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# **Differences in Metal Concentration by Particle Size in House Dust and Soil**

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# **Abstract**

The majority of particles that adhere to hands are  $<$  63  $\mu$ m in diameter yet risk assessments for soil remediation are typically based on soil samples sieved to  $\langle 250 \mu \text{m} \rangle$ . The objective of our study was to determine if there is a significant difference in metal concentration by particle size in both house dust and soil. We obtained indoor dust and yard soil samples from 10 houses in Tucson, Arizona. All samples were sieved to  $\langle 63 \mu \text{m} \rangle$  and 63 to  $\langle 150 \mu \text{m} \rangle$  and analyzed for 30 elements via ICP-MS following nitric acid digestion. We conducted t-tests of the log-transformed data to assess for significant differences that were adjusted with a Bonferroni correction to account for multiple comparisons. In house dust significant differences in concentration were observed for Be, Al, and Mo between particles sizes, with a higher concentration observed in the smaller particles size. Significant differences were also determined for Mg, Ca, Cr, Co, Cu, Ge, Zr, Ag, Ba, and Pb concentration in yard soil samples, with the higher concentration observed in the smaller particles size for each element. The results of this exploratory study indicate that current risk assessment practices for soil remediation may under estimate non-dietary ingestion exposure. This is of particular concern for young children who are more vulnerable to this exposure route due to their high hand mouthing frequencies. Additional studies with a greater number of samples and wider geographic distribution with different climates and soil types should be completed to determine the most relevant sampling practices for risk assessment.

# **Introduction**

Children living near hazardous waste sites contaminated with metals and other non-volatile chemicals receive the majority of their exposure via indirect ingestion of soil and dust that results from hand-to-mouth transfers of the contaminated media.1-3 Soil samples are typically taken from these sites and analyzed to estimate risk of exposure to the contaminants and develop soil remediation strategies. The United States Environmental Protection Agency (US EPA) guidelines for soil sampling recommend that the risk assessor should determine the most appropriate sieve size for their site.<sup>4</sup> However, typically soil samples are sieved to particle size  $\langle 250 \mu \text{m} \, (\text{mesh} \, #60) \rangle$  based on sampling recommendations for sites contaminated with lead.<sup>5</sup> This recommendation arises from a survey of the dermal adherence literature at that time. However upon closer inspection of the primary articles, the majority of these authors actually recommended sampling soil with particle sizes much smaller than  $250 \mu m$  (Table 1). More recent articles addressing dermal adherence have also determined that the majority of particles that adhere to hands are much

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smaller than the <250  $\mu$ m particle size typically used in risk assessments (Table 1). In particular Choate et al.<sup>10</sup> has demonstrated that the majority of particles that adhere to hands in several trials are  $\lt$ 63  $\mu$ m (i.e., delineation between silt and sand particles), regardless of the particle size distribution of the bulk soil. Soil particles in this smaller size fraction are also more likely to adhere to footwear or be resuspended by wind, allowing for subsequent penetration to the indoor environment where children's exposures are likely to occur through contact with house dust<sup>12</sup>.

Several studies have also demonstrated that concentration of several contaminants in soil and dust may vary by particle size.<sup>13-17</sup> Sheppard and Evenden<sup>7</sup> determined, using spiked soil with U, Pb, Hg, I, and hexachlorobenzene, that particles  $\leq 50 \,\mu$ m in diameter could have up to 10-fold higher concentration than the bulk soil. Gulson et al.<sup>18</sup> reported that the highest concentration of Pb for both soil and house dust was in the size fraction >38 μm to

53 um, but did not examine other metals or contaminants. Calabrese et al.<sup>19</sup> demonstrated that not only was the concentration of certain crustal elements higher in the smaller particle size fraction ( $\langle 250 \mu m \rangle$ ; using the concentration of these elements in the smaller particle size fraction improved inter-tracer consistency for soil ingestion studies. Thus, individuals are more likely to ingest soil from the smaller particle size fraction and the concentration of elements in the smaller size fraction is more representative of their exposure than the concentration in the bulk soil. Unfortunately, the smallest size fraction they examined was  $\langle 250 \mu \text{m}$ , rather than particle sizes in the range that adheres to hands more efficiently. Although Fergusson and  $Ryan^{20}$  did not perform any statistical analysis, they demonstrate visually how the concentration of 17 different elements in street dust from industrial areas decreases with particle size. This trend becomes most evident for the concentration of elements in size fractions with particles  $\langle 111 \mu m$ .

Given that the particles that are most likely to adhere to hands are much smaller and these particles may have a higher concentration of contaminants of concern, current soil sampling practices that sieve particles to  $\langle 250 \mu \text{m} \text{ may not be the most reliable for health-based risk}$ assessments.6, 9, 18 We examined differences in metal concentrations between particle sizes from matched soil and house dust samples obtained from residential environments potentially impacted by a hazardous waste site. Although others have examined differences in concentration of contaminants between particle sizes, our study is different for several reasons. Our samples were not spiked artificially or from industrial sites. As bioavailability may also affect the risk of exposure,  $18$  we analyzed the soluble fraction via an acid digestion rather than the total concentration in the samples. Previous studies did not report statistical analyses, and thus it is not clear if the differences in concentration between particle sizes are significant. We performed statistical analyses to compare the concentration between mutually exclusive particle sizes in the range that adheres most efficiently to hands (i.e., <63  $\mu$ m and 63 to <150  $\mu$ m).

#### **Methods**

#### **Sample Collection**

From January to May 2009, 10 households were recruited in Tucson, Arizona. These households were recruited through random mailings in a neighbourhood near an abandoned mine, where there was concern about contamination of the residences and their yards. Dust samples were taken using a 2.2 horsepower vacuum cleaner equipped with a special inlet containing a filter holder.21 A 6-inch square Teflon coated fiberglass backed filter was used to collect the dust. At each house a 1  $m^2$  template was laid on the floor in the room where residents spend the majority of their time. The area was vacuumed for two minutes. This was repeated until approximately 2 grams of dust were collected. The floor area sampled was in proportion with the type of flooring throughout the room. For example, if the room

Two trowels of soil were taken from each of the four sides of the house. The soil samples were taken at a location approximately 3 m perpendicular to the centreline of each side of the house. Soil samples were combined in one plastic bag per household. In addition, settled dust, indoor and outdoor air were sampled and a questionnaire was administered at each house but the results were not used for the current study.

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

Soil and dust samples were dried in an oven at 43°C for 8-10 hours. As recommended by the US EPA, homogenization was achieved through sicving.<sup>5</sup> The entire dust or soil sample was sieved using an electrical shaker (Ro-tap, Tyler, Mentor, OH) into the following particle sizes:  $>1$  mm (Mesh #18),  $<1$  mm to  $>150 \mu$ m (Mesh #100),  $<150 \mu$ m to  $> 63 \mu$ m (Mesh #230), and < 63 μm. 500 mg of each sample were microwave (Mars Xpress, CEM Corporation, Matthews, NC) digested in 10 mL of concentrated reagent grade nitric acid using EPA Method 3051. Only the smallest two size fractions were submitted for analysis.

Analytical work was performed by the Arizona Laboratory for Emerging Contaminants (ALEC) using inductively couple plasma mass spectrometry (ICP-MS) for multi-element determinations. Analyses were performed using an ELAN DRC-II ICP-MS (Perkin Elmer, Shelton, CT) instrument equipped with a PFA-ST nebulizer and a cyclonic quartz spray chamber. The instrument was operated with data acquisition by 'peak hopping.' RF power was maintained at 1450 W. The argon gas flow rates were 0.95, 15, and 1.3 l/min for the nebulizer, coolant and auxiliary respectively. Platinum sample and skimmer cones were used for all analyses. The sample uptake rate was 0.4 ml/min. The dwell time was 50 ms with 50 sweeps per replicate and 3 replicates.

There were 2 house dust and 2 yard soil sample duplicates submitted for analysis with the other samples. In addition, 3 nitric acid blank samples were submitted. 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, must fall 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 must fall 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%.

#### **Data Analysis**

Data analysis was performed using Microsoft Excel Version 12.3.0 (Microsoft Corporation, Seattle, WA) and STATA 11 (StataCorp, College Station, TX). The elemental concentrations in soil and dust were adjusted for the elemental concentration in the nitric acid blanks prior to analyses. Samples below the detection limit were assumed to be equal to half the minimum detection limit for data analysis. As environmental concentrations are log-

normally distributed,<sup>22</sup> all data were log-transformed prior to analysis. A two-tailed t-test on the log-transformed data was used to test for significant differences between the elemental concentration in the <63  $\mu$ m size fraction and the <150  $\mu$ m to <63  $\mu$ m size fraction in both soil and dust respectively. A Bonferroni correction was used to account for multiple comparisons (30 elements), thus concentrations were considered significantly different between particle sizes at p<0.0017.

# **Results**

The particle size distribution by mass is presented in Table 2 for the yard soil and house dust samples. A greater fraction of house dust is present in the smaller particles size ( $\leq 63 \,\mu m$ ) than for soil.

The concentrations of elements in house dust and yard soil are presented by particle size in Tables 3 and 4, respectively. Duplicate field samples from two households had an average relative percent difference of 14% and 17% for the two particle sizes in soil. Similarly they had a relative percent difference of 59% and 56% for the two particle sizes in house dust. The greater precision in soil has been observed in other studies and may be due to less heterogeneity.<sup>23</sup> House dust samples also had a greater geometric standard deviation than soil samples. This may indicate greater environmental variability house-to-house due to additional sources indoors or other household characteristics.

Significant differences were observed between particle sizes for the concentration of elements in both house dust and soil (Tables 3 and 4). Observed log mean differences, 95% confidence intervals and p-values are provided in the Supplementary information. After accounting for multiple comparisons, the concentration of Mg, Ca, Cr, Co, Cu, Ge, Zr, Ag, Ba, and Pb were significantly different between particle size fractions in soil. Similarly, the concentration of Be, Al, V and Mo were significantly different between particle sizes in house dust. The observed log mean difference and confidence interval were positive in all cases, indicating that concentrations are higher in dust and soil particles  $\langle 63 \mu m \rangle$  in diameter compared to the larger size fraction.

# **Discussion**

We analyzed the concentration of 30 elements for two particle size fractions (i.e., <63 μm,  $63$  and  $\lt 150 \,\mu\text{m}$ ) in matched house dust and soil samples. Our study confirms the results of previous studies,  $7, 13-20$  that the concentration was significantly higher for several elements in the smaller size fraction for both house dust and soil. However, we are the first study to focus on examining the  $\lt 63 \mu$ m particle size fraction. Particles in this size fraction are more likely to adhere to hands and thus be ingested regardless of the characteristics of the bulk soil.<sup>10</sup> Given that the concentration may be higher in the smaller size fraction, risk assessments and soil remediation strategies that rely on the concentration of the contaminants in bulk soil or larger particle size fractions (i.e.,  $\langle 250 \mu \text{m} \rangle$  may not be reliable.

Many reasons have been proposed for the higher concentration of contaminants in smaller particle sizes. For example, these differences may occur because smaller particles have a higher concentration of organic matter or a higher surface area to unit mass/volume.<sup>7, 13</sup> In addition, soil fractions with smaller particle sizes have a higher percentage of clay, which may provide more sorption sites for contaminants.20 Certain minerals like calcite, gypsum and dolomite are more soluble and prone to geologic chemical weathering.<sup>24, 25</sup> These minerals are composed of Ca and Mg, and this might explain the higher concentration of these elements in the smaller particle size fraction of soil in our current study.

Fergusson and Ryan<sup>20</sup> reported that there was less quartz and feldspar in the smaller outdoor dust size fractions that they studied indicating that these particles may be less likely to originate from the natural weathering of geologic formations and are more likely to arise from other anthropogenic sources. We determined that Cr, Co, Cu, Ge, Zr, Ag, Ba, and Pb were present at higher concentrations in the smaller size fraction of soil, and they are associated with anthropogenic sources present in Tucson.26 Some of these elements are associated with auto use. Cu arises from wearing of brake pads, Ag is associated with catalyst equipped vehicle emissions and Pb is present in soil near roadways due to historical use of leaded gasoline.<sup>27, 28</sup> There is a coal power plant in Tucson, and coal fly-ash can be a major atmospheric source of Cr, Co, Cu, Ba, Pb, Zr and Ge deposits in the top layers of soil.25, 28-31 Aluminum recycling and smelting is a common industry in this region of Tucson. These processes are associated with emissions of Ba, Cr, and Pb.<sup>32</sup> All of these anthropogenic sources produce particles that would be  $\lt 63 \mu$ m and could explain why these elements are higher in the smaller size fraction in our samples. As risk assessments are performed on hazardous waste sites contaminated by anthropogenic sources, this underscores the importance of sampling a smaller particle size of soil.

In our study we observed a higher concentration in house dust of Be, Al and Mo in the smaller particle size fraction. We have previously reported that the primary source of Be and Al in our house dust samples is most likely soil that is tracked in on shoes or settled resuspended soil from outdoor air.33 The majority of soil particles tracked into homes on shoes are  $\langle 38 \mu \text{m.}^{34}$  Similarly, the majority of settled dust in homes is  $\langle 50 \mu \text{m.}^{35}$  Although there is not a significant difference by particle size for the concentration of Be and Al in outdoor soil, it is likely that smaller soil particles are more likely than larger soil particles to be incorporated into house dust through track-in and aeolian resuspension. The concentration of Mo was higher in our dust samples than the matched soil samples indicating that Mo most likely has an indoor source.<sup>33</sup> Mo is an essential mineral present in many vegetables and other foods.<sup>36</sup> Cooking foods does increase the metal concentration in homes, and is the primary source of particles.<sup>37-39</sup> These combustion particles would be much smaller than 63 μm and could settle on surfaces and floors throughout the home. Mo is also a common component of residential heaters, air conditioners, cookware and light bulbs. 40, 41

Our results may be in part due to differences in extraction efficiency by particle size.18 We elected to digest our samples with nitric acid, which liberates the more soluble components of our samples and is more relevant for bioavailability.17, 18 However, nitric acid is not as efficient as stronger acids (i.e., hydrofluoric acid) at yielding a higher proportion of the metals encapsulated in crystalline minerals.42 These minerals, like feldspar and quartz contribute more to the larger particle size fractions. Some of the previous studies obtained total metal concentration via instrumental neutron activation analysis from outdoor dust samples and also observed that smaller size fractions have higher concentrations of metals.<sup>20</sup>

We observed a greater average relative percent difference in our duplicate house dust samples than in our soil samples. However, the average relative percent difference was relatively consistent for the two particle sizes per sampling matrix (i.e., 56% vs 59% for house dust; 14% vs 17% for soil) indicating that heterogeneity was pretty consistent across particle size. Our results demonstrate that house dust in general may have greater heterogeneity, perhaps due to additional indoor sources. Although this is a consistent finding with other studies, it may be important in the future to explore more robust sample homogenization techniques and determine how this may affection the concentration of contaminants across particle sizes. 21 Perhaps the most common soil homogenization technique is grinding followed by sieving. However, grinding will break down the larger particle sizes. Although this may increase their extraction efficiency, it is not clear if the

resulting concentration would be less relevant for risk assessment and not as representative of true exposure. Future studies should consider exploring additional homogenization techniques that do not affect particle size, such as the closed-bin riffle splitter, which apparently has the best ability of homogenization techniques to contain the loss of fines.<sup>43</sup>

Unfortunately for this study we were only able to obtain a limited number of samples  $(n=10)$ and analyze only two-size fractions (i.e.,  $\langle 63 \mu m, 63 \text{ and } \langle 150 \mu m \rangle$ ). However, even with the limited sample size, we were able to determine significant differences in concentration between the particle size fractions for several elements in house dust and soil even after adjusting for multiple comparisons. Although higher concentrations of metals have been reported before in smaller size fractions, this is the first study to report statistical analyses and confirm that these differences are significant. In the future these analyses should be repeated with larger sample size to confirm these differences. Additional analyses should also be performed with other size fractions to understand how these trends continue over the particles sizes relevant for human exposure.

# **Conclusions**

In conclusion, we determined that the smaller particle size of house dust and soil had higher concentrations of multiple elements. The majority of particles that adhere to children's hands are in this smaller particle size  $(<63 \,\mu m$ ).<sup>10</sup> The results of this exploratory study indicate that risk assessments and soil remediation strategies utilizing the current soil sampling practices that sieve particles  $\langle 250 \mu \text{m} \text{ may not be protective enough and underestimate}$ potential exposures. This is of special concern for young children whose primary exposures may be through dermal contact or ingestion of soil and dust particles adhered to their hands. Future investigations with a larger sample size and wider geographic distribution representing different climates and soil types should be completed to determine the most relevant sampling practices and sieving procedures for risk assessment aimed at protecting children's health.

#### **Supplementary Material**

Refer to Web version on PubMed Central for supplementary material.

#### **Acknowledgments**

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Beamer et al. Page 7

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#### **Environmental Impact**

Current risk assessment methods are based on soil samples sieved to a larger particle size than what typically adheres to hands. In our study we compared the concentration of several elements in two particle sizes in both house dust and yard soil samples. We determined that the concentration was significantly higher in the smaller particle size for several elements in both dust and soil. These results indicate that to ensure exposures are more adequately characterized, risk assessment for soil remediation should consider using soil samples sieved to the particle sizes more likely to adhere to human hands.

# Particle Size (μm) Fraction that Adheres Most to Skin



Percentage of soil and house dust mass by particle size



 ${}^{a}$ GM - geometric mean

 $b_{\text{GSD}}$  - geometric standard deviation

Element concentrations (mg/kg) in house dust by particle size from Tucson residences (n=10) Element concentrations (mg/kg) in house dust by particle size from Tucson residences (n=10)





 $p_{\rm DF-}$  detection frequency DF – detection frequency  $c_{0.5*\rm MDL}$  used for samples <BDL 0.5\*MDL used for samples <BDL

 $d_{\mathrm{GM}-$  geometric mean GM – geometric mean

 $e_{\rm GSD-}$  geometric standard deviation GSD – geometric standard deviation

 $\stackrel{*}{*}$  indicates sign<br>ficiance after Bonferroni correction (p<0.0017) indicates signficiance after Bonferroni correction (p<0.0017)

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Element concentrations (mg/kg) in yard soil by particle size from Tucson residences (n=10) Element concentrations (mg/kg) in yard soil by particle size from Tucson residences (n=10)









 ${}^{\Delta}\!\!\mathbf{A}\mathrm{D}\mathrm{L}$  – minimum detection limit, BDL – below detection limit MDL – minimum detection limit, BDL – below detection limit

 $b_{\mbox{\footnotesize DF}-\mbox{\footnotesize detection frequency}}$ DF – detection frequency

 $\rm \acute{o}$  0.5\*MDL used for samples <BDL 0.5\*MDL used for samples <BDL

 $d_{\mathrm{GM}-$  geometric mean GM – geometric mean

 $e_{\rm GSD-geometric\ standard\ deviation}$ GSD – geometric standard deviation

indicates signficiance after Bonferroni correction (p<0.0017)

\*