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Reconsideration of Methods and Standards: Digestion of Diaper Wipes and Use of Matrix-Matched Calibration Standards for Dust Lead Analysis

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Diaper wipes are widely used for sampling residential dust for lead analysis. A thicker type of diaper wipe was incompletely digested and had low recoveries (~40%) of lead on stock solution spikes using existing protocols. A modified protocol was applied to various quality control samples prepared with thicker diaper wipes in 134 batches of field samples. Modifications included a larger reagent volume (20 mL), more concentrated acid (50% HNO_3 followed by concentrated HNO_3), 3 h on the hot plate, and squeezing wipe residues during filtration. Seventeen batches were reanalyzed using matrix-matched standards. Acceptable lead recoveries were obtained for stock solution spikes (88%) and spikes prepared with leaded dust—SRM 2582 (88%), SRM 2589 (96%), and CRMO 14-050 (99%). Matrix-matched calibration standards increased mean lead recoveries by an additional 8%. Our protocol may provide a basis for a standard operational procedure for wipe digestion and analysis. Differences in estimates of dust lead loadings attributable to the type of wipe and to sample preparation and calibration procedures have implications for risk assessment, clearance testing, and comparability of laboratory data. Reconsideration of current protocols for wipe materials, wipe digestion, and judging laboratory performance (including the ELPAT external proficiency testing program for wipes) is warranted.

Introduction

Diaper wipes are routinely used to collect residential dust samples for assessment of lead hazards, clearance testing after lead-based paint abatement work, and research (*1*). Interim Federal clearance standards and guidelines for lead in settled dust after abatement (*1, 2*) and the U.S. Environmental Protection Agency's (EPA) recently proposed standard for lead in residential dust (*3*) are based on wipe sampling.

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Wipes have also been used in studies of the effectiveness of various practices for remediating residential lead-based paint hazards (*4*). The wipe method is practical and yields estimates of dust lead loadings that are associated with children's blood lead concentrations (*4, 5*), however, multiple types of diaper wipes and towelettes are in use. Department of Housing and Urban Development (HUD) guidelines mention multiple brands of diaper wipes (*6*); American Society for Testing and Materials (ASTM) standards specify the use of cellulose towelettes (thickness 0.02−0.10 cm and size 100−400 cm²) (*7, 8*); at least four brands of towelettes reportedly meet ASTM standards (*9*). Additionally, multiple sample preparation methods are used for the digestion of wipe samples for lead analysis (*10−13*).

In the course of analyzing the lead content of wipe dust samples collected in an ongoing study (*14*), we found that in using existing protocols (*10−12*) a commonly used thicker type of diaper wipe was not completely digested and had low recoveries of lead (~40%) on wipes spiked with lead stock solution. This paper reports a modified sample preparation protocol developed for thicker diaper wipes and its performance on a variety of quality control (QC) samples with and without the use of matrix-matched calibration standards.

Experimental Section

Little Ones Lightly Scented Baby Wipes (a thinner type of diaper wipe) were used in 1994 and 1995 for dust sampling in the environmental component of an ongoing study in Baltimore (*14*). Bigger and Thicker Baby Wash A Bye wipes were used after 1995 because the original brand was no longer available. These latter wipes were bigger (321 cm² versus 261 cm²) and had a higher average wet weight (4.3 g versus 2.1 g) but an average dry weight (1.3 g) similar to the thinner wipes formerly used. The similarity in dry weight may be due to the uneven texture of the thicker wipe as compared to the more uniform texture of the thinner wipe and to differences in materials. The manufacturer reported that the thicker wipe was a blend of cellulose and polyester (dry thickness of 0.05 cm and wet thickness of 0.08 cm) (Linda Volmar, personal communication).

Wipe samples using the thicker type of diaper wipe were prepared and analyzed at the Kennedy Krieger Research Institute's (KKRI) Trace Metals Laboratory using the modified protocol (Table 1). The following types of reagents were used: J. T. Baker nitric acid (trace metal grade, concentrated, 69.9%−70%), Mallinckrodt AR hydrogen peroxide (30% reagent ACS), and deionized water. Each batch of 60 samples included the following seven types of QC samples: a QC sample prepared as a wipe spiked with lead stock solution (spike), a spike duplicate, three wipes spiked with different standard reference materials (SRM) [National Institute of Standards and Technology (NIST) SRM 2582 (powdered paint), NIST SRM 2589 (lead-based paint), and certified reference material (CRM) CRMO 14-050 (baghouse dust)], and a method blank, all prepared using the thicker type of diaper wipe, as well as a reagent blank (Table 2). The weights of the SRMs and CRM used to spike wipes were selected so that the true values fell near the middle of the calibration curve. This paper is based on the lead recoveries of wipes spiked with stock solution and the three types of SRMs and CRM included in 134 batches. Fewer data are available for SRM 2589 and CRMO 14-050 because they were added to each batch later in the study. Three batches prepared with protocol deviations were excluded from the data analysis.

TABLE 1. Summary of Sample Preparation Protocol for Hot Plate Digestion of Thicker Wipes

	protocol description[a]
	remove wipe sample from sample centrifuge tube and place it in 125-mL Phillips beakers or 125-mL Erlenmeyer flasks
*	add 10 mL of 50% HNO_3 into sample tube
*	add 10 mL of concentrated HNO_3 acid into sample tube
	decant content of tube into respective beaker (flask) with wipe sample
	cover each beaker with nonribbed watch glass or flask with ribbed watch glass and place them on hot plate
*	reflux the samples at 95–100 °C for 3 h
	remove samples from hot plate
*	add 2 mL of deionized water to each sample by rinsing watch glass
*	add 3 mL of 30% H_2O_2 to each sample
*	return beakers (flasks) without watch glasses on hot plate (95–100 °C) for peroxide reaction for approximately 30 min (or more); change of sample color from white to yellow-amber means that reaction is over
	remove samples from the hot plate
*	add 3 mL of 30% H_2O_2 to each sample
*	return beakers to hot plate (95–100 °C) for peroxide reaction for approximately 30 min (or more); change of sample color from white to yellow-amber means that reaction is over
	remove samples from hot plate
	cool samples
	filter samples into 50-mL volumetric flask using VWR filter paper, grade 410, with following steps:
	rinse filter paper with deionized water
	pour sample on filter paper
	rinse flask with deionized water pouring solution on filter paper
*	squeeze undigested wipe residue using wood applicator sticks to remove as much sample solution as possible from its surface
	rinse filter paper of each sample with deionized water
	bring each sample to 50 mL volume with deionized water
	transfer each sample into original centrifuge tube
	if filtrate is cloudy, centrifuge sample for 15 min (4000 revertants/min); samples are ready for analysis

[a] An asterisk (*) indicates modifications/additions to existing protocols.

TABLE 2. Types of QC Samples and Preparation Procedures

type of QC sample	preparation procedure
wipe spiked with Standard Reference Material (SRM)	
NIST SRM 2589: powdered paint (nominal 10% lead)	Bigger and Thicker Baby Wash A Bye wipe plus 0.1 g of NIST SRM 2589 plus all reagents
NIST SRM 2582: lead-based paint (nominal 0.05% lead)	Bigger and Thicker Baby Wash A Bye wipe plus 0.25 g of NIST SRM 2582 plus all reagents
CRMO 14-050 baghouse dust (1914.0 ppm of lead)	Bigger and Thicker Baby Wash A Bye wipe plus 0.25 g of CRMO 14-050 plus all reagents
spike (wipe spiked with stock solution)	Bigger and Thicker Baby Wash A Bye wipe plus 0.5 mL of Perkin-Elmer pure atomic spectroscopy standard (lead, 1000 ppm) plus all reagents
spike duplicate (wipe spiked with stock solution)	same as for the spike sample
method blank	Bigger and Thicker Baby Wash A Bye wipe plus all reagents
reagent blank	reagents only

Compared to HUD's protocol (10), the modified protocol has a larger reagent volume (20 mL vs 14 mL); a higher concentration of nitric acid for digestion (50% and concentrated HNO_3 vs 20% HNO_3); an extended time on the hot plate (up to 3 h vs 20–30 min); and hydrogen peroxide additions. Our protocol differs from the EPA's 3050 protocol (12) in the following ways: the addition of nitric acid to the sample is done in one step; time on the hot plate is extended up to 3 h; and hydrogen peroxide is added in two steps (5 mL total vs additions in 1-mL aliquots up to 10 mL). Our protocol does not include the addition of HCl or the cutting of the wipes into small pieces because neither step was found to affect the completeness of digestion of the wipe or to improve lead recoveries, based on observation and limited testing. Differences between our protocol and the ASTM protocol E 1644-98 for hot plate digestion of towelettes (13) include the following: our protocol uses lower volumes of nitric acid (20 mL vs 35 mL) and final digestate (50 mL vs 100 mL) and specifies times for nitric acid digestion (3 h) and each peroxide addition step (30 min).

All field and QC samples were analyzed by flame atomic absorption spectroscopy (GBC Avanta (Australia), flame type: air–acetylene). The 217-nm wavelength was used for lead analysis to provide maximum sensitivity in our working range of concentrations (2.5–20.0 μg/mL) (15). Standard solutions used for calibration (0.25, 0.50, 1.0, 5.0, 10, and 20 ppm) were prepared in 10% nitric acid from GFS Chemicals Lead Standard Solution (1000 ppm of lead). A nonlinear calibration curve was used for the analysis.

QC sample type	mean (%) (*n*)		*t*-test *p*-value (one-sided)
	% recovery without squeezing step	% recovery with squeezing step	
spike	84 (67)	88 (62)	<0.001
spike duplicate	85 (69)	88 (62)	<0.001
SRM 2582	87 (67)	88 (62)	0.24
SRM 2589	93 (57)	96 (62)	0.02
CRMO 14-050	95 (23)	99 (62)	0.001

Matrix-matched calibration standards (0.25, 0.50, 1.0, 5.0, 10, and 20 ppm) were prepared by spiking Bigger and Thicker Baby Wash A Bye wipes with GFS Chemicals lead standard solution, followed by the addition of all reagents, heating on the hot plate, and filtering according to the modified protocol. A subset of 17 batches was analyzed using both matrix-matched and pure calibration standards.

Results

During method development, we found that increasing the temperature to approximately 150 °C sometimes resulted in complete digestion of the thicker wipe (within 3 h); in most cases, however, the digestate could not be analyzed due to spills and losses from boilover of the sample. Consequently, digestion was done at 95 °C according to the EPA's protocol to avoid spills and losses from boilover of the sample. Extending the time of hot plate digestion to 3 h did not result in total digestion of the thicker wipe but was found to increase lead recovery on the various QC samples. Increasing the volume of the nitric acid above 20 mL was found to prolong digestion time without providing complete digestion of the thicker wipe.

Using the modified protocol, the thinner type of diaper wipe was completely digested after 30−40 min heating on the hot plate; however, the thicker type of wipe was incompletely digested even after 3 h on the hot plate. After batch 71, we began squeezing the wipe residues during filtration to minimize the amount of digestate remaining in the wipe residues. Squeezing resulted in a mean increase of 3−4% in lead recoveries except for SRM 2582 (Table 3). No difference was found between the method blank samples (prepared with the wipe) with and without the squeezing step (means of 1.5 and 1.2 μg/sample, respectively, *t*-test *p*-value = 0.33).

The final protocol with the squeezing step was associated with mean lead recoveries ranging from 88% to 99% across all five types of QC samples and relative standard deviations ranging from 5% to 8% (Table 4). The absolute difference in mean recoveries between the QC sample types with the highest recoveries (CRMO 14-050) and the lowest recoveries (SRM 2582 and stock solution spikes) was 12%. For wipes spiked with stock solution or SRM 2582, less than 10% of the samples had lead recoveries below 80%. For wipes spiked with SRM 2589 or CRMO 14-050, lead recoveries were all within 20% of the target value.

The use of matrix-matched calibration standards was associated with a statistically significant 8% increase in mean lead recoveries on all types of QC samples, except for wipes spiked with SRM 2589. Mean lead recoveries across all the QC sample types ranged from 93% to 105% (Table 5).

The modified protocol was applied to eight wipes spiked with lead stock solution and eight wipes spiked with SRM 2582 prepared using four other brands of diaper wipes (Wash'n Dri Wipes, Wet Wipes, Baby Wipes, and Diaperene Wipes). For each brand of wipe, residues were present in the digestate, and the squeezing step was performed during filtration. The mean lead recoveries (91% for the eight spikes and 86% for the eight SRM 2582 samples) were similar to those in the main study.

Discussion

Wipe methods are likely to remain in wide use for the collection of residential dust samples for lead analysis. Other dust collection methods (e.g., vacuum, cyclone methods) are also being employed, however, these other methods are not likely to replace wipe methods soon because (a) estimates of dust lead loadings based on these methods are generally not better correlated with children's blood lead levels than wipe-based estimates (*16*), (b) the EPA's proposed residential dust lead standards are based on wipe sampling, and (c) because wipe sampling is more practical in the field in that it does not require electricity or special equipment.

In past studies, estimates of lead loadings were found to vary by dust collection method (wipe, cyclone, vacuum) (*17, 18*). This study shows that estimates of the lead content of dust wipe samples are influenced by the method of sample preparation and instrumental analysis. Sample preparation methodology was found to have the greatest impact on lead recoveries. The extended hot plate digestion time and the use of a higher concentration of nitric acid resulted in an increase in lead recoveries on diaper wipes spiked with stock solution from approximately 40% to above 80%. The squeezing of wipe residues during filtration increased mean recoveries by an additional 4%. With regard to instrumental analysis, the use of matrix-matched calibration standards increased mean recoveries by an additional 8%.

Results in Relation to Existing Laboratory Performance Criteria. Criteria for assessing the acceptability of laboratory performance in the analysis of dust wipe samples specified by HUD (*10*) and by the Environmental Lead Proficiency Analytical Testing (ELPAT) program (*19*) are based on lead recoveries of wipes spiked with a known amount of leaded dust. Only HUD criteria are directly applicable to the analysis of diaper wipes. Our modified method produced acceptable lead recoveries based on HUD criteria (i.e., ±20% of the "true value") when all three types of leaded dust (SRM 2582, SRM 2589, and CRMO 14-050) were used to spike the thicker type of diaper wipe. Mean lead recoveries and the lead recoveries for nearly all (97%) the individual wipe samples spiked with SRMs and CRM were within HUD's acceptable range. Our method also produced acceptable lead recoveries on wipes spiked with lead stock solution (Table 4).

ELPAT criteria are not applicable to the study findings and not directly comparable to HUD criteria because acceptability is judged by statistical comparisons to the consensus values of participating reference laboratories. Additionally, ELPAT is based on the use of PaceWipes towelettes spiked with sieved household dust and not SRMs. Although ASTM does not have criteria for judging laboratory performance on wipe samples, their criteria for wipe sampling materials (*7*) specify acceptable lead recoveries on towelettes spiked with NIST SRMs as 100 ± 10% (95% confidence level) of the lead recovery of the SRM alone without the wipe. Our method in combination with matrix-matched calibration standards met more stringent criteria; specifically, the 95% confidence intervals on mean recoveries were ±10% of the true value across all types of QC samples (Table 5).

Reconsideration of Current Protocols and Policy Implications. The results of the study show that lead recoveries are related to the type of wipe as well as to sample preparation method and the type of calibration standards used. These factors also can influence the estimates of dust lead loadings for field samples. For these reasons, reconsideration of the adequacy of the current protocols for the selection of wipe sampling materials and the preparation and analysis of wipe

TABLE 4. Descriptive Statistics for Calibration Percent Recovery on Laboratory QC Samples Prepared as Spiked Wipes Using Study Protocol (Table 1) without Matrix-Matched Standards

QC sample type	N	mean (%)	95% CI	SD (%)	min. (%)	10th PT	median	90th PT	max. (%)
spike	62	88	86−90	5.9	74	82	89	96	109
spike duplicate	62	88	87−89	4.6	79	81	88	93	100
SRM 2582	62	88	86−90	7.8	63	81	87	98	111
SRM 2589	62	96	95−97	5.2	82	89	95	102	111
CRMO 14-050	62	99	98−100	5.6	85	92	99	106	115

TABLE 5. Lead Recovery on QC Samples Prepared as Spiked Wipes and Analyzed with and without Matrix-Matched Calibration Standards

QC sample type	N	mean (%) (95% CI)		t-test p-value
		% recovery without matrix-matched standards	% recovery with matrix-matched standards	
spike	17	87 (82−91)	95 (91−99)	<0.001
spike duplicate	17	85 (82−88)	93 (91−95)	<0.001
SRM 2582	14	88 (85−91)	96 (94−98)	<0.001
SRM 2589	17	93 (90−96)	94 (89−99)	0.61
CRMO 14-050	15	97 (93−101)	105 (103−107)	<0.001

dust samples for lead may be warranted. In light of the findings that lead recoveries are related to the type of wipe and to the type of material used to prepare spiked wipes, a reconsideration of the adequacy of the ELPAT program for wipes and the various criteria for judging laboratory performance on wipes may be warranted.

Need for Standardizing Diaper Wipe Material. Consideration must be given to the variety of types and brands of diaper wipes and towelettes that are being used for dust collection and the fact that their availability changes over time. For example, during the course of our work the thicker type of diaper wipe became widely available, and most of the thinner brands of diaper wipes mentioned in the HUD protocol were no longer available in local stores. Also in recent years, the Maryland Department of Health and Mental Hygiene's Lead Laboratory has processed increasing numbers of thicker diaper wipes for lead analysis. Thicker diaper wipes are likely to be used increasingly for risk assessment and clearance testing in other states as well because they are more rugged and generally larger than towelettes and therefore better suited for sampling dust from a wider range of household surfaces and conditions (e.g., carpets and rough, deteriorated or porous surfaces). Despite the wide usage of diaper wipes, the only standard for wipe sampling materials (ASTM 1792-96) is limited to cellulose towelettes that can be used on hard smooth surfaces (e.g., tile, metal, plastic or wood) without tearing (7). Furthermore, the ELPAT program's exclusive reliance on a particular brand of towelette that meets ASTM standards greatly limits the applicability and relevance of this external proficiency testing program. For this reason, the types of wipes included in the ELPAT proficiency testing programs need further consideration.

Need for Standardizing Materials Used To Prepare Spiked QC Wipes. Consideration also needs to be given to which types of materials should be used to prepare spiked wipes for laboratory QC purposes and for establishing laboratory performance standards. First, existing sample preparation protocols are not uniform with regard to the types of spiked wipe. Stock solution spikes are included in the EPA's protocol and are specifically excluded from HUD's method, which specifies the use of wipes spiked with leaded dust (11, 12). ASTM's method for hot plate digestion of wipes describes the use of wipes spiked with leaded stock solution and certified reference materials (13). ASTM's standard for wipe sampling material employs towelettes spiked with NIST

SRMs (7). Second, lead recoveries vary by QC sample type prepared by the same method. For example, using the HUD method of digestion, wipes spiked with stock solution had lead recoveries of ∼40%, which was not acceptable according to HUD's criteria, but wipes spiked with SRMs had acceptable recoveries. Third, the selection of QC sample types (stock solution vs leaded dust) can be important for the efficiency of the sample preparation effort. Fourth, selection of QC sample types is important for maximizing comparability with the matrix of field samples as well as for increasing confidence in the comparability of the results provided by different laboratories.

The apparent interaction between the wipe matrix and the spiking materials, as evidenced by the increase in recoveries with the squeezing step and by the differences in lead recoveries, raises questions regarding which characteristics of the wipe matrix or the spiking material itself may account for the apparent interaction. Relevant characteristics of leaded dust used to spike wipes include the lead species present (e.g., lead carbonates, lead silicates), the potential presence of oils in the lead-based paint SRMs, and the particle size distribution. The relatively smaller particle size distribution of baghouse dust might explain the relatively high recoveries for CRMO 14-050 (Table 4).

Implications of Use of Matrix-Matched Calibration Standards. Increased lead recoveries were associated with the use of matrix-matched standards for all types of QC samples, except SRM 2589. The absence of a matrix-matching effect for SRM 2589 (the only QC type that required dilution) was likely due to the fact that the diluting solution used was not a matrix-matched solution (e.g., prepared as a method blank sample per Table 2). Increased lead recoveries were also observed for the field samples included in the 17 batches of wipes analyzed with matrix-matched standards. Despite the additional time and effort involved in the preparation of matrix-matched standards and matrix-matched diluting solution for samples requiring dilution, the use of such calibration standards to maximize lead recoveries may be important due to the concerns about lower levels of lead in residential dust (3, 5). The use of matrix-matched standards will potentially increase the number and proportion of field samples exceeding relevant standards for lead in residential dust, including post-abatement clearance standards. The use of matrix-matched calibration standards also adds another variable affecting the comparability of data across laboratories using different calibration practices.

Approaches for Addressing Methodological Issues. Approaches to addressing the various issues raised might include the following: (i) standardizing the type of wipe used for dust collection so that one type of wipe can be used for residential sampling, e.g., a standardized type of diaper wipe; (ii) employing a standardized protocol that is best suited for a standardized wipe or one that can handle a variety of wipes in the absence of standardized wipe; this protocol may include the use of matrix-matched standards to maximize lead recoveries; and (iii) standardizing the types of materials used to prepare QC samples for wipe dust analysis. These approaches would increase the comparability of dust wipe results across laboratories. Additionally, the various labora-

tory performance criteria employed by HUD, ASTM, and ELPAT should be reconsidered. Our modified method may provide a basis for the development of a standard operational procedure for a variety of types of wipes.

Systematic differences in estimates of dust lead loadings attributable to the type of wipe and to sample preparation, and analysis methods have practical implications for risk assessment, clearance testing of residences, and comparability of data across studies and laboratories. Such differences need to be considered in the establishment of standardized sampling and analytical protocols for lead in dust that may accompany any local, state, or federal standard for lead in residential dust. Furthermore, the growing understanding of the risk of children's exposure to low dust lead loadings argue for the best possible recoveries reasonably obtainable (*3, 5*). These issues are particularly relevant as the EPA finalizes its proposed standard for lead in residential dust, which is expected to result in an large increase in wipe sampling.

Acknowledgments

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