

**R207 – SPECIFIC REQUIREMENTS: ENVIRONMENTAL LEAD  
TESTING LABORATORY ACCREDITATION PROGRAM**

**(LEAD BASED PAINT, ETC.)**

September 2005

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## 1. Introduction

This document describes the accreditation requirements for the environmental lead (Pb) program under the A2LA Environmental Field of Testing. This program for accrediting environmental assessment activities associated with determining lead (Pb) contamination (i.e., laboratory analysis, field testing, and sample collection) is designed to meet the needs of users looking for quality environmental testing services. This program covers the analysis of lead (Pb) in various matrices and includes air, building debris<sup>a</sup>, dust, paint (unapplied), paint chips (residue), soil<sup>b</sup>, and water<sup>c</sup>. Laboratories may be accredited for the environmental lead program only or add the environmental lead program as part of their overall accreditation for other analytes under the Environmental Field of testing.

### NOTES.

- a,b = The analysis of building debris and soil for its Hazardous Waste Characteristics is covered under the A2LA Environmental Field of Testing.
- b = The soil analysis referred to in this program is for the determination of the hazard potential of lead exposure primarily to children under 6 years of age. This analysis involves a strong acid digestion.
- c = The water analysis referred to here is covered under the A2LA Environmental Field of Testing.

Environmental Lead (Pb) Measurement Techniques. This program is designed to accredit laboratories that conduct assessment activities associated with determining the presence of lead in environmental samples and the extent of this contamination. The activities include field testing, sample collection, and laboratory analysis in association with lead contamination suspected of originating from lead-containing paint and other similar sources of lead. This program and the attendant accreditation are available to laboratory organizations that conduct any or all of these activities. The lead of concern is usually found in several matrices that include air, building debris, dust, paint residue (chips), soil, and water. The main test technologies include:

- Atomic Absorption Spectroscopy - Flame (AAS-Flame) & Furnace (AAS-Furnace);
- Colorimetric Methods - HACH Field Methods;
- Inductively Coupled Atomic Emission Spectroscopy (ICP-AES);
- Solid State Anodic Stripping Voltametry (SS-ASV);
- X-Ray Fluorescence Spectroscopy (XRF).

The lead (Pb) testing area is currently undergoing extensive research and regulatory scrutiny that has resulted in a number of efforts to develop methods capable of providing valid analytical procedures for the analysis of lead contamination. A number of these methods are in the final draft stages. This A2LA program endorses the use of regulatory, validated national and/or international consensus methods as appropriate to the matrix of interest. The methods acceptable for use under this program are listed in Appendix A. This methods list will be updated by adoption of the final version of additional regulatory, national and/or international consensus methods or when research or best practice indicates that a specific method is no longer acceptable for use. The U.S. Environmental Protection Agency (USEPA) has developed measurement protocols for several different lead (Pb) measurement methods (40 CFR: 50, 136, 141, 261; and SW 846 3rd Ed.) and has several draft methods undergoing final development. The American Society for Testing and Materials (ASTM) has developed measurement protocols for several different lead (Pb) measurement methods and has a number of draft methods under development. The National Institute for Occupational Safety and Health (NIOSH) has developed measurement protocols for the analysis of airborne lead (Pb) and dust wipe measurement methods. A2LA provides accreditation for any of these methods.

There are strong opinions about the applicability of some of the procedures to certain types of sample matrices or types. A2LA does not intend to recommend the procedures to be used in particular situations except to require that methods used be from the Acceptable Methods List (Appendix A). The application of the method also must remain consistent with its scope. A2LA attests to the competence of laboratories performing to the current state of the art.

The choice of methods used to analyze for lead in environmental samples is very important. Method choice will depend on a number of variables such as sample matrix, concentration range, necessary sample preparation,

detection limit, dynamic range, precision, potential interferences, ease of use, and cost. There are at least seven sources of environmental samples that may be contaminated with lead: air, building debris, dust, paint (unapplied), paint residue, soil, and water. The choice of methods is limited to regulatory methods or, if allowed, those methods of demonstrated performance that are currently regarded as the best available technology and/or method. The methods associated with drinking water and hazardous waste are not listed here but are part of the Environmental Field of Testing. The methods listed in Appendix A apply directly to the analysis of lead (Pb) suspected to have originated from the use of leaded gasoline or lead-based paint. These methods are for analyses of paint, dust, and soil suspected of containing lead (Pb).

Accreditation Alternatives Both A2LA and the American Industrial Hygiene Association (AIHA) have been recognized by the USEPA as accreditors of lead (Pb) testing laboratories in its National Lead Laboratory Accreditation Program (NLLAP).

The A2LA program offers the lead (Pb) testing program with the broader scope of accreditation including organizations engaged in other kinds of environmental assessment activities. The scope of accreditation covers field testing, sample collection, and laboratory analysis including the matrices of air, building debris (hazardous waste), dust, paint (unapplied), paint chips (residue), soil (this program and hazardous waste), and water (drinking).

While A2LA accredits laboratories for any testing which they have been found competent to perform, the NLLAP includes only the matrices of paint, soil and dust and does not cover the analysis of test areas using in-situ techniques.

A2LA and the NLLAP recognize three types of laboratory operations: fixed-site, mobile and field operations, as defined below:

Fixed-Site: An operation that performs analytical testing from a permanent location associated with improved real estate.

Mobile Facility: A transportable, self contained facility that can perform analytical testing under controlled environmental conditions.

Field Operation: A temporary operation, using portable testing technologies, which performs analytical testing on-site, near the sampling location under evaluation.

Significance of A2LA Accreditation A2LA uses as the basis for all of its accreditations ISO/IEC 17025, "General Requirements for the Competence of Testing and Calibration Laboratories." These requirements have become the standard throughout the world and finding a laboratory competent to meet these requirements has become the basis for mutual recognition agreements with accreditation systems in other countries. A2LA currently has in force several foreign agreements and is negotiating with systems from additional countries. In these agreements, test data from accredited laboratories in the United States are accepted as if the laboratories were accredited in one of these other countries as well.

Several testing technologies are available and should be selected as appropriate to the sample type and associated action level (see Appendix C). The Department of Housing and Urban Development (HUD), the Environmental Protection Agency (EPA), the Occupational Safety and Health Administration (OSHA), and the Consumer Product Safety Commission (CPSC) all have legislative and regulatory responsibilities which they must exercise in dealing with problems associated with the use of paint, removal and disposal of lead based paint, paint residue, building debris and contaminated soil.

This program is intended to attest to the competence of organizations engaged in field testing, field sampling, and/or laboratory testing as described in its scope. (For the purpose of this document the term laboratory is used to cover fixed facilities as well as mobile, temporary, and field laboratory and sampling organizations.) This capability can include assessment/abatement activities associated with air, building debris, dust, paint, paint residue, soil, and water. The methods employed will be those required in meeting the HUD requirements, the CPSC requirements, the OSHA requirements, and/or the EPA compliance monitoring for air, water, waste water

and hazardous waste. Accredited laboratories are permitted to advertise the fact of their accreditation. Their scope of accreditation is specific, and users are encouraged to ask to see the scope of accreditation to review those specifics before employing an accredited laboratory. The A2LA website [www.a2la.org](http://www.a2la.org), contains a search directory that includes the scope of testing for each laboratory, and users may always contact the Association for specifics of a laboratory's competence.

## **2. General Criteria**

The general criteria for accreditation of laboratories and/or field testing organizations are contained in ISO/IEC 17025-2005.

## **3. Specific Criteria**

Specific criteria are an elaboration on or interpretation of the general criteria plus those additional requirements applicable to a certain field of testing, testing technology, type of test, or specific test. Lead (Pb) testing laboratories seeking accreditation for mobile or field testing must also meet the additional criteria outlined in the *R104 –General Requirements: Accreditation of Site Testing and Site Calibration Laboratories*.

The numbering system for each section of this document corresponds to the requirements sections of ISO/IEC 17025. The specific criteria applicable to the Environmental Lead (Pb) Program are as follows:

## **4. Management Requirements**

### **4.1 Organization** (No Additions)

### **4.2 Quality System** (No Additions)

### **4.3 Document Control** (No Additions)

### **4.4 Review of Requests, Tenders and Contracts** (No Additions)

### **4.5 Subcontracting of Tests**

4L.5.1 Lead testing included in a laboratory's A2LA Scope of Accreditation shall be sub-contracted only to other NLLAP-approved laboratories.

### **4.6 Purchasing Services and Supplies** (No Additions)

### **4.7 Service to the Client** (No Additions)

### **4.8 Complaints** (No Additions)

### **4.9 Control of Nonconforming Testing** (No Additions)

### **4.10 Improvement** (No Additions)

### **4.11 Corrective Action**

4L.11.1 If the reported values of QC samples fall outside of the acceptance limits stated in the method, samples associated with the batch are to be reanalyzed including a new set of QC samples; no sample values are to be reported unless the QC samples are within the acceptance limits. Laboratories shall document, investigate and take corrective action for all episodes where the QC data shows an out-of-control situation. No data shall be reported until the cause of the problem is determined and corrected, or the laboratory demonstrates the cause was a random event and no longer affects data. The laboratory shall keep records of all out-of-control events, the determined cause(s) and corrective actions taken. Laboratories shall respond to client quality complaints and maintain records of corrective action.

#### **4.12 Preventive Action** (No Additions)

#### **4.13 Control of Records**

The laboratory shall establish and maintain a records system ensuring that:

- 4L.13.1 All observations and calculations are recorded in a permanent manner (such as laboratory/field notebooks, pro-forma work sheets, or magnetic media) at the time they are made and that the units of measurement in which observations are recorded are stated;
- 4L.13.2 When Chain of Custody records are utilized or required (e.g., by the state or client), they shall establish an intact, contiguous record of the physical possession, storage and disposal of collected samples and shall include signatures of all individuals (including laboratory personnel) who had access to individual samples.
- 4L.13.3 Original records are uniquely identified and traceable to the tests or test items to which they refer and to any test reports based upon them;
- 4L.13.4 Records contain sufficient details of any significant departures or deviations from test specifications or other specified procedures including authorizations for such departures;
- 4L.13.5 Records identify the person or persons involved in sampling, preparation, calibration, analysis and final reporting as well as those responsible for checking data transcriptions and calculations;
- 4L.13.6 A log is maintained of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record;
- 4L.13.7 Corrections or amendments to test records are made with a single-line strike-through in a manner that does not obliterate the original data and are signed or initialed by the person responsible.
- 4L.13.8 Test records shall be protected from loss, damage, misuse or deterioration and shall be retained for an appropriate period in a manner that permits retrieval when required. Test records that are created and/or retained on magnetic media (e.g., computer disks) or photographic media (e.g., microfiche) shall be stored in a manner that protects them from the hazards that affect such media and provision shall be made for the printing of such records when required.
- 4L.13.9 The laboratory shall maintain records related to environmental lead (pb) analyses a minimum of 10 years. All hardware and software necessary for the historical reconstruction of data must be maintained by the laboratory. Alternatively, it is acceptable to utilize novel hardware and/or software to recreate historical files. However, the performance of the novel hardware/software must be demonstrated on historic files before they are substituted for the original system.
- 4L.13.10 The records system shall provide for retrievability and traceability of the sample source, the methodology of analysis/testing, results (including calibration and instrument checks), the person performing the analysis, the date, (in the case of mobile laboratories the location where the analytical work was performed);
- 4L.13.11 A secure archive is maintained where access, deposit and removal of records are controlled and documented.
- 4L.13.12 In instances where the laboratory is going out of business, clients of Pb analyses done under the NLLAP, are to be notified 60 days in advance of the closure of the laboratory. The laboratory shall have a plan for ensuring that records are maintained or transferred according to the client's instructions and applicable regulations. All final test reports generated by the laboratory as required in section 13 are to be submitted to the clients if not previously done.

#### **4.14 Internal Audits** (No Additions)

#### **4.15 Management Reviews**

4L.15.1 The quality system shall be reviewed at least once a year by the laboratory management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements.

### **5. Technical Requirements**

#### **5.1 General** (No Additions)

#### **5.2 Personnel**

5L.2.1 Technical Manager. The technical manager (however named) shall possess a college degree in chemistry or a related science and have at least 3 years of non-academic analytical laboratory experience of which at least 2 years shall be metals analysis experience. The technical manager shall be available during at least 50% of the laboratory operating hours to address technical issues for laboratory staff and customers. The technical manager position must be held by a laboratory employee and may not be contracted out.

5L.2.2 Quality Manager. The quality manager (however named) shall possess a college degree in a basic or applied science and have at least 1 year of non-academic analytical chemistry experience and training in statistics. Alternatively, the quality manager can have a college degree in other than the basic or applied sciences, with at least 4 years of non-academic analytical chemistry experience and training in statistics. The technical manager may also function as the quality manager so long as he/she does not act in the position as the sample analyst/technician analyzing the samples or act as the immediate supervisor of the analyst/technician involved with the analysis of the samples. The quality manager may be employed by the laboratory on a part-time basis or as a consultant in order to meet the external monitoring function of the position.

(NOTE: The "training in statistics" requirement can be met through an in-house training course sponsored by the laboratory or chemistry courses including a discussion and application of statistics.)

5L.2.3 Laboratory Supervisors. These individuals shall have a bachelor's degree in chemistry or related field with a minimum of one year of non-academic experience in metals analysis. Successful training in specific metals methods used in the laboratory shall be verified and documented using reference materials of the matrices of concern. Proficiency testing results must be documented. Individuals without a degree in chemistry or a related field can be recognized as laboratory supervisors as long as they meet the training and proficiency testing requirements stated above and have demonstrated to be proficient in metals analyses over a period of at least three years.

#### 5L.2.4 Analyst/Technician.

Analyst: an individual who performs sample analyses and possesses a bachelor's degree in chemistry or a related science.

Technician: an individual who performs sample analyses who does not have a degree in chemistry or a related science.

Analysts and technicians shall have a minimum of 30 calendar days of hands-on experience conducting analyses in an inorganic/metals laboratory before initiation of work on NLLAP-related samples.

5L.2.4.1 Prior to performing analyses on NLLAP samples, Lead (Pb) analysts/technicians shall have completed an external and/or internal training course for Lead or applicable metals analysis

and have demonstrated ability to produce reliable results through accurate analysis of standard reference materials (SRMs), proficiency testing samples, or in-house quality control samples. Their performance must be documented.

5L.2.4.2 Analysts/Technicians in training shall complete a minimum of four independent test runs of sample preparation and/or instrumental analysis. Independent runs are defined as analytical runs consisting of at least five known samples, one of which is a certified reference material or proficiency testing material, separated by a period of time sufficient to evaluate the performance of any previous independent runs.

5L.2.4.2.1 For sample preparation training, the recoveries of the associated reference materials or proficiency training samples for each run must be within  $\pm 20\%$  of the certified value, 75% of the time.

5L.2.4.2.2 For instrumental analysis training, the recoveries of the associated reference materials or proficiency training samples for each run must be within  $\pm 10\%$  of the certified value, 75% of the time.

(NOTE: The reference/proficiency test samples utilized shall: 1) be similar to matrices the analyst will encounter during routine sample analysis, 2) cover the sample mass range for which the analytical SOP has been designed and 3) cover the Lead (Pb) concentration for which the analytical SOP has been designed. In cases where there are several matrices of potential concern, four independent runs will not be sufficient to provide adequate demonstration of performance.)

5L.2.5 Analysts/Technicians involved in Lead (Pb) analyses shall periodically demonstrate their ability to adequately analyze samples for Lead (Pb) based on standard reference materials (SRMs) or certified reference materials. This demonstration shall be done at a minimum of every six months and can be a part of the analysis of proficiency testing materials or quality control samples associated with routine sample runs.

5L.2.6 The laboratory shall have documented evidence contained in their training records of analyst/technician initial and ongoing proficiency for each test method or activity performed on the matrices of concern. The documentation shall include a description of the training program content, the duration of the training, qualifications of the trainer and evidence that the analyst/technician has successfully performed the number of required test runs and successfully analyzed unknown reference samples of the matrices of concern within the specified acceptance criteria.

5L.2.7 The following additional training requirements shall be met for all mobile and field testing personnel:

5L.2.7.1 All mobile and field operation laboratory personnel involved in the designation of sampling areas as part of a Lead-based paint risk assessment in target housing and/or child occupied facilities shall be certified by the EPA or an authorized state or tribal program as a risk assessor pursuant to Section 402 of the Toxic Substance Control Act (TSCA).

5L.2.7.2 All mobile and field operation personnel shall have the capability to communicate with their supervisor or technical manager while on site at a field job location.

5L.2.7.3 All mobile or field operation technicians shall be accompanied by a qualified supervisor for their initial two NLLAP-related job sites.

5L.2.8 The criteria and training requirements for all laboratory personnel (including field and mobile laboratory personnel) shall be clearly defined and documented in the laboratory's quality system documentation.

### **5.3 Accommodation and Environmental Conditions**

5L.3.1 Contamination Control. For fixed-site and mobile laboratory facilities, laboratory dust wipe sampling and analysis shall be conducted at least quarterly to determine surface concentration levels of lead in the laboratory. Sample preparation and analysis is not to proceed until surface contamination is less than the specified maximum allowable concentration of 40 micrograms per square foot, (50 % of the lowest regulatory level). For field testing, appropriate contamination control blank samples shall be analyzed in order to monitor potential Lead contamination.

5L.3.1.1 Labware cleaning procedures shall be specified by the laboratory in a written SOP. The procedure must include a periodic monitoring of lead concentrations in cleaning baths, where applicable, or the monitoring of glassware contamination during the analysis of reagent or other blanks. The monitoring frequency must be at least once a month.

5L.3.1.2 Where the laboratory is responsible for taking dust sample wipes in the field, the laboratory must evaluate blank wipes representative of the lots to be used in the field for lead contamination analysis prior to field sampling.

The laboratory shall:

5L.3.2 Use distilled/demineralized water that it can demonstrate to be free of interferences at detection limits;

5L.3.3 Routinely check and record the conductivity of distilled/demineralized water (for a continuous system, check should be per batch or daily);

5L.3.4 Provide exhaust hoods for volatile materials (per 29 CFR 1910.1450, Occupational Exposure to Toxic Substances in Laboratories and ANSI/AIHA Z9.5-1992, American National Standard for Laboratory Ventilation);

5L.3.5 Have written detailed procedures and facilities in place for collection, storage, and disposal of chemical wastes (40 CFR 261);

5L.3.6 Appropriately store corrosive, reactive, or explosive chemicals safely in conformance with 29 CFR 1910; and

5L.3.7 Laboratory Safety. Laboratory personnel should apply general and customary safety practices as a part of good laboratory procedures. Each laboratory must have a safety and chemical hygiene plan (per OSHA rule 29 CFR 1910.1450) as part of their standard operating procedures. Where safety practices are included in an approved method, they must be strictly followed.

### **5.4 Test Methods and Method Validation**

The laboratory shall:

5L.4.1 Have documented procedures to check the validity of reported analysis values;

5L.4.2 Use at least ACS reagent grade chemicals to prepare standards;

5L.4.3 Use primary standard and QC reference materials;

5L.4.4 Prepare fresh analytical standards at a frequency consistent with good laboratory practices unless otherwise stated in the method (frequency is a function of concentration and type of matrix; generally, the lower the concentration the less stable the standard);

5L.4.5 Properly label reference materials/reagents with concentrations, date of preparation, expiration date and the identity of the person preparing the reagent; and

5L.4.6 Have standards preparation documentation such as a preparations record book.

5L.4.7 SOPs for test methods shall supply or refer to information addressing the following areas:

Interferences	Instrument Calibration
Scope and Application	Applicable Matrix or Matrices
Summary of the Method	Applicable Sample Mass Range
Definitions	Applicable Pb Concentration Range
Safety Considerations	Quality Control Procedures
Apparatus and Equipment	Detailed Step-by-Step Procedure
Reagents and Supplies	Sample Calculations
Sample Preservation and Storage	Method Performance (Accuracy and Precision)
Sample Preparation	Method Detection Limit
Sample Collection (as applicable)	Data Acceptance Criteria
Corrective Actions for Out-of-Control Data	
Contingencies for Handling Out-of-Control Data	
References	

5L.4.8 Acceptable Methodology. Procedures published by federal agencies (e.g., USEPA, NIOSH), nationally or internationally recognized technical authorities are acceptable to use once the laboratory has demonstrated and documented adequate performance of the method for each particular matrix as well as for the sample mass range and concentration of interest. The method performance procedures used must be documented.

5L.4.8.1 Methods under consideration for analytical testing shall demonstrate a quantitation limit equal to or less than 20% of the lowest relevant action level or regulatory limit of interest. For dust wipes it is 50 % of the regulatory requirement.

5L.4.8.2 Alternative or modified analytical methods may be used by a laboratory if they have been validated by the laboratory as meeting the minimum performance requirements specified in this document. The method validation must be documented.

(Note: Acceptable method validation procedures include:

- U.S. EPA Office of Solid Waste "Guidance for Method Development and Method Validation for the RCRA Program, June 1995."
- Guidance on Evaluation, Resolution and Documentation of Analytical Problems Associated with Compliance Monitoring, US EPA Office of Water, EPA 821-B-93-001, June 1993.
- Guidelines for Collaborative Study Procedure to Validate Characteristics of a Method of Analysis, J. Assoc. Off. Chem., Vol. 72, No. 4, 1989, p. 694.
- AOAC Peer - Verified Methods Program, Nov. 1993.)

5L.4.9 Method Performance Evaluation. Linear calibration ranges (or working calibration ranges) shall be established and routinely verified for each method. Method detection limits (MDLs) shall be established and statistically verified at least annually for each method and matrix of concern (paint chips, soil, and/or dust). For methods with stated MDLs, the laboratory shall demonstrate and document its ability to achieve such MDLs. MDLs shall be determined using procedures published or recognized by federal agencies (e.g., US EPA, NIOSH) or nationally or internationally acknowledged technical authorities (e.g., ISO, IUPAC).

5L.4.10 Where subsampling (obtaining sample aliquots from a submitted sample) is carried out as part of the analytical method, the laboratory shall use documented procedures and appropriate statistical techniques to obtain representative subsamples.

## **5.5 Equipment**

### For analytical balances/pan balances:

- 5L.5.1 Analytical balances shall be capable of weighing to 0.1 mg.;
- 5L.5.2 Records of balance calibration shall be kept for at least two ranges (no more than two decades apart) using weights that conform to at least Class 3 tolerances;
- 5L.5.3 Records showing functional/calibration checks each day of use for analytical balances and monthly for other balances shall be maintained; and
- 5L.5.4 The balances shall undergo metrological calibration at least annually.

### For labware and sample collection devices:

- 5L.5.5 All such devices shall be cleaned in a manner appropriate for the analytical procedures for which it is to be used.

### For ovens:

- 5L.5.6 Thermometers shall be graduated in increments no larger than 1°C;
- 5L.5.7 If oven temperature cannot be read without opening the door, the bulb of the thermometer shall be immersed in a sand bath; and
- 5L.5.8 Oven temperature shall be adequately monitored and controlled (e.g., beginning and end of each use cycle).

### For hot plates:

- 5L.5.9 Maintain the center of the hot plate at a temperature sufficient to sustain a mild reflux.

### For microwaves ovens:

- 5L.5.10 Calibrate the power available for heating weekly (This quality control function is performed to determine that the microwave has not started to degrade and that absolute power settings (watts) may be compared from one microwave unit to another).

### For thermometers:

- 5L.5.11 The laboratory shall have access to a NIST (NBS)-traceable thermometer for use in verifying working thermometers;
- 5L.5.12 The calibration of working mercury-in-glass thermometers shall be checked at least annually against a NIST (NBS)-traceable certified thermometer; and
- 5L.5.13 The calibration of dial-type thermometers shall be checked at least quarterly against a NIST(NBS)-traceable thermometer.

### For autopipetors/dilutors:

- 5L.5.14 The apparatus shall have sufficient sensitivity for the intended use; and
- 5L.5.15 Records shall be kept showing delivery volumes are checked gravimetrically at least monthly.

For reagents and standards, the laboratory shall:

- 5L.5.16 Specify their requirements in its documented quality system;
- 5L.5.17 Use ACS reagent grade or the quality specified by the analytical methods in use;
- 5L.5.18 Inspect, verify concentration (if appropriate), date, assign expiration date, and initial upon receipt;
- 5L.5.19 Not use reagents and standards beyond their expiration dates.

## **5.6 Measurement Traceability**

The laboratory shall, as appropriate:

- 5L.6.1 Use quality control materials and calibration standards that are traceable to NIST standards;
- 5L.6.2 Document the frequency, conditions, and standards used to establish calibration of all analytical/testing methodology; and
- 5L.6.3 Verify and document all working standards versus primary (reference) standards.
- 5L.6.4 Instrument performance checks shall be carried out before use for analysis of samples. Such checks shall include, as appropriate, evaluation of instrument sensitivity, noise levels and absorbance/emission levels versus historical values. Acceptance criteria shall be stated.
- 5L.6.5 Calibration curves shall be prepared daily (before the analysis of samples) to adequately cover the expected concentration ranges of the samples using at least 3 calibration standards (except for ICP) and one blank, unless otherwise specified by the method employed. Acceptance criteria shall be stated. New curves shall be prepared whenever an out-of-control condition is indicated and after new reagents are prepared. All calibration curves shall be dated and labeled with applicable method, instrument identification, analysis date, analyte concentration and instrument response information.
  - 5L.6.5.1 When used, the axes of the calibration curve shall be labeled. For electronic data processing systems that automatically compute the calibration curve, the equation for the curve and the correlation coefficient must be recorded. The equation for the line and the correlation coefficient shall also be recorded when the calibration is prepared manually.
  - 5L.6.5.2 A new curve shall be determined if two consecutive analyses of one continuing calibration check sample are outside acceptable limits. When the continuing calibration check limit is exceeded high (i.e., high bias) and there are non-detects for the corresponding analyte in all environmental samples associated with the continuing calibration check, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable check shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Additional sample analysis cannot occur until a new calibration curve is established and verified.
- 5L.6.6 For ICP analyses, where possible, a minimum of a two-point calibration plus a blank shall be performed each day of use before the analysis of samples. Linearity shall be confirmed by the calibration standards, their concentrations encompassing the concentration range of interest for the samples to be analyzed. Analysts using instruments with software utilizing only a single high standard for calibration, are to perform a calibration check using a reference sample with a concentration at the low end of the range of interest. In addition, an interference check standard shall be analyzed each day of use. Acceptance criteria shall be stated (see below).

- 5L.6.7 Calibration blanks must be successfully analyzed before and periodically with the analysis of samples. The calibration blank solutions consist of the same reagents used to digest samples. Performance criteria are stated in section 5L.8. Prior to analyzing samples, an initial calibration verification (ICV) standard must be analyzed. The source of the ICV standard must be independent from the instrument calibration samples and NIST traceable. Performance criteria are stated in section 5L.8. Continuing calibration verification (CCV) standards shall be analyzed in accordance with the analytical SOP. The CCV standard may be prepared from independent reference standards or from the same standards used to prepare the instrument calibration curve. Acceptance criteria shall be stated.
- 5L.6.8 Field testing devices shall be calibrated as required by the testing procedure. Acceptance criteria shall be stated. In the absence of a requirement in the testing procedure, calibration shall be in accordance with the manufacturer's specification.

## **5.7 Sampling (No Additions)**

## **5.8 Handling of Test Items**

The laboratory shall:

- 5L.8.1 Have documented procedures for the collection, shipment, receipt, unique identification (including multiple samples and subsamples), storage and disposal of samples as appropriate.
- 5L.8.2 Give samples an unambiguous sample number when collected and/or logged;
- 5L.8.3 Maintain a permanent record for sample collection and log-in data;
- 5L.8.4 Store samples in such a way as to maintain their identity, integrity, stability, and concentration and document the manner and duration of sample retention;
- 5L.8.5 Follow documented chain-of-custody procedures, when required.
- 5L.8.6 The laboratory shall have a sample custodian who shall be responsible for the sample control/logging. The procedures involved include the control, identity, preservation, condition of samples, and sample handling, storage, and disbursement for analysis. An identification scheme shall be documented and utilized, when applicable, in order to designate sample extracts, split samples and duplicates. The laboratory shall have a person responsible for ensuring that all analyses are performed within any USEPA/HUD or method-specified holding times, where appropriate.
- 5L.8.7 Along with a procedure for sample receipt, sample acceptance/rejection criteria shall be documented as well as procedures for advising field personnel and the client of problems with samples. The sample acceptance policy shall include, but is not limited to, the following areas of concern:
- 5L.8.7.1 Proper, full and complete documentation which shall include sample identification, the location, date and time of collection, preservation type (where relevant), sample matrix and any special remarks concerning the sample;
- 5L.8.7.2 Proper sample labeling that includes unique identification;
- 5L.8.7.3 Use of appropriate sample containers;
- 5L.8.7.4 Adequate sample mass in order to perform the analysis.
- 5L.8.8 If the sample does not meet the sample acceptance criteria, the laboratory shall:
- 5L.8.8.1 Retain correspondence and/or records of conversations concerning the final disposition of rejected samples or fully document any decision to proceed with the analysis of

compromised samples. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

5L.8.8.2 The analysis data shall be appropriately "qualified" on the final report.

5L.8.9 The laboratory shall utilize a permanent chronological record to document receipt of all samples. The following information must be recorded in the laboratory log:

5L.8.9.1 Date of laboratory receipt of sample;

5L.8.9.2 Sample collection date (if known);

5L.8.9.3 Unique laboratory identification code;

5L.8.9.4 Field ID code supplied by sample submitter;

5L.8.9.5 Sample matrix;

5L.8.9.6 Requested analyses, including approved method number, if applicable;

5L.8.9.7 Signature or initials of sample logger (where applicable); for electronic sample logging systems, the identity of the logging operator;

5L.8.9.8 Comments resulting from inspection for sample acceptance or rejection.

## **5.9 Assuring the Quality of Test Results**

5L.9.1 The laboratory shall comply with the quality control (QC) requirements of applicable federal or state environmental or public health agencies when testing specific matrices and the requirement as specified below:

5L.9.2 Quality Control Procedures. The laboratory shall have QC procedures (SOPs) specific to each test technology addressing, as appropriate, the use of:

5L.9.2.1 Reagent/method blank analyses;

5L.9.2.2 Replicate/duplicate or "side-by-side" field sample analyses;

5L.9.2.3 Spiked and blank sample analysis;

5L.9.2.4 Blind samples;

5L.9.2.5 Quality control samples;

5L.9.2.6 Control charts or equivalent;

5L.9.2.7 Calibration standards;

5L.9.2.8 Reference material samples;

5L.9.2.9 Internal standards; and

5L.9.2.10 Split/spiked field sample analyses;

5L.9.3 Quality Control Practices. The laboratory quality control program shall include the continual evaluation of its performance (system process control) for each matrix which includes the

determination of accuracy and precision. The required minimum performance criteria and QC sample frequency are stated below for analytical SOPs employing AAS or ICP in the absence of QC sample frequency determinations based on the use of system process control data produced by the laboratory for the specific method utilized.

- 5L.9.4 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.

Precision is evaluated by the reproducibility of analyses. Precision is commonly expressed as standard deviation or relative percent difference 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 samples where such is the case, the laboratory must select alternative QC options such as the analysis of duplicate laboratory control samples per batch in order to monitor laboratory performance.

- 5L.9.5 Accuracy and Precision Determinations for Paint Chip, Soil and Vacuumed Dust Samples. Matrix spiked samples shall be analyzed with a minimum frequency of five percent (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 one spiked sample for each matrix, per batch shall be digested and analyzed. Replicate (duplicate) samples shall be analyzed with a minimum frequency of five percent (5%) of samples for each matrix, per batch of samples. If there are fewer than 20 samples in a batch, at least one sample for each matrix, per batch shall be analyzed. In the event the analyte is not detected in the sample, replicate matrix spike samples may be analyzed.

- 5L.9.5.1 Matrix spike samples shall be prepared using a split field sample (before any digestion process). When possible, the split sample chosen shall be one identified with the lowest concentration of Lead detected and the level of Lead spiked shall be enough to result in a final Lead concentration of the prepared sample of twice the sample's observed native Lead concentration or five times the method detection limit, whichever is greater.

- 5L.9.5.2 For field samples too small and difficult to homogenize and split in order to obtain samples for matrix spike evaluation or replicate analyses, the laboratory shall select alternative QC options. One of these options is the analysis of duplicate laboratory control samples for each batch in order to monitor laboratory performance.

- 5L.9.6 Accuracy and Precision Determinations for Dust Wipe Samples. When analyzing wipe samples, method spike samples shall be prepared using blank collection media with a minimum frequency of 5%. If there are fewer than 20 samples per batch, at least one method spike/spike duplicate set shall be digested and analyzed per batch. The matrix samples are to be prepared using a lead-based paint (NIST SRM traceable) applied directly to the wipe.

- 5L.9.7 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 shall be processed and analyzed along with the samples. Method blanks shall be analyzed with a minimum frequency of five percent (5%) of the samples for each matrix, per batch of samples. If there are fewer than 20 samples in a batch, at least one method blank for each matrix, per batch shall be analyzed. The use of method blanks provides a measurement of laboratory and/or reagent contamination. Method blanks shall not be used to correct sample results.

5L.9.8 External Reference or Laboratory Control Sample Analysis. Prior to sample analysis, at least one independent lead reference or laboratory control sample (LCS) shall be digested and 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 reference or control sample shall be digested and analyzed per batch per matrix type. The concentration of the control sample shall be within the working range of the method and shall not require extensive pretreatment, dilution or concentration prior to analysis. Sources of these samples include but are not limited to: NIST Standard Reference Materials, proficiency testing samples from the ELPAT Program, commercially available certified reference samples, or samples prepared from different sources of analyte than calibration standards and whose concentrations were determined using definitive methods. All reference or laboratory control sample materials shall be NIST traceable.

(Note: The LCS must be a sample provided in each batch of like matrix for the samples being analyzed. The LCS may not be a pre-digested sample used as a spiking standard solution.)

5L.9.9 Acceptance Limits. Acceptable performance limits for analytical instrumentation as well as each method shall be established based upon the continuing statistical evaluation of 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 shall 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 statistical determination of adequate QC sample frequency, the following minimum QC sample frequencies are required (where applicable) for analytical SOPs employing AAS or ICP instrumentation:

<u>QC Sample</u>	<u>Frequency</u>	<u>Acceptance Limits</u>
Initial Calibration Verification (ICV)	Once per run after calibration	Within $\pm 10\%$ of known value.
<u>QC Sample</u>	<u>Frequency</u>	<u>Acceptance Limits</u>
Initial Calibration Blank (ICB)	Once per run at the beginning of run.	Absolute value not more than 10% of the regulatory limit or minimum level of concern.
Continuing Calibration Verification (CCV)	Before and at the end of a sample run as well as every 10 samples, or every 12 hours, or according to manufacturer's recommendations - whichever is most frequent.	Within $\pm 10\%$ of known value for ICP or FAAS; Within $\pm 20\%$ for GFAA.
ICP Interference Check Sample (ICS)	Beginning & end of each run or twice every 8 hours for ICP analysis.	Within $\pm 20\%$ of known value.

Continuing Calibration Blank (CCB)	After each ICS and CCV.	Absolute value not more than 10% of the regulatory limit or minimum level of concern.
Laboratory Control Sample (LCS)	1 per 20 samples or batch (5%).	Within $\pm 20\%$ of known value.
Matrix Spike	1 per 20 samples or batch (5%).	Within $\pm 25\%$ of known value.
Duplicate Field Sample	1 per 20 samples or batch (5%).	Within $\pm 25\%$ RPD for values $\geq 5$ times the detection limit
Method Blank	1 per 20 samples or batch (5%).	Absolute value not more than 10% of the regulatory limit or minimum level of concern.

5L.9.10 Control Charts. Control charts or a quality control data base shall be used to record quality control data and track laboratory performance with the associated acceptance limits for each matrix and to evaluate instrument performance. Control charts shall specify warning and action limits for acceptance or rejection of QC data. In the absence of a statistically sufficient data base to determine the necessary frequency for QC samples, the laboratory must default to the frequencies outlined in Section 5L.8.

## 5.10 Reporting the Results

5L.10.1 Test reports must be reviewed and signed by the technical manager or his/her designee taking responsibility for the test. Test reports must conform to the documentation requirements of Appendix B when more than twenty (20) analysis results are reported.

5L.10.2 The laboratory shall have documented procedures for data collecting, reducing, reporting and record keeping.

5L.10.2.1 The data reduction and review process shall include, but not necessarily be limited to: comparison of quality control data against established acceptance limits, computation verification, transcription of data and adherence to the procedures established in the laboratory SOPs. The review process shall be documented.

5L.10.3 All final test reports shall undergo the documented data review process before release to the client. All data reviews shall be conducted and signed by a qualified person not directly involved in the physical preparation and/or analysis of the samples in question. Qualified persons are defined as technicians, analysts, supervisors, technical managers and/or quality managers.

5L.10.4 The laboratory shall have documented procedures for correcting erroneously reported results.

5L.10.5 Method Quantitation Limit. The reporting of zero concentration is not permitted. The laboratory shall establish a method of limiting the lower reported values to a positive finite Lead (Pb) level that is appropriate for the technology being utilized. Measured Lead (Pb) levels below this positive finite value shall be reported as "less than" (" $<$ ") this positive finite value. The quantitation limit shall be reported as "less than" (" $<$ ") a value no greater than 10 times the determined method detection limit.

(NOTE: The term "ND" (Not Detected) can be used on test reports in place of "<". However, if "ND" is used, it must be defined on the report as being a value below the quantitation limit of the method being used for analysis and the method quantitation limit must be stated on the report.)

## **6. Proficiency Testing (Performance Evaluation) Requirements**

Please refer to the A2LA Proficiency Testing Requirements for Accredited Testing and Calibration Laboratories.

## **7. A2LA Obligations under NLLAP**

Notification of Accreditation Action. When a laboratory loses its accreditation under this environmental lead(Pb) program, A2LA shall notify in writing the authorized official of the USEPA National Lead Laboratory Accreditation Program within five (5) working days the result of such loss of accreditation.

Notification of Organizational Changes. A2LA shall also notify in writing the authorized official of the USEPA National Lead Laboratory Accreditation Program within thirty (30) days of any decision to implement major changes in organizational policies or management of A2LA that could affect the NLLAP.

Assessor Qualifications. A2LA environmental lead (Pb) assessors shall have attained at least a bachelors degree in chemistry or a related science and have at least 3 years of non-academic analytical laboratory experience of which at least 2 years shall be metals analysis experience. In addition to the current policy and procedures for selection, orientation, training and evaluation that all assessors must complete (as stated in the A2LA Assessor Acceptability Guide), A2LA shall have a training program for novice environmental lead (Pb) assessors and continuing education program for all environmental lead (Pb) assessors based on the most current version, including amendments, of the USEPA developed curriculum guidance document entitled, Pb-Based Paint Laboratory Accreditation: Curricula Recommendations for Assessor Training Programs. The A2LA training curricula now consists of a two-day lead (Pb) technical requirements and a five-day assessor course both with written examinations, which have been reviewed and approved by USEPA. All novice environmental lead (Pb) assessors shall pass both examinations before being used on an assessment. A2LA veteran assessors, other wise meeting the qualifications stated above and under contract before June 1, 1993, must also attend the two-day course and pass the examination before being used as an assessor for laboratories requesting accreditation under the environmental lead (Pb) program.

## APPENDIX A

### METHODS SELECTION LIST

The methods listed are acceptable to use with all the matrices (paint, dust, or soil) unless otherwise noted.

#### 1. SAMPLE PREPARATION.

ASTM E 1644-98: Standard Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead by Atomic Spectrometry.

ASTM E 1645-01: Standard Practice for the Preparation of Dried Paint Samples for Subsequent Lead Analysis by Atomic Spectrometry.

ASTM E 1726-01: Standard Practice for Sample Digestion of Soils for the Determination of Lead by Atomic Spectrometry.

ASTM E 1741-00: Standard Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry.

ASTM E 1979-98: Standard Practice for Ultrasonic Extraction of Paint, Dust, Soil and Air Samples for Subsequent Determination of Lead.

NTIS PB92-114172: Standard Operating Procedure for Lead in Paint By Hotplate- or Microwave-Based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Emission Spectroscopy.

EPA SW-846 Method No. 3050B, Revision 2, December 1996: Acid Digestion of Sediments, Sludges, and Soils.

EPA SW-846 Method No. 3051, Revision 0, September 1994: Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils.

NIOSH Method No. 7082: Lead (in air).

NIOSH Method No. 7105: Lead (in air).

NIOSH Method No. 7300: Elements (in air).

HUD Appendix 14.2: Procedure for the Digestion of Wipe Samples Using Diaper Wipes.

HUD Appendix 14.3: Procedure for the Preparation of Field Spiked Wipe Samples.

#### 2. LABORATORY SAMPLE ANALYSIS.

ASTM E 1613-99: Standard Test Method for Analysis of Digested Samples for Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption (FAAS) or Graphite Furnace Atomic Absorption (GFAAS) Techniques.

NTIS PB92-114172: Standard Operating Procedure for Lead in Paint By Hotplate- or Microwave-Based Acid Digestions and Atomic Absorption or Inductively Coupled Plasma Emission Spectroscopy. Matrix paint chips.

EPA SW-846 Method No. 6010B, Revision 2, December 1996: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).

EPA SW-846 Method No. 7420, Revision 0, September 1986: Lead [Atomic Absorption, Direct Aspiration (FAA)].

EPA SW-846 Method No. 7421, Revision 0, September 1986: Lead [Atomic Absorption, Furnace Technique (GFAA)].

NIOSH Method No. 7082: Lead [in air (FAA)].

NIOSH Method No. 7105: Lead [in air (GFAA)].

NIOSH Method No. 7300: Lead [in air (ICP-AES)].

HUD Appendix 14.1: Laboratory Analytical Procedures.

### 3. FIELD TESTING

ASTM 1753-01: Standard Practice for Use of Qualitative Chemical Spot Test Kits for Detection of Lead in Dry Paint Films.

ASTM E 1775-01: Standard Guide for Evaluating Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead.

### 4. SAMPLE COLLECTION TECHNIQUES

[with quality system documentation covering chain-of-custody, sampling procedures and training].

ASTM E 1727-99: Standard Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques.

ASTM E 1728-02: Standard Practice for Field Collection of Settled Dust Samples Using a Wipe Sampling Method for Lead Determination by Atomic Spectrometry Techniques.

ASTM E 1792-02: Standard Specification for Wipe Sampling Materials for Lead in Surface Dust.

ASTM E 1729-99: Standard Practice for Field Collection of Dried Paint Samples for Lead Determination by Atomic Spectrometry Techniques.

NIOSH Method 9100 (15 August 1994): Lead in Surface Wipe Samples.

NIOSH Method No. 7082: Lead [in air (FAA)]

HUD Appendix 13.1: Wipe Sampling for Settled Lead-Contaminated Dust.\*\*

HUD Appendix 13.2: Paint Chip Sampling.\*\*

HUD Appendix 13.3: Soil Sampling Protocol for Housing.\*\*

## 5. MISCELLANEOUS RELATED PROCEDURES

ASTM E 1605-02: Standard Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures.

ASTM E 1864-02: Standard Practice for Evaluating Quality Systems of Organizations Conducting Facility and Hazard Assessments for Lead in Paint, Dust, Airborne Particulate and Soil in and around Buildings and Related Structures.

\* For NIOSH references see DHHS (NIOSH) Pub. No. 84-100, 4th Ed., 1994, Peter Eller, Editor, Phone: 513 841 4256.

\*\* These sampling procedures are based on guidance provided in the U.S. Department of Housing and Urban Development, Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing.

## APPENDIX B

### DOCUMENTATION REQUIRED FOR LABORATORY RESULTS

Based on HUD requirements the following documentation requirements must be met. [Lead-Based Paint: Interim Guidelines for Hazard Identification and Abatement in Public and Indian Housing, Department of Housing and Urban Development, June 1995].

The laboratory must record and report data such that the data package can be validated and legally defensible. The information and data required can be categorized or classified as listed in the following section. Each classification listed also includes the minimum data elements required. In addition, each classification may be regarded as a separate data form to be completed by the laboratory in its documentation of the analysis. The list for each classification is not exhaustive and is intended to serve as minimum guidance to the laboratory regarding the information it is responsible for collecting, documenting and reporting.

1. Cover Page Information. General information about (Identification of the laboratory) the laboratory, identification of the sample preparation and analytical method employed and the conditions under which these methods were performed are to be presented on the cover page. The information provided must minimally include the following:

- a. Laboratory identification.
- b. Analytical run identification (sample numbers).
- c. Date of report preparation.
- d. Type of measurement detection system used (ICP, GFAA, FAA, etc.).
- e. Type of instrument used.
- f. Identification of the sample (digestion) procedure used
- g. Identification of the analytical method used.
- h. The signature of the authorized laboratory signatories.
- i. General comments and observations about the sample, sample preparation and/or analytical run as appropriate.
- j. Name and address of the client.
- k. Name and address of the laboratory performing the analysis.

2. Sample Information. A list of all samples analyzed in an analysis run (both field samples and QC samples), in the order in which they were analyzed is to be included in this section. Information on the particular sample run is also included. The chronological ordering of the samples allows: QC samples to be matched with the field samples; measurement system control to be evaluated; the data set to be validated; and the demonstration of legal defensibility. The information provided must minimally include the following:

- a. Sample identification
- b. Batch identification
- c. Sample type (paint chips, dust, air, etc.)
- d. Sample weight (in micro-milli-grams)/area (in mg/ft<sup>2</sup>)
- e. Matrix information
- f. Dilution factors
- g. Date (samples) collected and received in the laboratory
- h. Date and time analyzed
- i. Operator information

3. Results of Initial Precision and Accuracy Determination. Results of the initial analysis runs of 4 Matrix Based samples. The information provided must minimally include the following:

- a. Date and time of analysis
- b. Sample identification for the 4 aliquots
- c. Spiked concentrations

- d. Percent recoveries
- e. Mean, standard deviation, and relative standard deviation of the percent recoveries among the 4 aliquots
- f. Flag for problem detected by this analysis
- g. Corrective actions, if any.

4. Results of Calibration. Results of each determination of the calibration curve are summarized on this form:

- a. Date and time of calibration
- b. Identification of the standards
- c. Concentration of the standards
- d. Detection limits
- e. Instrument response
- f. Slope and intercept terms of the fitted calibration curve, if appropriate
- g. Mean-square error and correlation coefficient of the fitted calibration curve, if appropriate.

5. Results of Analysis on Blanks. Results of the analysis of the blank samples listed on the Sample Information form are summarized and minimally contain the following information:

- a. Date and time of analysis
- b. Sample identification
- c. Type of blank [initial calibration verification (ICV), continuing calibration verification (CCV), preparation blank, field blank]
- d. Detection limits
- e. Instrument response
- f. Estimated concentration based on the calibration curve
- g. Flag for problem detected by this analysis
- h. Corrective actions, if any.

6. Results of Calibration Verification. Results of the verification procedure on the calibration curve through verification standard samples are summarized and minimally contain the following:

- a. Date and time of analysis
- b. Identification of the calibration curve
- c. Sample identification of the calibration check samples
- d. Sample type (ICV, CCV)
- e. True concentration level of lead in the ICV and CCV
- f. Instrument response
- g. Estimated concentration based on the calibration curve
- h. Percent difference between the true and estimated concentration
- i. Flag for problem detected by this analysis
- j. Corrective actions, if any.

7. Results of Tests for Accuracy. Results of the analysis on spike samples, laboratory control samples, and linear range analyses are summarized and minimally include the following:

- a. Date and time of analysis
- b. Sample identification
- c. Sample type [regular spike, initial calibration spike, laboratory calibration spike, laboratory reference analyte]
- d. Spiking concentration
- e. Instrument response
- f. Estimated concentration of the sample
- g. Percent recovery
- h. Flag for problem detected by this analysis
- i. Corrective actions, if any.

8. Results of Tests for Precision. Results of the analysis on split and duplicate samples are summarized and should minimally contain the following:

- a. Date and time of analysis
- b. Sample identification
- c. Type of sample (duplicate spike samples, split samples, etc.)
- d. Number of duplicates
- e. Spiking concentration (if any)
- f. Instrument response
- g. Estimated concentration of the sample
- h. Relative percent difference for duplicate spike samples
- i. Relative standard deviation for split and duplicate samples
- j. Flag for problem detected by this analysis
- k. Corrective action, if any.

In addition, laboratories should document the data results from methods used to prevent and adjust for interferences and bias such as serial dilution methods and the Method of Standard Additions. Sources of standards used in the analysis should also be completely documented.

## APPENDIX C

### ACTION LEVELS

Some of the action levels reported here are regulatory; others are guidance from a recognized or responsible authority.

1. Lead free paint is by regulatory definition:  $\leq 0.06\%$  by weight lead (HUD and CPSC, References 1 and 2).
2. The lead level in applied paint coatings requiring abatement:  $> 0.5\%$  by weight or  $> 1\text{mg}/\text{cm}^2$  (HUD, reference 3).
3. The lead level in dust that triggers lead hazard management activities/abatement by sample area type is as follows (HUD, reference 3; EPA, reference 4):
  - a. Uncarpeted Floors ----- 40 ug/sq.ft.
  - b. Window Sills ----- 250 ug/sq.ft.
  - c. Window Wells ----- 400 ug/sq.ft.
4. The lead level in bare soil requiring specified action is as follows: (EPA Reference 4)
  - a. Entire yard ----- 2,000 ppm
  - b. Non-play area portion of the yard ----- 1,200 ppm
  - c. Final level in play area ----- 400 ppm
5. The target lead level in drinking water is zero (EPA, reference 5).
6. The target airborne lead level is  $30\mu\text{g}/\text{m}^3$ .

### REFERENCES.

1. Title 16, Code of Federal Regulations, 1992, Part 1303, Ban of Lead-Containing Paint and Certain Consumer Products Bearing Lead-Containing Paint.
2. Title 24, Code of Federal Regulations, 1992, Part 35, Lead Based Paint Poisoning Prevention in Certain Residential Structures.
3. Office of Public and Indian Housing, Department of Housing and Urban Development, Lead-Based Paint: Interim Guidelines for Hazard Identification and Abatement in Public and Indian Housing, June 1995. These action level procedures are based on guidance provided in the U.S. Department of Housing and Urban Development, Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing.
4. U.S. EPA Federal Register, Vol. 60, N. 244, Monday, December 20, 1995, Part V, Guidance on Identification of Lead-Based Paint Hazards; Notice.
5. Title 40, Code of Federal Regulations, 1992, Part 141, National Primary Drinking Water Regulations.
6. OSHA Occupational Safety and Health Standards, 29 CFR 1910.1025(b), October 11, 1995.

## APPENDIX D

### MICROWAVE OVEN CALIBRATION PROCEDURE

This power evaluation is accomplished by measuring the temperature rise in 1 kg (1.0 liter) of water exposed to microwave radiation for a fixed period of time. Water is placed in a Teflon beaker and stirred before measuring the temperature. The beaker is circulated continuously through the field for 2 minutes with the unit at full power. The beaker is removed, the water vigorously stirred, and the final temperature recorded. The final reading is the maximum temperature reading after the energy exposure. These measurements should be accurate to  $\pm 0.1$ oC and made within 30 sec of the end of heating. The absorbed power is determined by the following relationship:

$$P = \frac{(K)(C_p)(m)(T)}{t}$$

Where:

P = the apparent power absorbed by the sample in watts (W), [W=joule per sec].

K = the conversion factor for thermal capacity or specific heat (cal per gm per degree C) of water.

C<sub>p</sub> = the heat capacity, thermal capacity, or specific heat (cal per gm per degree C) of water.

m = the mass of the water sample in grams.

T = T<sub>f</sub>, the final temperature minus the T<sub>i</sub>, the initial temperature in degrees C.

t = time in seconds (s).

Using 2 minutes and 1 Kg of distilled water, the calibration equation simplifies to: P = (T) (34.87). The power in Watts can now be related to the percent power setting of the microwave unit. The microwave is then calibrated by simply plotting the percent power rating versus the experimentally determined Watts.

## APPENDIX E

### ACRONYMS AND GLOSSARY OF TERMS ASSOCIATED WITH THE PROGRAM

#### ACRONYMS

AA	Atomic Absorption
A2LA	American Association for Laboratory Accreditation
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
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 & Liability Act
CDC	Centers for Disease Control
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
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 (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
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
OSW	Office of Solid Waste (USEPA)
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
TQM	Total Quality Management
TSCA	Toxic Substances Control Act
XRF	X-Ray Fluorescence

## GLOSSARY

Accreditation:	A formal recognition that an organization (e.g., laboratory) is competent to carry out specific tasks (e.g., tests) or specific types of tasks. See also <u>Certification</u> .
Acceptance limits:	Data quality limits specified by the National Lead Laboratory Accreditation Program for analytical method performance.
Accuracy:	The closeness of agreement between a test result and an accepted reference value. The term accuracy, when applied to a set of observed values, will be a combination of a random component and of a common systematic error or bias component. Since in routine use, random components and bias components cannot be completely separated, the reported "accuracy" must be interpreted as a combination of these two components. See <u>Precision</u> and <u>Bias</u> .
Aliquot:	See <u>Subsample</u>
Analytical blank:	See <u>Digestion blank</u> .
Assessment:	The process of obtaining information on an entity to evaluate its conformance or non-conformance with specified criteria. An assessment generally includes or is intended to mean on an on-site visit of the entity.
Assessor:	An individual who carries out some or all functions related to laboratory assessment.
Batch:	A quantity of material produced or processed in one operation, considered to be a uniform, discrete unit.
Bias:	The difference between the population mean of the test results and an accepted reference value. Bias is a systematic error as contrasted with a random error. There may be one or more systematic error components contributing to the bias.
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 <u>Calibration curve</u> .

- 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 ensures 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.
- 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 an assessment. See Deficiency and Assessment.
- Deficiency: A failure to comply with one of the requirements of the program usually noted during an assessment. See Assessment.
- 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.
- 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 that 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  $\mu\text{g}/\text{ml}$  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.

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.

Lot: A set of samples submitted together for laboratory analysis which can be treated as one or more batches.

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 (MDL): The minimum concentration of an analyte that, in a given 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 such as A2LA.

Precision: The closeness of agreement between test results obtained under prescribed conditions. 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: Methods of checking laboratory testing performance by means of interlaboratory tests.

Quality assurance (QA): For laboratories, the activity of providing the evidence needed to establish confidence that laboratory data are of the requisite accuracy.

Quality control (QC): For laboratories, the process through which a laboratory measures its performance, compares its performance with standards and acts on any differences.

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

Quality system: For laboratories, the organization structure, responsibilities, procedures, processes and resources needed to ensure that laboratory services satisfy data requirements.

Quantitation Limits: The maximum or minimum levels or quantities of a target analyte that can be quantified with the certainty required by the data user.

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

$$RPD = \frac{|R_1 - R_2|}{R} \times 100$$

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

Run: A set of consecutive sample measurements.

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.

**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.

**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

Document Revision History

Date	Description
June 29, 2001	General Criteria updated to reference ISO/IEC 17025.  Specific Criteria numbering and order changed to match ISO/IEC 17025.  Document Revision History section added.  No other changes made.
January 9, 2004	Clauses under 4.12 Control of Records removed because of redundancy with ISO/IEC 17025 requirements.  Requirement added to section 4.14 requiring management review of quality system at least once a year.  Redundancies removed from section 5.3 and 5.4.  Update Appendix A, B and C.
September 14, 2004	In Clause 5L.3.1, 20 micrograms per square foot has been changed to 40 micrograms per square foot.
September 13, 2005	Updated to reference ISO 17025: 2005