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## Wipe Sampling Methodologies to Assess Exposures to Lead and Cadmium in Urban Canadian Homes

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# Wipe Sampling Methodologies to Assess Exposures to Lead and Cadmium in Urban Canadian Homes

## **Cover Page Footnote**

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## PART II: Heavy Metals

### Chapter 5

#### WIPE SAMPLING METHODOLOGIES TO ASSESS EXPOSURES TO LEAD AND CADMIUM IN URBAN CANADIAN HOMES

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#### ABSTRACT

Wipe sampling methods are widely used to quantify lead (Pb) loadings inside homes. In the present study we expand the wipe sampling method to investigate other elements in addition to Pb, namely cadmium (Cd) and the soil tracer yttrium (Y).

Following the ASTM 1728 sampling protocol, 1372 wipe samples (including field blanks and duplicates) were collected from 222 homes using Ghost Wipes™. All wipe samples were digested according to a modified version of the ASTM 1644 digestion protocol in which hydrofluoric acid was added to enhance extraction efficiency, and analyzed using ICP-MS. Recoveries assessed using NIST certified reference materials were 93±6% for Pb and 88±14% for Cd (n=66).

Results indicated that 43% of Pb and 23% of Cd samples were below LOD (932 ng m<sup>-2</sup> and 125 ng m<sup>-2</sup> respectively). Threshold values of 125 µg m<sup>-2</sup> for Pb and 4.4 µg m<sup>-2</sup> for Cd, identified using Q-Q plots, were used to distinguish “elevated” loading values from “background” loading values. Indoor sources and tracked-in soil were identified as potential contributors to elevated loading values. Spearman ranking indicated strong spatial associations amongst the metals. The study shows that wipe sampling provides useful information on room-to-room

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variability of metals, shedding light on possible sources of metals in residential environments.

Keywords: exposure assessment, housedust, metals, built environment

## **1. INTRODUCTION**

The average Canadian spends 90% of their time indoors (Health Canada, 2009), and thus there is a growing demand for information on contaminant levels and sources in the indoor environment. In particular, information about sources of metals in the home is necessary to assess childhood exposures caused by ingestion of dust. Industrial land use, traffic emissions, and geological weathering results in the dispersal and settling of metals in soil and street dust which are tracked indoors by residents and their pets. Soil and street dust is thought to contribute anywhere from 20% to 95% of house dust (Rasmussen et al., 2001 and references cited therein). Metals can also originate from interior sources such as paint, household products, crafts and hobbies, and tobacco use, and tend to accumulate in house dust (Rasmussen, 2004).

Cadmium's (Cd) non-corrosive properties allow its application in batteries, pigments, metal coatings, and plastics (Schoeters et al., 2006). At elevated levels of exposure, Cd acts as a nephrotoxicant (Rasmussen and Gardner, 2008 and references cited therein) and as a carcinogen (Bussi eres et al., 2004). The principle non-occupational exposures to Cd occur through diet (Gamberg and Scheuhammer, 1994; Kormaniki, 2005) and tobacco use (Yapici et al., 2006). A recent risk assessment of Cd in house dust in the Netherlands (Oomen et al., 2008) estimated that the above pathways represent 90% of the total daily intake of Cd, and therefore ingestion of house dust containing Cd could potentially play a significant role in an individual's overall exposure. Cadmium and lead (Pb) differ from essential metals such as copper and zinc which are required by the body in trace quantities for optimal health.

The Canadian government has significantly reduced the Pb content of paint and gasoline over the past few decades (Canada Mortgage and Housing Corporation (CMHC), 2009). Relics of Pb's past abundant use appear during renovations of older homes, potentially increasing Pb exposure (Reissman et al., 2002). The extensive literature on childhood exposures to Pb, pointing to the severe consequences of overexposure on early brain development resulting in learning and behavioral problems, has led to a re-evaluation of the 10 micrograms per deciLitre blood Pb regulatory action value (Lanphear et al., 2000; Canfield et al., 2003; Lanphear et al., 2005). Hornung et al. (2009) identified the need to maintain low Pb exposure throughout childhood as blood Pb at 6 years of age was

found to be more highly correlated with neurological health effects than at 2 years of age.

Presently, there are no Canadian guidelines for metal loadings in indoor residential dust. The United States Environmental Protection Agency (USEPA) has set a regulation for Pb in house dust at 40  $\mu\text{g ft}^{-2}$  on floors, based on the collection of house dust using the wipe method (USEPA, 2000). For the purpose of determining individual exposures to residential sources of Pb, many researchers view wipe sampling as superior to vacuum sampling. Wipe sampling mimics a child's hand contact with hard surfaces and it is thus argued that a child has greater potential to be exposed to metals in dust settled on smooth surfaces versus carpeted surfaces (Yiin et al., 2002). Additionally, Rodes et al. (2001) found that only one third of a child's hand actually touches a surface, therefore vacuum sampling may not represent tangible exposure. The question of where to sample was addressed by Wilson et al. (2006) as USEPA (2000) does not specify the optimal location or number of samples to be taken within the home. Wilson et al. (2006) identified the best predictor of elevated blood Pb in a child to be non-carpeted floor wipe samples collected in the home's entry area, living room, kitchen, and bedrooms.

The purpose of the present study is to quantify metal loadings in urban homes with the aim of establishing background levels for residential exposure in Canada. As such it represents the first published dataset of its kind for Canada. The only other published Canadian study to use wipes as the sampling medium was conducted by CMHC (1995) to test cleaning methods for Pb in paint dust. The present study also aims to identify differences in Pb and Cd loadings between rooms, thereby providing insight on possible sources. The entry of the home is a prime location to study metals that infiltrate the home from outdoor sources. Similarly, interior rooms such as kitchens, living rooms, and bedrooms are more likely to be influenced by indoor sources of metals. Ultimately, the information presented here will assist in quantifying typical Canadian exposures to Pb and Cd, and reveal ways to reduce exposures.

## **2. MATERIALS AND METHODS**

### **2.1 Selection of Wipe Brand**

Out of the many brands of wipes available on the market, the Ghost Wipes™ brand was selected for use in this study. Based on preliminary testing at Health Canada (Rasmussen, 2007a unpublished data) Ghost Wipes™ were determined to be optimal because they meet all criteria in the American Society for Testing and Materials (ASTM) method E 1792 (2002a); they completely digest in acid

(consistent with previous findings by Harper et al., 2002); and they contain very low background concentrations of the elements in this study. Ghost Wipes™ also meet criteria outlined by Millson et al. (1994) in their evaluation of market available wipes including robustness, high precision, and ease of use.

## 2.2 Sampling and Analysis

A total of 1372 wipe samples were collected from 222 homes between January and March 2008 in three Ontario cities: Barrie (57 homes), Greater Sudbury (86 homes), and Thunder Bay (79 homes). Homes were randomly selected as part of a sampling strategy designed for a larger nation-wide study (Rasmussen et al., 2007b), and therefore the sampling was not intended to be representative of individual cities. The present study incorporates all samples collected in the 2007-2008 sampling season. The collection of information by sampling technicians using questionnaires and interviews, and the communication of results and guidance to the participants, is described elsewhere (Rasmussen, et al. 2007b).

Up to ten wipe samples were collected from each home, from smooth surfaces in the middle of each room. Rooms that were sampled included: main entry, kitchen, living room, family room, adult's bedroom, child's bedroom, and child's primary play area, based on guidance provided by Wilson et al. (2006). Other rooms were occasionally included on an *ad hoc* basis. A total of 932 different rooms were sampled using wipes, yielding 932 individual wipe measurements plus 440 quality assurance measurements (total = 1372 wipes).

Wipe samples were collected according to ASTM E 1728 protocol (2002b), which prescribes a vertical and horizontal overlapping S-shaped movement applying even pressure to the floor surface. The collected wipe was folded inward to preserve the sample and placed directly into a labeled plastic digiPREP™ digestion tube which was sealed inside Ziploc™ bags for transport to the lab. The wipes were shipped to Health Canada, Environmental Health Centre, Ottawa Ontario, Canada, and stored frozen until time of analysis.

The analytical method employed in this study was based on a modification of ASTM method E 1644 (2004), a nitric acid digestion. The modifications consisted of (1) adding hydrofluoric acid during digestion to increase digestion efficiency, (2) multi-element determination by ICP-MS, and (3) incorporating the use of a digiPREP™ heating block. The goal of adding hydrofluoric acid was to quantify total metals in the dust (i.e. maximize recovery). It is noted that the human gastrointestinal tract is estimated to be capable of absorbing less than 30% of the total Pb in house dust (Turner and Ip, 2007).

### 2.3 Quality Assurance and Quality Control

A 12 square inch plastic template was used to constrain the wipe sampling area, as prescribed by the ASTM E 1728 protocol (2002b). The template was cleaned with an alcohol wipe between rooms and a new template was used for each home. In each home sampled, one field blank wipe and one field duplicate wipe were collected. The field blank wipe was exposed to all handling procedures used for the samples with the exception that no surface was wiped (ASTM, 2002b). The room from which the duplicate was collected rotated amongst homes. The sampling strategy yielded a quantity of blanks (n=220) and duplicates (n=220) in excess of the minimum frequency of 5% as outlined in the ASTM E 1728 protocol (2002b), incorporating a high proportion of quality assurance data into the study design.

Throughout sample digestion, three procedural reagent blanks and three procedural wipe blanks were included per batch (n = 37 batches). Three certified reference materials for Pb and Cd were included: NIST 2583 indoor dust, NIST 2584 indoor dust, and NIST 2711 Montana soil (certificates do not include yttrium). The mean recovery and standard deviation of these certified reference materials was  $93 \pm 6\%$  (n=66) for Pb and  $88 \pm 14\%$  for Cd (n=66).

The limits of detection (LOD) and quantification (LOQ) for each element were calculated based on three times and ten times the standard deviation of the lab procedural wipe blanks respectively (n=110). For sample results less than the LOD, half the LOD was substituted where required. The detection limits for Pb, Cd, and yttrium (Y) were calculated to be  $932 \text{ ng m}^{-2}$ ,  $125 \text{ ng m}^{-2}$ , and  $9.09 \text{ ng m}^{-2}$  respectively.

### 2.4 Data Analysis and Units

SPSS® Statistics (version 17.0) and Microsoft Excel® (2007) with the Analyse-it add-in (version 2.20) were used for statistical analyses. Shapiro-Wilk tests of the datasets resulting from this study revealed non-normal distributions, and therefore non-parametric statistical methods were employed. Spatial relations were investigated using Spearman rank correlation coefficients, and 50<sup>th</sup> and 95<sup>th</sup> percentiles were used to summarize Cd and Pb loadings within rooms and within homes.

To convert from SI units ( $\mu\text{g m}^{-2}$ ) used in the present study, to units of  $\mu\text{g ft}^{-2}$  (microgram per sq ft) used in the USA, multiply loading values expressed in  $\mu\text{g m}^{-2}$  by a factor of 0.0929.

### 3. RESULTS AND DISCUSSION

#### 3.1 Wipe Data Quality

Field blank and collocated duplicates were collected from all but two homes in this study (n=220). The LODs, LOQs, and medians of the field blanks are reported for each element in Table 1.

Table 1. Limits of detection (LOD) and limits of quantification (LOQ) for Cd, Pb, and Y are based on 37 analytical batches (three procedural wipe blanks per batch). Results for field wipe values (median) include all field wipe blanks collected in this study (n=220).

	LOD (ng m <sup>-2</sup> )	LOQ (ng m <sup>-2</sup> )	Field Wipe Blank (ng m <sup>-2</sup> )
Cd	125	416	< LOD
Pb	932	3110	< LOD
Y	9.09	30.3	< LOD

The relative percent difference (RPD) between collocated duplicate samples was calculated using the equation  $RPD = ((dup_2 - dup_1) / ((dup_2 + dup_1) / 2)) * 100$ . Note that the mean RPD in the range between LOD and LOQ is greater than the mean RPD above LOQ (Table 2). The greater variability in the lower range (between LOD and LOQ) reflects a combination of field and analytical sources of uncertainty, and points to the need to consider the more rigorous LOQ as the appropriate criterion for quality assurance.

Table 2. Mean Relative Percent Difference (RPD) of collocated duplicates categorized by limits of detection (LOD) and quantification (LOQ). Total number of pairs = 220. See Table 1 for LOD and LOQ values.

	Duplicates < LOD	Between LOD and LOQ		Duplicates > LOQ	
	No. of pairs	No. of pairs	Mean RPD	No. of pairs	Mean RPD
Cd	53	103	51.3	64	39.4
Pb	100	40	71.8	80	41.2

Results for Pb showed that 382 samples were below LOD, which equals 43% of the total of 932 wipe measurements. For Cd, 196 samples were below LOD, or



23% of the wipe measurements. In the case of Y, 34 samples were below LOD (or 3.6% of the measurements). With respect to the LOQ, 511 samples (55%) were below the LOQ for Pb, 672 (72%) were below LOQ for Cd, and 106 (11%) were below LOQ for Y (n=932).

There were a total of 36 homes in which all Pb loading values, in all rooms, were below LOD. In the case of Cd, there were 11 homes in which all samples were below LOD. This yielded a subset of 186 homes for Pb, and 211 homes for Cd, in which at least one wipe sample exceeded LOD. It is this subset of homes which is subjected to further analysis and interpretation in the discussion below.

### **3.2 Lead Loadings**

Figure 1 is a normality (Q-Q) plot of the maximum Pb loading observed in each of the subset of 186 homes having at least one wipe greater than the LOD. Note that the majority of the data fall on the line representing a lognormal distribution, with exceptions occurring at the extreme high and low ends. That portion of the dataset which falls on the lognormal line is considered the “background” subpopulation, for the purpose of this paper. Eight points occur above the breakpoint in the high end of the dataset, where the values start to trend away from the lognormal line (Figure 1). This breakpoint occurs at about  $125 \mu\text{g m}^{-2}$  (or  $12 \mu\text{g ft}^{-2}$ ). Three homes within the elevated subpopulation had wipe samples exceeding the USEPA (2000) regulation for Pb in floor dust, i.e.  $40 \mu\text{g ft}^{-2}$  or  $431 \mu\text{g m}^{-2}$ .

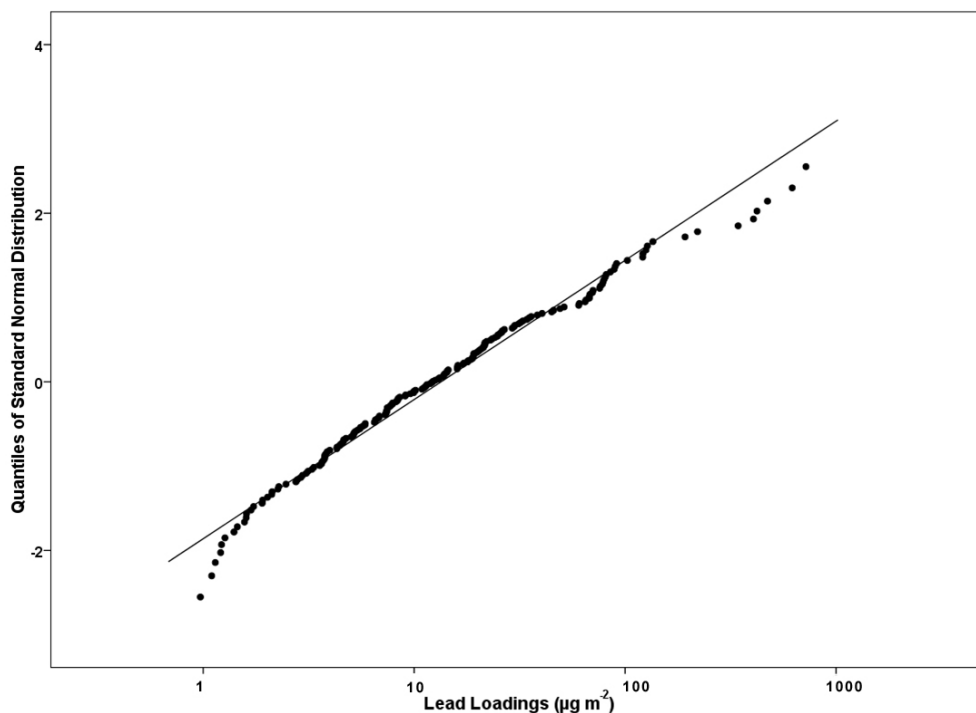
The upper breakpoint in the Q-Q plot at  $125 \mu\text{g m}^{-2}$  (or  $12 \mu\text{g ft}^{-2}$ ) is used herein as an empirical threshold to distinguish between “background” and “elevated” Pb loading subpopulations. This selection of a threshold at about  $12 \mu\text{g ft}^{-2}$  is coincident with a recent US residential study which concluded that Pb loadings less than  $12 \mu\text{g ft}^{-2}$  should be protective for the majority of children (Dixon et al., 2009).

Out of the eight above-threshold homes in Figure 1, three homes displayed the highest Pb loading in the entry way. In two homes the highest Pb loading occurred in an adult bedroom; in two homes the highest Pb loading occurred in children’s bedrooms; and in one home, the highest loading occurred in a child’s play room.

#### **3.2.1 Sources of Lead**

A room by room analysis was conducted with the aim of exploring potential sources of Pb (Table 3). Overall, the entry way displayed the highest median value however other noteworthy rooms include adult bedrooms and children play

rooms. Notably, the highest individual value was located in a child's bedroom. Lead loadings in the home's entry suggest outdoor sources such as track-in of dirt, whereas elevated loadings in bedrooms and play rooms suggest interior sources of Pb. It is concluded from the results shown in Table 3 that both indoor and outdoor sources of Pb contribute to Pb loadings in house dust.



*Figure 1.* Normality Plot (Q-Q plot) for log transformed Pb loadings occurring in a subset of homes having at least one wipe above the limit of detection ( $n=186$ ). Homes are represented by the wipe with the highest Pb loading, regardless of the room from which it was taken. The line through the data represents a lognormal distribution. Eight homes are above the breakpoint in the dataset which occurs at  $125 \mu\text{g m}^{-2}$ .

Table 3. Summary of Pb loadings ( $\mu\text{g m}^{-2}$ ) by room for all 222 homes sampled.  
(LOD =  $0.932 \mu\text{g m}^{-2}$ ).

Room	n	Percent Below LOD	50 <sup>th</sup> Percentile	95 <sup>th</sup> Percentile	Maximum value
Entry	208	22	5.64	87.5	619
Kitchen	218	49	1.02	21.2	165
Living / Family room	114	49	< LOD	40.9	485
Adult Bedroom	93	43	3.26	75.9	422
Child Bedroom	50	60	< LOD	133	720
Play room	23	26	3.50	66.9	220

Age of the home appears to be an important factor, as six out of the eight homes above threshold were built before 1960. The mean age of the eight above-threshold homes is  $1954 \pm 29$  years, which is (on average) nineteen years older than homes in which all wipe samples were below LOD ( $1973 \pm 22$  years;  $n=36$ ). A review of the literature indicates that Pb-based paint is most commonly cited as the primary cause of elevated Pb in older homes (Rasmussen, 2004).

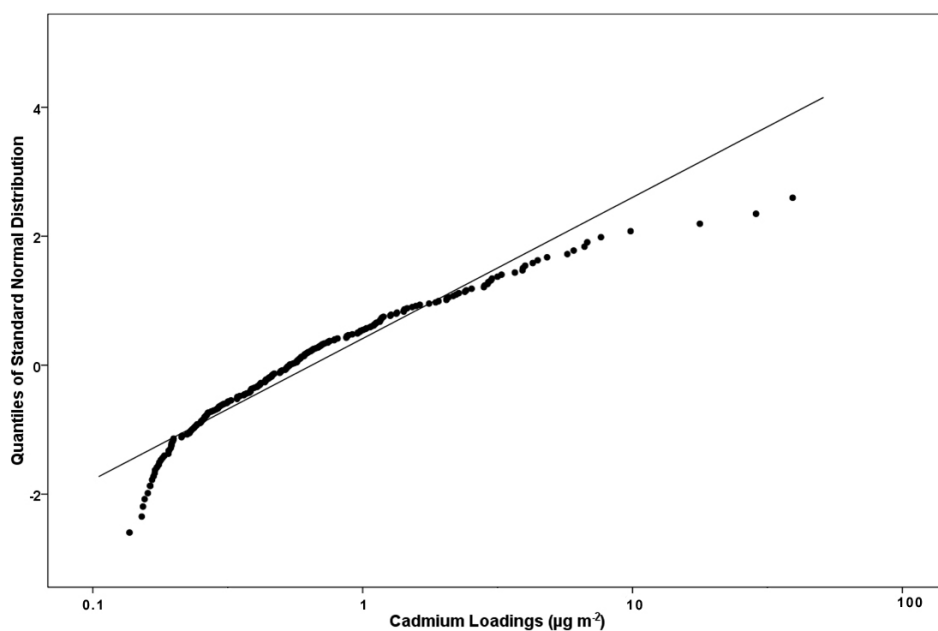
Potential sources of metals in the above-threshold homes may be hypothesized based on questionnaire responses. Residents of two of the above-threshold homes were employed in jobs where Pb may be encountered (mining and shipyards). In one home, Pb is stored in the house for craft and hobby use. A resident of another home habitually conducts bodywork on vehicles in the driveway: the entry wipe for this home displayed both elevated Pb and Cd loadings. Recent renovations involving painting (four homes) and plumbing (one home) were reported to have occurred in some homes with elevated Pb loadings. This may be relevant as renovation activity in older homes can increase Pb availability where high Pb content paint has been used in the past (CMHC, 2009). Sampling technicians noted that old paint was chipping off the wall in one home with elevated Pb loading built before 1960. Two homes with high Pb loadings reported that occupant(s) smoke indoors, which may be relevant as Pb from tobacco use has been indicated as a source of Pb on interior surfaces (Gaitens et al., 2009).

### 3.3 Cadmium Loadings

A normality (Q-Q) plot for Cd loadings (Figure 2) indicated a breakpoint in the dataset at about  $4.4 \mu\text{g m}^{-2}$  ( $0.4 \mu\text{g ft}^{-2}$ ). There were nine homes in this study with Cd loadings above this threshold value: three of these were kitchen wipes, three were entry wipes, two were office wipes, and one was from an adult bedroom.

### 3.3.1 Sources of Cadmium

As in the case of Pb, there are both indoor and outdoor sources of Cd. Cadmium loadings are relatively high in home entry ways and adult bedrooms compared to other areas of the home (Table 4). As exterior sources of Cd are associated with both industrial land use and geological sources, track-in of dirt by residents and their pets is a plausible explanation for the observation of relatively high loadings in entry ways. Higher Cd loadings in adult bedrooms versus child occupied rooms suggest tobacco use as a possible source. Questionnaire data indicated that 14% of homes in the study were occupied by at least one person who smoked inside the home. House age may also be a factor: but the dataset is too small to determine significance: the average age of homes with Cd loadings above threshold was  $1962 \pm 29$  years ( $n=9$ ), compared to homes where all wipe samples were below LOD ( $1975 \pm 23$  years;  $n=11$ ).



*Figure 2.* Normality Plot (Q-Q plot) for log transformed Cd loadings occurring in homes having at least one wipe above the limit of detection ( $n=211$ ). Homes are represented by the wipe with the highest Cd loading, regardless of which room it was collected from. The line through the data represents a lognormal distribution. Nine homes occur above the breakpoint in the dataset which occurs at  $4.4 \mu\text{g m}^{-2}$ .

Information gathered from the questionnaire results revealed clues about possible sources in the nine homes with above-threshold Cd loadings. In three homes where painting was listed as a hobby, elevated Cd loadings were observed

in the room where this hobby was conducted. Crafts and hobbies are known to have the potential to influence metal loadings in the home (Rasmussen, 2004), as Cd is a common component of certain paint pigments (Harte et al., 1991). In four other homes activities were reported that involve metal work such as welding, soldering, and body-work on vehicles. These activities are potential sources, as Cd is a common component of solders and fluxes (Harte et al., 1991). Of the two remaining elevated homes, one contained a resident who smokes and one had recent plumbing and painting renovations. Five of these nine homes above threshold were built before 1960.

*Table 4.* Summary of Cd loadings ( $\mu\text{g m}^{-2}$ ) by room for all 222 homes sampled (LOD=  $0.125 \mu\text{g m}^{-2}$ ).

<b>Room</b>	<b>n</b>	<b>Percent Below LOD</b>	<b>50<sup>th</sup> Percentile</b>	<b>95<sup>th</sup> Percentile</b>	<b>Maximum value</b>
Entry	208	16	0.314	3.11	8.14
Kitchen	218	28	0.207	1.24	9.84
Living / Family room	114	20	0.239	1.20	5.66
Adult Bedroom	93	22	0.276	2.74	19.0
Child Bedroom	50	28	0.199	1.68	3.27
Play room	23	17	0.320	1.06	1.63

### 3.4 Metal Correlations

Spearman correlation coefficients ( $r_s$ ) were calculated to compare Cd, Pb, and Y loadings for various rooms within the home. With regard to the entry wipe samples ( $n=208$ ; Table 5) correlations between Pb and other metals were greater than  $r_s = 0.5$ , which are strong relationships according to definitions by Reimann et al. (2008). No significant differences were observed for analyses of individual locales compared to analyses of the entire dataset. Correlations were similarly strong for bedroom and other interior wipe samples (i.e. kitchens, living rooms, bedrooms, and playrooms; Table 5). Since all correlations were strong, information about precise sources of these metals could not be determined from this type of analysis.

Calabrese and Stanek (1995) recommended Y as a soil tracer for use in the estimation of soil ingestion rates. Yttrium is a rare earth element with an average concentration of 30 ppm in the earth's crust (Bottrill, 2001). The comparison of Y

against Cd, and Pb in wipe samples was included in Table 5 in an effort to identify the relative contribution of outdoor and indoor sources of these metals in different areas of the home (Table 5). The highest proportion of soil is likely to be found in dust samples collected in the home's entry areas, due to track – in of outdoor dirt by residents and their pets.

The results in Table 5 are inconclusive as to whether indoor or outdoor sources dominate. Strong Pb – Y correlations ( $r_s = 0.7$ ) and strong Cd – Y correlations ( $r_s = 0.6$ ) are found for wipes collected in entry ways (Table 5). However, correlations are equally strong for wipes collected in bedrooms and other interior rooms (Pb-Y  $r_s = 0.6$ , Cd-Y  $r_s = 0.6$ ). These results suggest that both indoor and outdoor sources exist for all three elements.

Yttrium has limited use in household products: it is generally alloyed in small amounts with other metals, and is most commonly found as the oxide yttria ( $Y_2O_3$ ), used for making red phosphors in colour television picture tubes (Chemistry Encyclopedia 2007; Bottrill 2001). In summary it appears that Pb, Cd, and Y are contributed to house dust from both indoor and outdoor sources, based on the correlations in Table 5 observed in all entry and interior subsets.

Table 5. Spearman rank correlation coefficients for Cd, Pb, and Y sub-divided by room. "Interior room wipes" include living rooms, kitchens, bedrooms, and play rooms.

Location	n	Cd-Y	Pb-Cd	Pb-Y
Entry wipes	208	0.6	0.7	0.7
Bedroom wipes	189	0.6	0.7	0.6
Interior room wipes	575	0.6	0.6	0.6

#### 4. CONCLUSIONS

This research has generated the first multi-element wipe sampling database for background or baseline urban residential environments in Canada. The information obtained by applying the wipe methodology to Canadian residential environments assists in quantifying typical urban residential exposures to Pb and Cd, and reveals valuable information about variations in metal loadings amongst individual rooms within homes.

The results indicate that activities conducted in each room and the products used within them contribute to the metal level of that room. Similarly, outdoor sources contribute to differences in metal loadings of homes due to track-in of outdoor dirt by residents and their pets. The finding of strong correlations for Y against both Pb and Cd in entry areas as well as interior areas confirms that both indoor and outdoor sources are important for these three elements.

Ninety-nine percent of homes in this study fell below the USEPA regulation of  $40 \mu\text{g ft}^{-2}$  ( $431 \mu\text{g m}^{-2}$ ) for Pb in floor dust (USEPA, 2000). The finding that only a small percentage of homes had a wipe sample that exceeded the USEPA regulation (3 out of 222 in total) is consistent with US residential studies such as NHANES (National Health and Nutrition Examination Survey) which reported that the geometric mean of Pb in floor dust was  $1.1 \mu\text{g ft}^{-2}$  ( $12 \mu\text{g m}^{-2}$ ; Dixon et al., 2009). In the present study, analysis of the maximum wipe loading per home using Q-Q plots suggested thresholds of  $125 \mu\text{g m}^{-2}$  for Pb and  $4.4 \mu\text{g m}^{-2}$  for Cd, which are used to distinguish between background and elevated subpopulations. This dataset will contribute to the development of guidance for reducing exposures to residential Pb and Cd that is specific to the Canadian urban environment.

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