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Research and Development & Pesticides and Toxic Substances

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# **A Report on the Lead Reference Materials Workshop**

Washington, DC  
May 13-14, 1991



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## **EXECUTIVE SUMMARY**

The potential impact on health from environmental lead has resulted in increased interest in lead exposure by Federal, State and local governmental agencies. As a result, programs committed to sampling and analysis of lead are increasing nationwide. The development of reference materials that are appropriate to the media of interest is essential to the success of these programs. Therefore the USEPA sponsored a Lead Reference Materials Workshop that was held on May 13-14, 1991 at EPA Headquarters in Washington, DC. The purpose of the workshop was to review the status of analytical methods typically used for lead analysis and to determine, as specifically as possible, the types and characteristics of reference materials that would be most appropriate to environmental lead analyses.

The workshop was attended by 33 persons with experience in lead measurement methodologies. The morning session of the workshop began with a brief overview of the purpose and goals for the meetings and was followed by presentations on the following topics:

- need for reference materials,
- health-based performance criteria, regulations and other driving forces,
- portable and laboratory X-ray fluorescence,
- atomic absorption spectrometry/plasma emission spectrometry,
- field test kits, and
- blood analysis.

In the afternoon, the attendees divided into three subgroups (atomic absorption spectrometry/plasma emissions spectrometry, X-ray fluorescence and field test kits) to discuss these specific methodologies and the reference materials needed for each. At the end of the first day, all attendees reconvened to assess subgroup progress. The next day subgroup discussions continued through the morning, and all attendees met together again on the afternoon of May 14 to present conclusions and recommendations. As a result of this effort, parameters for reference materials in paint, soil, and dust were proposed.

Parameters specified for each medium include composition, concentration levels and acceptable error, rationale for selection of levels, analytical aliquot size, and an estimated

quantity needed for 2000-3000 laboratories. Composition was generally specified as "real-world", and concentration levels were selected to reflect health effects or regulatory levels. An error margin of  $\pm 10$  percent was established for all performance evaluation standards, except for low level soil and dust samples ( $\pm 20\%$ ) and dust wipes ( $\pm 25\%$ ). Aliquot sizes of 0.1 mL for blood, 100-250 mg for paint, soil and dust (atomic absorption spectrometry and plasma emission spectrometry), and 1-2 gm for soil and dust for X-ray fluorescence were considered appropriate. Total estimated quantities of 150 liters of blood, and 20-250 kilograms each of paint, soil and dust for each concentration level were recommended.

Because specific recommendations for performance evaluation standards were made on the basis of input from experts intimately familiar with sampling and analysis of lead, the Lead Reference Materials Workshop was successful and the resulting recommendations may be used with confidence.

## **ACKNOWLEDGEMENTS**

**This document was prepared under the direction of Dr. Ben S. Lim, Exposure Evaluation Division, Office of Toxic Substances, U.S. Environmental Protection Agency, Washington, D.C. (EED/OTS), and Ms. Sharon L. Harper, Atmospheric Research and Exposure Assessment Laboratory (AREAL), U.S. Environmental Protection Agency, Research Triangle Park, NC.**

**Special acknowledgement is given to Dr. Randall J. Cramer and Dr. Joseph Breen (EED/OTS/EPA) and Mr. Michael E. Beard and Mr. Darryl J. von Lehmden (AREAL/EPA), for their careful review.**

## **DISCLAIMER**

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## **Section 1**

### **Introduction**

#### **1.1 Goals of Workshop**

Programs supporting studies of the toxicity and bioavailability of lead, environmental monitoring of lead, and abatement and clearance of lead in housing are being initiated at ever-increasing rates. Examples include the Housing and Urban Development (HUD) National Survey, the EPA Three City Study, method evaluation studies sponsored by EPA, and abatement programs carried out in Maryland, Massachusetts, and in other states. The analytical methods being used in this work include portable and laboratory X-ray fluorescence (XRF), plasma emission spectrometry (ICP), atomic absorption spectrometry (AAS), and field test kits. In most of this work, the accuracy of the analyses is uncertain because appropriate reference materials (RMs) are not available. As a result, there are questions about the comparability of studies, an essential factor in evaluating the extent of lead contamination.

To deal with this important issue, the U. S. Environmental Protection Agency sponsored a Lead Reference Materials Workshop to review the availability of, and the requirements for, reference materials for lead in environmental media. The goal of the workshop was to delineate criteria for the preparation of reference materials appropriate to the following:

- sample composition - "real-world" materials
- concentration ranges pertinent to
  - health effects
  - regulations
  - typicality of "real-world" sampling
  - instrumental limitations
- chemical and physical (e.g., particle shapes and sizes) forms
- amounts of material necessary to serve lead programs nationwide, such as potential laboratory accreditation or proficiency testing programs for lead.

#### **1.2 Structure of Workshop**

Following planning efforts by EPA, National Institute for Standards and Technology (NIST) and EPA-contractor staff, letters of invitation were mailed to 49 representatives from Federal and

State governmental agencies and a number of EPA contractors with experience in sampling and analytical methodologies applicable to lead-based paint exposure studies. The intent was to invite representatives intimately familiar with measurement methodologies in environmental media (paint, soil, and dust), and in blood, while keeping the size of the workshop manageable and productive. A copy of "Analytical Performance Criteria for Lead Test Kits and Other Analytical Methods" (Williams, Estes and Gutknecht, 1991) was sent to the invitees as a means of providing background material supportive to the goals of the workshop.

Thirty-three representatives accepted the invitation to attend the workshop. These representatives were sent a list of questions for discussion, "Principal Concerns for the Lead Reference Materials Workshop," which is presented in Appendix A. Upon arrival, attendees received a notebook containing the following information:

- Workshop Program
  - Agenda
  - List of Attendees
- Available Reference Materials
- Description of Standard Methods for Lead
- Performance Criteria and Reports
  - Tables from "Analytical Performance Criteria for Lead Test Kits and Other Analytical Methods" (Williams, Estes, and Gutknecht, 1991).
  - Chapter 4: "Guidelines for Hazard Identification and Abatement in Public and Indian Housing" (U.S. Department of Housing and Urban Development, April, 1990).
  - Tables from "Comprehensive and Workable Plan for the Abatement of Lead-Based Paint in Privately Owned Housing" (U.S. Department of Housing and Urban Development, December, 1990).
  - "A Comparison of the Results of In-Situ Spot Tests for the Presence of Lead in Paint Films using Sodium Sulfide with the Results of Laboratory Measurement of Lead Concentrations in Paint Films using Flame Atomic Spectroscopy" (Blackburn, 1990).

The workshop began with a morning session of presentations of the status of available reference materials and measurement methodologies. (See workshop agenda presented on page 1-4.) In the afternoon, the attendees met in subgroups (listed on page 1-5) to discuss specific methodologies (AAS/ICP, XRF, Field Test Kits) and reference material requirements. At the end

of the first day, all attendees reconvened to assess progress of the subgroups. The subgroups reconvened the morning of May 14, and all attendees met together again during the afternoon of May 14 to present conclusions and recommendations.

**AGENDA FOR LEAD REFERENCE MATERIALS WORKSHOP  
MAY 13-14, 1991  
EPA HEADQUARTERS, WASHINGTON, DC**

<u>Time</u>	<u>Speaker</u>	<u>Topic</u>
<b>Day 1 - May 13</b>		
9:00 a.m.	Joe Breen (OTS)	Introduction and Welcome
9:15 a.m.	Jim DeVoe (NIST)	Need for Reference Materials
9:30 a.m.	Bill Gutknecht (RTI)	Health-Based Performance Criteria, Regulations and Other Driving Forces
9:50 a.m.	Mary McKnight (NIST)	Portable XRF
10:10 a.m.	Hal Vincent (EMSL-LV)	Laboratory XRF
10:30 a.m.	Break	
10:50 a.m.	Gary Dewalt (MRI)	ICP/AAS
11:10 a.m.	Bill Gutknecht (RTI)	Field Test Kits
11:30 a.m.	Dan Paschal (CDC)	Blood Test
11:50 a.m.	Ben Lim (OTS)	Direction for Subgroup Activities
12:00 noon	Lunch	
1:00-3:00 p.m.	Subgroup Meetings to Discuss Reference Material Requirements	
3:00-4:00 p.m.	Reports from Subgroups on Reference Material Requirements	
<b>Day 2 - May 14</b>		
9:00-11:30 a.m.	Subgroup Meetings to Define and Finalize Reference Material Characteristics and Requirements	
11:30 a.m. - 1:00 p.m.	Lunch	
1:00-2:00 p.m.	Subgroup Final Reports	
2:00-4:00 p.m.	Subgroup Chairman Committee Meeting	

**Tentative Subgroup Assignments  
for  
Lead Reference Materials Workshop  
May 13 - 14, 1991  
Washington, DC**

**AAS/ICP/GFAA/ASV**

J. Chisolm	S. Guyaux
J. DeVoe*	B. Lim**
G. Dewalt	W. Loseke
M. Epstein	A. Marcus
T. Gills	P. Parsons
P. Grohse	D. Paschal
G. Guirguis	S. Roda
	E. Williams

**XRF**

M. Beard**	J. Schirmer
A. Bober	J. Simpson
S. Dillman	H. Vincent*
J. Joseph	D. von Lehmden
M. McKnight	S. Weitz
J. Neefus	

**FIELD TEST KITS**

J. Breen  
R. Cramer\*\*  
W. Gutknecht  
S. Harper\*  
M. Huang  
D. Jacobs  
L. Smith

\* Group Leader

\*\* Assistant Group Leader

## **Section 2**

### **Summary of Presentations**

Presentations were made to provide a background for the need for reference materials, the current availability of reference materials and the status of the methodologies used for lead analysis. These presentations were the basis for subgroup discussion. The presentations are summarized below.

#### **Joe Breen - Introductory Comments**

Joe Breen presented a brief overview of EPA lead-based paint programs currently in progress as technical support to HUD. He also briefly discussed the proposed Lead Exposure Reduction Act of 1991 introduced by Senator Reid, Bill No. S.391, and its potential impact on EPA/NIST responsibility for the preparation of lead reference materials. He then introduced the speakers as follows:

#### **Jim DeVoe/NIST - "The Need For Reference Materials."**

Aspects of analytical chemistry quality assurance (ACQA) were discussed. Two "components" of ACQA are determination of analytical variability and systematic error. These components ensure consistency between labs and thus, reliability for decision making.

ACQA activities are generated through the use of two types of reference materials (RMs): primary reference (standard reference materials, SRMs), and performance evaluation (PE) standards. The characterization of the PE standards is less stringent and therefore the preparation is less costly than that for SRMs; thus, PE standards can be regenerated frequently. Because method development and intercomparison of methods are dependent upon the availability of reference materials, the development of methods and RMs is clearly an interactive process.

The requirements and activities of reference material laboratories were discussed in the light of the following:

- a high level of ACQA,
- preparation and distribution of PE standards,

- coordination and evaluation of round robin analysis results,
- assistance to production lab in ACQA improvements, and
- coordination of workshops on method development and requirements for RMs.

The question of privatization of RM preparation was raised. Jim DeVoe responded in the affirmative, but added that the activity should be heavily supported by government agencies. RM design considerations were discussed. These included: (1) purpose and requirements; (2) type; (3) concentrations (e.g., values should bracket real sample values); (4) required total uncertainty; (5) quantity; (6) distribution; and (7) cost.

As a rule, uncertainty in an SRM is 5 to 10 times less than the uncertainty usually established for a PE standard. The most important criterion for a PE standard is well-characterized homogeneity, so that each laboratory that uses the standard can analyze the same composition. Overall accuracy of the measurement follows from the measurement of SRMs.

Fabrication considerations include:

- availability on a continuing basis,
- stability,
- homogeneity,
- drying studies,
- packaging, and
- evaluation of prototype.

The existing SRM for lead in paint (NIST SRM 1579) was produced in 1973, and has a lead concentration of 11.87% (118,700 ppm). This level of lead is approximately 5 to 10 times greater than a concentration appropriate to current abatement standards.

Dr. DeVoe indicated that primary reference standards for lead in the following media were in preparation:

- lead in bovine blood,
- lead in powdered paint, and
- lead in soil.

A new SRM for lead in paint film is being planned. Currently there are no PE standards available for any of the principal media.

## **William Gutknecht/RTI - "Health-Based Performance Criteria, Regulations and Other Driving Forces."**

Currently regulations for lead in environmental media are based upon the following:

- instrumental limitations,
- practicality for clearance, and
- health effects.

Federal and State regulated levels for paint, as well as clearance levels for dust and levels in soil considered to be hazardous, are given in Table 2-1.

The need for reference materials was underscored by the estimations of the numbers of homes requiring testing/abatement. According to the HUD document, "Comprehensive and Workable Plan for the Abatement of Lead-Based Paint in Privately Owned Housing" (Department of Housing and Urban Development, December, 1990), more than 57 million dwellings in the U.S. have lead in paint in excess of the abatement level, while the number of dwellings with lead in dust in excess of the Federal clearance guidelines was reported to be greater than 10 million.

Finally, the limited number of different kinds of reference materials available was described.

In summary, the driving forces which would affect the development of new reference materials were listed as:

- levels related to health effects
- levels related to regulations
- precision requirements
- large number of dwellings to be tested
- current lack of reference materials.

## **Mary McKnight/NIST - "Portable XRF."**

The existing instrumentation was discussed including the Warrington, Princeton Gamma Tech (PGT) and Scitec models. Dr. McKnight noted that the Warrington and PGT models were filter instruments while the Scitec model was a spectrum analyzer. It was noted that other models were exhibited at the 1991 Pittsburgh Spectroscopy Conference.

The fundamentals of portable XRFs were briefly discussed, and the problems of variability



## Table 2-1. Concentrations Of Concern For Lead In Environmental Media

Medium	Concentration	Regulatory Agency	Regulatory Guideline	Rationale
Paint	1.0 mg/cm <sup>2</sup>	HUD	Abatement	Instrumental limitation
	5000 µg/g	HUD	Abatement	
	600 ppm	CPSC	"New" Paint	Impurity Level
Dust	200 µg/ft <sup>2</sup>	HUD	Clearance - floor	Health, clearance practicality
	500 µg/ft <sup>2</sup>	HUD	Clearance - window sill	Clearance practicality
	800 µg/ft <sup>2</sup>	HUD	Clearance - window sill	Clearance practicality
Soil	500-1000 ppm	CDC	Hazardous level; contributes to elevated PbB	Health

of results due to substrate composition and variability among instruments were noted.

The current HUD paint film standards prepared by NIST contain lead chromate, lead sulfate and lead molybdate and have total lead concentrations of 0.6, 1.5 and 3.0 mg lead/cm<sup>2</sup>. Proposed specifications for new film standards with concentrations of nominally 0.0, 0.3, 1.0, 1.5 and 5.0 mg lead/cm<sup>2</sup> were presented. These films were projected to be available in a year, to have a coefficient of variance of 2-3 percent, and to be durable in warm, humid environments.

### **Harold Vincent/EPA EMSL - Las Vegas - "Laboratory XRF."**

The fundamentals of XRF were discussed. Most laboratory XRFs will not excite and cause K-shell emission (approximately 100 KeV, about 7 times the energy of L-shell emission). Therefore, laboratory XRFs are typically set to monitor L-shell emission frequencies. Other issues such as (1) the ratio of the intensity of line to Compton Scatter, (2) effect of particle size, and (3) accuracy versus precision were discussed.

The detection limits stated for portable (i.e., hand-held) instruments were described as 50-500 ppm and 5-10 ppm for "mobile" laboratory instruments (those that can be set up in a trailer).

RM parameter considerations were described as: (1) homogeneity; (2) particle size; (3) film thickness; (4) spiking versus natural; (5) site-specific versus site typical; (6) density; and (7) chemical composition.

The effect of sample thickness was also discussed. A 2 mm x 30 mm disk, approximately 1.4 g, was considered the optimum configuration for XRF measurement. Problems (i.e., a sample that is too thin to "capture" all exciting X-rays) are associated with using less than 1.4 g.

XRF analysis of dust samples was briefly discussed. The dust matrix was described as being similar to soil, but not to paint. The required quantity of dust for XRF was suggested to be 400 mg, but it was noted that it was often difficult to obtain a 400 mg dust sample.

The uses of RMs for support of XRF analyses were summarized as follows: (1) instrument calibration (and check); (2) traceability; (3) diagnostics; (4) quality laboratories; (5) quality sample delivery groups; and (6) audits.

## **Gary Dewalt/MRI - "ICP/AAS."**

Fundamentals of ICP and AAS (flame and furnace) were discussed with respect to development of reference materials. Interferences present for these instrumental techniques fall into three major classifications: physical, chemical and spectral interferences. Physical interferences, occurring primarily in the inlet systems of these instruments, can be controlled through matrix matching of calibration standards and samples. Chemical interferences, though common to graphite furnace AAS, are not generally encountered in ICP. Control of these interferences is generally performed through addition of matrix modifiers to digested samples. Spectral interferences, which are most significant for ICP analysis, are controlled using a variety of background correction and spectral overlap correction methods. Reference materials should be matrix-matched as closely as possible to real-world samples in order to demonstrate that analysis data are not affected by the presence of these interferences.

Available digestion methods were summarized. These included a variety of wet and dry digestion methods, as well as microwave methods.

Accuracy and precision of various AAS/ICP methods were discussed. In the absence of any other quality control data for a group of samples, it was suggested that accuracies of 10 percent for ICP and flame AAS measurements, and 20 percent for graphite furnace AAS measurements, are expected for general analysis laboratories. These values were considered to be high by some attendees.

## **William Gutknecht/RTI - "Field Test Kits."**

The results of evaluation of five commercially available spot tests were presented. Four of these are based on the reaction of lead with rhodizonate to form a pink color, while the fifth is based on the reaction of lead with sulfide to form the dark lead sulfide. These test kits are:

	<b>Rhodizonate Based</b>	<b>Sulfide Based</b>
• LeadCheck (Hybrivet Systems)	X	-
• Verify LeadTest (Verify, Inc.)	X	-

• Frandon Lead Alert (Frandon Interprises)	X	-
• Merck EM Quant (EM Science)	X	-
• The Lead Detective (Innovative Synthesis Corp.)	-	X

The principle conclusion of the study is that the kits are inaccurate because they solubilize very little of the total lead available. Other conclusions of the study are as follows:

- Kits are sensitive, responding to  $<1 \mu\text{g Pb}^{+2}$ ;
- High salt concentrations cause negative interference;
- Dark colors of samples mask Lead Detective (sulfide) results;
- There is poor sensitivity with "real-world" dust and soils;
- There is adequate stability of color; and
- Numerous problems are associated with usage by non-technical staff.

### **Dave Jacobs/Georgia Tech - "Field Test Kits/Lead Paint."**

Dave Jacobs distributed "A Preliminary Evaluation of Commercially-Available Lead-Based Paint Field Test Kits," (Jacobs, 1991) that described an evaluation of the feasibility and utility of a series of lead-based paint field test kits. The Georgia Tech study used paint film standards with concentrations of nominally 0.0, 0.1, 1 and 4 mg lead/cm<sup>2</sup>. It was noted that the calcium sulfate in plaster substrates provided a significant interference. False negatives were far more prevalent than false positives.

### **Dan Paschal/CDC - "Blood Test."**

Dan Paschal described the determination of blood lead levels at the Centers for Disease Control (CDC). Analyses are carried out using graphite furnace atomic absorption spectrometry using either deuterium or Zeeman effect background correction. Interference from NaCl, common to biological samples, is minimized by dilution.

Dr. Paschal indicated the new CDC level of concern as 10-15  $\mu\text{g/dL}$ .

## **Section 3**

### **AAS/ICP**

#### **3.1 Introduction**

Lead reference materials development for atomic spectroscopic measurements were discussed in the context of the following considerations/driving forces:

1. Range of lead concentrations typically found
2. Lead concentrations related to health effects and regulations
3. "Real-world" source vs synthetic composition
4. Matrices - physical form of sample (e.g., powder, chip)
5. Matrix compounds/interferences that must be considered
6. Reference material quality level - primary standard or performance evaluation standard
7. Cost related to reference material level (primary standard vs performance evaluation standard)
8. Quantity requirements
9. Stability requirements

Analytical methodology, discussed from the standpoint of quantity required for analysis, was considered a secondary driving force. The nature of "real-world" samples, health effects, and regulations were considered to be the principal driving forces for the preparation of proposed performance evaluation standards.

Several questions were raised. These included:

1. How will concentrations be verified?
2. What should be the uncertainty values?
3. Who should assume responsibility in development and marketing?

With these considerations in mind, each matrix type was discussed.

#### **3.2 Blood**

Biologically bound lead was considered necessary. Currently, blood reference materials are

prepared from blood samples collected from cows orally dosed with lead nitrate. These samples seem to provide an appropriate matrix. Levels of lead in blood standards (provided by dosing animals with varying concentrations of lead nitrate) were proposed to reflect CDC levels of concern, 5-25 µg/dL, and the OSHA limit. The possibility of using freeze-dried blood was discussed. The stability for lyophilized blood was reported to be 3 years, as opposed to 5 years for frozen whole blood. Proposed levels are given in Table 3-1.

The quantity of blood specified for each unit of reference material should allow multiple determinations. A quantity of 3 mL was considered to be suitable for graphite furnace atomic absorption spectrometry (GFAAS) and anodic stripping voltammetry (ASV) determinations. It was noted that CDC has historically provided reference materials at no cost to requesting laboratories. The estimated cost per set of proposed reference materials would be about \$100, with the cost absorbed by the CDC or through a CDC/EPA interagency agreement.

### **3.3 Paint**

The current methodology for determination of lead-in-paint samples was briefly discussed. Most analytical problems associated with lead determination in paint appear to be dependent upon the extractability of the lead species. Current methods for lead extraction include:

1. Dry ashing followed by wet digestion with  $\text{HNO}_3$  or  $\text{HNO}_3/\text{H}_2\text{O}_2$
2. Wet digestion only with  $\text{HNO}_3$  or  $\text{HNO}_3/\text{H}_2\text{O}_2$
3. Microwave "digestion" with  $\text{HNO}_3$ ,  $\text{HNO}_3/\text{HCl}$ , or  $\text{HNO}_3/\text{H}_2\text{O}_2$ .

The sensitivity of flame AAS and ICP is appropriate to the determination of lead in the resulting digests using an analytical aliquot size of 250-mg. In order for a 250-mg aliquot to be representative of the bulk sample, it was proposed that the material be ground to a particle size equal to or less than 200 microns. It was believed that old paint (paint that has been removed from houses at least 40 years old and has been subjected to "weathering" effects) should be used since its lead extractability properties are different from new paint; e.g., post - 1978 paint containing less than 600 µg/g lead, as required by the Consumer Product Safety Commission (CPSC). The max-

**Table 3-1. Proposed Lead Reference Standards  
For AAS/ICP**

					Analytical		Quantity	
	Medium	Composition	Levels	Error, %	Rationale	Aliquot Size	Ref. Mat'l Amnt./Unit	Projected (Units)
3-3	Blood	Bovine - "Biologically bound" lead.	4-5	$\mu\text{g/dL} \pm 10$	Minimum measured level.	0.1 mL	3 mL per level	50,000
			10	$\mu\text{g/dL} \pm 10$	New defacto action level.			
			20	$\mu\text{g/dL} \pm 10$	Approximates past CDC action level (25 $\mu\text{g/dL}$ ).			
			40	$\mu\text{g/dL} \pm 10$	Mandatory OSHA limit.			
			60	$\mu\text{g/dL} \pm 10$	Upper levels ~100.			
	Paint	"Real world," old paint obtained from housing units $\geq$ 40 yrs. old from 3-4 locations. Interferents such as organics, Cr, and Ti should be present	500	$\mu\text{g/g} \pm 10$	Approx. CPSC limit.	250 mg	35 g per level	5000
			5000	$\mu\text{g/g} \pm 10$	Current abatement level.			
			50,000	$\mu\text{g/g} \pm 10$	Levels generally far lower than current NIST 1579 (11.87%)			
	Soil	"Real-world" compo- site from 3-4 cities; gathered near roof driplines.	20	$\mu\text{g/g} \pm 20$	Lower observable limit for rural area.	250 mg	50 g per level	5000
			1000	$\mu\text{g/g} \pm 10$	Upper limit CDC protective level.			
5000			$\mu\text{g/g} \pm 10$	(Dewalt, 1991)				
Dust	"Real world" composite from 3-4 cities	50	$\mu\text{g/g} \pm 20$	(Roda, 1991)	100 mg	10 g per level	5000	
		500	$\mu\text{g/g} \pm 10$					
		10,000	$\mu\text{g/g} \pm 10$	(Dewalt, 1991)				

**Table 3-1 (Cont'd). Proposed Lead Reference Standards  
For AAS/ICP**

<u>Medium</u>	<u>Composition</u>	<u>Levels</u>	<u>Error, %</u>	<u>Rationale</u>	<u>Analytical Aliquot Size</u>	<u>Ref. Mat'l Amnt./Unit</u>	<u>Quantity Projected (Units)</u>
Dust (wipe)	Above sample deposited on wipe, wipe folded and submitted for analysis	50	$\mu\text{g/g} \pm 25$	Low level encountered for ASV analysis of hand wipes (Roda, 1991)	100 mg	4 g per level	5000
		500	$\mu\text{g/g} \pm 10$				
		10,000	$\mu\text{g/g} \pm 10$				



imum lead content allowed by CPSC, 0.06 percent (600 µg/g), is based upon expected impurities in the paint components, rather than lead additives. Other than variations in extractability, analytical errors may result from ICP spectral interferences from metals such as titanium or chromium. It was suggested that data for the concentrations of major and minor elements, along with expected trace element interferences, be included as a part of the reference material certification. Proposed parameters for paint are presented in Table 3-1.

### **3.4 Dust - Bulk (vacuumed)**

The composition of "typical" dust samples (organic fibers, paint, hair and other biologicals, soil, etc.) was discussed. Questions about the feasibility/difficulty in obtaining large quantities of dust were raised, and, because of this, it was proposed that a synthetic dust be prepared from a composite of soil and paint. Drawbacks to this approach were discussed. The chief concern was that the lead forms in the "synthetic" material would not exhibit realistic extraction characteristics. Tom Gills of NIST believed that it would be possible to collect adequate quantities of dust. Consequently, it was suggested that "real-world" samples be collected from several (3-4) urban sites. Typically, a minimum of 100 mg of dust is required for analysis by AAS. The workgroup proposed that 5000 bottles of performance evaluation lead-in-dust be prepared, each containing 10 g of the reference material. Proposed parameters for bulk dust are given in Table 3-1.

### **3.5 Dust Wipes**

After discussion of action levels and proposed reference material levels for lead in dust on wipes, the question of a separate standard for dust wipe samples was considered. A number of procedures for wiping a preweighed quantity of dust from a container were considered. A suggestion that a weighed dust sample aliquot be placed on a smooth surface and the surface "wiped" as quantitatively as possible was proposed. Another suggestion for digesting a preweighed dust aliquot in its original container was proposed. However, pyrex containers were believed to be too expensive; flint glass bottles containing wipes were not considered suitable for in-situ hot plate

or microwave digestions. Finally it was suggested that a weighed aliquot (50 mg - 100 mg) of the bulk dust sample be placed onto a blank wipe, the wipe folded to contain the dust, and the entire sample (wipe plus dust) digested for analysis. Proposed parameters for dust for wipes are presented in Table 3-1.

Current methods for preparation of wipe samples were summarized as the following general types:

- Ashing, wet digestion with  $\text{HNO}_3/\text{H}_2\text{O}_2$
- Ashing, wet digestion with  $\text{HNO}_3/\text{H}_2\text{O}_2/\text{HCl}$
- Leaching with  $\text{HCl}$  to simulate stomach acid

It was concluded that further studies will be required to finalize procedures for the preparation of reference materials for dust.

### **3.6 Soils**

It was suggested that soil samples for reference material preparation be collected at roof drip lines. Proposed standard levels are presented in Table 3-1. Again, a variety of extraction methods are in use. Methods mentioned included a  $\text{HNO}_3$  leaching technique, EPA/OSW Method 3050 (and similar procedures), as well as two microwave methods using combinations of  $\text{HNO}_3$ ,  $\text{HCl}$  and  $\text{H}_2\text{O}_2$ . The expected analytical aliquot size for most methods is 250-500 mg.

It was concluded that further studies will be required to finalize procedures for the preparation of reference materials for soil.

## **Section 4**

### **Test Kits**

#### **4.1 Introduction**

Test kits are available for testing for lead in solids. These kits are sensitive, but are not accurate because they extract and respond to only a small fraction of the lead in the samples. In order for the kits to be useful, the accuracy must be improved. Therefore, the objectives of the subgroups were to identify reference materials for accurate test kits, currently not marketed. It was recommended that these kits be used primarily for screening purposes. Eventual development of two types of kits was suggested. These are kits for:

- (1) the consumer, and
- (2) a trained professional.

The consumer kit would offer a preliminary analysis (warning) that lead levels in solid materials were potentially harmful. A second more rigorous kit, available to the professional only, could be used for confirmatory analysis. The professional kit would be more accurate, principally because of a more rigorous, though field-safe, method of sample extraction.

#### **4.2 Paint**

As noted, tests performed by Research Triangle Institute showed that the commercial test kits on the market are not accurate. However, Miao Huang of the Consumer Product Safety Commission reported that she has developed a test kit satisfactory for measurements at the level of 0.06 percent lead in paint on toys, furniture, ceramics and other consumer products.

The principle use of a test kit for paint on walls, ceilings, trim and other dwelling components would be to determine the need for abatement. Since the current HUD level for abatement is  $1.0 \text{ mg/cm}^2$  or  $5000 \text{ } \mu\text{g/g}$ , it was decided that one reference paint material should be provided at  $5000 \text{ } \mu\text{g/g}$ . This material would also be satisfactory for those states using  $0.7 \text{ mg/cm}^2$  and  $1.2 \text{ mg/cm}^2$  as abatement decision levels. It was suggested that the solution resulting from

dissolution of these reference materials be diluted to check samples at lower lead levels, e.g., concentrations equivalent to  $0.1 \text{ mg/cm}^2$  or below. Because some states exercise the option of 0.06 percent (the CPSC level) as an abatement level, it was decided that a paint reference material at  $600 \text{ } \mu\text{g/g}$  should also be prepared. Again, the kits could be used to check lower concentrations by dilution of the dissolved sample material. These parameters are presented in Table 4-1.

### 4.3 Dust

The appropriateness of measurement results expressed as loading ( $\mu\text{g/ft}^2$ ) vs concentration ( $\mu\text{g/g}$ ) was discussed. Joe Schirmer believed that loading gave a better correlation to exposure. Sharon Harper noted that modeling studies require lead levels to be expressed as concentration ( $\mu\text{g/g}$ ). The concentrations of prepared dust reference materials would necessarily be determined as  $\mu\text{g lead/g medium}$ .

There is not a predictable correlation between loading and concentration, and both have been used to express levels of concern. CDC indicates  $500 \text{ } \mu\text{g/g}$  as a lower level of concern for lead in soil. Rufus Chaney (1990) suggested concentrations above  $300 \text{ } \mu\text{g/g}$  to be a level of concern. Studies have indicated "clean" dwellings to have lead levels of  $114\text{-}125 \text{ } \mu\text{g/ft}^2$  (Farfel and Chisolm, 1990). HUD has adopted clearance guidelines of  $200 \text{ } \mu\text{g/ft}^2$ ,  $500 \text{ } \mu\text{g/ft}^2$  and  $800 \text{ } \mu\text{g/ft}^2$  for floors, window sills and window wells, respectively. Given these health effects and regulatory levels, it was proposed that two levels,  $300 \text{ } \mu\text{g/g}$  and  $1000 \text{ } \mu\text{g/g}$ , be used as target values for dust reference materials, as shown in Table 4-1.

### 4.4 Soil

Lead in soil may come from a variety of sources including paint, gasoline, pesticides and natural minerals. The health effects range of concern, like that of dust, has been proposed to be  $150\text{-}450 \text{ } \mu\text{g/g}$ . The CDC states that the range of concern is  $500$  to  $1000 \text{ } \mu\text{g/g}$  for soil. Thus it was proposed that reference materials of  $300$  and  $1000 \text{ } \mu\text{g/g}$  be prepared. Proposed parameters for soil are presented in Table 4-1.

**Table 4-1. Proposed Lead Reference Standards for Test Kits**

	<u>Medium</u>	<u>Composition</u>	<u>Levels</u>	<u>Error, %</u>	<u>Rationale</u>	<u>Analytical</u>	<u>Ref. Mat'l</u>	<u>Quantity</u>
						<u>Aliquot</u>	<u>Size/Unit</u>	<u>Projected</u>
4-3	Paint	"Real-world" powder and/or films composed of recast "real-world" paint.	5000	$\mu\text{g/g} \pm 10$	HUD abatement level	250 mg; 1 cm <sup>2</sup>	35 g; 40 cm <sup>2</sup>	5000
			600	$\mu\text{g/g} \pm 10$	CPSC level; abatement level for some states.	250 mg; 1 cm <sup>2</sup>	35 g; 40 cm <sup>2</sup>	5000
	Dust	"Real-world" samples collected from several cities/sites.	300	$\mu\text{g/g} \pm 10$	Approx. CDC lower level of concern for soil.	100 mg	10 g	5000
			1000	$\mu\text{g/g} \pm 10$	CDC upper level of concern for soil.	100 mg	10 g	5000
	Soil	"Real-world" samples collected from several cities/sites.	300	$\mu\text{g/g} \pm 10$	Approx. CDC lower level of concern	250 mg	30 g	5000
			1000	$\mu\text{g/g} \pm 10$	CDC upper level of concern	250 mg	30 g	5000

## Section 5

### X-Ray Fluorescence

#### 5.1 Introduction

Reference materials are needed for field and laboratory X-ray fluorescence analyses. The typical range of performance with these instruments is from 50-10,000  $\mu\text{g/g}$ . Soil samples are normally in the range of 50-600  $\mu\text{g/g}$  but may be as high as 60,000  $\mu\text{g/g}$ . Dust samples may have lead levels up to 17,000  $\mu\text{g/g}$  (Dewalt, 1991), though quantities of dust collected are typically low. Therefore, even at high lead levels, the absolute amount of lead in the sample will be low; and measurement of lead in dust requires use of the more sensitive laboratory XRF instrument.

#### 5.2 Paint Films

It was noted that NIST is currently preparing standard paint films for HUD. Approximately 2000 sets of paint films having concentrations of nominally 0.0, 0.3, 1.0, 1.5 and 5.0 mg lead/ $\text{cm}^2$  will be prepared by May, 1992. These films will be used directly for testing instrument response only. Calibration will require the use of various blank substrates behind the films or the combination of films (for calibration at higher lead levels). Instrument linearity in the range of 0-3 mg/ $\text{cm}^2$  was suggested to be appropriate. Standard paint films are expected to be durable for 10 years if handled properly. Interferences such as As and Hg may be significant. In some cases, AAS/ICP analyses may be required to confirm the presence of lead.

NIST noted that the relationship of wt/wt units to wt/area units is as follows:

$$\mu\text{g/g (ppm)} = (\text{mg}/\text{cm}^2)(1/t)(1/\rho)(10^3)$$

where  $t$  = thickness of film layer in cm

where  $\rho$  = density of matrix in  $\text{g}/\text{cm}^3$

Therefore 1 mg lead/ $\text{cm}^2$  corresponds to 5000  $\mu\text{g/g}$  or 0.5% (w/w) lead in a 40 mil paint film (7 layers) assuming that  $\rho$  for paint is 2 g paint per  $\text{cm}^3$ .

Reference materials with known isotope ratios were discussed, and it was proposed that, while these materials are useful for research, they may be beyond the scope of this task force.

Parameters are given in Table 5-1.

### **5.3 Soils**

The expected performance range for soil samples was described as 50-10,000 µg/g. Several standards are needed to determine the response curve. It was noted that lead levels in field samples may reach 60,000 µg/g; other techniques, such as atomic absorption, could be used for samples with concentrations exceeding 10,000 µg/g. Particle sizes may vary and be as high as 250 microns. It was suggested that several matrices would be needed and that Rufus Chaney of the USDA be consulted to determine the appropriate matrix categories. Proposed parameters based on available information are presented in Table 5-1.

### **5.4 Dust**

Lead-in-dust levels in potential reference materials were described as being similar to soils (50-10,000 µg/g). The matrix for such standards was described as being sufficiently variable and complex so as to require further study of its physical and chemical characteristics. Because of small sample sizes and sensitivity requirements, it was noted that analysis of dust samples can presently be performed using laboratory XRF instruments only. Proposed parameters based on available information are presented in Table 5-1.

**Table 5-1. Proposed Lead Reference Standards for XRF Analysis**

	<u>Medium</u>	<u>Composition</u>	<u>Levels</u>	<u>Error, %</u>	<u>Rationale</u>	<u>Analytical Aliquot Size</u>	<u>Ref. Mat'l Size/Unit</u>	<u>Quantity Projected (Units)</u>
	Paint Film	New paint films	0.3 mg/cm <sup>2</sup>	± 10	Covers HUD and state abatement decision levels	3" x 4" film	Each	Minimum of 3000 - 4000 films, reusable
			1.0 mg/cm <sup>2</sup>	± 10	HUD action level			
			1.5 mg/cm <sup>2</sup>	± 10				
			5.0 mg/cm <sup>2</sup>	± 10	Values up to 20 mg/cm <sup>2</sup> have been observed			
5-3	Soils	"Real-world" samples. Particle size - 250 microns.	50	µg/g ± 10	50 µg/g detection limit for lab XRF	1-2 g for lab XRF	5-10 g per level	5000
			10,000	µg/g ± 10	Maximum field sample 60,000 µg/g			
	Dusts	"Real-world" samples. Possible "real-world" soil/dust composite?	50	µg/g ± 10	---	1-2 g for lab XRF	5-10 g per level	5000
			10,000	µg/g ± 10	17,000 has been observed (Dewalt, 1991)			



## **Section 6**

### **References**

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Dewalt, G., Personal Communication at Lead Reference Materials Workshop, May, 1991.

Farfel, M.R. and Chisolm, J.J., "Health and Environmental Outcomes of Traditional and Modified Practices for Abatement of Residential Lead-Based Paint", American Journal of Public Health, 1990, 80(10):1240-1245.

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# **APPENDIX A**

List of  
"Principal Concerns  
for the  
Lead Reference Materials Workshop"  
Sent to  
Potential Attendees

# **PRINCIPAL CONCERNS FOR THE LEAD REFERENCE MATERIALS WORKSHOP**

## **I. CONCERNS WITH METHODOLOGY**

- A. What is the status of each of the principal methodologies (XRF, AA, ICP, ASV) for determination of lead in paint, soil and dust? Accuracy? Precision? Limit of detection?
- B. What are specific problems for each method? Sample homogenization? Sample dissolution?
- C. What advances are needed for these existing methodologies and what advances are expected in the near future?
- D. What is the availability of the methods in laboratories around the country?
- E. What are the relative analysis costs and throughput?
- F. How are we to accommodate emerging measurement technologies, e.g., biomarker field kits?

## **II. CONCERNS WITH REFERENCE MATERIAL DEVELOPMENT**

- A. Identification of the driving forces for reference material development:

- Driven by proposed performance criteria?
- Driven by adverse health effects?
- Driven by real-world sample characteristics?
- Driven by regulations?
- **SHOULD NOT BE DRIVEN** by analytical methodology?

**B. General concerns with reference material selection:**

- What are the "normal" real-world concentration ranges? The table following this page should be filled in as a guide in determining compatibility of performance criteria, real-world concentrations and methodologies
- Variation in lead concentration, lead speciation, matrix (paint, dust, soil) composition with geographic source, urban vs. rural source, private vs. public housing, age of housing?
- Should the reference materials be blended from a variety of sources so as to be "universal"? That is, how do we assure that the materials are "representative" of the real world?
- How many different concentrations should be prepared?

- How should the concentrations be verified? What would be acceptable values for the ranges of uncertainty for these materials?
- How much material should be prepared?
- What is the role of NIST SRM's and EPA's Reference Materials Repository?
- What is the role of the private sector in development and marketing?

**C. Paint - Specific concerns with reference material selection:**

- How should the material be collected?
- What should the physical form be? Films? Chips? Powder?
- How should the material be homogenized?

**D. Dust - Specific concerns with reference material selection:**

- What kind of dust should be collected? House dust? Street dust?

- How should the reference material dust be collected when many kinds of dust collection methods (i.e., vacuum, hand-press, foam roller, wipe) are being used?
- How should the dust be prepared? Filtered? Homogenized?

**E. Soil - Specific concerns with reference material selection:**

- What kinds of soil should be collected? Dwelling drip-line soil? Playground soil? Soils adjacent to streets and highways? Landfill soils?
- How should the soil be collected? Digging? Scrapping?
- How deep should the soil be taken?
- How should the soil be prepared? Filtered? Homogenized?

## Reference Material Requirements

- A. Concentration in range of performance criteria
- B. Concentration in range of field samples
- C. Concentration in linear range of instrumentation
- D. Compensation for interferences found in sample matrix

	Field Kits	AA/ICP/GFAA	XRF	Bio Methods
Dust				
A.				
B.				
C.				
D.				
Soil				
A.				
B.				
C.				
D.				
Paint				
New/Replacement				
A.				
B.				
C.				
D.				
Abatement Std.				
A.				
B.				
C.				
D.				

# **APPENDIX B**

## **List of Attendees**



## LEAD REFERENCE MATERIALS WORKSHOP

May 13 - 14, 1991

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