

Development and Preparation of Lead-Containing Paint Films and Diagnostic Test Materials

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5 Disclaimer

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Abstract

Lead in paint continues to be a threat to children's health in cities across the United States, which means there is an ongoing need for testing and analysis of paint. This ongoing analytical effort and especially development of new methods continue to drive the need for diagnostic testing materials that provide the analytical challenges of real-world paints. To this end, 31 different types of paint test materials were developed and prepared. Preparation of the materials included development of lead-containing paint films yielding an overall relative standard error for one individual test sample being less than 10%. The 31 diagnostic test materials prepared with these paint films included two lead pigments; lead concentrations from nominally 0 to 2.0 mg lead/cm² (0 to 5% lead by weight); overlayers of both "lead-free," oil-based and water-based paints; Al, Ba, and Mg as potential chemical interferents; red and black potential color interferents; and substrates of wood, metal, masonry, and plaster. These materials challenge each step in method development and evaluation, including paint sample collection and preparation, lead extraction, and measurement of solubilized lead. When the materials were used to test performance of a new lead-in-paint testing method based on extraction using a rotor/stator method and measurement using turbidimetry, the results agreed to within $\pm 20\%$ of the expected lead values for 30 out of 31 of the diagnostic test materials, thereby demonstrating their levels of quality and utility.

TOC entry

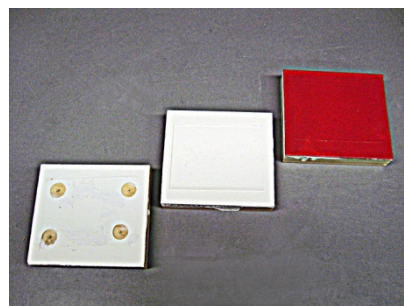
A procedure for preparing paint testing materials having controlled variations in substrate, lead form, layers, and potential analytical method interferences.

Introduction

Lead-based paint is a major source of lead poisoning for children and can also affect adults.^{1,2,3,4} In response to this threat to human health, lead in paint at or above 0.5% by weight or 1 mg of lead/cm² in most residential housing must be controlled or removed according to Title X of the Housing and Community Development Act.⁵ In addition, these federal regulated levels must now be applied in the renovation and repair industry according to the U.S. Environmental Protection Agency's (EPA's) Lead Renovation, Repair, and Painting (RRP) rule.⁶ Consequently the need for new reference or diagnostic test materials that simulate, as much as possible, real-world paints is ongoing. Such materials would not only be very useful for development of new methods such as test kits that meet the RRP rule requirements, but also for quality assurance (QA) for these new methods and methods already established, including x-ray fluorescence and methods involving sampling, grinding/homogenizing, digesting/dissolution, and measurement.

The test material that would best represent the type of paint sample brought back to the laboratory for routine analysis, which would include sample preparation such as grinding, would be paint applied to a real-world substrate that contains a known amount of lead. Though real-world paint shows great variability,⁷ the only reasonable approach is to prepare paint films with a homogeneous distribution of lead. The targets for the variability or uncertainty in the paint film thickness (microns), the paint film areal lead concentration (mg/cm²), and the paint diagnostic test material areal lead concentration (mg/cm²) would ideally be all less than $\pm 10\%$.

The goals of this research were, therefore, to develop, prepare, and evaluate intact test paint materials that (1) meet the variability goals stated previously; (2) contain known amounts of lead; and (3) as much as possible, have the characteristics of the prepared paint used in dwellings. In developing the materials described here (which include the paints and substrates), we considered the physical and chemical forms needed and whether the paint layers were realistically thick to provide challenges to the collection and extraction phases; had bottom leaded paint



layers that were adequately brittle to behave like real-world aged paints during processing/grinding; were chemically challenging to the extraction phase; and contained potential interferences like real-world paints to challenge the detection phase.

5 Standard Paint Films Design

Several intact paint test materials have been developed previously. The National Institute of Standards and Technology (NIST) offers paint film standard reference materials (SRMs) that consist of leaded paint on plastic backing with a protective
10 coating over the paint and, thus, do not represent real-world paints for methods other than XRF testing.⁸ NIST offers only one real-world reference material, RM 8680, which consists of paint on fiber board; it was collected in an old house in Durham, NC.⁹ QuanTech increased the sample complexity and, therefore, the
15 analytical challenge by developing a series of performance evaluation paint films by combining a linseed oil mixture and white lead and casting stand-alone films containing a bottom lead layer and multiple coats of non-leaded paint overlayers.¹⁰ Similar films were prepared for lead test kit evaluation.^{11,12} Though white
20 lead and linseed oil were used, these materials lacked the other complexities found with real-world paints such as potentially interfering colors and brittleness. In order to add these to the analytical challenge, research was undertaken to develop paint films that were more representative of real-world paints than
25 those previously developed. In this effort, the goals were to (1) use early 19th- or 20th-century lead-in-paint recipes, (2) use both white lead and lead chromate pigments, and (3) vary the lead concentration from <0.001% Pb to approximately 5% Pb or <0.01 mg lead/cm² to approximately 10 mg lead/cm². The 0.001%, 5%,
30 and 0.01 mg lead/cm² values are consistent with those prescribed in ASTM E 1828-01,¹³ while the top concentration is 10 mg lead/cm² instead of the 2 mg lead/cm² in the ASTM standard. It



Fig. 1. Gardco paint drawdown apparatus (www.gardco.com)

35 was further proposed that each paint layer be at least 35 to 80 μm thick, again in accordance with ASTM 1828-01. Development of the paint films started with a design that included the following:

- selection of early 20th-century lead-in-paint recipes
- method of mixing paint components
- 40 • method of casting uniform paint films
- methods of determining paint thickness and concentration uniformity
- method of synthetic aging

This design was followed in earlier work that included the
45 successful development of stand-alone paint films.^{14, 15, 16}

Following this initial work, it was decided that more complex, realistic synthetic paint materials were needed to diagnose the impacts paint components have on performance in order to develop and/or improve available analytical techniques.

50 Therefore, following the design described above, a second research effort targeted test materials that were more complex, including the following:

- Multilayer configurations—One or two non-leaded paint layers over one or two leaded paint layers, and three or four
55 non-leaded paint layers over two leaded paint layers of different composition
- Paint on different substrates such as wood, as a test kit non-reactive substrate, and on metal and plaster as test kit-reactive substrates
- 60 • Presence of potential chemical and color interferences

Experimental

Casting Paint Films

Based on earlier experience, it was decided that the best method for making a paint film was to use a drawdown apparatus. The device used was purchased from Gardco (Pompano Beach, FL). This apparatus, as shown in Fig. 1, consists of a flat glass plate mounted on a frame. Attached to this frame is a framework that holds a bar of metal wound tightly and uniformly with one or two
70 layers of wire of a uniform diameter. To make the paint film, we put an aliquot of paint on the glass at the top of the apparatus; the handle is then pulled down at a constant rate. As the paint flows through the grooves between the wires, a uniform layer of paint is created.

The best results were obtained with about 20 mL of paint put
75 on “release paper” (paper coated with a material that allows the removal of dried paint) on the machine with a large glass syringe and the paint drawn with a #90, 0.090 in diameter, single wire, drawdown bar. Thinner films were difficult to remove without tearing. Drying time for oil-based paint was found to be about
80 120 hours. Paint thickness was determined by carefully removing the paint film from the release paper and placing it on a flat steel sheet from Gardco. The paint was then marked in 3 cm x 3 cm squares, resulting in about 24 to 30 squares per sheet. The thickness was then measured with DeFelsko’s PosiTector 6000.¹⁷
85 The average dry-paint thickness measured for three sheets (approximately 24 squares each) of commercially available white paint prepared using the #90 drawdown bar was 72.94 ± 2.78 (3.81%) microns. The overall thickness RSD was 4.77% for 6 different sheets with 24 to 30 test squares from each sheet.

90 Paint Preparation

The first batch (RTI Batch 1) of laboratory-prepared paint was made according to the following recipe (by weight) for interior white paint:¹⁸

- white lead—35%
- 95 • zinc oxide—35%
- linseed oil—8%
- petroleum spirits—7%
- paint drier—15% (commercial paint drier, 6% cobalt NAP-ALL, OMG Americas, Inc., Franklin, PA)

100 The components were first mixed using a Ross and Son Engineering, LSK high-shear, Model HSM-100SK mixer.¹⁹

Mixtures similar to RTI Batch 1 (see Table 1) generated using this device did not flow well when cast. Varying the relative amounts of components, the order of mixing, and the time of mixing did not improve the results.

At this point, a commercial product known as Penetrol²⁰ was used in two batches because this material is supposed to make paint easier to spread smoothly. This material did result in considerable improvement of the paint flow as shown in Table 1. It is believed that the mixtures without Penetrol were not flowing because the white lead and/or zinc oxide pigment particles agglomerate to form much larger particles. Penetrol apparently provides for “wetting” of the small, pigment particles. Because we only wanted to use old recipes, we looked for other ways to “wet” the particles and make uniform paint films without using Penetrol. Also, the commercial paint drier (Table 1, Batch 4) was no longer used to remain true to old recipes. Assuming particle “wetting” was the problem, a large mortar and pestle was used to break up the agglomerates prior to using the Ross high-shear mixer. This procedure yielded some improvements (see Table 2) but was too laborious.

To develop a simpler method of mixing the paint ingredients, we purchased a Model 764AVM, single-tier ball mill from US Stoneware.²¹ One-liter grinding mill jars were purchased along with ½ in x ½ in (1.3 cm x 1.3 cm) cylindrical alumina grinding media. The expected advantage of using a ball mill was that all ingredients can be combined and mixed at one time. Batches 14 and 15 were prepared using this ball mill. The preparation of these batches is described in Table 2. As noted, the areal thickness variabilities for Batches 14 and 15 prepared with the ball mill were excellent—less than 5%.

Table 1. Composition of RTI paint batches 4 and 6 through 8

Mixture Components	Batch 4	Batch 6	Batch 7a	Batch 7b	Batch 8
White lead	400 g	350 g	550 g	550 g	700 g
Zinc oxide	400 g	350 g	550 g	550 g	500 g
Raw linseed oil	95 g	72 g	200 g	200 g	200 g
Boiled linseed oil	95 g	72 g	200 g	200 g	200 g
Mineral spirits	160 g	124 g	100 g	100 g	100 g
Paint drier	50 g	-	-	-	-
Penetrol	-	-	-	150 mL	130 mL
RESULT	Did not flow; visible ridges; brittle	Did not flow; visible ridges	Did not flow; visible ridges	Moderately smooth	Smooth; a few ridges
Thickness, μm	114 $\pm$ 26 (23%)	86.3 $\pm$ 10.4 (12.1%)	98.8 $\pm$ 10.3 (10.5%)	89.9 $\pm$ 5.5 (6.1%)	96.3 $\pm$ 5.9 (6.2%)

Table 2. Summary of preparation and results with RTI paint batches 12–15

	Batch 12	Batch 13	Batch 14	Batch 15
White lead	110 g	220 g	110 g	50 g
Zinc oxide	770 g	660 g	770 g	550 g
Raw linseed oil	157 g	176 g	150 g	70 g
Boiled linseed oil	152 g	181 g	150 g	70 g
Mineral spirits	100 g	83 g	110 g	70 g
METHOD	Mix with mortar and pestle; shear in mixer 25 min. at 6,500 rpm; cool down overnight before adding mineral spirits	Mix with mortar and pestle; shear in mixer 25 min. at 6,500 rpm; cool overnight before adding mineral spirits	Ball mill-69 R.P.M. 1 L jar 44 hrs	Ball mill-69 R.P.M. 1 L jar 72 hrs
RESULT	Slight ridges visible	Ridges visible	Looks very smooth	Looks most like commercial paint
Thickness, μm	88.0 $\pm$ 6.0 (6.9%)	90.8 $\pm$ 7.5 (8.3%)	98.7 $\pm$ 4.6 (4.6%)	99.4 $\pm$ 3.8 (3.8%)

Preliminary Analysis for Lead

To move ahead with testing for the ultimate goal of uniform lead concentration, we cut several 1.49 cm² squares from the sheet of paint film from RTI Batch 7b to perform a preliminary test of the uniformity of lead. The paint films as made were thin and rubbery. It must be noted here that real-world lead paint samples are generally brittle. This brittleness is a result of aging and exposure to the environment, including exposure to ozone and ultraviolet light. A long-term goal of this research is to artificially age the paint prepared in the laboratory so that it will have the brittleness and surface characteristics of old paint. The brittleness affects the ease of sample homogenization and lead extraction. It

is easier to grind hardened, brittle paint into fine particles than newer, more flexible paint, though such grinding usually results in some loss of fine lead-containing particles to the grinding device surfaces.²² There is experimental evidence that lead in fine particles of old, brittle paint is more easily dissolved in acid than lead in paint that has not undergone decomposition with time.²³

Though old leaded paint will most likely be brittle, overcoats of non-lead paint that have been applied recently (last 15 to 20 years or so) will not be as brittle as the older lead-containing paint and actually may be somewhat rubbery. Field samples with “rubbery” overcoats will be more difficult to grind and homogenize than those that are totally brittle.²⁴ In this work, the total combinations of the lead-containing paints and overcoats of non-lead, oil-based and water-based paints were heated to make the samples hard and brittle. Thus, this first generation of materials was not expected to be as challenging to methods of sample preparation as they would be if the lead-containing layers were heated while the overcoats were not. Making the samples more challenging, including using additional forms of lead, adding more potentially interfering chemicals, and using differential “aging,” could readily be done with future generations of these materials using the procedures presented in this paper.

Paint film squares were initially dried in an oven at various temperatures to approximate artificial aging. A batch was baked at 50°C for several days with no physical change. The same batch was then baked for another 2 days at 100°C, again with no physical change. The batch was then baked at 150°C. After only a few minutes, the paint turned brown and began to curl up on the edges. The paint was brittle and easily broken after about 60 hours at 150°C. This temperature and amount of time proved successful for subsequent work. Six baked paint film squares were ground up and extracted using the rotor/stator/nitric acid procedure^{25, 26} and analyzed by inductively coupled plasma optical emission spectrometry (ICP-OES).^{27,28} The result was a mean value of 4.7 ± 0.7 (14.9%) mg lead/cm². These values indicated that moderate lead uniformity was being achieved and, with further development, could ultimately show a variance of less than 5% in areal concentration.

To further test paint for lead films uniformity, we took aliquots from subsequently prepared and apparently improved sheets of paint and hardened them in the oven (150°C for 48 hours). These aliquots were prepared for ICP-OES analysis by a variety of methods, including (1) grinding in a mortar and pestle and extracting by ultrasonic/nitric acid digestion;²⁹ grinding in a test tube and microwave-aqua regia acid extraction;³⁰ and the rotor/stator-nitric acid extraction method.^{25,26} The results for these analyses showed great variability between methods and for each method. The lead results varied by 5 to 50%. Retests with both the same test film and the same method also showed major variability in results. At this point, there was no satisfactory explanation for the different values between methods and changing values with retesting. The methods used were consistent between original and retests. Also, errors in cutting the aliquots and weighing were highly unlikely, and there was little chance of any error in the measurement by ICP-OES because check samples and QC samples yielded the expected values. One

Table 3. Comparison of analysis results from three digestion methods using 0.2 g ground paint material (Lead concentration in %)

Paint ID	Expected Concentration (%) ^a	AREAL-Microwave ²⁷	Ultrasonic/25% HNO ₃ ²⁶	EPA Method 3051A ²⁹
Batch 22	13.4 ± 0.4	8.9 ± 0.9	7.3 ± 0.4	13.5 ± 0.1
Batch 26	2.69 ± 0.06	2.6 ± 0.2	1.04 ± 0.04	2.89 ± 0.01
ELPAT 52P3	2.14 ± 0.04	2.12 ± 0.01	2.18 ± 0.02	2.19 ± 0.02

^aExpected concentration based on AREAL microwave analysis of samples about or less than 0.1 g.

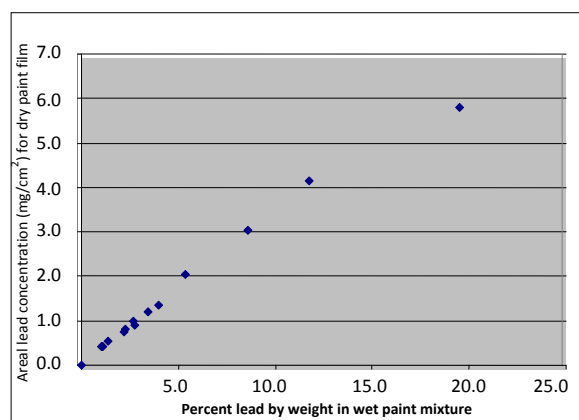


Fig. 2. Plot of paint film lead concentrations (mg lead/cm²) vs. lead concentration (% lead) in “wet” paint mixture.

thought was that the paint film extractability changes with age. However, replicate analyses of each of four batches analyzed at different times showed no apparent effect of age on the films. Therefore, the variability was believed to be in the sample preparation and most likely the extraction. That is, the extraction methods used were not sufficiently strong to digest the relatively “fresh” linseed oil-based paint when the sample amounts were greater than about 0.1 g; earlier work showed reproducible values for lead for batches when the individual samples were kept below about 0.1 g.³¹ To explore this further, samples weighing about 0.2 g were analyzed using the AREAL microwave method,³⁰ the ultrasonic/25% HNO₃ extraction method,²⁹ and EPA Method 3051A.³² This latter method consists of digestion in concentrated HNO₃ in a microwave vessel rather than dilute aqua regia and the microwave, tube-within-a-vessel configuration used with the AREAL method. Resultant data are presented in Table 3. As one can see, results acquired with EPA Method 3051A agreed well with the “Expected Concentrations,” which correspond to the analysis data collected earlier using about or less than 0.1 g of sample and the AREAL microwave method. One can see too that the AREAL microwave method and the ultrasonic extraction methods do not do well with about 0.2 g of sample. From this point on, it was decided to use EPA Method 3051A for all film analyses.

With procedures for preparing and analyzing the paint films established, examination of the analysis data collected showed that there was a general relationship between the composition of the “wet” paint mixture and the final lead concentration in mg lead/cm². Fig. 2 shows a plot of paint film value (mg lead/cm²) vs. percentage lead by weight in the “wet” paint mixture. The relationship was recognized with the first five or six batches

analyzed using EPA Method 3051A and subsequently served as a guide for producing target paint film lead concentration based on “wet” mixture lead concentration. The curve, as shown in Fig. 2, became more accurate for prediction as more paint films were prepared.

Here it should be noted that, among other purposes, this research was initiated to develop complex, test kit diagnostic paint materials that would be useful for response to the originally proposed RRP rule in 2006 of no more than 10% false positive responses and no more than 5% false negative responses, both at the 95% confidence level, at the action level of 1 mg lead/cm².⁶ The final RRP rule states, however, that lead test kits will not be tested in the 0.8 to 1.2 mg lead/cm² range.³³ Therefore, in the evaluation of any new methodology, false positive rates will be evaluated by a set of paint films that have lead concentrations less than 80% of the threshold. Similarly, false negative rates will be evaluated by a set of paint films that have lead concentrations greater than 120% of the threshold. Therefore diagnostic paint materials are only needed at two levels, 0.8 and 1.2 mg lead/cm².

Using the paint preparation procedures now well established, we prepared white lead paint films with targeted concentrations of 0.4 and 0.8 mg lead/cm². The recipes for these materials are presented in Table 4. These two paints were analyzed for lead and the results are presented in Table 5. One can see that the variability (RSD) is less than 5%.

Work to this point showed that the statistical variability of a set of samples cut from one sheet was generally less than 10% and gave indications that values of less than 5% could be achieved routinely with practice in preparing the paints. Of concern was the predicted variability of one sample. Statistical analysis was applied to the lead concentration data for multiple samples of four batches of paint, two each at 0.8 and 0.4 mg lead/cm² concentration.

Examination of the data shows that the variance in paint film lead concentration tends to increase as the expected value of lead increases. This was accounted for by allowing the variance to be a function of the expected value for lead. Specifically, a term was added to the model that allowed the variance to increase as the expected value of lead increased. The assumptions used for the data are that the observations are normally distributed, the mean varies by batch and that the variance is a function of the mean.

The model parameters were estimated using PROC NLMIXED in SAS.³⁴ The parameters “separate sheets of paint” and “intra-test correlation” were determined to be not significant and, therefore, were not included in the final analysis. The result was that observations within a batch were assumed to be independent of each other. The results of this analysis showed the relative standard error to be about 7%. Because the samples analyzed were each composed of two 1.49 cm² chips, the relative standard error of one chip would be about 1.4 times this value, or 9.8%. This means that the data quality objective of lead concentration uncertainty less than 10% was being met and production of the paint films could continue.

Diagnostic Test Material Preparation Plan

The characteristics of paint samples most likely to challenge testing methods include

- paint pigment,
- paint film layer structure,

Table 4. Recipes for preparation of white lead paints nominally 0.8 and 0.4 mg lead/cm²

Component	Batch 27 ^a (Target—0.8 mg lead/cm ²)	Batch 31 ^a (Target—0.4 mg lead/cm ²)
White lead	23 g	11.25 g
Zinc oxide	577 g	588.25 g
Raw linseed oil	70 g	70 g
Boiled linseed oil	70 g	70 g
Mineral spirits	70 g	70 g

^aEach rolled in ball mill for 96 hrs.

Table 5. Results of analysis of white lead paint Batches 27 and 31 with target values of 0.8 and 0.4 mg lead/cm²

	Sample Wgt (g)	Lead in Paint (mg/cm ²)
Batch 27, Test #1 n = 8	0.096 ± 0.002 (2.1%)	0.808 ± 0.017 (2.1%)
Batch 27, Test #2 n = 8	0.098 ± 0.002 (2.1%)	0.804 ± 0.028 (3.5%)
Batch 31, Test #3 n = 12	0.093 ± 0.004 (4.3%)	0.403 ± 0.019 (4.7%)
Batch 31, Test #4 n = 12	0.095 ± 0.003 (3.5%)	0.402 ± 0.013 (3.3%)

- chemical interferences,
- color interferences, and
- substrate variation.

In the real world, an enormous number of combinations of these variables exist. However, because of limited resources, a final plan was developed to prepare 31 different types of paint diagnostic test materials. Two lead pigments were selected: white lead, which has been used for thousands of years and is the pigment found in most old lead-containing paints, and lead chromate, which is much less soluble than white lead and, thus, presents a significant challenge to any sample digestion method. As noted above, two concentration levels were selected for each pigment type, 0.4 mg lead/cm² and 0.8 mg lead/cm². This allowed preparation of films at 0.8 mg lead/cm² and 1.2 (0.4 + 0.8) mg lead/cm², which are at ±20% of the federal standard for lead-based paint action level.^{6,33} To adequately represent paint samples from old buildings, we designed the materials to include multiple layers of lead-containing paint with overlayers of non-lead-containing, oil-based and water-based paints. Prior to testing for effects of pigment, interferences, and substrate, researchers need to test for method responsiveness over a range beyond 0.8 to 1.2 mg lead/cm². Therefore, films at 1.0, 1.2, and 2.0 mg lead/cm² were prepared using white lead.

Because of limited time and funding, only a limited number of potential chemical interferences could be used, so the elements Al, Ba, and Mg, which are common to old paints and may interfere with colorimetric tests, were selected.³⁵ As with chemical interferences, only a limited number of color interferences could be used. Red color provided by red iron oxide was chosen as a potential interference with the lead-positive pink color found with rhodizonate used in some colorimetric test kits, and black provided by black iron oxide was chosen as an extreme potential interference to all colorimetric tests. Finally, most common substrates found in field sampling are wood, masonry, plaster, and metal, so it was decided that each of these be used for the test materials. These provide not only different challenges to

Table 6. Proposed lead-in-paint diagnostic test materials

Type	Diagnosis	Substrate	No. Films and Lead Compound ^a	White Paint Overlayers: No. and Type ^b	Chemical Interferences Al, Ba, Mg, Fe	Lead Layer Colors ^c
A	Sensitivity, accuracy, and precision	Wood	1 non-Pb	1-OB, 2-WB	---	White
B	Sensitivity, accuracy, and precision	Wood	1-WPb	1-OB, 2-WB	---	White
C	Sensitivity, accuracy, and precision	Wood	1-WPb	1-OB, 2-WB	---	White
D	Sensitivity, accuracy, and precision	Wood	1-WPb	1-OB, 2-WB	---	White
E	Sensitivity, accuracy, and precision	Wood	1-WPb	1-OB, 2-WB	---	White
F	Sensitivity, accuracy, and precision	Wood	1-WPb	1-OB, 2-WB	---	White
G	Accuracy and precision (A & P) with form of Pb	Wood	1-CrPb	1-OB, 2-WB	---	Yellow
H	A & P with form of Pb	Wood	2-CrPb	1-OB, 2-WB	---	Yellow
I	A & P with multiple layers	Wood	1-WPb	1-OB, 2-WB	---	White
J	A & P with multiple layers	Wood	2-WPb	1-OB, 2-WB	---	White
K	A & P with substrate effects	Steel	1-WPb	1-OB, 2-WB	---	White
L	A & P with substrate effects	Steel	2-WPb	1-OB, 2-WB	---	White
M	A & P with substrate effects	Masonry	1-WPb	1-OB, 2-WB	---	White
N	A & P with substrate effects	Masonry	2-WPb	1-OB, 2-WB	---	White
O	A & P with substrate effects	Plaster	1-WPb	1-OB, 2-WB	---	White
P	A & P with substrate effects	Plaster	2-WPb	1-OB, 2-WB	---	White
Q	A & P with chemical interferences	Wood	1-WPb	1-OB, 2-WB	Al, Ba, Mg	White
R	A & P with chemical interferences	Wood	2-WPb	1-OB, 2-WB	Al, Mg, Ba	White
S	A & P with color interferences	Wood	1-WPb	1-OB, 2-WB	Fe	Red
T	A & P with color interferences	Wood	2-WPb	1-OB, 2-WB	Fe	Red
U	A & P with color interferences	Wood	1-WPb	1-OB, 2-WB	Fe	Black
V	A & P with color interferences	Wood	2-WPb	1-OB, 2-WB	Fe	Black
W	A & P with all potential interferences	Steel	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
X	A & P with all potential interferences	Steel	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
Y	A & P with all potential interferences	Steel	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
Z	A & P with all potential interferences	Masonry	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
AA	A & P with all potential interferences	Masonry	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
AB	A & P with all potential interferences	Masonry	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
AC	A & P with all potential interferences	Plaster	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
AD	A & P with all potential interferences	Plaster	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black
AE	A & P with all potential interferences	Plaster	1-CrPb, 1-WPb	3-OB,4-WB	Al, Ba, Fe, Mg	Yellow, Black

^aLead compounds: 1-WPb = 1 white lead film, 2-WPb = 2 white lead films; 1-CrPb = 1 lead chromate film, 2-CrPb = 2 lead chromate films

^bOverlayers: 1-OB = 1 oil-based lead-free paint overcoat; 2-WB = 2 water-based lead-free paint overcoats

^cColors added and sources: Red iron oxide added to paint film for red color; black iron oxide added to paint film for black color; yellow from lead chromate.

Table 7. Summary of paint films prepared and their use in different types of diagnostic paint test materials described in Table 6

Nominal Lead Concentration (mg/cm ²), in Prepared Paint Films	To be Used for Type
Blank	A
0.4 white lead	B, J, L, N, P
0.8 white lead	C, I, J, K, L, M, N, O, P
1.0 white lead	D
1.2 white lead	E
2.0 white lead	F
0.4 lead chromate	H, W, X, Z, AA, AC, AD
0.8 lead chromate	G, H, Y, AB, AE
0.4 white lead with Al, Ba, Mg	R
0.8 white lead with Al, Ba, Mg	Q, R
0.4 white lead with black iron oxide	V
0.8 white lead with black iron oxide	U, V
0.4 white lead with red iron oxide	T
0.8 white lead with red iron oxide	S, T
0.4 White lead with Al, Ba, Mg, black iron oxide	W, Y, Z, AB, AC, AE
0.8 White lead with Al, Ba, Mg, black iron oxide	X, AA, AD

remove the paint from the substrate, but also the potential of chemical interference (e.g., calcium in plaster interfering with rhodizonate and metal interfering with a colorimetric method based on lead ion complexation). With these parameters in mind, we developed the plan shown in Table 6, which shows the chemical and structural parameters of each of the 31 types of test materials to be prepared. A summary of the paint films made and their use in the different types of diagnostic test materials to be made are shown in Table 7.

Few difficulties were encountered in preparing the paint films. The white lead and chromate lead paints were prepared without difficulty following the established procedures. The concentration of the added metals, Al, Ba, and Mg, to the white lead paint was based on multielement data from previous paint sample analyses performed in our laboratory. Because levels of these metals vary so much in real-world paints, it was decided that setting up a worst case would be best. Subsequently, wet paint concentrations for Al, Ba, and Mg were chosen to be 4.3%, 3.2%, and 2.0%, respectively. After several failed attempts, a red paint was achieved with red iron oxide (Hoover Color Corporation, Hiwassee, VA, "HR-1201 Red" [Fe₂O₃]) at 15% concentration in the wet paint, and black paint was achieved with black iron oxide (Strem Chemicals, Newburyport, MA, Black Iron Oxide, [Fe₃O₄]) at 8% concentration in the wet paint.

With the methods and approaches for making the paint films needed for the different types of diagnostic test materials established, the paint films were prepared as listed in Table 7. The time for preparation of one batch of paint film included 4 days of mixing in the ball mill; 3 days for drying on the release paper; 3 days for baking; and 1 to 2 days for digestion and lead measurement. As noted, 16 different paint films were needed to make the 31 types of diagnostic material. The total wet weight for each of these 16 was nominally 810 g. The range of component concentrations to prepare each film was as follows:

White lead	0 to 6.7%
Chromate lead	1.7 to 3.2%
Zinc oxide	44 to 74%
Boiled linseed oil	8.6%

Table 8. Results of analysis of multiple samples taken from cast paint films needed to prepare all planned types of diagnostic test materials.

Batch	Nominal Areal Lead Concentration (mg/cm ²) and Film Description	Number of Films Prepared	Overall Thickness ^a (μm)	Areal Lead Concentration (mg/cm ²)	Mass Lead Concentration (%)	n
19	Blank	8	101.9 ± 9.5 (9.4%)	Blank	-	8
39	0.4 white lead	11	107.2 ± 7.3 (6.8%)	0.391 ± 0.027 (6.97%)	1.21 ± 0.02 (1.3%)	12
35	0.8 white lead	24	102.5 ± 6.2 (6.1%)	0.808 ± 0.051 (6.25%)	2.56 ± 0.06 (2.3%)	12
41	1.0 white lead	8	100.7 ± 5.1 (5.1%)	1.02 ± 0.05 (4.7%)	3.07 ± 0.03 (0.9%)	8
29	1.2 white lead	6	100.9 ± 4.0 (3.9%)	1.31 ± 0.06 (4.4%)	4.07 ± 0.05 (1.1%)	8
45	2.0 white lead	6	100.8 ± 7.3 (7.3%)	1.98 ± 0.12 (6.2%)	6.16 ± 0.11 (1.7%)	8
46	0.4 lead chromate	16	99.7 ± 6.7 (6.8%)	0.378 ± 0.028 (7.42%)	1.24 ± 0.03 (2.7%)	20
37	0.8 lead chromate	19	98.6 ± 6.1 (6.2%)	0.763 ± 0.056 (7.39%)	2.41 ± 0.02 (0.8%)	12
49	0.4 white lead with Al, Ba, Mg	15	103.2 ± 6.0 (5.8%)	0.358 ± 0.027 (7.64%)	1.21 ± 0.02 (1.4%)	12
50	0.8 white lead with Al, Ba, Mg	11	102.4 ± 6.8 (6.6%)	0.784 ± 0.096 (12.3%)	2.60 ± 0.05 (1.8%)	12
53	0.4 white lead with black iron oxide	8	88.0 ± 6.0 (6.8%)	0.406 ± 0.050 (12.25%)	1.20 ± 0.02 (1.3%)	8
54	0.8 white lead with black iron oxide	8	89.5 ± 6.9 (7.7%)	0.765 ± 0.045 (5.82%)	2.55 ± 0.04 (1.5%)	8
55	0.4 white lead with red iron oxide	6	101.5 ± 6.2 (6.1%)	0.364 ± 0.012 (3.39%)	1.24 ± 0.02 (1.4%)	8
56	0.8 white lead with red iron oxide	8	99.2 ± 6.5 (6.6%)	0.734 ± 0.026 (3.53%)	2.45 ± 0.04 (1.5%)	8
51	0.4 white lead with Al, Ba, Mg, black iron oxide	18	90.2 ± 6.9 (7.7%)	0.358 ± 0.033 (9.35%)	1.19 ± 0.01 (0.6%)	12
52	0.8 white lead with Al, Ba, Mg, black iron oxide	12	88.5 ± 6.4 (7.3%)	0.747 ± 0.046 (6.18%)	2.66 ± 0.04 (1.3%)	12

^aThickness tested on all films.

- Raw linseed oil 8.6%
- Mineral spirits 8.6 to 9.8%
- Al₂O₃, BaCO₃, MgCO₃ 8.0% 4.6% 6.9%, respectively
- Red iron oxide 15%
- Black iron oxide 8.0%

The number of films cast from each batch was varied depending on the need for test sample production. For lead concentration verification, typically two samples were taken for analysis from the first, middle, and last films cast from each batch, though more were taken when more than eight or nine films were cast from one batch (or duplicate batches with the same recipe). These were then baked and analyzed using Method 3051A and ICP/OES. The results are presented in Table 8. As noted, all batches of paint films had thickness variability less than 10%, but two of the batches had lead areal concentration uncertainty (mg lead/cm²) greater than 10%. All batches had lead mass concentration uncertainty (%) less than 3%.

Preparation of Diagnostic Test Materials

With the procedures for preparing the paint films established, we turned to developing the procedures for preparing the actual test materials. Each individual test unit was to consist of a combination of a substrate, one or two layers of paint films, and then overcoats of oil-based and/or water-based paints; in this paper, each is referred to as a “coupon.”

Substrate Development and Preparation

As noted, the substrate materials selected and prepared included metal, wood, plaster, and masonry. Fig. 3 shows the final versions of each of the four substrate materials.

The wood substrate pieces were cut from hard New Zealand Select Pine (Claymark Brand) into sections 9 cm x 10 cm x 1.2 cm. The metal substrate pieces were cut from metal sheet from the Paul N. Gardner Co. Inc. (Gardco), Pompano Beach, FL; the substrate pieces were cut to be 4 in x 3.5 in (9 cm x 10 cm). The bottom of each metal piece was sprayed with Rust-Oleum Gray #7582 metal primer (Rust-Oleum, Vernon Hill, IL) to prevent



Fig. 3. Photograph of each of four substrate materials identified and/or prepared for construction of diagnostic test materials.

rusting. The masonry substrate pieces were prepared from a special production from Shale Brick (Raleigh, NC), which was a half brick 7 1/2 in x 3 1/2 in x 1/2 in (18 cm x 9 cm x 1.2 cm) intended for use as a paver. These were cut in half to yield substrate pieces 3 5/8 in x 3 1/3 in x 1/2 in (9 cm x 9 cm x 1.2 cm). Plaster presented some challenges. After several different failed attempts, it was found that USG Hobby Plaster from United States Gypsum Company, Chicago, IL (Plaster.com) worked well. The plaster was cast in a pan and cut into 9 cm x 9 cm x 1.2 cm pieces. Two problems were incurred with the plaster. First, the plaster needs to be primed in order to seal the surface; if not primed, the paint films could not be attached to the plaster. Second, the plaster tended to crack after baking. The solution was to glue the back of each plaster unit to a wood backing using Goop (Eclectic Products, Inc., Pineville, LA). This adhesive and wood backing did not have any apparent effect on the results.

Preparation of Paint Films and Construction of Diagnostic Test Materials

The first diagnostic test materials were made with wood substrate. Sheets of RTI laboratory-prepared paint film were cut into 6 cm x 8 cm pieces. A small paint roller (3 in length, 1 in diameter) was used to put a thin layer of lead-free primer paint

(Behr Premium Plus oil-based primer and sealer, White No. 434 Behr Process Corp., Santa Ana, CA) on each block of wood, and then the paint film pieces were carefully laid at the center of each wood block. A glass tube or metal rod was used to smooth out the paint. These were then allowed to dry for about 2 days. If two layers of paint film were needed, then the second film was added using the primer as an adhesive. After the primer dried, and if planned, the film or films were overcoated using a small, low-knap roller with lead-free oil-based paint (Sherwin Williams ProMar200 Low VOC Alkyd Semi Gloss 6403-36590 Extra White B334-WZ251, Sherwin Williams Co., Cleveland, OH) with 2 days allowed for drying of this overcoat. A second layer of oil-based paint was added, if required, and dried and then two to four coats of a water-based lead-free paint (Glidden Evermore Semi-Gloss EM 6413, Base 3, ICI, Strongsville, OH) were added with 1 day of drying after each coat. The prepared diagnostic test materials (substrate piece plus paint film(s) plus overcoats) were then baked at 150°C for 48 hours. A completed coupon is shown in Fig. 4. Samples were taken from representative diagnostic test materials using the modified drill bit method²⁶ and analyzed using EPA Method 3051A and ICP/OES.³² It was found that the results (mg lead/cm²) for the samples from the diagnostic test materials agreed well with the values expected from the paint film, as illustrated in Table 9.

The approach used for the wood diagnostic test materials was then applied to produce the metal, masonry, and plaster diagnostic test materials. A problem encountered with the metal diagnostic test materials was that the paint bubbled during baking. It was thought that there were small amounts of solvents in the primer and paint films, though they were thought to be dry. To reduce this problem, all subsequently prepared diagnostic test materials were baked at 40°C for 24 hours before baking at 150°C for 48 hours. Detailed step-by-step instructions for paint test material preparation can be found in SOP EPA 600/R-10/070.¹⁶

Samples were taken from each type of diagnostic test material. Sampling consisted of taking two samples from opposite corners of each of three diagnostic test materials wherein these were typically the first, ninth, and eighteenth coupon prepared; thus, six samples were collected for each type. The modified drill bit method was used to collect the paint samples from the wood and plaster substrate diagnostic test materials, while the chisel method was used to collect the samples from the metal and masonry substrate diagnostic test materials.²⁵ The results of analysis of these samples are presented in Table 10. Only three types, O, Z, and AE, did not meet the quality objective of the RSD for the areal concentration (mg lead/cm²) because they were less than 10%.

Application of Lead-in-Paint Diagnostic Materials

A new lead-in-paint field analysis method that may have application by those following the RRP rule has been developed.³⁶ This method involves reaction of dissolved lead with molybdate and measurement of the reaction product by turbidity. The method was thoroughly evaluated with a variety of laboratory-prepared and real-world reference materials. Analysis of 14 samples from six reference materials with lead concentrations near 1 mg cm⁻² yielded a correlation to inductively coupled plasma-atomic emission spectroscopy (ICP-



Fig. 4. Photograph of bare wood substrate (right) and substrate with lead paint film on wood with overcoats of oil-based and water-based paint.

Table 9. Results of analysis of wood substrate diagnostic test materials

Coupon Construction on Wood Substrate	Expected Areal Lead Concentration (mg/cm ²) ^a	Mean Areal Lead Concentration (mg/cm ²), n=2 Method 3051A-ICP/OES
1 - Primer + 0.89 mg/cm ² film	0.89 ± 0.060 (6.69%)	0.87
2 - Primer + 0.89 mg/cm ² film + 0.46 mg/cm ² film	1.35 ± 0.045 (3.35%)	1.32
3 - Primer + 0.89 mg/cm ² film + 2 oil-based layers + 2 latex (water-based) layers	0.89 ± 0.060 (6.69%)	0.84
4 - Primer + 0.89 mg/cm ² film + 3 oil-based layers	0.89 ± 0.060 (6.69%)	0.85

^aExpected value based on paint film analysis values.

QC samples included:

SRM NIST 2581 (0.449%) – Method 3051A value: 0.445%

Blank – Method 3051A value: 8.13 µg

AES) analysis of 0.97, with an average bias of 2.8%.

The diagnostic materials described in this paper were tested using this new method. The paint samples were collected using a drill or chisel, the lead was extracted from the paint using a rotor/stator procedure,^{25,26} and the lead was quantified by the new turbidimetric method. The weights of the paint diagnostic material samples collected for the rotor-stator/turbidity method varied from 0.04 to 0.30 g, which reflects the variation in the complexity of the diagnostic materials (number of paint films, overlayers of paint, etc.) for a fixed-size collection area. Because of limited resources, only a few replicates could be performed. The resulting values, plotted in Fig. 5, were compared with the final concentrations generated from EPA Method 3051A extraction and ICP/OES quantitation. The rotor-stator/turbidity data either showed no statistical difference or agreed to better than ±20% for all but one diagnostic material (Type H). The plot shows excellent results for the most complex materials Types W to AE, which included all of the potential interferences in the materials' design (lead compounds, multiple layers, substrates, chemical interferences, and color interferences).

Table 10. Summary of EPA 3051A and ICP/OES results for prepared diagnostic test materials

Type Diagnostic Test Material	Number Prepared	n	Average Areal Lead Concentration (mg/cm ²)	Average Mass Lead Concentration (%)
A	18	6	Blank	Blank
B	18	6	0.341 ± 0.014 (4.11%)	0.359 ± 0.023 (6.41%)
C	18	6	0.759 ± 0.017 (2.24%)	1.018 ± 0.063 (6.19%)
D	18	6	0.955 ± 0.050 (5.24%)	1.141 ± 0.078 (6.84%)
E	18	6	1.167 ± 0.069 (5.91%)	1.369 ± 0.140 (10.2%)
F	18	6	1.917 ± 0.136 (7.09%)	1.973 ± 0.080 (4.05%)
G	18	6	0.744 ± 0.051 (6.85%)	1.021 ± 0.100 (9.78%)
H	18	6	1.196 ± 0.040 (3.34%)	0.980 ± 0.059 (6.02%)
I	18	6	0.741 ± 0.034 (4.59%)	0.851 ± 0.053 (6.23%)
J	18	6	1.115 ± 0.079 (7.09%)	0.946 ± 0.113 (12.0%)
K	18	12	0.716 ± 0.067 (9.36%)	1.555 ± 0.101 (6.50%)
L	18	6	1.147 ± 0.052 (4.53%)	1.162 ± 0.048 (4.13%)
M	18	6	0.701 ± 0.034 (4.85%)	1.463 ± 0.065 (4.41%)
N	18	6	1.164 ± 0.115 (9.88%)	1.355 ± 0.027 (1.99%)
O	18	7	0.821 ± 0.114 (13.9%)	0.421 ± 0.093 (22.1%)
P	18	7	1.322 ± 0.056 (4.24%)	0.660 ± 0.098 (14.9%)
Q	18	6	0.658 ± 0.038 (5.78%)	0.617 ± 0.088 (14.3%)
R	18	6	1.143 ± 0.085 (7.44%)	0.858 ± 0.123 (14.3%)
S	18	6	0.675 ± 0.031 (4.59%)	0.908 ± 0.143 (15.7%)
T	18	6	1.08 ± 0.07 (6.54%)	0.829 ± 0.069 (8.32%)
U	18	6	0.699 ± 0.044 (6.29%)	0.898 ± 0.059 (6.57%)
V	18	6	1.134 ± 0.098 (8.64%)	0.853 ± 0.054 (6.33%)
W	16	8	0.734 ± 0.045 (6.13%)	0.494 ± 0.062 (12.6%)
X	16	6	1.078 ± 0.061 (5.66%)	0.646 ± 0.066 (10.22%)
Y	18	6	1.061 ± 0.084 (7.92%)	0.666 ± 0.063 (9.46%)
Z	16	5	0.717 ± 0.072 (10.0%)	0.523 ± 0.033 (6.31%)
AA	16	6	1.062 ± 0.090 (8.47%)	0.825 ± 0.080 (9.70%)
AB	18	6	1.018 ± 0.044 (4.32%)	0.776 ± 0.023 (2.96%)
AC	16	6	0.696 ± 0.064 (9.20%)	0.387 ± 0.054 (14.0%)
AD	16	6	1.03 ± 0.10 (9.71%)	0.644 ± 0.050 (7.76%)
AE	18	6	0.945 ± 0.112 (11.9%)	0.641 ± 0.073 (11.4%)

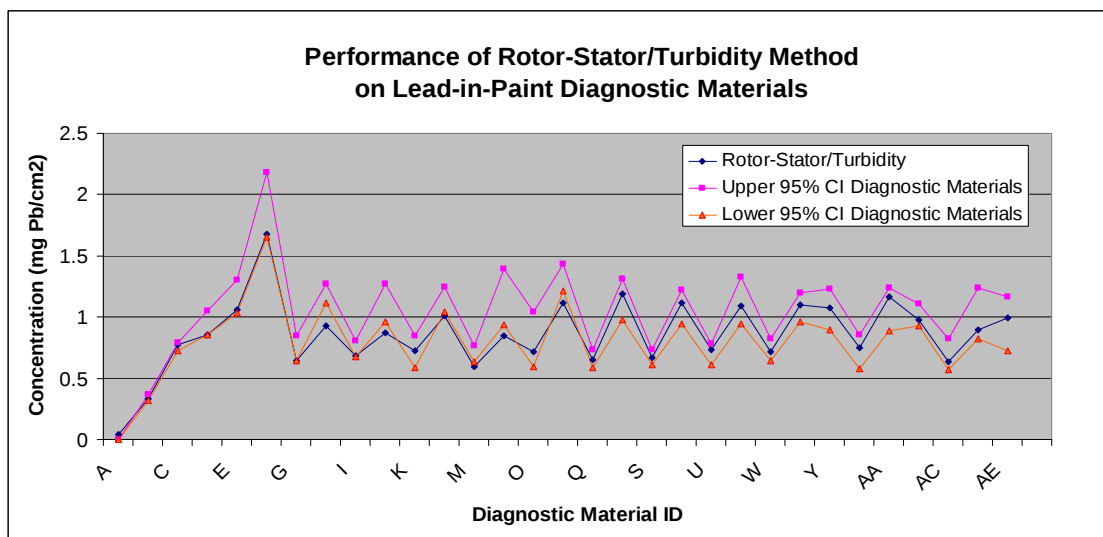


Fig. 5. Performance of rotor-stator/turbidity method with lead-in-paint diagnostic materials

Conclusions

Lead-containing paints were successfully developed from old recipes, and uniform lead-containing paint films were cast. The 31 types of lead-in-paint diagnostic test materials developed

using these films provide most of the types of analytical challenges found with real-world paints. These include variation in lead pigment and lead concentration, multiple layers of leaded and non-leaded paint, potential chemical and color interferences, and variations in substrate. Though these test materials are more

representative of real-world paints than any made previously, they do not cover all possible variables. For example, baking the paint provides brittle paint like that found in real-world paints, but developing a synthetic aging process that adequately
 5 simulates the physical/chemical changes that occur over 50+ years remains a challenge. The utility of the new diagnostic test materials has been demonstrated when used to evaluate the rotor-stator/turbidity method with potential use for the RRP rule. These materials indicate that this new method works successfully at
 10 each analytical stage—sample collection and preparation, lead extraction, and lead measurement.

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