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## Evaluation of a Handwipe Disclosing Method for Lead\*

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**ABSTRACT:** A qualitative chemical screening method for lead in wipe samples was evaluated for its utility in detecting the presence of lead in collected dust; preliminary evaluation of the performance of the method is reported here. In evaluating the method on pure lead compounds, the observed intensity of the characteristic color change due to the presence of lead was generally consistent with the relative solubilities of the tested compounds. Some pure (non-lead) metal compounds (e.g., those of Ag, Ba, Bi, Ca, Cd, Hg, and Sr) were found to give false positive results. Several representative lead-containing reference materials were also tested, and the qualitative test results differed for different materials. For materials collected on wipes, the method was found to be effective for detecting lead in several sample matrices commonly found in occupational settings. The technique was also applied on-site on dermal samples collected at field locations.

**KEYWORDS:** handwipe, lead, qualitative analysis, screening, workplace

### Introduction

Qualitative field screening methods are sometimes used in environmental health and occupational hygiene applications for the on-site detection of toxic substances of concern, e.g., heavy metals such as lead [1–3]. Such methods may be used to assess whether toxic agents of concern are present or absent, even with respect to regulatory action levels [4]. Field screening tests can provide a timely means for assessing potential human exposures to toxic materials, but their validity is called into question if their performance has not been evaluated or verified [5,6].

A qualitative colorimetric (screening) method for disclosing the presence of lead in dust wipe samples has been developed [7,8]. The method entails the use of a wetted handwipe to collect dust on surfaces such as skin, floors, and myriad other substrates that are potentially contaminated with lead. The wipe is subsequently treated with a weakly acidic leaching solution in order to extract soluble lead that may be present in the collected dust matrix. The presence of lead is then disclosed by use of an aqueous solution of rhodizonic acid, which forms a characteristic pink- to red-colored complex under acidic conditions [9,10].

The goal of this study was to evaluate the handwipe disclosing technique for lead by subjecting the method to a series of validation tests. In previous work, we have evaluated qualitative colorimetric screening methods for lead in air filter samples [11] and in paint coatings [12]. Because the performance characteristics of qualitative spot tests are matrix-dependent [13,14], it is recommended to evaluate these techniques for each analytical matrix of interest. In the present study, we have evaluated the response of the handwipe screening method for lead using chemically pure standards of known lead compounds. Also, the method was tested on representative matrix chemical standards, i.e., certified reference materials (CRMs), and on-site on dermal samples collected in field locations where lead was a suspected contaminant.

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## Experimental

### Reagents

All chemicals used in this work were reagent grade. Rhodizonic acid (disodium salt),  $\text{Ca}(\text{NO}_3)_2$ ,  $\text{KCl}$ ,  $\text{K}_2\text{CrO}_4$ ,  $\text{KNO}_3$ ,  $\text{NaCl}$ ,  $\text{Pb}_3\text{O}_4$ ,  $\text{PbS}$ ,  $\text{TlCl}$ , and  $\text{TlNO}_3$  were obtained from Sigma-Aldrich (Milwaukee, WI).  $\text{Al}_2\text{O}_3$ ,  $\text{BaCO}_3$ ,  $\text{Cd}(\text{NO}_3)_2$ , and  $\text{NaNO}_3$  were from J. T. Baker (Philipsburg, NJ).  $\text{PbO}$  and  $\text{PbBr}_2$  came from Johnson Matthey (Royston, Herts., United Kingdom).  $\text{Pb}$  metal (granular) and  $\text{PbO}_2$  were from Merck (Rahway, NJ).  $\text{AgNO}_3$ ,  $\text{Al}(\text{NO}_3)_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Hg}(\text{NO}_3)_2$ ,  $\text{K}_2\text{SO}_4$ ,  $\text{PbCrO}_4$ , and  $\text{PbSO}_4$  were purchased from Fisher Scientific (Fair Lawn, NJ).  $\text{AgCl}$ ,  $\text{PbCl}_2$ ,  $\text{Pb}(\text{NO}_3)_2$ ,  $\text{Pb}_3\text{O}_4$ , and  $\text{Sr}(\text{NO}_3)_2$  were obtained from Mallinckrodt (St. Louis, MO).  $\text{Cd}(\text{CH}_3\text{COO})_2$ ,  $\text{Pb}(\text{CH}_3\text{COO})_2$ , and  $\text{Te}$  powder came from Matheson, Coleman, and Bell (Norwood, OH).  $\text{Ba}(\text{NO}_3)_2$ ,  $\text{BaCl}_2$ ,  $\text{Bi}(\text{NO}_3)_3$ ,  $\text{Ca}(\text{CH}_3\text{COO})_2$ ,  $\text{Mg}(\text{NO}_3)_2$ ,  $\text{NiCl}_2$ ,  $\text{Ni}(\text{CH}_3\text{COO})_2$ , and  $\text{Zn}(\text{CH}_3\text{COO})_2$  were from Chem Service (Media, PA). Metal standard solutions ( $\approx 1000 \mu\text{g/mL}$ ) were obtained from Inorganic Ventures (Lakewood, NJ).

Deionized water (18  $\text{M}\Omega$ ) was produced using a Barnstead Nanopure system (Thermolyne, Dubuque, IA). White vinegar (5 % aqueous acetic acid) was purchased from a local grocery store (Bigg's, Harrison, OH).

### Materials

Dust wipes meeting standard performance specifications [15] were obtained from Palintest (Lot No. 080245; Gateshead, Tyne, and Wear, United Kingdom). Wax paper was found in a local convenience store (Ameristop, Barnesburg, OH). Disposable nitrile laboratory gloves were ordered from Fisher Scientific. Plastic pump spray bottles (vol.  $\approx 150 \text{ mL}$ ) yielding fine-mist aerosols were purchased from U.S. Plastics (Lima, OH).

Representative lead-containing CRMs consisted of National Institute of Standards and Technology (NIST) Standard Reference Materials<sup>®</sup> (SRMs) (NIST, Gaithersburg, MD). The SRMs used were as follows: (a) 1579a, lead-based paint; (b) 1633b, coal fly ash; (c) 1648, urban particulate matter; (d) 2580, powdered paint I; (e) 2581, powdered paint II; (f) 2582, powdered paint III; (g) 2583, trace elements in indoor dust I; (h) 2584, trace elements in indoor dust II; (i) 2709, San Joaquin soil; (j) 2710, Montana soil I; (k) 2711, Montana soil II; and (l) 8704, Buffalo River sediment.

### Sample Handling

All laboratory and field procedures and manipulations were carried out while wearing clean nitrile gloves (Kimberly-Clark, Roswell, GA). In order to prevent cross-contamination, gloves were changed frequently, and at least each time a new sample was tested.

For preparation of the colorimetric indicator solution, 0.135 g of sodium rhodizonate was dissolved in 105 mL of very cold ( $\approx 2^\circ\text{C}$ ) deionized water within a spray bottle, and was kept on ice to prevent deterioration of the rhodizonate solution. A fresh indicator solution was prepared daily. Vinegar extraction solution was contained in a separate spray bottle, but was not refrigerated.

In order to prevent biasing the results, tests were carried out blind by the investigator performing the colorimetric wipe procedure for lead. That is, samples were prepared for testing by a different person from the one who actually carried out the tests on the samples.

Single-element standard solutions of Ag, Al, Ba, Bi, Ca, Cd, Fe, Na, Pb, Sn, Te, and Tl (Inorganic Ventures; metal concentrations  $\approx 1000 \mu\text{g/mL}$ ) were diluted 10-fold in order to prepare solutions containing lead and/or potential interferants at desired concentrations. Also, two multielement standard solutions (Inorganic Ventures) were similarly diluted. The compositions of the multielement standard solutions were as follows: (Solution A) Sb at 500  $\mu\text{g/mL}$ , Te and W at 250  $\mu\text{g/mL}$ , Sn and Ti at 125  $\mu\text{g/mL}$ , and Mo and Zr at 50  $\mu\text{g/mL}$ ; (Solution B) K at 3750  $\mu\text{g/mL}$ , Cu at 2500  $\mu\text{g/mL}$ , Zn at 750  $\mu\text{g/mL}$ , As at 500  $\mu\text{g/mL}$ , Se at 375  $\mu\text{g/mL}$ , Al, Cu, Fe, Pb, Mg, P, and Ti at 250  $\mu\text{g/mL}$ , La, Ag, and V at 100  $\mu\text{g/mL}$ , and Ba, Be, Cd, Cr, Co, Li, Mn, Ni, Sr, and Y at 50  $\mu\text{g/mL}$ . Aliquots of the diluted solutions (500  $\mu\text{L}$ ) were spiked onto clean watch glasses or Petri dishes and were allowed to dry overnight.

NIST SRMs were weighed to  $\pm 0.0001 \text{ g}$  on an analytical balance (Mettler model AE163, Greifensee, Switzerland). For each SRM, a weighed amount of sample ( $\approx 50 \text{ mg}$ ) was deposited lightly and uniformly

TABLE 1—*Qualitative colorimetric screening results from using the handwipe method on ≈50 mg of various lead compounds.*

Compound(s)	Test Response <sup>a</sup>
PbCrO <sub>4</sub>	—
PbS, PbO <sub>2</sub>	+
Pb metal, Pb <sub>3</sub> O <sub>4</sub>	++
Pb(CH <sub>3</sub> COO) <sub>2</sub> , Pb(NO <sub>3</sub> ) <sub>2</sub> , PbO, PbCl <sub>2</sub> , PbBr <sub>2</sub> , PbSO <sub>4</sub>	+++

<sup>a</sup>(—: negative; +: weak positive; ++: definite positive; +++ brilliant positive).

on a sheet of wax paper within a 10 × 10 cm area (demarcated by a plastic template). A newly opened wipe was then unfolded and used to quantitatively collect the SRM onto the wipe surface [16]. The wipe was then laid face-up on the wax paper surface, with the collected material exposed. In order to leach lead in the collected sample, a few sprays (3–5) of vinegar were applied to the exposed wipe. After a few seconds, the wipe was then sprayed with 2–4 sprays of the rhodizonic acid indicator solution. Observation of the characteristic color change (from yellow/orange to pink/red) on the wipe surface was recorded as a positive result, while absence of this observation was recorded as a negative. Watch glasses and Petri dishes that were spiked with standard solutions and then dried were similarly tested.

### Field Samples

Dermal wipe samples were collected and tested qualitatively for the presence of lead at field sites using commercial colorimetric kits (“Full Disclosure,” SKC, Inc., Eighty Four, PA) in accordance with NIOSH method 9105 [8]. Dermal wipe sampling and qualitative lead testing was carried out at a metal cutting/brazing operation in northwestern Ohio (United States) and at an outdoor firing range in southwestern Ohio (United States). After colorimetric testing, the wipe samples were placed into 50 mL plastic centrifuge tubes (Becton-Dickinson, Franklin Lakes, NJ), and securely capped. The samples were then transported to the laboratory following standard chain-of-custody procedures [17]. In the laboratory, lead in these wipe samples was extracted by means of strong acid hot plate digestion (Lindberg, Thermo-Fisher Scientific, Pittsburgh, PA) following the procedure described in ASTM 1644 [18]. Subsequently, lead in wipe sample extracts was determined using inductively coupled plasma-atomic emission spectrometry (Spectro EOP, SpectroAnalytical, Fitchburg, MA) in accordance with ASTM E1613 [19].

## Results and Discussion

Results from use of the lead handwipe disclosure method on ≈50 mg of various lead compounds are shown in Table 1. The observed intensity of the characteristic red color bloom varied for different lead compounds, and this was generally related to the solubility of each compound in aqueous solution [20]. Because lead chromate is insoluble in weak acid solution, a false negative result was observed for this matrix. For compounds giving intense positive responses, it was possible to detect the characteristic pink or red color due to the lead-rhodizonate complex for very small amounts of material (as low as ≈1 mg). With such materials, the effective identification limit for lead is apparently established by what can be discerned by the human eye [21].

Qualitative results from a number of non-lead compounds are shown in Table 2. As can be seen, it was

TABLE 2—*Qualitative colorimetric screening results from using the handwipe method on ≈50 mg of various non-lead compounds.*

Compounds	Test Response <sup>a</sup>
Al(NO <sub>3</sub> ) <sub>3</sub> , Al <sub>2</sub> O <sub>3</sub> , Fe <sub>2</sub> O <sub>3</sub> , HgCl, KCl, K <sub>2</sub> CrO <sub>4</sub> , K <sub>2</sub> SO <sub>4</sub> , KNO <sub>3</sub> , Mg(NO <sub>3</sub> ) <sub>2</sub>	—
AgCl, Bi(NO <sub>3</sub> ) <sub>3</sub> , Cd(CH <sub>3</sub> COO) <sub>2</sub> , Hg(NO <sub>3</sub> ) <sub>2</sub> , NaCl	+
AgNO <sub>3</sub> , BaCO <sub>3</sub> , BaCl <sub>2</sub> , Ca(CH <sub>3</sub> COO) <sub>2</sub> , Ca(NO <sub>3</sub> ) <sub>2</sub> , CdCl <sub>2</sub> , Cd(NO <sub>3</sub> ) <sub>2</sub> , HgCl <sub>2</sub> , Zn(CH <sub>3</sub> COO) <sub>2</sub>	++
Sr(NO <sub>3</sub> ) <sub>2</sub> , Ba(NO <sub>3</sub> ) <sub>2</sub>	+++

<sup>a</sup>(—: negative; +: weak positive; ++: definite positive; +++ brilliant positive).

observed that several other metals (besides Pb) gave positive responses with sodium rhodizonate. Many of these potential positive interferences, e.g., from  $\text{Cd}^{2+}$  and  $\text{Hg}^+$ , have been identified previously; in fact, sodium rhodizonate has been used for decades as a qualitative test for  $\text{Ag}^+$ ,  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ , and  $\text{Tl}^+$  [22]. However, the sensitivity of the reaction with some of these other metals is less than with  $\text{Pb}^{2+}$  [10]. Nevertheless, observation of false positives underscores one of the limitations of using qualitative screening methods.

It was desired to investigate the use of the handwipe colorimetric lead screening test on lesser masses of metals than can be investigated by weighing bulk pure materials. Therefore, it was necessary to prepare test surfaces of desired metal masses from standard solutions having target metal concentrations (as described in the experimental section). Spiked masses on watch glasses or Petri dishes ranged from  $\approx 5 \mu\text{g}$  to  $\approx 500 \mu\text{g}$ , depending on the metal(s) and applicable concentration ratio(s). Results from these trials are illustrated in Table 3. It is seen that Ba, Cd, and Ag give false positive results for Pb, while Fe causes a negative interference, as evidenced by a false negative. Other Pb/metal mixtures are unaffected by potentially interfering coexisting metals.

To examine data from CRMs, results from using the handwipe colorimetric lead screening test on various NIST SRMs are given in Table 4. With the exception of SRM 2709, results are negative for SRMs having lead concentrations below  $\approx 400 \mu\text{g/g}$ . The (albeit weak) false positive result from SRM 2709 is likely due to a colorimetric reaction of rhodizonate with barium, which is present in this material at a level of nearly  $1000 \mu\text{g/g}$ . At and above  $\approx 450 \mu\text{g Pb/g}$  ( $\approx 20 \mu\text{g Pb/wipe}$ ), all test results (save one, i.e., that for SRM 2711) are positive, although the intensity of the positive response varies appreciably with the matrix. For materials giving positive responses, the intensity of the characteristic color bloom was generally greater for paint samples versus soils and sediments. This is probably due to solubility differences of the lead compounds extant in the various media.

Results from field studies of the colorimetric screening test, as applied to dermal wipe samples ( $n = 130$ , with similar numbers of samples from each site), are summarized in Table 5. Comparable results were observed from each of the two field sites, both in terms of ranges of lead concentrations obtained and colorimetric responses; thus, the data from the two sites were grouped together. It can be seen from Table 5 that negatives or weak positives are prevalent below about  $10 \mu\text{g Pb/sample}$ , while definitive positive results predominate above  $50 \mu\text{g Pb/wipe}$ . A significant overlap of positive and negative results is obtained for masses between  $10$  and  $50 \mu\text{g Pb/sample}$ , which is consistent with what is to be expected from performance curves for qualitative test methods [13]. These data suggest that the handwipe disclosing method for lead may be useful for screening of dermal samples for this element at levels below  $10 \mu\text{g}$  (for absence of Pb) and above  $50 \mu\text{g}$  (for presence of Pb) per wipe. However, more study is needed in order to obtain sufficient data to accurately model the performance curve of the test method [14], and thereby obtain reliable estimates of the method's performance parameters.

## Conclusions

The results of this work have highlighted some of the attributes and limitations of this qualitative screening method. Certain test matrices are obviously problematic, and false positive/false negative rates need to be investigated for each matrix of interest. For some sample matrices, the method is very sensitive; for instance, less than  $20 \mu\text{g Pb}$  was detected in wipe samples collected at a firing range. However, some of the limitations of the handwipe method are obvious; thus, previous knowledge of the test matrix is highly recommended if the procedure is used for screening purposes.

TABLE 3—Qualitative colorimetric screening results from using the handwipe method on smooth surfaces (watch glasses or Petri dishes) prepared from dried test solutions.

Test Metal or Mixture <sup>a</sup>	Test Response <sup>b</sup>
Mixture A; Al; Ca; Fe; K; Na; Sn; Te; Zn; Pb/Fe	—
Ba; Cd; Pb/Al; Pb/Ba; Pb/Ca; Pb/Na; Pb/Sn; Pb/Te	+
Mixture B; Ag; Pb; Pb/Ag; Pb/Cd	++

<sup>a</sup>(1) Test surfaces of mixtures A (no Pb) and B ( $\approx 25 \mu\text{g Pb}$ ) were prepared from the corresponding multielement solutions A and B described in the experimental section. (2) Metal masses  $\approx 50 \mu\text{g}$  for single element spikes. (3) For binary (Pb/M) mixtures, M mass  $\approx 500 \mu\text{g}$  and Pb mass  $\approx 50 \mu\text{g}$ .

<sup>b</sup>(—: negative; +: weak positive; ++: definite positive).

TABLE 4—Qualitative colorimetric screening results from using the lead handwipe disclosure method on  $\approx 50$  mg of various representative NIST SRMs.

SRM Number and Matrix <sup>a</sup>	SRM Pb Content ( $\mu\text{g/g}$ )	Wipe Pb Content ( $\mu\text{g}$ )	Test Response <sup>b</sup>
1579a (paint)	11 900	595	+++
2584 (dust)	9761	488	++
2710 (soil)	5532	277	+
2580 (paint)	4340	217	+++
2711 (soil)	1162	58.1	—
1648 (particulate matter)	655	32.8	+
2581 (paint)	449	22.5	+
2582 (paint)	208.8	10.4	—
8704 (sediment)	150	7.5	—
2583 (dust)	85.9	4.3	—
1633b (fly ash)	68.2	3.4	—
2709 (soil)	18.9	0.95	+

<sup>a</sup>1579a: Lead-based paint; 1633b: Coal fly ash; 1648: Urban particulate matter; 2580: Powdered paint I; 2581: Powdered paint II; 2582: Powdered paint III; 2583: Trace elements in indoor dust I; 2584: Trace elements in indoor dust II; 2709: San Joaquin soil; 2710: Montana soil I; 2711: Montana soil II; 8704: Buffalo River sediment.

<sup>b</sup>(—: negative; +: weak positive; ++: definite positive; +++ brilliant positive).

A few potential applications of the handwipe disclosing method for lead can be predicted from this preliminary work. The method was effective for the detection of lead in many lead compounds employed in industry, such as oxides of lead and lead salts. Hence, the use of the handwipe method to detect appreciable levels of lead on workers' hands and on surfaces in affected workplaces is especially appealing. The procedure also appears to be effective for detecting lead at significant concentrations in indoor dust, and might be used for risk assessment and clearance purposes. However, the performance of the method on some matrices, e.g., soils, is less effective. Nevertheless, the colorimetric handwipe disclosing procedure was designed mainly as a lead screening tool for occupational settings where lead is a contaminant in dust produced during work activities. Examples of workplace environments where the use of this screening tool may be applicable include battery manufacturing/recycling, lead-based paint abatement/renovation, and firing ranges. Indeed, the vast majority of lead compounds used in industry are soluble or sparingly soluble [23], and the colorimetric handwipe procedure is effective for detecting lead in these materials.

As this was a preliminary evaluation, it remains of interest to carry out a full validation of the handwipe screening method for lead. This should be carried out in a manner that is consistent with new standard guidelines for quality assurance of qualitative analysis [24,25]. To this end, it is recommended to carry out round-robin studies so that interlaboratory data can be compared and contrasted on representative performance evaluation materials. Also, extensive field studies are necessary in order to fully evaluate the method performance in specific matrices and workplace environments.

TABLE 5—Summary of qualitative colorimetric screening results ( $n = 130$ ) obtained using the handwipe disclosing method on dermal samples tested at field sites, versus Pb mass ranges obtained from quantitative laboratory analysis.

Number of Test Responses <sup>a</sup>					
Range ( $\mu\text{g Pb/Sample}$ )	(—)	(+)	(++)	Percent (—)	Percent (++)
<5	39	6	1	85	2
5–10	19	6	1	73	4
10–20	13	5	5	57	22
20–50	6	5	3	43	21
>50	1	1	19	5	90

<sup>a</sup>(—: negative; +: weak positive; ++: definite positive; note that only negatives and definite positives are used to compute percents of “meaningful” responses).

*Disclaimers*

Mention of company names or products does not constitute endorsement by the Centers for Disease Control and Prevention. The findings and conclusions in this paper are those of the authors and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

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