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Evaluation of a Chemical Spot-Test Kit for the Detection of Airborne Particulate Lead in the Workplace

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A commercial rhodizonate-based test kit was evaluated for its potential use in the detection of lead in airborne particulate samples at work sites. Over 350 air samples were collected at abrasive blasting lead paint abatement sites using cellulose ester membrane filters and personal sampling pumps. The filter samples were tested with the chemical spot test and then analyzed by graphite furnace atomic absorption spectrophotometry. No positive readings were recorded for lead masses below 1.3 µg Pb/filter, and no negative readings were observed for lead amounts above 8.1 µg Pb/filter. Experimental data were statistically modeled in an effort to estimate the performance parameters of the spot test kit. The identification limit of the kit was found to be approximately 3.6 µg/filter sample. For lead mass values above approximately 10 µg Pb/filter, 95% confidence of a positive reading was found; while 95% confidence of a negative reading was found for lead masses below approximately 0.6 µg Pb/filter. Based on the results of this study the rhodizonate-based test kit for lead demonstrates potential for use in field screening for lead in personal breathing zone and area air samples.

Keywords: spot test kit, airborne particulates, lead detection

Workers involved in the abatement of lead-based paint or renovation of structures that are coated with lead-containing paint are known to be at risk of airborne lead exposure.(1-4) Protocols for monitoring airborne lead exposures in the workplace include National Institute for Occupational Safety and Health (NIOSH) methods,(5) which involve laboratory analysis by atomic absorption or emission spectrometry following collection on filter membranes using personal sampling pumps. Laboratory analyses of personal breathing zone (PBZ) samples for compliance purposes may be time-consuming, and more rapid screening and measurement techniques for lead are desired. In light of the short-term duration of lead abatement and renovation jobs, and also due to the transient nature of the workforce involved in abatement activities, rapid detection and analysis methods are needed to assess worker exposures to lead in these work environments. Hence, field-based methods for the qualitative detection and quantitative determination of PBZ lead are under development in this laboratory.

The study described herein involved the evaluation of a qualitative spot test for lead in filter samples, which were obtained mainly at work sites where abrasive blasting was used to remove lead-based paint from highway bridges. The spot test is based on the use of the rhodizonate dianion, which has been employed as a qualitative test for the presence of lead for many years.(6-9) Rhodizonate reacts with Pb^{2+} to form a complex with a pH-dependent characteristic color. Uncomplexed rhodizonate is yellow to orange, while the lead-rhodizonate complex is pink to red under acidic conditions and violet to blue in neutral to basic solutions.(7) Rhodizonate also forms colored complexes with several other metals such as barium and strontium,(6,7) but only the lead-rhodizonate complex yields the pink or red characteristic color in acidic solution. Hence, the use of rhodizonate as a spot test for lead is highly specific for this metal.

Most commercially available spot test kits for lead are based on the reaction of this metal with either rhodizonate or sulfide ions. Rhodizonate and sulfide-based spot tests for lead have been evaluated recently on paint, dust, and soil for their potential use in detecting lead in buildings.(10,11) The sulfide dianion, S^{2-}, forms a black precipitate (PbS) when it reacts with Pb^{2+}; this precipitation reaction is the basis of the sulfide spot test for lead. Fewer interferences to the detection of lead have been observed with rhodizonate than with sulfide,(10) in part because sulfide can form dark precipitates with other metals besides lead. Also, the use of the rhodizonate spot test has been found to be less subject to operator error and interferences compared with the sulfide kit.(10,11)

Other spot tests for lead have been used in the past,(8) but the selectivity and sensitivity of those spot tests were found to be inferior to sulfide and rhodizonate. Due to the nature of airborne particulate samples, it was decided that a rhodizonate spot test would be the most appropriate choice for this matrix. The formation of a PbS precipitate was found to be difficult to observe on workplace air filter samples, since the particulate matter collected on air filter samples is typically dark brown or black. Also, a rhodizonate-based kit was selected that was found to be easy to use on cellulose ester air filter samples.

This work represents the first effort to identify performance parameters for the test kit measurement of lead in airborne particulate samples. For a given matrix, a qualitative spot test can be evaluated through the use of performance curves.(12) A performance curve for a given spot test is a plot of the spot test response versus the amount of lead as determined by a reference quantitative analytical method. Equivalently, the performance curve is a plot of the probability of a positive test kit response versus the "known" analyte concentration. From the performance curve it is possible to determine the identification limit of the spot test and the ranges of lead mass that result in either positive or negative readings. (The identification limit is the amount of analyte at which one has a 50% chance of observing either a positive or negative result.)(8) However, many data points are necessary to define the performance curve for lead detection adequately for a given spot test and a given matrix.(12) Hence, this study was limited to the evaluation of a single lead test kit. However, the protocol employed in this investigation may be applied to the evaluation of other candidate test kits, analytes, and sample matrices.

EXPERIMENTAL

Sample Collection

Airborne particulate samples were collected at the following abrasive blasting and lead paint removal sites during 1992 and 1993: Athens, Dayton, Milford, and Springdale, Ohio. The sample collection protocol was as described in the NIOSH methods(5) and in ASTM practice E1553.(13) Samples were collected closed-faced at a flow rate of 2.0 (± 0.1) L/min on preloaded 0.8-µm pore size cellulose ester 37-mm filter cassettes (SKC, Eighty-Four, Pa.) using personal sampling pumps (DuPont Model P2500A, Wilmington, Del.). To obtain a range of loadings on the filters, sampling times varied from a few minutes to several hours (ca. 10 L to 500 L). Once collected, samples were transported to the laboratory in sealed plastic bags.

Spot Test Detection

A commercial rhodizonate spot-test kit (Merck EM Quant, Darmstadt, Germany) was used on each filter sample according to manufacturer's instructions. The test strips consisted of thin strips of flexible plastic on which rhodizonate-impregnated pieces of felt were attached at one end. The filter cassettes were opened manually, and two or three drops of reagent solution were placed on the center of each filter. The piece of felt on the test strip was then applied with manual pressure for 2 minutes. A pink or red color change on the felt piece and/or on the filter itself was recorded as a positive reading, while an absence of color change was recorded as a negative result. The test kit included a scale that may allow for semiquantitative measurement of the lead concentration (in ppm), based on the intensity of pink to red color that develops. However, since this application of the kit is intended ordinarily for water samples, no effort was made to estimate the lead concentration based on the results of the tests on airborne particulate samples. Rather, a characteristic color change of any intensity was recorded as a positive result.

Laboratory Analysis

After testing with the spot test kit, the filters, test strips, and backup pads were transferred to 125-mL borosilicate glass beakers. Added to each beaker were 3 mL of concentrated reagent grade nitric acid (Aldrich, Milwaukee, Wis.) and 1 mL of 30% reagent grade hydrogen peroxide (Aldrich, Milwaukee, Wis.). The samples were heated on a hotplate at a surface temperature of 140°C until the volume was reduced to about 0.5 mL. (The corresponding temperature of the beaker contents was ca. 100°C.) Additional 2–3 mL aliquots of nitric acid were added to digest the test strips. Digested samples were brought to volume with ASTM Type I(14) water in a 125-mL Class A borosilicate volumetric flask. The digested and diluted samples were then analyzed at 283.3 nm using a graphite furnace atomic absorption spectrophotometer (GFAAS; Perkin-Elmer Zeeman 5100, Norwalk, Conn.). The detection limit of the spectrophotometric technique was ~ 0.01 µg/filter.

Computational Methods

Statistical analyses were conducted using PC-SAS® (Cary, N.C.) software. Maximum likelihood and numerical calculations were carried out with SAS programs.(15)

RESULTS AND DISCUSSION

Spot Test Data

Four sets of data were collected to determine the operational characteristics of the lead spot-test kit on cellulose ester membrane filter samples. The samples were collected from locations known to be subject to lead contamination, and were not manufactured in the laboratory. When the spot test is applied to the sample, it returns either a positive response indicating the presence of lead, or a negative result indicating that no lead was detected. The data consisted of 371 observations of environmental samples that had been tested by the rhodizonate chemical spot test, followed by GFAAS analyses. A positive test result was scored a value of 1 while a negative result was scored a value of 0. No positive test kit readings were observed for lead loadings below 1.3 µg, and no negative spot test readings were obtained for lead levels above 8.1 µg. Thus there is a region of uncertainty between ~1 µg Pb/filter and ~10 µg Pb/filter, where positive and negative results overlap. Data from four different sample sets were collected to examine results obtained from various work sites.

The experimental data can be modeled to yield a characteristic performance curve for the test kit. The performance curve would be different for other manufacturers' test kits and other sample matrices. Specific objectives of the analysis of the performance curve concern the interpretation of a positive or negative test kit response. First, is the spot test result consistent with the assumption that as the amount of lead in the sample increases, the probability of a positive response will increase monotonically? If so, this would imply that a model for predicting the probability of a positive response as a function of the amount of lead could be fit to these data. Second (and third), what can a user infer, concerning the amount of lead on the sample, from a positive (or negative) test kit response?

Obtaining precise solutions for these three objectives requires data for which there is the greatest uncertainty of the test kit response being positive or negative; i.e., for amounts of lead such that the probability of a positive response is ~0.5. Since samples were obtained in real work environments (rather than being prepared artificially), there are some limitations to the overall analysis. For one, the study is subject to the variability of the amount of lead in the environmental samples; that is, lead loadings are not known *a priori*. The amount of lead is determined analytically (by spectrophotometric means) and is subject to error, rather than being known without error as this treatment assumes. This limitation was ignored, and its effects on the findings of the study are unknown (although the error introduced here is thought to be minimal). Furthermore, the composition of the sample matrix is variable and was not experimentally controlled. This may influence the leachability of lead from samples collected at different work sites; also, the effects of interferences are unknown. These limitations (due to possible differences in sample matrices) were addressed by collecting sets of samples from several work environments, rather than just one sample set from a single work site. Statistical comparison of results obtained from the four work sites indicated that the sample matrices were similar for the locations chosen for this study.

Statistical Modeling

Statistical modeling methods were employed in efforts to determine the performance curve and estimate performance parameters of the observed experimental spot-test kit data. For a qualitative test the performance curve describes the probability of a positive test result as a function of the mass or concentration of analyte. This function should be a continuous, increasing function with values (ideally) beginning at zero and ending at unity. An example of a generalized performance curve is shown in Figure 1.

The following distribution function(16) was used to fit the observed spot test kit data:

$$p(x;a,b) = 1 - \exp[-(x/a)^b] \text{ (for } x \geq 0),$$

where p, the probability of observing a positive response, is a function of lead mass, x, and parameters a and b. Parameters a and b are location and scalar parameters, respectively. Increasing the value of *a* will shift the performance curve to the right, while increasing the b value will make the curve steeper.

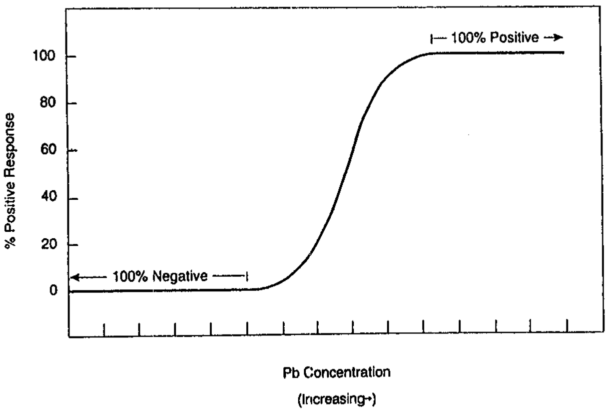

FIGURE 1. Expected performance curve (probability of positive response versus mass or concentration of analyte) for a qualitative chemical spot test

Based on the above model, the maximum likelihood method(17,18) was used to estimate the two unknown parameters a and b. However, test kit performance parameters that are of interest are the lead masses that give 5, 50, and 95% chances (p=0.05, 0.50, and 0.95) of positive response. These performance parameters can be expressed as functions of the two primary parameters a and b:

$$x_p = a[-\ln(1-p)]^{1/b} \text{ (for } p = 0.05, 0.50, \text{ and } .095),$$

where x_p is the lead mass at a given probability of positive response.

The 50% positive response point $x_{0.50}$ is the identification limit defined previously. The 5% (95%) positive response point $x_{0.05}$ ($x_{0.95}$) provides the information that when a positive (negative) test kit response is observed, we are at least 95% confident that the lead mass is above $x_{0.05}$ (below $x_{0.95}$). The parameters $x_{0.05}$, $x_{0.50}$ and $x_{0.95}$ may be used to characterize the performance of the spot test kit.

Estimates and confidence intervals for the two primary parameters a and b, and the three positive response points (test kit performance parameters of interest) $x_{0.05}$, $x_{0.50}$, and $x_{0.95}$ are listed in Table I. Details regarding the estimation procedure employed for this work are presented in the Appendix. A two-sided confidence interval was used for the identification limit, while single-sided confidence limits were used for the 5% and 95% response points. The reasons for the use of single-sided confidence intervals for $x_{0.05}$ and $x_{0.95}$ are explained as follows: only the lower limit of the 5% response point and the upper limit of the 95% response point are of interest for application of the test kit. Results for estimation of the performance curve are plotted in Figure 2.

The identification limit $x_{0.50}$ of the spot test kit for airborne particulates collected in this work was found to be on the order of a few micrograms of lead (Table I). The identification limit is of interest in that it is equivalent to the detection limit of a quantitative analytical measurement in the following sense: both the identification limit of a qualitative measurement tool(8) and the detection limit of a quantitative measurement device(19) correspond to the values of analyte mass (or concentration) at which one has a 50% chance of a negative result. The values $x_{0.05}$ and $x_{0.95}$ are of interest in that these parameters enable the user to employ the test kit for exposure assessment purposes.

TABLE I. Point and Interval Estimates of the Spot Test Kit Performance Parameters for the Detection of Lead in Airborne Particulate Samples

Parameter	Point Estimate^A	Interval Estimate (95%)
a	4.69 ± 0.39	(3.92, 5.46)
b	1.41 ± 0.17	(1.07, 1.75)
$x_{0.05}^B$	0.57 ± .015	(0.27, ∞)
$x_{0.50}^C$	3.62 ± 0.32	(2.84, 4.41)
$x_{0.95}^D$	10.21 ± 1.29	(0, 13.94)

[A] ± values are standard deviations
[B] 5% positive response point
[C] 50% positive response point (identification limit)
[D] 95% positive response point

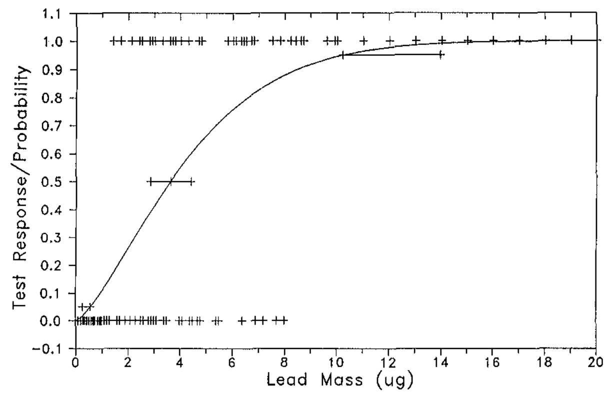

FIGURE 2. Data fitted by statistical modeling, and 95% confidence intervals about the estimated parameters $x_{0.05}$, $x_{0.50}$, and $x_{0.95}$. Maximum likelihood estimates of parameters a and b were those values given in Table I. For the test kit response, a negative result is given a value of zero, while a positive reading is attributed a value of one. +: individual data points; solid line: modeled performance curve.

Let us consider explicitly what the user may infer from a positive or a negative test kit result, based on calculated test kit performance parameters (Table I). If a negative result is observed, we may be 95% confident that the mass of lead in the sample is less than 10.21 μg. On the other hand, if a positive result is observed, we may be 95% confident that the mass of lead in the sample is greater than 0.57 μg. These values can then be used, with knowledge of sampling duration and flow rates, to evaluate compliance with applicable action levels and compliance levels, e.g., Occupational Safety and Health (OSHA) action levels and permissible exposure limits (PELs).

The implications of the test kit evaluation are as follows. Here the performance parameters for point estimates obtained from the maximum likelihood treatment, listed in Table I, are utilized. The case of the use of a chemical spot test on a personal breathing zone sample obtained at a flow rate of 2 L/min is considered. If a positive result is obtained after sampling for 5 min, one would expect the OSHA PEL of 50 μg/cm^3 to be exceeded over an 8 hr time-weighted average. On the other hand, if a negative result is obtained after sampling for 2 hr, it can be predicted that the OSHA PEL probably will not be exceeded over an 8-hr workday. Of course, these implications assume that the sampling periods yield exposures that are representative of the entire workday. Also, these conclusions are specific for the test kit evaluated here. Performance parameters for other chemical spot test kits may be different, and so other test kits must be evaluated similarly before being used to screen for lead in workplace air.

CONCLUSION

A major potential advantage of the use of spot tests for airborne particulate samples is that there is no action level constraint, since the flow rate, collection time, etc., can be adjusted to obtain measurable quantities of lead. Thus the method may be useful for on-site screening of lead in workplace air. Lead spot tests could be employed on-site to give an indication of whether personal exposures are in compliance with applicable federal or state regulations. Knowledge of the test kit performance parameters, provided by a statistical fit of the experimental data, may allow for the use of rhodizonate-based spot tests in field screening of airborne lead in workplace environments.

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APPENDIX

The maximum likelihood estimates for the parameters a and b in the performance function can be obtained by maximizing the following logarithmic function:

$$
\begin{aligned}
&L((a, b); (x_i, y_i), i = 1, \ldots, n)\\
&\quad = \sum_{i=1}^{n} (y_i \ln (p(x_i; a, b)) + (1 - y_i) \ln (1 - p(x_i; a, b)))\\
&\quad = \sum_{i=1}^{n} \ln (1 - |y_i - p(x_i; a, b)|),
\end{aligned}
$$

where the second equality is due to $y_i = 0$ or 1. Since explicit expressions for the maximum likelihood estimates are not available, a numerical procedure (described below) was employed to find estimates for parameters a and b (see Table I). According to the large sample theory,(16,17) maximum likelihood estimators for a and b have a joint asymptotic bivariate normal distribution with mean $(a,b)^t$ (where superscript t means transpose of a vector) and variance-covariance matrix:

$$
\begin{aligned}
\hat{V} &= \begin{pmatrix} \hat{\sigma}^2_{\hat{a}} & \hat{\sigma}_{\hat{a}\hat{b}} \\ \hat{\sigma}_{\hat{b}\hat{a}} & \hat{\sigma}^2_{\hat{b}} \end{pmatrix}\\
&= \begin{pmatrix} \dfrac{\partial^2 L}{\partial a^2} & \dfrac{\partial^2 L}{\partial a \partial b} \\[2ex] \dfrac{\partial^2 L}{\partial b \partial a} & \dfrac{\partial^2 L}{\partial b^2} \end{pmatrix}^{-1}_{(\hat{a},\hat{b})} = \begin{pmatrix} 6.47 & -0.05 \\ -0.05 & 33.93 \end{pmatrix}^{-1}\\
&= \frac{1}{\dfrac{\partial^2 L}{\partial a^2}\dfrac{\partial^2 L}{\partial b^2} - \left(\dfrac{\partial^2 L}{\partial a \partial b}\right)^2} \begin{pmatrix} \dfrac{\partial^2 L}{\partial b^2} & -\dfrac{\partial^2 L}{\partial a \partial b} \\[2ex] -\dfrac{\partial^2 L}{\partial b \partial a} & \dfrac{\partial^2 L}{\partial a^2} \end{pmatrix}_{(\hat{a},\hat{b})}\\
&= \begin{pmatrix} 0.1546 & 0.0002 \\ 0.0002 & 0.0295 \end{pmatrix}.
\end{aligned}
$$

Asymptotic confidence intervals for a and b are given by:

$$(\hat{a}^{0.025}, \hat{a}^{0.975}) = (\hat{a} - 1.96\hat{\sigma}_{\hat{a}}, \hat{a} + 1.96\hat{\sigma}_{\hat{a}})$$

and

$$(\hat{b}^{0.025}, \hat{b}^{0.975}) = (\hat{b} - 1.96\hat{\sigma}_{\hat{b}}, \hat{b} + 1.96\hat{\sigma}_{\hat{b}}).$$

Let $x = f(p; a, b) = a[-\ln(1 - p)]^{1/b}$ be the inverse function of $p(x; a, b)$ and $\hat{a}$, $\hat{b}$ be the maximum likelihood estimates of a and b, respectively. Then estimates for the $\gamma 100\%$ positive response points with $\gamma = 0.05, 0.5, 0.95$ are given by

$$\hat{x}_\gamma = f(\gamma; \hat{a}, \hat{b}).$$

The standard deviation of the estimate for x_γ may be estimated by:

$$
\begin{aligned}
s(\hat{x}_\gamma) = \Bigg[&\left(\hat{\sigma}_{\hat{a}} \frac{\partial f(\gamma; \hat{a}, \hat{b})}{\partial a}\right)^2\\
&+ 2\hat{\sigma}_{\hat{a}\hat{b}} \frac{\partial f(\gamma; \hat{a}, \hat{b})}{\partial a} \frac{\partial f(\gamma; \hat{a}, \hat{b})}{\partial b} + \left(\hat{\sigma}_{\hat{b}} \frac{\partial f(\gamma; \hat{a}, \hat{b})}{\partial b}\right)^2\Bigg]^{1/2}.
\end{aligned}
$$

To obtain a confidence interval for a performance parameter x_γ, we consider the statistic $(\hat{a}-a, \hat{b}-b)\hat{V}^{-1}(\hat{a}-a, \hat{b}-b)^t$. According to normal theory,(18) it has an asymptotic χ^2-distribution with 2 degrees of freedom. Therefore, a $\gamma 100\%$ confidence region for a and b can be obtained by

$$
\begin{aligned}
C_\gamma(\hat{a}, \hat{b}) &= \{(a, b) : (\hat{a} - a, \hat{b} - b)\hat{V}^{-1}(\hat{a} - a, \hat{b} - b)^t\\
&\qquad \le \chi^2_2(\gamma)\}\\
&= \{(a, b) : 6.47(a - 4.69)^2\\
&\qquad - 0.1(a - 4.69)(b - 1.41)\\
&\qquad + 33.93(b - 1.41)^2 \le \chi^2_2(\gamma)\},
\end{aligned}
$$

where $\chi_2{}^2(\gamma)$ is the $\gamma 100$ percentile of the χ^2-distribution with 2 degrees of freedom. For $\gamma = 0.9$ and 0.95, $\chi_2{}^2(\gamma) = 4.61$ and 5.99, respectively.

Based on the confidence region for a and b, interval estimates for performance parameters can be derived as follows: (1) A two-sided 95% confidence interval for a $\gamma 100\%$ positive response point x_γ is given by:

$$
\begin{aligned}
(\hat{x}^{0.025}_\gamma, \hat{x}^{0.975}_\gamma) = (&\min\{f(\gamma; a, b) : (a, b) \in C_{0.95}(\hat{a}, \hat{b})\},\\
&\max\{f(\gamma; a, b) : (a, b) \in C_{0.95}(\hat{a}, \hat{b})\}
\end{aligned}
$$

and (2) a one-sided 95% confidence interval for a $\gamma 100\%$ positive response point x_γ is given by:

$$(0, \hat{x}^{0.95}_\gamma) = (0, \max\{f(\gamma; a, b) : (a, b) \in C_{0.9}(\hat{a}, \hat{b})\})$$

or

$$(\hat{x}^{0.05}_\gamma, \infty) = (\min\{f(\gamma; a, b) : (a, b) \in C_{0.9}(\hat{a}, \hat{b})\}, \infty).$$

Estimates of performance parameters determined from this statistical treatment are shown in Table I.