



Report on the State of Development, Availability, Evaluation, and Future Use of Test Kits for the Measurement of Lead in Paint

SCIENCE



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Prepared by

W. F. Gutknecht, W. Winstead, C.A. Salmons, and D.A. Binstock

RTI International
3040 Cornwallis Road
Research Triangle Park, NC 27709

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1.0 INTRODUCTION

The purpose of this issue paper is to address the availability and performance characteristics of portable lead test kits especially suited for lead in paint, procedures for evaluating the performance of these test kits, and the availability of performance evaluation materials suitable for test kits. Knowledge and understanding of these issues will provide a platform for identifying approaches or modifications that can be used with current or newly developed test kits for lead in paint to adjust their performance to meet the new Federal performance standards found in Lead; Renovation, Repair, and Painting Program; Proposed Rule (EPA, 2006).

Lead (Pb)-based paint is a major source of Pb poisoning for children and also can affect adults. In children, Pb poisoning can cause irreversible brain damage, impair mental functioning, retard mental and physical development, and reduce attention span. In adults, it can cause irritability, poor muscle coordination, and nerve damage to the sense organs and nerves controlling the body. Pb poisoning also may cause problems with reproduction (such as a decreased sperm count) and also may increase blood pressure. Thus, young children, fetuses, infants, and adults with high blood pressure are the most vulnerable to the effects of Pb.

Pb-based paint is a major source of Pb that poses this risk to children and adults. Pb-containing paint may be a direct hazard if eaten, but it poses its greatest hazard when it is broken down into small particles and becomes a component of house dust or soil around the house. It is these particles, which stick to the hands of children and are ingested through hand-to-mouth activity, that pose the most significant potential for harm.

Some 38 million houses (Jacobs et al., 2002) in this country still contain leaded paint. The U.S. Department of Housing and Urban Development (HUD) has an ongoing major effort that includes grants to communities and citizen organizations for removal of Pb hazards from dwellings. This effort and the efforts of other groups, including commercial paint testing, control, and abatement firms, all need to analyze the paint in dwellings to determine if Pb is present at or above the levels of concern identified by the U.S. Environmental Protection Agency (EPA) and HUD (Title X, 1992; HUD, 1995). Those levels of concern are 0.5% Pb by weight or 1.0 mg Pb/cm², the Federal regulated levels. Some States and localities may use other definitions of Pb-based paint; for example, Wisconsin defines "lead-bearing" paint as surface coating material containing more than 0.06% Pb by weight or more than 0.7 mg Pb/cm² in the dried film of applied paint (Wisconsin Statutes, 2004).

Methods of analysis for Pb in paint include portable X-ray fluorescence (XRF), anodic stripping voltammetry (ASV), atomic absorption spectrometry (AAS), inductively coupled plasma (ICP) emission spectrometry, and quantitative test kits. Homeowners also may perform Pb testing using spot or qualitative test kits specifically designed for in-home use by untrained people. An alternative is for the homeowner to collect a paint sample and send it to a commercial laboratory for analysis; kits for this purpose can be purchased that give guidance for collection of a paint sample and the materials for submitting it.

Test kits offer the potential for performing field measurements that will indicate whether Pb levels in paint are above or below the Federal regulated levels. Suitable performance of these kits could lead to rapid decisions about the presence of unacceptable risk, faster and lower cost testing, immediate indication of need for some form of control or abatement, and real-time tracking of the effectiveness of performance of control or abatement. The Federal regulated level is defined as paint containing Pb at or above 1 mg/cm² or 0.5% by weight. For a test kit to

be recognized by EPA, it must meet the following performance standards and goals (from *Lead; Renovation, Repair, and Painting Program; Proposed Rule* [EPA, 2006]).

- Has a demonstrated probability (with 95% confidence) of a false negative response to Pb levels above the regulated level of less than 5%
- Has a demonstrated probability (with 95% confidence) of a false positive response to Pb levels below the regulated level of no more than 10%
- Can be used reliably by a person with minimal training
- Provides results within an hour
- Costs less than \$2 per test

To further clarify whether these goals can be met, a study of the current status of test kits has been performed. The primary objectives of this study are to perform a literature search and prepare an issue paper discussing the availability and performance characteristics of portable Pb test kits especially suited for testing Pb in paint, procedures for evaluating the performance of these test kits, and the availability of performance evaluation materials suitable for test kits. This study included the following.

- Conduct literature search and contact experts
- Identify portable Pb test kits and their sources
- Develop a matrix that presents the key operating parameters for each test kit identified
- Locate in the literature and summarize performance evaluations, if available, of each test kit identified
- Identify performance evaluation materials suitable for use with test kits
- Describe protocols suitable for determining the performance of test kits, especially for Pb in paint

1.1 Test Kits

Test kits are analytical systems based on relatively simple measurement technologies. The most common type is based on the development of a color resulting from the reaction between Pb and some other chemical agent; this color can be noted visually or its intensity measured with a simple colorimeter. Test kits are generally of two types: (1) spot test kits (Sections 1.1.1 and 3.1) and (2) semi-quantitative or quantitative test kits (Sections 1.1.2 and 3.2).

Semi-quantitative test kits are those that give an approximate value for the Pb level. They typically work by visually comparing the intensity of the color formed with some standard chart of colors or through a color comparator. The quantitative test kits usually involve an instrumental measurement of the product of reaction with Pb. This may be measurement of the intensity of the colored product of Pb with rhodizonate or some other reagent or measurement of some other unique property of Pb, such as its electrochemical properties, using a Pb-ion-selective electrode.

1.1.1 Spot Test Kits

Qualitative spot test kits are those wherein the formation of a color is observed visually. The paint is either tested directly (i.e., on the substrate material to which it has been applied) or after removal from the substrate. The color-forming reagent may be applied directly on the paint or to a paint sample that has been treated with a reagent that releases the Pb from the organic paint matrix to some extent and, thereby, increases the potential for color formation. Spot tests have great potential to complement other laboratory and field methods currently in use. Spot tests currently are being used as a qualitative test for the presence of Pb (i.e., a level equal to a method-defined limit of detection). Nevertheless, the analytical performance and reliability of the tests remain unvalidated or incompletely validated. Spot tests often serve as an initial testing or screening tool, followed by portable XRF or laboratory analysis after considering the results of the spot tests. A spot test that meets the proposed Federal performance standards would be

very useful in that it potentially provides a way to perform the tens of thousands of onsite analyses required for future work. There are, however, limitations to the spot tests, which include the following.

- The technique is qualitative, so no analytical reference standards for accuracy and precision are available; performance will be judged by the rates of false positive and false negative results (Song et al., 2001).
- The presence of ions other than Pb (e.g., barium) can give rise to positive results, so there is a built-in false positive factor.
- Detection is by visual comparison of color changes, so the results are subjective and may be inconsistent.
- Results for colored paints may be difficult to interpret. An observed darkening may be the result of wetting with the solution, rather than the formation of a colored precipitate.
- Detection in layers below the surface may be affected by the briskness of application and, thus, extraction. This effort may not be reproducible.
- Interpretation is often a function of available lighting.

In the early 1990s, two principle chemistries were used for Pb spot tests: (1) reaction with sodium sulfide to form the dark gray or black lead sulfide precipitate and (2) reaction with rhodizonate to form a pink complex. These chemistries are discussed in more detail in the following subsections.

1.1.1.1 Sodium Sulfide

Detection. In the sodium sulfide test, a drop of sodium sulfide solution is placed on exposed layers of paint. Layers that contain Pb will turn gray or black as a lead sulfide precipitate is formed. In a test of this method at the Naval Civil Engineering Laboratory (Vind and Mathews, 1976), positive results for Pb were observed at a minimum concentration of 0.5% (w/w). The authors stated that, even though the detection limit of the test was approximately the regulatory limit (0.5% [w/w]), detection of Pb at this level in darker paints would not be possible. This is not necessarily the case when one considers that, in pre-1978 housing, there likely are many layers of paint and layers of different colors; the dark sulfide may be difficult to differentiate from some layers but likely not all layers.

Studies by McKnight et al. (1989) and Blackburn (1990) have shown inconsistencies in the detection limit of the sulfide-based spot test. Blackburn tested 377 paired paint chips. The concentration of Pb for one chip in each pair was determined by flame atomic absorption spectroscopy (FAAS) and converted to milligrams per square centimeter. The concentration of the other chip in the pair was determined using the spot test method. The author found variations in the color of the precipitates: black, gray, green, blue, brown, copper, and orange. The observation of black or gray precipitates was correlated with 96% of the "positive results." Blackburn observed positive results (black coloration) to increase with Pb concentration from 28.3% at a concentration of 0.7 to 0.9 mg/cm² to 80.4% at FAAS concentrations of ≥ 10.0 mg/cm². The frequency of negative results was found to be technician dependent. On wood substrates only, negative test results at 0.7 to 0.9 mg/cm² were 51.1%; whereas negative results at concentrations of ≥ 10.0 mg/cm² decreased to 20.5%. Blackburn (1990) concluded that the overall false negative results on wood were 25.0%. This is inconsistent with the findings of McKnight et al. (1989) who estimated the false negative results of sodium sulfide spot tests to be about 10%.

Selectivity. A number of inorganic compounds contain metals whose sulfides are dark (see Table 1). Vind and Mathews (1976) and studies by Midwest Research Institute (1990) evaluated the formation of colored precipitates with sodium sulfide solution for inorganic materials having potential uses in paint formulations (biocides or pigments). The authors observed positive

Table 1. Metallic Elements Having at Least One Black Sulfide

Element	Colors of Sulfides	Some Uses of Compounds in Paints
Antimony	Black, red	Pigment
Bismuth	Black, brown, gray	Pigment
Cadmium	Black	Pigment
Chromium	Black, brown, gray	Pigment, corrosion inhibitor
Cobalt	Black, gray, red	Pigment, drier
Copper	Black	Biocidal pigment
Iron	Black, green, yellow	Pigment
Lead	Black	Pigment, drier, corrosion inhibitor
Manganese	Black, green, pink	Pigment, drier
Mercury	Black, red	Pigment, biocide
Molybdenum	Black, brown, gray	Pigment, corrosion inhibitor
Nickel	Black, gray, yellow	Pigment

results for mercuric oxide, mercuric iodide, and phenylmercuric oleate, all of which are used as biocides.

Cobalt naphthenate and manganese naphthenate, used as curing or drying agents, also turned black with the application of sodium sulfide solution. Bismuth trioxide changed from greenish-white to light brown in the presence of the sodium sulfide solution. The most common pigments in older paints included Pb, chromium, iron, and cadmium; common driers included cobalt, Pb, manganese, and zinc (Gooch, 1993). Despite the plethora of reactions of different metals with sulfide, sulfide serves as a useful indicator for Pb in that Pb is usually the predominant metal in older paints.

1.1.1.2 Sodium Rhodizonate

Sodium rhodizonate forms a pink complex with Pb in acidic solutions (Feigl and Suter, 1942). It may be used to detect Pb in

- paint,
- dust,
- soil,
- dilute solutions,
- ores and minerals,
- alloys, and
- pigments and glass.

Detection. The test is rapid and sensitive. In evaluation studies performed at the Research Triangle Institute (RTI) (Luk et al., 1993b), four commercially available rhodizonate-based kits yielded a positive reaction to Pb ranging from about 0.5 µg Pb (absolute in solution) to 5 µg Pb, with the reproducibility being ± 0.05 to ± 0.5 µg, respectively. Consumer Reports (1995) presented levels yielding positive responses for several of the rhodizonate-based test kits as follows.

- Lead Zone 5%
- Know Lead 0.5%
- LeadCheck 0.5%
- Merck EM Quant 5%

Selectivity. The rhodizonate is known to react with sulfate as found in plaster and wallboard. This reaction depletes the amount of rhodizonate available to react with the Pb and, thus, can cause false negative results. The National Institute of Occupational Safety and Health (NIOSH) Manual of Analytical Methods, Fourth Edition, 1996, Methods 7700 and 9105, lists Cd^{2+} and Sn^{2+} as interferences. The EM Quant Lead Test Catalog No. 10077 from Gallade Chemical Incorporated, Santa Ana, CA, lists the anions iodite, oxalate, sulfide, and sulfite and the cations Cu^{2+} , Sr^{2+} , Fe^{3+} , and Ba^{2+} as significant interferences. Of these potential interferents, only cadmium, iron, and barium are likely to be found in old paints and, therefore, be potential problems.

1.1.2 Semi-quantitative and Quantitative Test Kits

Semi-quantitative and quantitative test kits generally measure the intensity of a Pb concentration-dependent parameter against some standard. A variety of kits involve reaction of Pb ion in solution to form a colored complex with a Pb-specific reagent; the intensity of the absorbance measured with a colorimeter gives a measure of concentration (see Section 3.2.3). Reflectometry is a technique wherein the intensity of the color complex is measured by reflectance of incident wavelength-specific light from the complex on a substrate into a detector (see Section 3.2.2).

One quantitative test kit available is not based on color intensity measurement, but on the electrochemical reaction of Pb that has been dissolved to form the Pb ion, Pb^{2+} (see Section 3.2.2). This method, ASV, involves reducing the Pb ion in solution to form Pb metal on an electrode (i.e., $\text{Pb}^{2+} + 2\text{e} \rightarrow \text{Pb}^0$). After a period of time, the Pb metal is rapidly oxidized back to the ion form in solution, and the electrical current associated with this oxidation is related to the concentration of the Pb ion in the original solution.

1.2 Early Issue Papers

In the early 1990s, EPA assigned RTI two tasks: (1) to write a paper that would lay out the required performance parameters for test kits and (2) to conduct outreach to producers of test kits. Test kit manufacturers were very responsive to these efforts, which had a positive effect on the direction of early test kit development; today's kits are probably better because of this effort. The early work described here indicates the beginning of a concerted effort to advance the science and harmonize test kit performances.

1.2.1 Proposed Performance Parameters

In the first effort (EPA, 1991), performance requirements for test kits were proposed with respect to

- sensitivity,
- selectivity,
- accuracy,
- response time,
- safety,
- appearance,
- reproducibility, and
- stability.

The requirements proposed are described as follows.

Sensitivity. The optimum criteria for test kit sensitivity is the detection of Pb at the lowest concentration associated with adverse health effects (i.e., increases in blood lead levels). Criteria (EPA, 1991) were proposed for Pb in paint; these are shown in Table 2. Because a quantitative relationship between Pb-based paint and elevation of blood lead levels had not

Table 2. Proposed Analysis Performance Criteria for Pb In Paint as of 1991

<i>Standards for New/Replacement Paint</i>	
<i>Reference</i> CPSC, FDA	<i>Concentration</i> 600 ppm, 0.12 mg/cm ²
<i>Proposed Performance Criteria</i> Concentration Level of Concern: 0.06 % (w/w), 600 ppm 95% of results positive ≥ 0.045% (w/w), 450 ppm 95% of results negative ≤ 0.015% (w/w), 150 ppm	
<i>Standards for Abatement</i>	
<i>Reference</i> HUD State of Maryland State of Wisconsin	<i>Concentration</i> 1.0 mg/cm ² 0.7 mg/cm ² 0.7 mg/cm ²
<i>Proposed Performance Criteria</i> Concentration Level of Concern: 0.7 mg/cm ² 95% of results positive ≥ 1.0 mg/cm ² 95% of results negative ≤ 0.1 mg/cm ²	
<i>Comments</i> <ul style="list-style-type: none"> • No quantitative relationship between Pb level in paint and health effects had been established. • With pica activities, difficulty arises in transforming XRF values to average daily intake. • HUD considers 1.0 mg/cm² (5000 ppm) a positive XRF measurement for Pb and requires abatement at this concentration. • CDC considers 0.7 mg Pb/cm² paint a positive XRF measurement for Pb. • CPSC level of concern for new paint is 0.06% (600 ppm). • FDA = Food and Drug Administration 	

been established, criteria for paint were proposed for both abatement and clearance on the basis of guidelines already in existence. For abatement, levels considered positive from an instrumental standpoint were used to propose measurement criteria. Concentrations of 0.7 mg/cm² (positive by Centers for Disease Control and Prevention [CDC] standards) and 1.0 mg/cm² (positive by HUD standards) were considered unacceptable risks (i.e., abatement is necessary) and should result in a positive detection. A minimum level of approximately one-fifth of the CDC “positive” concentration (i.e., 0.1 mg/cm²) was proposed as negative for Pb.

Clearance standards were proposed on the basis of the 1978 maximum level for Pb in new paint (600 ppm) proposed by CPSC, and consideration of abated paint as a dust source (i.e., a clearly positive concentration of 450 ppm). Accordingly, clearance performance criteria recommended were 95% positive results at 0.45% and 95% negative results at 0.15%. Results of evaluations of test kits showed that a threefold range from clearly negative to clearly positive results is achievable for total Pb in solution (Luk et al., 1991). Test kit sensitivity is limited by the ability to extract Pb from the medium.

Selectivity. The test kits be selective for Pb over potential interferences. Through selection of the primary color-forming reagent; use of chemical agents to mask interferences; and other chemical parameters, such as pH; the selectivity ratio for Pb to any other potential interferences shall be 100 to 1.

Accuracy. Test kits on the market were shown to have poor accuracy. Results (EPA, 1991) were found to depend on the ability to extract Pb from the matrix, which is a function of the Pb species and the physical form of the matrix, rather than of the concentration of Pb in the matrix.

Criteria for accuracy, 95% of the results positive at a specified sensitivity, were believed to be achievable for concentrations proposed if test kit solutions extract Pb quantitatively (EPA, 1991).

Response Time. The test kits develop full color or change within 30 s and be stable for a minimum of 1 h to allow for a delay in noting the results or confirmation, if needed.

Safety. Hazard of materials (i.e., sodium rhodizonate) should be evaluated. Information on dermal effects, toxicity, etc., shall be indicated, if necessary, on enclosures similar to package inserts for medications or Material Safety Data Sheets for chemicals. Precautions and personal protection (e.g., gloves) shall be included if special handling needs are required.

The use of fracture- or splatter-resistant containers is important. The design of containers is particularly important when kits contain solutions. Special considerations for child safety, such as child-proof containers and vials, must be given to kits used by homeowners. Testing solutions, strips, etc., shall be sealed so that they are inaccessible to children.

Disposal instructions for solutions, paper strips, test ware (e.g., vials, cups, wands) shall be included in the test kit. Options, including flushing into the sanitary sewer or wrapping in newspaper for disposal in a landfill, shall be specified.

Appearance. Warnings be included in the test kit about physical properties that may affect accuracy and reproducibility of the test kit, including change in color or reagents, precipitates, etc.

Reproducibility. The test kits include some reference device or material to assure the reproducibility of the test kit. Options for this material include

- a standard test solution or Pb-impregnated strip, and
- a color chart or wheel.

Reproducibility shall be $\pm 10\%$ between individual test kits and between production lots.

Stability. Test kits be labeled with a production log number and an expiration date. Test kits shall have a shelf life of a minimum of 6 mo.

1.2.2 Manufacturer Outreach

In the outreach effort (Luk et al., 1992), manufacturer's of five test kits ([1] Frandon Lead Alert, [2] Verify LeadTest, [3] Hybrivet LeadCheck, [4] Merck EM Quant; and [5] Lead Detective) were asked to review a draft of the report, "Evaluation of Lead Test Kits for Analysis of Paint, Soil and Dust," (Luk et al., 1991) and two RTI/EPA documents, (1) "Options for a Lead Analysis Laboratory Accreditation Program" (Estes et al., 1991a) and (2) "Options for a Test Kit Certification Program" (Estes et al., 1991b) and to provide comments on the test kit evaluation report. Only three of the five companies successfully completed the outreach process.

Those three companies all indicated a serious interest in being of service to their customers through the availability of a toll-free, 800 number. This interest also was demonstrated through their plans or willingness to clarify the instructions included with each kit. None of the manufacturers considered the reagents in their kits to be hazardous. None thought laboratory gloves were needed. Frandon, Hybrivet, and Merck all stated that they had the capability to manufacture as many kits as could be sold. The Frandon test kit was designed for home use, whereas the Hybrivet and Merck kits were designed for both home and professional use. Hybrivet was promoting their kit as an adjunct screening tool to XRF, with their protocol for detection of more than 200 $\mu\text{g}/\text{ft}^2$ of Pb on surface. Also, all three manufacturers planned to develop and market quantitative kits. They also stated that the availability of reference materials would be extremely useful, both for development of new, quantitative kits and also as quality assurance materials for both qualitative and quantitative kits. Finally, all three manufacturers

agreed that verification of test kit performance is desirable. However, there were reservations about a verification process slowing the development and marketing of new products.

1.3 Preparation of This Issue Paper

As noted in the introduction, EPA's Proposed Rule *Lead; Renovation, Repair, and Painting Program* (EPA, 2006), provides the following performance standards and goals for a test kit to be recognized by EPA.

- Has a demonstrated probability (with 95% confidence) of a false negative response to Pb levels above the regulated level of less than 5%
- Has a demonstrated probability (with 95% confidence) of a false positive response to Pb levels below the regulated level of no more than 10%
- Can reliably be used by a person with minimal training
- Provides results within an hour and costs less than \$2 per test

The principle issue to be considered is whether test kits already available meet these standards of performance, and, if not, whether there is the potential for modified or newly developed kits to meet these standards. The principle purpose of this issue paper is to provide a detailed picture of the current status of test kits for Pb in paint, put in perspective with a limited history of their development and performance.

This issue paper has involved several tasks. First, a literature search was performed, and experts and test kit manufacturers were contacted. From this effort, a matrix was produced that describes all spot and quantitative tests that have been identified. Second, reports of past studies of the performance of test kits were identified and reviewed; summaries of these studies are provided in this issue paper. Next, a search for standard test materials was conducted and also is reported on in this paper. Finally, methods for evaluation of test kits were identified and are described.

2.0 AVAILABLE TEST KITS

An extensive search for available test kits was performed. Sources of information included the Internet, lists of test kits in previous reports, and communication with those in the field. Table 3 presents the results of this search in a matrix format. The parameters covered in this matrix are listed below.

- Kit name or type
- References
- Description
- Summary of method
- False negative rate
- False positive rate
- Training required
- Cost
- Analysis time
- Response range
- Types of coatings and Pb compounds that can be tested
- Interference
- Hazardous materials
- Target and current users

Because of the scope of all this information, the table is organized in the following manner. The first page shows the first five columns (through false negative rate) for the first group of test kits.

Then the second page shows the remaining columns for those same test kits (with the names of the test kits repeated for reference). The third and fourth pages show the same data for next group of test kits, and so on.

The information for each parameter for each test kit was taken from the literature that comes with the kit, from manufactures or sellers of the kits, and from written reports of studies of the test kits. Information for many of the parameters for many of the kits is not available because it has not been determined by the manufacturers or by researchers evaluating the kits. In some cases, manufacturers have indicated the information is proprietary. Although a thorough search was performed, it is likely that not all studies have been identified. Table 3 does contain all well-known brands of test kits.

3.0 PREVIOUS STUDIES OF TEST KITS

Following are summaries of several past studies of test kits. These summaries are provided to give an overview of the types of evaluations these kits have been subjected to and also to provide a history of the performance of the kits. They are presented to provide knowledge and understanding that hopefully will serve as a platform for identifying approaches or modifications that can be used with current or newly developed test kits for Pb in paint to adjust their performances to meet the new performance standards found in *Lead; Renovation, Repair, and Painting Program; Proposed Rule* (EPA, 2006).

3.1 Spot Test Kits

The majority of studies of test kits identified are focused on the spot test kits because the majority of the kits developed are spot test kits. They offer the simplest, fastest, and cheapest means of testing, and homeowners can use them, so they offer the greatest opportunity for retail sale and profit to the manufacturer.

3.1.1 RTI Laboratory Evaluation

In an early laboratory evaluation, RTI tested the performance of five spot test kits in the laboratory (Luk et al., 1993b). Through a search of the literature and trade journals and through contact with experts, the following five test kits were identified.

- (1) LeadCheck (Hybrivet Systems)
- (2) Verify LeadTest (Verify, Inc.)
- (3) Frandon Lead Alert (Frandon Enterprises)
- (4) Merck EM Quant (EM Science)
- (5) The Lead Detective (Innovative Synthesis Corp.)

A limited study of these five kits was performed to identify both positive attributes and limitations. Tests were performed with trained analytical chemists or technicians, except for the nontechnical user tests (which are described below). The following tests were performed.

- Response relative to test sample Pb content
- Potential metal interferences
- Potential salt interferences
- Response to laboratory-prepared and real-world paint, dust, and soil samples
- Color stability
- Accuracy of use by nontechnical personnel

Lower Level of Response. The first test determined the range of Pb content in test samples over which test kit responses went from negative to positive. This experiment was intended to estimate the identification limits (lower limits of response) of the kits.

Table 3. Summary of Available Test Kits and Their Operational Parameters

Kit Name or Type	References	Description	Summary of Method	False Negative
Accukits, 970-330-4238	Available from www.professional-equipment.com	Sample is sent to a lab for atomic absorption or inductively coupled plasma-atomic emission spectrometry (ICP) analysis.	The user sends a sample to the laboratory. SW846 7000 methods are used, either AA or ICP. Homeowners can send in wipes or paint samples.	No data
Acc-U-Test, South Shore Lead Paint Testing, 781-337-5546	www.sslpt.com ; Rossiter et al. (2000)	Color change, sodium sulfide	Place a drop of the reagent in a notch in the paint and look for formation of a dark or black color.	13% at 1.0 mg/cm ²
Bionanotechnology, DzymeTech, Dr. Lu, 217-333-2619	Lu (2005)	Under development. Small Business Innovative Research Contract from EPA. Dzyme proposes to develop a spot test kit based on Dr. Yi Lu's work at University of Illinois. The technology is a catalytic DNA-gold nanoparticle colorimetric sensor for Pb.	DNAzyme is selective for Pb (II). Gold nanoparticles assembled by the DNAzyme to form blue aggregates; Pb causes substrate cleavage, inhibition of assembly of nanoparticles, and red color formed.	No data; currently testing
Cole's Test, Cole Environmental, Sandra Cole, 216-961-7030	Information from EPA Docket and U.S. patents 6800485 and 6489170	Proprietary. Patented. Not yet commercially available. The test is based on the reaction of Pb with sodium rhodizonate under strong acid conditions. Dilute hydrochloric acid is used.	Cole's test forms a blue-purple color when Pb is present. The reaction is immediate. Two solutions are used—an acid and sodium rhodizonate. A positive paint chip for quality control (QC) would be provided. A color chart would be used for comparison.	None known
D-Lead, Esca Tech, 877-532-5323, Dan Askin	www.esca-tech.com	Two solutions. Color change. Trade secret but not patented. Originally developed for industrial applications but can be used by homeowners.	Contact one surface with one solution and then the second solution. Appearance of yellow color indicates Pb.	Does not work for lead chromate
EM Quant, 888-830-9092 (number is for a distributor)	Gallade Chemical Incorporated, www.emdchemicals.com , Gutknecht et al. (1997)	Rodizonic acid; test strips and reagent available	Pb reacts with rodizonic acid in an acidic solution (acetic acid is used) to form a red complex. Graduations of 0, 20, 40, 100, 200, and 500 ppm Pb available. The test detects ionic Pb.	>90% on surface, about 22% with notch

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	False Positive	Training	Cost	Time	Response Range	Types of Coatings and Pb Compounds That Can Be Tested	Interferences	Hazardous Materials	Target and Current Users
Accukits	No data	Unknown	\$12.95	Sample collection	ICP or AAS range	Paint	None of significance	None	Homeowner
Acc-U-Test	41% at 1.0 mg/cm ²	Required	\$12.95	5 min	0 to > 0.5%	Paint	Metals that form sulfide precipitates	Sulfide toxic, caustic	Paint testers
Bionanotechnology, DzymeTech	No data; currently testing	Two sensors are being investigated. With the colorimetric approach, the test would be a dipstick (yes or no answer) and a homeowner could use it. For the fluorescence sensor (quantitative answer), a trained inspector would use it.	Unknown	Testing is ongoing, but the sensing step takes about 2 min. The extraction step currently takes about 20 min.	The color change is clearly visible at 1 mg/cm ² . The range can be adjusted for the sensor or by using 2 sensors.	Pb (II)	Currently iron is one, but they are working to overcome the problem.	Dilute acid (such as vinegar) used	Homeowners for the simpler test; inspectors for the more complex sensor
Cole's Test	No data	Approximately 5 min	\$0.25 per test to manufacture; not yet commercially available	Immediate reaction	"99.999%" accurate; low end of range around 0.05%	Mainly paint	None known, according to company. According to the patent, this test avoids interference from barium.	Dilute hydrochloric acid is used	Contractors and homeowners
D-Lead	No data	For the test, read the instructions. Taking a good sample is critical.	\$75/120 tests; three sizes available	Immediate	As low as 20 µg Pb	Paint, hands, ceramics, etc., for surfaces; the kit does not work for chromate.	Dirt and bleach	None	Mainly commercial applications, homeowners
EM Quant	Essentially 0%	None	To be determined	5 min	>0.0+%	Ionic Pb only	Potentially red or pink paint	None	Homeowner

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	References	Description	Summary of Method	False Negative
First Alert, BRK, 800-323-9005	www.firstalert.com	Can use for "Rapid Method" or "Patented Leach Method;" yellow, black, or brown on the swab tip indicates that Pb is present.	For the rapid method, the swab is dipped into the indicator solution, then rubbed on the surface. The leach test uses vinegar to leach the Pb before the test. The company representative indicated that all of their information is available on their Web site.	No data
Frandon All-In-One (Card), 800-359-9000—number is now Caremark, not Frandon. EMS, 727-530-3602, sells a Pace Environs Lead Alert Professional Lead Test Kit; also Web site www.pactape.com has a Pb alert kit.	Rossiter et al. (2000)	Colorimetric with a test card	Pink or red on the applicator tip or test card indicates the presence of Pb. They recommend grinding the sample. A leaching solution is used.	24%
Full Disclosure (Surface Wipe), SKC, 724-941-9701, Connie Kelly	SKC, www.skcinc.com	Surface wipe with a color change if Pb is present; designed for occupational uses, including workers' hands	The wipe turns pink or red in the presence of Pb. The wipes also can be sent for atomic absorption spectrometry or anodic stripping voltammetry. Rhodizonate compound and acetic acid are used.	Unknown
Hach LeadTrak Pocket Colorimeter II, 800-227-4224. Hach Co., P.O. Box 389, Loveland, CO 80539	Simplified Testing for Lead and Copper in Drinking Water, Hach, Technical Information Series-Booklet 19, www.hach.com	Colorimeter	The sample is preserved and fixed with reagents. A column is used to separate the Pb. pH is adjusted. The sample is mixed with an indicator. The level is read with the colorimeter. Uses 4-(2 pyridylazo)-resorcinol as color-forming reagent.	No data
HazCat Lead in Paint Test Kit, David Mandeville, 800-543-5487. Associated with HazTech System, Inc., Mariposa, CA	www.hazcat.com, Costa (1961)	Chloranilic acid as indicator. A positive detection results in a brown precipitate.	A pea-size amount of crushed paint is placed on a watch glass. Three drops of metal extraction solution and four drops of Pb test are added. Pb is indicated by brown solution or precipitate.	Unknown
Home Free (MACS); Jim Richards, 800-622-7522	Available from www.professional equipment.com and mall.ballparks.com	Atomic absorption spectroscopy	The user sends a sample to the laboratory.	Unknown

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	False Positive	Training	Cost	Time	Response Range	Types of Coatings and Pb Compounds That Can Be Tested	Interferences	Hazardous Materials	Target and Current Users
First Alert	No data	Read the manual before use	Locally owned hardware store, \$9.99	Up to 4 h	As low as 1 to 3 ppm.	Paint, ceramics, dust, etc.	Copper and bismuth are potential interferences.	Sulfide, toxic, caustic	Paint tester and homeowner
Frandon All-In-One (Card)	15%	Unknown	EMS sells the Frandon's professional kit for \$29.95.	Unknown	At a concentration of 0.3 mg/cm ² , there is a 95% probability of a negative result.	Paint	Red paint may cause a problem. Barium may produce an orange response. Gypsum, plaster, and stucco may interfere.	None	Homeowner and paint tester
Full Disclosure (Surface Wipe)	Unknown	No more than 5 min training would be required.	\$29.95 for kit	Immediate reaction	18 µg is the lower limit of detection.	Designed for testing hands; not meant for detecting lead chromate, alkyl Pb, or other less soluble Pb types	Silver, cadmium, barium, mercury, and titanium	Dilute acetic acid is used.	Designed for occupational uses
Hach LeadTrak Pocket Colorimeter II	No data	1 h+	Set up is \$300 to \$1600; cost per sample is \$4 to \$5.	3 min to set up; 10 min per test	Minimum detection limit in the manual is 5 µg/L. Manual lists precision of 70 ± 10 µg/L. Detection limit of 2 µg/L; range of 2 to 150 µg/L.	Water	The manual lists several metal interferences, including aluminum, copper, iron, and zinc.	Buffers minimal	Professional water tester
HazCat Lead in Paint Test Kit	Unknown	Unknown	\$111 for 25 to 100 tests	Unknown	Limit of identification is 5 µg Pb.	Paint	Co (II), Ag (I), Hg (I), Hg (II), Bi (III)	Chloranilic acid	Designed for anyone
Home Free (MACS)	No data	Unknown	\$21.95	Sample 15 min, read 20 min	Unknown	Paint, water, soil	Unknown	Unknown	Homeowner

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	References	Description	Summary of Method	False Negative
Know Lead Kit by Carolina Environment, 704-598-1397, 800-448-LEAD	Unable to contact; neither phone number was working.			
Lead Alert Wipe/Sanding Test Kit (according to OSHA notes, Lead Alert was developed by Frandon Enterprises, formerly sold by Sensidyne, owned by Pace Environs; number for Pace Environs from Web, 467-7578, no answer, no matches with AnyWho)—EMS, 727-530-3602, sells a Pace Environs Lead Alert Professional Lead Test Kit; see more details under Frandon.	EMS, www.emssales.net ; PPI Pace, www.pactape.com	Color change	According to the OSHA Web site, the kit includes an indicating solution, leaching solution, and indicating tablet.	20% to 50%
LeadCheck (Hybrivet), 800-262-LEAD	www.leadcheck.com , Luk et al. (1993b), EPA (1995b), Gutknecht et al. (1997), Rossiter et al. (2000)	Colorimetric	The swab contains two ampules, which are broken open and mixed. The swab is squeezed until yellow liquid comes out. A distinctive pink or red color forms if Pb is present.	Less than 5%
Chromate Check (Hybrivet), 800-262-LEAD, Marcia Stone	www.leadcheck.com	Colorimetric	Lead chromate is not often used in household paints, but may be found in marine or industrial paints. A pink to purple color change indicates the presence of chromate pigments.	No known cross-reactions
Lead Detective, 617-965-5653	www.gis.net/~mtf/tldhome.htm	Sodium sulfide solution	Sodium sulfide is mixed with paint. If Pb is present, the sample turns black.	<5% to 25%
JNJ Lead Detector, 800-554-9994	www.jnj-industries.com	The kit includes LeadCheck test swabs, detecting powder, acetic acid extraction solution, and developing solution. The Web site has all of the information, the representative said. JNJ_Industries.com	Pink or red color develops if Pb is present. Positive wipes and swabs can be sent to lab for quantitative analysis.	No data
Lead Inspector (Abotex), 800-268-LEAD	www.leadinspector.com	Sodium sulfide solution	The swab is dipped into the indicator vial. The surface is rubbed with the swab. A color change indicates that Pb is present.	If Pb is bound

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	False Positive	Training	Cost	Time	Response Range	Types of Coatings and Pb Compounds That Can Be Tested	Interferences	Hazardous Materials	Target and Current Users
Know Lead Kit	—	—	\$11.95 for four (old data)	—	—	—	—	—	—
Lead Alert Wipe/Sanding Test Kit	19% to 20%	None	\$20/kit according to OSHA Web site	5 min per test	0.5 µg (-) to 0.7 µg (+) 0.6 mg/cm ² (-) to 1.2 mg/cm ² (+)	Paint	Thallium, silver, cadmium, tin	None	Homeowner and paint tester
LeadCheck (Hybrivet)	20% to 70%. See EPA (1995b), Gutknecht et al. (1999), Rossiter et al. (2000)	None	Local Home Depot, \$5.67; locally owned hardware store \$6.99; EcoKitchen sells a four-pack for \$9.99.	Less than 1 min	0.38% w/w 0.5 µg (-) to 1 µg (+) 1.2 mg/cm ² (-) to 1.9 mg/cm ² (+)	Wood, paint, metal, ceramics, vinyl, costume jewelry, etc.	Potentially red or pink paint	None	Anyone
Chromate Check (Hybrivet)	No known cross reactions	Read and follow directions	\$39.95 for eight; lower if more are purchased; expect the price to come down	Immediate reaction	0.5 µg is the low end.	Plated surfaces and paints; chromate	No known	Acid, but no hazardous materials	Commercial ship builders and renovators; those testing plated surfaces
Lead Detective	40% to 70%	Required	\$29.95	5 min	0.5 µg (-) to 2 µg(+)	Paint	Metals that form sulfide precipitates	Sulfide toxic, caustic	Paint testers
JNJ Lead Detector	Same as LeadCheck	None		5 min	Same as LeadCheck	Paint	Potentially red or pink paint	None	Homeowner
Lead Inspector (Abotex)	Copper, bismuth, and iron		Eight-pack for \$12.99	Immediate reaction.	1-ppm detection limit	Any surface—vinyl, paint, ceramic, etc.	Copper, bismuth, iron		Homeowners

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name orType	References	Description	Summary of Method	False Negative
MacLellan Water Technology	William Vandervilp, 800-200-0865, www.mwater.ca	Color change	Fill pipette with water; add the test strip; turns black if greater than 15 ppb.	No data
Palintest SA-1000 Scanning Analyzer, 800-835-9629, George Belarski	www.palintestusa.com; Sussel and Ashley (2002)	Voltammetry, disposable electrode	The test sample is mixed with a conditioning tablet in a tube. The electrode is immersed in the sample.	AA vs ASV: Slope ~0.7, Intercept ~0.30g, r^2 ~0.8
Pro-Lab, 800-427-0550	www.prolabinc.com	Impregnated test pad. No additional information that the company representative could send.	The test pad is moistened and then rubbed over the surface. Pink or purple color indicates the presence of Pb.	No data
PurTest Lead Test for Water	www.silverlakeresearch.com	Immunoassay test	Add Lead-a-Finders to water. Place test strip in reaction bottle. Pink line appears on strip if Pb is greater than 15 ppb.	None observed
Rapid immunochromatographic strip tests, Silver Lake Research, Mark Geisberg, 888-438-1942	www.silverlakeresearch.com	Under development. Small Business Innovative Research Contract from EPA. Silver Lake intends to develop a paint test strip using immunoassay. They have an immunoassay test for water (see above).	The test is still under development.	No data
SenSafe Lead Paint Test Kit	www.sensafe.com	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below
Watersafe, Silver Lake, 888-438-1942 is a distributor, Industrial Test Systems is the manufacturer, 888-861-9712	Industrial Test Systems, Inc., www.sensafe.com; Watersafe, www.watersafetestkits.com	Test strips with dithizone	Vinegar (reagent A) is used to extract the Pb. Dithizone is used as a color indicator. Pink is indicative of Pb.	No data

Table 3. Summary of Available Test Kits and Their Operational Parameters (cont'd.)

Kit Name or Type	False Positive	Training	Cost	Time	Response Range	Types of Coatings and Pb Compounds That Can Be Tested	Interferences	Hazardous Materials	Target and Current Users
MacLellan Water Technology	No data	None	Paint \$14.99, soil \$24.99, water \$23.99	10 min	> 600 ppm and > 5000 ppm	Water	Unknown	Unknown	Homeowner
Palintest SA-1000 Scanning Analyzer	No data	1 to 2 h	\$1028 for instrument with sensor pack	Collection, extraction, 10 min measure	2 to 100 µg/L	Water only for the 1000	None	Extraction reagent	Water tester
Pro-Lab	No data	Read the instructions before use	Locally owned hardware store, \$9.99	5 min	Unknown	Paint, ceramics, etc.	Unknown	No	Homeowner
PurTest Lead Test for Water	None observed	Unknown	\$14.95	Within 10 min	At 13 ppb 10/10 are negative; at 18 ppb, 10/10 are positive	Water	No cross- reactivity observed.	Unknown	Homeowner
Rapid immunochro- matographic strip tests	No data	No needed training is anticipated.	Planned to be under \$5	Expected to take under 10 min	Unknown	Paint	Unknown	No	Homeowners
SenSafe Lead Paint Test Kit	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below	Same as Watersafe below
Watersafe	No data	Unknown	\$14.95 for 10	Within 5 min	At 600 ppm, 80% read positive. Range of 600 to 5000 ppm	Lead chromate would not be extracted with vinegar and would not be detected.	Zinc, cadmium, silver, tin, gold, molybdenum, and chromium may give a positive result. Nitrate and chloride may reduce sensitivity.	Unknown	Unknown

Following instructions provided with the test kits, each brand of kit was tested with solutions prepared with $\text{Pb}(\text{NO}_3)_2$ and PbCl_2 to determine range of response. The test kits were reacted with 10 to 80 mL quantities of Pb solution from well below the point of color development to well above the point of color development. The test sample Pb content ranges corresponding to all negative responses to all positive responses are given in Table 4.

Table 4. Test Kit Response to Pb^{2+} in Solution (All Negative to All Positive)

Test Kit	$\text{Pb}(\text{NO}_3)_2$	PbCl_2
LeadCheck (original and new)	0.5 – 1.0 μg	<0.4 – 0.8 μg
Verify LeadTest	0.1 – 0.3 μg	<0.1 – 0.2 μg
Frandon Lead Alert	0.5 – 0.7 μg	0.1 – 0.3 μg
Merck EM Quant (A)	0.5 – 0.6 μg	NA
Lead Detective Kit	0.5 – 2.0 μg	0.5 – 1.0 μg

Based on these results, the kits rank as follows from lower level of response to higher.

- (1) Verify LeadTest (0.3 $\mu\text{g/g}$)
- (2) Merck EM Quant (A) (0.6 $\mu\text{g/g}$)
- (3) Lead Alert (0.7 $\mu\text{g/g}$)
- (4) LeadCheck (1.0 $\mu\text{g/g}$)
- (5) Lead Detective (2.0 $\mu\text{g/g}$)

The chemical form of the Pb solution affects the responsiveness of the kits. This effect may be result from a combination of competition for complexation of Pb^{2+} by species other than rhodizonate ion and/or changes in pH or ionic strength. The size of the range over which rhodizonate-based kits went from all negative to all positive varied from 0.1 μg for the Merck EM Quant A (all negative at 0.5 μg to all positive at 0.6 μg) to 0.5 μg for the LeadCheck (all negative at 0.5 μg to all positive at 1.0 μg).

Metal and Salt Interferences Tests. Paints, dusts, and soils may contain metal species other than Pb that react with the rhodizonate ion or sulfide to form a colored product and thus yield false positive results. Other species in the samples may inhibit color formation by reacting with the Pb or causing shifts in pH or ionic strength and thus yield false negative results.

Color-forming (positive) interferences by metals were investigated for the Frandon Lead Alert kit using atomic absorption standard solutions. The Frandon Lead Alert kit was used because it appeared to represent the average rhodizonate-based kit. The standard solutions were usually acidic (2% HNO_3 , dilute HCl) as were the kit reagent; thus the test conditions were assumed to be acidic. When nominally 100 μg (100 μL , 1000 ppm) of potentially interfering metal ions were put in contact with the test element (i.e., swab) of each kit, only Ba^{2+} (which gives an orange color with Pb) and Ni^{2+} yielded responses that could be interpreted as positive for Pb. Feigl and Suter (1942) reported that Ag^{1+} , Hg^{2+} , Tl^{1+} , Pb^{2+} , Cu^{2+} , Sn^{2+} , Zn^{2+} , Ba^{2+} , and Sr^{2+} all gave responses to sodium rhodizonate in neutral and/or pH 2.8 solution. Their test procedure involved mixing high levels of the metal (1%, 10,000 ppm) with 0.2% sodium rhodizonate, which could account for the difference in results. They reported that the selectivity of sodium rhodizonate favors Pb^{2+} over the majority of these metals and, in particular, that the selectivity for Pb over barium is 10,000 to 1. The sulfide-based Lead Detective kit tested with these same samples showed responses to Ag^{1+} , Cd^{2+} , Co^{2+} , Cu^{2+} , Fe^{3+} , Hg^{2+} , Ni^{2+} , and Ti^{2+} , all of which are known to form insoluble sulfides. Both high levels (2000:1, $\text{Cl}^-:\text{Pb}^{2+}$) and moderate levels (200:1, $\text{Cl}^-:\text{Pb}^{2+}$) of chloride (as NaCl) were found to result in decreased response (negative interference) for the Frandon Lead Alert, Verify LeadTest, and Lead Detective kits. Other salts were tested as possible interferences. A series of solutions was prepared with different

concentrations of NaNO_3 , KNO_3 , $\text{Na}(\text{C}_2\text{H}_3\text{O}_2)$, and $\text{K}(\text{C}_2\text{H}_3\text{O}_2)$ mixed with $1\text{ }\mu\text{g}$ of Pb^{2+} and tested in duplicate using the LeadCheck test kit. The purpose for testing these materials was that they had potential for use as buffers, which might be needed to make Pb paint extracts compatible with test kits chemistries. The Na^{1+} (or K^{1+}) to Pb^{2+} ratios at which negative interferences occurred are as follows.

Compound	$\text{Na}^{1+}(\text{or } \text{K}^{1+}):\text{Pb}^{2+}$ Ratio
NaNO_3	1000:1
KNO_3	1300:1
$\text{Na}(\text{C}_2\text{H}_3\text{O}_2)$	200:1
$\text{K}(\text{C}_2\text{H}_3\text{O}_2)$	200:1

Thus, it appears that the sodium and potassium salts interfere, although it is not clear if the Na^{1+} and/or K^{1+} interfere. The effect of the salts may be a result of a change in ionic strength. The acetate presents even a greater extent of interference, which may result, in part, to a pH effect or formation of a lead acetate complex.

Response to Laboratory-Prepared Paint Films. After tests with National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs), the test kits were further challenged by measuring responses to laboratory-prepared paint films (EPA, 1995a). Seven oil-based paint films spiked with white lead were prepared. Following kit instructions, paint sections averaging 1.1 cm^2 in area were tested. Concentration ranges over which the color appeared (i.e., all negative to all positive) are given in the Table 5. Because a limited number of paint films were prepared and used, transition curves (response versus concentration) were not well defined.

Table 5. Test Kit Response to Pb in Paint Films (All Negative to All Positive)

Test Kit	Response Range
LeadCheck (original)	$1.9 - 2.6\text{ mg/cm}^2$
LeadCheck (new)	$1.2 - 1.9\text{ mg/cm}^2$
Verify Lead Test	$0.6 - 1.2\text{ mg/cm}^2$
Frandon Lead Alert	$0.6 - 1.2\text{ mg/cm}^2$
Merck EM Quant (A)	$1.2 - 1.9\text{ mg/cm}^2$
Lead Detective Kit	$<0.11\text{ mg/cm}^2$ (i.e., transition occurs below 0.11 mg/cm^2)

Color Stability Tests. A concern with the test kits was the rate of formation and stability of the color formed as a result of a positive response. Slow formation or rapid fading of the color could lead to a positive response being interpreted as a negative response. To test color formation and stability, the rhodizonate-based kits were tested with respect to time stability of the color developed. When exposed to amounts of Pb in solution just above the detection limit, only the Verify LeadTest and EM Quant Method A kits showed fading of the color from pink to yellow within 30 min. All kits showed no fading for at least 15 min after reaction with Pb.

Nontechnical User Tests. The test kits were designed for use by homeowners or professionals. Any improper use of the kits could affect the outcome of the tests. Therefore, ease and accuracy of use were tested by having nontechnical personnel use the kits while being observed by an experienced chemist. Two nontechnical staff members were provided with kits, written procedures, and RTI-prepared paint films for analysis. Each was instructed to perform duplicate analyses. There was considerable variation in the results, as shown in Table 6.

Table 6. Results of Nontechnical User Tests

<i>Number of Negative and Positive Responses</i>				
Test Kit	Tester #1		Tester #2	
Test Paints (1.6 mg/ cm ²)	Negative	Positive	Negative	Positive
LeadCheck (new)	1	1	2	0
Verify LeadTest	2	0	0	2
Frandon Lead Alert	0	2	0	1
Merck EM Quant	4	0	3	1
Lead Detective	0	2	0	2

Example problems noted by an experienced observer included those that follow.

- Not following instructions
- Confusion over the order of use of the two reagent tubes in the LeadCheck kit
- Variation in firmness of rubbing paints
- Stirring with the reaction zone of the Merck Em Quant test strips rather than the “upper end” as called for in the instructions

Relationship to Proposed Performance Criteria (EPA, 1991). EPA had developed target criteria for performance of the test kits for different media (Estes et al., 1991b). The approach proposed a 95% negative response at levels corresponding to minimal known health effects or not requiring regulatory action and a 95% positive response at levels corresponding to suspected significant health effects or requiring regulatory action. The target and actual results are shown in Table 7, which shows that the measured ranges of response to paint (negative to positive) were higher than the proposed target levels for all the rhodizonate-based kits. The opposite is true for the sulfide-based Lead Detective kit, which had a response range (negative to positive) below target levels.

Table 7. Comparison of Target Performance Criteria (EPA, 1991) and Actual Performance Results on Paint (Luk et al., 1993b)

Test Kit	Early Target EPA Performance Criteria	Actual Performance Results
LeadCheck (new)	95% positive at 0.7 mg/cm ² 95% negative at 0.1 mg/cm ²	All positive at 1.9 mg/cm ² All negative at 1.2 mg/cm ²
Verify LeadTest	95% positive at 0.7 mg/cm ² 95% negative at 0.1 mg/cm ²	All positive at 1.2 mg/cm ² All negative at 0.6 mg/cm ²
Frandon Lead Alert	95% positive at 0.7 mg/cm ² 95% negative at 0.1 mg/cm ²	All positive at 1.2 mg/cm ² All negative at 0.6 mg/cm ²
Merck EM Quant	95% positive at 0.7 mg/cm ² 95% negative at 0.1 mg/cm ²	All positive at 1.9 mg/cm ² All negative at 1.2 mg/cm ²
Lead Detective	95% positive at 0.7 mg/cm ² 95% negative at 0.1 mg/cm ²	All positive at 0.6 mg/cm ² All negative at 0.1 mg/cm ²

The general conclusions from this earlier study (Luk et al., 1993b) were as follows.

- (1) The kits generally respond to less than 1 µg of Pb²⁺ in solution.
- (2) Positive interferences were not found for the rhodizonate kits for the limited set of paint, dust and soil samples used in this study. However, barium, which yields a “pinkish” orange color with rhodizonate, may be interpreted by some as Pb.

- (3) The dark colors of certain dust samples masked observation of formation of lead sulfide at low levels with the Lead Detective Kit. Positive responses with the Lead Detective resulted from Ag^+ , Co^{2+} , Cu^{2+} , Fe^{2+} , Fe^{3+} , Hg^{2+} , Ni^{2+} , and Ti^{2+} . Many of these metals may be found in paints, dusts, and soils.
- (4) All kits showed adequate stability (>15 min) of the developed color.
- (5) Tests with untrained, nontechnical personnel showed significant variability in usage and, consequently, in results.
- (6) The measured response ranges (negative to positive) of the rhodizonate-based kits were generally above the targeted ranges set by EPA. The sulfide-based Lead Detective kit yielded positive responses to “blank” paint (i.e., with no Pb added), and, therefore, for RTI test paint films, response ranges were below the targeted ranges.

Based on the results of this limited study, the rhodizonate kits were considered to have adequate sensitivity to measure available Pb in solution to meet the EPA target criteria. That is, the chemistry of the kits allowed easy detection of Pb at the lower levels of concern, provided that the Pb is available to react with the test kit reagent(s). The sulfide-based kit responds to levels below the target level for most samples.

The issues addressed in this work included the following.

- *False positive and negative results*
- *Interferences*
- *Need for training*

3.1.2 HUD/QuanTech Field Study

Midwest Research Institute and QuanTech, Inc., performed a large field study in 1993 that included evaluation of portable XRF and field test kits (EPA, 1995b). The study was conducted in Louisville (pilot study), Denver, and Philadelphia. The study involved both multifamily and single-family dwellings. Including those locations in the pilot study, a total of 1290 individual test locations on six substrate types in 22 housing units were tested. The breakdown of testing locations by substrate was as follows: 93 brick, 226 concrete, 124 drywall, 217 metal, 242 plaster, and 388 wood.

The test kits in this study represented the range of kits available at the time the study was conducted. Test kits from five different manufacturers were tested in this study: three rhodizonate based kits, two sodium sulfide based kits, and one proprietary kit. The rhodizonate-based kits included were LeadCheck and the sanding and coring versions of Lead Alert; the sodium sulfide kits were Lead Detective and the Massachusetts State-approved kit. The pilot study also included the Lead Zone kit, which uses proprietary chemistry. The results of the spot tests performed in the field were reported as either negative or positive. Paint was taken from the 1290 test locations and returned to the laboratory for analysis for Pb using acid digestion and measurement using ICP. These ICP results were then used to evaluate the test kits responses.

Table 8 shows overall false positive and false negative rates for the test kits compared to laboratory analytical results using the 1-mg Pb/cm² threshold. Table 9 shows the corresponding rates for the 0.5% threshold.

One immediately notes a great deal of variability with these spot tests. For example, for both the 1.0-mg/cm² and the 0.5% thresholds, false negative percentages vary from a few percent to more than 50%. Also, the high levels of false positive results indicate the spot test kits are very sensitive and are responsive to amounts of Pb that are effectively less than the threshold values.

Table 8. Overall False Positive and False Negative Rates for Test Kits Compared to Laboratory Analytical Results Using the 1.0 mgPb/cm² Threshold

Test Kit	False Positive Percentage	False Negative Percentage
LeadCheck	46%	6%
Lead Alert: Coring	15%	24%
Lead Alert: Sanding	9%	53%
Lead Detective	36%	23%
Lead Zone	28%	14%
MA Sodium Sulfide	65%	1%

Table 9. Overall False Positive and False Negative Rates for Test Kits Compared to Laboratory Analytical Results Using the 0.5% Pb Threshold

Test Kit	False Positive Percentage	False Negative Percentage
LeadCheck	42%	11%
Lead Alert: Coring	11%	36%
Lead Alert: Sanding	19%	67%
Lead Detective	32%	27%
Lead Zone	25%	25%
MA Sodium Sulfide	62%	6%

It is possible to have interferences from the substrates as described in Section 3.1.1. The calcium in plaster apparently ties up the rhodizonate resulting in false negatives for kits based on this reagent. On the other hand, metals such as iron react with sulfide, and, therefore, a steel substrate will lead to false positives with kits based on the sulfide reaction with Pb. In this study, as noted above, the paint samples were taken from a variety of substrates. Table 10 shows the Pb level in mg/cm² at which there is a 50% probability of a positive test kit result, as estimated from a statistical model developed in this study. Table 11 shows the Pb level in percent by weight. These values represent the 50:50 point or inflection in the normal response curve. One sees very few combinations of test kit and substrate yielding 50:50 points at either 1.0 mg/cm² or 0.5% Pb.

Here too, one sees that the substrate has an impact on the results for the individual spot test kits, and, again, there is great variability between the spot test kits.

Table 10. Pb Level (mg/cm²) at Which There Is a 50% Probability of a Positive Test Kit Result

Test Kit	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.02	0.19	1.14	0.34	0.13	0.03
Lead Alert: Coring	0.33	1.84	NA	0.65	NA	0.77
Lead Alert: Sanding	NA	NA	NA	NA	NA	1.24
Lead Detective	0.05	0.60	NA	0.55	0.98	0.20
Lead Zone	0.08	1.38	0.31	0.82	0.71	0.15
MA Sodium Sulfide	0.01	0.01	0.08	0.08	0.02	0.04

Table 11. Pb Level (Percent Pb by Weight) at Which There Is a 50% Probability of a Positive Test Kit Result

Test Kit	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.02	0.16	0.56	0.32	0.14	0.07
Lead Alert: Coring	0.13	1.14	NA	1.09	NA	0.97
Lead Alert: Sanding	NA	0.88	NA	NA	NA	1.68
Lead Detective	0.01	0.33	NA	0.63	0.58	0.38
Lead Zone	0.07	0.49	0.35	1.03	0.44	0.26
MA Sodium Sulfide	0.01	0.01	0.13	0.08	0.02	0.09

High levels of Pb were not always detected with complete certainty using test kits. In a number of cases, the limiting probability of a positive test kit result was much lower than the desired value of 100%. This occurred for four of the six kits: Lead Alert (Coring) on metal; Lead Alert (Sanding) on concrete, metal, and wood; Lead Detective on concrete, metal, and plaster; and Lead Zone on plaster.

The principle issue addressed in this work was the following.

- *False positive and negative results*

3.1.3 OSHA Study

In an OSHA study (Adler, 1994; www.osha.gov/SLTC/leadtest/leadkits.html), the LeadCheck and Lead Alert All-in-One Professional kits were evaluated. Interference tests showed that barium yielded an orange color, which could be misinterpreted, and that gypsum, plaster dust, and stucco yielded negative interferences with these kits. Samples tested in this study included soluble Pb films in Petri dishes and paint from wood and plaster of older homes. Soluble salt

films were prepared in Petri dishes at 0.1, 0.6, 1.0, 1.4, and 24 mg/cm². Both test kits showed positive responses at the 0.1 mg/cm² level. The expected values for the real-world paints were determined using inductively coupled plasma-optical emission spectrometry (ICP-OES) and AAS. Some of the results are presented in Table 12.

From experiments both with the Petri dish films and the real-world paints, one can see the kits demonstrate their “protective” nature and respond to well below 1 mg Pb/cm² and 0.5% Pb.

The issues addressed in this work included the following.

- *False positive and negative results*
- *Interferences*

3.1.4 Laboratory Evaluation of the LeadCheck Test Kit

The LeadCheck test kit was evaluated by Scharman and Krenzelok (1996). These researchers tested 26 paint chip samples in triplicate following the manufacturer’s instructions. The samples ranged from <0.00007% to 28.0%, as determined by AAS; 14 of the samples contained less than 0.5% Pb. A false positive was defined as a failure of the pink or red color change when the paint sample contained more than 0.5% Pb. The results were reported as “sensitivity” and “specificity,” where

Sensitivity = (# true positives)/(# true positives + # false negatives), and

Specificity = (# true negatives)/(# true negatives + # false positives).

Table 12. OSHA Study Results

Paint Source	LeadCheck Results	Lead Alert Results	ICP Analysis (wt%)
Garage (green paint) (home #1)	Cut made at angle into paint - intense pink. Pealed paint, side adjacent to wood - intense pink	Total Pb test - immediate dark pink color on swab	Pb 3.1 Zinc 14
	Surface test - immediate distinct pink on swab	Surface test - immediate distinct pink on swab	
Paint chips from fence (home #1)	Surface test, side of paint formerly toward wood (dirty), cleaned with wet wipe - distinct pink	Total Pb test - no pink color observed on swab.	Pb 0.03 Zinc 3.0
	Surface test, side of paint away from wood - no pink	Surface test, side of paint formerly toward wood (dirty), cleaned with wet wipe - faint pink, obscured by dirt	
Paint chips from bottom of doorpost outside living room door (home #2)		Surface test, side of paint away from wood - no pink color observed.	Pb 0.14 Zinc 6.9
	Surface test, side of paint formerly toward wood (green) - pink patches on paint	Total Pb test - no pink color observed on swab.	
		Surface test, side of paint formerly toward wood (green) - faint but distinct pink on swab	
		Surface test, side of paint away from wood (white) - no pink, even after 15 min	

At room temperature, the sensitivity was found to be 91.7% and the specificity to be 77.8%. Similar to other studies, the false negatives were in the range of 5 to 10% and the false positives were in the range of 20% to 30%.

The principle issue addressed in this work was the following.

- *False positive and negative results*

3.1.5 RTI Pilot Field Study

A pilot field study (Gutknecht et al., 1997) was conducted for EPA that served as means of establishing procedures for a larger field study to follow (see Section 3.1.7). The research involved the evaluation of three brands of portable XRF instruments, seven qualitative test kits, and a quantitative colorimetric test kit. The tests were conducted on a set of nine wood cabinet doors and nine locations on a painted fiberboard wall in residential dwellings in Durham, NC. Paint samples returned to the laboratory were analyzed using microwave digestion and inductively coupled plasma emission spectrometry (MW/ICP). This method is well established (Binstock et al., 1991).

The method of sample preparation was critical. In this laboratory study, the paint from the wood was first ground using a mortar and pestle. This ground material showed the presence of flecks of paint, probably a latex overcoat that did not readily grind into fine powder. When this same paint was ground at dry ice temperature, a finer powder was obtained. The cryogenic grinding reduced the standard deviation of the MW/ICP analyses by about 80% relative to that achieved with the mortar and pestle.

The variability between test point locations determined by MW/ICP was considerable. The Pb-in-paint-on-wood levels varied from $3.30 \pm 0.02\%$ ($1.73 \pm 0.01 \text{ mg/cm}^2$) to $4.79 \pm 0.12\%$ ($2.40 \pm 0.06 \text{ mg/cm}^2$), and the Pb-in-paint-on-fiberboard levels varied from $1.34 \pm 0.21\%$ ($2.26 \pm 0.36 \text{ mg/cm}^2$) to $1.97 \pm 0.19\%$ ($2.14 \pm 0.21 \text{ mg/cm}^2$).

Seven qualitative kits were selected for testing. Each kit was tested on three different 1 x 1-in squares in the same location used for the quantitative kit test. The test area, which was adjacent to the area used for XRF measurements and collection of paint for MW/ICP analysis, was 2 x 12 in and divided into 24 1 x 1-in squares. Of the 24, 3 squares were used for the quantitative kit and 21 squares were used for the qualitative test kits; the individual squares were assigned randomly to the different kits.

The kits tested were as follows.

Sulfide Based

- Accu-U-Test
- Lead Detective
- Lead Inspector

Rhodizonate Based

- EM Merck
- Frandon Lead Alert
- LeadCheck
- Frandon-All-in-One

Table 13 summarizes the method of usage of these kits.

Table 13. Summary of Method of Usage for Test Kits Evaluated in RTI Pilot Field Study

Sample Form	Method of Testing	Brand of Kit
Surface of paint	Press test strip	EM Merck
	Rub with swab	Frandon Lead Alert
Invasive	Notch paint and test	LeadCheck
		Acc-U-Test
		Lead Detective
	Core paint and test	Frandon All-in-One
Extract into solution		Lead Inspector

Five of the seven test kits showed all positive responses to all the tests for both the paint on wood and the paint on fiberboard. The EM Merck and Frandon Lead Alert showed a mixture of negative and positive responses to the paint on wood and all negative responses to the paint on fiberboard. This difference is due in large part to the method of testing. When the invasive method of notching was used with the EM Merck and Frandon Lead Alert kits, the paint on the wood yielded all positive responses for these two brands; using the invasive technique of notching resulted in some positive responses on the fiberboard using the EM Merck kit and all but one positive response using the Frandon Lead Alert kit.

The principle issue addressed in this work was the following.

- *False positive and negative results*

3.1.6 Field Evaluation of the LeadCheck Test Kit

A study was carried out by Ashley et al. (1998) that involved testing of XRF, ASV, and qualitative test kits at some 200 locations in an old school building. The paint substrates included plaster, wood, metal, and brick. The expected values were determined by removing a paint sample, grinding it, extracting the Pb in acid using ultrasonication, and measuring the Pb level using AAS.

Only one brand of qualitative test kit was used, the LeadCheck swabs (HybriVet Systems, Inc.). In situ spot test kit analysis was conducted according to ASTM Standard E1753 (ASTM, 2004). First, the surface was cleaned using a new individually wrapped wet wipe (Wash N Dry). A notch was then cut into the center of the paint film sample area down to the substrate so as to expose all paint layers. The chemical spot test then was conducted in the cut following the ASTM procedure and the manufacturer's instructions, and the presence or absence of the characteristic color for Pb was noted. Also, the relative intensity of the pink color formed was noted. Swabs showing a negative response were checked after 24 h for any color formation. Any negative result was confirmed with the use of a positive control, wherein a test card that contained lead nitrate was tested using the same LeadCheck swab that gave a negative in situ reading on the paint film sample.

The resulting response curves were very similar to those reported previously (as described in Section 3.1.4), with a relatively large number (about 30%, based on estimation from the plots in the publication) of positive responses below the action levels of 0.5% or 1 mg/cm². The test kit data showed 4.5% false negative readings for samples with Pb levels above the 0.5% action level. The test kit results also showed about 4% false positive readings for samples with Pb levels below the CPSC action level of 0.06% Pb. The number of false negative results and false positive results by substrates are shown in Table 14.

Table 14. False Negative and False Positive Rates for Field Evaluation of LeadCheck Kit (Ashley et al., 1998)

Substrate	Number of Observations	Number of Negative Results at >0.5%	Number of Positive Results at <0.06%
Plaster	44	1	0
Metal	41	1	1
Wood	40	0	1
Brick	41	1	2
All substrates	166	3	4

The principle issue addressed in this work was the following.

- *False positive and negative results*

3.1.7 RTI Large Field Study

RTI performed a large field study in the mid-1990s for EPA (Gutknecht et al., 1999). In this study, samples were analyzed using test kits, XRF, and ICP at 115 different primary test locations. These primary test locations were found in five buildings associated with a residential hospital and four buildings associated with an old school. Substrate materials encountered in these buildings included plaster, metal, concrete, fiberboard, and wood. At each primary test location, three measurements were made of the Pb in the paint with each of nine test kits:

- (1) Acc-U-Test,
- (2) EM Merck,
- (3) Frandon All-in-One (card),
- (4) Frandon All-in-One (solution),
- (5) Frandon Lead Alert (card),
- (6) Frandon Lead Alert (solution),
- (7) LeadCheck,
- (8) Lead Detective, and
- (9) Lead Zone.

The tests were performed by a number of different staff. In addition, three different samples were taken for ICP-AES analysis at each of these 115 locations. The responses of the qualitative test kits were rated as follows: no apparent color change (N), weak color change (P⁻), clearly positive response (P), and strong color formation (P⁺). These four ratings were assigned numeric values of 0, 0.1, 1.0, and 1.2, respectively. The average of the three numbers for each location was compared to the average ICP-AES values. Because of the lack of resources, a full analysis of these results, including statistical analyses, never was completed. However, simple visual comparisons were made. Figures 1 and 2 show the results for the LeadCheck in mg/cm² (Figure 1) and percent Pb (Figure 2). There are a large number of positive responses below the 1.0 mg/cm² level (in Figure 1) and the 0.5% level (in Figure 2). Figure 1 shows 1 of 81 results as false negatives and 35 of 81 results as false positives. Figure 2 shows 5 of 88 results as false negatives and 16 out of 88 results as false positives. The same trends were observed with the other rhodizonate-based kits and even more so with the sulfide-based Lead Detective kit.

The principle issue addressed in this work was the following.

- False positive and negative results

3.1.8 NIST Laboratory Study

A laboratory study was performed to determine the reliability of spot test kits using a large number of laboratory-prepared paint samples (Rossiter et al., 2000) For the study, four

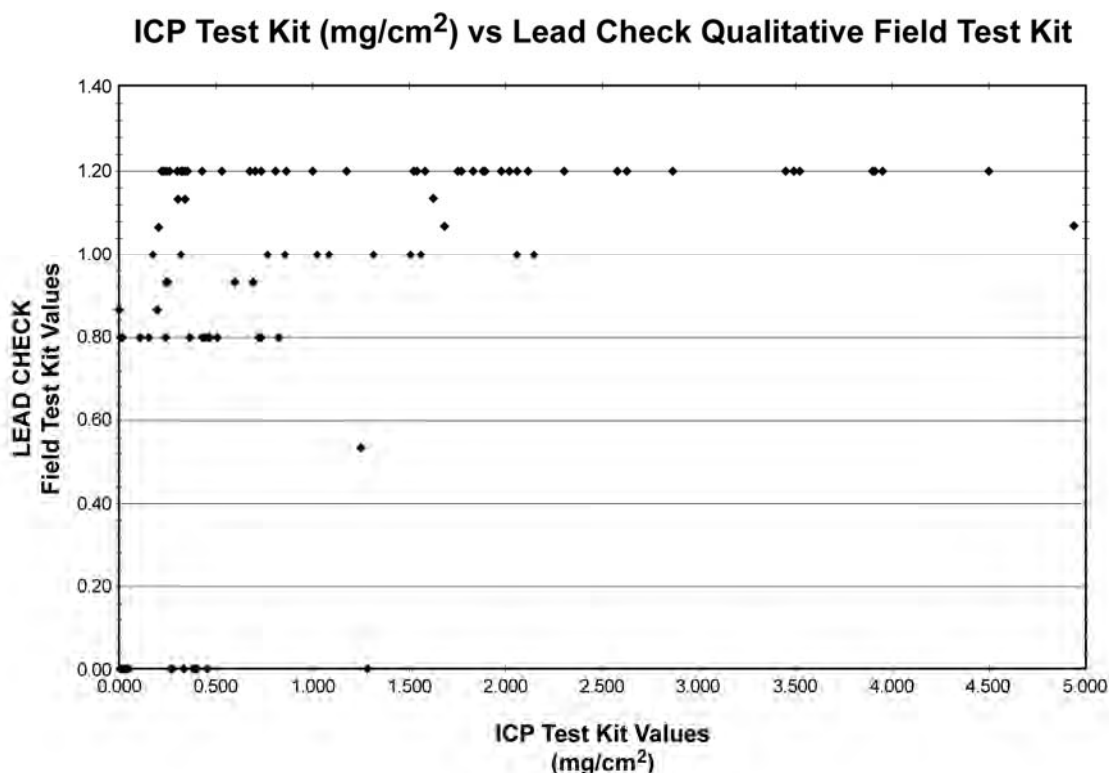


Figure 1. ICP results versus LeadCheck results (mg/cm²).

Note: Responses presented as numeric average of three results from each site rated as 0 - no apparent color change, +0.1 - a weak color change, +1.0 - a clearly positive response, and +1.2 - a strong color formation. All positive results at concentrations less than 1 mg Pb/cm² represent false positive results relative to the proposed Federal performance standards.

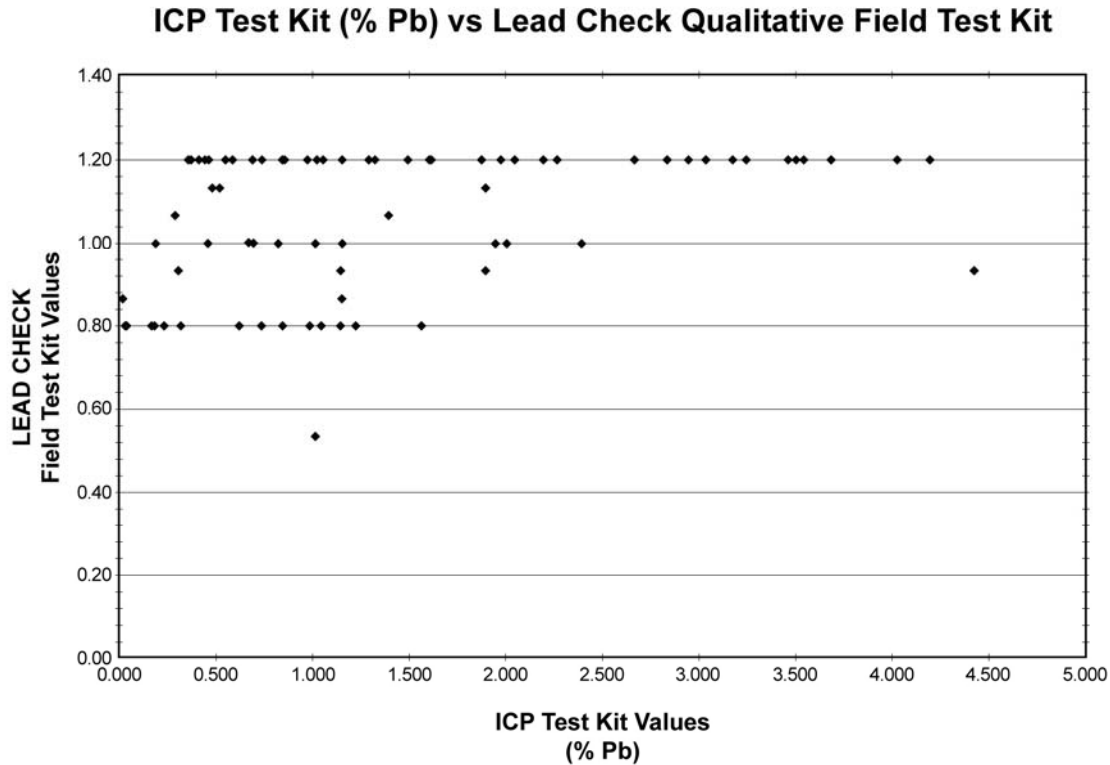


Figure 2. ICP results versus LeadCheck results (percent Pb).

Note: Responses presented as numeric average of three results from each site rated as 0 - no apparent color change, +0.1 - a weak color change, +1.0 - a clearly positive response, and +1.2 - a strong color formation. All positive results at concentrations less than 0.5% Pb represent false positive results relative to the proposed Federal performance standards.

rhodizonate-based kits ([1] Lead Zone, [2] LeadCheck, [3] Lead Alert Home Kit, and [4] Lead Alert Professional Kit) and four sulfide-based kits ([1] Acc-U-Test, [2] Heads Up, [3] Lead Detective, and [4] Sulfide Massachusetts [MA]) were used to investigate the effects of Pb level, Pb pigment type, operator, substrate, overlayer paint type, and overlayer paint thickness.

Three substrates were used for the test panels: (1) wood, (2) plaster, and (3) steel. For the purposes of experimental design, each substrate was categorized as either reactive (i.e., causing interference) or nonreactive to the test kit reagent. Wood was considered to be nonreactive for both rhodizonate and sulfide test kits. Plaster substrates may interfere with the development of the characteristic color for rhodizonate test kits and result in false negative responses (ASTM, 2004), so plaster is considered to be reactive for rhodizonate test kits. Metals present in steel substrates may react with sulfide test kits resulting in false positives (ASTM, 2004), so steel substrates were considered to be reactive for sulfide-based test kits. The experiments were designed so that rhodizonate reagents were applied to specimens having plaster and wood substrates, and sulfide reagents were applied to specimens with steel and wood substrates.

The leaded-paint films were prepared by spreading a Pb-spiked commercial paint on a smooth, nonporous surface, using a drawdown blade to provide films having uniform thickness. The leaded-paint film/substrate assemblies were overcoated with a thin or thick layer of latex or oil-based household paint to assess the impact of overlayer paint type and thickness.

The Pb levels for each test panel were determined quantitatively by a commercial laboratory accredited in the National Lead Laboratory Accreditation Program (NLLAP) using ICP spectrometry according to NLLAP protocols. Operators conducted the spot tests according to protocols written for each of the eight spot test kits. For each protocol, the basic steps for the spot test kit were taken from the manufacturer's instructions.

Several of the test kits were tested in different ways. For the LeadCheck test kit, Type 1 refers to the first swab examined within 2 min, Type 2 refers to the first swab reexamined after a period of time, and Type 3 refers to a second swab checked for up to 18 h. For the Lead Detective and Sulfide MA test kits, Type 1 refers to testing a notch, and Type 2 refers to testing a paint chip surface.

Table 15 summarizes the false negatives and false positives at the 1.0 mg/cm² Pb level for paints spiked with white lead and lead chromate on wood substrate. Regarding false negatives, the results varied considerably depending on the Pb pigment type. For white lead, the percent of false negatives was generally low (<4%), except for Acc-U-Test and Lead Alert Home Kit. In five

Table 15. False Negatives and False Positives at Pb Levels of Less Than and Greater Than 1.0 mg/cm²

Pb Pigment Type	Kit	Total No. of Observations	False Negatives		Total No. of Observations	False Positives	
			No.	%		No.	%
White lead	Acc-U-Test	40	5	13	200	81	41
	Heads Up	30	0	0	114	91	80
	Lead Zone	45	0	0	195	132	68
	LeadCheck – Type 1	45	1	2	195	124	64
	LeadCheck – Type 2	45	1	2	195	124	64
	LeadCheck – Type 3	45	1	2	195	125	64
	Lead Alert Home Kit	45	12	27	195	87	45
	Lead Alert Prof. Kit	45	0	0	195	88	45
	Lead Detective Type 1	40	1	3	200	97	49
	Lead Detective – Type 2	40	0	0	200	126	63
	MS Sulfide – Type 1	24	1	4	120	41	34
	MS Sulfide – Type 2	24	0	0	120	67	56
Lead chromate	Acc-U-Test	115	57	50	125	34	27
	Heads Up	69	29	42	75	41	55
	Lead Zone	125	75	60	115	18	16
	LeadCheck – Type 1	125	33	26	115	34	30
	LeadCheck – Type 2	125	25	20	115	36	31
	LeadCheck – Type 3	125	1	1	115	57	50
	Lead Alert Home Kit	125	100	80	115	12	10
	Lead Alert Prof. Kit	125	97	78	115	9	8
	Lead Detective – Type 1	115	6	5	125	64	51
	Lead Detective – Type 2	115	2	2	125	70	56
	MS Sulfide Type – 1	69	3	4	75	31	41
	MS Sulfide Type – 2	69	1	1	75	39	52

cases (Heads Up, Lead Zone, Lead Alert Professional Kit, Lead Detective – Type 2, and MA Sulfide – Type 2), no false negatives were observed. In contrast, for lead chromate, only LeadCheck – Type 3, Lead Detective – Types 1 and 2, and MA Sulfide – Types 1 and 2 had low percentages (<5%) of false negatives. Regarding false positives, the vast majority (about 85%) of the spot tests for both Pb pigment types showed percentages greater than 30%. That is, most spot test kits gave positive responses when the true value was less than 1.0 mg/cm².

The probabilities of positive response as a function of Pb concentration and other covariates were estimated using logistic regression models. Plots for the LeadCheck – Type 1 and MA Sulfide – Type 1 are shown in Figures 3 and 4. The filled circles represent the proportions of

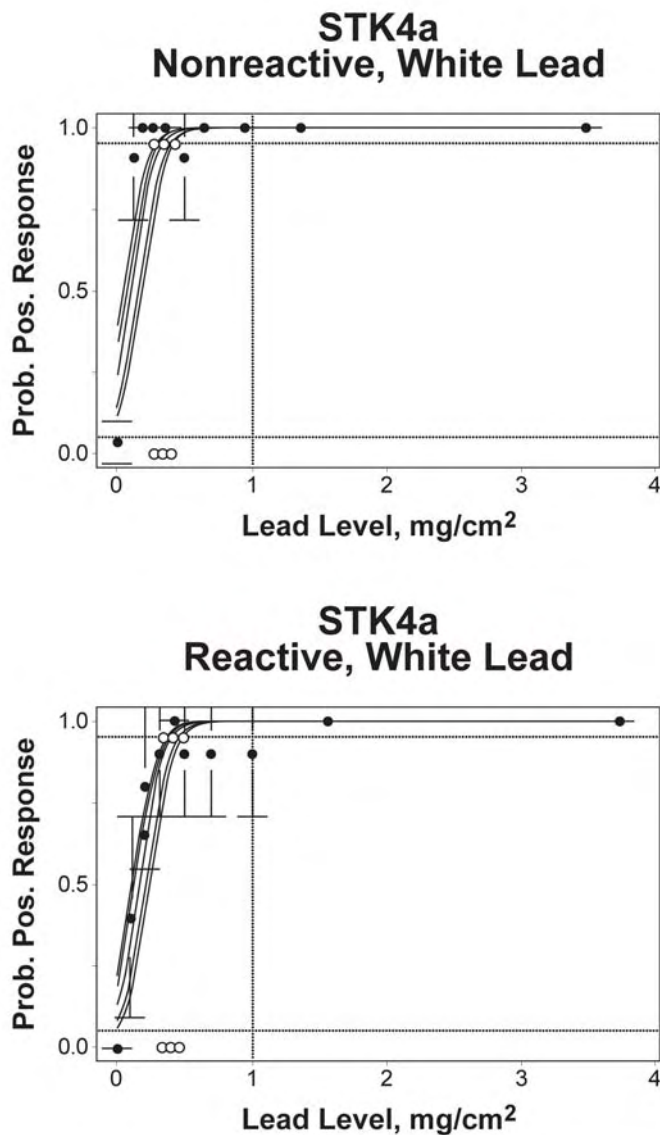


Figure 3. Probability of a positive response versus Pb level for LeadCheck – Type 1 (here presented as STK4a) on nonreactive (wood) and reactive (plaster) substrates (reproduced from Rossiter et al., 2000).

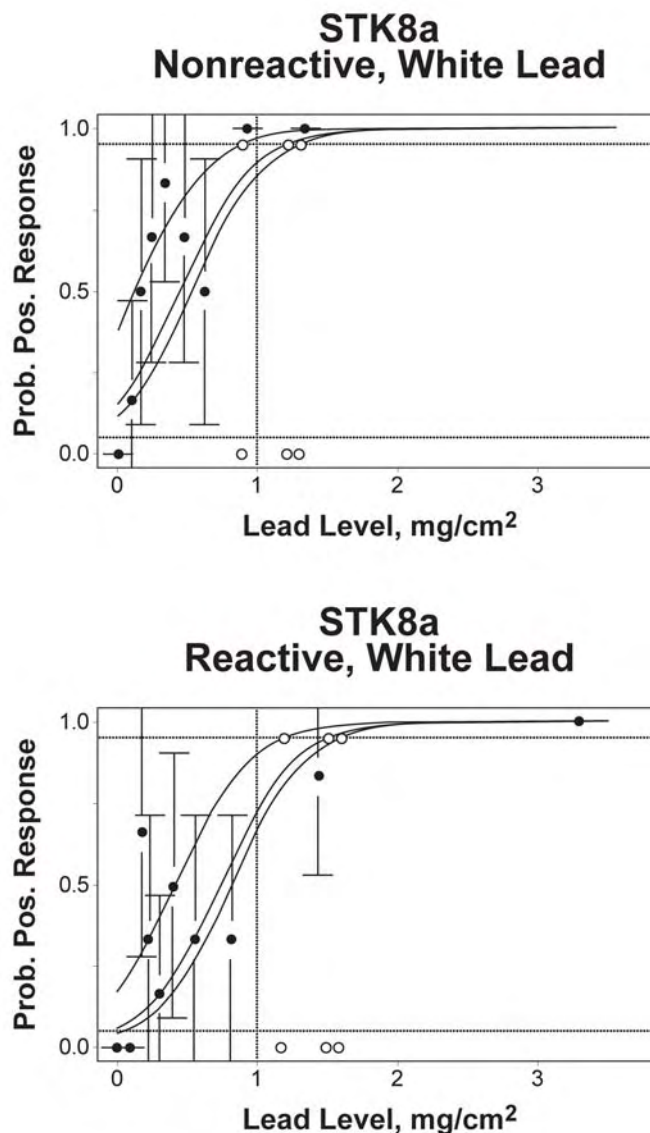


Figure 4. Probability of a positive response versus Pb level for MA Sulfide – Type 1 (here presented as STK8a) on nonreactive (wood) and reactive (steel) substrates (reproduced from Rossiter et al., 2000).

positive responses at a given Pb level, and the error bars are the associated 95% binomial confidence intervals. The two horizontal dashed lines represent the 0.5 and 0.95 probabilities of a positive response. Circles are the 0.95 probability point for each operator; they are repeated above the horizontal axis for clarity.

A separate model was fit for each kit, as well as for white lead and for lead chromate. The substrate effect was treated as a fixed effect in the regression model (except for Heads Up), and the operator effect was modeled as random. The results of these calculations are presented in Table 16.

Table 16. Pb Level Corresponding to a 95% Probability of a Positive Response for an “Average” Operator

Spot Test Kit	Pb Level (mg/cm ²)			
	White Lead		Lead Chromate	
	Reactive Substrate	Nonreactive Substrate	Reactive Substrate	Nonreactive Substrate
Acc-U-Test	1.9	2.3	6.2	8.4
Heads Up	—	0.4	—	89.8
Lead Zone	0.1	0.1	8.1	7.7
LeadCheck – Type 1	0.5	0.4	4.1	3.5
LeadCheck – Type 2	0.5	0.4	3.6	2.8
LeadCheck – Type 3	0.5	0.4	0.6	0.7
Lead Alert Home Kit	3.9	2.1	24.6	21.6
Lead Alert Prof. Kit	0.9	0.7	14.3	11.6
Lead Detective – Type 1	1.1	1.2	1.1	1.5
Lead Detective – Type 2	0.3	0.4	0.8	1.0
MS Sulfide – Type 1	1.9	1.6	1.3	1.5
MS Sulfide – Type 2	0.5	0.5	0.8	0.9

The issues addressed in this work included the following.

- False positive and negative results
- Interferences
- Need for training

3.1.9 Testing Using the HUD Archive

In a study led by QuanTech, four rhodizonate-based test kits and one sulfide-based test kit were applied to a set of the real-world painted building components from the HUD archive materials that are used for evaluating portable XRF instruments (Cox et al., 2001). The objectives were to (1) determine the extent to which manufactured paint films previously tested by NIST (Rossiter et al., 2000) are representative of field paint samples with respect to the evaluation of test kit performance, and (2) investigate the development of Performance Characteristic Sheets from the data collected at NIST using the manufactured paint samples. The real-world samples and manufactured samples had similar Pb levels. The same protocols were used for testing both the NIST and archive samples. The substrates were divided into reactive and nonreactive substrates based on the potential for chemical interaction between test kit reagents and the substrate. Wood was the nonreactive substrate for both types of kits. The reactive substrate was plaster for the rhodizonate kits and metal for the sulfide kit.

Testing of the archive samples was carried out by two of the five trained staff involved in the NIST testing. The results achieved with the white lead-based paint are shown in Table 17.

The number of false negatives at or above 1.0 mg/cm² is small, averaging about 6%; as noted, three out of five of the kits gave no false negatives. The number of false positives below 1.0 mg/cm² is large, averaging close to 50%. Even at close to 0 mg/cm², false positives occur.

Table 17. Results of HUD Archive Testing on White-Lead-Based Paint

Response	Low Pb Level > 0.01 mg/cm ²	Low Pb Level < 0.01 mg/cm ²	Pb Level ≥ 1.0 mg/cm ²	Pb Level < 1.0 mg/cm ²
False negatives	1.7% to 35.6% av 17% ± 14%	—	0% to 26.7% av 6% ± 12% ^a	—
False positives	—	0% to 11.7% av 5.3% ± 4.3%	—	44.6% to 67.7% av 60% ± 11%

^aThree out of five kits gave 0.0% (no false negatives).

As noted earlier, a test kit that meets the proposed Federal performance standards must have a high probability of obtaining a positive result when the Pb level is at or above 1.0 mg/cm² and a high probability of obtaining a negative result when the Pb level is below 1.0 mg/cm². As stated by the authors, “None of the test kits in this research have this capability as shown in the response curves.”

As to the goals of this study, the test kit evaluation system using manufactured samples was found to be acceptable for predicting performance of test kits on nonreactive substrates; that is, the manufactured samples appeared to be representative of the archive samples in that the response curves for the archive samples fell between the response curves for white lead-based and lead chromate-based paints (which were taken as most soluble and least soluble common Pb pigments, respectively). For reactive substrates, the response curves generated with the archive samples were generally outside of the two curves for the white lead-based and lead chromate-based paints, indicating significant differences between the archive and manufactured samples.

The principle issues addressed in this work were the following.

- False positive and negative results
- Need for training

3.1.10 Reports from Jim Weydt of Acc-U-Test Test Kit

Jim Weydt of Acc-U-Test, Weymouth, MA, provided a summary of a number of reports of work with the sulfide-based kits (Weydt, 2006). In one report (Vind et al., 1978), tests were made of the sensitivity of the sodium sulfide reagents to various Pb compounds. In that work, Vind and colleagues report that the minimum concentration of Pb that could be detected in light-colored paints was approximately 0.5%, although the basis of the relationship between the test compounds and paint concentrations is not clear. In a different report in Consumer Reports in July 1995, the sensitivity of the sulfide-based Acc-U-Test is given as 0.05%, which is more consistent with the sensitivities reported by others, such as Rossiter et al. (2000). In other work, Weydt (1993) reported that 16 metal pigments considered to be potential interferents with the sulfide-based test kit were reacted with 6% to 8% sodium sulfide test solution. These metal pigments were antimony, bismuth, cadmium, chromium, cobalt, copper, iron, manganese, magnesium, mercury, molybdenum, nickel, silver, tin, titanium, and zinc. All but one of these metals are used as fire retardants, biocides, fungicides, and drying agents in paints for industrial, commercial, and marine use. The possibility of these materials causing false positive test results in paints intended for residential use is very unlikely. If present, the levels are expected to be so low as to not interfere with an in situ sulfide-based test.

The principle issues addressed in this work were the following.

- False positive and negative results
- Interferences

3.1.11 Test Kit Based on DNAzyme Nanoparticles

Lu (2005) describes a new Pb test kit based on the reaction of Pb with a component of an enzyme. Studies have shown that DNA has enzymatic properties. A component of the DNA is termed a DNAzyme, which consists of a substrate strand and an enzyme strand. The DNAzyme is highly selective towards Pb(II) as a cofactor over other metal ions to carry out hydrolytic cleavage of the substrate strand. In the presence of Pb(II) ion, the enzyme cleaves its substrate into two pieces. The strand is extended on both ends, with the extended fragment complementary to DNA attached to gold nanoparticles. As a result, gold nanoparticles can be assembled by the DNAzyme to form blue aggregates. In the presence of Pb, the substrate is cleaved and the assembly of nanoparticles is inhibited, which results in a red color. Then, the ratio of spectrophotometric absorptions at 522 nm and 700 nm (which correspond to blue and red) can be used to determine Pb(II) ion concentration.

Synthetic samples with varying concentrations and latex overlays were prepared for the study. Results reported are shown in Figure 5. The change from essentially all negative responses to essentially all positive responses occurs over the range of about 1 mg/cm² to about 2 mg/cm². This compares to a range of about 0 mg/cm² to 0.5 mg/cm² for LeadCheck (see Figure 3) and a range of about 0 mg/cm² to 1.5 mg/cm² for MA Sulfide (see Figure 4).

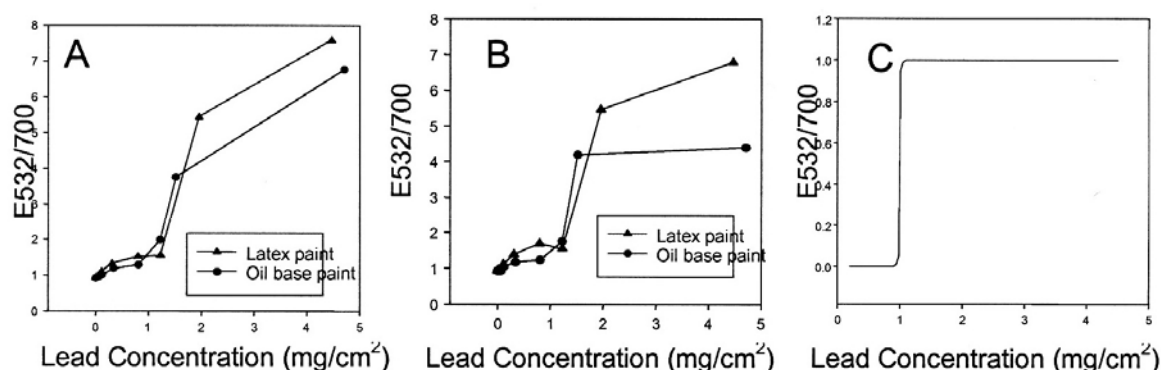


Figure 5. Response of DNAzyme-based test kit using (A) benchtop and (B) portable colorimeters; (C) ideal sensor defined by Rossiter et al. (2000) (reproduced from Lu, 2005).

Additionally, the tests of the method were performed with paint samples made in the laboratory by mixing commercially available white latex or white oil-based paints with Pb (II) carbonate, basic powder. 10% acetic acid and soaking and ultrasonication for about 40 min yielded quantitative recovery with diced paint samples.

The principle issue addressed in this work was the following.

- False positive and negative results

3.1.12 Immunoassay-Based Test Kits

Pb test kits may be based on the use of an immunoassay. This is a biochemical test that measures the level of a substance using the reaction of an antibody or antibodies to its antigen (in this case, Pb). The assay takes advantage of the specific binding of the antibody to its antigen. Monoclonal antibodies often are used, as they usually bind to only one site of a particular molecule and, therefore, provide a more specific and accurate test. The antibodies picked must have a high affinity for the antigen (if there is antigen available, a very high

proportion of it must bind to the antibody). In a competitive immunoassay, the antigen in the unknown sample competes with labeled antigen (e.g., Pb ion bound in a fluorescent complex) to bind with the antibodies. The amount of antigen that transfers from the labeled state to being bound to the antibody site is then measured by a change in the label's color, fluorescence, etc.

The PurTest Immunoassay Lead test kit is used for Pb in water (www.silverlakeresearch.com). In less than 10 min, the test can be used to detect Pb in water at or below the EPA action level of 15 ppb.

The principle issue addressed in this work was the following.

- *False positive and negative results*

3.1.13 Summary for Qualitative Test Kits

In summary, a number of qualitative test kits have been developed and studied. The majority of these are based on the rhodizonate or sulfide reactions with Pb. The kits generally demonstrate a level of false negatives that meets the proposed Federal performance standard of less than 10%, along with a high level of false positives, as high as about 70%. The high level of sensitivity found with these kits seems appropriate if the manufacturer is concerned that the users have a tool that is conservative and reveals very low levels of Pb because there is no way to determine how much of the actual Pb present will be extracted to react with the kit reagents.

3.2 Semi-quantitative and Quantitative Test Kits

3.2.1 Semi-quantitative Test Kits

Semi-quantitative test kits are those that give an approximate value for the Pb level. They typically work by visually comparing the intensity of the color formed with some standard chart of colors or through a color comparator. To get some estimate of the concentration, the sample must be removed from the substrate and homogenized to some degree and subjected to extraction to get a total sample value. One such kit is the Merck EM Quant, which uses rhodizonic acid in an acidic solution to form a red complex with the Pb ion. The test procedure is as follows.

- Add three drops of reagent (acetic acid) to the sample solution and swirl cautiously.
- Immerse the test strip in the solution for 1 s so that the reaction zone is thoroughly moistened. Remove excess liquid by stroking the edge of the strip against the rim of the vessel.
- Wait 2 min and compare the reaction zone with the color scale on the package.

The scale on the package is graduated in different concentrations: 0, 20, 40, 100, 200, and 500 mg Pb²⁺/L (ppm). The pH of the solution being tested should be between 2 and 5. This is normally the case once the reagents have been added. If the quantity of reagent specified in the directions is not sufficient to adjust the solution to this pH (which can be checked with a pH indicator strip), strongly acidic solutions must be buffered prior to the determination with 1 M sodium hydroxide solution, and alkaline solutions with 1 M nitric acid.

In the study by Luk et al. (1993b) the Merck EM Quant was used to test for Pb on the surface of laboratory-prepared paints. Method A for this kit calls for wetting the surface and then contacting the surface with the test strip after the reaction has gone on for 1 min; Method B calls for wetting the strip and then contacting the strip with the paint surface. For paints with 0.1 and 0.6 mg/cm², Method A values for three samples of each paint were all 0 ppm. At 1.9 mg/cm², the three readings were all 10 ppm. For paint at 2.6 mg/cm², the three readings were 40, 100, and 200 ppm. Again, it must be noted that this semi-quantitative mode is intended for solubilized Pb (i.e., Pb ion). No study has been identified that evaluated the Merck EM Quant with solubilized paint.

Other test kits intended for the measurement of Pb in water using visual comparison are available and could be used for testing solubilized Pb, including PurTest Lead and Lead Inspector Lead Test Kit.

3.2.2 Quantitative Test Kits—Laboratory Study

The quantitative test kits usually involve an instrumental measurement of the product of reaction with Pb. This may be measurement of the colored product of Pb with rhodizonate or some other reagent or measurement of some other unique property of Pb. A number of ASTM methods for quantitative measurement of Pb in paint that are or could be applied in the field have been developed and are available, including

- E1729-05 Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination;
- E1979-04 Standard Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead;
- E2051-01 Standard Practice for the Determination of Lead in Paint, Settled Dust, Soil, and Air Particulate by Field-Portable Electroanalysis; and
- D3559-96 Standard Test Methods for Lead in Water (using ASV).

Three different quantitative kits have been studied by RTI. These are based on color intensity measured by reflectance, color intensity measured with a simple colorimeter, and electrochemical oxidation of Pb that has been concentrated on an electrode through a period of reduction or plating out of the sample extract (ASV).

In a laboratory study (Williams et al., 1996), the EM Science Reflectoquant (based on reflectance) and the Pace Environs PaceScan 2000 (based on ASV) were evaluated with a series of paint reference materials that were either from the Environmental Lead Proficiency Analytical Testing (ELPAT) program or other paints that were ground to a powder and verified by multilaboratory analysis. Pb concentrations ranged from 222 µg/g to 118,700 µg/g. Materials were extracted according to the manufacturer's recommendations. Pace Environs, Inc., prescribed the RTI Ultrasonic/Acid Extraction Method (Luk et al., 1993a) to extract paint, dust, and soil samples for analysis by the PaceScan 2000. However, EM Science did not prescribe an extraction method for the Reflectoquant, so RTI modified its original ultrasonic/acid extraction method (Luk et al., 1993b) to address sensitivity requirements for the Reflectoquant.

EM Science Reflectoquant. With this instrument, the Pb-containing samples (detection limit goals determine appropriate sample weight) were digested (or extracted) in a nitric acid solution and diluted to 50 mL, and 10-mL aliquots were removed. The aliquots were buffered into a pH range of 3 to 4 (previously determined to optimize the formation and stability of the lead rhodizonate complex [Luk et al., 1993b]). The test strips were removed from the vial and immersed in the buffered solutions (10 mL of extract plus volume of buffer) for approximately 2 s, then placed in the optics of the reflectometer. The measurement requires a total of 2 min (immersion and optical measurement). At the end of the 2-min interval, the Pb concentration in the solution (µg Pb/mL) is displayed on the Reflectometer.

The Lead Test instructions enclosed with each Reflectoquant kit of test strips, bar code, and Lead Test reagent indicated that the instrument measured Pb in the range of 20 to 200 mg Pb/L (equivalent to 20 to 200 µg Pb/mL or 20 to 200 ppm). The instrumental detection range of 20 to 200 µg Pb/mL correlates to a method detection range of 10,000 to 100,000 µg Pb/g paint (for a 100-mg sample extracted using the RTI ultrasonic/acid extraction method [Luk et al., 1993b]).

The maximum recovery of Pb with the test samples (151%) was observed for ELPAT Sample 5P1 extracted and buffered with NH₄OH/NH₄OAc. The measured means for three of the six samples were statistically different from the expected mean concentration. When ELPAT

performance criteria were applied to the ELPAT samples analyzed, three out of five measured mean concentrations were found to be in the ELPAT acceptance range (mean $\pm 3\sigma$). Results are summarized in Table 18.

Table 18. Summary of Results for the Analysis of RTI Core Paint Materials Using the EM Science Reflectoquant

Analytical Result	Buffer System	
	NaOH/NaOAc	NH ₄ OH/NH ₄ OAc
Sample weight extracted	500 mg	500 mg
Method detection limit (MDL) (calculated, n=7)	7.1 µg Pb/mL (900 µg Pb/g)	10.2 µg Pb/mL (1300 µg Pb/g)
Method quantitation limit (MQL) (calculated, n=7)	24 µg Pb/mL (3000 µg Pb/g)	34.0 µg Pb/mL (4320 µg Pb/g)
Range of recoveries (all samples)	63% ^a to 132% ^b (n=18)	80.1% to 151% ^b (n=17)
Mean recovery	98.5 \pm 23.9% (n=18)	114 \pm 22.0% (n=17)
Range of reproducibility	1.3 to 16% (1 σ , n=6)	1.0% to 22% (1 σ , n=6)
Pooled relative standard deviations (RSDs)	8.2% (n=6)	11.8% (n=6)
Equivalency of measured mean and expected concentration at the 95% confidence level	Measured means for three out of six samples were statistically different from the expected concentration at the 95% confidence level.	Measured means for three out of six samples were statistically different from the expected concentration at the 95% confidence level.
Comparison of measured mean with ELPAT performance range (ELPAT samples only)	Three out of five samples ^c were acceptable.	Three out of five samples ^c were acceptable.
pH criteria	pH=4, particularly for samples with low levels of Pb	Three to four
Cost of analyses	Cost of instrument: \$495.00 (in 1995 dollars) Materials: \$1.11/test strip + ultrasonicator + reagents	

^aAt low end of linear operating range

^bAt high end of linear operating range

^cELPAT samples only

The Pb concentration of each extract buffered with the NaOH/NaOAc system was verified by ICP analysis to evaluate the extraction efficiency of the modification of the RTI ultrasonic/acid extraction method (Luk et al., 1993b). A linear regression of the measured concentration versus the RTI ICP concentration was generated to eliminate variables from the extraction method and to allow only the measurement methods to be compared. The linear regression curve, provided in Figure 6, shows a correlation coefficient of 0.986 and a regression $y = 1.16x - 3510$. The slope of the curve suggests the importance of verifying calibration before samples are analyzed with this instrument.

EnviroScan PaceScan 2000. With this instrument, 5-mL aliquots of the extract were taken from the extraction tube, the extracts were buffered with the packaged PacePrep tablet (used to adjust pH and add an electrolyte for stabilizing the ionic strength of the extract), and analyzed

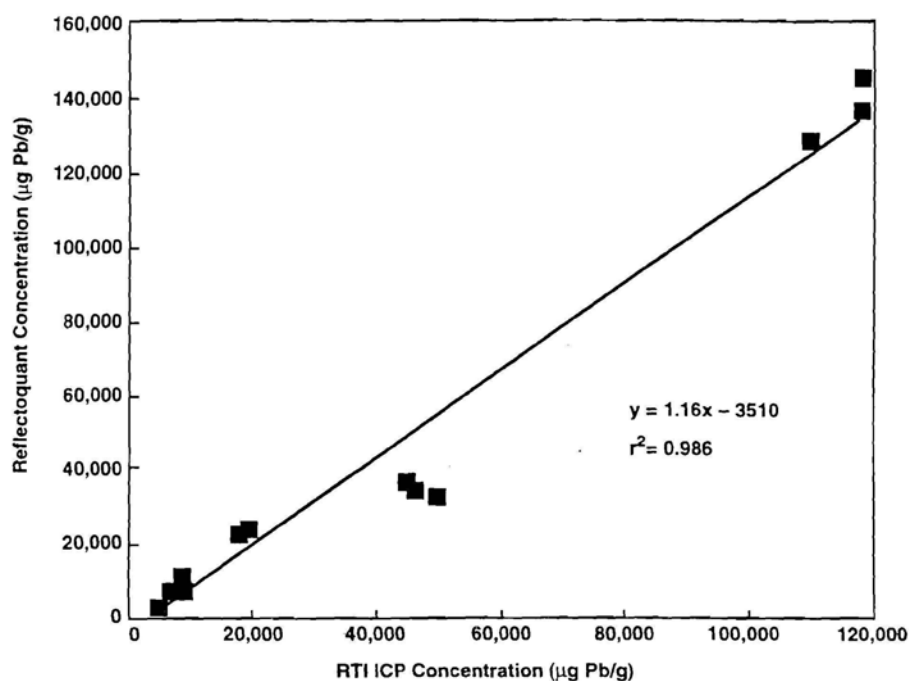


Figure 6. Plot of the Pb concentration (µg Pb/g) measured by the EM Science Reflectoquant versus ICP-AES for RTI paint materials.

using the anodic stripping analysis instrument. A new, separate electrode is used for each analysis.

The PaceScan offers two ranges, a low range (LR), and a high range (HR), for the measurement of Pb in paint.

LR: 0.025 to 1.5 mg Pb/sample (50 mL extract)
0.050 to 3.0 mg Pb/cm²

HR: 0.1 to 5.0 mg Pb/sample (50 mL extract)
0.2 to 10.0 mg/cm²

Measured mean concentrations of the ELPAT samples were compared to the ELPAT range of acceptable performance, mean \pm 3 σ (Esche et al., 1995). An MDL (254 µg Pb/g paint) and an MQL (848 µg Pb/g paint) were calculated from the standard deviation of seven analyses of the 100-mg paint extract with the lowest Pb concentration (MEM Low Paint, measured concentration 1420 µg Pb/g). The MDL is consistent with the instrument's low-range operating specification, where 0.025 mg/sample equates to 250 µg Pb/g paint for a 0.100-g aliquot extracted using the RTI ultrasonic/acid extraction method (Luk et al., 1993b). Of the 20 samples measured, only three showed relative recoveries less than 90%. The high recovery of Pb (131%) for ELPAT Sample 5P2 (expected concentration 222 µg Pb/g paint) suggests a positive bias at lower Pb levels. Results for the analysis of paint are summarized in Table 19.

To compare analytical performance only, a linear regression of the measured mean concentration versus the RTI ICP concentration was generated; the regression plot is provided as Figure 7. The regression equation, $y = 0.900x - 103$, $r^2 = 0.9996$, suggests a good correlation of PaceScan data to ICP analysis.

Table 19. Summarized Results for the Measurement of RTI Core Paint Materials Using the PaceScan 2000

Performance Parameter	Measurement Range	
	Low Range (0.025 to 1.5 mg Pb/sample)	High Range (0.1 to 5.0 mg Pb/sample)
Sample weight extracted	100 mg	100 mg
MDL	0.0254 mg Pb/sample 254 µg Pb/g	Not determined
MQL	0.0848 mg Pb/sample 848 µg Pb/g	Not determined
Mean recovery	88.1± 6.4% (n=18)	89.0% ± 17.1% (n=21)
Range of individual recoveries	76.1% ^a to 99.9%	50% ^b to 110%
Range of RSDs	3.2% to 10.8% (n=6)	0% to 10% (n=7)
Pooled RSD	6.1% (n=6)	5.6% (n=7)
Equivalency of measured mean and expected mean concentration at 95% confidence level	Measured means for three out of six samples were statistically different from the expected mean concentration at the 95% confidence level.	Measured mean for one out of seven samples was statistically different from the expected mean concentration at the 95% confidence level; 2 samples were indeterminate (divided by 0) at the 95% confidence level.
Comparison of mean result to ELPAT acceptable performance range (mean ± 3σ)	The measured means for six out of six samples are acceptable according to the ELPAT Performance Range.	Not determined

^aMeasurement is 1.468 mg Pb/sample, near the upper end of the low range.

^bMeasurement is 0.1 mg Pb/sample, at the low end of the high range.

3.2.3 Quantitative Test Kits—Field Studies

The pilot field study described in Section 3.1.5 included evaluation of a colorimetric test kit (Gutknecht et al., 1997). The procedure used in this study was developed by Luk et al. (1993a).

The method involves weighing 0.1 to 0.25 of paint, soil, or dust into a 50-mL plastic centrifuge tube. Then 15 mL of 25% (v/v) nitric acid is added and the sample in the tube is placed in an ultrasonic bath for 30 min. Following dilution to a fixed volume, thorough mixing, and separation of solids by centrifugation, filtration, or decanting, an aliquot of the resulting extract is pH-adjusted and further diluted in two successive steps. The sample solution is then analyzed for Pb using the Hach colorimetric kit for Pb (Hach). First a Hach reagent is added. The resulting solution is then passed through an ion exchange column provided in the Hach kit to collect and concentrate the Pb. The Pb is flushed from the column with an eluent provided in the kit.

A complexing agent then is added to the eluted Pb solution, and the resulting colored complex is measured using a small portable colorimeter, provided in the test kit. Results from the laboratory evaluation of the method generally showed a recovery of greater than 90% with standard samples and an RSD of less than 10%.

Samples for this field evaluation of the Hach method were taken from the area used for XRF testing and also the qualitative test kit area. The accuracy of the colorimetric test kit was estimated by comparison of the MW/ICP values for areas with the test kit results for these

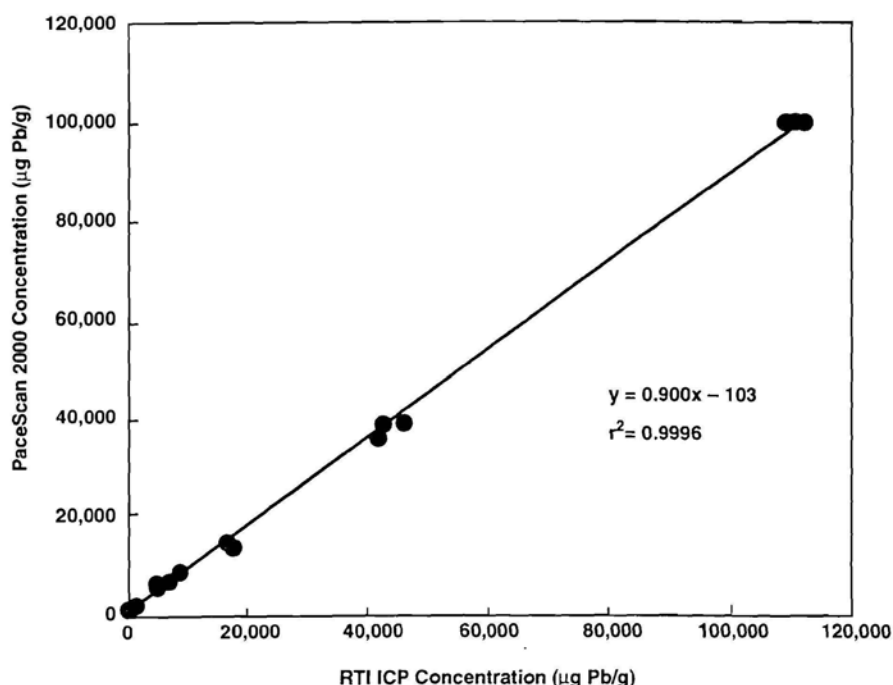


Figure 7. Plot of the Pb concentration (µg Pb/g) measured by the PaceScan 2000 versus ICP-AES for RTI paint materials.

areas. The average mass recoveries (100 plus percent difference) for the wood and fiberboard components were $97\% \pm 17\%$ and $90\% \pm 14\%$, respectively. These recovery values of $\geq 90\%$ are consistent with performance reported by Luk et al. (1993b). Comparison of MW/ICP values determined from the test kit locations is more tenuous because of the variability of Pb in the paint from location to location. Nevertheless, average mass recoveries (100 plus percent difference) for the wood and fiberboard components were $99\% \pm 15\%$ and $92\% \pm 10\%$, respectively. However, the range of recoveries is relatively large, as presented in Table 20.

Table 20. Range of Recovery Values for the Colorimetric Test Kit Test

Sample	Mass Concentration	Area Concentration
Paint on wood, area B	71% to 118%	71% to 119%
Paint on wood, test kit locations	78% to 127%	67% to 134%
Paint on fiberboard, area B	65% to 107%	66% to 103%
Paint on fiberboard, test kit locations	77% to 109%	68% to 121%

The range of concentrations was very limited, as reported above. This, in conjunction with large variability in the results, made any regression analysis meaningless.

The precision was estimated from the replicate analyses in the test kit locations, because it is reasonable to assume that variation in this 2 x 12-in limited area would be minimal. The pooled standard deviations for the analyses of three different samples in each of the different test kit

locations are presented in Table 21, along with these same values for the MW/ICP analyses. As noted, the pooled RSD for the quantitative field kits is about 15% for mass concentration.

Table 21. Pooled Standard Deviation for Triplicate Analyses Performed within Each Test Kit Location

Analysis Performed	Mass Concentration, % (pooled RSD)	Area Concentration, mg/cm² (pooled RSD)
MW/ICP, mean of all sample areas (A-D3), wood	0.63 (15)	0.47 (22)
Colorimetric field kit, wood, test kit locations	0.58 (14)	0.42 (19)
MW/ICP, mean of all sample areas (A-D3), fiberboard	0.22 (14)	0.29 (19)
Colorimetric field kit, fiberboard, test kit locations	0.21 (14)	0.18 (15)

The study described in Section 3.1.5 included evaluation of three quantitative test kits, the Reflectoquant; the PaceScan 2000; and a third kit, the Hach Colorimeter. It should be noted that other field colorimeters are available for measurement of Pb in water, such as the Orbeco-Hellige colorimeter (Orbeco Analytical Systems, Farmingdale, NY), which claims a range of 1 to 150 µg Pb/L.

The Hach DR 100 analog colorimeter is precalibrated over the range of 0 to 150 µg/L. A three-point calibration is recommended for this instrument. In a standard operating procedure (SOP) developed by Luk et al. (1993a), the paint samples are to be digested/extracted in 25% nitric acid in a small, field portable ultrasonic bath. This SOP gives a measure of the bias expected with well-characterized homogenized real-world paint and NIST SRMs. Values reported are shown in Table 22.

Table 22. Accuracy and Precision Values for Real-World and NIST SRM Samples

Sample	Referee Value, % Pb (n)	Mean Kit Value % Pb (n)	Accuracy as % Bias	% RSD
Paint 1	0.162 ± 0.004 (3)	0.181 ± 0.014 (15)	11.7	7.7
Paint 2	0.646 ± 0.023 (3)	0.624 ± 0.023 (15)	-3.4	3.7
Paint 3	3.60 ± 0.03 (3)	3.25 ± 0.20 (15)	-9.7	6.2
SRM 1579	11.87 ± 0.04	10.8 ± 0.8 (25)	-9.0	7.4

The large field study described in Section 3.1.7 involved taking samples from the same location for ICP analysis, spot tests, and quantitative test kits. As noted previously, some 115 locations in an old hospital and an old school that included plaster, wood, metal, and concrete were tested. The data for the Reflectoquant, PaceScan, and Hach kits were not fully statistically analyzed because of the lack of resources. Mathematical modeling based on paired values should be performed for proper comparison of the kits with the ICP values. Currently, only simple regression analysis has been performed. These results are presented in Table 23.

One can see that the agreement of the ICP values with the test kits values is highly variable. One source of the variability is the source of the samples; the slope for the PaceScan for wood is 0.63 (see Table 23) for all samples tested, but 1.00 if one looks only at samples from the old hospital and excludes samples from the old school.

Table 23. Results of Regression Analysis Comparing ICP Results with Quantitative Test Kits

Substrate (n)	Reflectoquant Reflectometer		PaceScan ASV		Hach Colorimetric	
	Slope	R ²	Slope	R ²	Slope	R ²
Wood (59)	2.15 ± 0.26	0.56	0.63 ± 0.065	0.62	2.16 ± 0.25	0.56
Plaster (19)	0.58 ± 0.43	0.096	1.25 ± 0.11	0.89	0.41 ± 0.40	0.057
Concrete (10)	0.85 ± 0.39	0.37	0.77 ± 0.30	0.46	0.85 ± 0.39	0.38
Metal (19)	1.01 ± 0.12	0.80	0.62 ± 0.12	0.63	1.12 ± 0.10	0.87

Ashley et al. (1998) also included the PaceScan ASV system in a field study. Expected values were determined by AAS analysis of samples returned to the laboratory. The results were reasonably well correlated, with $R^2 = 0.81$; the slope was 0.868. This study explained outliers as possibly resulting from the samples not being grinded adequately in the field. Ashley and co-workers also performed regression analysis; their results are presented in Table 24.

Table 24. Results of Regression Analysis Comparing AAS Results with PaceScan Kit Results

Substrate Type (n)	Slope	Intercept (%)	R ²
Wood (39)	0.894	0.055	0.731
Plaster (44)	1.28	-0.018	0.821
Brick (40)	1.09	-0.027	0.965
Metal (42)	0.762	0.057	0.872
All (165)	0.868	0.072	0.814

The slopes and correlation coefficients are similar for the two studies for the PaceScan kit. Differences could arise from numerous sources, including the nature of the paints collected, the age of the paints, and the difference in extent of homogenizing and extracting the paints. The differences also could arise from the variability of the paint across the sampling location. It is well known that real-world paint samples, even from the same wall or door frame, show considerable point-to-point variability in Pb concentration. This makes sense if one considers the mechanics of painting. At the start of application, the brush is full and the layer may be thick, but as the paint continues to flow from the brush onto the surface, the layer may become thinner. If one multiplies this possibility over several coats of paint, one can see that there is a strong likelihood of great variability in the point-to-point Pb concentration.

Finally Sussell and Ashley (Sussell and Ashley, 2002) performed onsite measurement of Pb in paint chip samples by ultrasonic extraction and ASV in the field during renovation and remodeling activities. Pb in sample extracts subsequently was determined by AAS in a fixed-site laboratory. The remaining sample extracts plus undissolved material were then subjected to hot plate digestion in concentrated nitric acid-30% hydrogen peroxide prior to AAS analysis for Pb. Pb measurements by AAS were compared to Pb determinations by hot plate digestion-AAS; these data were highly correlated and demonstrated not-significant bias, thereby showing that the ultrasonic extraction procedure is equivalent to the hot plate procedure. Field ASV data were compared to AAS and results from the fixed-site laboratory Pb measurements. A significant negative bias associated with the ASV measurement was observed and attributed to a matrix effect, such as a chemical interference (Rossiter et al., 2001). Additional leaching of Pb from the

paint sample matrix after ultrasonic extraction in the field (i.e., during transport and storage) is another possible confounder that could result in the observed negative bias in onsite measurements.

3.2.4 Immunoassay-Based Test Kits

An area of ongoing development is the use of immunoassay for quantitative measurement of Pb. In one study, Pb concentrations were determined by a fluorescence polarization of immunoassay method that used polyclonal antibodies raised against the Pb (II) chelate of ethylenediaminetetraacetic acid (EDTA) (Johnson et al., 2002). The technique is based on competition for a fixed concentration of antibody binding sites between Pb-EDTA, formed by treating the sample with excess EDTA, and a fixed concentration of a fluorescent analogue of the Pb-EDTA complex. The limit of detection was approximately 1 ppb and cross reactivity with 15 nontarget metals was below 0.5% in all cases. The authors claim the methods are simple and amenable to field testing, allowing “timely and cost effective characterization of suspected sources of Pb.”

4.0 AVAILABILITY OF PERFORMANCE EVALUATION MATERIALS

ASTM E 1828, “Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Spot Test Kits for Lead in Paint” (ASTM, 2001b) sets forth requirements and provides guidance for the preparation of standard synthetic dry paint films. The ASTM standard addresses leaded paint components, substrate, overlayers, thickness, concentration ranges, and number of samples. These reference paint films are intended for method evaluation. The approach would be to prepare a large number of paint films that are made up of different types of Pb pigments, different concentrations of Pb, and different structure (thicknesses and overlayers) and to use the kits under consideration to test all these samples. A select subset of these materials could be used as quality control (QC) materials for the kits.

Current test kit manufacturers do not use or recommend using performance evaluation materials. Among test kit manufacturers, the accepted practice is to include a QC card (loaded with a known amount of Pb) or, in the case of one supplier, an actual paint chip that will provide a positive kit response. Inclusion of QC samples with the kits would be very beneficial, both to assure the user that the kit is being used properly and that the kit has the expected sensitivity.

The only current sources of performance evaluation materials for evaluating test kits are QuanTech and NIST. QuanTech provides leaded film standards with certified Pb levels from <0.06 thru 3.51 mg/cm² prepared using a single layer of old formulation white lead paint mounted on a nylon substrate support overlaid with 10 layers of nonleaded paint to produce a 2.9 x 2.5-in rectangular coupon. NIST provides Pb paint films ranging in concentration from blank to a high level Pb of 5.59 mg/cm² (Table 25). The NIST SRM paint films are intended for XRF analysis and are coated with a laminate layer that would have to be removed for use with test kits. Recently (in 2006), Dr. Kim Rogers of EPA developed a protocol for the removal of the laminate from NIST SRM paint films and reported on testing the protocol with the 1.0 mg/cm² standard (Harper, 2006).

NIST also has available RM 8680, Pb in paint on fiberboard, available in a 10.2 x 15.2 x 1.3-cm section of painted fiberboard, whose concentration is individually value assigned from 1 to 2 mg/cm².

For those test kits involving extraction of the Pb in paint followed by colorimetric, reflectance, or electrochemical determination, numerous Pb-in-paint reference materials are available. NIST produces several powdered Pb-in-paint SRMs, and RTI has accumulated 55 rounds of ground

Table 25. NIST SRMs for Leaded Paint Films

SRM	Description	Pb Concentration (mg/cm²)
2570	Pb paint film, blank	<0.001
2571	Pb paint film, nominal 3.5 mg/cm ²	3.58
2572	Pb paint film, nominal 1.6 mg/cm ²	1.527
2573	Pb paint film, nominal 1.0 mg/cm ²	1.040
2574	Pb paint film, nominal 0.7 mg/cm ²	0.714
2575	Pb paint film, nominal 0.3 mg/cm ²	0.307
2576	Pb paint film, high level	5.59

Pb-in-paint ELPAT materials. In coordination with this work assignment, RTI is preparing intact paint films using ELPAT materials for Pb, which, if successful, would more closely mimic real world conditions for test kit evaluation.

In addition, there have been studies reported in the literature that involved both evaluating spot test kits and the preparation of paint standards. Two reports used manufactured test samples of leaded paint and paint films for test kit evaluations (Rossiter et al., 2000; Cox et al., 2001). Cox and co-workers describe in detail the steps involved in the preparation of the manufactured performance evaluation samples. Lu (2005) describes a method for extracting Pb from paint and quantifying the amount of Pb present using a colorimetric Pb biosensor. For this study, Pb paint samples of different concentrations were prepared, with the preparation described.

5.0 STANDARD PROCEDURES FOR EVALUATION OF TEST KITS

An examination of the literature has identified several standard methods for evaluating test kits. One is ASTM Method E 1828-01, “Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Spot Test Kits for Lead in Paint.” (ASTM, 2001b). The steps of the standard (described just below) are straightforward.

- Collect paint samples or reference materials, or prepare synthetic standard paint films.
- Test paint samples with the test kit according to ASTM Practice E 1753-04, “Standard Practice for Use of Qualitative Chemical Spot Test Kits for Detection of Lead in Dry Paint Films” (ASTM, 2004).
- Prepare and analyze tested paint samples for quantitative Pb content using ASTM standards.
- Compare the quantitative Pb content data from chemical analysis to the qualitative Pb spot test kit results.
- Determine the performance parameters of a particular spot test for a particular paint matrix by statistically modeling the comparative data.

Section 7.3 of the standard states that “hundreds of samples spanning the Pb concentration range of interest must be obtained for each combination of spot test kit and sample matrix that is to be tested.”

Although modeling the qualitative spot test kit data is very complex, it enables the determination of the level of confidence of obtaining a positive or negative response at a given concentration.

Another method for evaluation is E 1775, “Standard Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead” (ASTM, 2001a). This method

- provides guidelines for determining the performance of field-portable quantitative Pb analysis instruments;
- applies to field-portable electroanalytical and spectrophotometric (including reflectance and colorimetric) analyzers; and
- addresses sample matrices of concern including paint, dust, soil, and airborne particulate.

Another study was performed as a test of manufactured paints for test kit evaluation, although it is not a standard method. In this study directed by QuanTech, four rhodizonate-based test kits and one sulfide-based test kit were applied to a set of the real-world painted building components that are included in the HUD archive materials used for evaluating portable XRF instruments (Cox et al., 2001). The objectives were to (1) determine the extent to which manufactured paint films previously tested by NIST (Rossiter et al., 2000) are representative of field paint samples with respect to the evaluation of test kit performance, and (2) investigate the development of Performance Characteristic Sheets from the data collected at NIST using the manufactured paint samples. The real-world samples and manufactured samples had similar Pb levels. The same protocols were used for testing both the NIST and archive samples. The substrates were divided into reactive and nonreactive substrates based on the potential for chemical interaction between test kit reagents and the substrate. Wood was the nonreactive substrate for both types of kits. The reactive substrate was plaster for the rhodizonate kits and metal for the sulfide kit. As to the goals of this study, the test kit evaluation system using manufactured samples was found to be acceptable for predicting performance of test kits on nonreactive substrates; that is, the manufactured samples appeared to be representative of the archive samples in that the response curves for the archive samples fell between the response curves for the white lead- and lead chromate-based paints. For reactive substrates, the response curves generated with the archive samples were generally outside of the two curves for the white lead- and lead chromate-based paints, indicating significant differences between the archive and manufactured samples. One reviewer of this issue paper noted that in Table 12 of the Rossiter and co-workers report, there appears to be very little difference between substrate types within pigment types for the three best performing kits in the NIST study: LeadCheck Type 3, Lead Detective Type 2, and MS Sulfide Type 2. The archive study by Cox and others appears to have set aside these results for these three kits in favor of following the traditional approach of regarding plaster and drywall as not feasible for a rhodizonate kit and metal as not feasible for a sulfide kit. However, it appears possible that collected data may have supported the use of these three kits as negative screens on their reactive substrates.

Song, Schlecht, and Ashley (Song et al., 2001; Ashley et al., 2002) present a statistical procedure that enables the estimation of performance criteria and characteristics of field screening test methods. These methods, along with subsequent confirmatory analysis, allow one to evaluate qualitative, semi-quantitative, and quantitative field methods for their potential use in screening analysis. False negative rates, false positive rates, sensitivity, and specificity are key characteristics of screening methods that can be determined from performance characteristic curves. The authors present various options for using multiple test results to improve decisions based on test results.

Examples of other methods for evaluating and validating quantitative analytical procedures established by various organizations, such as EPA, include the following.

- “Environmental Monitoring and Assessment Program (EMAP), Chemical Method Evaluation Guidance,” EPA/620/R-96/001, 1996.
- NELAC Standard, National Environmental Laboratory Accreditation Conference, Quality Systems, Appendix C, “Demonstration of Capability,” June 2003.
- NIOSH Publication No. 95-117: Guidelines for Air Sampling and Analytical Method Development and Evaluation, May 1995.

These procedures typically would be followed for the quantitative test kits such as the PaceScan kit.

The Association of Official Analytical Chemists (AOAC) offers the Performance Tested Methods Program (<http://www.aoac.org/testkits/perftestedmtd.html>), which provides an independent third-party review of test kit performance claims. Test kits found to be in conformance with their claims are granted Performance Tested Methods status by AOAC. The Performance Tested Methods status assures the test kit user that independent assessment has been conducted, and the kit performs as claimed.

6.0 SUMMARY AND RECOMMENDATIONS FOR FUTURE KIT EVALUATION

A summary of the studies reviewed in this issue paper are presented in Table 26. Overall, these studies give a comprehensive picture of the performance of qualitative and quantitative test kits.

6.1 Historical Perspective

Evaluations of Pb test kits have been performed for some 20 years. The original qualitative test kits were based on the reaction of Pb with rhodizonate or sulfide to form pink or gray/black colors, respectively. Most of the kits developed over the past 15 years also have been based on one of these two reagents because of their specificity for Pb and the ease of visualizing the formation of the characteristic color. The sensitivity of the qualitative kits has changed very little. Evaluations performed in the early 1990s and more recently demonstrated that most of the rhodizonate- and sulfide-based test kits show a positive visual response to only a few micrograms of Pb, which makes them conservative and protective but also results in a large percentage of false positive responses relative to the Federal regulated level of 1 mg Pb/cm² or 0.5% Pb. Figure 3 in this document shows the inflection point of the response curve for LeadCheck to be at about 0.3 mg/cm², whereas Figure 4 in this document shows the inflection point of the response curve for MA Sulfide to be at about 0.8 mg/cm²; both show positive responses at levels less than 1 mg/cm².

Qualitative Pb test kits now are being developed using enzymes and immunochemistry. These biochemical systems provide high selectivity for Pb and may show fewer metal interferences than the rhodizonate or sulfide approaches. They also provide very sensitive measurements, reacting to the same low levels that are detected with rhodizonate and sulfide. One of the issues to be addressed is the stability of these biochemical systems. Being organic, they are subject to oxidation, loss of activity (denaturation), and sensitivity to the pH and ionic strength of the test solution.

The quantitative methods (colorimetry, ASV, reflectometry, and fluorescence) have been in the analyst's collection of tools for many, many years. What is relatively new in the last 15 years or so is the miniaturization of the instruments, which makes them suitable for use in the field. These newer, miniaturized versions are small, rugged, and relatively easy to use. The same is true for instruments used for preparation of the paint samples, such as field-portable microwave ovens and ultrasonic baths.

6.2 Issues with Qualitative or Spot Test Kits

In the recent past, a number of controlled laboratory studies and field studies of qualitative or spot test kits have been performed. Standard test solutions and real-world and synthetic paint

Table 26. Summary of Studies Reviewed in This Issue Paper

Report Section	Study Year	Reference	Kit Types			Testing Location		Application		Reference Materials Used						Number of Samples/ Number of Tests	Other Measurements				Number of Testers
			Rhodonate	Sulfide	Quantitative	Lab	Field	In situ	Ex situ	Real World	Synthetic	Ground	Films	Chips	Solution		XRF	AA	ICP	ASV	
1.1.1	1990	Blackburn (1990)		•		•			•							Unknown/ 377		•			Unknown
3.1.1	1993	Luk et al. (1993b)	•	•		•			•				•		•	27/144			•		2
3.1.2	1993	EPA (1995b)	•	•			•	•		•						1290/7740			•		Unknown
3.1.3	1994	Adler (1994)			•	•			•			•				1/179				•	Unknown
3.1.4	1998	Sharman and Krenzelok (1996)	•			•			•			•				26/78		•			Unknown
3.1.5	1995	Gutknecht et al. (1997)	•	•	•		•	•				•				18/378			•		3
3.1.6	1998	Ashley et al. (1998)	•		•		•		•		•		•			166/166	•	•	•	•	Unknown
3.1.7	1997	Gutknecht et al. (1999)	•	•			•	•				•				115/2415			•		4
3.1.8	2000	Rossiter et al. (2000)	•	•		•			•		•		•			20/3000			•		5
3.1.9	2000	Cox et al. (2001)	•	•		•				•	•		•			124/1420			•		5

Table 26. Summary of Studies Reviewed in This Issue Paper (cont'd.)

Report Section	Study Year	Reference	Kit Types			Testing Location		Application		Reference Materials Used						Number of Samples/ Number of Tests	Other Measurements				Number of Testers
			Rhodizonate	Sulfide	Quantitative	Lab	Field	In situ	Ex situ	Real World	Synthetic	Ground	Films	Chips	Solution		XRF	AA	ICP	ASV	
3.1.10	2006	Weydt (2006)		•		•				•					•	Unknown					Unknown
3.1.11	2005	Lu (2005)	Enzyme			•					•		•			26+/ Unknown			•		1
3.2.2	1996	Williams et al. (1996)			•	•			•			•				8/55				•	1
3.2.3	1993	Gutknecht et al. (1997)			•		•	•				•				115/345			•		2

samples have been used for these evaluations. The data presented in the preceding sections of this paper, which are representative of the data collected in these studies, clearly show that the spot or qualitative test kits have great variability in their responses to Pb in paint samples. The results of these studies also show that most kits show a relatively large number of positive responses to levels below 1 mg Pb/cm², but many fewer negative responses for levels above 1 mg Pb/cm². As shown in the work by Luk et al. (1991), the test kits are very sensitive, responding positively to only a few micrograms of Pb. Therefore, as most data show, these rhodizonate- and sulfide-based spot test kits are generally useful as screening tools for the presence of Pb in paint, including very low or “zero” Pb concentrations.

Various factors account for the lack of reproducible results with direct testing of the paint surface. One is that the extraction reagents used in the kits are mild, and, therefore, the amount of Pb released for reaction depends on the form of the Pb. Harper et al. (1995) point out that inconsistent extraction is one of the primary sources of varying responses. Also, the substrates give rise to interferences; calcium in plaster reduces the rhodizonate response, and metal substrates cause false positive responses with the sulfide-based kits. The structure of the layers of the paint also affects the results. Variation in the structural composition that could include thin layers of highly leaded paint overcoated with other layers of either leaded or nonleaded paint will have an impact on the tester’s ability to get a reliable visual response. A homogeneous thin layer of paint at 1 mg Pb/cm² would be expected to test differently than a homogeneous thick layer of paint at 1 mg Pb/cm².

With qualitative or spot test kits applied to whole or intact paint, it appears to be very difficult to meet the measurement goal, “Demonstrated probability (with 95% confidence) of a false positive response no more than 10% of the time to Pb levels below the regulated level.” However, it appears the goal, “Demonstrated probability (with 95% confidence) of a false negative response less than 5% of the time to Pb levels above the regulated level,” already has been met. That is, a test kit easily can be made to react with a minimum amount of Pb expected (e.g., a few micrograms), and, therefore, usually be positive above the action level. However, having a qualitative or spot test kit applied to whole or intact paint that shows no reaction until very close to 1 mg Pb/cm² or 0.5% Pb will not be reliable because the amount of Pb released for reaction with the test kit reagent(s) cannot, as of this time, be controlled.

Reviewers who commented in EPA Docket # EPA-HQ-OPPT-2006-0044 expressed concerns (by more than 10 to 1) about the “reliability, acceptability, and availability of the test kits” (see www.regulation.gov).

In summary, issues that make it difficult for the direct contact, qualitative, or spot test to meet the proposed Federal performance standards described previously include

- variability in the effective sample size (e.g., paint surface versus cut notch);
- weakness of the kit’s Pb extraction reagent;
- variability in the chemistry/extractability of the extraction reagent from kit type to kit type;
- variability in the amount of Pb exposed for extraction from test point to test point on the same paint;
- rate of extraction;
- effects of varying Pb-containing layer thickness and the presence of additional layers of Pb-containing and low-Pb paint;
- presence of interfering metals or substrates; and
- capability of the reagents rhodizonate and sulfide to form visually apparent responses with only a few micrograms of Pb in solution, thereby yielding a large number of false positive results relative to the proposed Federal performance standards.

6.3 Addressing the Basic Issue of Performance

The most basic issue leading to “false” positive results (as defined by the proposed Federal performance standards) is the uncontrolled extraction of Pb from paint during direct-contact testing. The high sensitivity of the currently available qualitative test kits, in a sense, compensates for this variability, making these tests very well suited for screening for Pb and especially useful to the homeowner as a conservative, protective tool. However, to meet the goal of less than 10% false positives, there is need to quantitatively control the fraction or amount of Pb extracted from the paint. It appears that the only way to control this extraction is to extract an entire, well-defined sample.

The qualitative test kits have potential for meeting the proposed Federal performance standards if we assume the following.

- The paint samples are well defined by collecting a known area or mass.
- The samples are homogenized.
- At least 90% of the original Pb is “available” for reaction with the test kit reagents.

This view is based on the fact that the transition from negative response to positive response is narrow, as available amount of Pb ion changes. Table 27 summarizes the ranges from all negative to all positive from Luk et al. (1993b).

Table 27. Test Kit Response to Pb Ion [from $\text{Pb}(\text{NO}_3)_2$] in Solution: 100% Negative to 100% Positive (Luk et al., 1993b)

Test Kit	Range from All Negative to All Positive
LeadCheck	0.5 to 1.0 μg
Verify LeadTest	0.1 to 0.3 μg
Frandon Lead Alert	0.5 to 0.7 μg
Merck EM Quant	0.5 to 0.6 μg
Lead Detective	0.5 to 2.0 μg

Development of a qualitative test kit that provides a sharp transition or “end point” with a solution representing 1 mg/cm^2 (or 0.5%) concentration of Pb possibly could achieve the proposed Federal performance standards. One possible way to achieve such an end point is to use quantitatively controlled amounts of reagents that visually indicate the complete reaction of an exact amount of Pb.

Solubilizing the Pb opens up the possibility of using semi-quantitative or quantitative test kits for the analysis. Use of these kits most likely will be more costly in terms of time and equipment than will the qualitative kits. Nevertheless, quantitative kits should be considered as tools for meeting the proposed standards because they already have been shown to work well. One can see in the laboratory work of Luk et al. (1993b) presented in Section 3.2.3 that the Hach colorimeter yields results within about 10% of those expected with real-world paints. In the work of Williams et al. (1996), as described in Section 3.2.2, the mean recovery with laboratory-prepared samples averaged about 100% for the Reflectoquant and about 90% for the PaceScan ASV system.

6.4 Method Selection and/or Development

Any manufacturer or research group that identifies a current method, modifies a current method, or develops a new method that meets the proposed Federal performance standards will need to address the main issue of performance described above (uncontrolled extraction of Pb from

paint during direct-contact testing). To successfully address this issue of quantitative recovery of Pb, new or modified methods will need to

- collect a defined sample,
- prepare the sample for extraction/solubilization of the Pb, and
- perform actual quantitative extraction/solubilization of the Pb.

As noted in previous sections of this document, Pb in solution can be measured by a variety of field portable methods. One of the challenges to be met by the developers is dealing with any adverse reactions between the chemistry of the Pb measurement method and components of the paint other than Pb and/or the reagents used to extract/solubilize the Pb.

6.5 Method Evaluation

When a test kit has been identified or developed that potentially will meet the proposed Federal performance standards, it must be put through a thorough series of tests to validate its performance. Evaluation of a candidate test kit should be performed both in the laboratory and the field. One of the standard methods described in Section 5 is recommended. In the laboratory, the evaluation would include testing with standard Pb solutions, standard paint extracts, and known synthetic and real-world paint samples.

6.5.1 Performance Evaluation Samples

6.5.1.1 Sample Preparation

The size of the sample will be determined by the sample requirements of the measurement method, with more sensitive methods requiring less sample than less sensitive methods. Preparation of standard Pb solutions is straightforward. Standard paint samples to be used include ground real-world paint samples, such as NIST SRMs and those used in the ELPAT program; paint films prepared in the laboratory also could be included.

Using laboratory-prepared paint film samples and real-world paint samples adds the challenge of preparing these intact paint chips or films for extraction. As noted, the size of the sample will be determined by the sample requirements of the measurement; sample size is also important because a larger sample is generally more representative of the whole area (e.g., painted wall) than a small sample. In past studies, field-collected paint samples sizes have varied from circles 1 cm in diameter to squares 2 in on a side.

Pulverizing, grinding, or mashing the paint samples into pieces or particles small enough for efficient extraction is affected by the thickness, layering, and brittleness/hardness of the sample. Harper and Gutknecht (2001) collected paint samples of varying sizes (areas and masses) at different locations across a variety of surfaces, including metal, plaster, concrete, and wood, and compared a variety of grinding techniques. Manual mortar and pestle grinding for at least 1.5 min and mechanized grinding techniques were found to generate similar homogenous particle size distributions required for aliquots as small as 0.10 g. However, simple grinding in a centrifuge tube with a glass rod is unlikely to yield this same degree of homogeneity. When samples were about 0.1 g or less, there was a significant amount of sample left in or on the grinding apparatus. Homogenization and subsampling steps were found to be the principal sources of variability related to the size of the sample collected. In addition, suitable homogenization takes time and, therefore, increases costs.

The challenge for preground, homogenized paint samples is the efficiency of the extraction. The efficiency of the extraction reagent/procedure for such samples already may be known from previous studies. If not known, the extraction efficiency would be determined by (1) “hard” or total digestion of an aliquot of the extract collected by the candidate method; (2) “hard” or total digestion of the postextraction paint residue; and (3) measurement of both these total digestion

extracts by AAS or ICP-OES to determine the true amount extracted versus the true or expected amount present in the original sample, respectively. One also could perform total digestion and ICP analysis of the original, well-homogenized ground paint samples to determine the expected concentration.

The Pb concentration of a real-world paint chip cannot be estimated accurately before analysis. Every chip collected, even if from the same painted surface, is different; no two are identical because of differences in application of paint across the surface. At the beginning of an application, the paint brush or roller is loaded heavily and the paint application is relatively thick. At the end of the application, the brush loading is light and the paint application is relatively thin. Multiple applications of paint over time compound this difference in the paint composition from point to point across a surface. The only way to determine the original Pb concentration value for a real-world paint chip is to perform a “hard” or total digestion of the solution and paint residue remaining after drawing an aliquot for the test kit measurement and then to analyze this digestate with AAS or ICP-OES. The acceptability of the total Pb solubilization procedure—sample grinding or pulverizing plus extraction—is determined by the amount of Pb in the extract relative to the original or expected amount in the paint sample.

6.5.1.2 Laboratory-Prepared Paint Films

Laboratory-prepared paint films have been used by other researchers (Luk et al., 1993b; Rossiter et al., 2000) for evaluation of test kits. These materials offer the potential of a supply of reproducible reference materials that are well characterized and can be used by different researchers and manufacturers to evaluate a variety of different test kits, thereby giving comparability of evaluation. However, making these films is a challenge. The variables to be considered include the following.

- Chemical composition of the films (e.g., lead carbonate, lead chromate, red lead, linseed oil [raw and/or boiled], organometallic driers, or, alternatively, Pb-spiked, available oil-based and/or water-based paints)
- Substrate selection (reactive or nonreactive)
- Potential range of Pb concentrations
- Paint film thickness
- Layering (single layer versus multiple layers)
- Coatings
- Achieving acceptable area uniformity
- Chemical analysis
- Artificial aging

Preparing paint mixtures according to old paint recipes is not technically difficult. However, a significant effort must be given to preparing very uniform paint films that will be suitable for use as reference materials; this is especially true when multiple layering is performed. A major consideration is artificial aging of the laboratory paints such that they exhibit the hardness and “exposed” state of old paints; there are no fast and easy methods for this process. Because every real-world paint is different in terms of the combination of composition, layers, mix of paint types, age, and condition, it is impossible to have a set of synthetic test materials that represent all paints that will be found in the field. Limits may include (1) thick and thin, (2) brittle and rubbery, (3) few layers and many layers, and (4) white lead-based and other Pb-based pigments such as chromate. (Most old paints used white lead as the primary pigment.) It is assumed that the Pb concentrations needed will include a few (2 to 3) at levels well below and well above the action levels (1 mg/cm² or 0.5% by weight) and a moderate number (6 to 10) within $\pm 20\%$ of the action levels to fully and accurately characterize the transition range for the test kit from negative to positive. This means 10 to 15 different concentrations for each different paint

material/structure and substrate combination. As per the tests of Rossiter et al. (2000), one might simply use wood as a nonreactive substrate and iron and plaster as potentially reactive substrates. With 3 different Pb pigments, 2 different overlayers, and 3 substrates, there is the potential need for close to 300 test samples for a thorough evaluation of the most promising test kit method or methods.

6.5.2 Evaluating the Testing/Measurement Results

The evaluation of the measurement component of a kit is relatively straightforward if we assume that the only way to meet the proposed Federal performance standards for test kits is to have the Pb removed from the paint sample and in solution, and that this can be done successfully. Using both standard Pb solutions and “known” paint extracts, the accuracy of a qualitative test end point (i.e., the number of negative values when the concentration is less than a value equating to 1 mg/cm² [or 0.5%] or the number of positive values when the concentration is greater than a value equating to 1 mg/cm² [or 0.5%]) can be determined. The response versus solution concentration data from this correct/incorrect analysis also can be plotted to produce characteristic response curves. The response could be quantified several ways: (1) 0 for no response or 1 for a positive response; (2) an average numeric value for replicates at each concentration; or (3) giving the intensity of the response a numeric value, as done by Gutknecht et al. (1999). The curve could be modeled as done by EPA (1995b) to yield predictions of the probability of positive response as a function of test sample concentration.

6.6 Summary

In summary, if it is assumed that the accuracy and precision are improved by ex situ testing (i.e., analysis of the paint removed from its source), there are still a number of issues to be met to meet the proposed Federal test kit performance standards (see just below).

- Collecting a representative sample or samples of paint
- Removing an intact sample without substrate (which may have high Pb levels)
- Accurately controlling or knowing the paint sample area for subsequent area concentration calculations
- Breaking, crushing, grinding, etc., the paint sample to allow rapid Pb extraction and dissolution
- Finding and using an extraction reagent that will extract 90+% of the Pb in the prepared (ground) paint sample while still being safe and relatively convenient to use in the field
- Identifying an apparatus (e.g., ultrasonic bath or field microwave oven) that can be used in the field to promote the dissolution of the Pb
- Applying an analytical method that will yield an accurate ($\pm 10\%$) measure of the Pb in solution and be relatively safe to use, with minimum waste generation

These challenges go beyond the technical issues. Labor requirements and material costs must be optimized to meet those proposed goals that relate to time of analysis and cost—less than 1 h and less than \$2 in materials per sample. Finally, the final test kit procedure will need to be reasonably easy to perform with some minimum level of training.

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