



Ultrasonic extraction and field-portable anodic stripping voltammetric measurement of lead in dust wipe samples

Kevin Ashley^{a,*}, Tamara J. Wise^a,
Wandaliz Mercado^{a,1}, Diane B. Parry^b

^a *US Department of Health and Human Services Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, Cincinnati, OH 45226, USA*

^b *Procter and Gamble Company, Ivorydale Technical Center,
5299 Spring Grove Avenue, Cincinnati, OH 45217, USA*

Abstract

Dust wipe samples were subjected to ultrasonic extraction (UE) in diluted nitric acid, and then analyzed for lead content using field-portable anodic stripping voltammetry (ASV). Recoveries of lead were determined from wipe materials which were spiked with certified reference materials (CRMs) containing known quantities of lead. Four different wipe materials and four different CRMs were tested, with and without filtration of aliquots of sample extract through 0.45 μm hydrophilic polytetrafluoroethylene filters. The CRMs consisted of paint, soil, particulate, and dust matrices. Wipe materials were chosen from those which have been found to meet the performance aspects of an ASTM standard specification. UE/ASV experiments were carried out in accordance with newly published ASTM procedures for on-site extraction and electroanalysis. Recoveries were found to vary for different wipe materials and CRMs. For several CRMs, quantitative (80–120%) recoveries for UE/ASV were observed for one wipe material whether filtration was used or not, while other wipe materials required filtration for quantitative recovery. In the case of one wipe material which contained detergents, quantitative recoveries could not be achieved whether filtration was used or not. The total analysis time for a sample set of 6–12 samples was 60–90 min, including extraction time and sample manipulation. The results of this work have provided information on the choice of wipe materials that can be used for quantitative lead measurements by UE/ASV in materials that are representative of sources of lead in surface dust. Published by Elsevier Science B.V.

Keywords: Anodic stripping voltammetry; Ultrasonic extraction; Wipes; Lead; On-site analysis

* Corresponding author. Tel.: +1-513-841-4402; fax: +1-513-841-4500.

E-mail address: kashley@cdc.gov (K. Ashley).

¹ Hispanic Association of Colleges and Universities summer student intern, Universidad Interamericana, San German, Puerto Rico.

1. Introduction

Dust wipe samples are used widely to assess lead contamination in surface dust present in residences and in occupational settings [1]. The laboratory analysis of dust wipes for lead content typically requires hot plate extraction in a concentrated acid solution [2], followed by atomic spectrometric determination of lead [3]. However, it is often desired to have analysis results rapidly available on-site in the field, thereby avoiding the need for samples to be sent to a fixed-site laboratory for analysis. The capability to conduct lead measurements in the field would allow for rapid assessment of potential lead hazards to workers, children, or others. On-site analysis results would enable corrective actions to be taken rapidly, such as cleaning prior to vacating a job site [4]. Thus, user-friendly field-portable methods for determining lead in dust wipe samples are desired.

In this study, an on-site analytical method involving the use of ultrasonic extraction (UE) of dust wipe samples in diluted nitric acid, followed by field-portable anodic stripping voltammetry (ASV) for the measurement of lead, was investigated. Previous studies of UE/ASV on environmental certified reference materials (CRMs) [5] and workplace air [6] samples have demonstrated the potential of this method for field-based measurements of lead in a variety of sample matrices. The UE/ASV procedure has also been evaluated in the field for the measurement of lead in paint film samples [7]. However, the UE/ASV technique has not yet been fully evaluated for the determination of lead in dust wipe samples, although the method was shown to give promising results in an interlaboratory evaluation [8].

For the purposes of this study, wipes were chosen which have been found to meet the performance requirements of an ASTM specification for the collection and analysis of lead in surface dust [9]. The wipes were spiked with CRM paint, soil, particulate, and dust, and were then subjected to ultrasonic extraction in accordance with a new ASTM procedure [10]. Extracts of the sonicated wipes were then analyzed for lead content by portable ASV in accordance with an ASTM procedure [11]. For purposes of comparing ASV results to data from atomic spectrometry, aliquots of some wipe ultrasonic extracts were also analyzed for lead content by inductively coupled plasma-atomic emission spectrometry (ICP-AES) [12]. Owing to the newness of the ASTM procedures, performance data were lacking for UE/ASV as applied to dust wipe samples; hence the need to fill this knowledge gap by performing the present study.

A wipe presents a challenging matrix for on-site analysis by UE/ASV, due to the large amount of organic material comprising the wipe, and the possible presence of surfactants and other potential interferants within the wipe. Preliminary studies suggested that filtration of aliquots from extracts of wipes for analysis might alleviate potential interferences to ASV analysis by organic material, surfactants, etc. Therefore, experiments were conducted to investigate whether filtration with hydrophilic polytetrafluoroethylene (PTFE) filters enhanced method performance over UE/ASV lead determinations in the absence of filtration.

2. Experimental

2.1. Dust wipes

Wipe materials, which were chosen for this study, were those which had been found to meet analytical performance requirements of ASTM Specification E1792 [8,13]. This standard specification covers a number of aspects, most importantly: (1) performance requirements for the collection efficiency of wipe materials used to sample for lead in surface dust [14]; (2) specifications for thickness, width, and length; (3) demonstrated ruggedness during use for sampling; (4) requirements for moisture content and uniformity; (5) a requirement for the absence of background lead within the wipe and (6) a requirement for demonstrated quantitative lead recoveries using reference sample preparation and analytical methods [15]. The four wipes chosen for this work were Wash'n Dri[®] (Softsoap Enterprises, Chaska, MN; lot #942103), Pace Wipe[™] (Pace Environs, Cary, NC; lot #1296-01), Dust Wipe (Palintest USA, Erlanger, KY; lot #12974321), and Lead Wipe[™] (AramSCO/Lynx Products, Thorofare, NJ; lot #2OD365).

2.2. Certified reference materials

The CRMs used included three National Institute of Standards and Technology (NIST) Standard Reference Materials[®] (SRMs) (NIST, Gaithersburg, MD): (1) SRM #1579a, Lead-based paint (11.87% Pb by mass); (2) SRM #1648, Urban particulate (0.655% Pb by mass) and (3) SRM #2704, Buffalo river sediment (0.0161% Pb by mass). A fourth CRM used was Bag House Dust (Fisher Scientific, Fair Lawn, NJ; #SRS014-50) (0.1914% Pb by mass). For SRMs 1579a and 1648, and also for CRM SRS014-50, ~0.05 g of material was loaded onto each wipe; for SRM 2704, about 0.5 g of material was spiked onto each wipe. The CRMs were weighed to the nearest ± 0.0001 g on an analytical balance (Mettler Model AE163, Greifensee, Switzerland). The spiking procedure consisted of unfolding each wipe and depositing a weighed amount of CRM onto the center of the wipe. Wipes were then refolded to contain CRM within the wipes. In some cases, CRMs alone (i.e. not spiked onto wipes) were weighed for subsequent UE/ASV analysis (in similar amounts as were weighed onto wipes). An aqueous lead standard (1000 $\mu\text{g Pb/ml}$, J.T. Baker, Philipsburg, NJ) was used for the preparation of calibration check solutions by serial dilution with ASTM Type I water [16] (Barnstead Nanopure[™] system, Thermolyne, Dubuque, IA).

2.3. Ultrasonic extraction

The UE procedure was carried out in accordance with ASTM Practice E1979 [9]. Wipes spiked with CRMs (or alternatively CRMs only, i.e. no wipe) were placed into 50 ml Falcon[™] polypropylene centrifuge tubes (Becton Dickinson, Lincoln Park, NJ). Instrumental grade nitric acid (15 ml of 25% (v/v)) (EM Science, Gibbstown, NJ) was then introduced into each centrifuge tube by means of a mechanical precision pipet (Eppendorff, Hamburg, Germany). Wipes were pushed to the bottoms of the tubes using clean plastic or glass stirring rods, and were compressed in order to remove bubbles. Blank wipes (unspiked with CRMs) were treated in the same fashion.

The centrifuge tubes containing blank or CRM-spiked wipes, or CRMs only, were capped and placed into a 110 W ultrasonic bath (Sonicor Model SC-150/H, Farmingdale, NY), with the water level in the bath at least 2.5 cm above the liquid level within the tubes. The sonicator was then activated for a period of 20–25 min with no deliberate heating. The centrifuge tubes were then opened, and the wipes were compressed with clean plastic or glass rods in order to remove bubbles that may have accumulated within the wipes during sonication. The tubes were then re-capped, and the wipes were again subjected to ultrasonic agitation for a second 20–25 min period (for a total extraction time of 45 min). The centrifuge tubes were then removed from the ultrasonic bath, shaken vigorously, and allowed to cool to room temperature before final dilution to 50 ml with water. The bath temperature was $\sim 40^{\circ}\text{C}$.

2.4. Portable ASV analysis

The ASV analysis was carried out in accordance with ASTM E2051 [10]. The portable ASV device used was a Palintest[®] SA5000 instrument (Palintest USA, Erlanger, KY), which weighs about 400 g. This instrument is dedicated to lead analysis, is powered by eight AA batteries, and employs disposable screen-printed electrodes [17] (Type SE-1, Palintest USA). The disposable electrodes utilize a proprietary screen-printed three-electrode design, with each electrode being used for a single analysis. The ASV device measures the current due to the lead stripping peak at approximately -470 mV versus the Ag/AgCl reference electrode, and automatically converts this current to a measured lead concentration [18].

For field-portable ASV analysis, 5 ml aliquots of extraction solution were removed from the centrifuge tubes using a mechanical pipet (Eppendorff). For some extracted samples, 5 ml aliquots were filtered through $0.45\text{ }\mu\text{m}$ hydrophilic polytetrafluoroethylene (PTFE) filters (Gelman Acrodisc, Cole-Parmer, Vernon Hills, IL) by using 5 ml syringes (Gelman). For each sample aliquot that was filtered, the filtration was accomplished in the following manner: using the syringe, a 5 ml aliquot of the extract was removed. The PTFE filter was then attached to the syringe, and the aliquot was filtered through the filter and into a 6 ml polypropylene sample measurement vial (Palintest USA) by pushing the syringe plunger. Hydrophilic PTFE filters were used owing to their inertness to acid and their compatibility with aqueous solutions. Sample solutions (whether filtered or unfiltered) were placed into the 6 ml sample vials. An electrolyte tablet (Type SP-B, Palintest USA) was introduced into each sample vial containing a 5 ml aliquot of sample solution. Each tablet was crushed with a clean plastic or glass stirring rod, and each vial was then capped and vigorously shaken to ensure complete dissolution of the electrolyte. The electrolyte tablet contains a proprietary mixture of inert salts, oxygen scavenger and pH indicator. The sample solutions were then analyzed for lead content using the portable ASV pre-programmed instrument software and disposable electrodes. The ASV analysis time is 45 s per sample.

2.5. ICP-AES analysis

Aliquots of selected sample extracts (5 ml) were analyzed directly by ICP-AES (Jarrell-Ash Model 1160, Waltham, MA) for lead content using the 220.4 nm emission line. For certain samples, both unfiltered sample extracts, as well as extracts filtered through $0.45\text{ }\mu\text{m}$

hydrophilic PTFE filters, were analyzed by ICP-AES (for comparison with ASV results from sonicated samples).

3. Results and discussion

Observed recoveries of lead from the ASV analyses of wipes spiked with paint, soil and particulate CRMs, and from the CRMs alone, are shown in Tables 1 and 2 for unfiltered and filtered sample extract solutions, respectively. (Recall that sample extracts were prepared for subsequent analysis by ultrasonic extraction in dilute nitric acid.) Results from blank (unspiked) wipes (which were run in triplicate for each type of wipe) were below the method

Table 1

Lead recoveries from UE/ASV analysis of CRMs and CRM-spiked wipes: analysis of unfiltered sample extract solutions^a

Certified reference material				
Wipe	SRM 1579a ^b	SRM 1648 ^c	SRM 2704 ^d	CRM SRS014-50 ^e
Wash'n Dri	94.2 ± 1.6	57.9 ± 8.0	60.5 ± 10.8	69.5 ± 9.7
Dust Wipe	61.0 ± 8.7	75.5 ± 5.6	57.9 ± 13.4	119.1 ± 4.0
Pace Wipe	115.0 ± 8.0	96.1 ± 6.5	84.2 ± 1.2	106.8 ± 1.4
Lead Wipe	89.7 ± 5.5	34.2 ± 5.2	77.5 ± 10.3	69.4 ± 2.3
None	112.6 ± 5.4	91.3 ± 4.9	95.7 ± 1.5	104.4 ± 0.3

^a Mean recoveries of lead ($n = 6$) are given in percent recovery versus the certified lead content; \pm values are relative standard deviations (RSDs).

^b Lead-based paint, 11.87% Pb.

^c Urban particulate, 0.655% Pb.

^d Buffalo river sediment, 0.0161% Pb.

^e Bag House Dust, 0.1914% Pb.

Table 2

Lead recoveries from UE/ASV analysis of CRMs and CRM-spiked wipes: analysis of sample extract solutions filtered through 0.45 μ m PTFE filters^a

Certified reference material				
Wipe	SRM 1579a ^b	SRM 1648 ^c	SRM 2704 ^d	CRM SRS014-50 ^e
Wash'n Dri	118.6 ± 3.6	58.6 ± 3.9	57.0 ± 3.2	59.4 ± 12.9
Dust Wipe	125.1 ± 3.3	107.0 ± 1.6	112.2 ± 3.8	106.1 ± 6.1
Pace Wipe	112.9 ± 1.0	89.1 ± 8.4	93.3 ± 3.7	106.8 ± 4.9
Lead Wipe	106.9 ± 3.1	96.0 ± 8.5	90.1 ± 2.0	76.1 ± 1.6
None	104.4 ± 1.5	91.2 ± 3.6	94.1 ± 2.6	101.9 ± 1.5

^a Mean recoveries of lead ($n = 6$) are given in percent recovery versus the certified lead content; \pm values are relative standard deviations (RSDs).

^b Lead-based paint, 11.87% Pb.

^c Urban particulate, 0.655% Pb.

^d Buffalo river sediment, 0.0161% Pb.

^e Bag House Dust, 0.1914% Pb.

detection limit (MDL) of 2.5 $\mu\text{g Pb}$ per wipe in all cases. The CRMs tested were chosen as representative sample matrices of the kinds of materials that may be present in surface dust within residences and other locales. It can be seen that the recoveries of lead are quite variable, especially for the case of unfiltered sample solutions (Table 1). There are some notable differences in lead recoveries among CRMs as well as among wipes, both for unfiltered (Table 1) and filtered (Table 2) sample extracts.

In all cases shown in Tables 1 and 2, quantitative recoveries of lead (between 80 and 120%) were obtained from CRMs alone, i.e. when no wipes were present in the samples (Tables 1 and 2, bottom rows). Recoveries were statistically different (*t*-tests, 95% confidence limits) for one material, SRM 1579a, wherein the mean lead recovery for unfiltered sample extracts was slightly greater than that for filtered sample extracts. These recovery results for UE/ASV analysis of CRMs are similar to previously published data from UE/ASV analyses of lead-containing performance evaluation materials [5,6,8,19].

Anodic Stripping Voltammetry (ASV) analysis of unfiltered sample extracts resulted in quantitative recoveries for all CRMs spiked onto Pace Wipes, and for SRM 1579a spiked onto Wash'n Dri and Lead Wipes, and also for CRM SRS014-50 spiked onto Dust Wipes (Table 1). Low lead recoveries (range 34–78%) were obtained for all other “unfiltered” cases (Table 1). Only the Pace Wipes gave analysis data for all four matrices which met the performance criteria of an ASTM standard guide for evaluating field-based extraction and analysis methods [20]. This standard guide, ASTM E1775, lists an accuracy criterion of $\pm 25\%$ of the values obtained by using reference analytical methods, and it also lists an analytical precision requirement of $\pm 15\%$ for CRMs. For the “unfiltered” UE/ASV analysis of Pace Wipes spiked with the three CRMs investigated here, both ASTM performance criteria are met. For results from the other CRM-spiked wipes shown in Table 1, performance criteria for precision ($\pm 15\%$) are met in all instances. However, the $\pm 25\%$ accuracy criterion is met for only a few cases: Wash'n Dri spiked with SRM 1579a; Lead Wipe spiked with SRMs 1579a and 2704; and Dust Wipe spiked with SRM 1648 and CRM SRS014-50.

ASV analysis of filtered sample extracts resulted in quantitative recoveries for all cases except for Wash'n Dri wipes spiked with either SRMs 1648 or 2704 or CRM SRS014-50 (which gave low results), Lead Wipes spiked with CRM SRS014-50 (which also gave a slightly low result), and Dust Wipes spiked with SRM 1579a (which gave a somewhat high result) (Table 2). With the exception of a few cases (the three low results for Wash'n Dri wipes and the one high result for Dust Wipes), performance data met the performance criteria of ASTM E1775. In most cases lead recoveries from the wipe materials were increased significantly if the sample extracts were filtered through 0.45 μm hydrophilic PTFE filters prior to ASV analysis (compare data of Tables 1 and 2).

Filtration of sample extracts may help to remove interferences from surfactants that may be present within the wipes. Ultrafiltration has been shown to be effective for removing surfactants from aqueous solutions, as surfactants can form micelles which can be separated from the aqueous phase [21]. Surfactants and other organic materials can interfere with ASV analysis by binding to the surface of the working electrode and thereby blocking active sites [22]. Hence, the measured analyte signal (i.e. current) is lower than it would otherwise be in the absence of such an interference. This phenomenon is probably responsible for the low results observed in many instances where no filtration was used before analyzing extracted samples (Table 1). As evidenced by quantitative recoveries of lead, significant

Table 3
Ingredients in wipe sampling materials listed on packaging

Wipe	Ingredients listed
Wash'n Dri	Benzalkonium chloride, ^a water, SD alcohol 40, ^b PEG-75 ^b lanolin, benzoic acid, nonoxynol-9, ^b fragrance.
Dust Wipe	Deionized water, benzalkonium chloride. ^a
Pace Wipe	Distilled water, Ledizolv TM , ^c benzalkonium chloride. ^a
Lead Wipe	None; stated to not contain lanolin.

^a Cationic surfactant.

^b Nonionic surfactants.

^c Proprietary lead-complexing reagent.

removal of surface-active agents by filtration of extract solutions prior to ASV analysis apparently resulted for two of the four wipe materials tested, i.e. Dust Wipes and Lead Wipes (Table 2). No improvement in lead recoveries was observed for Wash'n Dri wipes spiked with SRMs 1648 and 2704 and CRM SRS014-50 (compare Tables 1 and 2). For all four CRMs, quantitative lead recoveries from Pace Wipes were obtained by ASV analysis whether sample extracts were filtered or not (Tables 1 and 2).

In view of the various results observed for the different wipe materials, it is of interest to consider the ingredients present within the wipe materials. Table 3 lists information regarding the ingredients within the wipes that were provided on the wipe packaging. (Proprietary ingredients are not listed.) Benzalkonium chloride is added to the wipes as an antibacterial and antifungal agent. The Pace Wipes contain a substance named LedizolvTM, which is a complexing agent meant to bind lead [23]. On the packaging of Pace Wipes and Dust Wipes, it is stated that the wipes conform to ASTM specifications for wipe sampling materials for lead in surface dust. The packaging of the Lead Wipe mentions that it “. . . does not contain lanolin which may alter lead test results.”² It is noted that Wash'n Dri wipes are not marketed for the collection of lead in surface dust, but they have been used for this purpose [24] and have been found to give quantitative results when using analytical methods such as hot plate digestion followed by atomic spectrometry [15]. Apart from benzalkonium chloride, which is a cationic surfactant, the Wash'n Dri wipes also contain a series of non-ionic surfactants as well as lanolin (Table 3).

ICP-AES analysis of ultrasonic extracts of Wash'n Dri wipes spiked with SRMs 1648 and 2704, both filtered and unfiltered, yielded low recoveries which were statistically equivalent to the results presented in Tables 1 and 2 (Table 4). This confirms that the ultrasonic extraction procedure did not successfully solubilize lead in the case of Wash'n Dri wipes spiked with SRMs 1648 and 2704. Therefore, the low recoveries observed for this wipe material spiked with these two reference materials were not due to an electrode fouling problem. If this were indeed the case, then quantitative recoveries would have been observed by ICP-AES.

² The source of this statement is unknown. The presence of lanolin within a wipe apparently does not alter lead analysis results when hot plate digestion and atomic spectrometric analysis is employed [15].

Table 4

Mean lead recoveries from selected NIST SRM-spiked wipe sample extracts, as determined by ICP-AES analysis ($n = 3$)^a

Wipe	SRM 1579a ^b	SRM 1648 ^c	SRM 2704 ^d
Wash'n Dri	–	48.6 ± 16.2 ^e	71.0 ± 16.8 ^e
	–	47.8 ± 16.7 ^f	70.9 ± 17.1 ^f
Lead Wipe	118.6 ± 0.8 ^e	94.1 ± 2.4 ^e	85.9 ± 1.2 ^e
	118.6 ± 0.2 ^f	94.1 ± 1.4 ^f	87.1 ± 1.1 ^f

^a (± values are relative standard deviations).

^b Lead-based paint, 11.87% Pb.

^c Urban particulate, 0.655% Pb.

^d Buffalo river sediment, 0.0161% Pb.

^e Unfiltered.

^f Filtered.

ICP-AES analysis of Lead Wipes spiked with SRMs (Table 4) yielded results which were quantitative (80–120%), whether sample extracts are filtered or not. This confirms that lead was successfully solubilized in the case of SRM-spiked Lead Wipes. The low recoveries observed for unfiltered sample extracts (Table 1) are ascribed to electrode fouling by species which can apparently be successfully removed by filtration (see results of Table 2).

The results for Lead Wipes and Dust Wipes (compare Tables 1 and 2) suggest that benzalkonium chloride species may be effectively removed from extract solution by filtration with hydrophilic PTFE filters. For Pace Wipes, however, the presence of a lead-complexing agent (Ledizolv) within the wipe material appears to alleviate effects of interferences from benzalkonium chloride, and allows for quantitative lead measurements to be successfully undertaken by UE/ASV, both for unfiltered as well as filtered sample extracts; note the quantitative recoveries obtained for this wipe material for all cases (Tables 1 and 2). Metal chelants used for complexation purposes are generally more surface-active than cationic surfactants such as benzalkonium chloride, so this may be responsible for the absence of observed interference (as evidenced by no significant diminution in lead recoveries) for the Pace Wipe analyses.

It is interesting to note that a number of non-ionic surfactants and lanolin (which are present in Wash'n Dri wipes) may cause interferences (as evidenced by low recoveries) in the determination of lead in SRMs 1648 and 2704 and CRM SRS014-50, but not in SRM 1579a (see Tables 1 and 2). Apparently lead pigments in paint are not influenced by the presence of the surfactant mixture within the Wash'n Dri wipes, which results in the ability to quantitatively measure lead in paint. Alternatively, the high level of lead in SRM 1579a may result in a high lead concentration within the extract solution which is less affected by the presence of un-removed surfactant material. However, non-ionic surfactants and/or lanolin apparently can bind irreversibly to lead compounds that are present in SRMs 1648 and 2704, and in CRM SRS014-50, and may form an insoluble "detergent" phase [25], thereby resulting in low recoveries whether sample extracts are filtered or not. In any case, the reasons for the differences in recoveries observed for Wash'n Dri wipes spiked with the different CRMs (Tables 1 and 2) are not completely clear. The inability of the extraction procedure to effectively solubilize lead in these cases of SRMs 1648 and 2704

was confirmed by ICP-AES analysis (Table 4). Clearly the detergent system present within Wash'n Dri wipes makes these wipes unsuitable for UE/ASV determination of lead in three of the four representative sample matrices studied here.

The Lead Wipes and Dust Wipes do not contain non-ionic surfactants or lanolin, but apparently they do contain species which, if not removed, can cause low lead recoveries for certain sample matrices (compare data of Tables 1 and 2). The only difference in ingredients between the Dust Wipes and Pace Wipes is the presence of a lead-complexing agent in the latter. It, therefore, appears that if it is desired to analyze unfiltered sample extracts of dust wipes by portable UE/ASV, a lead-complexing agent is needed. It is possible that the complexation agent promotes solubilization as well as favors desirable electrochemical response through complexation with the lead ion [26].

4. Conclusions

The results of this study indicate that three of the wipe materials tested (Dust Wipes, Pace Wipes and Lead Wipes) can be used for portable UE/ASV quantitative analysis of lead in all four sample matrices tested (paint, particulate, sediment, and dust) if sample extracts are filtered through 0.45 μm hydrophilic PTFE filters prior to the analysis step. However, only one of the wipe materials (Pace Wipes) can be used to obtain quantitative recoveries of lead by UE/ASV in both unfiltered and filtered samples. Nevertheless, filtering of extracts for all types of wipes is recommended as a precautionary measure. Wash'n Dri wipes are not acceptable for portable UE/ASV quantitative analysis, for the presence of a detergent system in these wipes precludes their use for three of the four representative sample matrices examined here. Further study is required to more thoroughly explain the findings in this paper. The estimated total analysis time for a sample set of 6–12 samples is 60–90 min, which includes the 45 min extraction period and sample manipulation.

Acknowledgements

Dr. R. Streicher (NIOSH), A. Sussell (NIOSH) and Dr. W. Gutknecht (RTI) critically reviewed the draft manuscript. ICP-AES analyses were performed by M. Millson (NIOSH). We thank Dr. M. McKnight (NIST) for helpful discussions, and the referees for their thorough review and commentary.

References

- [1] US Department of Housing and Urban Development (HUD): Guidelines for the Evaluation and Control of Lead-Based Paint in Housing, HUD, Washington DC, 1995.
- [2] ASTM E1644, Standard Practice for Hot Plate Digestion of Dust Wipe Samples for Subsequent Determination of Lead by Atomic Spectrometry, ASTM Standards on Lead Hazards Associated With Buildings, American Society for Testing and Materials (ASTM), West Conshohocken, PA, 1998.
- [3] ASTM E1613, Standard Test Method for the Analysis of Digested Samples for Lead by Inductively Coupled Plasma Atomic Emission Spectrometry, Flame Atomic Absorption Spectrometry, or Graphite Furnace Atomic Absorption Spectrometry, ASTM Standards on Lead Hazards Associated With Buildings, ASTM, West Conshohocken, PA, 1998.

- [4] A. Sussell, C. Hart, D. Wild, K. Ashley, An evaluation of worker lead exposures and cleaning effectiveness during removal of deteriorated lead-based paint, *Appl. Occup. Environ. Hyg.* 14 (1999) 177.
- [5] K. Ashley, Ultrasonic extraction and field-portable anodic stripping voltammetry of lead from environmental samples, *Electroanalysis* 7 (1995) 1189.
- [6] K. Ashley, K.J. Mapp, M. Millson, Ultrasonic extraction and field-portable anodic stripping voltammetry for the determination of lead in workplace air samples, *Am. Ind. Hyg. Assoc. J.* 59 (1998) 671.
- [7] K. Ashley, M. Hunter, L. Tait, J. Dozier, J.L. Seaman, P.F. Berry, Field investigation of on-site techniques for the measurement of lead in paint films, *Field Anal. Chem. Technol.* 2 (1998) 39.
- [8] K. Ashley, R. Song, C.A. Esche, P.C. Schlecht, P.A. Baron, T.J. Wise, Ultrasonic extraction and portable anodic stripping voltammetric measurement of lead in paint, dust wipes, soil, and air: an interlaboratory evaluation, *J. Environ. Monit.* 1 (1999) 459.
- [9] ASTM E1972, Standard Specification for Wipe Sampling Materials for Lead in Surface Dust, ASTM Standards on Lead Hazards Associated With Buildings, ASTM, West Conshohocken, PA, 1998.
- [10] ASTM E1979, Standard Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead, Annual Book of ASTM Standards, Vol. 04.11, ASTM, West Conshohocken, PA, 2000.
- [11] ASTM E2051, Standard Practice for the Determination of Lead in Paint, Settled Dust, Soil, and Air Particulate by Field-Portable Electroanalysis, Annual Book of ASTM Standards, Vol. 04.11, ASTM, West Conshohocken, PA, 2000.
- [12] NIOSH Method 7300: Elements by ICP-AES, in: P.M. Eller, M.E. Cassinelli (Eds.), NIOSH Manual of Analytical Methods, 4th Edition, National Institute for Occupational Safety and Health, Cincinnati, OH, 1994.
- [13] D.A. Binstock, E.D. Estes, J.D. Neefus, E.E. Williams, W.F. Gutknecht, S.L. Harper, M.E. Beard, in: M.E. Beard, S.D.A. Iske (Eds.), Preparation and Evaluation of Lead-Contaminated Dust Method Evaluation Materials, Lead in Paint, Soil and Dust (ASTM STP 1226), ASTM, Philadelphia, PA, 1995.
- [14] C. Chavalitnikul, L. Levin, A laboratory evaluation of wipe testing based on lead oxide surface contamination, *Am. Ind. Hyg. Assoc. J.* 45 (1984) 311.
- [15] M. Millson, P.M. Eller, K. Ashley, Evaluation of wipe sampling materials for lead in surface dust, *Am. Ind. Hyg. Assoc. J.* 55 (1994) 339.
- [16] ASTM D1193, Standard Specification for Reagent Water, Annual Book of ASTM Standards, Vol. 11.01, ASTM, Philadelphia, PA, 1993.
- [17] J. Wang, Decentralized electrochemical monitoring of trace metals—from disposable strips to remote electrodes, *Analyst* 119 (1994) 763.
- [18] K. Ashley, On-site extraction and anodic stripping voltammetric determination of lead, *Appl. Occup. Environ. Hyg.* 13 (1998) 94.
- [19] US Environmental Protection Agency (EPA), Evaluation of the Performance of Reflectance and Electrochemical Technologies for the Measurement of Lead, Characterized Paints, Bulk Dusts, and Soils (EPA 600/R-95-083), EPA, Research Triangle Park, NC, 1996.
- [20] ASTM E1775, Standard Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead, ASTM Standards on Lead Hazards Associated With Buildings, ASTM, West Conshohocken, PA, 1998.
- [21] E. Pramauro, A. Bianco Prevot, Solubilization in micellar systems — analytical and environmental applications, *Pure Appl. Chem.* 67 (1995) 551.
- [22] J. Wang, Electrochemical Preconcentration, in: P.T. Kissinger, W.R. Heineman (Eds.), Laboratory Techniques in Electroanalytical Chemistry, 2nd Edition, Marcel Dekker, New York, 1996.
- [23] N.R. Wilson, private communication, 1993.
- [24] A. Sussell, A. Weber, D. Wild, D. Wall, K. Ashley, An Evaluation of Airborne and Surface Lead Concentrations from Preliminary Cleaning of a Building Contaminated with Deteriorated Lead-Based Paint, in: M.E. Beard, S.D.A. Iske (Eds.), Lead in Paint, Soil and Dust (ASTM STP 1226), ASTM, Philadelphia, PA, 1995.
- [25] W.L. Hinze, E. Pramauro, A critical review of surfactant-mediated phase separations (cloud-point extractions): theory and applications, *Crit. Rev. Anal. Chem.* 24 (1993) 133.
- [26] F.A. Cotton, G. Wilkinson, Advanced Inorganic Chemistry, 4th Edition, Wiley, New York, 1980, (Chapter 3).