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Pb-Based Paint Laboratory Operations Guidelines: Analysis of Pb in Paint, Dust, and Soil

Revision 1.0

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Pb-Based Paint
Laboratory Operations Guidelines:
Analysis of Pb in Paint, Dust, and Soil

Revision 1.0

Technical Programs Branch
Office of Pollution Prevention and Toxics
U. S. Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

John V. Scalera, Work Assignment Manager
Janet C. Remmers, Project Officer

May 14, 1993

DISCLAIMER

This document has been reviewed and approved for publication by the Office of Prevention, Pesticides, and Toxic Substances, U.S. Environmental Protection Agency. The use of trade names or commercial products does not constitute Agency endorsement or recommendation for use.

NOTICE

This guide is limited to the analysis of Pb in paint film or chips, Pb-based paint contaminated soils, and deposited dust (vacuum dust and wipe samples). There are many programs covering Pb in other matrices, such as air (occupational and environmental), drinking water, eating utensils, solid waste, hazardous waste, gasoline, and blood. Separate regulatory guidance is already in place for Pb in these matrices. This guide provides needed information for laboratory chemists and managers in laboratories that seek accreditation for Pb in paint, soil, and deposited dust matrices. The guideline also provides recommendations for good laboratory practices for these laboratories.

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AUTHORS AND CONTRIBUTORS

Midwest Research Institute (MRI) was requested by the Environmental Protection Agency (EPA) to develop a good laboratory practices guideline for laboratory chemists. This Laboratory Operations Guideline provides guidance to laboratories seeking accreditation by organizations participating in the EPA National Lead Laboratory Accreditation Program (NLLAP). The NLLAP recognizes laboratories which demonstrate, through proficiency testing and systems audits, the capability to analyze for Pb in paint, soil, and deposited dust, including vacuumed dust and wipe samples.* The guideline was developed with the cooperation of the Technical Programs Branch, Office of Pollution Prevention and Toxics (OPPT) under EPA Contract 68-DO-0137. A working meeting to gather information for the basis of this report was held in Gaithersburg, Maryland, from June 24 to 26, 1992, with a group of metals laboratory experts. The affiliations of the working group and the role of each organization are presented below.

MIDWEST RESEARCH INSTITUTE (MRI)

MRI was responsible for the planning and the conduct of the working meeting. Dr. Larry Lowry, the Work Assignment Leader, had primary responsibility for conducting the meeting and writing the final reports with input from working group members and Mr. Paul Constant, Dr. Gary Dewalt, and Mr. Jack Balsinger from MRI.

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* In order to avoid confusion in the terms lead (for Pb) and lead (for leader), the following conventions are used. Pb will be used for the heavy metal. Lead will be used for all other uses. The term, good laboratory practices, will not be used here since it refers to the EPA/TSCA GLP standards. The preferred term is Laboratory Operations Guidelines (LOG). The term, paint, in this document refers to dried paint film or paint chips and not to liquid, uncured paint.

WORKING GROUP PARTICIPANTS

The following individuals actively participated in the working meeting and in the review of this document. Their affiliations and the organizations they represent are listed.

` Joseph J. Breen, Ph.D.
Chief, Industrial Chemistry Branch
Economics, Exposure and Technology Division
Office of Pollution Prevention and Toxics
U.S. Environmental Protection Agency
401 M Street, SW
Washington, DC 20460

` Michael S. Epstein, Ph.D.
National Institute of Standards and Technology
B-220 Chemistry Building
Gaithersburg, MD 20899

Research chemist in the Inorganic Analytical Research Division of the Chemical Science and Technology Laboratory. Currently responsible for Atomic Absorption (AA) and DC Plasma Emission Spectrometry, he has 15 years of experience in AA and plasma emission, including the analysis of Pb paint film materials. He helped to coordinate the certification of SRMs for soils and sediments.

` Mr. Lee Friell, M.S.
Manager, Iowa Hygienic Laboratory
Henry A. Wallace Building
Des Moines, IA 50319

Represents the Association of State and Territorial Public Health Laboratory Directors (ASTPHLD) and state laboratory accrediting bodies. An inorganic chemist with experience in Inductively Coupled Plasma Emission Spectrometry (ICP-AES) EPA work on soil and water. His laboratory is a CLP-certified laboratory that conducts Pb analyses in many different matrices.

` Mr. Fred Grunder
Manager, Laboratory Accreditation Program
American Industrial Hygiene Association
2700 Prosperity Avenue, Suite 250
Fairfax, VA 22031

Site assessor and member of the American Industrial Hygiene Association (AIHA) Environmental Lead Laboratory Accreditation Committee (ELLAC).

` Larry K. Lowry, Ph.D.
Senior Advisor, Exposure Assessment
Midwest Research Institute
425 Volker Blvd.
Kansas City, MO 64110

Work Assignment Leader (WAL) and principal author of the report.

` Mr. John V. Scalera
Technical Programs Branch,
Chemical Management Division,
Office of Pollution Prevention and Toxics
U.S. Environmental Protection Agency
401 M Street, SW
Washington, DC 20460

Work Assignment Manager (WAM) and principal EPA reviewer.

` James Scott, M.S.
Georgia Power Company
Environmental Laboratory
5131 Maner Road
Smyrna, GA 30080

A lead assessor for the American Association for Laboratory Accreditation (A2LA).

` Barbara J. Weaver, C.I.H.
Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601

Represents the laboratory director, Dr. Wilson Hershey, the American Council of Independent Laboratories (ACIL) and the Committee on National Accreditation of Environmental Laboratories (CNAEL).

EPA CHEMICAL MANAGEMENT DIVISION, TECHNICAL PROGRAMS BRANCH (CMD/TPB)

EPA was responsible for oversight in the development of the study plan; managing and coordinating the conduct of the overall project; and reviewing, editing, and finalizing this report. Key staff included Mr. John V. Scalera, Work Assignment Manager; Janet C. Remmers, Project Officer; and Dr. Joseph J. Breen, former Project Officer.

EXTERNAL REVIEWERS

The following individuals served as external reviewers of this document. Their participation in this review is gratefully acknowledged.

Kevin Ashley, Ph.D., NIOSH, Cincinnati, OH.
Daniel Paschal, Ph.D., CDC, Atlanta, GA.
Barbara J. Weaver, C.I.H., Lancaster Laboratories, Lancaster, PA.
Michael S. Epstein, Ph.D., NIST, Gaithersburg, MD.

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CONTENTS

EXECUTIVE SUMMARY

The hazards of Pb-based paint have become a leading public health issue of the 1990s, and Pb-paint abatement of homes is a high priority among many different health and environmental organizations. The Environmental Protection Agency (EPA) estimates there are several hundred laboratories, not currently performing analysis, that will be involved in new, extensive Pb-abatement programs.

Following the lead of the Interagency Lead-Based Paint Task Force (U.S. EPA, 1992), the EPA Office of Pollution Prevention and Toxics (OPPT) is establishing the National Lead Laboratory Accreditation Program (NLLAP). The NLLAP will provide federal oversight for state and private sector laboratory accreditation programs involved in the accreditation of laboratories analyzing paint, soil, and dust samples associated with the abatement and control of Pb-based paint contaminated housing. The NLLAP will recognize accrediting organizations that meet NLLAP minimum requirements through a Memorandum of Understanding (MOU). Each NLLAP-recognized accrediting organization will administer its laboratory accreditation program under NLLAP oversight. In order to be recognized by the NLLAP, laboratories must meet the following criteria:

- The laboratory must successfully undergo a systems audit inclusive of an on-site assessment by an analytical laboratory accrediting organization recognized by EPA through an MOU.

- The laboratory must successfully participate in the Environmental Lead Proficiency Analytical Testing (ELPAT) program.*

The purpose of this Laboratory Operations Guide is twofold: to provide information for **laboratory chemists** performing analysis for Pb in paint, soil, and

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* The Environmental Lead Proficiency Analytical Testing (ELPAT) Program is a cooperative effort to improve and evaluate the performance of laboratories involved in the analysis of Pb in paint, dust, and soil matrices. The National Institute for Occupational Safety and Health (NIOSH) performs ELPAT data analysis under a Memorandum of Understanding (MOU No. PW593570-01-0) with the U.S. Environmental Protection Agency (EPA). The American Industrial Hygiene Association (AIHA) contracts for ELPAT sample production and administers the ELPAT program as permitted under a Cooperative Research and Development Agreement (CRADA No. NIOSH-92-1) with NIOSH covering cooperation in analytical research and proficiency test programs.

deposited dust, including wipe samples and vacuumed dust; and to assist those laboratories seeking accreditation.

The guideline was prepared following a working meeting of experts in metals analysis from government and independent laboratories. It begins with a general overview of the collection of paint, soil, and deposited dust samples so that the laboratory staff will have a good understanding of the types of samples that will be analyzed and will be able to provide the client with suggestions on field-sampling procedures. Then issues such as laboratory management, personnel qualifications, quality assurance, proficiency testing, analytical methods, and instrumentation, relevant to the analysis of paint, soil, and deposited dust for Pb, are discussed. Practical aspects of laboratory operations for these matrices are stressed. This guideline provides specific recommendations that should be considered for laboratories seeking NLLAP recognition for analyses of Pb in paint, soil, and dust (including wipe samples and vacuumed dust).

SECTION 1

INTRODUCTION

1.1 BACKGROUND

The hazards of Pb-based paint have become a leading public health issue of the 1990s. Pb-paint abatement of homes is a high priority among many different health and environmental organizations. The Environmental Protection Agency (EPA) estimates there are several hundred laboratories not currently performing these analyses that will be involved in new extensive Pb-abatement programs.

1.2 EPA RECOGNITION OF LABORATORIES

Following the lead of the Interagency Lead-Based Paint Task Force (U.S. EPA, 1992), the EPA Office of Pollution Prevention and Toxics (OPPT) is establishing the National Lead Laboratory Accreditation Program (NLLAP). The NLLAP will provide federal oversight for state and private sector laboratory accreditation programs involved in the accreditation of laboratories analyzing paint, soil, and dust samples associated with the abatement and control of Pb-based paint contaminated housing. The NLLAP will recognize accrediting organizations that meet NLLAP minimum requirements through a Memorandum of Understanding (MOU). Each NLLAP-recognized accrediting organization will administer its laboratory accreditation program under NLLAP oversight. In order to be recognized by NLLAP, laboratories must meet the following criteria:

- The laboratory must successfully undergo a systems audit inclusive of an on-site assessment by an analytical laboratory accrediting organization recognized by EPA through an MOU.

- The laboratory must successfully participate in the Environmental Lead Proficiency Analytical Testing (ELPAT) program. (See footnote on page xi.)

The completion of a proficiency testing-based program alone is not sufficient proof that a laboratory can perform successfully on real world samples. The proficiency testing sample, even though it is a matrix-based material, will usually receive special treatment in the laboratory. The systems audit is necessary to ensure that a laboratory has the required staff, methods, facilities, quality assurance plans, and other essentials necessary to perform the analysis within a stated level of confidence.

1.3 OTHER PROGRAMS

This guideline is limited to the analysis of Pb in paint film or chips, Pb-based paint contaminated soils, and deposited dust (vacuum dust and wipe samples). There are many programs covering Pb in other matrices. Some of these are Pb in air (occupational and environmental), drinking water, eating utensils, solid waste, hazardous waste, gasoline, and blood. Separate regulatory guidance is already in place for Pb in these matrices. The guidelines in this document are intended to avoid a duplication of efforts for existing programs.

1.4 PURPOSE

This laboratory guideline provides needed information for laboratory chemists and laboratory managers in laboratories that seek accreditation for Pb in paint, soil, and deposited dust matrices, including vacuumed dust and wipe samples. The guideline identifies major issues that must be included in a laboratory training program. It also provides minimum recommendations for good laboratory practices in a laboratory that analyzes paint, soil, and deposited dust for Pb. The guideline should help a laboratory make an informed commitment to participate in the NLLAP. When finalized, the availability of specific quality system requirements for laboratories wishing to participate in the NLLAP will be announced in the *Federal Register.*

Quality laboratories that understand the idiosyncracies of Pb analysis are needed by federal agencies, including HUD and EPA. Data from these laboratories must be of high quality to support decisions regarding the suitability of habitation in buildings undergoing Pb abatement.

1.5 APPROACH

EPA was requested to develop a laboratory operations guideline for use by laboratories that seek accreditation in the Pb-abatement area. A working meeting of experts in metals analysis from government and from independent laboratories was convened in Gaithersburg, Maryland, on June 24 to 26, 1992. Information gathered at this meeting was compiled into this document.

This guideline includes a general overview of the collection of paint, soil, and deposited dust samples in order that the laboratory staff will have a good understanding of the types of samples that will be analyzed and can provide the client with suggestions on field-sampling procedures. The guideline then addresses many issues, such as laboratory management, personnel qualifications, quality assurance, proficiency testing, analytical methods, and instrumentation, relevant to the analysis of paint, soil, and deposited dust for Pb.

This document builds on existing laboratory guidelines, such as the TSCA GLP standards (U.S. EPA, 1989), and the various federal task forces and professional committee reports recently published on the laboratory aspects of Pb. This document includes a discussion of major issues related to Pb matrices and a glossary of acronyms and terms. An extensive reference list is also included.

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SECTION 2

FACILITIES AND PERSONNEL QUALIFICATIONS

This section gives recommendations for facilities seeking to be accredited for the analysis of paint, soil, and deposited dust for Pb, and requirements for the training and experience of laboratory personnel.

2.1 FACILITIES

These laboratory guidelines apply to fixed location, temporary, and mobile laboratories.

A laboratory should have the space, equipment, instruments, ventilation, utility services, storage, safety equipment, and manuals necessary to accomplish Pb analyses of paint, soil, and deposited dust. The facility should have restricted access for security reasons and controlled access to sample preparation areas to reduce contamination. Other recommendations are listed in the TSCA Good Laboratory Practices Standards (U.S. EPA, 1989). Minimum recommendations for metals laboratories include:

- Appropriate area for sample receipt, processing, and storage (secured, controlled temperature).

- A laboratory hood for digestion of samples that meets the requirements specified in the Industrial Hygiene Ventilation Manual of the American Conference of Governmental Industrial Hygienists (ACGIH, 1991).

- An adequate number of grounded electrical circuits that meet local electrical codes and ensure stable electrical supply to instruments and data systems. Uninterrupted power supplies may be needed in some areas to protect data systems.

- Ambient temperature and humidity control adequate to insure reliable operation of instrumentation and sample/digest stability.

- Cross-contamination control procedures to prevent sample contamination and contamination of work areas. Controlled access to sample preparation areas and other procedures to minimize sample contamination. Documentation of effectiveness of contamination control by use of surface wipe samples.

- A glassware-cleaning facility with SOPs and monitoring requirements.

- Approved procedures for disposal of hazardous waste.

2.2 PERSONNEL AND QUALIFICATIONS

The laboratory management should provide technical and quality managers who operate the laboratory in conformance with ISO Guide 25 (ISO/IEC Guide 25, 1990), NLLAP requirements, and the accrediting organization requirements. Following are the personnel and minimum qualifications that are needed.

Technical Manager, or however named

This individual should have a B.S. degree in Chemistry, or related field, with a minimum of 3 years' nonacademic laboratory experience, two of which are in metals analysis. This individual is responsible for the technical effort and should be available to the analyst at the laboratory at least 50% of the normal work day. The technical manager may also serve as the inorganic chemistry supervisor.

Quality Manager, or however named

This individual should have a B.S. degree in basic science and have at least 1 year of nonacademic analytical chemistry experience and training in statistics, or 4 years nonacademic analytical chemistry experience and training in statistics. Experience or knowledge of ISO Guide 25 is essential. The quality manager should be separated from the analytical chemistry operations. In some small laboratories, the technical manager may also function as the quality manager as long as this person is not involved in the direct supervision of the lead analyst/technician doing the routine sample analysis.

Inorganic Chemist, Spectroscopist, or however named

This individual should have a B.S. degree in Chemistry, or related field, with a minimum of 1 year in metals analysis. Training in specific metals methods used in the laboratory must be documented; proficiency in analysis must also be documented. This category includes the following persons:

- Inductively Coupled Plasma-Emission Spectroscopist
  Experience: 1 year minimum recommended (nonacademic)
  Training: Satisfactory completion of a short course on ICP-AES. An in-house training program is acceptable.

- Flameless Atomic Absorption Spectroscopist
  Experience: 1 year minimum recommended (nonacademic)
  Training: Satisfactory completion of a short course on Graphite Furnace Atomic Absorption (GFAA). An in-house training program is acceptable.

` Flame Atomic Absorption Spectroscopist
Experience: 1 year minimum recommended (nonacademic)
Training: Satisfactory completion of a short course on Direct Flame Aspiration Atomic Absorption Spectrometry (FLAA). An in-house training program is acceptable.

Analyst, Technician, or however named

Two years of technical education at the college level is recommended. This individual must have documented training in specific metal methods used in the laboratory and must have documented proficiency in performing assigned tasks. This category includes the following persons:

` Inorganic Sample Preparation Technician
Experience: 3 months minimum recommended (nonacademic)

` Routine Sample Analyst (instrumentation)
Experience: 6 months minimum recommended (nonacademic)

The above staff must have documented training on instruments specific to the laboratory and have demonstrated proficiency in these techniques. Junior staff, such as analysts or technicians, must work under the direct supervision of a degreed scientist in one of the "Chemist/Spectroscopist" categories. Junior staff may also work under the supervision of the Technical Manager, or a sample analyst/technician who has performed successfully over a period of three years in the analysis of metals, using the same technologies that will be used for the analysis of Pb-containing samples.

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SECTION 3

QUALITY ASSURANCE

Quality assurance programs are required for laboratories that analyze paint, soil, and deposited dust for Pb. The ISO Guide 25 (ISO/IEC Guide 25, 1990), the internationally accepted quality system for testing laboratories, should serve as the basis of the laboratory quality system. There are several good general references for quality assurance programs listed in the bibliography. The reference by Liabastre (Liabastre, 1992) is recommended as it addresses all aspects of quality assurance for environmental assessment laboratories. The reference by Ratliff (Ratliff, 1990) is also recommended. Information specific for laboratories that analyze paint, soil, and deposited dust for Pb is located in the HUD Interim Guidelines (HUD, 1990) and in the other various referenced publications.

3.1 THE QUALITY SYSTEM

A laboratory must have a quality system documented in a quality manual. The manual should document the policies and objectives of the quality system. The specific program requirements are found in individual accrediting organization policies. The major components of a typical quality system, which are listed below, should be addressed and documented in a quality manual and in related supporting documents. The components are listed according to ISO Guide 25 headings (ISO/IEC Guide 25, 1990).

- QA management should be directed by a full-time employee with power to oversee the situation, identify problems, and make corrections, while being independent of the analyses.

- A quality policy statement, including objectives and commitments by top management.

- Organization and management structure of the laboratory, its place in any parent organization, and relevant organizational charts.

- Relationship between management, technical operations, support services, and the quality program.

- Procedures to control and maintain documentation of the quality manual and related supporting documents.

- Job descriptions of key staff and reference to other staff.
- The introduction of new employees to the quality manual and the requirement that all employees periodically review the manual.
- A documented training program for employees that includes site-specific SOPs.
- Identification of the laboratory sign-off person for reports.
- Traceability of calibration standards to SRMs.
- Scope of the laboratory operation and services offered.
- Procedures for review of incoming work to assure adequate facilities and staff.
- Reference to the calibration, verification, and test procedures used.
- Procedures for handling calibration and test items.
- SOPs for sample log-in procedures.
- SOPs for sample preparation, including debris removal, substrate removal, drying, grinding, sieving, and mixing.
- SOPs for sample and subsample identification, including digests and extracts.
- SOPs for the preparation of working standards and calibration solutions.
- SOPs for digestion procedures, methods of analysis, and calibration procedures.
- SOPs for major equipment calibration, reference standards used, and maintenance of equipment.
- References to verification practices, including interlaboratory comparisons, proficiency testing programs, use of reference materials, and internal quality control schemes.
- SOPs for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur.
- Arrangements for exemptions permitting departures from documented policies/procedures as specified in SOPs.

- References to procedures for dealing with complaints.

- References to procedures for protecting confidentiality of results.

- SOPs for data audit and review.

- Quality system audits should be conducted to ensure that the documented quality system is implemented as written.

3.2 REVIEW OF THE QUALITY SYSTEM

The quality system requires frequent inspection and audits to ensure its effectiveness. The following are recommendations for quality system audits.

- Quality system audits should be conducted at regular intervals by trained and qualified staff to verify the system is implemented as written. Discrepancies found should be corrected, and any client whose reported data are affected should be notified in writing immediately.

- The quality system should be reviewed at least once per year by management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements.

- All audit and review findings, and any corrective actions that arise from them, should be documented by the Quality Manager and resolved in a timely manner.

3.3 QUALITY CONTROL

The quality manual and related supporting documents should contain the following sections on quality control:

3.3.1 Quality Control System

- QC procedures required by applicable federal or state environmental or public health agencies should be listed, documented, and followed.

- A sample tracking system must be maintained.

- Control chart data or equivalent should be maintained for each analytical technique. See *Handbook for SRM Users* (Taylor, 1985) for recommendations on control charting.

- Supervisory personnel should review the data calculations and QC results (internal data review).

- Deviations or deficiencies in QC should be reported to management immediately.

- A documented corrective action plan should be implemented when analytical results fail to meet QC criteria.

- QC data must be retrievable for all analytical results.

3.3.2 Calibration and Quality Checks

- Standard calibration curves should be prepared to adequately cover the expected concentration ranges of the samples and the expected "action levels" of Pb (HUD, 1990).

- Standard calibration curves should be prepared using at least three standards and one blank, unless otherwise specified by the method.

- New calibration curves should be prepared whenever out-of-control conditions are indicated and after new reagents are prepared and used.

- Method detection limits should be determined and documented (40 *CFR* 136, Appendix B).

- The laboratory should ensure the quality of results by implementing and reviewing quality checks, as appropriate, but not limited to:

  - Internal quality control charting based upon statistical techniques. This is most useful for identifying trends and out-of-control incidents.

  - Regular use of standard reference materials and certified reference materials as primary reference materials.

  - Participation in the ELPAT Pb-proficiency testing program.

  - Replicate testings using the same or different methods.

  - Re-testing of retained items as needed.

3.3.3 SOPs

- The QC procedures (SOPs) should be specific to each test technology and matrix addressing the following:

  - Reagent and method blanks

  - Glassware cleaning

- Trip and field blanks
- Sampling and subsampling
- Replicate/duplicate analysis
- Spiked and blank sample analysis
- Blind samples
- Quality control samples
- Control charts
- Calibration standards
- Reference samples and SRMs
- Internal standards

3.4 QUALITY CONTROL PRACTICES

The laboratory quality control program should include the continual evaluation of its performance (system process control) for each matrix, which includes the determination of accuracy and precision. One possible method used for laboratory system process control is the use of control charts to monitor the performance of a specific QC sample. Control charts should specify warning and action limits. In the absence of a statistically sufficient data base to determine the necessary frequency for QC samples, the laboratory should default to the use of a set frequency for QC samples stated in its analytical standard operating procedure.

Quality control practices can be broken down into those processes that are affected by the instrumentation and those that are related to the sample matrix. The following recommendations for matrix-based quality control practices applicable to AA or ICP-AES should be used in the absence of laboratory-based process control data.

3.4.1 Precision and Accuracy Determinations

Accuracy studies are performed to determine how close a measurement comes to an actual or accepted reference value. Accuracy can be expressed as percent recovery and evaluated by analysis of matrix spike samples. A matrix spike is an aliquot of a sample fortified (spiked) with a known quantity of the analyte of interest and subjected to the entire analytical procedure. The spike should be prepared from a standard stock, which is different from the calibration standard stock, and should have a Pb concentration that is within the range of the sample to be run.

Precision is evaluated by the reproducibility of analyses. Precision is commonly expressed as standard deviation or relative percent difference (RPD) and can be

evaluated by the analysis of replicate samples. Replicate sample analyses are one or more additional analyses on separate portions of a given sample in order to assist in the evaluation of method variance. Most commonly, two replicate analyses (defined as a duplicate analysis) are performed.

In the analysis of soil, dust (vacuum) and paint chip matrices, samples may be too small and difficult to homogenize and split in order to obtain samples for matrix spike evaluations or replicate analyses. For these sample matrices, the laboratory should select alternate QC options, such as the analysis of duplicate laboratory control samples per batch.

Paint chips, soil, and vacuumed dust samples

Accuracy determination. Matrix spiked samples should be analyzed with a minimum frequency of 5% of the samples for each matrix, per batch of samples (samples processed at a single time). If there are fewer than 20 samples in a batch, at least 1 spiked sample for each matrix per batch should be analyzed.

Precision determination. Replicate (duplicate) samples should be analyzed with a minimum frequency of 5% of samples for each matrix per batch of samples. If there are fewer than 20 samples in a batch, at least 1 sample for each matrix per batch should be analyzed. In the event the analyte is not detected in the sample, replicate matrix spike samples may be analyzed.

Dust wipe samples—accuracy and precision determinations

When analyzing wipe samples, method spike samples are prepared using blank collection media and analyzed with a minimum frequency of 5% of samples for each matrix per batch of samples. If there are fewer than 20 samples per batch, at least 1 method spike/spike duplicate set should be run per batch. The matrix samples are to be prepared using a Pb-based paint NIST SRM applied directly to the wipe. It is recommended that the client submit blank wipes representative of the lots to be used in the field for lead contamination analysis prior to field sampling.

3.4.2 Method Blanks

When using methods requiring sample pretreatment not performed on calibration standards, a method blank containing all reagents and subject to all preparation steps should be processed and analyzed along with the samples. Method blanks should be analyzed with a minimum frequency of 5% of the samples for each matrix per batch of samples. If there are fewer than 20 samples in a batch, at least 1 method blank for each matrix per batch should be analyzed. The use of method blanks provides a measurement of laboratory and/or reagent contamination. Method blanks are not to be used to correct sample results.

3.4.3 External Reference or Laboratory Control Sample Analysis

At least one external reference or laboratory control sample (LCS) should be analyzed with each matrix per batch of samples with a minimum frequency of 5%. If there are fewer than 20 samples per batch, then at least 1 LCS should be run per batch per matrix type. The concentration of the LCS should be within the working range of the method and should not require extensive pretreatment, dilution, or concentration prior to analysis. Sources of these samples include but are not limited to: NIST Standard Reference Materials, commercially available certified reference samples, or samples prepared from different sources of analyte than calibration standards and whose concentrations were determined using definitive methods. If available, all these reference materials should be NIST traceable.

3.4.4 Recommended QC Sample Criteria

The following recommendations for analytical instrument quality control practices should be used in the absence of laboratory-based process control data.

Acceptable performance limits for analytical instrumentation, as well as each method, should be established based upon the continuing statistical evaluation of the data generated by the analysis of quality control samples, unless specific minimum acceptance limits are established by the method. The laboratory's calculation procedures for statistically derived acceptance limits should be documented. Some methods have listed acceptance criteria for applicable analytes based upon determinations by a single laboratory, the compilation of data from many laboratories, or limits that are assumed or expected. These limits may be too broad to define accurate acceptance criteria for routine use. These limits are best used as guidelines during the initial phases of method use and are superseded when the laboratory has collected sufficient self-generated data for proper statistical evaluation.

In the absence of sufficient data for the determination of QC sample frequency and acceptance criteria, the following minimum QC sample frequencies and acceptance limits are recommended (where applicable) for analytical SOPs employing AA or ICP-AES instrumentation:

QC sample	Frequency	Acceptance limits
Initial calibration verification (ICV)	Once per run after calibration	Within ±10% of known value
Initial calibration blank (ICB)	Once per run at the beginning of run	Absolute value not more than 20% of the regulatory limit or level of concern
Continuing calibration verification (CCV)	Before and at the end of a sample run, as well as every 10 samples	Within ±10% of known value for ICP or FLAA; within ±20% for GFAA

Interference check sample (ICS)	Beginning and end of each run or twice every 8 hr	Within 20% of known value
Continuing calibration blank (CCB)	After each ICS and CCV	Absolute value not more than 20% of the regulatory limit or level of concern
Laboratory control sample (LCS)	1 per 20 samples or batch (5%)	Within ±20% of known value
Matrix spike	1 per 20 samples or batch (5%)	Within ±25% of known value
Duplicate sample	1 per 20 samples or batch (5%)	Within ±25% relative percent difference (RPD)
Method blank	1 per 20 samples or batch (5%)	Absolute value not more than 20% of the regulatory limit or level of concern

A detailed recommended analysis protocol is listed in Section 11.

SECTION 4

REQUIRED STANDARD OPERATING PROCEDURES (SOPs)

4.1 OVERVIEW

All methods, including sample collection, subsampling, digestion, and analysis, should have laboratory-generated standard operating procedures (SOPs). There are no standard methods from EPA or other organizations/agencies with published validations for the analysis of Pb in paint and deposited dust matrices. There is a standard method for the digestion of soils (U.S. EPA SW-846 Method 3050). Modifications of methods must be documented in revised SOPs. Minor modifications, for example, the use of more acid, should be specified in SOPs and include the reasons to make such adjustments. No deviations should be permitted during routine sample analysis beyond those limits specified in the laboratory SOP, but deviation within stated limits is acceptable. Guidelines for the preparation of SOPs have been published by the EPA Office of Solid Waste (U.S. EPA, 1990c).

4.2 ELEMENTS OF SOPs

SOPs for analytical methods should address the following basic elements:

- Scope and application of the laboratory method
- Summary of the method
- Definitions and abbreviations
- Interferences
- Safety considerations
- Apparatus and equipment
- Reagents and consumable supplies
- Sample collection, preservation, and storage
- Sample preparation (debris removal, substrate removal, drying, grinding, sieving, and mixing)
- Instrument calibration
- Quality control procedures (internal and external)
- Detailed step-by-step procedure
- Sample calculations
- Method performance, including accuracy and precision
- References

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SECTION 5

FIELD SAMPLING

5.1 OVERVIEW

Laboratories that perform analyses of Pb in paint, soil, and deposited dust will generally not be involved in the sample collection phase; therefore, specific knowledge of sample collection is not needed. This section provides the laboratory chemist with a general understanding of the sampling procedures so that:

- There is a better understanding of the nonuniformity of the sample that arrives at the laboratory;
- There is an understanding of the types of samples, contaminants, and debris that may arrive at the laboratory;
- The laboratory is able to recommend minimum sample sizes to the client to ensure that there is sufficient sample to meet analytical requirements, such as minimum detection limits, spike samples, and duplicate analyses; and
- The laboratory can better comply with TQM principles by knowing the entire history of the sample from collection site to report (the process).

The HUD Interim Guidelines (HUD, 1990) and the HUD Risk Assessment Protocol (HUD, 1992) provide recommended sampling protocols for the collection of paint chips, dust, soil, and airborne Pb dust.

Some laboratories may be asked to perform actual field sampling. In those cases, the HUD Interim Guidelines need to be studied thoroughly to develop the appropriate sampling strategies to comply with their directives. Laboratories that may also be asked by their clients for recommendations on sample collection need to be prepared to offer suggestions, if asked.

5.2 MINIMUM SAMPLE SIZE

The minimum sample size collected is based on the ability to detect Pb at the action level recommended in the HUD Interim Guidelines (HUD, 1990) with a defined degree of confidence. There are no federal standards at present, but the HUD

"clearance" guidelines (Section 10.4.3) provides the following guidance for **dust** collected with a wipe sample:

- 200 µg Pb/sq ft for floors (includes carpet)
- 500 µg Pb/sq ft for window sills
- 800 µg Pb/sq ft for window wells

The HUD Interim Guidelines (HUD, 1990) recommendation for Pb in paint film is 1.0 mg/cm^2 or 0.5% by weight. The Consumer Product Safety Commission (CPSC) limit for Pb in paint film, established in 1978, is 0.06%. Recently, a proposal was made by CPSC to reduce the Pb content of paint to 0.01% by weight (*Federal Register* 57, 18418, April 30, 1992). CDC suggests that 500-1000 µg/g of Pb in soil leads to elevations in blood Pb in children (CDC, 1991). ASTPHLD suggests that if a child with a blood Pb level of zero ingests 1 g of soil containing 1000 mg/kg Pb, then the blood Pb level of the child could rise to 10 µg/dL, the current CDC action level. ASTPHLD suggests that a soil Pb level of less than 200 µg/g (200 mg/kg) would not result in a significant elevation of blood Pb level in children, unless an unusually large amount of soil is ingested (ASTPHLD, 1991).

The recommended minimum sample size to submit to the laboratory is:

- Soil: 1.5 g
- Dust: 300 mg
- Wipe: 1 wipe used to sample dust in a 1 sq ft area
- Paint: 300-mg paint chips

The absolute minimum sample size needed for analysis taken from a submitted sample using FLAA methods is:

- Soil: 0.5 g or as specified in the digestion procedure
- Dust: 100 mg or as specified in the digestion procedure
- Wipe: 1 wipe taken over a 1 sq ft area or as specified in the digestion procedure
- Paint: 100 mg or as specified in the digestion procedure

The minimum sample size is dependent on the concentration of Pb in the sample, on the dilution of the digested Pb containing sample prior to instrumental analysis, and on the instrumental method of analysis. Wipe samples containing less than 200 µg Pb/sq ft (the HUD, 1990 clearance value) may not be detected with FLAA if only 1 sq ft is sampled and the final digest volume is 100 mL. Adequate sensitivity exists for other matrices. GFAA and ICP-AES have adequate sensitivity, even at clearance levels and minimum sample size recommendations.

Minimum sample sizes required for other sources of Pb, such as from steel structures and non-HUD buildings, must be determined from relevant guidelines.

5.3 WIPE SAMPLING

5.3.1 Acceptable Criteria for Wipes

There should be minimum wipe sample acceptability criteria that would either specify acceptable dust wipe media for collection of deposited dust samples or require laboratory evaluation of the collection media for blanks and digestion recovery/ interference. If a laboratory is to accept a sample for analysis in a matrix other than specified in this guideline, it must develop an SOP as described in Subsection 6.2.

5.3.2 Characteristics of a Good Wipe

- Durability during sample collection. The wipe should not disintegrate during the sampling process.

- Controlled background. The wipe should be made to rigid specifications regarding its background levels of Pb from the manufacturing process. ASTM Subcommittee E06.23 suggests a Pb level of < 5 µg Pb/wipe.

- Digestibility of the wipe should be compatible with analytical sample preparation SOPs and leave little or no residue.

- Wipes should have good dust pickup capacity and not just "push the pile of dust around the surface."

- Wipes should be individually-wrapped and pre-moistened, or there should be a well-defined sampling protocol for multiwipe dispensers in order to minimize contamination.

5.3.3 Wipe Sampling Recommendations

Procedures for collection of wipe samples are detailed in the HUD Interim Guidelines, Appendix A5-24 (HUD, 1990). Details are also given in the HUD Risk Assessment protocol (HUD, 1992), pp. 28926-28927. An SOP for wipe sample collection must be followed. The type of wipe is not specified in the HUD Interim Guidelines (HUD, 1990), which creates a difficulty for the laboratory, because a variety of dust wipe samples are likely to arrive in the laboratory. Wipes, including "baby wipes" (including those containing lanolin and aloe), gauze pads, filter paper, napkins, "wet naps," alcohol swabs, and duct tape, have been received to date in some laboratories offering Pb analyses. The laboratory should request the following regarding all wipe samples:

- Rigid contamination control should be maintained by (1) use of an individually wrapped pre-moistened wipe, or (2) use of wipes from a multiple dispenser pack accompanied by a specific protocol.

- Unused wipes should be submitted to the laboratory for (1) Pb blank determination and (2) digestibility studies. If existing SOPs include digestion data for the submitted wipe, then only a Pb blank determination is necessary.

- If wipes from a dispenser pack are used, the HUD sampling guidelines should be followed. (Discard first wipe and handle wipes with gloved hand to avoid contamination. Each new wipe sample should be handled with a clean glove to avoid cross-contamination.)

5.4 PAINT CHIPS

The HUD Interim Guidelines (HUD, 1990) and the HUD Risk Assessment Protocol (HUD, 1992) provide sampling protocols. An SOP for paint chip collection must be followed. The laboratory should request that those collecting the paint chips adhere to the following recommendations:

- Collect paint chips free of other debris, if possible.

- Remove as much substrate as possible, if necessary, because results may be expressed as Pb per weight.

5.5 DUST—VACUUMED SAMPLES

The HUD Interim Guidelines (HUD, 1990) do not suggest protocols for collection of vacuumed dust, and there are no published guidelines. The procedure for the collection of vacuumed dust samples should be detailed in an SOP.

5.6 SOIL SAMPLES

The HUD Interim Guidelines (HUD, 1990) and the EPA (U.S. EPA, 1990c) have protocols for collection of soil samples. Generally, the soil sample represents a composite of samples collected from several adjacent areas and at different depths. Soil collection should be detailed in an SOP.

5.7 BLANKS AND BACKGROUND SAMPLES

The HUD Interim Guidelines (HUD, 1990) suggest some of the types of field blanks and background samples that should be submitted to the laboratory. The collection of blanks and background samples should be covered by an SOP. The suggestions below are based on good laboratory practice for metals laboratories.

5.7.1 Blanks

The following types of blanks should be submitted to the laboratory.

Trip blank: A clean sample, including collection media, that is carried to the sampling site and transported back to the laboratory for analysis **without being opened**. This blank is analyzed as a regular sample through all steps. The trip blank evaluates the integrity of the sample container.

Field blank: A clean sample of matrix (e.g., paint, soil, dust, or wipe) carried to the sampling site, exposed to the sampling conditions (e.g., bottle caps removed) and returned to the laboratory, treated as an environmental sample, and carried through all steps of the analysis. For example, clean quartz sand, non-Pb containing paint, or a clean wipe could be used as a field blank matrix. The field blank evaluates possible site contamination sources such as airborne contaminants.

Rinseate blank: A sample of a "used" cleaning fluid rinse solution, also called an equipment blank. Rinseate blank examples include a final rinse of the device used to collect soil or vacuumed dust or the final rinse to clean a scoop used to collect soil or vacuumed dust samples. The rinseate blank is used in rinsing collection media and equipment prior to use to monitor possible cross contamination. The rinseate blank goes through all steps in the analysis including the digestion.

5.7.2 Background Samples

A background sample is a sample of matrix collected at or near the site that is uncontaminated with Pb from paint. It is often difficult to obtain a true background sample in the field; therefore, the collection of background samples is not recommended.

5.8 DUPLICATE FIELD SAMPLES

The HUD Interim Guidelines (HUD, 1990) suggest collection of "duplicate" samples adjacent to areas of concern. This term is incorrectly used (see Glossary) since "duplicate" implies a uniform distribution of sample. The distribution of Pb in paint, soil, and dust is not uniform; therefore, the usage of the term "duplicate" is incorrect. A second sample could be collected in an adjoining area to provide a better representation of Pb deposition, but this is not a duplicate sample.

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SECTION 6

LABORATORY SAMPLING

This section covers the handling of the sample after it is received from the field and prior to digestion. Cleanup activities (removal of rocks, substrate, hair, etc.) and subsampling or aliquoting of the bulk sample into uniform portions suitable for analysis are also covered. Subsection 6.6 gives recommendations for sample tracking and storage.

6.1 SOLIDS—GENERAL CONSIDERATIONS

Particle size distribution, debris, and stratification of solid samples is a major problem. The following general considerations apply to all solid samples.

- Samples must be examined for debris, such as hair, paper clips, pins, and insects, prior to subsampling the sample. Debris should be removed with tweezers or by screening through a course #4 mesh (4 to 7 mm) sieve.

- Samples of dust or finely ground paint or soil are subject to stratification from vibration in the laboratory. Therefore, thorough mixing is essential prior to removing an aliquot for analysis.

- A representative sample must be obtained. A device such as a "riffle" box, or equivalent, should be used for separating and allocating fractions of dust or soil that have been ground to a #10 mesh (1.9 mm) and sieved. A riffle box randomly aliquots one-half of the sample to one side and one-half of the sample to the other. Several passes may be necessary to reach usable sample sizes for digestion.*

- Humidity control is very important in sampling of solids, particularly if results are expressed on a weight basis. Oven drying at 105` C to a constant weight is recommended.

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* Use of a riffle box to separate coal and coke is described in ASTM Method (D5).

6.2 WIPE SAMPLES

The handling of wipes in the laboratory should be detailed by an SOP. Wipes are used to collect deposited dust over a defined surface area. In most cases, the Pb content in the wipe material is unknown and appears to vary from lot to lot and among suppliers. The pickup efficiency (ability to pick up and retain dust on the wipe material) and the digestibility properties are also unknown. Research on the development of standardized wipes is in progress. Until such time as some "standardized wipe" is developed, the laboratory must perform the following tests on each type of wipe submitted.

- Determine the Pb background level in the "blank" wipe submitted with the samples. Ideally, Pb background levels of wipe samples should be determined prior to sample collection. If the background level is greater than 5 µg Pb per wipe, blank correction may be necessary. Blank correction can be used if the blank is < 20% of the regulatory limit or level of concern. If blank correction is used, it must be documented on the report. It should be noted that blank values of 5 µg per wipe are insignificant at HUD clearance recommendations of 200 µg per wipe collected over a 1 sq ft area.

- Perform a recovery study of a spiked wipe (extractable Pb) using the laboratory standard digestion technique. The digestion technique does not have to destroy wipe fibers (total Pb) but should be able to digest Pb from dust deposited on the wipe (extractable Pb).

Results should be expressed per wipe or per area sampled. If the area is less than 1 sq ft, results should be corrected and reported as µg Pb/sq ft.

6.3 PAINT CHIPS

Appropriate steps must be taken to ensure uniformity of the sample before subsampling. The presence of "substrate" compromises the results, particularly if the results are presented on a weight basis. The handling of paint chips must be covered by an SOP. Because paint chips containing substrate present special problems, the following should be addressed:

- Attempts should be made to remove the paint from substrate. If the paint cannot be removed from the substrate, the analytical report must include an annotation that results may be invalid. Substrate contamination of paint must be noted because large amounts of nonlead containing substrate will produce low lead concentrations and may lead to false negative results.

Paint chips relatively free of substrate should be handled as follows:

- Cut paint chips into small pieces with scissors or a knife.

- Grind up the pieces of paint chips into a powder with a mortar and pestle or other nonmetal contaminating material to improve digestibility. Paints with a high latex content may not be suitable for grinding due to their tendency to form "gummy" residues.

6.4 DUST—VACUUMED SAMPLES

There are no defined procedures for preparation of vacuumed dust samples. An SOP must be developed covering the steps listed below.

- Dust samples should be sieved (#10 mesh, 1.9 mm) to remove debris (metal, glass, hair, pins, etc.) prior to digestion because this debris is not dust.

- Dust samples must be subjected to humidity control. If dust is moist, it should be dried in an oven at 105` C to a constant weight. Cross-contamination during drying can be reduced by placing dust samples in covered drying bottles.

6.5 SOILS

The handling of soil samples should be covered by an SOP that addresses:

- Screening to remove debris, including metal, glass, plant material, rocks, plaster, and bricks. If large paint chips are present in the soil, these should be removed and digested separately.

- Drying of the soil at 105` C to a constant weight to control for variable moisture content.

- Grinding to a fine mesh (#10) to aid digestion.

- Thorough mixing prior to analysis to avoid stratification.

6.6 SAMPLE TRACKING AND STORAGE

A sample tracking system should be detailed in an SOP and referred to in the QA manual. A subsampling system of unique numbers should be used for all digests and dilutions of the original sample so that original sample identification is not lost. If computer log-in procedures are used, the sample log-in procedure should include hard copy backup of computer log-in records. Although a legal chain of custody is not usually required, a client may demand it in some cases that may involve potential litigation.

The SOP should detail the specifics for storage of unused samples during the analysis cycle and after completion of analysis. Digests can be kept for 2 to 4 weeks, as long as digest volumes are monitored gravimetrically for evaporation loss. Holding

times for matrix samples are not a problem. It is recommended that the laboratory establish an automatic discard date for digests and samples, such as 90 days after reporting to the client, unless contacted by the client.

The requirements for storage of samples and digests include:

- Secure storage in a locked or controlled-access area.
- Uniform environmental conditions must be maintained, such as a cool, dry storage area.
- Storage of digests require special conditions. Fluid loss in digests by evaporation should be monitored gravimetrically.

SECTION 7

SAMPLE DIGESTION PROCEDURES

At this time, there are no standard EPA-approved digestion methods for paint and dust matrices. EPA SW-846 Method 3050 is approved for soils, but not necessarily for analysis of Pb in soils contaminated with paint chips. There are three basic sample digestion techniques that have been applied, often with some modification, to digest paint chips, dust (wipes and collected dust), and soil samples. These methods are outlined below and cited in Section 9.0. However, little published information is available to document the suitability of these digestion methods.

- Dry ashing followed by wet digestion with HNO_3 or HNO_3/H_2O_2
- Wet digestion using a hot plate with HNO_3 or HNO_3/H_2O_2
- Microwave digestion with HNO_3, HNO_3/HCl, or HNO_3/H_2O_2

Dry ashing is not recommended because it is difficult to control and has the possibility of uneven heating and splatter/cross-contamination of samples. Therefore "wet digestion" techniques are preferable. "Wet digestion" techniques using HNO_3 alone are not generally recommended because digestion may be incomplete. Wet digestion techniques, such as U.S. EPA SW-846 Method 3050, a validated method for soils, are suggested and may be suitable for digestion of most samples. But this method has not been validated for other matrices. NIOSH Method 7082 is also suitable for digestion of dust samples. Perchloric acid has also been used in combination with nitric acid with acceptable results. However, since the use of perchloric acid requires special safety precautions (i.e., a perchloric acid hood), it is generally not recommended.

Research is currently underway to develop a standard digestion procedure that would work with all matrices using 200-250 mg of sample. The ASTM has prepared draft wet digestion methods for wipes, dust, soils, and paint chips, which are currently undergoing review and testing. Digestion acids should be chosen with analytical instrumentation in mind because of incompatibility of HCl with GFAA instrumentation. Whatever digestion technique is used, it is recommended that the digest be filtered or centrifuged prior to instrumental analysis. The specified digestion procedure should be documented in an SOP.

Other methods, including digestion with perchloric and hydrofluoric acids and fusion with lithium metaborate, have been suggested, but are not recommended for general use because of safety considerations.

SECTION 8

INSTRUMENTATION

Three general types of instrumentation are suitable for analysis of Pb in paint, soil, and dust matrices. These are listed below and are available from a variety of vendors in many different configurations.

8.1 ATOMIC ABSORPTION SPECTROMETRY USING DIRECT FLAME ASPIRATION

This instrumentation, which is widely available with and without autosamplers, has adequate sensitivity for most samples, depending on dilution volumes used in the digestion process. Following are characteristics of FLAA:

- Instrument detection limits: Instrument detection limits are adequate for most samples, but are the highest of the three techniques considered in this guide. Since direct aspiration is required, a minimum of 5 mL of digest is needed for aspiration and measurement of a stable signal.

- Principal interference: Light scattering and molecular absorption by matrix components are common for measurements near detection limit and can cause a false positive signal. They can best be corrected using methods such as a continuum source or Zeeman background correction schemes. Correction using alternate nonabsorbing Pb lines is possible but not optimal. Matrix enhancement or suppression of the Pb absorbance signal is possible at all concentration levels and can be corrected by using the method of standard additions.

- Cost: Initial instrument cost is low and consumables, such as acetylene gas, are inexpensive.

- Sample throughput: Sample throughput is good using either autosamplers or manual aspiration and can be several samples per minute.

- Maintenance and potential downtime: Routine maintenance is limited to periodic cleaning of the aspirator, mixing chamber, and burner, as well as external optics as necessary. Of the three methods, maintenance and potential downtime is lowest.

- Principal Pb lines: The 283.3-nm line is preferred because of less molecular absorption and scattering. The 217.0-nm line, however, is more sensitive and can be used if a continuum source background correction is employed.

- Range of analysis: The linear range of analysis is approximately two orders of magnitude, from about 0.2 µg Pb/mL to 20 µg Pb/mL, but can be extended by rotating the burner head.

- Potential problem areas: The method detection capabilities are borderline for wipes below HUD "Clearance" levels. For such samples, the 217.0-nm line and background correction must be used.

8.2 ATOMIC ABSORPTION SPECTROMETRY USING THE GRAPHITE FURNACE

This instrumentation is widely available and is the most sensitive technique of the three techniques. Throughput is good with autosamplers and sample size requirements are very small. The following are GFAA characteristics:

- Instrument detection limits: Detection limits are the lowest of the three instrumental techniques. Because only 20 µL of sample are used for analysis, digest volume requirements are the smallest (10 to 25 mL). If the laboratory is also involved with blood Pb determinations, GFAA is the instrumentation of choice.

- Principal interference: Light scattering and molecular absorption by matrix components are common for most measurements and can cause a false positive signal. They can best be corrected by methods such as a continuum source (Deuterium Arc) or Zeeman or Smith-Hieftje background correction schemes. Matrix enhancement or suppression of the Pb absorbance signal is often significant and can be corrected by using the method of standard additions. Matrix modifiers, such as magnesium nitrate, lanthanum nitrate, palladium, or ammonium dihydrogen phosphate, minimize loss of Pb during the sample charring step and allow higher charring temperatures. This also minimizes, but does not eliminate, matrix enhancement or suppression effects. Chloride arising from the use of HCl in a digestion can cause significant interferences in GFAA.

- Cost: Initial instrument cost is intermediate. Maintenance and consumable costs are significant, since the graphite furnace tubes must be replaced approximately every 500 firings and argon gas must constantly flow through the system to prevent oxidation of the graphite.

- Sample throughput: Autosamplers are required to increase precision and throughput. Manual sample introduction is tiresome and often irreproducible. Throughput is approximately one sample every 2 to 3 min.

- Maintenance and potential downtime: The primary difference between maintenance of GFAA and FLAA is the alignment and cleaning of furnace components of the former. Because of the complexity of some graphite furnace systems, downtime may be greater than with FLAA.

- Principal Pb lines: The 283.3-nm line is preferred because of less interference. The 217.0-nm line, however, is more sensitive and may be used as needed, provided the interference and noise are not severe.

- Range of analysis: The linear range of analysis for an intermediate sample size is from about 0.001 µg Pb/mL to 0.1 µg Pb/mL, but can be varied by adjusting sample size and dilution.

- Potential problem areas: Matrix interference and contamination.

- Advantages: This instrumentation is widely available and is the most sensitive technique of the three. Throughput is good with autosamplers and sample size requirements are very small.

8.3 INDUCTIVELY COUPLED PLASMA EMISSION SPECTROMETRY

This instrumentation is available in many laboratories and offers the advantage of simultaneous multielement determinations. Sensitivity for Pb is intermediate, but adequate for all Pb matrix samples. Sample volume requirements are moderate because the digest is aspirated into the plasma torch.

- Instrument detection limits: Instrument detection limits, which are similar to the FLAA technique, are adequate for most samples, but may present analysis difficulties at the lowest level of wipe samples. Because the direct aspiration rate of ICP-AES is less than FLAA, less sample is required. If other elements are desired in environmental samples, this is the instrumentation of choice.

- Principal interference: Spectral interferences caused by radiation from lines of other elements present in the sample are most common and can be corrected by several methods. Background correction can be performed by selecting wavelengths near the Pb line, or an alternate Pb line can be used. It is important to include an interfering element check sample that contains high levels of suspected elements (aluminum, titanium, chromium, calcium, or iron).

- Cost: Initial instrument cost is high, but major consumable cost is only argon gas, unless the instrument is operated incorrectly and the torch is destroyed.

- Sample throughput: Sample throughput is intermediate between FLAA and GFAA. Samples that are directly aspirated require a longer period for equilibration and washout. Throughput is typically slightly less than one sample per minute.

- Maintenance and potential downtime: Maintenance costs are the highest of all the instruments discussed because of the complicated design of ICP-AES instruments and the requirements for critical alignment of components.

- Principal Pb lines: Usually the 220.35-nm line is used, although an alternate line is at 217.0 nm.

- Range of analysis: The linear range of analysis for the 220.35-nm line is from about 0.2 µg Pb/mL to 3,000 µg Pb/mL.

- Potential problem areas: Spectral interferences from high levels of other metals and insufficient washout of mixing chamber can occur after the analysis of a sample of high Pb concentration.

- Advantages: This instrumentation is available in many laboratories and offers the advantage of simultaneous multielement determinations. Sensitivity for Pb is intermediate, but adequate for all Pb matrix samples. Sample volume requirements are moderate because the digest is aspirated into the plasma torch.

8.4 OTHER INSTRUMENTATION

In addition to these instruments, there are others that are not currently recommended. X-ray fluorescence (XRF) is currently being evaluated for laboratory use and may be suitable. However, sample preparation steps, including sample loading, can significantly affect precision and bias. Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), although a powerful and sensitive technique, is not recommended at this time because of a lack of need for this level of instrumentation sophistication and costs. Anodic stripping voltametry may be suitable, provided that the method is compatible with digestion techniques. Methods using spectrophotometric instrumentation for Pb, such as the dithizone method, are not recommended because of the potential for contamination and interference. The latter method also may not be compatible with digestion procedures.

SECTION 9

ANALYTICAL METHODS AND CALIBRATION

This section gives a list of published methods and discusses calibration standards applicable to paint, soils, and deposited dust matrices. References from agencies of the Federal Government can be obtained from the National Technical Information Service (NTIS), (703) 487-4650.

9.1 LIST OF METHODS

Many of these methods have not been validated with paint, soil, and deposited dust matrices.

AOAC 5.009 (1984) - Lead in Paint Using Direct Aspiration Atomic Absorption.

ASTM D-3335-85a - Test Method for Low Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectrometry (direct aspiration).

ASTM D 3618 - Test Method for the Detection of Lead in Paint/Dried Paint Films.

U.S. EPA Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air (40 *CFR* Part 50, Appendix G).

NIOSH 7082, Lead in Air Collected on Cellulose Ester Filters. Nitric acid/ hydrogen peroxide hot plate digestion followed by direct aspiration atomic absorption at 283.3 nm.

NIOSH 7105, Lead in Air Collected on Cellulose Ester Filters, Nitric/Hydrogen Peroxide Hot Plate Digestion Followed by GFAA.

NIOSH 7300, Elements in Air Collected on Cellulose Ester Filters, Nitric/ Perchloric Acid Hot Plate Digestion Followed by ICP-AES at 220.4 nm.

*U.S. EPA SW-846 Method 7420, Pb - Atomic Absorption, Direct Aspiration (U.S. EPA, 1990c).

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* These methods do not include a digestion technique and are for digests of Pb prepared by one of the digestion techniques listed above.

*U.S. EPA SW-846 Method 7421, Pb - Atomic Absorption, Graphite Furnace (U.S. EPA 1990c).

*U.S. EPA SW-846 Method 6010A, Metals - Inductively Coupled Plasma Emission Spectroscopy (U.S. EPA 1990c).

**U.S. EPA SW-846 Method 3050A - Acid Digestion of Sediments, Sludges and Soils (Metals) (U.S. EPA 1990c).

**U.S. EPA SW-846 Method 3051 - Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils (Metals) (U.S. EPA 1990c).

In addition to these cited methods, the ASTM E36 subcommittee is working on several standard methods for Pb. These include GFAA, FLAA, and ICP-AES. These draft methods include digestion techniques for paint, dust, wipes, and soil and include hot plate wet digestion techniques and microwave digestion methods. These draft methods are not yet available for distribution.

9.2 CALIBRATION

9.2.1 Primary Standards

Primary standards are solutions of standards that are traceable to aqueous-based SRMs from NIST and should be used for instrument calibration. The preparation of primary standards should be detailed in an SOP. The SOP should detail the traceability of the primary standard to primary calibrant SRMs from NIST.

The NIST aqueous 10,000 ppm Pb (in 10% HNO_3) SRM is suitable for calibrant material and should be used to check laboratory working standards. The SRM is available as SRM 3128 (50 mL of a 10 mg/mL solution in 10% HNO_3). Matrix-based SRMs are not primary standards and are not suitable for instrument calibration.

9.2.2 Working Standards

Stock primary standards must be prepared from material traceable to NIST SRM 3128. These stock standards are stable, but are subject to evaporation and loss of Pb to the container wall. The possible loss of solvent can be monitored by weighing the stock solution at regular intervals. The preparation of stock and working standards, including storage conditions, should be detailed in an SOP. Acids used in standards should match the acids used in the matrix. Purchased stock standards should include certifications that standards are traceable to SRM-3128.

** These methods are general digestion techniques for the matrices listed. The mild conditions used in SW-846 methods must be evaluated for their efficiency in digesting these matrices. They must be combined with an analytical method such as the EPA SW-846 Methods 6010A, 7420, or 7421 for completion of analysis.

Working standards should be prepared from stock primary standard solutions of 1,000 to 10,000 ppm Pb. Working standards are used for initial calibration of the instrument and to verify the calibration at intervals dependent on the instrumental method. The recommended minimum intervals are at the beginning, midpoint, and at the end of a batch of samples (usually 20 samples) run on any particular day. Results that are reportable should be in the calibration range.

9.2.3 Matrix-Based Quality Control Samples

A variety of matrix-specific materials (LCS) contain Pb and can be used for quality control samples. These internal QC samples must be independent of the instrument calibrant and used only to monitor the performance of the entire process, including the digestion step.

9.2.4 SRMs from the NIST

NIST prepares a variety of SRMs. These reference materials are rigorously characterized and analyzed by definitive methods. They are expensive and are not intended to be used for routine quality control. They are intended to be used in the development and validation of methods and as a real-world tool to evaluate method performance. Examples of NIST SRMs available for Pb-based matrices are listed in the table below. Certificates are available from NIST.

SRM	Description and date	Certified Pb value
1579a	Powdered Pb-based paint, Feb. 3, 1992	11.995% ± 0.031
—[a]	Powdered Pb-based paint (in progress)	4.0%
—[a]	Powdered Pb-based paint (in progress)	0.5%
1648	Urban particulate matter, Nov. 16, 1978	0.655% ± 0.008
2704	Buffalo River sediment, July 9, 1990	161 µg/g ± 17
2709	Baseline agricultural soil, Oct. 16, 1992	18.9 µg/g ± 0.5
2710	Highly contaminated soil, Oct. 16, 1992	5532 µg/g ± 80
2711	Moderately contaminated soil, Oct. 16, 1992	1162 µg/g ± 31
2579	Lead paint film on Mylar sheet, set of 5, July 27, 1992	3.53 mg/cm^2 ± 0.24 1.63 mg/cm^2 ± 0.08 1.02 mg/cm^2 ± 0.04 0.29 mg/cm^2 ± 0.01 < 0.0001 mg/cm^2

[a] These NIST SRMs are under development.

9.2.5 Other Reference Materials

Reference materials from other sources are available, but they are not NIST-certified and may be less well-defined and characterized. However, they may be suitable for use as internal quality control materials.

There are three CRADA certified materials available. They are labeled: "This product was verified for accuracy and stability under a cooperative research and development agreement (CRADA) with the U.S. Environmental Protection Agency." They are manufactured by Resource Technology Corporation, Laramie, Wyoming, and are available from Fisher Scientific. These reference materials have also been certified by A2LA. Current research is being conducted to better characterize the homogeneity of these materials. These materials are not characterized like SRMs and cannot be used as substitutes for NIST SRMs. Their characteristics are shown in the following table.

No.	Description	Certified concentration
SRS 013-50	Paint Blasting Waste	643.2 ± 129.4 ppm
SRS 006-50	Paint Sludge	753.0 ± 114.7 ppm
SRS 014-50	Bag House Dust	1914.2 ± 410.6 ppm

ELPAT samples may be available for use in evaluation of method performance. Call (703) 849-8888 for more information.

9.3 VALIDATION OF METHODS

Analytical methods should include validation studies conducted with matrix-based SRMs, if available, or with other matrix-based reference materials. Guidelines for analytical methods validation studies have been published in the *Journal of the Association of Official Analytical Chemists International* (JOACI, 1989).

9.4 SUMMARY OF INSTRUMENT- AND MATRIX-SPECIFIC PARAMETERS

9.4.1 Instrument-Specific Parameters

The following table is a summary of typical instrument-specific parameters identified by the working group.

Parameter, instrument-specific	ICP-AES	FLAA	GFAA
IDL (µg/mL)	0.05	0.03	0.001
MDL (µg/g)[a,c]	5	3	0.1
Interference, spectral[b]	Al, Cr, Ti, Ca, Fe	not common	not common
Interference, matrix and corrective action[b]	Possible. Matrix-matching internal standards	Common. Method of standard additions	Common. Matrix modifiers and background correction; method of standard additions
Sample size, preferred	600 mg	750 mg	150 mg
Sample size, lab minimum[c]	200 mg	250 mg	50 mg

[a] MDL: Calculation: see 40 *CFR* 136, Appendix B.

[b] Interference, matrices: Other matrices, such as the substrate and debris (hair, glass, sticks, needles, insects, etc.), will interfere if not screened or removed from the matrix of interest.

[c] These values will vary depending on the digestion procedure used, the final volumes, and sample sizes.

9.4.1.1 Precision, Accuracy, and QC Frequency—

Precision, accuracy, and frequency of QC should be nearly the same for all methods. Precision and accuracy should be charted for the particular measurement system with performance characterized by an SOP. Generally precision for all methods is about ±10% at 5X MDL. Accuracy, measured as percent recovery, varies from 85-115%.

Matrix spikes and QC check samples independent of the calibrant should be run at a minimum frequency of 5% (1 per 20 or 1 per batch).

Stability checks of instrumentation are laboratory and instrument specific and should be detailed in an SOP.

9.4.2 Matrix-Specific Parameters

The following table is a summary of parameters specific for the matrix.

Parameter, matrix specific	Paint chips	Soil	Dust	Vacuumed dust
Sample size, bulk	250 mg[a]	1-2 g[a]	1 ft^2 wipe[a]	300 mg[a]

[a] Sample sizes are dependent on the digestive procedure, final volumes, and instrumentation used for the analyses. These values are typical of FLAA techniques.

Other sample parameters, such as homogenization and digestion techniques, are specific to a given matrix, instrument, and collection technique. Specifics of these parameters should be detailed in an SOP.

SECTION 10

DATA QUALITY AND REPORTS

This section discusses proficiency testing, rejection criteria, and reports and record management.

10.1 PROFICIENCY TESTING

Laboratories must demonstrate proficiency in the Environmental Lead Proficiency Analytical Testing (ELPAT) Program to be "recognized" by NLLAP (See footnote on p. xi). Laboratories may participate in this program independently without participating in an accreditation program.

10.1.1 Characteristics of Proficiency Testing Materials from ELPAT

10.1.1.1 Wipes—

Currently, the wipe proficiency testing (PT) material is a Whatman No. 40 filter with added analyzed paint dust. This filter is manufactured to rigid quality standards that provide a consistently low background level of Pb. However, in general, these types of filters have poor durability and poor pickup efficiency and are, therefore, not a suitable collection medium. Currently available "baby wipes," though not necessarily of consistent quality for use as dust collectors, are more durable than the above-noted filters, but have not been manufactured to provide a consistently low background level of Pb. When a standardized, laboratory-grade wet wipe is developed for dust collection, that material may be used as a "real world" testing medium.

10.1.1.2 Powdered Paint Chips—

The PT material is prepared from a composite of paint collected from the outside of old buildings. The composite is then ground to a fine mesh and blended with paint from different sources to achieve target concentrations.

10.1.1.3 Soil—

The PT material is prepared from a composite of soils taken from different sources. The composite is then ground to a fine mesh and blended with soils from difference sources to achieve target concentrations.

(PT matrix materials, including ELPAT samples, are not to be used for instrument calibration or primary standards. These materials have not been subjected to rigorous characterization for their target concentrations.)

10.1.2 Target Concentrations for ELPAT Proficiency Testing Materials

Specifications and target concentrations of the NIOSH/AIHA PT samples are shown below.

Wipes (Whatman No. 40 filters spiked with paint dust)*

- 20 µg Pb/wipe
- 200 µg Pb/wipe
- 500 µg Pb/wipe
- 5000 µg Pb/wipe

Paint chips**

- 0.05% Pb
- 0.4% Pb
- 0.7% Pb
- 5.0% Pb

Soil*

- 20 mg Pb/kg (background levels in rural environments)
- 500 mg Pb/kg
- 1000 mg Pb/kg
- 5000 mg Pb/kg

It should be noted that PT materials and SRMs that are fine powders are subject to significant stratifications from vibration in the laboratory. Therefore, thorough mixing is essential prior to removing an aliquot for analysis.

There also are problems with paint dust and soil because of the non-uniformity of the matrix. The PT program must assess the skills of the laboratory and not the uniformity of the PT materials. Work continues on characterization of the uniformity of the ELPAT PT materials to better characterize the material.

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* The HUD Clearance Guideline recommendation is 200 µg Pb/sq ft on floors.

** CDC guidelines for paint chips are 500-1000 ppm (0.05-0.10%).

* ASTPHLD suggests that a soil Pb of less than 200 µg/g (200 mg/kg) may not result in the significant elevation of blood Pb in children, unless an unusually large amount of soil is ingested.

10.2 REJECTION CRITERIA AND CORRECTIVE ACTION

The following guidelines are recommended as minimum rejection criteria that require corrective action prior to release of data. Data should be thoroughly evaluated, even if one of these criterion is out of range, and corrective action taken prior to release of data.

- Within day or intra-day variation of the calibration curve as measured by continuing calibration verification is greater than 10%.
- Any blank that exceeds 20% of the regulatory limit or minimum limit of concern.
- Spike recoveries of extractable Pb less than 75% or greater than 125% at the mid-range concentration.
- Matrix-based quality control or check sample (also called control or laboratory control sample) outside 80% to 120% of stated value.
- Unacceptable precision (> ±25% RPD) of duplicate samples (two aliquots of the same bulk sample carried through the entire procedure.) Precision is based on the concentration of the sample and the method detection limit.

Corrective actions include reanalysis of QC check samples. If these QC samples are out of range, then repeat the entire analysis including recalibration and all QC samples.

10.3 REPORTS AND RECORD MANAGEMENT

Reporting and record-keeping requirements are outlined in the HUD Interim Guidelines (HUD, 1990).

> "All information relating to field sample analysis and QA/QC sample analysis, along with information on laboratory facilities, equipment, methods, and procedures should be documented by the laboratory, so that an analytical event can be recreated for an audit or investigation."

The HUD Interim Guidelines (HUD, 1990) recommend that the following general categories of records should be kept.

- Cover page information including methods, dates, instruments, digestions, and sign-offs by the laboratory director.
- Sample information including identification, blanks, QC samples, sample weights, dilution factors, and batch identification.
- Results of initial precision and accuracy runs.
- Results of calibration including sources of standards and detection limits.

- Results of blanks including type of blank and any corrections used.
- Results of calibration verification checks.
- Results of tests for accuracy and precision.
- Data reduction and reporting procedures including data calculations, outliers, and data archiving.

More details are given in the HUD Interim Guidelines (HUD, 1990). The client may have more specific needs, so the laboratory should be prepared to provide that data.

There are no regulatory requirements for record retention for these matrices. The HUD Interim Guidelines and NLLAP requirements suggest 10 years. Record retention policies must be established with the client, with the realization that there may be future regulatory requirements.

SECTION 11

GENERAL RECOMMENDATIONS FOR AN ANALYSIS PROTOCOL

The analysis protocol for a digest may be specified in individual method citations. Individual laboratory SOPs must provide specifics. The quality control program should be based on the laboratory's continuous evaluation of its performance (system process control). In the absence of laboratory-generated process controls, the recommendations in Section 3.4 should be used regarding frequency of blanks, calibration, and controls.

Since Pb is ubiquitous in the environment and in the laboratory, rigorous steps must be specified in an SOP on how contamination control is to be achieved during subsampling, digestion, and analysis. Cross-contamination should be documented by monitoring of surfaces, glassware, and reagents. A protocol to reduce cross-contamination from Pb is described by T. J. Murphy (Murphy, 1976).

The following are general recommendations for an analysis protocol:

- The instrument should be calibrated daily with an aqueous working standard traceable to an aqueous-based SRM (SRM 3128).
- Stock working standards for Pb (10,000 ppm) are stable. However, evaporation should be monitored by periodic weighing to document and correct for evaporative losses. Sealed containers help control evaporation loss; however, loss to container walls is possible.
- The daily calibration curve should consist of one initial calibration blank and <u>at least</u> three standards covering the concentration range of the samples.*
- The 3-standard calibration curve should have a correlation coefficient of at least 0.995.
- The calibration curve should be verified by the periodic use of continuing calibration blank (CCB) and continuing calibration verification samples throughout the run.
- The LCS (matrix-based and near the midpoint of the calibration curve) should be ±20% of stated value.

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* Calibration requirements are both instrument and method specific. SOPs for specific analytical methods should be followed.

- One spiked matrix sample or duplicate matrix sample should be included per batch of up to 20 samples. A suitable duplicate matrix sample would be split digest samples because duplicate field samples cannot be collected.

- Instrument drift should be documented and corrected using continuing calibration verification (CCV) and continuing calibration blanks (CCB) according to the method SOP.

- Interference check samples (ICSs) for ICP-AES instrumentation (background shifts and interelement interference) should be determined prior to performing analyses to correct for potential interferences from components in the sample matrix. The ability of the instrument to measure lead in the presence of potential interference should be determined at the beginning, during the run, and after the sample is run. Correction factors should be applied if available on the specific ICP-AES instrument in use.

- Background correction for GFAA using simultaneous methods (e.g., Zeeman, Smith-Hieftje, Deuterium Arc) should be used at all times.

- Matrix modifiers, used in GFAA, should be verified to be free of Pb contamination.

- Matrix-based SRMs at action levels, if available, should be used to verify working standards and CRMs at monthly intervals.

- All samples exceeding the upper limits of the calibration range should be diluted to fit within the calibration range.

- The SOP should provide for a means to control carryover following samples with high concentrations (memory effect). Reruns of samples following a high sample is recommended.

- The SOP should provide for possible resampling of the submitted sample if the result is at or above an "action level" to confirm a "positive" result.

- Sample analysis priorities: Although the following scenario has been suggested, consideration should also be given to development of an analysis protocol using randomization of samples and blanks to minimize bias.

    - Assemble all samples, standards, blanks, and background samples.

    - Analyze those samples expected to contain Pb first.

    - If a significant amount of Pb is found, analyze blanks and background samples to determine if there is contamination.

    - Blank collection media (wipes) should also be analyzed to determine the background Pb levels.

- QC data should be control charted in order to monitor trends and QC excursions. The SOP must specify what is done in the event of unacceptable trends or excursions.

Table 1 shows the recommended process quality control blanks and control materials to be included in each batch. Table 2 shows the recommended instrumental QC standards and their specifications.

TABLE 1. QUALITY CONTROL SAMPLES AND PROCESS CONTROL

QC samples	Definition	Frequency
Method blanks	Type 1 water—digest as a sample with addition of all reagents. Should reflect the maximum treatment given any one sample within the batch.	1 per 20 samples, a minimum of 1 per batch
Spiked samples	A portion of a sample is fortified with all the target analytes before preparation and analyzed independently.	1 per 20 samples per matrix type, a minimum of 1 per batch
Spiked sample duplicates	A portion of a same sample used for the spiked sample is fortified with all the target analytes before preparation.	1 per 20 samples per matrix type, a minimum of 1 per batch
Reference material (standard reference)	A material of known composition, where analyte levels are certified by the manufacturer. These materials should be traceable to NIST standards.	1 per batch of samples

TABLE 2. RECOMMENDED INSTRUMENTAL QC STANDARDS AND SPECIFICATIONS

Name	Use	Specification
ICB—Initial calibration blank	Used for initial calibration and zeroing instrument response.	Calibration standard which contains no analyte. Must be measured during calibration and after calibration. Measured value to be less than 5 times the instrumental detection limit.
Calibration standards	Used to calibrate instrument. The high standard rerun is used to check for high response rollover.	Must be matrix matched to acid content present in sample digestates. Must be measured prior to measuring any sample digestates. Correlation coefficient of ` 0.995, as measured using linear regression on instrument response (y) versus concentration (x). The highest level calibration standard must be measured after calibration. The measured value to fall within ±10% of known value.
ICV—Initial calibration verification	Used to verify calibration standard levels.	Concentration of analyte to be near midrange of linear curve. The ICV is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. Must be measured after calibration and before measuring any sample digestates. Measured value to fall within ±10% of known value.
ICS—Interference check sample (for ICP-AES only)	Used to verify accurate analyte response in the presence of possible spectral interferences from other analytes present in samples.	Concentration of analyte to be less than 25% of the highest calibration standard, concentrations of interferant will be 200 µg/mL of Al, Ca, Fe, and Mg. Must be analyzed at least twice, once before and once after all sample digestates. Measured analyte value to fall within ±20% of known value.

TABLE 2 (CONTINUED)

Name	Use	Specification
CCV—Continuing calibration verification	Used to verify freedom from excessive instrumental drift.	Concentration to be near midrange of linear curve. Must be analyzed before and after all sample digestates and at a frequency not less than every 10 sample digestates. Measured value to fall within ±10% of known value for ICP-AES or FLAA (±20% for GFAA), run 1 every 10 samples.
CCB—Continuing calibration blank	Used to verify blank response and freedom from carryover.	Calibration standard that contains no analyte. Must be analyzed after the CCV and after the ICS. Measured value to be less than 5 times the instrumental detection limit.

SECTION 12

SAFETY, HEALTH, AND HAZARDOUS WASTE

Laboratories must comply with OSHA Standard 29 *CFR* 1910.1450, "Occupational Exposure to Hazardous Chemicals in Laboratories." This regulation requires a Chemical Hygiene Plan that addresses all aspects of laboratory operations.

Certain Pb materials may be classified as hazardous waste. A solid waste containing more than 200 ppm of Pb may fail the TCLP (Toxicity Characterization Leaching Procedure) used to define a hazardous waste (U.S. EPA SW-846 Method 1310 for TCLP, followed by Methods 3050/6010). By failing the TCLP, a waste is classified as hazardous and consequently requires special handling and disposal. Therefore, steps must be detailed in an SOP for the handling of potentially hazardous waste to include compliance with applicable local, state, and federal regulations.

Digests, which are acidic in nature, also contain Pb and perhaps other metals. These digests must be disposed of according to local state and federal regulations.

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SECTION 13

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APPENDIX

ACRONYMS AND GLOSSARY OF TERMS

ACRONYMS

AA	Atomic Absorption
A2LA	American Association for Laboratory Accreditation
ACIL	American Council of Independent Laboratories
AIHA	American Industrial Hygiene Association
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
APHA	American Public Health Association
ASTM	American Society for Testing and Materials
ASQC	American Society for Quality Control
ASTPHLD	Association of State and Territorial Public Health Laboratory Directors
AWWA	American Water Works Association
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Responsibility, Compensation and Liability Act
CDC	Centers for Disease Control
CMD	Chemical Management Division
CNAEL	Committee on National Accreditation of Environmental Laboratories
CRADA	Cooperative Research and Development Agreement
CLP	Contract Laboratory Program
CRM	Certified Reference Material
EDL	Estimated Detection Limit
ELLAC	Environmental Lead Laboratory Accreditation Committee (AIHA)
ELPAT	Environmental Lead Proficiency Analytical Testing (AIHA/NIOSH)
EMPC	Estimated Maximum (Protocol) Concentration
FLAA	Direct Flame Aspiration Atomic Absorption Spectrometry
GFAA	Graphite Furnace Atomic Absorption Spectrometry
GLP	Good Laboratory Practices Standards (TSCA)
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma Emission Spectrometry
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICV	Initial Calibration Verification
ICS	Interference Check Standard
IDL	Instrument Detection Limit
IMVL	Interlaboratory Method Validation Study
ISO	International Organization for Standardization
LCS	Laboratory Control Sample
LOQ	Limit of Quantitation

LSA	Laboratory Systems Audit
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MOU	Memorandum of Understanding
MRI	Midwest Research Institute
NATA	National Association of Testing Authorities (Australia)
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NLLAP	National Lead Laboratory Accreditation Program
NTIS	National Technical Information Service
NVLAP	National Voluntary Laboratory Accreditation Program
OSW	Office of Solid Waste (U.S. EPA)
PE	Performance Evaluation
PM	Preventive Maintenance
PT	Proficiency Testing
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAMS	Quality Assurance Management Staff
QAPjP	Quality Assurance Project Plan
QAPP	Quality Assurance Program Plan
QC	Quality Control
QM	Quality Manual
RCRA	Resource Conservation and Recovery Act
RE	Relative Error
RPD	Relative Percent Difference
SAP	Sample Analysis Plan
SARA	Superfund Amendments and Re-authorizations Act of 1986
SOP	Standard Operating Procedure
SRM	Standard Reference Material Produced by NIST
TCLP	Toxicity Characteristic Leaching Procedure
TPB	Technical Programs Branch
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
XRF	X-Ray Fluorescence
WAL	Work Assignment Leader (L. K. Lowry)
WAM	Work Assignment Manager (J. Scalera)
WPCF	Water Pollution Control Federation

GLOSSARY

Accreditation:	A formal recognition that an organization (e.g., laboratory) is competent to carry out specific tasks or specific types of tests. See also Certification.
Accredited laboratory:	A laboratory that has been evaluated and given approval to perform a specified measurement or task, usually for a specific property or analyte and for a specified period of time.

Acceptance limits:	Data quality limits specified by the National Lead Laboratory Accreditation Program for analytical method performance.
Accuracy:	The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. See Precision and Bias.
Aliquot:	See Subsample
Analytical blank:	See Digestion blank.
Bias:	The systematic error manifested as a consistent positive or negative deviation from the known true value.
Blind sample:	A subsample submitted for analysis with a composition and identity known to the submitter but unknown to the analyst and used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
Calibrate:	To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the calibration standards should bracket the range of planned measurements. See Calibration curve.
Calibration blank:	See Initial calibration blank.
Calibration-check:	See Calibration verification.

Calibration-check standard:	See Calibration verification.
Calibration curve:	The graphical relationship between the known values for a series of calibration standards and instrument responses.
Calibration drift:	The difference between the instrument response and a reference value after a period of operation without recalibration. See Continuing calibration verification.
Calibration standard:	A substance or reference material used to calibrate an instrument.
Calibration solution:	See Calibration standard.
Calibration verification:	See Initial or continuing calibration verification.
Certification:	The process of testing and evaluation against specifications designed to document, verify, and recognize the competence of a person, organization, or other entity to perform a function or service usually for a specified time. See also Accreditation.
Certified Reference Material (CRM):	A reference material that has one or more of its property values established by a technically valid procedure and is accompanied by or traceable to a certificate or other documentation issued by a certifying body. See Certification and Reference material.
Chain of custody:	An unbroken trail of accountability that insures the physical security of samples, data, and records.
Check sample:	An uncontaminated sample matrix spiked with known amounts of analytes, usually from the same source as the calibration standards. It is generally used to establish the stability of the analytical system, but may also be used to assess the performance of all or a portion of the measurement system. See also Quality control sample.

Continuing Calibration Blank (CCB)	A standard solution which has no analyte and is used to verify blank response and freedom from carryover. The CCB should be analyzed after the CCV and after the Interference Check Standard (ICS).
Continuing Calibration Verification (CCV)	A standard solution (or set of solutions) used to verify freedom of excessive instrumental drift. The concentration to be near mid-range of linear curve. The CCV should be matrix matched to acid content present in sample digestates. The CCV should be analyzed before and after all sample digests and periodically throughout the analyses of sample digests.
Control chart:	A graph of some measurement plotted over time or sequence of sampling, together with control limit(s) and, usually, a central line and warning limit(s).
Control sample:	See Laboratory control sample.
Corrective action:	Action taken to correct a deficiency noted in a technical systems audit. See Deficiency and Technical systems audit.
Deficiency:	A failure to fully comply with the requirements of the NLLAP program usually noted during a technical systems audit. See NLLAP and Technical systems audit.
Digestion blank:	A mixture of all reagents used for the digestion of paint, soil, or dust matrices but without the matrix. This blank, is carried through all steps of the analysis starting with the digestion step. This blank evaluates the process for contamination from the laboratory.
Duplicate analyses or measurements:	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation, or storage internal to the laboratory.

Duplicate samples:	Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.
External quality control:	Activities that are routinely initiated and performed by persons outside of normal operations to assess the capability and performance of a measurement process.
Field blank:	A clean sample of matrix (e.g., paint, soil, dust, wipe) carried to the sampling site, exposed to the sampling conditions (e.g., bottle caps removed), returned to the laboratory, treated as an environmental sample, and carried through all steps of the analysis. For example, clean quartz sand, non-Pb containing paint, or a clean wipe could be used as a field blank. The field blank, which should be treated just like the sample, evaluates possible site contamination sources such as airborne contaminants.
Initial calibration blank (ICB):	A standard solution that contains no analyte and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates. The ICB should be measured during calibration and after calibration.
Initial calibration verification (ICV):	A standard solution (or set of solutions) used to verify calibration standard levels. Concentration of analyte to be near mid-range of linear curve which is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. The ICV must be matrix matched to acid content present in sample digestates. The ICV should be measured after calibration and before measuring any sample digestates.
Instrument maintenance log:	A chronological record of preventive and emergency maintenance performed on an analytical instrument. The logs include record of calls, service technician summaries, records of calibration etc.
Interference check standard (ICS):	A standard solution (or set of solutions) used for ICP-AES to verify accurate analyte response in the presence of possible spectral interferences from other analytes present in samples. The concentration of analyte to be less than 25%

of the highest calibration standard, concentration of interferant will be 200 µg/Ml of Al, Ca, Fe, and Mg. The ICS must be matrix matched to acid content present in sample digestates.

Internal quality control: See Intralaboratory quality control.

Internal standard: A standard added to a test portion of a sample in a known amount and carried through the entire demonstration procedure as a reference for calibration and controlling the precision and bias of the applied analytical method.

Intralaboratory precision: A measure of the method/sample specific analytical variation within a laboratory, usually given as the standard deviation estimated from the results of duplicate/replicate analyses.

Intralaboratory quality control: The routine activities and checks, such as periodic calibrations, duplicate analyses, and spiked samples, that are included in normal internal procedures to control the accuracy and precision of measurements.

Laboratory blank: See Digestion blank.

Laboratory control sample (LCS): A matrix-based reference material with an established concentration obtained from a source independent of the instrument calibration and traceable to NIST or other reference materials. The LCS is carried through the entire procedure from digestion through analysis as a field sample. The purpose of the LCS is to evaluate bias of the method.

Laboratory systems audit: See Technical systems audit.

Matrix blank: A sample of the matrix (paint chips, soil, dust) but without the analyte (Pb). This sample goes through the complete analysis including digestion.

Method blank: See Digestion blank.

Method performance: A general term used to document the characteristics of a method. These characteristics usually include method detection limits, linearity, precision, accuracy and bias.

Method detection limit The minimum concentration of an analyte that, in a given

(MDL):	matrix and with a specific method, has a 99% probability of being identified, qualitatively or quantitatively measured, and reported to be greater than zero.
Mobile laboratory:	A mobile laboratory is a self-contained, mobile facility that moves under its own power or is conveyed on a trailer, and does not remain at a site for more than two years.
NLLAP requirements:	Requirements specified by the EPA National Lead Laboratory Accreditation Program (NLLAP) in order to be accredited for lead analysis in paint, soil and dust matrices by an EPA-recognized laboratory accreditation organization.
Precision:	The degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.
Primary standard:	A substance or device with a property or value that is unquestionably accepted (within specified limits) in establishing the value of the same or related property of another substance or device.
Proficiency testing:	A systematic program in which one or more standardized samples is analyzed by one or more laboratories to determine the capability of each participant.
Quality assurance (QA):	An integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality within a stated level of confidence.
Quality assurance program:	See Quality assurance.
Quality assurance coordinator:	See Quality manager.
Quality control (QC):	The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. The aim is to provide quality that is satisfactory, adequate, dependable, and economical.

Quality manager: The manager of the quality system. The Quality Manager is independent of the analyst and reports directly to management.

Reagent blank: See Digestion blank.

Reference material: A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or assigning values to materials.

Reference standard: See Calibration standard.

Relative percent difference: A term defined as

$$RDP \quad \frac{| R_1 \quad R_2 |}{\overline{R}} \times 100$$

where $| R_1 - R_2 |$ represents the absolute difference in two values and $\overline{R}$ represents the average of two values.

Replicate analysis or measurements: The analysis or measurement of the variable of interest performed identically on two or more subsamples of the same sample within a short time interval. See Duplicate analysis or measurement.

Replicate sample: Two or more samples representing the same population characteristic, time, and place, which are independently carried through all steps of the sampling and measurement process in an identical manner. Replicate samples are used to assess total (sampling and analysis) method variance. Often incorrectly used in place of the term "replicate analysis." See Duplicate samples and Replicate analysis.

Report sign-off: The Technical Manager or designee authorized to review and sign analysis reports.

Reproducibility:	The extent to which a method, test or experiment yields the same or similar results when performed on subsamples of the same sample by different analysts or laboratories.
Rinseate blank:	A sample of a "used" cleaning fluid rinse solution, also called an equipment blank. Rinseate blank examples include a final rinse of the device used to collect soil or vacuumed dust or to clean the scoop used to collect soil or vacuumed dust. The rinseate blank is used in rinsing collection media and equipment prior to use to monitor possible cross contamination. The rinseate blank goes through the complete analysis, including the digestion.
Sample log:	The document where sample identification, condition, etc is noted when samples arrive at the laboratory. The log is part of the sample tracking system. See Sample tracking.
Sample tracking:	A system of following a sample from receipt at the laboratory, through sample processing and analysis, and to final reporting. The system includes unique numbering or bar coding labels and the use of a sample log.
Secondary standard:	A standard whose value is based upon comparison with a primary standard.
Site blank:	See Field blank.
Site visit:	An on-site visit to a laboratory for the purpose of conducting a technical systems audit.
Site visitor:	A person who conducts technical system audits. The terms site visitor, auditor and assessor are often used interchangeably. See Technical systems audit.
Spiked matrix:	See Spiked sample.
Spiked reagent blank:	A specified amount of reagent blank fortified with a known mass of the target analyte, usually used to determine the recovery efficiency of the method.

Spiked sample:	A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Spiked samples are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Split samples:	Two or more representative portions taken from a sample or subsample and analyzed by different analysts or laboratories. Split samples are used to replicate the measurement of the variable(s) of interest.
Standard addition:	The procedure of adding known increments of the analyte of interest to a sample to cause increases in detection response. The level of the analyte of interest present in the original sample is subsequently established by extrapolation of the plotted responses.
Standard operating procedure (SOP):	A written document that details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
Standard reference material (SRM):	A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content independent of analytical method.
Standardization:	The process of establishing the quantitative relationship between a known mass of target material (e.g., concentration) and the response variable (e.g., the measurement system or instrument response). See Calibrate and Calibration curve.
Stock solution:	A concentrated solution of analyte(s) or reagent(s) prepared and verified by prescribed procedure(s), and used for preparing working standards or standard solutions.
Stratification:	The division of a target population into subsets or strata which are internally more homogeneous with respect to the characteristic to be studied than the population as a whole.
Subsample:	A representative portion of a sample. A subsample may be taken from any laboratory or a field sample.

Substrate:	This term has a very specialized use in the Pb-abatement area. It refers specifically to the material to which paint is attached, such as wallboard, concrete, wood, steel, etc.
Systems audit:	See Technical systems audit.
Technical systems audit:	A thorough systematic on-site, qualitative review of facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.
Trip blank:	A clean sample, including collection media, that is carried to the sampling site and transported back to the laboratory for analysis **without being opened**. This blank is analyzed as a regular sample through all steps. The trip blank evaluates the integrity of the sample container.
Validation:	The process of substantiating specified performance criteria.
Working standard:	See Secondary standard.