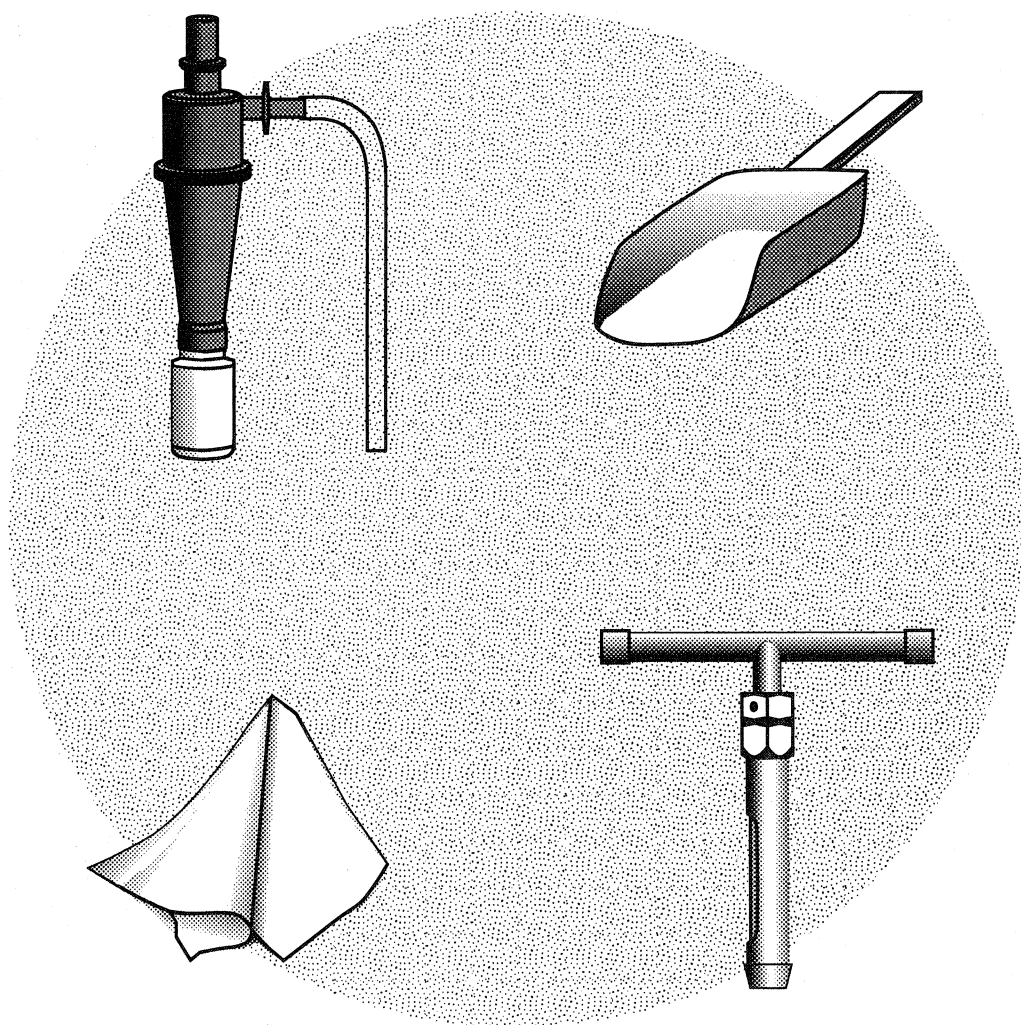




Residential Sampling for Lead: Protocols for Dust and Soil Sampling

Final Report



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**Residential Sampling for Lead:
Protocols for Dust and Soil Sampling**

Final Report

**For U.S. Environmental Protection Agency
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Technical Programs Branch, MC-7407
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A. Introduction

1.0 Overview

This document provides guidance for the collection of soil and settled dust samples for subsequent determination of lead. Collection of soil samples is performed using either coring or spooning methods. Collection of settled dust samples are performed using wipe or vacuum methods. Advantages and disadvantages of each method are discussed. Protocols presented in this document are capable of producing samples for lead determination results in μg per gram of soil, μg per gram of dust, and μg per ft^2 of dust.

2.0 Purpose

The purpose of this document is to provide detailed sampling procedures to maintain uniformity in data collection of lead in soil and settled dust samples. Analytical results obtained from samples collected using these sampling procedures will eventually be used for comparison to numerical health-based standards developed by the U.S. Environmental Protection Agency (EPA) for discerning unsafe lead levels, according to Title X, Section 403: *Identification of Dangerous Levels of Lead*.

Today, a variety of approaches to sample collection for lead determinations are being utilized. Substantial resources are applied to the generation of lead data. The use of standardized protocols should improve the value of lead data gathered by a wide variety of organizations and individuals.

3.0 Related Documents

Currently, EPA is developing health-based standards for lead in soil and house dust under the Toxic Substances Control Act (TSCA), Title X, Section 403. Furthermore, EPA will soon publish standards on the conduct of residential lead risk assessments for the purposes of training risk assessors (TSCA, Title IV, Section 402). Both standards are interwoven and extremely important in addressing public health issues regarding lead.

For guidance to identify, control, or abate lead hazards in housing, consult the U.S. Department of Housing and Urban Development document, Guidelines for the Evaluation and Reduction of Lead-Based Paint Hazards in Housing (HUD, 1994).

To obtain more information on lead, call the National Lead Information Center Clearinghouse at (800) 424-LEAD. In the Washington, D.C., area, call (202) 833-1071.

($\mu\text{g}/\text{ft}^2$). Vacuum dust collection can generate both lead loading and lead concentration results. However, vacuum dust collection is generally more difficult and costly to perform than wipe dust collection.

6.0 Laboratory Analysis

Samples collected for lead determination are generally analyzed using atomic spectrometry methods. These instrumental methods require samples to be converted from a solid to a liquid form prior to lead measurement. This conversion process is commonly referred to as sample preparation. Sample preparation generally includes several initial handling steps, followed by a digestion process, that solubilize the lead contained in the sample. The accuracy and precision of the lead determination is dependent on both the sample preparation and instrumental analysis activities. An estimate of accuracy and precision can be obtained by submitting Quality Control (QC) samples to the laboratory together with field samples. Preparation of appropriate QC samples is discussed in the sample collection protocols presented in this document. Although this document is not intended as a guide on laboratory analysis procedures, a brief discussion of sample preparation and instrumental analysis activities, as they relate to the collection of soil and settled dust sample, is presented below.

6.1 Sample Preparation of Soil Samples

A number of laboratory sample preparation methods can be used for the preparation of soil samples. Hot plate digestions, such as SW846 method 3050³ or ASTM ES 36-94⁴, utilize nitric acid (sometimes with hydrochloric acid) and hydrogen peroxide for oxidation of sample components and solubilization of lead. Microwave digestions, such as 3051³, utilize nitric acid for oxidation of sample components and solubilization of lead. Sample size limits exist for both digestion methodologies. Samples collected in the field and submitted to the laboratory are generally much larger than the digestion methods are capable of accommodating. Therefore, a subsample of the original soil sample must be used for the lead determination. This requires the use of a homogenization process prior to digestion to generate a representative subsample. The water content of collected soils can be widely variable. Therefore, to provide consistency for lead data comparisons under variable soil and weather conditions, soil samples must be reported on a dry weight basis. This requires inclusion of a drying process into sample preparation method used for soil analysis.

In summary, any sample preparation method used for lead determination in soil samples must include a homogenization and drying process prior to subsampling for digestion and solubilization of the lead.

Absorption Spectrometry (GFAAS), and Inductively Coupled Plasma Atomic Emission Spectrometry (ICPAES). A variety of methods covering the use of these techniques, such as SW846 methods 7420³, 7421³ and 6010³, or ASTM E 1613-94⁵, can be used for lead measurements. In general, FAAS and ICPAES both have sufficient detection capability for lead determinations in all soil samples and most dust samples. However, for clean environments, GFAAS detection, which has approximately a 10-fold improvement in detection capability over FAAS and ICPAES, may be more appropriate for some dust samples. Lead measurement data must incorporate sample preparation variables, such as sample weights and digestion volumes, prior to reporting lead results.

- 2.2.3 Sample collection container, resealable plastic bags (1 quart or 1 gallon) or sealable rigid walled container with 50-mL minimum volume. If plastic bags are used, samples should be double bagged to protect against breakage and potential sample loss.
- 2.2.4 Spoon, plastic or stainless steel. Used for scoop sampling.
- 2.2.5 Steel or plastic measuring tape or ruler, divisions to at least $\frac{1}{8}$ inch.

2.3 General Supplies

- 2.3.1 Field notebooks, bound with individually numbered pages, see subsection 4.1.
- 2.3.2 Indelible ink marker, black or blue.
- 2.3.3 Ink pens, black or blue.
- 2.3.4 Packaging tape, used for sealing shipping containers.
- 2.3.5 Plastic bags, trash bags with ties.
- 2.3.6 Plastic gloves, powderless. Gloves with powder should not be used to avoid potential contamination of samples from powder material.
- 2.3.7 Preprinted field forms, preprinted with sufficient entry lines to address documentation needs presented in subsection 4.1
- 2.3.8 Shipping containers, cardboard or plastic for interim storage and shipment of sample collection containers.

2.4. Cleaning Supplies

- 2.4.1 Water, drinking water. Drinking water is used to assist in cleaning sampling equipment for soil sample collection. High purity water is not required for cleaning of sampling equipment because action levels for lead in soils are relatively high with respect to lead levels in drinking water.
- 2.4.2 Wipe, Disposable towelette moistened with a wetting agent. Used for cleaning sampling equipment. Wipe brands or sources should contain insignificant background lead levels. Laboratory analysis of replicate blank wipes should be used to determine background lead levels prior to

through the soil surface while maintaining the scooping depth of the tube (0.5 inch) in the soil. Move the tube a distance of 6-12 inches across the soil surface to complete collection of the soil into the tube. The movement of the tube across the sample location will result in a composite type soil sample.

3.1.1.5 Remove the tube from the ground, and wipe off any excess soil clinging to the outside of the tube and cap threads with a gloved finger. Replace the cap. Label the plastic centrifuge tube with sufficient information to uniquely identify the sample. Discard any gloves used during sample collection in a trash bag.

3.1.2 Scoop Sampling Using a Spoon

3.1.2.1 Label a new resealable plastic bag for use as a sample collection container (See subsection 5.5).

3.1.2.2 Pull on a pair of clean, powderless, plastic gloves. Gloves are used to protect the workers' hands and the integrity of the samples (to aid in avoiding cross-contamination between samples).

3.1.2.3 Using a measuring tape and a clean spoon, dig a small test hole adjacent to the sampling location to the depth of 0.5 inch. Use this hole as a visual aid during soil collection to help limit collection to a depth of 0.5 inch. Clean the spoon using a wipe until soil is no longer visible on the spoon.

3.1.2.4 Scoop the soil with the spoon down to the depth indicated by the test hole and place the sample in a sample collection container. Continue to collect soil until a circular hole of approximately 2 inch (0.5 inch deep) has been created.

3.1.2.4 Collect soil from two more locations within a 1 foot diameter circle around the first sample location, using the same procedure described above (subsections 3.1.2.2 through 3.1.2.4). Composite these scoop samples into the same sample collection container and seal the container in a manner that will minimize the air contained in the container. Discard any gloves used during collection in a trash bag after all three scoop samples have been collected and composited.

3.1.2.5 Pull on a pair of clean, powderless, plastic gloves. Clean the spoon using wipes and water until soil is no longer visible on the spoon. Discard any wipes and gloves used during cleaning in a trash bag. An alternative approach to cleaning is to use disposable spoons.

The top end is the opposite opening.) Push out all but 0.5 inch of the soil from the probe with the plunger. Using a gloved finger, wipe off the excess soil protruding from the probe. Allow the soil pushed out of the probe to fall on the ground near but not on the sampling location.

3.2.8 Using a clean plunger (without stop), push the remaining 0.5 inch section of the sample core into a sample collection container.

3.2.9 Collect two more soil cores within a 1 ft diameter circle around the first sampling location, using the same procedure described above (subsections 3.2.2 through 3.2.8). Composite these cores into the same sample collection container and seal the container in a manner that minimizes the air contained in the container. Discard any gloves used during collection in a trash bag after all three core samples have been collected and composited.

3.2.10 Pull on a pair of clean, powderless, plastic gloves. Clean the coring probe, coring plungers, and plastic inserts (if used) using wipes and water until soil is no longer visible on the equipment. Discard any wipes and gloves used during cleaning in a trash bag.

4.0 Quality Control

Adherence to quality control (QC) procedures is an important part of field sample collection. QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

4.1 Documentation

All field data related to sample collection must be documented. A field notebook or sample log form can be used to record field collection data. It is recommended that both types of documentation records (field notebooks and preprinted sample log forms) be utilized to assure collection of all relevant field data. Field data entries on documentation records must adhere to the following requirements:

4.1.1 General Documentation Requirements:

- All entries must be made using ink.
- Each page (notebook or form) must include the name of the person making the entries and the date of entries found on the page.
- Any entry errors must be corrected by using only a single line through the incorrect entry (no scratch outs) accompanied by the initials of the person making the correction and the date of correction.

- 4.2.2 Blind Reference Material Samples. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods. Prepare a blind reference material by placing a portion (1-2 grams) of a reference material into a labeled sample collection container. It is recommended that the frequency of these QC samples be at least 1 per 20 field samples. Reference materials from NIST⁶, such as SRMs 2709, 2711, and 2704, are readily available and can be used for preparing blind reference materials. Other sources of materials with known lead levels, such as soil materials from the ELPAT⁷ program, may also be used as blind reference materials.

4.3 Contamination Avoidance

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid tracking soil from one location to another by:
 - identifying and clearly marking all sampling locations upon arrival at the sampling site, and
 - instructing field team members to avoid walking through or over any of the marked sampling location areas.
- Use a new pair of powderless gloves at each sampling location.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample containers until needed to collect each sample.
- When using bulk packed wipes, at each sampling location, discard the first two wipes pulled from the wipe container.

5.0 Glossary

- 5.1 Digestion, Sample preparation process that solubilizes lead present in the sample. The digestion process produces an acidified, aqueous solution called the digestate. A lead determination is made on the digestate during an instrumental measurement process.
- 5.2 Field Data, Any information collected at the sampling site.
- 5.3 Field Sample, Physical material taken from the sampling site that is targeted for lead determination.
- 5.4 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large

C. Protocol for Collection of Dust Samples for Lead Determination Using Wipe Sampling

1.0 Introduction

This protocol provides for the collection of settled dust samples from hard, relatively smooth, nonporous surfaces using wipe methods. The protocol is not applicable for the collection of settled dust samples from highly textured surfaces, such as brickwork and rough concrete, and soft fibrous surfaces, such as upholstery and carpeting. The protocol is capable of producing samples for lead determination results in loading terms ($\mu\text{g}/\text{ft}^2$).

2.0 Equipment and Supplies

2.1 Sampling Equipment

- 2.1.1 Disposable shoe covers (optional), see subsection 4.3.
- 2.1.2 Masking tape, used for holding down sampling templates and marking sampling locations.
- 2.1.3 Sample collection container, sealable rigid-walled container with 50-mL minimum volume. Use of a resealable plastic bags for holding and transporting the settled dust wipe sample is not recommended due to the potential losses of settled dust within the plastic bag during laboratory handling. Quantitative removal and processing of the settled dust wipe sample by the laboratory is significantly improved through the use of sealable rigid walled containers.
- 2.1.4 Sampling template, 1 ft² inside area reusable aluminum or plastic, or disposable cardboard or plastic template. A variety of shapes are recommended for use in variable field situations such as square, rectangular, square "U" shaped, rectangular "U" shaped, and "L." All templates must have accurately known inside dimensions. Templates should be thin (less than $\frac{1}{8}$ inch) and capable of lying flat on a flat surface.
- 2.1.5 Steel or plastic measuring tape or ruler, divisions to $\frac{1}{16}$ inch.
- 2.1.6 Wipe, disposable towelette moistened with a wetting agent. Wipe brands or sources should contain insignificant background lead levels. Laboratory analysis on replicate blank wipes should be used to determine background lead levels prior to use in the field. Background lead levels less than 10 μg per wipe are considered insignificant for most

assumes that the depth is no larger than the dimensions of a wipe. If this is not true, then the Template Assisted Sampling Procedure should be used.

3.1 Template Assisted Sampling Procedure

Following is a summary of this procedure:

1. Select a sampling location.
2. Mark the sampling location using a template.
3. Perform first wiping: Side-to-side, fold the wipe.
4. Perform second wiping: Top-to-bottom, fold the wipe.
5. Perform third wiping: Clean-up the corners, fold the wipe, and store the sample.

The detailed procedure is as follows:

- 3.1.1 Pull on a pair of clean, powderless, plastic gloves.
- 3.1.2 Carefully place a clean template on the surface in manner that minimizes disruption of settled dust at the sampling location. Either tape or place a heavy object on the outside edge of the template to prevent it from moving during sample collection. An alternative to using a template is to mark an outline of the sampling location using masking tape as described in subsection 3.2.2.
- 3.1.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.
- 3.1.4 At the beginning of a sampling period (or if a new bulk-packed container of wipes is opened), remove a minimum of the top 2 wipes from the container and wipe off gloved fingers with each wipe as they are removed. Use the next wipe from the container to collect the sample.
- 3.1.5 First Wiping, Side-to-Side: Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area, starting at either corner furthest away from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion. During wiping, apply pressure to the finger tips.

At the end of the first pass from one side to the other, turn the leading edge of the wipe (the portion of the wipe touching the surface) 180 degrees, pulling the wipe path slightly closer to the operator and make a

4. Perform second wiping: One direction (reverse), Side-to-side, fold the wipe.
5. Perform third wiping: Clean-up the corners, fold the wipe, and store the sample.

The detailed procedure is as follows:

- 3.2.1 Pull on a pair of clean, powderless, plastic gloves.
- 3.2.2 Mark an outline of the sampling location using masking tape. Care should be taken to minimize any disruption of dust at the sampling location. For areas that are dirty or contain high dust levels, new tape may have to be applied more than once to get adhesion to the surface. Discard any soiled tape in a trash bag.
- 3.2.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.
- 3.2.4 At the beginning of a sampling period (or if a new bulk-packed container of wipes is opened), remove a minimum of the top 2 wipes from the container and wipe off gloved fingers with each wipe as they are removed. Use the next wipe from the container to collect the sample.
- 3.2.5 First Wiping, One Direction, Side-to-Side: Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area, starting at either corner furthest away from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion. During wiping, apply pressure to the finger tips. At the end of the first pass from one side to the other, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half with the sample side folded inside the fold.
- 3.2.6 Second Wiping, One Direction, Side-to-Side: Using a clean side of the wipe, repeat step 3.2.5 using a wiping motion in the reverse direction.
- 3.2.7 Third Wiping, Clean Corners: Using a clean side of the wipe, perform a third wiping around the perimeter of the sampling area to collect any dust remaining in the corners. Start from the middle of one edge of the area and use the same wiping technique as described above. When the perimeter has been wiped and the starting location reached, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half one more time with the sample from this third wiping folded inside the fold.

4.1.3 Documentation Required for Each Sample Collected:

- An individual and unique sample identifier and date of collection. This must be recorded on the sample container in addition to the field data records (notebook or form).
- Name of person collecting the sample and specific sampling location data from which the sample was removed.

4.2 QC Samples

4.2.1 Blank Samples. Blank samples should be periodically collected at random throughout the sampling day at each sampling site. Two types of blank samples should be collected: field blanks and QC blanks. Both these blanks are collected in the same manner; however, they are used for different purposes.

4.2.1.1 Field blanks. Field blank samples are used to identify any potential systematic lead contamination present in the wipe and during the handling of samples during field collection and laboratory analysis activities. Field blanks should be collected in the same manner as used to collect field samples with the exception that no surface is wiped. Each wipe designated as a field blank should be removed from the bulk pack, folded to match the field samples, and placed into a labeled sample collection container.

Each field blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blanks can then be submitted in a blind manner to the laboratory). It is recommended that field blanks be collected at a frequency of 1 per 20 field samples. At a minimum, three should be collected at each sampling site for each new pack of bulk wipes used for sample collection (i.e., one near the beginning of the sampling period at the site, one in the middle, and one near the end). Field blank lead results should not exceed 20 µg/sample. Lead results above this value should trigger an investigation into the potential cause and resampling of samples associated with the field blank may have to be undertaken. Large blank lead values can often be sporadic and not systematic; therefore, blank correction of field sample results using field blank data is not recommended.

4.2.1.2 QC blanks. QC blank samples are used for preparation of blind reference material samples described in subsection 4.2.2. QC blanks should be collected in exactly the same manner as described

- Clean sampling equipment and measuring tapes frequently with wipes.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample collection containers until needed to collect each sample.
- When using bulk packed wipes, at each sampling location, discard the first two wipes pulled from wipe container.

5.0 Glossary

- 5.1 Digestion, Sample preparation process that solubilizes lead present in the sample. The digestion process produces an acidified, aqueous solution called the digestate. A lead determination is made on the digestate during an instrumental measurement process.
- 5.2 Field Blank, See subsection 4.2.1.
- 5.3 Field Data, Any information collected at the sampling site.
- 5.4 Field Sample, Physical material taken from the sampling site that are targeted for lead determination.
- 5.5 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large amount of lead determinations to develop a lead result known to a high degree of confidence.
- 5.6 Sample Collection Container, Container for holding and transporting the samples from the field to the laboratory. The internal volume of the container must be sufficient to hold the entire collected sample.
- 5.7 Sampling Location, Specified area within a sampling site that is subjected to sample collection. Multiple sampling locations are commonly designated for a single sampling site. An example would be at the bottom of a specific slide in a specific playground area.
- 5.8 Sampling Site, Local geographical area that contains the sampling locations. A sampling site is generally limited to an area that can be easily covered on foot. An example would be John Smith's house at 3102 Nowhere Avenue, Detroit, MI.

D. Protocol for Collection of Dust Samples for Lead Determination Using Vacuum Sampling

1.0 Introduction

This protocol provides for the collection of settled dust samples from surfaces using vacuum methods. The protocol is suitable for the collection of settled dust samples from both hard or smooth and highly textured surfaces, such as brickwork and rough concrete, and soft, fibrous surfaces, such as upholstery and carpeting.

Procedures presented in this protocol are intended to provide a method for collection of dust from surfaces that can not be sampled using wipe collection methods. In addition, these procedures are written to utilize equipment that is readily available and in common use for other environmental sampling applications (i.e., air particulate sample collection).

Due to the flow dynamics inherent in the vacuum method, results for vacuum dust samples are not likely to reflect the total dust contained within the sampling area. This protocol generally will have a collection bias toward smaller, less dense, dust particles. However, the protocol, if performed as written, will generate dust lead data that will be consistent and comparable between operators performing the method. This protocol can be used to produce samples for lead determination results in both loading ($\mu\text{g}/\text{ft}^2$) and concentration ($\mu\text{g}/\text{g}$). It is recommended, however, that it not be used for the generation of concentration results due to particle size collection bias and potential errors intrinsic to processing and handling preweighed filters (or entire filter cassettes), which are required to determine total collected sample weight. Even though it is not normally recommended, this protocol includes procedures for generation of total collected sample weight.

Other vacuum sampling methods that utilize less common equipment, such as cyclone sample collectors, may be useful for collection of settled dust, particularly with respect to generation of more quantitative dust lead concentration results.

2.0 Equipment and Supplies

2.1 Sampling Equipment

- 2.1.1 Air-sampling pump. A portable, battery-powered air pump that is capable of a flow rate of 2.5 L/min through a filter cassette equipped with the nozzle specified in subsection 2.1.2. Inlet of the pump must be fitted with a nipple to accept the tubing sized to fit tightly on the outlet side of a filter cassette.

2.2 General Supplies

- 2.2.1 Field notebooks, bound with individually numbered pages, see subsection 4.1.
- 2.2.2 Indelible ink marker, black or blue.
- 2.2.3 Ink pens, black or blue.
- 2.2.4 Packaging tape, used for sealing shipping containers.
- 2.2.5 Plastic bags, trash bags with ties.
- 2.2.6 Plastic gloves, powderless. Gloves with powder should not be used to avoid potential contamination of samples from powder material.
- 2.2.7 Preprinted field forms, preprinted with sufficient entry lines to address documentation needs presented in subsection 4.1
- 2.2.8 Shipping containers, cardboard or plastic for interim storage and shipment of sample collection containers.

3.0 Sampling Procedure

Two types of sampling procedures are presented. The first, Loading Only Vacuum Collection, is intended for collection of dust for lead loading determinations ($\mu\text{g}/\text{ft}^2$) only. The second, Collection on Prew weighed Media, is intended for collection of dust for both lead loading ($\mu\text{g}/\text{ft}^2$) and lead concentration ($\mu\text{g}/\text{g}$) determinations. The latter type has two options that differ in the methods used for determining the total collected sample weight.

3.1 Calibration of Air-Sampling Pump

Regardless of the type of the sampling procedure used (see subsection 3.2 or 3.3), the air-sampling pump used for sample collection must be calibrated prior to sample collection for any given day. The procedure for air pump calibration is as follows:

- 3.1.1 Label a filter cassette with an ink marker to distinguish it as one used for pump calibration (and not to be confused with or used for collection of a field sample). Remove the inlet and outlet plugs and place them in a labeled, resealable plastic bag.

- 3.2.2.2 Manual Marking of Sampling Area, Mark an outline of the sampling location using masking tape. Care should be taken to minimize any disruption of dust within the sampling location. For areas that are dirty or contain high dust levels, new tape may have to be applied more than once to get adhesion to the surface. Discard any soiled tape in a trash bag.
- 3.2.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.
- 3.2.4 If not prelabeled from prefield processing, label a filter cassette with an ink marker. Remove the inlet and outlet plugs and place them into a labeled resealable plastic bag. Attach the outlet to the air-sampling pump with a piece of flexible tubing. Attach collection nozzle to the inlet side of the filter cassette using a short section of new tubing (less than $\frac{1}{2}$ inch). Always use a new section of tubing for the inlet side of the filter cassette.
- 3.2.5 First Vacuuming: One Direction, Side-to-Side: With the air-sampling pump on, vacuum the selected sampling surface area, starting at either of the corners furthest from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion while holding the collection nozzle at an angle of approximately 45° to the sampling surface. Avoid pressing down hard on the sampling surface during sample collection. Move the nozzle at a rate of approximately 2-4 inches per second. At the end of the first pass from one side to the other, carefully lift the collection nozzle and repeat the vacuuming sweep in the same direction as the first, using a slightly closer overlapping pass. Care must be taken to avoid overloading of the filter cassette. Repeat the procedure until the entire sampling area has been covered using the one-direction, side-to-side sweeping motions.
- Overloading will result in decreased air flow and a reduction in sampling efficiency and increased sampling bias toward smaller, less dense particles. A drop of air flow of more than 10% is an indicator of overloading. If overloading of samples becomes evident, reduce the sampling area to prevent filter overloading or use multiple cassettes for collection within the same sampling area.
- 3.2.6 Second Vacuuming: One Direction, Top-to-Bottom: With the air-sampling pump on, vacuum the selected sampling surface area, starting at a far corner, using a slow top-to-bottom sweeping motion in the same manner as described in subsection 3.2.5. Repeat the procedure until the entire sampling area has been covered using the one-direction, top-to-bottom sweeping motions.

3.3.1 Prefield Stabilization and Gravimetric Procedure, Option 1—Prewriteghed Filter

This procedure suffers from the lack of quantitative transfer of all dust clinging to the cassette during postfield processing. Therefore, this option is considered somewhat more qualitative than Option 2. However, unlike Option 2, it is not susceptible to weight errors resulting from inadvertent touching or improper handling of the filter cassettes between pre- and postfield processing.

3.3.1.1 Prefield Procedure. The filter inside the cassette (not the backup support pad) must be weighed to constant weight prior to sample collection (prefield) at known temperature and humidity conditions (i.e., desiccated at room temperature). This can be performed for preloaded filter cassettes as follows:

- a. Pull on a new pair of clean, powderless, plastic gloves.
- b. Place a unique sample identifier on the outside of each cassette targeted for preweight generation using indelible ink and allow to dry.
- c. Using a clean screwdriver, separate the cassette rings that hold the filter in place. Place the rings on a clean, dry area, such as a plastic bag or equivalent surface.
- d. Using clean plastic tongs, lift the filter from the cassette and place it in a clean, dry, labeled beaker, watch glass, or other equivalent labeled container.
- e. Place the container with filter into a desiccator and allow the filter to stabilize to a constant weight. Periodically weigh and record the filter on a clean balance to determine weight stability. (Record all weights to ± 0.0001 g.) A constant weight for this protocol is one that does not change more than ± 0.002 gram for repeated measurements (minimum of 2) taken over a minimum of a 24-hour period. Using clean plastic tongs, replace the filter back into the cassette, reassemble the cassette, reweigh the container, and record the empty container weight. The prefield filter weight is the difference between the container plus filter weight and the container-only filter weight.
- f. Place the preweighed filter inside the sample cassette into a resealable plastic bag container for transport to the field.

3.3.1.2 Postfield Procedure. The filter and dust inside the cassette (not the backup support pad) must be weighed to constant weight prior to laboratory sample preparation (postfield) at the same known temperature and humidity conditions used for prefield processing. This can be performed as follows:

- a. Pull on a new pair of clean, powderless, plastic gloves for each sample handled.

- c. Remove the inlet and outlet plugs and place them into a labeled, resealable plastic bag.
- d. Place the filter cassette into a desiccator in a manner that allows air to flow freely through the inlet and outlet holes. Allow the filter cassette to stabilize to a constant weight. Record the weight of the entire filter cassette without plugs. (Record all weights to ± 0.0001 g.) A constant weight for this protocol is one that does not change more than ± 0.002 grams for repeated measurements (a minimum of 2) taken over a minimum of a 24-hour period.
- e. Replace the inlet and outlet plugs and place the entire filter cassette with plugs into a labeled resealable plastic bag for transport to the field.

3.3.2.2 Postfield Procedure. The filter cassette with dust (without plugs) must be weighed to constant weight prior to laboratory sample preparation (postfield) at the same known temperature and humidity conditions used for prefield processing. This can be performed as follows:

- a. Pull on a new pair of clean, powderless, plastic gloves.
- b. Remove the inlet and outlet plugs and place them back into the original labeled, resealable plastic bag.
- c. Place the filter cassette into a desiccator in a manner that allows air to flow freely through the inlet and outlet holes and that does not allow any spillage of dust out the holes. Allow the filter cassette to stabilize to a constant weight. Record the weight of the entire filter cassette plus dust without plugs. (Record all weights to ± 0.0001 g.) A constant weight for this protocol is one that does not change more than ± 0.002 grams for repeated measurements (minimum of 2) taken over a minimum of a 24-hour period. It is recommended (not required) that no initial weight data be attempted until the sample has remained in the desiccator for at least 72 hours.
- d. The contents of the filter cassette should be prepared for lead analysis. A quantitative transfer procedure that utilizes the backup support pad for wiping dust out of the inside of the cassette combined with rinsing out the cassette with dilute acid can be used to transfer the entire sample to the digestion vessel. The total sample weight for use in determining lead concentration is the difference between the postfield filter cassette weight and the prefield filter cassette weight.

4.0 Quality Control

Adherence to quality control (QC) procedures is an important part of field sample collection. QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

Both these blanks are collected in the same manner; however, they are used for different purposes.

- 4.2.1.1 Field blanks. Field blank samples are used to identify any potential systematic lead contamination present in the filter cassette and handling of samples during field collection and laboratory analysis activities. Field blanks should be collected in the same manner used to collect field samples with the exception that no air is drawn through the filter cassette. Each cassette designated as a field blank should be removed from the plastic bag, inlet and outlet caps pulled off, the tubing and sampling nozzle attached, and then this procedure is reversed. The vacuum pump is not turned on.

Each field blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blanks can then be submitted in a blind manner to the laboratory). It is recommended that field blanks be collected (or designated) at a frequency of 1 per 20 field samples. At a minimum, one field blank should be collected at each sampling site. Field blank lead results should not exceed 20 µg/sample. Lead results above this value should trigger an investigation into the potential cause of the problem and resampling of samples associated with the field blank may have to be undertaken. Large blank lead values can often be a sporadic and not systematic; therefore, blank correction of field sample results using field blank data is not recommended.

- 4.2.1.2 QC blanks. QC blank samples are used for preparation of blind reference material samples described in subsection 4.2.2. QC blanks should be collected in exactly the same manner as described for field blanks. Each QC blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blind reference materials prepared from the blanks can then be submitted in a blind manner to the laboratory). It is recommended that QC blanks be collected (or designated) at a frequency of 1 per 20 field samples. At a minimum, two should be collected at each sampling site (an extra should be collected to assure sufficient QC blanks are available in case problems are experienced during preparation of blind reference material samples).

- 4.2.2 Blind Reference Material Samples. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods. Prepare a blind reference material by placing an

- 5.3 Field Data, Any information collected at the sampling site.
- 5.4 Field Sample, Physical material taken from the sampling site that is targeted for lead determination.
- 5.5 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large number of lead determinations to develop a lead result known to a high degree of confidence.
- 5.6 Sample Collection Container, Container for holding and transporting the samples from the field to the laboratory. The internal volume of the container must be sufficient to hold the entire collected sample.
- 5.7 Sampling Location, Specified area within a sampling site that is subjected to sample collection. Multiple sampling locations are commonly designated for a single sampling site. An example would be at the bottom of a specific slide in a specific playground area.
- 5.8 Sampling Site, Local geographical area that contains the sampling locations. A sampling site is generally limited to an area that can be easily covered on foot. An example would be John Smith's house at 3102 Nowhere Avenue, Detroit, MI.
- 5.9 Sample Preparation, Process used to ready a sample received from the field for lead determination using instrumental measurement methods. The process is dependent on the sample type and can include a large number of steps such as homogenization, drying, splitting, weighing, digestion, dilution to a final known volume, and filtering.
- 5.10 QC Blank, See subsection 4.2.2.

