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Standard Operating Procedure for the Laboratory Analysis of Lead in Paint, Bulk Dust, and Soil by Ultrasonic, Acid Digestion and Inductively Coupled Plasma Emission Spectrometric Measurement

STANDARD OPERATING PROCEDURE FOR THE LABORATORY ANALYSIS OF LEAD IN PAINT, BULK DUST, AND SOIL BY ULTRASONIC, ACID DIGESTION AND INDUCTIVELY COUPLED PLASMA EMISSION SPECTROMETRIC MEASUREMENT

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16. ABSTRACT

The details and performance of a simplified extraction procedure and analysis for three media are provided. Paint, bulk dust, and soil are collected using standard or referenced methods. Paint is ground to the consistency of coarsely ground coffee or cornmeal and bulk dust and soil are sieved using a 60-mesh (250 µm) sieve. Up to 0.25 g of paint, bulk dust, or soil is weighed out and placed in a 50-mL centrifuge tube. Five mL of 25% (v/v) nitric acid is added and the sample is ultrasonicated for 30 minutes. Deionized water is added to a total of 50 mL. The sample is shaken then allowed to settle. The plasma emission spectrometer is calibrated and the samples are analyzed. Quality control samples analyzed include blanks, duplicates, and secondary and primary reference materials. Other quality control activities followed include checking for matrix and spectral interferences. For lead in these three media, the typical minimum detection limit is estimated to be 10 µg/g, bias <20%, and precision <15% RSD.

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SECTION 1.0
PRINCIPLE AND APPLICABILITY

1.1 SCOPE AND APPLICATION

The adverse health effects resulting from exposure of young children to environmental lead (Pb) has received increasing attention in recent years. Studies have shown that chronic exposure even to low levels of lead can result in impairment of the central nervous system, mental retardation, and behavioral disorders.[1,2] Although young children are at the greatest risk, adults may suffer harmful effects as well.[3]

The major sources of exposure to lead in housing units are thought to be paint, dust, and soil. Food, water, and airborne lead are also potential sources but are considered to be minor avenues of exposure. Currently, lead-based paint is receiving emphasis as a critical area of concern and a principal medium for lead contamination and exposure. Although less consideration has been given to soil contaminated with lead from petroleum additives or from the leaching of exterior paint (near driplines, etc.), contaminated soil may be tracked into homes. Like dust, it can collect on hands, toys, and food and be ingested. Concentrations in paint, dust, and soil must be determined if a comprehensive approach to the problem of lead ingestion from housing sources is to be developed.

Under Section 302 of the Lead-Based Paint Poisoning Prevention Act, as amended, Public Housing Authorities (PHAs) are required to randomly inspect all their housing projects for lead-based paint.[4] Currently, the device most frequently used for testing in housing is the portable X-ray fluorescence (XRF) spectrometer, which gives rapid results and is nondestructive. However, uncertainty as to the accuracy and precision of XRF measurements is a major problem, especially near and below the abatement level for paint (i.e., 5,000 μg/g or 1 mg/cm^2).[5] Inconclusive XRF measurements must be confirmed in the laboratory using a more accurate method such as microwave or hotplate digestion followed by atomic absorption spectrometry (AAS) or inductively coupled plasma (ICP) emission spectrometry.[5] This standard

operating procedure describes use of a third lead solubilization method applicable to paint, bulk dust, and soil consisting of an ultrasonic extraction with dilute nitric acid followed by ICP analysis.[6,7]

1.2 SUMMARY OF METHOD

1.2.1 Sampling and Measurement

Samples are collected in the field according to the Department of Housing and Urban Development (HUD) guidelines.[5]

Following a suitable sample homogenization procedure,[8] the sample is extracted using 25% (v/v) nitric acid for 30 minutes in a centrifuge tube placed in an ultrasonic bath. After dilution to a fixed volume with thorough mixing and separation of solids, the lead content is measured by ICP emission spectrometry using the 220.35-nm emission line and the optimum instrumental conditions recommended by the manufacturer.

1.2.2 Quantitation Range, Sensitivity, and Minimum Detection Limit

1.2.2.1 Quantitation Range --

For paint samples, the typical range is 50 to 100,000 μg Pb/g (0.005 to 10 percent) assuming (1) the instrument is linear up to 200 μg/mL, (2) a sample mass of 0.1 g was used, and (3) the final extract volume was 50 mL. The range is 20 to 40,000 μg Pb/g (0.002 to 4 percent) with a 0.25-g sample.

For dust and soil samples, the typical range is 20 to 40,000 μg Pb/g (0.002 to 4 percent) assuming instrument linearity up to 200 μg/mL, a final extract volume of 50 mL, and a sample mass of 0.25 g.

1.2.2.2 Sensitivity --

ICP sensitivity is a function of the photo current integration time as well as other instrumental parameters. However, an indication of ICP sensitivity at a given wavelength is the ratio of net analyte intensity to background analyte intensity, I_n/I_b. For the 220.35-nm line, a reasonable value for this ratio is 50 to 100, which would result in a detection limit of approximately 0.050 μg/mL (50 ppb).[9]

Many ICPs manufactured in the last five years employ torches arranged in an axial configuration as compared to the radial torch configuration used to collect the data for this SOP. These axial systems are considerably more sensitive (at least an order of magnitude) and would be expected to provide correspondingly lower detection limits.

1.2.2.3 Minimum Detection Limit (MDL) --

The typical MDL for lead in paint, bulk dusts, and soils is estimated to be 10 μg/g (0.0010 percent), with the MDL defined as 3 times the standard deviation of replicate (n = 5 to 15) analyses of low-level samples. This value is based on conditions described in Section 1.2.2.1 (0.25-g sample).[10]

Detection limits using ICPs with axial torches may be expected to be considerably lower (see Section 1.2.2.2).

1.2.3 <u>Interferences</u>

Interferences for ICP can be manufacturer and model specific.[9]

1.2.3.1 Spectral Interference --

The efficient excitation of sample constituents at high temperature results in the possibility of spectral overlap interferences. A mathematical correction can be applied for the interference if the interfering element and the magnitude of the interference are determined. As an alternative, an interference-free line may be chosen if the line exhibits an adequate detection limit. Background shifts due to stray light, line broadening, and recombination continuum and other less well-defined sources require correction by background measurement near the analysis line. This correction normally is done dynamically within the instrument.

1.2.3.2 Physical Interferences --

Paint, dust and soil extracts may contain unknown species that affect the efficiency of their nebulization relative to standards, thereby making matrix matching of sample extracts and standards essentially impossible. The existence of these

physical interferences may be checked for by: 1) analyzing a post-extraction spike and determining the recovery of that spike; or, 2) analyzing serial dilutions of the original sample extract and determining if an increase in concentration (after the appropriate dilution factor has been applied) over the original extract of greater than 10% is observed. These effects may be compensated for using either: (1) the method of standard additions; (2) sample dilution; or (3) internal standardization.

1.2.3.3 Chemical Interferences --

Chemical interferences, that is, interactions between molecular and/or ionic species during the emission process, are insignificant for ICP because of the completeness of sample destruction by the high energy of the plasma.

1.2.4 Precision and Bias

In a laboratory evaluation of the method, homogenized real-world paint, bulk dust, and soil samples were analyzed along with the National Institute for Standards and Technology (NIST) Standard Reference Materials (SRMs) using both the subject method and a referee method, microwave acid extraction, and measurement using ICP.[7,11] The accuracy (as percent bias) and precision (relative standard addition [RSD]) achieved are presented in Tables 1 and 2.

The combined extraction-analysis RSDs are summarized as follows:

Paint

$RSD_{0.1\,g}$ = 2% to 10% at Pb concentrations of 0.02% to 11.9%

$RSD_{0.25\,g}$ = 3% to 9% at Pb concentrations of 0.02% to 11.9%

Dust

$RSD_{0.25\,g}$ = 3% to 14% at Pb concentrations of 0.01% to 0.45% (104 to 4,550 μg/g)

Soil

$RSD_{0.25\,g}$ = 1% to 4% at Pb concentrations of 0.06% to 0.26% (581 to 2,690 μg/g)

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Table 1. ICP Measurement of Ultrasonic Extracts of Paint Samples - Comparison to Referee Method Values

Sample	Aliquot Size (g)	Mean Value, % Pb (N)	% RSD	Ref. Value % Pb	Accuracy as % Bias
ELPAT 5P2	0.10 0.25	0.0185 (3) 0.0169 (12)	10.3 6.5	0.022 ± 0.003[a]	-16.7 -23.9
MEM Low Paint	0.10 0.25	0.154 ± 0.016 (5) 0.157 ± 0.015 (5)	10.5 9.4	0.169 ± 0.063[b]	-8.8 -7.1
ELPAT 4P2	0.10 0.25	0.478 (6) 0.505 (4)	5.4 8.7	0.526 ± 0.039[a]	-9.2 -4.0
ELPAT 6P2	0.10 0.25	0.856 ± 0.039 (5) 0.779 ± 0.016 (5)	4.6 2.1	0.899 ± 0.082[a]	-5.0 -13.4
ELPAT 5P1	0.10 0.25	1.79 (5) 1.73 (3)	3.2 2.9	1.76 ± 0.17[a]	1.7 -1.7
ELPAT 5P3	0.10 0.25	4.51 (3) 4.43 (5)	2.0 3.8	4.40 ± 0.58[a]	2.5 0.7
NIST SRM 1579	0.10 0.25	11.6 (3) 11.6 (6)	3.0 4.8	11.87 ± 0.02	-2.6 -2.6

[a] Based on the hot plate and microwave acid digestion and atomic absorption and emission spectrometric analysis performed by over 30 reference laboratories in the AIHA/ELPAT laboratory accreditation program.[12]

[b] Based on hotplate and microwave acid digestion and atomic absorption and emission spectrometric analysis by over 25 laboratories in a round robin study.[8]

Table 2. ICP Measurement of Ultrasonic Extracts of Bulk Dust and Soil Samples - Comparison to Referee Method Values

Sample	Aliquot Size (g)	Mean Value, µg/g Pb (N)	% RSD	Ref. Value µg/g Pb	Accuracy as % Bias
MEM Low Dust	0.25	88.6 (6)	13.9	104 ± 6[a]	-14.8
ELPAT 3D3 Dust	0.25	540 (9)	7.0	551 ± 55[b]	-2.0
MEM High Dust	0.25	4,820 (6)	2.9	4,550 ± 120[a]	6.0
ELPAT 5S3 Soil	0.25	560 (10)	4.0	581 ± 38[b]	-3.6
ELPAT 6S3 Soil	0.25	735 (6)	3.3	790 ± 52[b]	-7.0
ELPAT 4S2 Soil	0.25	2,390 (9)	3.3	2,690 ± 170[b]	-11.2
NIST SRM 2711 (Montana Soil)	0.25	991 (6)	1.4	1,162 ± 31	-14.7

[a] Based on hotplate and microwave digestion and atomic absorption and emission spectrometric analysis by over 25 laboratories in a round robin study.[8]

[b] Based on the hot plate and microwave acid digestion and atomic absorption and emission spectrometric analysis performed by over 20 reference laboratories in the AIHA/ELPAT laboratory accreditation program.[13]

SECTION 2.0
APPARATUS AND REAGENTS

NOTE: Before use, all apparatus should be scrupulously cleaned. The recommended procedure is:

1. Wash with warm laboratory detergent solution or ultrasonicate 10 minutes with laboratory detergent.
2. Rinse and then soak a minimum of 1 hour in 50% (v/v) nitric acid.
3. Rinse three times with doubly deionized water and air dry.

2.1 SAMPLE COLLECTION

2.1.1 Paint

A paint sample collection procedure is described in Chapter 5 of the HUD Guidelines.[5]

2.1.2 Bulk Dust

A standard method for collection of lead-contaminated bulk dust from carpets has been developed by the American Society for Testing and Materials (ASTM).[14] One research device to be considered for bulk dust collection from hard surfaces is the HVS-3 vacuum sampler as modified by the Kennedy-Krieger Institute.[15]

2.1.3 Soil

A standard procedure for collection of soil samples was used in the EPA Urban Soil Lead Abatement Demonstration Project.[16]

2.2 SAMPLE EXTRACTION

The following apparatus and reagents are used in this procedure:

2.2.1 Polypropylene centrifuge tubes with screw caps: 50 mL (PGC Scientifics Corp. No. 000-2090-STR (ELKAY), or equivalent).

2.2.2 Glass rods: 1/4 to 3/8 inch diame..., 6 to 8 inches long, tapered at one end to conform to shape of bottom of centrifuge tube (Pyrex glass, or equivalent).

2.2.3 Sieves: 10 mm and 250 μm (Nalgene #4233-0012 U.S. Standard HDPE sieves, or equivalent), plus collection tray, rubber mallet to tap sieves, and nylon brush and some form of moist wipe to clean sieves.

2.2.4 Glass Mortar and pestle (optional apparatus for sample grinding).

2.2.5 Analytical balance: reliable to ±0.001 g (Denver Instrument Co., Model 400D, XE Series, or equivalent).

2.2.6 Ultrasonic bath: 53 W or greater, 2-1/4-pt capacity (Fisher Model FS5, equivalent or larger). Operation of ultrasonic bath must be confirmed prior to use following one of the procedures given in Appendix A.

NOTE: The centrifuge tubes placed in the ultrasonic bath must be kept in an upright position, with the bath water about 1 inch above the level of liquid in the centrifuge tubes. This can be done by placing the tubes in a rack that will fit into the ultrasonic bath. A rack to hold six centrifuge tubes in the Fisher FS5 ultrasonic bath can be obtained by cutting a section from a larger plastic rack (Fisher Scientific Catalog No. 14-809-30, or equivalent). The bottom of the tubes should be kept from touching the metal bottom of the bath to maximize transfer of ultrasonic energy. The cover of the

bath or some other weight, such as a block of polyethylene or Teflon, must be placed on top of the tubes to keep them immersed in the water in the bath.

2.2.7 Sample riffler (Carpco, Inc. Model SS-16-3 Microsplitter, or equivalent).

2.2.8 Deionized water: Unless otherwise indicated, references to deionized water shall be understood to mean reagent water as defined by Type 1 of ASTM Specification D1193.[17] (ASTM Type I Water: Minimum resistance of 16.67 megohm-cm, or equivalent).

2.2.9 Extraction solution (25% v/v HNO_3): To a 1-liter volumetric flask, add 50 mL of deionized water. Cautiously add 250 mL of nitric acid (Baker Instranalyzed, or equivalent) and dilute to volume with deionized water.

CAUTION: Nitric acid fumes are toxic. Prepare in a well-ventilated fume hood while wearing safety glasses.

2.2.10 Nitric acid cleaning solution (50% v/v HNO_3): Prepare by diluting American Chemical Society (ACS) reagent-grade HNO_3 1:1 with deionized water.

2.2.11 Nitric acid rinsing solution (10% v/v HNO_3): Prepare by diluting ACS reagent-grade HNO_3 1:10 with deionized water.

2.2.12 Chemical fume hood (Kewaunee Scientific Equipment Corp., Adrian, MI. 49221, Airflow Supreme Model or equivalent).

2.3 MEASUREMENT

2.3.1 Inductively Coupled Argon Plasma Emission Spectrometer

Computer-controlled plasma emission spectrometer with background correction

and radio frequency plasma generator. The Leeman Labs Plasma Spec I ICP (or equivalent) may be used.

2.3.1.1 **Argon Gas Supply --**

Ensure that adequate argon, water, and electrical power are available. Liquid argon is the most desirable source of argon, especially for daily use, from a cost and labor perspective. If gas is used, ensure adequate purity.

2.3.1.2 **Cooling Water --**

Recirculating or fresh water that meets flow rate and temperature specifications.

2.3.2 Reagents - Measurement

Master Stock solution: 1,000 μg Pb/mL. Commercial standard; alternatively, weigh out 1.5985 g ACS reagent-grade $Pb(NO_3)_2$ that has been dried for 2 hours at 110° C and dissolve in 200 mL water in a 1-L volumetric flask. Add 25 mL concentrated HNO_3 and dilute to volume with water. Store in a linear polyethylene or Teflon bottle. Stable - 1 year.

Nitric acid diluent: 2.5% v/v nitric acid. Place about 100 mL deionized water in a 1-L volumetric flask. Add 25 mL concentrated HNO_3 and dilute to volume with deionized water. Mix well and store in a linear polyethylene or Teflon bottle.

2.4 Protective Equipment

Safety equipment to be used for protection from both lead and nitric acid during sample extraction and measurement is as follows:

1. Safety glasses with side shields
2. Laboratory gloves, latex, powder-free, disposable (Fisher Scientific Catalog No. 11-393-52 or Equivalent)
3. Laboratory coat

SECTION 3.0
PROCEDURE

3.1 SAMPLE COLLECTION

Samples will be collected as described in Section 2.1.

3.2 SAMPLE PREPARATION

The precision and bias values presented in Section 1.2.4 are based on analyses of paint samples mechanically ground to $<120\ \mu m$ and dust and soil samples sieved to $<150\ \mu m$. Because real-world samples of these materials tend to be relatively non-homogeneous, the quality of the results will depend upon sample preparation. More thorough preparation (e.g., more thorough grinding, sieving, mixing) will improve the quality of the analytical results but will also increase the costs of the analysis.

3.2.1 Paint

Collected paint may be in the form of a chip or chips lifted or scraped off a surface, chips swept off a floor, or small pieces of paint swept, for example, off a window sill. A standardized collection method may be found in the 1995 HUD guidelines.[5] The paint must be ground to a small particle size (equivalent to coarsely ground coffee or cornmeal) to maximize extraction efficiency.

3.2.1.1 Preliminary Treatment --

The first step taken is to remove by hand any foreign objects in the sample, including pieces of wood, plaster, or other debris.

The second step is to weigh the paint sample. If the cleaned or initial sample is less than or equal to about 0.25-g, the entire sample is analyzed; otherwise, only a portion is analyzed.

The third step is to grind the chip(s) in a 50-mL centrifuge tube, beaker, or mortar to particles equivalent to coarsely ground coffee or cornmeal using a glass rod or pestle. If the sample is less than about 2 g, the entire sample is to be ground. If the sample is more than 2 g, use a mechanical riffler (Carpco, Inc. Model SS-16-3 Microsplitter, or equivalent) to split the sample. The sample is split until a final portion of about 1 g is obtained. Alternatively, 1 g may be obtained from the crushed bulk sample by grabbing four to six small portions from different locations in the bulk material using a spatula. The grabbed samples are placed together in the grinding vessel.

3.2.1.2 Grinding --

Weigh and record the weight of a 50 mL centrifuge tube. Place the total original sample (if < 2 g) or sample portion in the tube. Proceed to grind the paint in the tube using a Pyrex glass rod that has been tapered at one end to conform to the bottom shape of the centrifuge tube. The rod should be moved in a rotary manner similar to that used with a pestle in a mortar. Grind until a particle size equivalent to coarsely ground coffee or cornmeal is achieved. This may require 3 to 5 minutes of grinding. Upon withdrawing the glass rod from the tube, tap it gently on the inside of the tube to knock off any particles of paint. Weigh the tube plus contents and record the weight. Take the difference between the empty tube and the tube plus paint to determine the weight of the paint. If the sample is less than or about equal to 0.25 g, the entire sample is analyzed; if the sample is greater than 0.25 g, weigh out between 0.1 and 0.25 g of the ground paint into a second, preweighed, labeled centrifuge tube for analysis.

An alternative to grinding in a centrifuge tube is to use a mortar and pestle. This device will permit preparation of a finer powder. However, the mortar and pestle must be thoroughly cleaned between each sample. This is best done using a brush and a moist wipe to remove all particles. The wipes are placed in a plastic bag as waste. The mortar and pestle are then rinsed in 10% nitric acid (v/v) and deionized

water and thoroughly dried. The mortar and pestle should be constructed of a material (such as glass) with low lead content.

NOTE: Latex paint or layers of latex paint on oil-based paint will not grind well. The latex paint can be ground into small chips 1 - 2 mm in diameter, but these pieces stretch like rubber rather than break into a powder. If the latex-containing sample collected must be aliquoted before extracting, then attempts must be made to grind the latex as finely as possible through several extra minutes of grinding; also, efforts must be made to take an aliquot that is representative of the whole, i.e., includes a representative portion of the latex material. Alternatively, the latex-containing paint can be cryogenically ground. Manual cryogenic grinding is performed by placing the 50-mL centrifuge tube with the paint sample into a small, wide-mouth dewar containing crushed Dry Ice for several minutes to chill the paint, and then proceeding to grind the paint with a glass rod while the tube remains in the Dry Ice.[18] The tube should be capped both before grinding and after grinding to minimize condensation of water vapor into the sample. The paint sample can also be ground using a commercially available cryo-mill (Spex Model 6700 Freezer/Mill, or equivalent)[19] that takes 1 to 2 g of sample and produces a fine powder with several minutes of grinding at liquid nitrogen temperature. The paint is ground in a tube that is a component of the device; the tube must be cleaned between samples.

3.2.2 Bulk Dust

Bulk dust may be swept from a surface or collected using a vacuum cleaner (Section 2.1.2). The dust may be mixed with large amounts of other materials including lint, hair, etc., and other large debris which must be separated from the dust through sieving.

While wearing gloves and a dust mask, remove large foreign objects from the dust with a tweezer. Then stack the 60-mesh (250-μm) sieve on the collection pan. Transfer the dust to the sieve and proceed to gently tap the side of the sieve with a rubber mallet for about 30 seconds. Stir the dust and repeat the tapping process.

Repeat the stirring and tapping process three more times. Then transfer the dust collected in the pan to a preweighed, labeled centrifuge tube and weigh the tube again. The material remaining in the sieve may be saved in another container for further study or emptied into a plastic bag for later disposal. The centrifuge tube is capped and then rotated (pitch and yaw) for about 15 seconds and then rolled for 15 seconds. If the sample is less than or about equal to 0.25 g, the entire sample is analyzed; if the sample is greater than 0.25 g, weigh out 0.25 g of the sieved dust into a second, preweighed, labeled centrifuge tube for analysis.

The sieve and collection pan must be thoroughly cleaned between samples. First, brush the sieve with a soft nylon brush. This should be done in an area where the cleaning process will not contaminate other samples. Then wipe both the sieve and pan with moist wipes. The wipes are placed in a plastic bag as waste. The pan and sieve are allowed to air dry and then inspected visually for any residue. The cleaning process is to be repeated if residue is observed.

3.2.3 Soil

Soil samples are typically scooped from the surface or collected with an auger type of device. The first step is to manually remove large objects such as stones, pebbles, and sticks from the soil.

The next concern is moisture. Samples that are too wet will not sieve properly. Also, analysis of wet samples would add uncertainty to the analysis since percent moisture is unknown. Therefore, some form of drying is recommended. The samples are spread in pans such as aluminum pie tins and allowed to air dry for 8 hours or overnight. The samples in the pan should be mixed occasionally to facilitate drying. The pans should be placed in an area where the samples could not be accidentally contaminated by other operations in progress or by airborne dust.

When the samples appear dry, they are sieved. Place the 60-mesh (250-μm) sieve on the collection pan and the 10-mm sieve on the 250-μm sieve. Place the soil in the 10-mm sieve and, using a gloved hand, gently rub the soil round and round to break up clumps and facilitate sieving. Continue the process until no more material

passes through the 10-mm sieve. Remove the 10-mm sieve and discard the material in the 10-mm sieve.

Next, proceed to manually rub the soil in the 250-μm sieve until it appears that no more material is breaking up into smaller pieces or passing through the sieve.

Separate the 250-μm sieve from the collection pan, discard the material left in the 250-μm sieve and transfer the material in the collection pan to a preweighed, labeled 50-mL centrifuge tube and weigh the tube again. Proceed to mix the soil as described in Section 3.2.2 for bulk dusk. If the sample is less than or about equal to 0.25 g, the entire sample is analyzed; if the sample is greater than 0.25 g, weigh out 0.25 g of the sieved soil into a second, preweighed, labeled centrifuge tube for analysis. Clean the sieving apparatus as described in Section 3.2.2 for bulk dust.

3.3 ULTRASONIC SAMPLE EXTRACTION

WARNING: Safety glasses, a laboratory coat, and gloves are to be worn when performing the extractions with nitric acid. It is recommended that they also be worn during performance of the analysis procedure.

3.3.1 Bulk Samples - Paint

The extraction procedure for powdered paints is as follows:

3.3.1.1 Weigh out 0.1 to 0.25 g (nearest milligram) of sample into a graduated 50-mL centrifuge tube (if not already done under Section 3.2.1.2).

3.3.1.2 With a pipet, add 5 mL of extracting acid (25% v/v HNO_3, Section 2.2.9) and then cap the centrifuge tube.

3.3.1.3 Using an ultrasonic bath that has been tested for acceptable operation (see Appendix A), place the centrifuge tubes in the ultrasonic bath (Section 2.2.6) so that the bath water is about 1 inch above the level of liquid in the tubes, and sonicate the samples for 30 minutes following the manufacturer's instructions.

3.3.1.4 Following sonication, allow the samples to cool and add deionized water (Section 2.2.8) to the 50-mL mark.

3.3.1.5 Shake the tube briefly (5-10 seconds) and allow to settle for 15 minutes. The clear solution (supernatant) above the solid residue is now ready for analysis.

3.3.2 Bulk Samples - Dust

Weigh out a maximum of 0.25 g (nearest milligram) of sample into a graduated 50-mL centrifuge tube (if not already done under Section 3.2.2). Proceed with the extraction as in Section 3.3.1.2.

3.3.3 Bulk Samples - Soil

Weigh out a maximum of 0.25 g (nearest milligram) of sample into a graduated 50-mL centrifuge tube (if not already done under Section 3.2.3). Proceed with the extraction as in Section 3.3.1.2.

CAUTION: Sample aliquots greater than 0.25 g for paints, dust, and soils may result in lower extraction efficiency and therefore a negative bias.[6]

3.4 CALIBRATION

3.4.1 Working Standard, 100 μg/mL

Prepare by diluting 10 mL of the 1,000 μg/mL master stock solution to 100 mL in 2.5% HNO_3. The working standard should be prepared at least weekly; daily preparation is preferred.

3.4.2 Calibration Standards

Normally two to five standards are used for ICP calibration. Typical concentrations are shown in the following table. Prepare daily by diluting the working standard as indicated.

Volume of 100 μg Pb/mL working standard, mL	Final volume, mL	Concentration, μg Pb/mL
0	100	0
1.0	200	0.5
3.0	100	3.0
10.0	100	10.0
30.0	100	30.0
100.0	100	100.0

Higher lead concentrations may be used as long as the linearity of response is verified.

3.4.3 Calibration Curve

The calibration curve (integrated photocurrent [or equivalent] vs. concentration) will be calibrated automatically. When first calibrating the system or after any significant change to or work on the instrument, a manually plotted standard curve should be prepared and then compared to the standard curve calculated by the system. Any difference in the curves of more than 10 percent needs to be investigated and corrective action taken.

3.5 QUALITY CONTROL

Quality control is necessary to ensure that resulting data are of adequate quality. These are described in the following sections.

3.5.1 Blank Check

Laboratory or reagent blanks are analyzed to determine the background or contamination levels. Contamination levels above the detection limit must be accounted for and eliminated, if possible, before proceeding with sample analysis.

3.5.2 Duplicates

It is recommended that one duplicate sample be analyzed for every 20 samples. A duplicate (split) sample is one brought through the whole sample preparation and measurement process. The duplicate analysis gives a measure of overall precision, which is a combination of the precision of sample preparation (grinding, sieving, mixing), sample extraction, dilution (if necessary), and analysis.

A suitable method for evaluating the acceptability of the results of a duplicate analysis is through calculation of the maximum acceptable Relative Percent Difference (RPD) between the individuals in a duplicate pair. The RPD is calculated as

$$100 \cdot |X_1 - X_2| / [(X_1 + X_2)/2]$$

where X_1 and X_2 are values of the individuals of a duplicate pair. The maximum acceptable RPD will vary with the proximity of the analytical result to the MDL, the precision of the method, and the acceptable probability of a false positive wherein a "false positive" would be failing the QC check when, in fact, the true precision (unknown) is at an acceptable level. On the basis of laboratory tests and limited field use, the true relative standard deviation (σ) of the method used in the field is estimated to be 100% at 0 to 2 times the MDL, 15% at 2 to 10 times the MDL and 7% at >10 times the MDL.[6] A reasonable probability for a false positive is 5%. Under these limitations, maximum allowable RPDs, which are calculated as 2.77σ,[20] are as follows:

Average Analyte Concentration (Multiples of Minimum Detection Limit, MDL)	Maximum Acceptable Relative Percent Difference, RPD
0 - 2	277%
2 - 10	42%
>10	19%

The procedure is to choose a sample for duplicate analysis. It is preferable for the lead level to be >2 x MDL. Perform the duplicate analysis and calculate the RPD. If the calculated RPD value exceeds the maximum acceptable value given above, corrective action is to be taken including review of all original data and calculations, and possible analysis of a second duplicate sample.

3.5.3 Reference Materials

Depending on the matrix, it is recommended that a secondary reference material be analyzed once per sample batch or, at a minimum, once per day to check the entire extraction/analysis procedure. Environmental Lead Proficiency Analytical Testing (ELPAT) materials, which are available from the American Industrial Hygiene Association, Fairfax, VA, may be used as secondary reference materials, or such materials may be purchased from commercial suppliers of reference materials.

It is recommended that a primary reference material (SRM) be analyzed once per week. Lead recovery should be within 80 to 120 percent of the known value. Appropriate SRMs for paint, which are currently available from the National Institute for Standards and Technology (NIST), Gaithersburg, MD, include NIST 2580 (nominal 4% Pb), 2581 (nominal 0.5% Pb), 2582 (nominal 200 mg Pb/kg) and 2589 (nominal 10% Pb). Suggested SRMs for dust are NIST 2583 (nominal 90 mg Pb/kg); and 2584 (nominal 1% Pb) and suggested SRMs for soil are NIST 2709 (Agricultural Soil, 18.9 $\pm$ 0.5 μg Pb/g), 2711 (Montana Soil II, 1162 $\pm$ 31 μg Pb/g), 2710 (Montana Soil I, 5532 $\pm$ 80 μg Pb/g), 2586 (500 mg Pb/kg) and 2587 (3000 mg Pb/kg). If the

sample is out of control, sources of error must be identified and appropriate corrective action taken.

3.5.4 Matrix Interferences

It has been observed that the high concentrations of dissolved materials in paints, soils and dusts depress the values measured by ICP.[7] Testing for physical interferences may be performed by either (1) spiking the sample extract with lead solution and determining the recovery of the spike or (2) performing serial dilution(s) of the extract and determining if there is a significant increase (>10%) in the lead value from the analysis of the diluted sample(s) compared to the analysis of the original extract. These checks as well as compensation techniques are described below.

3.5.4.1 Method of Addition (Extract Spike) Check --

Aliquots of extracts representing each source of paint, dust and soil samples are spiked with lead solution after initial analysis to approximately double the original extract concentration, and then analyzed. The spike recovery must be within 80 to 120 percent of the expected value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix interference/suppression is likely. The use of a standard-addition analysis, such as the Method of Standard Addition (MSA) procedure can usually compensate for this effect.

CAUTION: The standard-addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

3.5.4.2 Dilution Check --

Matrix suppressions in the nebulizer can also be tested for by analyzing a set of serial dilutions of the original extract (e.g. 1:9, 1:24, 1:99). An increase in the

lead value of more than 10% (properly corrected for the dilution) indicates a matrix effect. Such a dilution test should be performed for each new matrix type. The final dilution ratio used will be limited by the lead concentration, which should be at least 1 µg/mL and within the linear range of the calibration curve. If all samples in the analysis batch are sufficiently high in lead content, then sample dilution may be used to compensate for this type of suppression. This technique should not be applied to sample extracts with a lead content < 100 times the instrument detection limit.

3.5.4.3 Matrix Interference Compensation by Internal Standardization --

A fairly simple and effective means of compensating for matrix effects while allowing for determinations near the detection limit is internal standardization. Internal standardization (I.S.) allows the analyst to correct the signal without compromising the detection limit. Most instrumentation manufactured in the past five years provides the analyst with automated I.S. correction through the instrument software. The analyst spikes all standards, field samples, and QC sample digests with an element not likely to be present in detectable quantities in the sample background (such as yttrium [Y]), ensuring that the I.S. concentration is identical for all standards and samples. The signal from both lead and the I.S. is then measured by ICP. Any suppression of signal should be equivalently proportional for both elements. The instrument software then recalculates the lead concentration based on the ratio of the I.S. concentration in the sample to the I.S. concentration in the standard. For example, using a 5 µg Y/mL final known concentration in all standards and samples, it is found that the measured yttrium signal in the sample is 4.5 µg/mL or a 10% suppression. The instrument software then automatically corrects (increases) the lead signal in the sample by a factor of 5/4.5 or 1.111 to compensate for the matrix effect. This technique compensates for subtle differences in both sample and acid matrix in the sample digest. If added to the digest prior to dilution to volume, this approach will also correct for slight errors in sample dilution.

Consult the ICP operator's manual for more detailed instructions.

3.5.5 Spectral Interferences

When lead is being measured by ICP, it is important to be aware of the potential for spectral interferences due to the existence of potentially high levels of interferences (e.g., Ti, Al, Cr). It is important to periodically analyze interfering element check samples that contain known high levels (200-1,000 µg/mL) of each suspected interfering element. Such solutions are available from a variety of vendors. Once the solutions are analyzed, the data must be evaluated to determine the existence of false lead values that are more than 2 times the solution detection limit. If the false values do exceed this criterion, an interfering element correction factor (F_{IEC}) must be determined as follows:

$$F_{IEC} = \frac{\text{False analyte signal}}{\text{Concentration of interferant}}$$

For example, 1,000 µg/mL of aluminum causes a false lead signal of approximately 0.250 µg/mL (7 x DL_{Pb}) for many optical systems. Therefore, F_{IEC} = (0.25/1000) = 0.00025. This value is then used to correct lead data in the presence of high aluminum. The interfering element identified in the above manner is therefore added to the analytical program. This procedure must be applied to all potential interfering elements. The presence and magnitude of interferences will vary by instrument due to differences is optical designs. Follow the operator's manual to establish automated interfering element correction through the instrument software.

3.5.6 Calibration Check Samples

High and low independently prepared check samples are to be run alternately after every 10 samples to determine that calibration has not drifted. If a change of more than 10 percent is measured, the system must be recalibrated and all samples run since the last calibration check must be reanalyzed.

The results should be plotted on a control chart at the end of each sample analysis session, although real-time checking is preferred.[21] The analysis is concluded to be out of control if any one or more of the following criteria is met.

1. One or more points outside of the control limits.
2. A run of at least eight points, where the type of run could be either a run up or down, a run above or below the center line, or a run above or below the median.
3. Two of three consecutive points outside the 2-sigma warning limits but still inside the control limits.
4. Four of five consecutive points beyond the 1-sigma limits.
5. An unusual or nonrandom pattern in the data.

3.6 SAMPLE DETERMINATION

3.6.1 ICP

1. Ensure that adequate argon, water, and electrical power are available. Liquid argon is the most desirable source of argon, especially for daily use, from a cost and labor perspective. If gas is used, ensure adequate purity.
2. Adjustment of Nebulizer Spray - See operator's manual for procedure.
3. Ignition of Torch - Check that argon supply is on.
4. After startup, be sure plasma does not flicker or present an orange corona around torch. If the plasma flickers, be sure the spray chamber is draining properly. If the orange corona is observed, make sure that the nebulizer argon is on. Otherwise some residual salt may be present in the nebulizer spray that must be flushed out or the entire spray chamber assembly must be cleaned.
5. Warmup - Allow the instrument to warm up at least 30 minutes to permit the standard readings to stabilize and allow analyses to begin.
6. Optical Calibration/Torch Alignment Procedures - Before analytical calibration procedures are performed, it is important to perform the optical calibration procedures and the torch alignment operation. Each of these is described in the operator's manual.

7. Select a program that includes wavelength, integration time, number of replicate readings, sample uptake time, and rinse time. Consult the instrument operator's manual for other parameters, such as plasma forward power, gas pressures and flow rates, etc.

8. Aspirate the calibration standards and establish a calibration curve.

9. Run a calibration check sample as described in Section 3.5.6.

10. Aspirate a sample solution and obtain the concentration readout.

11. Analyze a calibration blank at least every ten (10) samples. This result must be less than three (3) times the instrument detection limit. If not, sample carryover due to an insufficient rinse time setting is likely. Correct the problem and reanalyze all samples measured since the last successful calibration blank analysis.

12. Analyze a check sample (Section 3.5.6) every ten (10) samples to evaluate instrument drift. This result should be within 10 percent of the expected value. If not, recalibrate the instrument and reanalyze all samples measured since the last successful check sample analysis.

13. Analyze the Interfering Element Check Sample(s), (See Section 3.5.5) to ensure that the "false" lead signal is less than three times the instrument detection limit.

14. Conclude the ICP measurement run with successful calibration blank and check sample analyses.

3.7 WASTE DISPOSAL

All leftover extract solutions, reagent wastes, and rinse water are poured into a carboy for waste containment. Any centrifuge tubes or other vessels containing nitric acid as one of the reagents are to be rinsed with water before being placed in a plastic bag for disposal or reuse after cleaning. The waste collected in the carboy is disposed of according to applicable regulations.

SECTION 4.0
DATA PROCESSING

4.1 DIRECT DETERMINATION

For direct determination, read the element value (μg/mL) from the calibration curve or readout. If dilution of the sample has been performed, then

μg/mL Pb in the sample = μg/mL in the diluted solution X D

$$\text{Where } D = \frac{(\text{mL of aliquot}) + (\text{mL of diluent})}{\text{mL of aliquot}}$$

4.2 CALCULATION - FIELD SAMPLE CONCENTRATION

4.2.1 Area Concentration

The area concentration of lead in a paint chip is calculated as follows:

$$\text{mg Pb/cm}^2 = (C_{TS} \times V_{TS} \times M_{OS}/M_{SA})/(1000 \times A_{OS})$$

where
- C_{TS} = lead concentration in test solution, corrected for dilution, μg Pb/mL
- V_{TS} = volume of sample digest solution, mL
- M_{OS} = mass of original sample, g
- M_{SA} = mass of sample aliquot digested, g
- A_{OS} = area of original sample, cm^2.

4.2.2 Mass Concentration

The mass concentration of lead in a paint chip is calculated as follows:

$$\mu\text{g Pb/g} = (C_{TS} \times V_{TS})/M_{SA}$$

where
- C_{TS} = lead concentration in test solution, corrected for dilution, μg Pb/mL
- V_{TS} = volume of sample digest solution, mL
- M_{SA} = mass of sample aliquot digested, g.

SECTION 5.0

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APPENDIX A

PROCEDURES FOR TESTING OPERATION OF ULTRASONIC BATH

Procedure for Testing Operation of Ultrasonic Bath

The test is performed using apparatus available from:

Blackstone Ultrasonics
P.O. Box 220
Jamestown, NY 14702-0220

The apparatus is sold as a Performance Kit, Part No. 4050114. The general procedure is to place a set of ceramic rings coated with pencil lead into the ultrasonic bath. The bath is operated for a set period of time and the amount of pencil lead removed is taken as an indication of acceptable or unacceptable operation. The Performance Kit consists of the following items.

- Qty (5) Ceramic Rings
- Qty (5) Pencils
- Qty (1) Bottle of Water Wetter

The procedure to follow is:

1. Fill the ultrasonic tank with water to a depth of 12 inches and add two ounces of Water Wetter.

2. Bring the water temperature to 100°F.

3. Using a pencil supplied with the kit, coat the smooth side of each of the 5 ceramic rings with pencil lead. At least 95% of each ring's surface should be covered.

4. Place the rings, face up, in an "X" pattern in the cleaning basket. One ring should be in the center of the basket and the others approximately 1 inch from each corner.

5. De-gas the liquid in the tank by operating the ultrasonics continuously for 10 minutes.

6. Turn OFF the ultrasonics and allow the system to settle for 30 seconds.

7. Turn ON the ultrasonics and lower the basket into the tank. Start timing as soon as the ceramic rings are immersed in the liquid.

8. Gently move the basket up and down approximately ½ inch for thirty seconds.

9. After 30 seconds, turn the ultrasonics OFF and remove the basket.

10. Compare each ring with the comparator chart provided in the kit. (The chart shows ten rings ranging from essentially no removal of pencil lead [score of 1] to essentially total removal of the pencil lead [score of 10]). From the chart, grade the rings accordingly.

11. Add the five scores together. A total of 30 is acceptable.

Alternative Procedure for Testing Operation of Ultrasonic Bath

This alternative procedure has been provided by Mr. Lee Seaman, formerly of Pace Environs, Inc., and now of J and L Environmental Services, Elfers, FL.:

1. Obtain any standard "household" aluminum foil. Standard foil rather than "freezer thickness" foil should be used. Cut a piece of the foil large enough to cover ½ to 3/4 of the tank bottom. Since there are differences in foil manufacturing, try to buy enough foil at one time to keep as a working standard.

2. Fill tank with warm water (approximately 120° F) with a known amount of wetting agent. Three drops of a dishwashing detergent will be sufficient.

3. Run bath for 5 minutes to properly degas the solution.

4. Turn ultrasonics off and lower foil into the tank at an angle to avoid air getting trapped under the foil. Foil should end up almost parallel and centered to bottom.

5. Turn ultrasonics on for 45 seconds. Turn off machine and remove foil.

6. Examine the piece of foil. Notice a peening effect and/or perforations on the foil. The location and size of these peening and/or perforation patterns indicate the cleaning intensity and uniformity of the ultrasonic sound waves throughout the tank. If the foil appears unchanged following this process, the unit most likely needs repair.

7. Date and save foils in a protective box. If foils show a sudden decline in the magnitude of the peening effect and/or perforations, the unit may need to be repaired.