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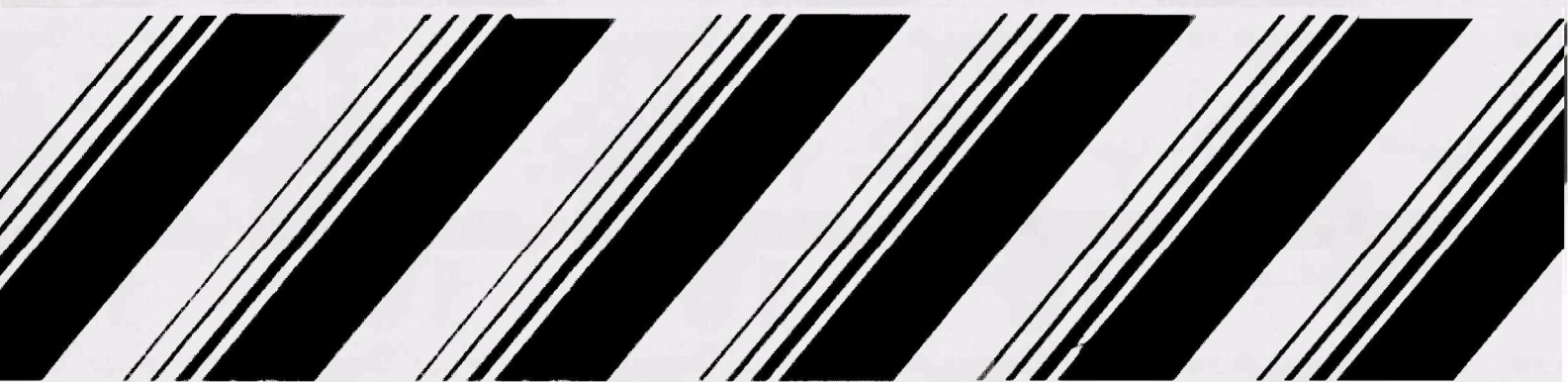
Standard Operating Procedure for Solubilization of Lead on Dust Wipes by Hotplate Acid Digestion



STANDARD OPERATING PROCEDURE FOR SOLUBILIZATION OF LEAD ON DUST WIPES BY HOTPLATE ACID DIGESTION

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1.0 PRINCIPLE AND APPLICABILITY

1.1 SCOPE AND APPLICATION

The adverse health effects resulting from exposure of young children to environmental lead have received increasing attention in recent years. Studies have shown that chronic exposure even to low levels of lead can result in impairment of the central nervous system, mental retardation, and behavioral disorders.[1,2] Although young children are at the greatest risk, adults may suffer harmful effects as well.[3]

As a result of the growing concern about these adverse health effects, the identification and assessment of hazards from lead-based paint (LBP) and LBP-containing dust and soil have become critical environmental issues. To quantify the magnitude of these hazards and to develop and implement strategies for their reduction, methods for measuring lead in paint, soil, and dust must be developed and validated.

Measuring lead in dust involves collecting the dust, in most cases extracting the lead from the dust, and finally instrumentally measuring the extracted lead. Current methods of dust collection include vacuuming,[4,5] which may result in the collection of bulk dust or dust on filters, and surface collection using a wipe. A large number of wipe methods have been developed, several of which are described in Table 1. Protocols for evaluating wipes have been published, including the protocol by Chavalitnikul and Levin.[19] Procedures have been developed for measuring lead in dust[20,21] and lead in dust on air filters.[22,23] This standard operating procedure (SOP) describes a hotplate acid digestion of lead in dust collected on wipes. This procedure is based on a method provided by Ms. Harriotte Hurley, formerly of Azimuth Laboratories, Charleston, SC.[24]

1.2 SUMMARY OF METHOD

1.2.1 Sampling and Analysis

The dust wipe sample collection procedures vary considerably (See Table 1). The basis of each method, however, is to manually move the wipe material (natural or synthetic fabric, paper, etc.) across a fixed area of surface. The material may or

TABLE 1. WIPE SAMPLING METHODS

Method/ Researcher	Surface Type	Wipe Matrix	Area	Sampling Procedure	Container	Units	Reference
ASTM ES-30-94	Hard (floors, windows)	Disposable towelette moistened with wetting agent	Template 30 cm x 30 cm (12" x 12") measured area	• Wipe with "S" motion L ↔ R, F → B • Fold wipe • Repeat with "S" motion F ↔ B, L → R, • Fold wipe • Repeat with "S" motion L ↔ R, F → B	Hard-walled container recommended	$\mu g/cm^2$	6
Georgia Tech	Hard (floors, windows)	Little Ones Baby Wash Cloths® (K-Mart)	Measured area	• Wipe with "S" motion F → B, L ↔ R • Fold wipe • Repeat with "S" motion F ↔ B, L → R • Fold wipe • Repeat with "S" motion L ↔ R, F → B	Centrifuge tube (50 mL)	$\mu g/ft^2$	7
HUD	Hard (floors, windows)	Commercial non-alcohol wipes	Template 12" x 12", or measured area	• Wipe with "S" motion L ↔ R, F → B, • Fold wipe inside • Wipe with "S" motion 90° to first wipe • Fold wipe • Repeat with "S" motion L ↔ R, F → B	Polypropylene tube (50 mL)	$\mu g/ft^2$	8
Kennedy-Krieger (Farfel)	Hard	Non-alcohol wipes	Template 12" x 12", or measured area	• Wipe with "S" motion L ↔ R, F → B, • Fold wipe inside • Repeat with "S" motion F ↔ B, L → R • Fold wipe • Repeat with "S" motion L ↔ R, F → B	Ziploc bags	$\mu g/ft^2$	9
Lepow (1974)	Hard	Preweighed adhesive labels	---	---	---	$\mu g/g$	10

(continued)

TABLE 1. (Continued)

Method/ Researcher	Surface Type	Wipe Matrix	Area	Sampling Procedure	Container	Units	Reference
Rabinowitz	Hard	Preweighed filter paper (Whatman 541)	930 cm^2	---	Resealable polyethylene bags	μg/wipe	11
LWW	Hard	3 polyethylene filters (preweighed) 3.8 cm x 6.35 cm	Template 27.3 cm x 4 cm	• Wipe within template with filter 1 (moistened with Type I water) • Wipe within template with filter 2 (moistened with Type I water)* • Wipe within template with filter 3 (dry)	Aluminum pouch inside of plastic bag	$\mu g/ft^2$ $\mu g/g$	12
NIOSH	Hard	• Gauze 2" x 2" wetted with 1-2 mL distilled water* • Wash'n Dri® (Canaan Products, Inc.) wipes (individually wrapped) or equivalent	10 cm x 10 cm disposable template	• Wipe with "S" motion 3-4 vertical strokes • Fold wipe inside • Wipe with "S" motion 3-4 horizontal strokes • Fold wipe inside • Wipe with "S" motion 3-4 vertical strokes	Ziploc bag	$\mu g/ft^2$ (mg/m^2)	13 14
Stark	Hard	Prewetted cotton gauze	---	---	---	$\mu g/g$	15
State of MA	Hard (floors, windows)	Individually wrapped alcohol wipes (Triad Medical, Inc.)	Template 1 ft^2	Wipe area once L → R	Plastic tube	$\mu g/ft^2$	16
State of NC	Hard (floors, windows)	Johnson & Johnson Baby Wipes	Template 1 ft^2 or measured area	• Wipe with "S" motion • Wipe with "S" motion 90° to first pass	Ziploc bag	$\mu g/ft^2$	17

(continued)

TABLE 1. (Continued)

Method/ Researcher	Surface Type	Wipe Matrix	Area	Sampling Procedure	Container	Units	Reference
Vostal	Hard (uncarpeted)	Disposable paper towel (14 cm x 20 cm) moistened with 20% denatured alcohol or 1:150 benzalkonium chloride	Template $1ft^2$	---	---	$\mu g/ft^2$	18

LEGEND:

L ↔ R = Left to Right, Right to Left in Overlapping "S" Motion
F → B = Front to Back (Top to Bottom)
* American Society for Testing and Materials (ASTM) Type 1 water: resistance ≥ 16.67 megaohm-cm

may not be pretreated with liquid solubilization or wetting agents (detergents). The wipe is folded to enclose the collected dust and may be used to wipe a second or even third time before being placed in a labelled container or bag.

Using the digestion method described in this SOP, the loaded dust wipe is placed into a 250-mL beaker and digested using 30% hydrogen peroxide and concentrated nitric acid. The digestion mixture is quantitatively transferred to a 50-mL polyethylene centrifuge tube, and the tube is filled to the mark with doubly deionized water. The residue is separated from the supernatant by centrifugation or simply by permitting it to settle overnight. The supernatant is analyzed for lead by inductively coupled argon plasma (ICP) emission spectrometry, using the 220.35-nm emission line and the optimum instrumental conditions recommended by the manufacturer.[25]

1.2.2 Precision and Bias

As noted in Table 1, many different types of wipes are commercially available. In this study, three types of wipes were selected for testing the wipe extraction procedure because (1) they were representative of the various types in use and (2) they were used in methods provided by Federal organizations or proposed for use in federal programs. Included were a baby wipe used in a procedure published in the U.S. Department of Housing and Urban Development's (HUD's) Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing,[8] a wipe used in a procedure proposed by the National Institute for Occupational Safety and Health (NIOSH),[13] and a Nuclepore® filter used in a procedure known as the Lioy-Weisel-Wainman (LWW) method proposed for use in the EPA National Human Exposure Assessment Survey (NHEXAS).[12,26]

The three types of dust wipes (Little Ones Baby Wash Cloths®,[7,27] Wash'n Dri Moist Disposable Towelettes®,[13,28] and Nuclepore® filters[12,29]) were spiked with standard reference materials (SRMs) and method evaluation materials (MEMs) and digested using the hotplate procedure summarized in Section 1.2.1. The reference materials used included National Institute of Standards and Technology (NIST) SRM

1579, powdered lead-base paint; NIST SRM 2704, Buffalo River sediment; and two dust MEMs from the American Industrial Hygiene Association's Environmental Lead Proficiency Analytical (ELPAT) program.[30]

Accuracy as percent bias and precision as relative standard deviation (RSD) were determined and are presented in Tables 2 through 4.[31] Accuracy as percent bias ranged from -8.7% to +3.7% and precision as RSD ranged from 2.54% to 11.7%. The average bias was -2.9% and the pooled RSD was 7.8%.

TABLE 2. Dust Wipe Hotplate Digestion - K-Mart Little Ones Baby Wash Cloths® Spiked with 0.100 g Reference Material (μg/g Except Where Noted)

Sample	Reference Value	Mean Value ± SD (N=3)	Accuracy as % Bias	RSD %
NIST 1579	11.87 ± 0.04%	11.9 ± 0.32%	0	2.69
NIST 2704	161 ± 17	147 ± 14.1	-8.7	9.60
ELPAT 3D2	551 ± 55[a]	516 ± 27.5	-6.8	5.33
ELPAT 3D4	2210 ± 401[a]	2150 ± 247	-2.7	11.5

[a] ELPAT program mean value of 34 reference laboratories.[30]

Table 3. Dust Wipe Hotplate Digestion - Wash'n Dri Moist Disposable Towelettes® Spiked with 0.100 g Reference Material (μg/g Except Where Noted)

Sample	Reference Value	Mean Value ± SD (N=3)	Accuracy as % Bias	RSD %
NIST 1579	11.87 ± 0.04%	11.5 ± 0.35%	-3.4	3.04
NIST 2704	161 ± 17	167 ± 19.5	3.7	11.7
ELPAT 3D2	551 ± 55[a]	537 ± 13.6	-2.5	2.54
ELPAT 3D4	2210 ± 401[a]	2110 ± 222	-4.5	10.5

[a] ELPAT program mean value of 34 reference laboratories.[30]

TABLE 4. Dust Wipe Hotplate Digestion - Nuclepore® Filters Spiked with 0.100 g Reference Material (μg/g Except Where Noted)

Sample	Reference Value	Mean Value ± SD (N=3)	Accuracy as % Bias	RSD %
NIST 1579	11.87 ± 0.04%	10.9 ± 1.11%	-8.4	10.2
NIST 2704	161 ± 17	162 ± 6.11	0.6	3.77
ELPAT 3D3	551 ± 55[a]	543 ± 45.1	-1.4	8.30
ELPAT 3D2	2210[b] ± 401[a]	2190 ± 75.8	-0.9	3.46

[a] ELPAT program mean value of 34 reference laboratories.[30]

2.0 APPARATUS

The apparatus used for sampling and analysis of the dust wipes is described below.

NOTE: Before use, all labware should be scrupulously cleaned. The recommended procedure is:

1. Wash with hot laboratory detergent in water or ultrasonicate 10 minutes with laboratory detergent in water.
2. Rinse and then soak a minimum of 1 hour in 50% volume/volume (v/v) nitric acid.
3. Rinse three times with double deionized water and air dry or oven dry at 105°C.

2.1 SAMPLING

The dust collection procedures referenced in this SOP use the materials and apparatus as follows:

2.1.1 HUD Method Using Baby Wipe[7,27]

2.1.1.1 Disposable baby wipe (Little Ones Baby Wash Cloths® or equivalent).

2.1.1.2 Gloves, latex, powder-free, disposable (Fisher Scientific Catalog No. 11-393-53 or equivalent).

2.1.1.3 Centrifuge tubes: 50-mL disposable polypropylene (Fisher Scientific Catalog No. 05-526B or equivalent).

2.1.1.4 Template: either masking tape or reusable made of laminated paper, metal, or plastic; typically 1 ft^2 (929 cm^2) to enclose the area to be wiped.

2.1.2 NIOSH Method Using Gauze or Hand Wipe[13,28]

2.1.2.1 Gauze pads, 2" x 2" sterile cotton (Curity, Johnson & Johnson, or equivalent), or Wash'n Dri Moist Disposable Towelettes® or equivalent.

2.1.2.2 Bags, plastic, sealable (for example, with attached wire, tape, or zip-type seal); (Ziploc 1-quart freezer bags or equivalent).

2.1.2.3 Gloves, latex, powder-free, disposable (Fisher Scientific - see Section 2.1.1.2 or equivalent).

2.1.2.4 Template, plastic, 10 cm x 10 cm or other standard size to enclose the area to be wiped.

2.1.2.5 Water, distilled, ASTM Type 1, in plastic squeeze bottle.

2.1.3 LWW Method Using Nuclepore® Filter[12,29]

2.1.3.1 Nuclepore® filter preweighed and stored in folded aluminum foil pouch (Nuclepore® Catalog No. 232300, Costar Corp., Cambridge, MA 02140).

2.1.3.2 Wipe sampler template with sampling area of 27.3 cm x 4 cm.

2.1.3.3 Wipe sampler movable plate-filter retainer consisting of two sections: 1) a 4-cm square block that is 7-mm thick and 2) a 5.1-cm x 4.7-cm frame.

2.1.3.4 Gloves, latex, powder-free, disposable (Fisher Scientific - See Section 2.1.1.2 or equivalent).

2.1.3.5 Bags, plastic, sealable (for example, with attached wire, tape, or zip-type seal); (Ziploc 1-quart freezer bags or equivalent).

2.2 EXTRACTION

The applicability of this method has been tested with three of the most commonly used lead-in-dust collection wipes (Section 1.2.2) Applicability of this method to other types of wipes will be verifiable through quality control (QC) activities (sections 3.2 and 4.0). The apparatus and reagents used in this procedure are as follows:

2.2.1 Hotplate with temperature control from 70° to 200° Celsius (Corning 6795, Fisher Scientific Catalog No. 11-495-52A or equivalent).

2.2.2 Beakers, 250-mL (borosilicate glass, Fisher Scientific Catalog No. 02-540K or equivalent).

2.2.3 Disposable wipes for preparation of laboratory control standards (LCS). These wipes are to be selected at random from the set to be used for sample collection.

2.2.4 Electronic top-loading balance for preparation of LCSs (Mettler AJ 100 balance, Mettler Instruments Corp., P.O. Box 71, Highstown, NJ 08520, or equivalent).

2.2.5 Pipetter, 200- to 1000-μL (Oxford Benchmate, Fisher Scientific Catalog No. 21-229 or equivalent).

2.2.6 Glass (4-mm) rod to push wipe into beaker.

2.2.7 Forceps, Nalgene polypropylene scissor-type (Fisher Scientific Catalog No. 10-309 or equivalent).

2.2.8 Hydrogen peroxide: 30% H_2O_2, volume/volume, ACS reagent grade.

2.2.9 Nitric acid: Concentrated, ACS spectrographic grade.

2.2.10 Watch glass, ribbed, borosilicate glass (Fisher Scientific Catalog No. 02-613B or equivalent).

2.2.11 Centrifuge tubes: 50-mL disposable polypropylene (Fisher Scientific Catalog No. 05-526B or equivalent).

2.2.12 Centrifuge (International Equipment Company Model CL or equivalent).

2.2.13 Deionized water in dropper or squirt bottle: Unless otherwise indicated, references to deionized water shall be understood to mean reagent water as defined by Type 1 of ASTM Specification D 1193[32] (ASTM Type 1 Water: minimum resistance of 16.67 megohm-cm or equivalent).

2.2.14 NIST Traceable Secondary Reference Materials for LCS. If these are not

available, use a NIST SRM such as NIST SRM 1648 (Urban particulate matter); SRM 2583 (Lead in dust); SRM 2704 (Buffalo River sediment); or SRM 1579 or 2582 (Lead in paint).

2.2.15 Automatic pipettor: 10-mL capacity, 0.1-mL subdivision (Universal Repipet Dispenser, VWR Scientific, 1230 Kennestone Circle, Marietta, GA 30066, Catalog No. 53528-168, or equivalent).

2.2.16 Two- and three-mL Class A volumetric pipettes.

2.2.17 Chemical fume hood, Kewaunee Scientific Equipment Corp., Adrian, MI 49221, Airflow Supreme Model.

2.3 PROTECTIVE EQUIPMENT

Safety equipment to be used for protection from both lead and biological contamination during dust collection and wipe extraction is as follows:

2.3.1 If significant airborne dust is present or being generated by the collection activity, wear a fitted respirator (Occupational Safety and Health Administration (OSHA) Standard 29 CFR 1910.134) for dust collection (half-mask respirator, Fisher Scientific Catalog No. 17-632 [size], and high-efficiency particulate air (HEPA) filter/cartridge, Fisher Scientific Catalog No. 17-635-7, or equivalent).

2.3.2 Disposable booties, coveralls, and head covers for dust collection in high-lead environment (coveralls with shirt collar, tacked-on hood, elasticized wrists, bound seams, Fisher Scientific Catalog No. 01-361-51 [size], or equivalent).

2.3.3 Safety glasses with side shields for wipe extraction.

2.3.4 Laboratory gloves (See Section 2.1.1.2) and coat for wipe extraction.

2.3.5 Dust mask to be worn while handling dust wipes (mask for dusts and mists, NIOSH/OSHA TC-21C-132; Fisher Scientific Catalog 17-653 or equivalent).

2.3.6 Heavy-duty plastic bags (6-mil or greater) for disposal of personal protection products and dust collection waste (IPCO, Inc., Catalog No. R335OBP, IPCO, Inc., Norcross, GA 30091, or equivalent).

2.3.7 Detergent (heavy-duty cleaner with sodium metasilicate; Bondex International, Inc., St. Louis, MO 63122, or equivalent) and wet wipes for cleanup (Wash 'n Dri®, Softsoap Enterprises, Inc., Chasha, MN 55318, or equivalent).

2.3.8 Filter for waste water filtration: 20-μm pore size (Spectra/Mesh Nylon; Fisher Scientific Catalog No. 08-670-204 or equivalent).

3.0 PROCEDURES

3.1 COLLECTION OF MATERIAL

As stated earlier (Section 1.1), a variety of wipe methods is described for collection of dust on surfaces. Check the references for complete descriptions of operating procedures. Three methods chosen for testing are described below:

3.1.1 HUD Method Using Baby Wipes[8] - The HUD method uses a disposable wipe material meeting the following criteria: a) low lead background (less than 5 μg/wipe[14]); b) a single thickness; c) durable; d) does not contain aloe or alcohol; e) can be digested in the laboratory; f) yields 80-120% spike recovery rates; and g) remains moist during the sampling process. A template of paper, metal, or plastic, typically with a sampling area of 1 ft^2, is placed on the surface to be sampled.

The wipe is placed at one corner of the template and wiped side to side with as many horizontal "S" strokes as necessary to cover the entire template area. The wipe is folded in half with the contaminated side facing in and placed in the template top corner, and the wiping is repeated but in a top-to-bottom direction. Finally, the side- to-side wiping is repeated. When all the visible dust is removed, the wipe is inserted into a labelled centrifuge tube.

3.1.2 NIOSH Method Using Gauze or Hand Wipe[13] - The NIOSH method uses a 2" x 2" gauze pad or a Wash'n Dri® towelette and a plastic template with a sampling area of 10 cm x 10 cm. The template is placed over the area to be sampled, and the surface is wiped using three to four vertical "S" strokes. The wipe is folded with the exposed side in, and the area is again wiped with three to four horizontal "S" strokes. Finally, the wipe is folded once more and the area wiped with three to four vertical "S" strokes. The wipe is then folded with the exposed sides in and placed into a labelled plastic bag.

3.1.3 LWW Method Using Nuclepore® Filter[12,26] - The LWW method is a new dust wipe sampling technique utilizing the LWW sampler. The sampler consists of two sections constructed from Delrin®: (1) a movable plate-filter retainer consisting

of a 4-cm square block 7 mm thick and a 5.1-cm x 4.7-cm frame that holds the 4-cm block; and (2) a template 3 mm thick with a sampling area of 27.3 cm x 4 cm. The frame plus block traces the rectangular opening within the template.

For sampling, a Nuclepore® filter cut into a rectangle of 3.8 cm x 6.35 cm is preweighed, folded, and placed into the sample frame so that the filter edges are touching the frame sides. The 4-cm square block is inserted into the frame to hold the filter in place. The filter is wetted with deionized water and slid across the length of the template. It is passed back and forth three times, removed from the plate-filter retainer, and placed into an aluminum foil pouch.

3.2 QUALITY CONTROL SAMPLES

3.2.1 Blank - If a blank wipe was not submitted from the field, initiate a laboratory blank by placing a clean, unused wipe in a clean, labelled 250-mL glass beaker. The filter should come from the same lot as those used to collect the samples. Record the sample number in the laboratory notebook.

3.2.2 Quality Control Check - Initiate a QC check by placing a wipe spiked with 250 μg of lead in a clean, 250-mL beaker.

Prepare the spiked wipe as follows:

3.2.2.1 -- Place an unused, clean wipe in a clean 250-mL glass beaker.

3.2.2.2 -- Using a 500-μL micropipette, measure 250 μL of commercial 1000-μg/mL Pb standard onto the wipe.

3.2.2.3 -- Allow the wipe to air dry.

3.2.3 Laboratory Control Standard - Initiate an LCS by using the calibrated Mettler balance to carefully weigh out an NIST-traceable reference material (Section 2.2.1.4) on a piece of clean glassine weighing paper. Record the exact weight. Quantitatively transfer the entire amount into a clean, unused dust wipe. Fold the dust wipe so that all the weighed material is contained, and place it in a clean, labelled 250-mL beaker. Record the sample number in the laboratory notebook. For example,

to prepare a 1000 μg Pb LCS, 0.1527 g of a 6550 μg/g reference material would be used.

3.3 EXTRACTION PROCEDURE

The extraction process should be conducted in a chemical fume hood.

3.3.1 Carefully remove the field sample wipe from its container using Teflon® tipped tweezers or clean, gloved fingers.

3.3.2 Place the wipe in a clean, labelled 250-mL glass beaker and push down with an acid-washed glass rod.

3.3.3 Using a volumetric pipette, add 3 mL of 30% hydrogen peroxide.

3.3.4 Add 20 mL of concentrated nitric acid using the automatic pipettor. Use the glass rod to mix and agitate the wipe and extraction reagents until the wipe is thoroughly wetted. Withdraw the glass rod, rinsing it with about 1 mL of deionized water. Allow the sample to sit until any visible reaction ceases.

3.3.5 Place the beaker on a hotplate and cover with a ribbed watch glass.

3.3.6 Turn on the hotplate to the level necessary to achieve a slow reflux. The temperature setting required to achieve a slow reflux will depend upon the type of wipe being extracted, the temperature of the laboratory, and the model of the hotplate. Do not allow the extraction solution to boil.

3.3.7 After 20 minutes, cautiously add 2 mL of 30% hydrogen peroxide.

3.3.8 Continue to reflux until the volume has decreased and the sample is nearly dry. Do not allow to evaporate completely. There must be some liquid left to ensure that the Pb is not lost through splattering or formation and loss of aerosols.

3.3.9 Add 5 mL of concentrated nitric acid and repeat the reflux step to near dryness.

3.3.10 Carefully remove the beaker from the hotplate and allow to cool.

3.3.11 Using a volumetric pipette, add 5 mL of concentrated nitric acid to the sample beaker.

3.3.12 Using a squirt bottle or dropper, rinse the watch glass with 3 - 5 mL of deionized water, being careful to catch all the rinsate in the sample beaker.

3.3.13 Carefully pour the sample liquid into a clean, labelled, 50-mL polyethylene centrifuge tube. If any undissolved material remains in the beaker, press the wipe material down using a clean glass rod to extract as much solution as possible.

3.3.14 Using a squirt bottle or dropper and small quantities of deionized water, carefully rinse all interior surfaces of the beaker and pour the rinsate into the same centrifuge tube that contains the sample. Repeat the rinse step at least three times, but do not exceed a final volume of 50 mL.

3.3.15 Dilute to the mark on the centrifuge tube with deionized water to achieve a final volume of 50 mL. This dilution results in a nitric acid concentration of approximately 10%.

3.3.16 Allow any undissolved particulate matter to settle out overnight, if necessary, or centrifuge at 2500 rpm for 25 minutes. If the extract is not to be analyzed immediately, decant the solution into a separate clean, labelled centrifuge tube.

3.3.17 The sample is now ready for lead analysis using the selected instrumental technique, atomic absorption spectroscopy (AAS) or ICP.[21,25]

3.4 DATA PROCESSING

AAS or ICP analysis will result in values of μg Pb/mL for the extracts. To convert from μg Pb/mL to μg Pb/wipe, use this formula:

$$\mu\text{g Pb/wipe} = \mu\text{g/mL measured} \times \text{DF1} \times 50\ \text{mL/wipe}$$

where DF1 = any dilution performed prior to analysis due to high lead concentrations.

4.0 QUALITY CONTROL

4.1 SAMPLE COLLECTION

Check original references for details of QC procedures for sample collection. General procedures to be applied include the following:

4.1.1 Only use wipes that are uniform from one to another in size, thickness, and appearance.

4.1.2 Only use wipes that, from wipe to wipe, appear to have similar loadings of solubilization or wetting agents.

4.1.3 Avoid contamination of wipes before and after use.

4.1.4 Make all reasonable attempts to maintain constant and uniform pressure while using an individual wipe and from wipe to wipe.

4.1.5 Maintain uniform wiping motions from wipe to wipe.

4.1.6 Place wipes in labelled containers.

4.2 WIPE EXTRACTION

4.2.1 Remove used wipes from containers carefully and deliberately to avoid dust sample loss.

4.2.2 Immerse each wipe as completely as possible in extraction reagents.

4.2.3 Do not allow extraction solution to boil as this may result in splattering and sample loss.

4.3 QUALITY CONTROL SAMPLES

Preparation of the QC samples is described in Section 3.2. Criteria for the extraction procedure being under control are as follows:

4.3.1 Blank - The blank wipe shall contain $\leq$ 5 μg Pb. If a higher level is measured, repeat the blank analysis. If this blank is also $>$ 5 μg Pb per wipe, either identify a new source of wipes that have blank values $\leq$ 5 μg Pb per wipe and discard the current batch, or make replicate measurements of the blank (n = 5 to 7) and calculate an average blank value. Report this value with the field sample results.

4.3.2 Quality Control Check - The QC check shall yield a recovery of $>$ 90%. If the recovery is $<$ 90%, repeat the test. If the repeat value is also $<$ 90%, identify the source(s) of error, including, for example, overheating of the digestion beaker or miscalibration of the measurement instrument, and make appropriate corrections.

4.3.3 Laboratory Control Standard - The LCS shall yield a recovery between 80 and 120% relative to that which would be achieved with direct HNO_3/H_2O_2 digestion of the reference material. If the recovery is outside these limits, repeat the test. If the recovery remains outside the 80 to 120% window, either (1) identify the source(s) of error and make appropriate corrections, or (2) make replicate measurements of the LCS (n = 5 to 7) and calculate an average recovery value. Report this value with the field sample results.

5.0 SAFETY

5.1 COLLECTION

Appropriate safety procedures must be followed during collection of dust material from paint abatement sites or other sites that might have high airborne lead levels. Collection from areas where paint chips and dust could contaminate a person requires full-body protection to protect the worker and to prevent take-home lead contamination. This includes wearing disposable booties, coveralls, head cover, and gloves during collection. A respirator and safety glasses with side shields or goggles must also be worn. At the end of the collection period, remove the disposable safety wear in a relatively clean area and place it in a plastic bag for disposal (Section 6.0). After collecting the dust samples, take a shower as soon as is practically possible.

If worker contamination is not a potential problem, gloves still need to be used to prevent contamination of the samples. In some cases, the use of booties may also be needed.

5.2 ANALYSIS

Take normal laboratory precautions when digesting the dust wipe samples. Wear gloves, a laboratory coat, and safety glasses with side shields when performing acid-based extractions. Also, follow the manufacturer's instructions for safe usage of the ICP or AAS.

6.0 WASTE DISPOSAL

6.1 PROTECTIVE CLOTHING

Disposable protective clothing including dust masks and spent respirator dust filter cartridges will generally not be categorized as hazardous waste because they typically pass the EPA Toxicity Characteristic Leaching Procedure (TCLP).[33] If there is any uncertainty about the characterization of these materials, subject them to the TCLP. This procedure involves an extraction in an aqueous solution at constant pH in a closed vessel for 18 ± 2 hours followed by analysis of the solution for lead. If the extracted lead exceeds 5 mg/L, consider the material a hazardous waste, and place it in a State-licensed or -permitted hazardous waste landfill. If this level is not exceeded, place the material in a municipal landfill.

6.2 WASH WATER

Filter wash water using a 20-μm pore size filter (Section 2.3.8) and allow to drain into a sanitary sewer system. A coarse screen may be used as a prefilter.

6.3 EXCESS DUST WIPE SAMPLES

Subject excess dust wipe samples to the TCLP and treat them as hazardous waste should the extract exceed the allowed limit of 5 mg/L.

6.4 SOLUTION WASTE

Pour all leftover extract solutions, reagent wastes, and rinse water into a plastic carboy. Rinse with water any centrifuge tubes or other vessels containing nitric acid as one of the reagents before putting them into a plastic bag for disposal or reuse after cleaning. Dispose of waste solutions according to applicable regulations.

7.0 CORRECTIVE ACTION

See Section 4.3 for corrective actions in response to unacceptable QC sample analysis results.

Corrective action for the measurement of lead is described in the "Standard Operating Procedure for Lead in Paint by Hotplate- or Microwave-Based Acid Digestion and Atomic Absorption of Inductively Coupled Plasma Emission Spectrometry."[25]

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