**Preliminary Draft Report** 

# A Comparison of Five Sampling Methods for Settled Lead Dust: A Pilot Study

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# **Table Of Contents**

			Page
List of Tables		i	i
Introduction		1	L
Methods		3	3
Results		e	5
Conclusions		2	27
Recommendations		2	28
Acknowledgements		3	30
Appendices		3	31
Appendix A Appendix B Appendix C	Sampling and Analytical Methods Raw Data Wipe Sampling Recovery Data		

# List of Tables

- CC

		Daga
Table 1	Sampling Design	Page 5
Table 7.	Summary of Performance Characteristics	10
Table 2.	Summary Statistics for Manufacturistics	10
Table 5.	Jummary Statistics for Measurement of Lead Loading	11
<b>T</b> 11 4	Using Four Methods $\dots \dots \dots$	11
Table 4.	Statistics for Measurement of PDD Loading ( $\mu g/It^2$ )	10
	by Bioavailable Lead Wipe Method	12
Table 5.	Statistics for Measurement of PbD Loading ( $\mu g/ft^2$ )	10
	by Total Lead Wipe Method	12
Table 6.	Statistics for Measurement of PbD Loading ( $\mu g/ft^2$ )	10
	by DVM Method Using All Samples	13
Table 7.	Statistics for Measurement of PbD Loading ( $\mu g/ft^2$ ) by HVS Method	
	Using All Samples	13
Table 8.	Statistics for Measurement of PbD Loading ( $\mu g/ft^2$ ) by DVM Method	
	Using Sample Weights Greater Than 2 mg	14
Table 9.	Statistics for Measurement of PbD Loading ( $\mu g/ft^2$ ) by HVS Method	
	Using Sample Weights Greater Than 2 mg	14
Table 10.	Statistics for Measurement of PbD Concentration (ppm) by	
	DVM Method Sample Weights Greater than 2 mg	15
Table 11.	Statistics for Measurement of PbD Concentration (ppm) by	
	HVS Method Sample Weights Greater than 2 mg	15
Table 12.	Summary of Method Differences for All Surface Types for	
	Log-Transformed Loadings	17
Table 13.	Regression Models and Residual Variance by Sampling Method	18
Table 14.	Nested Factorial ANOVA for Log-Transformed Loadings	
	for Differences Among Sampling Methods Using All Data	18
Table 15	Residual Variances for Each Method's Loadings from the	
1000 15.	Nested Factorial Model	19
Table 16	Residual Variance for the DVM Method after Dividing the	
14010 10.	Samples into High and Low Values	20
Table 17	ANOVA For Method and Surface Type Effects by House on	20
	Log Transformed PhD Logdings	21
Table 18	Summary Statistics for the Measurement of Lead Dust Concentration	21
14010 10.	by Vacuum Methods (Evoluting Samples < 2 mg)	22
Table 10	Nosted Easterial ANOVA for Log transformed PhD	
Table 19.	Concentration (nnm) for Vacuum Sampling Methods	22
T-11-00	Concentration (ppin) for vacuum Sampling Methods	23
Table 20.	Nested Factorial ANOVA Limited to Method, House and Surface Type	22
m 11 01	Effects for Log-Transformed PDD Concentrations (ppm)	23
Table 21.	Summary of Surface Differences for Log-Transformed Concentrations	24
Table 22.	Correlations among Log-Transformed PbD Loading and Concentration	25
	Collected at All Locations	25
Table 23.	Correlations Between Methods By Surface Types	26

# Introduction

#### Purpose

The purpose of this pilot study is to determine relative collection efficiencies and the reproducibility of five settled dust sampling methods. The National Center for Lead-Safe Housing (the Center) is funding this study for two reasons:

- 1. The Environmental Protection Agency requires assistance in interpreting data from a number of previous studies examining the lead dust/blood lead relationship. Unfortunately, these studies used a variety of lead dust sampling methods with differing collection efficiencies, making it difficult to compare results. A study comparing relative collection efficiencies of different sampling methods may help the agency interpret previous findings, and thus inform the agency's proposed standard for establishing "dangerous" levels of settled lead dust.
- 2. With funding from the Department of Housing and Urban Development, the Center and the University of Rochester will be directing a large, carefully controlled study of the blood lead/dust lead relationship later this summer in 200-400 dwellings in Rochester, NY. It is likely that this larger study can evaluate only one wipe sampling method and one vacuum method. To conduct the larger study, the Center needs to decide which two sampling methods should be used. The Center requires assistance and data to inform that decision.

Relative collection efficiencies and estimates of reproducibility could conceivably have been studied in a laboratory setting. There is certainly much utility in studying collection efficiencies in a setting where confounding influences can be eliminated or carefully controlled. However, it is unlikely that surfaces spiked with known amounts of lead dust would actually reflect realworld conditions, where lead dust is often ground into surfaces or embedded in porous substrates like carpet or concrete. In order to retain real-world characteristics, this study assumes that levels of settled lead dust in adjacent areas in the same rooms are equal and that the only variability encountered is due to sampling method. Duplicate samples were collected in each room to estimate the reproducibility of each method.

This study is not designed to determine <u>absolute</u> collection efficiencies from surfaces spiked with known amounts of lead dust. Instead, this study attempts to determine relative collection efficiencies with an eye toward selecting two methods (one wipe and one vacuum) that are practical, informative, and cost-effective.

# Criteria For Settled Lead Dust Sampling Methods

Previous research has demonstrated a clear relationship between levels of settled lead dust and lead poisoning in children. These and other studies have employed a variety of sampling methods to attempt to characterize settled lead dust levels and to relate those levels to blood lead levels of resident children. Since there are many factors other than lead dust that determine a child's blood lead level, establishing a sampling method that can predict the relationship between lead dust and blood lead is a difficult proposition. Historically, research studies have employed a variety of techniques to capture the dust that is thought to contribute most to blood lead levels.

There is still no consensus on how to measure exposures to lead in dust. Broadly speaking, such exposure measurements would need to consider particle size, ease of liberation from various environmental matrices, location, and chemical speciation. Except for location, none of these are easily measured through routine analysis.

The nation is about to embark on sampling of settled lead dust in literally millions of dwellings to characterize immediate risks and help focus control efforts. In order to make this a reality, lead dust sampling methods must be:

- Sufficiently descriptive of lead contamination on a variety of surface types to permit responses that are based on real hazards (i.e., blood lead level);
- Adequately reproducible on different surface types so that different individuals collecting the same samples on the same types of surfaces find similar results;
- Relatively inexpensive, with only a modest investment in equipment, supplies, and laboratory analytical costs;
- Relatively simple to practice, so that with a modest degree of training, dust sampling can be implemented widely; and
- Lightweight and portable
- Able to be completed in a short time in the field.

#### Types of Sampling Methods

This report provides field and laboratory observations of the ease of use, analysis, and speed of five settled lead dust sampling methods. The report also provides a very preliminary statistical analysis of the relationship of the different sampling methods to each other by type of method, type of surface, level of contamination, and by unit of measure (i.e., loading ( $\mu$ g/ft<sup>2</sup>) and concentration (ppm)). Of course, concentration data are reported for vacuum samples only.

It is beyond the scope of this study to determine which method is most biologically significant, i.e., which one best characterizes blood lead levels. Although blood lead data from health department records were collected for this study, it is likely that there were serious confounding influences. These include extensive cleaning with HEPA vacuums and the variable times at which blood lead levels were determined (2 - 12 months prior to dust sampling). The larger study for which this pilot was conducted will examine the question of the relationship between blood lead and dust lead levels.

# Methods

This pilot study was completed within a time span of three months in order to meet certain deadlines for both the EPA standard-setting process, defined by Title X, and the full Rochester study schedule.

# Sampling Strategy

One diaper wipe sample, one tared wet wipe sample, and two vacuum samples were collected on a side-by-side basis from four adjacent areas on floors. The diaper wipe sample was digested twice using a "bioavailable" weak acid digestion technique, followed by a "total" strong acid digestion technique. This produced a total of five sampling methods.

A grid with each sampling area equalling approximately one square foot was constructed to delineate exact sampling areas. Each grid was therefore equal to four square feet, with 4 adjacent areas of 1 square foot. The exact area within each grid used for each sample was randomly selected. The grid was located at the midpoint of 3 rooms in each house. An additional grid was located on a smooth concrete surface on the interior or exterior of the house, such as the front porch, walkway, or driveway (if such a surface is available). Rough concrete was not sampled.

One grid area was sampled using "Little Ones" diaper wipes, available from K-Mart. This wipe sample was analyzed using two digestion procedures. The first was the cold hydrochloric acid digestion (so-called "bioavailable") procedure used at the Kennedy Krieger Institute in Baltimore, Maryland. The second was analyzed using the hot nitric acid/peroxide digestion method (socalled "total" lead) typically used in HUD-related work. Dust wipes with known amounts of lead dust were inserted blindly into the sample stream at a rate of one per twenty samples submitted to determine if there was significant sample loss caused by this second digestion of wipe samples.

A second grid area was sampled using the tared wet wipe procedure developed at the University of Medicine and Dentistry of New Jersey. The statistical data for this sampling method are not included in this report, since laboratory analysis has not yet been completed.

A third grid area was sampled using the University of Cincinnati's dust vacuum method (DVM), which involves the use of a personal air sampling pump operating at a nominal flowrate of 2.5 liters/min fitted with Tygon tubing. The tubing was connected to a 37 mm cassette housing a mixed cellulose ester filter with a pore size of  $0.8 \mu$ . The inlet of the cassette was fitted with a specially-made nozzle. Blind spike samples were also inserted into the sample stream for this analysis.

The fourth grid area was sampled using the High Volume Small Surface Sampler (HVS-3) vacuum sampler, which uses a cyclone and a higher flowrate (estimated at 16 liters per minute). Dust samples were collected in tared teflon vessels, which were subsequently weighed. The dust was transferred quantitatively to another vessel and shipped to the laboratory for analysis. Blind spiked samples with known amounts of lead dust were inserted into the sampling stream to determine if this transfer was accomplished quantitatively. The procedure for this type of sampling followed the protocol established by the Kennedy-Krieger Institute in Baltimore.

In order to estimate reproducibility, a second set of four 1 square foot grids were placed adjacent to the first four 1 square foot grids and sampled in exactly the same fashion. Thus, each method was used twice in the same room on the same surface in the same house.

The complete sampling protocol for each method and the field sampling manual is provided in Appendix A. This includes the random selection grid assignment method for each method.

Twenty dwelling units in Butte, Montana were selected by a research group at the University Environmental Health Foundation (associated with the University of Cincinnati), based on previous sampling that indicated significant lead dust levels were present. These twenty dwellings exhibited a mix of different floor surface types, including smooth vinyl, wood, low pile carpet, high pile carpet, and concrete.

Table 1 summarizes the sampling design for this study.

Description	Number
Dwellings	20
Sampling Methods	5ª
Sampling Locations at Each Dwelling	4 (3 interior floors and 1 exterior surface)
Duplicate Samples/Room	2
Total	800 <sup>b</sup>

Table 1. Sampling Design

<sup>a</sup> New Jersey method not included in this report.

<sup>b</sup> Only 640 samples are considered in this report, since the New Jersey method results are not yet available.

#### Laboratory Analysis

Except for the New Jersey sampling method, all analysis was performed by Azimuth Laboratories, an AIHA-accredited laboratory that participates in the Environmental Lead Laboratory Proficiency Analysis Test program. This laboratory has extensive experience in lead analysis, but is not a research-type laboratory. Such a laboratory was selected to determine if the analysis for each procedure was feasible for a commercial laboratory. The New Jersey dust wipe samples were analyzed in the New Jersey laboratory where the method was developed, since this analysis is quite unusual.

Initially, all samples were to be analyzed by flame atomic absorption spectroscopy or inductively-coupled plasma emission spectroscopy in order to determine if the analysis could be completed inexpensively. Analysis by graphite furnace atomic absorption was originally not permitted, since such analysis is probably too costly to implement on a national scale. However, many of the samples in this study were below the limit of detection for flame AA and had to be reanalyzed by the more sensitive graphite furnace AA, since many dust lead levels were lower than anticipated.

Laboratory personnel provided observations on the ease of each analytical method.

# Data Analysis

Summary statistics of central tendency and variance were calculated for each sampling method on each surface type. A four factor nested factorial analysis of variance (ANOVA) was used to analyze the loading and concentration data. For vacuum samples, separate analyses were performed, first using all samples, then using only those samples that weighed more than 2 mg (total dust) to determine if the prevalence of low weight samples in this studies affected the results. Both the loading and concentration data were transformed to their natural logarithm to approximately normalize the statistical distributions. The four factors used in the ANOVA were:

- 1. Sampling Method
- 2. House in which the samples were collected
- 3. Locations within the house
- 4. Locations within the same room

Regression equations were modelled for each sampling method and correlation coefficients were calculated.

At this writing, a full statistical analysis of the data has not been completed. The results presented here are <u>preliminary</u>, and may be modified somewhat following further review.

Data from the New Jersey method are not yet available and are not included in the statistical analysis.

#### Results

### Field Observations

Each of the five sampling methods were practiced by Center staff in the field. University Environmental Health Foundation of Cincinnati personnel also reported observations regarding feasibility of each of the techniques.

1. Wipe Sampling

The wipe sampling protocol in this study is difficult to implement on rough or carpeted surfaces. Typically, the wipe will curl, making it difficult to achieve complete contact with the surface. The diaper wipes are not manufactured from analytical grade materials, making consistency of materials uncertain.

Differing pressures and slightly different wiping styles may be practiced by different technicians, possibly resulting in different recovery rates. It was also reported that the moisture in the wipes was somewhat variable, with the last wipes in a container having noticeably higher levels of moisture content.

Analytical costs for this method were approximately \$12/sample for this study. Results can be reported in loading only.

2. University of Cincinnati Dust Vacuum Method (DVM)

The plastic nozzle, which consists of a round plastic tube crimped into a small slot at one end became blocked with dust on several occasions, especially on vinyl floors. Although some of this dust could be sucked onto the filter by using toothpicks or briefly placing a gloved finger over the opening to increase the flowrate and force the dust through the slot, some sample loss appeared inevitable. It is worth noting that previous versions of this sampling method employed round tubing that is less likely to become blocked, although the return to round tubing could mean that the time period required for sampling the surface would be lengthened slightly, since the slot opening is wider than the round tube.

Another deficiency of this method involves the difficulty in using sampling strokes that do not overlap and that do not miss areas. There is no way to consistently guide the sampling inlet so that the entire surface is covered. A sampling template with guide wires could solve this problem.

The plastic nozzles are not commercially available at this time; they are prepared by heating plastic tubing and forming to a jig and are manufactured by a research group at the University of Cincinnati.

Analytical costs were about \$20 per sample for this study. The method is capable of reporting both loading and concentration.

## 3. HVS-3 Sampling

The HVS-3 sampler used in this study is a modified version developed at the Kennedy-Krieger Institute in Baltimore. It employs a Dirt Devil mini-vacuum cleaner that requires AC current, which in some cases will mean use of an generator. This reduces the portability of the device. The flowrate for the vacuum cleaner is not specified, and is not determined for each application. However, researchers at the Kennedy-Krieger Institute report the flowrate with the cyclone in line is approximately 16 liters/minute. It is likely that the flowrate changes somewhat on surfaces presenting differing pressure drops (e.g., carpeting vs. vinyl floors). On the other hand, the DVM uses a constant flow pump that can handle a pressure drop of up to 20 inches water gauge.

Although the sampling inlet is larger, the HVS-3 method shares the same problem of overlapping sampling strokes, or missing some areas shown by the DVM.

The cyclone used in the HVS-3 method is a non-standard item that is not widely available at this time. In addition, the cyclone has to be broken down and cleaned after each sample was collected, a process that requires 5-10 minutes. There are some difficult-to-reach areas that can only be cleaned by a thorough washing that is not feasible in the field.

The exterior of the tared microwave digestion tubes must be kept clean of hand oil and other material so that the final weight reflects only the captured dust. This means that it is necessary to double bag the tubes and never place them on surfaces which could contaminate them.

There remains some concern that the cyclone does not capture small particulate. This could affect the device's ability to capture lead dust in the size range of interest. Smaller particles have been shown to be related to increased lead uptake rates, and lead concentrations are typically higher in small particles.

Analytical costs for this method were approximately \$10/sample for this study. The method is capable of reporting both loading and concentration.

4. New Jersey Tared Wet Wipe Method

The tared sampling media, which consisted of three small rectangular wipes about 3.8 cm x 6.4 cm, had to be mounted on a pressure block with tweezers. This proved to be quite difficult in the field. There appeared to be significant danger of media contamination from handling. Since the aluminum foil housing the filter media is included in the weighing process, there is concern that the outside of the aluminum could be contaminated. A clean plastic sheet or piece of aluminum foil was used to prepare each sample, but it was still usually necessary to touch the aluminum foil with a gloved finger.

On some warped linoleum and wood flooring, the block clearly did not make contact with the entire surface wiped. Also, grains of sand or other minute objects on the floor prevented the block from making contact with the surface. The template into which the block fits is designed to standardize the pressure applied to the surface. However, the template appears to be too wide to fit in some window wells.

Some sample loss during several steps in the protocol was identified visually in the field. When the filter media are removed from the block and placed in the aluminum, some visible dust remains on the block. It seems to be virtually impossible to prevent some dripping and sample loss when deionized water is added. After the new filter is applied, the application of water may also result in sample loss, especially since the excess water is shaken off. Finally, it appeared to be impossible to avoid touching the part of the filter media with the dust on it with the tweezers--some of the sample (probably very little) may have been lost to the tweezers.

There is nothing in the protocol about cleaning the block and template. Wet diaper wipes were used, but it is not known whether the deposition of oils from the wipe could affect the tare weight. More detailed instructions on cleaning should be provided.

The sampling also took about 10-15 minutes per spot sampled, the longest of any of the sampling methods employed here.

Analytical costs were about \$40/sample for this study. The method is capable of reporting both loading and concentration.

#### Laboratory Observations

1. Cincinnati DVM Vacuum

The analytical laboratory reported that many samples contained very low levels of total dust. Since the method involves transfer of dust from the filter and the filter cassette by rinsing into a tared beaker, followed by drying and reweighing, the detection limit was limited by the relatively small amount of dust compared to the large total weight of the beaker. The laboratory suggested the use of a tared PVC filter instead of a MCEF filter (which is hygroscopic and cannot be tared). However, this does not solve the problem of loose material and dust retention by the filter cassette.

# 2. HVS-3

Since this is a bulk sample, the laboratory reported no difficulties in analysis, since this is a relatively routine process.

#### 3. New Jersey Sampler

No laboratory observations are available for this preliminary report.

Performance Characteristic	Total Lead Wipe	Bioavailable Lead Wipe	Tared Wet Wipe (New Jersey)	DVM	HVS-3
Time/Sample Collected, including cleaning (minutes)	1	1	10-15	4	10-15
Portability	High	High	High	High	Low
Laboratory Analysis	Simple	Complex	Complex	Simple	Simple
Cost/sample for Flame AA in this study	\$12	\$24	\$40	\$25	\$10ª
Cross-sample contamination potential	Moderate	Moderate	High	Low	Moderate
Visual Performance on Warped or Rough Surfaces	Poor on rough surfaces or carpet	Poor on rough surfaces or carpet	Poor on warped or rough surfaces	Acceptable	Acceptable

 Table 2.

 Summary of Performance Characteristics

<sup>a</sup> The laboratory was not responsible for weighing, acid washing, drying, and reweighing the microwave digestion collection vessel. This would add perhaps another \$5-\$7 to the cost of analysis.

#### Data Analysis

Table 3 shows summary measures of central tendency and variance for all methods except the New Jersey method. Of the 800 samples proposed in the original design, 160 New Jersey samples are not yet available. Of the remaining 640 samples, 24 exterior dust samples could not be collected due to house characteristics and two HVS-3 samples were destroyed during laboratory analysis. This resulted in a total of 614 samples analyzed for this study. Raw data for each house is included in Appendix B.

Method	N <sup>a</sup>	Mean (µg/ft²)	Standard Deviation (µg/ft <sup>2</sup> )	Geometric Mean (µg/ft <sup>2</sup> )	Geometric Standard Deviation $(\mu g/ft^2)$	Upper 95 % Confid ence Interval	Lower 95% Confid ence Interval
Bioavail able Wipe	154	44.4	115	11.1	4.68	232	1.1
Total Wipe	154	111.4	386	15.1	5.61	641	1.4
DVM	154	41.9	98.2	6.8	8.83	227	0.2
HVS-3	152 <sup>b</sup>	1492	6514	132	11.65	5562	1.2

Table 3.Summary Statistics for Measurement of Lead Loading Using Four Methods

<sup>a</sup> Houses 1, 12, and 19 were sampled in only 3 rooms, not four (i.e., no exterior dust samples could be collected). Therefore, of the 640 samples proposed in the design, 24 were not collected (3 houses x 4 methods x 2 samples = 24). This results in a loss of 6 samples for each method, yielding 154 samples per method (160 - 6 = 154)

<sup>b</sup> Two HVS-3 samples were lost due to lab error.

# Loading

Table 3 shows that overall, the loadings were highest for the HVS-3 and lowest for the DVM. The bioavailable lead wipe collected less lead than did the total lead wipe. Using the geometric means, both wipe methods collected about twice as much lead as did the DVM, while the HVS-3 collected nearly nine times as much lead as the wipe.

However, performance of each sampling method varied to some extent by surface type. Of special interest is carpets, where wipe samples were much lower than the DVM and HVS-3. For low-pile carpets, loadings for the DVM samples were almost four times higher than those from the wipes and about twice as high for high-pile carpets. Average loadings using the HVS-3 method were about 30 to 70 times higher than the DVM on the low- and high-pile carpets, respectively. On vinyl surfaces, the total lead wipe method recovered significantly more lead than did either vacuum method.

Performance of each sampling method by surface type is shown in Tables 4-11. Two separate analyses were performed for the DVM and HVS-3 samplers to determine if the prevalence of low sample weights resulted in a difference in the variability. The first analysis used all

samples, while the second analysis excluded all samples with total weights less than 2 mg. A summary table of the relative performance of each method by loading for each surface type appears as Table 12.

# Table 4.

# Statistics for Measurement of PBD Loading ( $\mu g/ft^2$ ) by Bioavailable Wipe Method Using All Samples

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	28	13.1	14.3	8.6	2.50	109.2	55.7	2.0
Wood	26	22.2	35.5	13.7	2.33	160.2	135.9	5.4
L-Carpet	60	6.8	11.9	4.2	2.53	174.1	13.9	1.0
H-Carpet	10	6.2	4.7	3.9	3.45	75.9	15.0	0.4
Concrete	28	137.7	119.8	99.6	2.25	87.0	439.1	29.9
Other	2	807.0	196.6	795	1.28	24.4	946.0	668

# Table 5.

# Statistics for Measurement of PbD Loading (µg/ft<sup>2</sup>) by Total Pb Wipe Method Using All Samples

Surface	N	Mean	S.D.	GM	GSD	C.V.	95 %	5%
Vinyl	28	19.0	20.8	11.9	2.70	109.2	74.6	1.6
Wood	26	53.2	164.8	16.8	3.21	310.1	581.3	5.1
L-Carpet	60	8.0	12.0	5.2	2.26	151.4	22.2	1.3
H-Carpet	10	4.6	2.4	3.8	2.11	51.6	7.8	0.9
Concrete	28	364.1	571.7	194	2.82	157.0	2066.5	45.5
Other	2	2265	1056	2138	1.62	45.6	3012	1518

# Table 6.

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	28	2.7	6.2	0.8	4.95	233.0	21.6	0.1
Wood	26	7.7	14.0	1.5	7.88	180.6	50.0	0.1
L-Carpet	60	56.5	110.7	15.3	5.42	195.9	303.7	1.3
H-Carpet	10	9.5	7.8	7.5	2.08	81.8	30.0	2.4
Concrete	28	70.0	101.1	33.2	3.72	144.5	392.9	2.2
Other	2	361.5	381.1	241	3.90	105.4	631.0	92

# Statistics for Measurement of PbD Loading (µg/ft<sup>2</sup>) by DVM Method Using All Samples

# Table 7.

Statistics for Measurement of PbD Loading (µg/ft<sup>2</sup>) by HVS Method Using All Samples

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	28	9.7	11.0	5.0	3.66	112.9	38.5	0.53
Wood	26	503.5	1600	34.3	11.30	317.7	6139	0.8
L-Carpet	59	1403	3518	453.0	3.71	250.8	5640	64.0
H-Carpet	10	966.1	1035	504.0	4.02	107.2	3520	47.0
Concrete	27	1630	4268	413.0	4.40	261.9	16368	39.6
Other	2	38525	48048	18162	7.08	124.7	72500	4550

# Table 8.

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	19	3.9	7.3	1.7	3.40	190.3	33.0	0.2
Wood	16	9.4	13.6	3.3	5.24	145.7	51.0	0.2
L-Carpet	58	58.5	112.2	16.8	5.13	191.9	315.8	1.5
H-Carpet	10	9.5	7.8	7.5	2.08	81.8	30.0	2.4
Concrete	28	70.0	101.1	33.2	3.72	114.5	392.9	2.2
Other	2	361.5	381.1	241	3.90	105.4	631.0	92.0

# Statistics for Measurement of PbD Loading (µg/ft<sup>2</sup>) by DVM Method Sample Weights Greater Than 2 mg

# Table 9.

Statistics for Measurement of PbD Loading (µg/ft<sup>2</sup>) by HVS Method Sample Weights Greater Than 2 mg

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	25	10.5	11.3	5.5	3.65	108.0	41	0.5
Wood	25	523.6	1629	39.7	10.55	311.2	6372	1.4
L-Carpet	59	1402	3518	453.0	3.71	250.8	5640	64.0
H-Carpet	10	966.1	1035	504.0	4.02	107.2	3520	47.0
Concrete	26	1674	4347	411.0	4.53	260.0	16935	39.4
Other	2	38525	48048	18162	7.08	124.7	72500	4550

# Table 10.

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	19	727.9	1598	317	2.97	219.6	7173.9	84.3
Wood	16	882.5	1002	527	2.96	113.5	4909.9	76.4
L-Carpet	58	1224.0	5294	417	2.79	432.4	2369.3	83.5
H-Carpet	10	319.2	179.5	267	1.95	56.2	598.6	86.3
Concrete	28	1056.0	1006	740	2.32	95.2	3752.5	234
Other	2	1893.0	725.7	1822	1.48	38.3	2405.6	1379

# Statistics for Measurement of PbD Concentration (ppm) by DVM Method Sample Weights Greater Than 2 mg

# Table 11.

# Statistics for Measurement of PbD Concentration (ppm) by HVS Method Sample Weights Greater Than 2 mg

Surface	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
Vinyl	25	239.60	266.6	122	4.08	111.2	1030.5	4.7
Wood	25	575.0	817.4	303	3.10	142.2	3172.4	48.9
L-Carpet	59	579.4	424.5	440	2.20	73.3	1712.5	102
H-Carpet	10	561.6	507.6	365	2.85	90.4	1736.9	86.2
Concrete	26	1284	1781	659	3.10	138.7	6246.7	94.9
Other	2	108417	143951	37328	11.5	132.8	210206	6629



Figure 1

Ln(HVS) ug/m<sup>2</sup>

16

Figure 1 shows how each of the methods performed on each surface type, and then compares the results to the HUD clearance standards. The HUD clearance standards were intended to be used for wipe sampling <u>only</u>, not for vacuum methods. For the wipe method (total digestion), 8 out of 77 floors were above 200  $\mu$ g/ft<sup>2</sup>; for the bioavailable digestion 5 out of 77 floors were greater than 200  $\mu$ g/ft<sup>2</sup>. For the DVM method, 5 of 77 floors were greater than 200  $\mu$ g/ft<sup>2</sup>, which is similar to the two wipe methods. However, for the DVM method, 45 of 77 floors exceeded the clearance standard. This demonstrates the importance of setting a clearance standard related to a specific sampling method

Table 12 is a summery of the performance of each method in order of capture efficiency for each surface type based on the log-transformed loadings.

Summ	ary of Method Differences for All Surface Types For Log-transformed Loadings
Surface	Methods in Order of Capture Efficiency By Loading
Vinyl	Total, Bioavailable > HVS, DVM
Wood	HVS > Total, Bioavailable, DVM
Low-Carpet	HVS > DVM > Total, Bioavailable
High-Carpet	HVS > DVM > Bioavailable, Total
Concrete	HVS > Total, Bioavailable, DVM
Other	HVS > Total, Bioavailable, DVM

Best fit regression equations were developed to compare the total lead wipe and the two vacuum methods; the residual variance for each was calculated. The equations should prove helpful to EPA in attempting to compare the results of the sampling methods employed in earlier studies to other sampling methods. These models are shown in Table 13. They show that the DVM results are more variable than the total wipe and HVS methods (no regression equation was developed for the bioavailable wipe method).

			Table	13.			
Regression	Models	and	Residual	Variance	by	Sampling	Method

Regression Equation	Residual Variance <sup>a</sup>
Ln DVM = 0.55 Ln (HVS) + 0.11	0.16
Ln DVM = 0.23 Ln (Total Pb Wipe) + 0.4	0.47
Ln HVS = $0.26$ Ln (Total Pb Wipe) + $0.06$	0.16

<sup>a</sup> All samples included

Table 14 shows the ANOVA results for sampling method, house, location within each house, and location within each room (shown as co-location in the table).

# Table 14.

Source	Degrees of Freedom	Sum of Squares	F-Test	P-Value
Method	3	677.39	71.02	0.0001
House	19	291.74	1.02	0.45
Location (House)	57	856.41	46.17	0.0001
Method*House	57	181.21	0.88	0.71
Method*Location (House)	163	591.44	11.15	0.0001
Colocate(House(Location))	77	56.09	2.24	0.0001
Error	211	68.66		

# Nested Factorial ANOVA for Log-transformed PbD Loadings for Differences Among Sampling Methods Using All Data

Note:  $F_{method} = MS_{method} / MS_{method*house}$ 

 $F_{house} = MS_{house} / MS_{Location(house)}$ 

 $F_{method*(house)} = MS_{method*(house)} / MS_{method*Location(house)}$ 

The ANOVA showed that the lead dust loadings were significantly different according to:

- The method of sampling
- Locations within houses
- Locations within rooms

This last finding is especially important, since it suggests that lead loadings in adjacent areas within the same rooms are in fact not similar. This is contrary to the assumption stated at the outset of this study, that is, that lead loadings in areas immediately adjacent to each other within rooms are essentially the same. However, it is still possible to estimate the comparative reproducibility of each method by comparing residual variances for each method's loadings from the nested factorial model. This analysis was performed using all residuals <u>and</u> after removing one pair of outliers. These results are shown in Table 15.

Residu	al Variances for Ea From the Nested H	ch Method's Loadings Factorial Model
Methods	Using All Residuals <sup>a</sup>	After Removing the Highest Outlier Pair For Each Method
Bioavailable	0.1902	0.1640
Total	0.1635	0.0919
DVM	0.3617	0.2568
HVS	0.1567	0.1322

Table 15.

<sup>a</sup> Samples with weights less than 2 mg were eliminated

Residuals from an ANOVA which pooled the co-location variance with the error term were calculated and were used to estimate the residual variance for each sampling method. For the wipe samples, the residual variance was 0.19 and 0.16 for the bioavailable and total lead sampling methods, respectively. Residual variances of 0.36 and 0.16 were estimated for the DVM and HVS methods, respectively. After removing outliers, the residual variances were 0.16 for bioavailable wipe loading, 0.09 for total lead wipe loading, 0.26 for DVM lead loading, and 0.13 for HVS loading. This implies that the DVM is much less reproducible than the other methods when measuring loading and that the total lead wipe had the highest degree of reproducibility. The reproducibility of the total wipe is also considerably higher than the bioavailable wipe.

To determine if the lower reproducibility of the DVM is due to its ability to preferentially collect only smaller particles, residual variances were calculated after dividing the samples into "high" and "low" values by using both the arithmetic mean and the geometric mean as the dividing line. After performing this split, the residual variances declined, although it is not clear whether the reproducibility is greater or smaller for high and low loadings. This analysis is presented in Table 16.

# Table 16.Residual Variance for the DVM Method After Dividing the Samples into High and Low<br/>Values

Split at the Arithmetic Mean (48  $\mu$ g/ft<sup>2</sup>)

>49 <48 n=29 n=104 Res. Variance = 0.1342 Res. Variance = 0.1888

Split at the Geometric Mean (11.3  $\mu$ g/ft<sup>2</sup>)

$> 11.3 \ \mu g/ft^2$	$< 11.3 \ \mu g/ft^2$
n=67	n = 66
Res. Variance $= 0.2036$	Res. Variance $= 0.0795$

A second ANOVA model was constructed to examine the effect of differential efficiency on surface types. Terms that were confounded with surface type were removed. This ANOVA included only the sampling method, house, surface, and method by surface interaction.

The ANOVA model for log-transformed lead dust loadings indicated that each of this model's effects were significant (p < 0.0001). These results are shown in Table 17. The HVS-3 was again found to recover more of the lead dust than did the other methods and that the other methods were not significantly different. *A posteriori* comparisons between methods for each surface indicated that the HVS method recovered significantly more lead dust than did the other methods for each surface except vinyl. On vinyl surfaces, the wipe method was significantly better than either vacuum method. The DVM recovered the least lead dust on all surfaces except carpet, for which its recovery was second to HVS and performed significantly better than the wipe method analyzed by either bioavailable or total lead.

## Table 17

# ANOVA For Method And Surface Type Effects by House on Log-transformed PbD Loadings

Source	Degrees of Freedom	Sum of Squares	F-Test	P-Value
Method	3	249.97	68.20	0.0001
House	19	216.30	9.32	0.0001
Surface	5	609.44	99.77	0.0001
Surface*Method	15	514.05	28.05	0.0001
Error	545	665.82		

## Concentration

Summary statistics for measurement of concentration of lead dust by the two vacuum methods are shown in Table 18. The data show that the geometric mean concentration using the DVM method is about 100 ppm higher than the concentration found by the HVS-3 sampler. The spread in the data was higher for the HVS-3 sampler.

Method	N	Mean	S.D.	GM	GSD	C.V.	95%	5%
DVM	133	1019	3586	460	2.79	351.9	2782	86.7
HVS	147	2111	17313	374	3.67	820.0	3153	48.1

# Table 18.Summary Statistics for the Measurement of Lead DustConcentration by Vacuum Methods (Excluding Samples < 2 mg)</td>

A four factor nested factorial ANOVA was used to analyze the log-transformed concentrations calculated for the vacuum collection methods (see Tables 19 and 20). Only the house, location within house, and the method by location within house effects were significant. The lack of a significant effect for method (p = 0.38) indicates that there was not a significant overall difference between the two sampling methods when considering concentration. This is somewhat surprising, since the DVM is expected to pick up smaller particles (and thus higher lead concentrations) than the HVS-3, which picks up larger particles. This could be explained by the different performance efficiencies which are dependent on the type of surface sampled (see Tables 10 and 11). Significantly higher concentrations were found by the DVM method when the sample was taken on vinyl or wood. Significantly higher concentrations were found by the HVS method for high pile carpets and "other" surfaces. The differences in lead dust concentration between these methods were not significant for samples from low pile carpets or concrete (see Table 21).

Source	Degrees of Freedom	Sum of Squares	F-Test	P-Value
Method	1	0.79	0.81	0.38
House	19	88.71	1.96	0.03
Location (House)	56	133.56	4.71	0.0001
Method*House	19	18.37	0.95	0.53
Method*Location (House)	50	50.68	2.00	0.005
Colocate(House(Location))	74	38.15	1.02	0.47
Error	60	30.37		

 Table 19.

 Nested Factorial ANOVA for Log-transformed PbD Concentration (ppm) for Vacuum Sampling Methods

Note: F<sub>method</sub> = MS<sub>method</sub> / MS<sub>method\*house</sub>

 $F_{house} = MS_{house} / MS_{Location (house)}$ 

 $F_{method*(house)} = MS_{method*(house)} / MS_{method*Location (house)}$ 

# Table 20.

Nested Factorial ANOVA Limited to Method, House and Surface Type Effects for Logtransformed PbD Concentrations (ppm)

Source	Degrees of Freedom	Sum of Squares	F-Test	P-Value
Method	1	1.97	2.30	0.13
House	19	70.86	4.35	0.0001
Surface	5	45.25	10.56	0.0001
Surface*Method	5	20.81	4.85	0.0003
Error	249	213.48		

# Table 21.

# Summary of Surface Differences For Log-transformed Concentrations

Surface	Methods in Order of Capture Efficiency By Concentration
Vinyl	DVM > HVS
Wood	DVM > HVS
Low-Carpet	no difference (HVS = $DVM$ )
High-Carpet	HVS > DVM
Concrete	no difference (HVS = $DVM$ )
Other	HVS > DVM

## Correlations

Correlations among the average of the paired log-transformed lead loading samples in each room for each of the four methods and the average paired log-transformed lead dust concentrations for the two vacuum methods were also calculated. All of these correlations were significant at the 0.05 level. The two digestion methods used to analyze the wipe samples were very highly correlated with each other (r=0.96) but correlated less well with the lead dust measures obtained by vacuum sampling (r values ranged between 0.26 and 0.48). The two vacuum methods' loadings correlated highly with each other (r=0.75), while the concentrations correlated less highly (r=0.45). These results are shown in Table 22.

Table 22.Correlations among Log-transformed PbD Loading and Concentration Collected at All Locations (n=77) (Co-located Samples Are Averaged)									
	Bioavailable Wipe	Total Wipe	DVM (µg/ft²)	DVM (ppm)	HVS (μg/ft²)				
Total Wipe $DVM (ug/ft^2)$	0.96	0 44							
DVM (ppm)	0.48	0.44	0.68						
HVS $(\mu g/ft^2)$	0.26	0.29	0.75	0.44					
HVS (ppm)	0.43	0.46	0.52	0.45	0.68				
All r's are sta	tistically signi	ficant (j	o < 0.05)						

Correlations among methods were also calculated by surface type (see Table 23). However, the sample sizes are extremely small, so the utility of these measures is doubtful. The two wipe digestion methods remained highly correlated regardless of surface, ranging between r values of 0.79 for samples from vinyl surfaces to 0.97 for samples from wood surfaces. The correlation of wipe samples with the loadings found for the vacuum samples tended to be best for vinyl (r values ranged from between 0.74 and 0.93) and worst for high pile carpets (r values ranged between 0.17 and 0.85). The two vacuum methods' loadings correlated highly regardless of surface, but were most highly correlated for concrete samples (r=0.58 for hi-pile carpet, r=0.56 for low pile carpets) and vinyl (r=0.59). The correlations between these methods' concentrations were quite variable, ranging between r=0.16 for vinyl and r = 0.98 for high pile carpet.

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Correlations between Methods by Surface Types Using All Samples								
Surfaces	Bioavailable (µg/ft <sup>2</sup> )	Total (µg/ft <sup>2</sup> )	DVM (µg/ft²)	DVM (ppm)	HVS (µg/ft²)			
Vinyl (n=14 pairs)								
Total $(\mu g/ft^2)$	0.79							
DVM ( $\mu$ g/ft <sup>2</sup> )	0.64	0.75						
DVM (ppm)	0.75	0.62	0.85					
HVS $(\mu g/ft^2)$	0.62	0.60	0.59	0.42				
HVS (ppm)	0.28	0.32	0.03	0.16	0.43			
Wood (n=13 pairs)								
Total ( $\mu g/ft^2$ )	0.97							
DVM ( $\mu g/ft^2$ )	0.35							
DVM (ppm)	0.50	0.45	0.81					
HVS $(\mu g/ft^2)$	0.59	0.53	0.66	0.75				
HVS (ppm)	0.49	0.44	0.48	0.41	0.77			
Low-Carpet (n=30 pairs)								
Total $(\mu g/ft^2)$	0.88							
DVM ( $\mu g/ft^2$ )	0.67	0.66						
DVM (ppm)	0.37	0.40	0.67					
HVS ( $\mu$ g/ft <sup>2</sup> )	0.28	0.49	0.58	0.31				
HVS (ppm)	0.11	0.20	0.32	0.43				
High-Carpet (n=5 pairs)								
Total ( $\mu g/ft^2$ )	0.88							
DVM ( $\mu g/ft^2$ )	0.17	0.26						
DVM (ppm)	0.38	0.67	0.35					
HVS ( $\mu$ g/ft <sup>2</sup> )	0.54	0.85	0.56	0.92				
HVS (ppm)	0.21	0.54	0.42	0.98	0.87			
Concrete (n=14 pairs)								
Total ( $\mu g/ft^2$ )	0.95							
DVM ( $\mu$ g/ft <sup>2</sup> )	0.74	0.76						
DVM (ppm)	0.67	0.61	0.79					
HVS $(\mu g/ft^2)$	0.85	0.93	0.80	0.65				
HVS (ppm)	0.43	0.58	0.50	0.37	0.68			

 Table 23.

 Correlations Between Methods by Surface Types Using All Samples

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# Conclusions

This study supports the following conclusions:

- 1. Each method yielded significantly different results, indicating that the choice of sampling method is important.
- 2. The bioavailable and total lead wipe sampling techniques are highly correlated with each other; each of these wipe methods is also correlated with the DVM method to a lesser extent and to the HVS-3 method even less.
- 3. Both wipe sampling techniques performed relatively poorly on carpets, suggesting that one of the vacuum methods must be used there. Wipe sampling performed very well on smooth surfaces.
- 4. The DVM had a relatively poor level of reproducibility for loading, compared with wipe sampling and the HVS-3. For concentration, the DVM had a lower coefficient of variation than did the HVS-3.
- 5. The DVM generally found higher (but not significantly higher) <u>concentrations</u> than did the HVS-3, while the HVS-3 found higher <u>loadings</u>. This is not surprising, since lead dust levels are known to be more concentrated in the smaller particle size ranges, which the DVM samples preferentially.
- 6. The HVS-3 and the New Jersey tared wet wipe sampling method both display serious feasibility issues with regard to widespread field implementation. The HVS-3 method is less portable, requires AC electrical current, is relatively difficult to clean in the field (causing a longer sampling time), and shows a significant potential for cross-sample contamination. The New Jersey method also has significant field implementation problems, mostly with regard to handling of tared media in the field. Both of these are essentially research methods still undergoing development.

There are important limitations to this study that are worth noting. First, many of the sampling results in this study were below the range of interest, at least for loading, which is  $50 \ \mu g/ft^2$  or more by wipe sampling. Since it is likely that efficiency and reproducibility for each of the methods will vary depending on the extent of pre-existing contamination, the findings here may not be representative of conditions close to existing standards (200  $\mu g/ft^2$  for floors by wipe sampling).

Second, a limited number of homes were included in this study, due to time and financial constraints. A larger study involving sampling of all different types of floor surfaces may have yielded more robust findings.

Finally, the fact that a HEPA vacuum cleaning and educational intervention was done in some of the study homes some time ago may have altered the "normal" particle size distribution and dust lead loadings. Specifically, the removal of fine particles may have affected the relative efficiency of each of the methods.

# Recommendations

Regression equations and correlation coefficients have been calculated for each of the sampling methods examined here, providing at least some preliminary means of relating sampling methods used in previous studies to each other. This should enable EPA to make better use of previously-conducted epidemiological studies in setting a standard for "dangerous" levels of lead in dust, as required under Title X. It may also prove to be quite helpful in any meta-analysis. Of course, these factors require confirmation in additional studies. This meets the first objective of this study.

The decision on which sampling methods to use in the Rochester study is a complex one, since there are important additional factors that lie beyond this study that must be considered. Most importantly, earlier studies that established a correlation between blood lead level and the DVM method are perhaps the most robust for any of the sampling methods. If it is true that the DVM method is the best in terms of yielding biologically-significant information, and if it is also true that the DVM method is more feasible in the field, then it would be a serious mistake not to use it in the Rochester study. In terms of its ability to correlate with blood lead levels, it must be said that the HVS-3 method is entirely unproven at this point. Some of the problems with the DVM sampling technology can be readily fixed by replacement of the slotted nozzle with a round tubing one, guide wires on area templates, and perhaps replacement of the MCEF filter with a tared PVC filter. In addition, the sampling equipment is already widely available, since many state and local governments perform air sampling and may already have the pump required for the DVM method. These constant flow pumps cost about \$750 - \$1,000 each (about the same as the HVS-3 cyclone) and have a long track record of rugged field use in the industrial hygiene community. Detection limit concerns could be most adequately addressed by enlarging the sampling area for relatively clean surfaces. Finally, quality control would be fairly straightforward, since NIST standards are available for the cassette filters used here and since cleaning or handling of sampling media in the field would not be required.

The HVS-3 sampler is in many respects still under development and not quite ready for widespread field use. The sampling pump (Dirt Devil vacuum) is not a constant flow device and therefore may be more subject to surface variations than the DVM method (i.e., it may perform more poorly on porous surfaces). However, one would have expected to see poorer reproducibility in this study for the HVS-3 if this were in fact a major difficulty. The power supply issue will be a major problem in housing where no power exists, whereas the DVM is battery-operated. It is likely that the HVS-3 sampler will take longer to operate, while the DVM sampling train can be put together simply and quickly. The main advantage of the HVS-3

sampler is that it appears to collect far more lead dust more consistently and therefore may prove to be a more useful tool in estimating both current and potential risk.

In short, if the HVS-3 method is selected for the Rochester study, it is possible that in the end a method will have been used that does not correlate well with blood lead levels and may not be capable of being widely and quickly implemented in the field.

On the other hand, this study did demonstrate the superior reproducibility for loading and collection efficiency of the HVS-3 method.

There are similar, but perhaps less critical issues in comparing the two wipe sampling methods. Although the "bioavailable" extraction is the historical basis for the 200  $\mu$ g/ft<sup>2</sup> standard, this study suggests there may be little difference between the two (the geometric mean for bioavailable lead in this study is 11  $\mu$ g/ft<sup>2</sup>, while for total lead it is 15  $\mu$ g/ft<sup>2</sup> - with similar geometric standard deviations, hardly a large difference). Reproducibility was also quite similar and the correlation between the two methods was quite high. The bioavailable sample preparation method is considerably more labor-intensive in the laboratory, requiring long-term agitation and pH adjustment, which is likely to increase costs. Finally, quality control will be quite difficult for the bioavailable extraction, whereas for total lead digestion, it has been shown that consistently acceptable recoveries of lead from field spiked wipe samples containing known amounts of lead dust is feasible (see Appendix C for blind spiked wipe samples inserted into the sample stream for the public housing risk assessment program). No one truly knows (as of this writing) how much lead from any of the common lead NIST standards leaches out under the bioavailable digestion protocol, although this could probably be determined with exhaustive testing. It would be far simpler to weigh out a known amount of lead dust on a routine basis and determine if the laboratory can achieve recoveries of 80 -120%.

The National Center for Lead-Safe Housing believes that it is important to use <u>three</u>, not two lead dust sampling methods on a side-by-side basis in the Rochester study. Due to its carefully-controlled nature, this study should enable us to conclude which of the two vacuum techniques is superior in terms of correlation with blood lead level. Wipe sampling must be one of the methods used, since it is by far the simplest and easiest method and has been shown to exhibit some correlation with blood lead level.

We believe that it is both feasible and necessary to collect three side-by-side samples, although this will necessitate acquiring at least one more additional environmental sampling team and approximately \$100,000 in additional funding. To conclude, we recommend that the following lead dust sampling methods be employed on a side-by-side basis in the Rochester study:

- Wipe sampling with total lead digestion
- HVS-3 vacuum dust sampler
- DVM vacuum dust sampler

# Appendix A

# Sampling and Analytical Methods

## Acknowledgments

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