In addition to the 64 field samples, six blanks (roughly 10% of the total samples) were collected. A sample size of 16 was necessary to detect significant paired differences between the cassette types, based on a power analysis of data from Demange et al., (2002). The total number of samples needed was 64 with 16 samples per cassette type for both dust size fractions. The relative humidity and room temperature were measured and recorded throughout the sampling period with a Kestrel pocket weather tracker model 4000 and sample volumes were adjusted accordingly from calibration conditions.

Sampling		Number of	Number of Non-
Method	Days	Conductive	Conductive
Wiethod		Samples	Samples
Respirable	1	Q	Q
Dust	1	0	0
Respirable	n	0	0
Dust	2	0	0
Total Dust	3	8	8
Total Dust	4	8	8

Table II: Summary of the sampling schedule

For the respirable dust sampling days, sampling trains were separately paired: one having a conductive, open face, cassette with an SKC aluminum cyclone attachment and the other having a non-conductive, open face, cassette with an SKC aluminum cyclone attachment. The paired cassettes hung side-by-side in a vertical position along the back wall of the rock crusher roughly 4 feet above floor level. A 5.0 µm pore size PVC filter was positioned in each 37 mm cassette. With the use of Tygon <sup>TM</sup> tubing, the sampling media was placed in line with SKC Aircheck air sampling pumps operating at a nominal airflow rate of 2.5 liters per minute (l/m). All Tygon <sup>TM</sup> tubing were of similar length. Pumps were calibrated before and after sampling with a Bios Defender 510 primary flow meter. An illustration of respirable dust sampling is shown below in Figure 1.



Figure 1. Illustration of paired sampling methodology employed for each sampling trial

For the total dust sample days, sampling trains were paired: one having a conductive, closed-face, cassette and the other having a non-conductive, closed-face, cassette. The paired cassettes hung side-by-side with the air inlet facing approximately 45° downwards along the back wall of the rock crusher roughly 4 feet above floor level. A 5.0 µm pore size PVC filter was positioned in each 37 mm cassette. With the use of Tygon tubing, the sampling media was placed in line with SKC Aircheck air sampling pumps operating at a nominal airflow rate of 2 l/m. All Tygon tubing were of similar length. Pumps were calibrated before and after sampling with a Bios Defender primary flow meter.

All filters were desiccated pre and post sampling with a NIKKO, AD-101F desiccator equipped with fresh DRIERITE (blue) desiccant for a minimum duration of 24 hours. The filters were weighed pre and post sampling with a self-calibrating Microbalance MYA 5.3 Y electronic scale.

After sampling, each cassette inlet was capped, placed upright in a storage container, and transported by hand to the lab for analysis. Latex gloves, tweezers, and ACL 520 staticide spray were used when handling cassettes and filters. The outside of each cassette was cleaned with a moist paper wipe, cassette plugs were removed, and the cassette gently disassembled. The 37 mm filter was removed from each cassette and placed on a lab dish which was then placed inside the desiccator cabinet.

The 3-piece cassette has an inner extended ring used for open face sampling which helps evenly distribute particles. The 3rd inlet piece is removed and replaced with a cyclone when sampling for respirable dust. The 2-piece cassette consists of two pressed fitted pieces and has a smaller sized air inlet compared to the open face 3-piece cassette. A single 25 mm PVC filter was used to wipe the inside walls of each cassette as described by Hendricks et al (2009).

Since the 3-piece cassettes used for respirable sampling are positioned with the cassette base and inner portion (no cassette inlet cap) to allow even distribution of the aerosol on the filter, only the interior of the inner cassette piece was wiped and analyzed. For consistency, only the cassette walls and not the air inlet (cowl) to the two-piece cassettes used to sample total dust were wiped. The inner cassette wall surface area of the closed-face cassette is 703.5mm<sup>2</sup>. The inner cassette wall surface area of the open face cassette is 1005mm<sup>2</sup>. The wiping motion was circular, beginning and ending in the same location of the inner cassette wall.

Initial assessment of wipe recovery was performed to determine the mass recovery on multiple wipe samples. The walls of non-conductive cassettes used to sample for total dust were wiped using two consecutive filters. The filters used for wiping were desiccated, placed through a static neutralizing strip, pre/post weighed prior and after wiping. An amount of mass was recovered from the first wipe and little to no mass was recovered from the second wipe.

Considering the sensitivity of the scale, one wipe per cassette was used to recover mineral wall losses. This single wipe approach is consistent to the Ceballos et al (2015) study, which revealed that the second and third wipe showed minimal recovery compared to that of the first wipe. However, it should be noted that a wet method was used verses a dry wipe method in this study. It is anticipated that wetting the wipes with de-ionized water would increase wall loss recovery, but this technique is not conducive to a gravimetric analytical technique.

Two tweezers were used to handle the filter during wiping: the first tweezer retained the filter when wiping. The second tweezer was used to transfer the filter from the first tweezer onto the scale. At no point did the filter used for wiping contact anything other than the tweezers, inner cassette walls, and scale. The cassette filter was weighed pre-and post-sampling yielding a "filter mass" and the filter used to wipe the inside of the cassette was weighed pre and post wiping yielding a "wall loss mass". The particulate mass of the cassette filter and wall wipe were added to yield a "total mass". The percent of wall loss recovered was calculated by dividing the "wall loss mass" by the "total mass" which was then multiplied by 100.

#### 4.1 Statistical Analysis

To assess the research hypothesis, descriptive statistics were first used to summarize mean filter and wall loss masses and standard deviation for each cassette and sampling type. Due to the data not being normally distributed, a 1-Sample Wilcoxon Signed Rank test was used to compare the mean percent wall loss difference between each paired conductive and nonconductive cassettes for both respirable and total dust data. The null hypothesis for each method is that conductive cassette mean percent wall losses would be greater than or equal to nonconductive cassette mean percent wall losses. A Spearman correlation was used to determine if a significant association existed between wall loss mass vs. filter mass for conductive and nonconductive cassettes when sampling both respirable and total dust. The null hypothesis is that there was no correlation between wall loss mass vs filter mass. The level of statistical significance for all analyses was set at  $\alpha = 0.05$ . Minitab Statistical Software version 19 (State College, PA) was used for the data analysis.

#### 5. Results

Average particulate mass and gravimetric mass concentration values were categorized based on cassette type and sampling method. These mean mass and mass concentration data for respirable and total dust are summarized in Table 3 with raw data provided in Appendix A. As noted in the table, the average respirable conductive filter mass was 0.957 mg, and the average wall loss mass was 0.042 mg with a combined average mass of 0.999 mg. The average respirable non-conductive filter mass was 0.551 mg, and the average wall mass was 0.138 mg with a combined average total conductive filter mass was 1.716 mg, average wall mass was 0.082 mg with a combined average mass of 1.798 mg. The average total non-conductive filter mass was 1.637 mg, average wall mass was 0.083 mg with a combined average mass of 1.719 mg. There is a larger measured difference between wall loss mass and filter mass for respirable dust sampling using a conductive cassette. The measured difference between wall loss mass and filter mass for respirable dust sampling using a conductive cassette. The measured difference between wall loss mass and filter mass for respirable dust sampling using a conductive cassette. The measured difference between wall loss mass and filter mass for respirable dust sampling using a conductive cassette.

Since respirable and total dust measurements are most often expressed as mass concentration, data are also provided in Table 3 as gravimetric mass concentration. The mass concentration results reflect observations made with the mass data, but accounts for differences in flow rates between total and respirable and dust sampling. The air pumps were calibrated in the lab before being used in the field. Originally, the temperature and pressure were not considered adjusting for pump volume because particulate mass and not concentration were the focus of the research. The sampling took place during the winter months and most likely having lower humidity levels which could result in an increase of static charge for both cassette and particulate during sampling.

		Mass Par	ticulate from F	liter (mg) by c	assette ty	ре				
Tumo		Respira	ble Dust	Total Dust						
Type	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	0.96	0.53	2.86	0.37	1.72	0.53	2.79	1.12		
Non-conductive	0.55	0.45	2.02	0.00	1.64	0.89	4.62	0.63		
Differences	0.41	0.08	0.84	0.37	0.08	0.37	1.83	0.49		
Wall loss mass (mg) by cassette type										
Tuno		Respira	Total Dust							
Туре	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	0.04	0.03	0.11	< 0.01	0.08	0.10	0.40	0.02		
Non-conductive	0.14	0.13	0.40	0.04	0.08	0.08	0.32	0.02		
Differences	0.10	0.10 0.10 0.30 0.04 0.00 0.02 0.08		< 0.01						
		Р	ercent wall los	s by cassette t	ype					
Tuno	Respirable Dust					]	Fotal Dust			
Туре	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	4.93%	0.03	0.13	< 0.01	4.55%	0.05	0.23	0.01		
Non-conductive	25.24%	0.24	0.98	0.07	5.83%	0.06	0.20	0.01		
Differences	20.31%	0.21	0.85	0.07	0.01	0.00	0.03	0.00		
		Filt	er Mass Conc	entrations (mg	/m^3)					
Tumo	Respirable Dust					]	Fotal Dust			
Туре	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	0.94	0.56	2.94	0.4	2.07	0.67	3.47	1.36		
Non-conductive	0.55	0.46	2.06	< 0.01	1.97	1.06	5.50	0.79		
Differences	0.39	0.09	0.88	0.40	0.10	0.39	2.04	0.57		
		Wall	Loss Mass Co	ncentrations (r	ng/m^3)					
Tumo		Respira	ble Dust		Total Dust					
Type	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	0.04	0.03	0.10	< 0.01	0.10	0.12	0.47	1.36		
Non-conductive	0.14	0.12	0.39	0.04	0.10	0.09	0.38	0.79		
Differences	0.09	0.10	0.29	0.04	0.00	0.02	0.09	0.57		
		Gravimetric	Mass Concent	tration Percent	tage Wall	Loss				
Tumo		Respira	ble Dust			]	Fotal Dust			
Туре	Mean	SD	Range (max)	Range (min)	Mean	SD	Range (max)	Range (min)		
Conductive	4.93%	0.03	0.13	< 0.01	4.55%	0.05	0.23	0.01		
Non-conductive	25.24%	0.24	0.98	0.07	5.83%	0.06	0.20	0.01		
Differences	20.31%	0.21	0.85	0.07	1.28%	0.01	0.03	0.00		

Table III. Summary Results of Farticulate Mass Confected per Casselle Type/Meth	Table	<b>III: Summary</b>	<b>Results of Parti</b>	iculate Mass Col	lected per Cassett	e Type/Metho
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The gravimetric mass concentration percentage of wall loss was 4.9% and 25.2% for respirable conductive and non-conductive cassettes, respectively. The gravimetric mass concentration percentage of wall loss was 4.5% and 5.8% for total conductive and non-conductive cassettes, respectively. However, underestimation of concentrations may not be the case when sampling for total dust using either cassette type. The table consists of mean filter mass, mean wall loss mass, and mean percent wall loss mass for each method and cassette type.

The greatest difference in means between cassette types was measured when sampling for respirable dust. The mean differences between cassette types when sampling for total dust was minimal compared to respirable dust sampling. The non-conductive cassette mean percent wall loss has the greatest difference in range and highest standard deviation compared to the remaining data.

Percent wall loss values were categorized based on cassette type and sampling method. The mean percent wall loss for non-conductive cassettes when sampling for respirable dust was 25.24%. The mean percent wall loss for conductive cassettes when sampling for respirable dust was 4.93%, which was significantly lower than the non-conductive cassette (P<0.001).

The mean percent wall loss for non-conductive cassettes when sampling for total dust was 5.83%. The mean percent wall loss for conductive cassettes when sampling for total dust was 4.55%, which was not significantly lower than the non-conductive cassettes (P=0.183).

Box plot's representing the mean wall loss % differences between conductive and nonconductive cassettes for respirable dust and total dust sampling are shown below in Figures 2 & 3, respectively.



Figure 2. A comparison of mean wall loss percentage differences between conductive vs. non-conductive cassettes when sampling for respirable dust



Figure 3. A comparison of mean wall loss percentage differences between conductive and non-conductive cassettes when sampling for total dust

A Pairwise Spearman test was used to determine if a there was a correlation between the wall loss mass and filter mass. The particulate mass from the filter and the mass particulate from the cassette walls of conductive and non-conductive cassettes for both respirable and total dust sampling were plotted on an X, Y graph as illustrated in Figures 4 and 5, respectively.



Figure 4: Respirable wall loss mass vs. filter mass



Figure 5: Total wall loss mass vs. filter mass

While a slightly significant correlation was observed (r = -0.502, P = 0.048) between wall loss mass vs. corresponding filter mass for respirable dust sampling with conductive cassettes. Correlations were not observed between wall loss mass and corresponding filter mass data for non-conductive cassettes when respirable dust sampling(r = 0.197, P = 0.464), between wall loss mass vs. corresponding filter mass for conductive cassettes when total dust sampling (r = 0.057, P = 0.833), and between wall loss mass vs. corresponding filter mass for nonconductive cassettes when total dust sampling (r = -0.403, P = 0.121), respectively. Although no correlation was found statistically, the graph visually shows less wall loss mass with the conductive cassette's vs non-conductive cassettes when sampling for respirable dust.

#### 6. Discussion

The objective of this study was to characterize the wall loss associated with nonconductive and conductive cassettes while sampling for mineral dust utilizing respirable and total dust methodologies. The first aim of this research was to assess if mean percent wall loss from conductive cassettes applied in respirable dust sampling would be greater to or equal to the mean percent wall loss from non-conductive cassettes. The results indicated that the mean percent wall loss from conductive cassettes was significantly lower (p<0.001) than the mean percent wall loss from non-conductive cassettes when sampling for respirable dust, therefore, we will reject the null hypothesis.

The second aim was to assess if the mean percent wall loss from conductive cassettes applied in total dust sampling would be greater to or equal the mean percent wall loss from the non-conductive cassettes. The results indicated that the mean percent wall loss from conductive cassettes was not significantly lower (p=0.183) than the mean percent wall loss from non-conductive cassettes when sampling for total dust. Therefore, we fail to reject null hypothesis 2.

The third aim was to assess if there was a correlation between wall loss mass vs. filter mass in non-conductive and conductive cassettes applied in respirable and total dust sampling. If a significant negative correlation were found between wall loss and the corresponding filter mass, we postulated that this would indicate that as more PM adheres to the filter during sampling, less PM would adhere to the cassette inner walls. Of these objectives, one slightly significant correlation (p=0.048) existed between wall loss mass and filter mass for conductive cassettes when sampling for respirable dust. Therefore, we reject null hypothesis 3a.

Of the remaining hypothesis, 3b through 3d, the results indicated no correlation (p=0.464) between wall loss mass and filter mass for non-conductive cassettes when sampling

for respirable dust, no correlation (p=0.833) between wall loss mass and filter mass for nonconductive cassettes when sampling for total dust, and no correlation (p=0.121) between wall loss mass and filter mass for non-conductive cassettes when sampling for total dust. Therefore we reject the null hypothesis for 3b, 3c, and 3d. However, a weak negative correlation existed between wall loss mass and filter mass for non-conductive cassettes when sampling for total dust. A weak positive correlation existed between wall loss mass and filter mass for conductive cassettes when sampling for total dust.

The mean percent wall loss for respirable sampling using conductive cassettes was approximately 4% and approximately 25% when using non-conductive cassettes. The mean percent wall loss recovered from the non-conductive cassettes was substantially larger than the mean percent wall loss recovered from conductive cassettes. These results were similar to Charm et al., (2014) results which compared cassette wall losses between cassette types when sampling for respirable coal dust. Charm et al., (2014) study found a mean percent wall loss of 17.9% for non- conductive cassettes and a mean wall loss of 3.73% for conductive cassettes when using a SKC aluminum cyclone and/or Dorr-Oliver Nylon cyclone, revealing a significant (p<0.05) difference in wall loss between both cassette types. In some cases, we recovered wall losses over 50% for non-conductive cassettes when sampling for respirable dust.

Reports, as early as the 1990's, were based on a respirable silica study which demonstrated that a large proportion of particles may adhere to the inside surfaces of both twopiece and three-piece cassettes when using a Dorr-Oliver Nylon Cyclone (Reichmann, 2005).

Majority of studies measuring particulate wall loss are done on metal sampling methods. Studies measuring particulate dust using closed face 37 mm cassettes are limited, and because of, studies measuring wall loss quantities for metal analyte using 37 mm cassettes were used to compare our wall loss results for total mineral dust sampling. The mean percent wall loss for total dust sampling using conductive cassettes in this study was approximately 4% and a mean percent wall loss of 5% for non-conductive cassettes. These results were not similar to Ceballos et al (2015) results which reported and average wall loss of 29% from non-conductive cassettes when sampling for lead dust using NIOSH method 7303. It's important to note that the analytical method for lead is different from the analytical method used for this study, and the size and composition of lead particles varies from the size and composition of mineral dust.

Even though mass concentrations were well below the OSHA PEL when including wall deposits, there is a risk of underreporting when analyzing only the filter of the 3-piece nonconductive cassette when sampling for respirable mineral dust. The risk of not including a portion of the sample when using non-conductive cassettes for respirable sized particles is more likely than sampling with conductive cassettes. Health effects can be misrepresented, especially if the contaminant toxicity is high such as silica or asbestos where exposure to the smallest amounts of silica or asbestos can have significant impact on human health.

The median wall loss results from the Ashley & Harper (2013) study comparing filter and wall deposits from paired IOM and CFC cassettes when collecting airborne lead dust recovered a median wall loss ranging from 0 to 30%. The higher percent range of wall loss is not consistent with our total dust sampling mean wall loss percent recovery of 4-5%. It's important to note the study suggests that mechanisms influencing wall deposition for particles smaller than 20 µm AED are not size-selective, and it postulates a combination of electrostatic charge, gravitational settling, diffusion, turbulence and inertial impaction were among dominant mechanisms contributing most to wall loss effects. Total dust sampling collects particle sizes ranging from respirable sized fractions to fractions larger than 20 µm and may have different dominant

mechanisms than those mechanisms dominant to respirable dust sampling which collects 4  $\mu$ m sized particles with 50% efficiency. This may be one contributing factor as to why the results between total dust sampling and respirable dust sampling were not similar.

Humidity affects the electrostatic charge found on both the cassette and particle. A lower humidity will increase static charge. Sampling was done during the winter months which typically have lower humidity levels compared to warmer months of the year. Since cassettes where paired, humidity levels were not considered.

A study by Hendricks et al (2009) measured filter and wall mass for metal analyte using 37 mm cassettes. They found metal particulate on most the sample's inner cassette surfaces. The study did not give specific wall loss mass percentages, but did find metal particulate wall losses for a majority of their samples, which is consistent with our results of finding mineral particulate on the walls of respirable dust samples.

Recovering wall loss by wiping inner cassette surfaces is tedious, time consuming, and may introduce unwanted errors by not recovering particulate adhering to the cassette's corners or crevices. Ashley & Harper (2013) suggest using an internal digestible capsule during sampling. The capsule is fused to the filter eliminating the need for separate rinsing and/or wiping. After sampling, the capsule can be easily removed and analyzed along with the filter.

#### 6.1 Study Limitations

Transporting the samples from the field site to the laboratory could have caused particulate to transfer from the filter onto the walls as noted in Ashley and Harper (2013) study. Careful consideration and handling were taken to prevent contamination of the cassette walls from particulate collected by the filter. Each cassette was placed upright into a secured container with the filter facing up and was carried by hand from the site to the laboratory. Prior to opening cassettes in preparation for analysis, the caps were removed to eliminate any suction effect when dissembling the cassettes. This was done to prevent particulate from being sucked off the filter and potentially adhering to the cassette walls.

For the total dust samples, by wiping the inner cassette walls only, material may have been missed and may have underestimated the total wall loss. As noted in Puskar et all, 2010, up to 62% of dust may be on the inside surface of the cassette tops.

A combination of stability, leveling, vibration, filter placement within the balance, thermal drafts, temperature, humidity, and electrostatic charge can affect a balance and should be considered when gravimetrically weighing a filter (Baron, 2009). In addition to the scale, static from the filter, tweezers, and gloves could have affected results during handling and when weighing filters. Static effects were greatly reduced by the use of static neutralizing strips and staticide spray.

### 7. Conclusion

This study evaluated the impact of conductive vs. non-conductive sampling cassettes for respirable as well as total mineral dust. Previous studies have revealed that the application of conductive cassette materials may reduce particle loss on cassette walls. No significant differences were observed in wall loss concentrations between conductive vs. non-conductive cassettes when sampling for total dust. However, a significant difference in wall loss between conductive vs. non-conductive cassettes when sampling for total dust. However, a significant difference in wall loss between conductive vs. non-conductive cassettes when sampling for respirable mineral dust was observed. Conductive cassettes produced less wall loss than the non-conductive cassette when sampling for respirable mineral dust under similar sampling conditions. These results suggest that future respirable dust sampling for mineral dusts should consider the application of conductive cassettes to reduce wall loss. Substantial wall loss, such as that which was observed in this study, may result in underestimation of actual worker' exposure which may increase the risks of disease.

In terms of total dust sampling, the results of this study suggest that wall loss may not be a significant factor, regardless of which cassette type is used. However, further research is warranted, since these results are inconsistent with previous studies. A potential limitation that may have influenced the total dust sampling reported in wall losses is that the inner walls were wiped vs. the inner walls along with the inside surface of the cassette tops.

#### 8. References

- American Conference of Governmental Industrial Hygienists (2019). *Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices*. Cincinnati, OH: Signature Publications.
- Anna, D. H. (2011). The Occupational Environment: Its Evaluation, Control, and Management. *American Industrial Hygiene Association*.
- Ashley, K., & Harper, M. (2013). Analytical Performance Issues. *Journal of Occupational and Environmental Hygiene*, 30.
- Ashley, K., & Harper, M. (2013). Closed-Face Filter Cassette (CFC) Sampling—Guidance on Procedures for Inclusion of Material Adhering to Internal Sampler Surfaces. *Journal of Occupational and Environmental Hygiene*. Retrieved from Journal of Occupational and Environmental Hygiene.
- Baron, P. A. (2003). Factors affecting aerosol sampling. NIOSH. Manual of Analytical Methods, fourth ed., third supplement, 184, 207.
- Baron, P., & Deye, G. (1990). Electrostatic Effects in Asbestos Sampling: Experimental Measurements. American Industrial Hygiene Association, 51.
- Brisson, M., & Archuleta, M. (2009). The real issue with wall deposits in closed filter cassetteswhats the sample? *Occupational & Environmental Hygiene*.
- Ceballos, D., King, B., Beaucham, Brueck, & Scott (2015). 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. *Journal of Occupational and Environmental Hygiene*.

- Charm, J., Lee, T., Kashon, M., Kusti, & Harper, M. (2014). Quartz coal dust deposited on Internal Surface of Respirable Size Selective Samplers. *Journal of Occupational and Environment Hygiene*, 1.
- Demange, Gendre, Herve-Bazin, Carton, & Peltier. (1990). Aersoal evaluation difficulties due to particle deposition on filter hodler inner walls . *British Occupational Hygiene Society*, 399-403.
- Demange, M., Gorner, P., Elcabache, J., & Wrobel, R. (2002). Field comparison of 37 mm CFC& IOM Sampler. Applied Occupational & Environmental Hygiene.
- Harper, M., & Ashley, K. (2012). Analytical Performance issues Preliminary Studies on the Use of Acid-Soluble Cellulose Acetate Internal Capsules for Workplace Metals Sampling and Analysis . *Journal of Occupational and Environmental Hygiene*, 128.
- Harper, M., & Demange, M. (2007). Analytical Performance Criteria. *Journal of Occupational* and Environmental Hygiene, 1.
- Hart, J. F., Autenrieth, D. A., Cauda, E., Chubb, L., Spear, T. M., Wock, S., & Rosenthal, S. (2018). A comparison of respirable crystalline silica concentration measurements using a direct-on-filter Fourier transform infrared (FT-IR) transmission method vs. a traditional laboratory X-ray diffraction method. *Journal of occupational and environmental hygiene*, *15*(10), 743-754.
- Hendricks, W., Stones, F., & Lillquist, D. (2009). On wiping the interior walls of 37 mm Closed face Cassettes: An OSHA Perspective. *Journal of Occupational and Environmental Hygiene*.
- Hinds, W. (1999). Aerosol Technology: properties, behavior, and measurement of airborne particles, Second edition. New York: Wiley-Interscience.

- Klaassen, C., Casarett, L., & Doull, J. (2018). *Casarett & Doulls Toxicology The Basic Science* of Poisions Eighthth Edition . New York: McGraw-Hill Education .
- Lee, T., Chisholm, W. P., Slaven, J. E., & Harper, M. (2009). Size distributions of 0.5 to 20 μm aerodynamic diameter lead-containing particles from aerosol sampler walls and filters. *Aerosol Science and Technology*, *43*(10), 1042-1050.

Mineral Dust-Gravimetric Method. (2006, October). Retrieved from msha: https://arlweb.msha.gov/Readroom/HANDBOOK/MNMInspChapters/Chapter5.pdf

National Institute for Occupational Safety and Health (NIOSH) (2016): NIOSH Manual of analytical methods 5th (NMAM) Fifth Edition. Cincinnati, OH: DHHS (NIOSH)
Publication. Retrieved from

https://www.cdc.gov/niosh/nmam/pdf/NMAM 5thEd EBook-508-final.pdf

- O'Connor, S., O'Connor, P., Feng, A., & Ashley, K. (2014). Gravimetric Analysis of Particulate Matter using Air Samplers Housing Internal Filtration Capsules. *National Center for Biotechnology Information*.
- Puskar, M., Harkins, J., Moomey, J., & Hecker, L. (2010). Internal Wall Losses of Pharmaceutical Ducts During Closed-Face 37mm Polystyrene Cassette Sampling . *American Industrial Hygiene Association Journal*.
- Reichmann, D. (2005). Evaluation of quartz residue on cassette interior of AIHA proficiency samplers. *American Society of Testing and Materials*.

Witschger, O., Grinshpun, S. A., Fauvel, S., & Basso, G. (2004, June 1). *The Annals of Occupational Hygiene*. Retrieved from Performance of Personal Inhalable Aerosol Samplers in Very Slowly Moving Air when Facing teh Aerosol Soource : https://academic.oup.com/annweh/article/48/4/351/145103

# Appendix A

Cassette type wall loss and filter concentrations for respirable and total dust sampling are shown below:

Table IV: Wall Loss, Fliter, and Combined Mass Concentration											
Concentrations (mg/m^3)											
	Respirable Dust Total Dust										
	Conductive	2	No	on-Conduc	tive	C	onductive		No	n-Conducti	ve
Wall Loss	Filter	Total	Wall Loss	Filter	Total	Wall Loss	Filter	Total	Wall Loss	Filter	Total
0.037	0.792	0.829	0.389	0.386	0.775	0.257	3.470	0.037	0.072	0.787	0.829
0.103	0.707	0.809	0.062	0.423	0.485	0.042	2.477	0.103	0.021	2.631	0.809
0.040	0.354	0.394	0.349	0.651	1.000	0.030	1.727	0.040	0.063	2.213	0.394
0.024	0.697	0.720	0.102	0.553	0.655	0.045	1.361	0.024	0.042	1.873	0.720
0.061	0.734	0.794	0.094	0.292	0.386	0.053	1.937	0.061	0.083	1.774	0.794
0.015	0.690	0.704	0.063	0.225	0.288	0.060	2.745	0.015	0.061	2.393	0.704
0.000	0.814	0.814	0.086	0.002	0.088	0.080	2.617	0.000	0.146	1.737	0.814
0.029	0.747	0.776	0.072	0.653	0.725	0.056	3.410	0.029	0.064	1.673	0.776
0.015	2.943	2.958	0.125	0.510	0.635	0.018	1.877	0.015	0.379	1.647	2.958
0.059	1.008	1.067	0.039	0.474	0.513	0.050	1.476	0.059	0.134	1.380	1.067
0.039	0.874	0.913	0.061	0.290	0.351	0.037	1.444	0.039	0.043	1.510	0.913
0.082	0.921	1.003	0.385	0.300	0.685	0.044	1.638	0.082	0.030	1.470	1.003
0.024	0.951	0.976	0.048	0.660	0.708	0.082	1.919	0.024	0.093	1.535	0.976
0.050	0.920	0.971	0.054	0.325	0.379	0.473	1.583	0.050	0.245	0.982	0.971
0.055	0.887	0.942	0.150	2.062	2.212	0.192	1.778	0.055	0.088	5.505	0.942
0.027	0.992	1.019	0.084	0.986	1.069	0.065	1.696	0.027	0.030	2.383	1.019

Table IV: Wall Loss, Filter, and Combined Mass Concentration

Cassette type wall loss and filter mass for respirable and total dust sampling are shown below:

Mass (mg)											
Respirable Dust					Total Dust						
	Conductive		No	on-Conduc	tive	C	onductive		No	n-Conducti	ve
Wall Loss	Filter	Total	Wall Loss	Filter	Total	Wall Loss	Filter	Total	Wall Loss	Filter	Total
0.039	0.825	0.864	0.402	0.399	0.801	0.202	2.725	2.927	0.057	0.625	0.682
0.106	0.730	0.836	0.065	0.441	0.506	0.035	2.055	2.090	0.017	2.155	2.172
0.042	0.367	0.409	0.362	0.675	1.037	0.025	1.429	1.454	0.052	1.817	1.869
0.025	0.730	0.755	0.106	0.575	0.681	0.037	1.115	1.152	0.035	1.550	1.585
0.063	0.761	0.824	0.097	0.301	0.398	0.044	1.607	1.651	0.068	1.449	1.517
0.015	0.711	0.726	0.065	0.233	0.298	0.049	2.260	2.309	0.050	1.960	2.010
0.000	0.836	0.836	0.089	0.002	0.091	0.067	2.182	2.249	0.120	1.430	1.550
0.030	0.775	0.805	0.074	0.675	0.749	0.046	2.793	2.839	0.053	1.391	1.444
0.015	2.855	2.870	0.122	0.499	0.621	0.015	1.573	1.588	0.319	1.387	1.706
0.057	0.978	1.035	0.040	0.486	0.526	0.042	1.243	1.285	0.113	1.168	1.281
0.040	0.898	0.938	0.061	0.290	0.351	0.031	1.213	1.244	0.036	1.265	1.301
0.085	0.953	1.038	0.395	0.308	0.703	0.037	1.386	1.423	0.025	1.232	1.257
0.025	0.987	1.012	0.048	0.654	0.702	0.069	1.616	1.685	0.078	1.293	1.371
0.052	0.951	1.003	0.050	0.302	0.352	0.396	1.326	1.722	0.205	0.821	1.026
0.057	0.924	0.981	0.147	2.018	2.165	0.162	1.497	1.659	0.074	4.624	4.698
0.028	1.023	1.051	0.081	0.952	1.033	0.055	1.435	1.490	0.025	2.017	2.042

Table V: Wall Loss, Filter, and Combined Masses

#### SIGNATURE PAGE

This is to certify that the thesis prepared by Curtis Carroll entitled "Assessing Particulate Matter Wall Loss with Conductive vs. Non-Conductive Cassettes used for Airborne Mineral Dust Sampling" has been examined and approved for acceptance by the Safety, Health, and Industrial Hygiene Department, Montana Technological University, on this 24th day of April, 2020.

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