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### **Use of dust fall filters as passive samplers for metal concentrations in air for communities near contaminated mine tailings**

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#### **Abstract**

Mine tailings are a source of metal exposures in many rural communities. Multiple air samples are necessary to assess the extent of exposures and factors contributing to these exposures. However, air sampling equipment is costly and requires trained personnel to obtain measurements, limiting the number of samples that can be collected. Simple, low-cost methods are needed to allow for increased sample collection. The objective of our study was to assess if dust fall filters can serve as passive air samplers and be used to characterize potential exposures in a community near contaminated mine tailings. We placed filters in cylinders, concurrently with active indoor air samplers, in 10 occupied homes. We calculated an estimated flow rate by dividing the mass on each dust fall filter by the bulk air concentration and the sampling duration. The mean estimated flow rate for dust fall filters was significantly different during sampling periods with precipitation. The estimated flow rate was used to estimate metal concentration in the air of these homes, as well as in 31 additional homes in another rural community impacted by contaminated mine tailings. The estimated air concentrations had a significant linear association with the measured air concentrations for beryllium, manganese and arsenic (p<0.05), whose primary source in indoor air is resuspended soil from outdoors. In the second rural community, our estimated metal concentrations in air were comparable to active air sampling measurements taken previously. This passive air sampler is a simple low-cost method to assess potential exposures near contaminated mining sites.

#### **Environmental Impact**

Multiple air samples are necessary to characterize exposures and determine potential health impacts in communities near contaminated mining sites. Given that there are hundreds of thousands of inactive or abandoned sites in the Western United States alone, it is not feasible to use conventional sampling techniques to characterize these exposures, particularly in rural

communities. We present a technique to estimate metal concentrations in air using dust fall filters as passive samplers. This technique could be used to screen communities and provide temporal and spatial distributions, so that active air sampling techniques could be more effectively targeted.

#### **Introduction**

The United States Environmental Protection Agency (US EPA) Office of Inspector General identified 156 "mega" hard rock mining sites nationwide with the potential to cost \$24 billion to clean up and maintain, which is over 12 times the annual US EPA budget for remediation of large hazardous waste sites (i.e., Superfund site).<sup>1</sup> However, there are thousands more inactive and abandoned mines across the arid Western United States, with approximately 80,000 sites covering 136,653 acres in the state of Arizona alone.<sup>2</sup> Mine tailings and smelter ash at these sites can be resuspended by wind and blown for hundreds of miles and potentially have widespread impacts on human health.<sup>3</sup> These particles, which may contain high concentrations of metals, can be inhaled or ingested. They pose a particular risk for children, who breathe more air and ingest more dust on a per body weight basis than adults and whose developing bodies are more susceptible to the adverse health effects of metals.<sup>4</sup>

Traditionally, these risks are assessed from soil and outdoor air samples in potentially affected communities, which are used to estimate exposures.<sup>5</sup> However, on average, Americans spend 87% of their time in enclosed buildings.<sup>6</sup> Thus, the indoor environment is where they have the greatest risk of exposure and where samples should be collected. In addition, air samples are collected with expensive equipment that requires electricity and regular visits by specialized personnel for deployment, calibration and maintenance. Multiple samples are also necessary to understand the spatial and temporal distribution of these exposures, but are often limited by equipment and personnel constraints. Given the scale of the problem (i.e., 80,000 sites in Arizona alone) and that many of these sites are in rural areas where access to trained professionals is limited, simpler low-cost methods are needed to screen for potential impacts from metals in communities near abandoned mines.

Passive samplers have been developed to provide low-cost simple alternatives to traditional air sampling techniques and are one of the few practical technologies that could be used to achieve the vast sampling requirements posed by these mining sites.<sup>7</sup> Traditionally, passive samplers have been used for measuring organic gases and vapors,  $8$  where principles of diffusion and permeation into a sorbent are used to estimate sampling rates and air concentrations. Several devices have also been developed for passive collection of aerosols, which utilize a variety of physical structures and collection media.<sup>9–11</sup> However, it is not clear how feasible it would be to distribute some of these more complicated passive sampling devices, which still require specialized personnel, in rural communities. Additionally, these samplers have been primarily evaluated in occupational environments where concentrations are much higher, and it is not clear if they would be adequate at typical ambient levels (i.e., in a home). $9,10$  Furthermore, the few passive samplers that have been used to measure relatively low concentrations do not contain substrates that are readily adaptable for chemical analysis of aerosols.<sup>11</sup> Natural vegetation including leaves, tree rings,

bark, and lichens have been used as passive samplers for measuring metal concentrations in air near mines, smelters and even from traffic.<sup>12–16</sup> However, they are designed for outdoor air collection and are probably not practical for assessing indoor air concentrations. Deposited dust has been used to monitor outdoor air pollution by metals near a smelter plant<sup>17</sup> and to assess their spatial distribution in mining regions.<sup>18</sup> Similarly, dust collection via dry deposition onto settling plates has been used to characterize potential exposures in homes.<sup>19</sup> These studies typically report a dust loading in mass of dust deposited per surface area per unit time. However, while measures of dust loading do allow for comparisons between homes and assessment of spatial and temporal distributions, they are not readily comparable to screening levels for metal concentrations in air, which are expressed as mass of a contaminant per volume of air.

The objective of our study was to determine if dust fall filters could be used to estimate air concentrations in rural communities impacted by mining wastes. The first part of our study consisted of collecting settled dust on large filters concurrently with active air sampling to estimate a flow rate for the passive filters. We also wanted to determine if there were meteorological factors that might affect these flow rates, even if the samples were taken indoors. We then used the estimated flow rates and metal loadings collected on the dust fall filters to estimate the metal concentrations in air and compared them to metal concentrations in air measured with active samplers using traditional methods. The second part of this study consisted of evaluating the use of these passive samplers to characterize the potential for exposures in a second rural community impacted by mining wastes.

#### **Methods**

#### **Study Populations and Recruitment**

Between January and May 2009, we recruited 10 households through random mailings in the Valencia West and Drexel Heights neighborhoods of Tucson, Arizona. These neighborhoods are adjacent to the Bureau of Land Management Saginaw Hill site, where there is an abandoned mine. We selected these particular neighborhoods because there was concern about contamination of the residences and yards from wind resuspension of mine tailings. At one household, samples were obtained on two separate occasions.

Between July 2012 and June 2013, we recruited 31 households living within 5 miles of the Iron King Mine and Humboldt Smelter Superfund Site in Dewey-Humboldt, Arizona. Households were recruited through advertisements at community festivals and venues, doorto-door solicitation, and mailings to households in the study area. Households had to have at least 1 child between 1–11 years of age to participate. In this part of the study, recruitment and all sample collection were completed by trained community members. We selected this community because there is concern about exposure to arsenic, beryllium, nickel, cadmium, lead,, aluminium, and chromium from resuspension of the mine tailings and smelter ash.<sup>20</sup> The study was approved by the University of Arizona Human Subjects Protection Program. Informed consent was obtained prior to sample collection.

#### **Sample Collection**

In both communities, we collected indoor dust fall by modifying the ASTM Standard Test Method for Collection and Measurement of Dust Fall (Settleable Particulate Matter).<sup>21</sup> Glass fiber filter (GFF) or mixed cellulose ester (MCE) filters, of 120 mm diameter, were placed in petri dishes that were then placed in 3.79 L stainless steel cylinders (190 mm high by 159 mm diameter). These cylinders were placed at a height of 1 m in each home for 7 days. One GFF was used for gravimetric analysis and 3 MCE filters were used for analysis of metals in the dust fall, for a total of 4 cylinder collection devices per home. New petri dishes were used for each collection and the steel cylinders were cleaned with isopropyl alcohol between collections.

In the first part of the study near Saginaw Hill, two parallel active air samples were collected with SKC AirCheck XR5000 at 4 liters per minute for 7 days, concurrently with the collection of dust fall samples. Particulate samples were collected with a SKC Button Aerosol Sampler, following the American Conference of Governmental Industrial Hygienists sampling criteria for inhalable particulates. One sample was taken with a GFF for gravimetric analysis, while the other was taken with an MCE filter for elemental analysis. All samples (i.e. air and dust fall) were transported on blue ice and stored in the refrigerator at 4°C until analysis. Meteorological variables (i.e., temperature, relative humidity, wind speed, and precipitation) corresponding to the collection period were obtained from the MesoWest Database.<sup>22</sup>

#### **Sample Preparation and Laboratory Analysis**

The GFFs were weighed on a microbalance. Each MCE dust fall filter from Saginaw Hill was digested in 2.5 mL of distilled water and 7.5 mL of concentrated nitric acid in a microwave (Mars Xpress, CEM Corporation, Matthews, NC) using EPA Method 3051. After digestion, the three samples from each household were combined and reduced to 15 mL on a hot block prior to analysis. The MCE filters from the active air samples in Saginaw Hill and the MCE dust fall filters from Dewey-Humboldt were digested in 10 mL of nitric acid in the microwave. All samples were centrifuged, and the supernatant was submitted for analysis.

The Arizona Laboratory for Emerging Contaminants (ALEC) performed the analyses using inductively coupled plasma mass spectrometry (ICP-MS) for multi-element determinations. For this study, the concentration of arsenic, beryllium, manganese, nickel, cadmium, lead, aluminium, and chromium was measured. Samples collected from Saginaw Hill were analyzed using an ELAN DRC-II ICP-MS (Perkin Elmer, Shelton, CT) instrument equipped with a PFA-ST nebulizer and a cyclonic quartz spray chamber. Argon, platinum sample and skimmer cones were used for all analyses. Samples collected in Dewey-Humboldt were analyzed using an Agilent 7700x ICP-MS (Agilent Technologies, Santa Clara, CA) equipped with a collision cell using helium gas and nickel cones.

For analytical quality control, a check solution (from an independent source and comparable to a low-to-midrange standard) was analyzed after the calibration and before each sample set. Also, a NIST sample (NIST 1643e Trace metals in water) was included at the beginning

and end of each sample set to assess quality control on the dissolved metals in solution. According to US EPA Method 6020, these quality control checks, referred to as Initial Calibration Verification standards and Independent Calibration Verification, were within 10% of their expected value. A mid-range standard was analyzed after every 10 samples and at the end of the run as a Continuing Calibration Verification, and the results were within 25% of the expected value. Calibration standards were prepared from multi-element stock solutions purchased from AccuStandard (New Haven, CT). The stocks were diluted in 1% nitric acid to provide a working calibration curve of at least 5 points. Samples were also diluted with 1% nitric acid until their response was determined to be within the calibration range. Internal standards (Rh, In and Ga) were added to both standards and samples prior to analysis. All analytical measurements were performed in triplicate with an average relative standard deviation of 3%.

As the two parts of this study were conducted during different time periods and with different analytical instruments, blanks and duplicates were submitted during each collection period. There were 3 field filter blanks, 3 laboratory filter blanks, 4 duplicates, and 3 samples of nitric acid submitted with the samples from Saginaw Hill. There were 3 field filter blanks, 15 laboratory filter blanks, 3 duplicates, and 6 samples of nitric acid submitted with the samples from Dewey-Humboldt.

#### **Calculations and Statistical Analyses**

We used the air concentration and the dust fall rate obtained concurrently in Saginaw Hill to estimate a flow rate for the filters, according to:

$$
Q{=}\frac{D_{\rm \scriptscriptstyle F}\times SA}{C_{\rm \scriptscriptstyle S}}
$$

where  $D_F$  is the dust fall rate ( $\mu$ g/cm<sup>2</sup>/hr), *SA* is the surface area of the passive filter (cm<sup>2</sup>) and  $C_s$  is the concentration of inhalable particulates in the air sample ( $\mu$ g/m<sup>3</sup>). For these calculations we used the bulk particle mass obtained from gravimetric analysis of the active and passive GFFs. These calculations were performed in Microsoft Excel 2011 (Microsoft Corporation, Seattle, WA).

We calculated the average temperature, relative humidity, wind speed and daily precipitation for each of the concurrent active and passive sampling periods near Saginaw Hill. We used classification tree analysis in S-PLUS 8.0 (Insightful Corporation, Seattle, WA) to identify meteorological factors that have the strongest impact on the estimated flow rate. Because of our small sample size, we used Wilcoxon rank sum tests to assess if the differences in flow rates were significant as a function of these groups.

Using the estimated flow rates and the metal loading obtained on the passive MCE samplers, we estimated the concentration of the metals in air. We used linear regression to assess the association between metal concentrations obtained from the passive and active samplers. We applied the estimated flow rates from the first part of the study (i.e., near Saginaw Hill) to calculate the concentration of metals in the air from Dewey-Humboldt.

With the exception of the tree analysis, all statistical analyses were performed in STATA SE 12 (StataCorp, College Station, TX). An alpha level of 0.05 was considered significant for all statistical tests. After blank correction, values below the laboratory limit of quantification were substituted with the limit of quantification divided by the square root of two<sup>23</sup> prior to all subsequent calculations.

#### **Results**

A summary of the dust fall rates, inhalable particulate matter concentrations, estimated flow rates, and meteorological variables for the samples collected near Saginaw Hill are provided in Table 1. Duplicate inhalable particulate matter concentrations had a relative percent difference of 7%. The relative percent difference between dust fall rates measured during different sampling periods in the same house was 26%. Because these samples were taken during different sampling periods, variability in household activities may have contributed to the larger difference.

Using classification tree analysis, we identified the value for each of our meteorological variables that provided the greatest difference in subsets of our estimated flow variable. Summary statistics for the estimated flow rates as a function of the meteorological variables are presented in Table 2. The flow rates were most affected by relative humidity and precipitation. The flow rates for the passive filters during sampling periods with almost no precipitation were significantly lower than the flow rates during sampling periods with precipitation.

Because of the significant difference in estimated flow rates as a function of precipitation, the mean estimated flow rate for each precipitation category was used in subsequent analyses. Using the mean estimated flow rate corresponding to the precipitation category during the active air sampling period (Table 2), we then estimated metal concentrations in air from our passive filters. The estimated air concentrations from the passive samplers had a significant linear association with the measured air concentrations for arsenic, beryllium, and manganese, but not for nickel, cadmium, lead, or aluminium (Table 3). Chromium was only detected on one dust fall filter from Saginaw Hill and thus was not used in these analyses.

We estimated indoor air concentrations in Dewey-Humboldt, near the Iron King Mine and Humboldt Smelter Superfund Site, using the mean estimated flow rate corresponding to the appropriate precipitation category (Table 2) during the passive filter sampling period (Table 4). Although we did not take any active air samples in Dewey-Humboldt, we were able to compare our results with those from active air sampling measurements reported in the Remedial Investigation Report for the Superfund Site performed for the US EPA. 20 With the exception of beryllium, the distribution of our results was within the same range as other measurements obtained in the town. Our results underestimated the concentration of beryllium by almost an order of magnitude. When comparing our results to the appropriate screening levels, our estimates indicate that arsenic, nickel, and chromium air concentrations are of most concern in this community.

#### **Discussion**

In this study, we evaluated the use of dust fall filters as passive air samplers for metals in communities that may be impacted by wind resuspension of contaminated mine tailings. Our estimated flow rates, obtained from concurrent active and passive air sampling, were most influenced by precipitation during the sampling period. Significant linear associations were observed between the concentrations measured using both methods for arsenic, beryllium and manganese. Deployment of these passive samplers in Dewey-Humboldt, near the Iron King Mine and Humboldt Smelter Superfund Site, resulted in estimated air concentrations that were comparable to those measured previously in the same community. These passive filters are a cost-effective and simple method that could be used to screen and prioritize the thousands of communities residing near potentially contaminated mining sites.

In other studies, temperature and relative humidity were found to have the greatest effect on collection of aerosols with passive air samplers.<sup>7</sup> However, in our study we determined that precipitation had the greatest effect on the estimated flow rates for our passive samplers. The estimated flow rate was significantly higher if there was precipitation during the sampling period (Table 2). We estimated the flow rates as a function of the mass of settled dust, surface area of the passive filter, and the concentration of bulk inhalable particles in the air measured through concurrent active air sampling. The concentration of particles in air was significantly lower if there was precipitation during the sampling period (Wilcoxon rank sum, p=0.005). No significant differences were observed for the dust fall rates or the sampling surface area as a function of precipitation. Wet removal of particulates by precipitation lowers the concentration of particles in outdoor  $\arctan 24$  and likely reduced the amount of resuspended soil in outdoor air. However, we determined a significant decrease in particle levels in indoor air as a function of precipitation. It is possible that this is a result of less outdoor air particle penetration during those sampling periods as well as wet deposition associated with increased humidity in the indoor environment during precipitation events. Others have also reported a significant negative correlation between rain and particulate matter in an unoccupied classroom.<sup>25</sup> Future work is warranted to investigate this phenomenon and how it may affect exposures near contaminated sites.

In a previous publication, we discussed how house dust and soil ratios could be used to prioritize metals for future air quality investigations and delineate those metals that primarily arise from outdoor soil from those that have other indoor sources.26 House dust and soil were collected at the same time and in the same homes as our concurrent active and passive air sampling near Saginaw Hill. Manganese, beryllium and arsenic had the lowest dust/soil ratios of all 35 elements assessed with a geometric mean of 0.55, 0.63, and 0.70, respectively. Interestingly, these are the same metals that demonstrated a significant linear association between the active and passive air sampling in the same homes. As our passive filters primarily rely on gravitational settling of the suspended particles, they are more likely to be enriched in larger heavier particles, which primarily arise from soil track-in on footwear and wind resuspension of soil.

Conversely, significant linear relationships between the air concentrations measured using the passive and active methods were not observed for nickel, cadmium, and. lead However,

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these metals all had dust/soil ratios greater than one, indicating that they may have indoor sources. In Tucson, chromium was detected on only one dust fall filter but in all but one of our active air filters. Chromium also had a dust/soil ratio greater than 4, indicating that there are substantial indoor sources. Both environmental tobacco smoke and cooking are associated with increased indoor particulate concentrations for nickel, cadmium, lead, and chromium in residential environments.27,28 Environmental and tobacco smoke are combustion sources that primarily generate ultrafine and fine particles, which have much lower gravitational settling velocities.<sup>29</sup> In particular, the ultrafine particles begin to behave more like gas molecules. Our dust fall filters would not be as efficient at capturing these molecules, which may explain the lack of association between the active and passive filters. In a study that examined the size of particles collected on dust deposition plates, significant differences were found in relation to the height of the plates for large particles but not for small particles.<sup>19</sup> In the future, dust fall filters could be used at different heights to help define sources of the metal particles and provide better characterization of the contribution from outdoor sources like contaminated mining sites.

Although aluminium also had a low dust/soil ratio and most likely arises in the indoor environment from outdoor soil, there was not a significant association between the active and passive samples (Table 3). However, the blank dust fall filters, submitted at the time of the collection in the Saginaw Hill area had a high aluminium concentration (mean=2994 μg/L) resulting in many non-detectable values after blank correction. The blank dust fall filters submitted at the time of the Dewey-Humboldt collection had a much lower concentration (mean=902 μg/L). It is possible that the lack of correlation between the active and passive air samples may be due to the high background concentration in the filters during that sampling period relative to the concentration of aluminium in the air. In the future, if aluminium is an analyte of interest, it may be important to identify filters with lower background levels or use a much longer sampling duration unless the air concentrations are known to be very high. In Dewey-Humboldt, where the aluminium concentrations in air were much higher than near Saginaw Hill (Table 4) and the background levels in the dust fall filters were lower, we only had one non-detectable value.

Not only are residents who live near contaminated mine tailings exposed via inhalation of respirable aerosols that can penetrate deep in the gas-exchange region of their lungs, they are also exposed to the metal contaminants of inhalable particles that can deposit anywhere in their lungs because these can also be coughed up and subsequently ingested. Furthermore, they can be exposed to these metals via ingestion of the contaminated dust on their hands or other surfaces in their homes. The majority of dust that adheres to hands is  $\langle 100 \mu m.30 \rangle$ Thus, it is important to highlight that we collected inhalable aerosol samples using the SKC Button Sampler, which has the efficiency to collect at least 50% of the particles <100 microns. We did not collect respirable aerosol samples, which should be collected with a device that has 50% efficiency at 4.5 microns and less for larger particles. Future work should be completed to understand how the relationships between the passive and active samples in the current study would be impacted by the size distribution of particles collected in the active air samples. However, in a previous study of settled dust on glass plates in homes on average 88% of the particles were 5 microns, and only 1 particle in the 31 samples was greater than 100 μm.19 Although we do expect our passive sampler to under

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sample the smaller particles in proportion to their distribution in the air, we do think that it has the potential to collect the small respirable aerosols that arise from Aeolian resuspension of contaminated mine tailings and are capable of infiltrating the indoor environment. The bias created from under sampling the smaller size fraction, should be consistent between homes and there should not impact our goal of using this device as a screening mechanism. However, it would be important in the future to conduct a robust analysis of the passive and active air samples both indoors and outdoors to understand how the accuracy and precision of the passive samplers is impacted by the size distribution of particulates in the air.

Despite the limitations we have identified, this passive sampling method has many advantages. It provides a simple low-cost alternative or complement to active air sampling techniques. Because of their purchase and maintenance costs, the number of samples that can be taken by active samplers is limited and it is impractical to use them for extended periods to measure chronic exposures, as is necessary for epidemiology studies. Passive samplers can be deployed for much longer, providing a time-weighted average that is difficult to obtain with active/grab sampling technologies. For example, it has been demonstrated that moss bags provide better chronic estimates of exposure for epidemiological investigations when compared to active air samplers.<sup>13</sup> In addition to air concentrations, the deposition rate of metals on indoor surfaces, such as our passive samplers, is also a potentially important metric for assessing dust ingestion of contaminants. For example, in another study near a smelter, children's blood lead levels were significantly correlated with the dust deposition rate of lead on surfaces in their homes, but not with the concentration of lead measured in their indoor air or floor dust.<sup>31</sup> The passive samplers provide metric of the settled dust concentrations in the home, which is deposited on every household surface and available for subsequent ingestion. Thus, these passive samplers have the potential to help characterize, not just one, but two important exposure routes (i.e. inhalation and ingestion) for contaminated dust in mining and smelting communities. Furthermore, passive samplers do not require a power source and are silent, making them more amenable and less obtrusive for sampling in participants' homes. Using these samplers in people's homes provides an exposure metric that accounts for the particles capable of infiltrating the indoor environment, where people spend the majority of their time<sup>6</sup> rather than relying on outdoor measurements. In rural settings they could even be mailed to participants, substantially lowering study costs and increasing the number of samples that could be obtained.

#### **Conclusions**

Dust fall filters are a reasonable approach for measuring concentrations of metals from resuspended soil in air, but further work is needed to determine if they can be used to assess metal concentrations from other sources. Our estimated air concentrations from these passive samplers were comparable to those measured using traditional methods. Metal concentration estimates from the passive samplers were higher in Dewey-Humboldt, near the Iron King Mine and Humboldt Smelter Superfund Site, than those from our measurements near Saginaw Hill, which is confirmed with the results from the active air sampling measurements in both communities. It is important to note that while these dust fall filters should not be used for regulatory purposes without further evaluation, these

results demonstrate that they could be used as a screening method to identify high-risk communities or in epidemiological investigations where relative concentrations are needed. Given their low cost and simplicity, these dust fall filters could be very useful for community-based participatory research studies near the thousands of inactive or abandoned mines in the Western United States.

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Summary of dust fall rates, inhalable particulate matter air concentrations, surface area, estimated flow rates and meteorological variables obtained near Saginaw Hill.

**Table 1**



*\** standard deviation

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# **Table 2**

Estimated flow rates (m<sup>3</sup>/hr) as a function of meteorological variables from samples taken near Saginaw Hill Estimated flow rates (m3/hr) as a function of meteorological variables from samples taken near Saginaw Hill



## **Table 3**

Linear regression of metal concentration (ng/m<sup>3</sup>) measured from the active and estimated from the passive samplers near Saginaw Hill Linear regression of metal concentration (ng/m<sup>3</sup>) measured from the active and estimated from the passive samplers near Saginaw Hill



*+*

β - coefficient from linear regression

### **Table 4**

Estimated air concentrations of metals from passive samples taken in Dewey-Humboldt, and comparison to measured air samples and screening levels Estimated air concentrations of metals from passive samples taken in Dewey-Humboldt, and comparison to measured air samples and screening levels obtained from the Remedial Investigation Report for the Iron King Mine and Humboldt Smelter Superfund Site <sup>20</sup> obtained from the Remedial Investigation Report for the Iron King Mine and Humboldt Smelter Superfund Site 20



measurements taken in the Town of Humboldt;

 $+$  for Pb this is the Annual Ambient Air Quality Standard, for all other metals it is the US EPA Residential Regional Screening Level  $20$ *+*for Pb this is the Annual Ambient Air Quality Standard, for all other metals it is the US EPA Residential Regional Screening Level 20