Open Vessel Microwave Assisted Sample Preparation for Lead Analysis In Paint Chips, Wipes and Soil

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Lead pollution from manmade materials is reasonably controlled today, however lead pollution remains a concern because of remediation of various sites which are suspected environmental and or occupational health hazards. Indeed section 302 of the Lead-Based Paint Poisoning Prevention Act requires Public Housing Authorities to randomly inspect all housing projects under their control for lead based paint. The existence and abatement of lead based paints has placed a burden on analytical laboratories as they are called upon to perform increasingly large numbers of lead determinations. Sample preparation in general is the rate-determining factor in sample turn around time.

A study was done to develop a method to reduce the sample preparation time as well as provide a safe effective procedure for lead determination. The critical components of the study (Slide 1) include the objective of the work, a discussion of the equipment used, an audit of the details in the optimized procedure, and examination of the analytical results and the conclusions drawn from the study.

The objective in this study (Slide 2) was to develop a method which would provide a quick sample preparation technique for lead analysis in paint chips, wipes and soil. The success of the procedure was dependent on the development of a digestion vessel set which was safe, easy to use, disposable, and therefore inexpensive. These features allowed for rapid turn around time and high sample throughput.

Several pieces of equipment were necessary to execute the developed procedure. (Slide 3) Digestions were carried out in an MDS-2000 CEM microwave digestion system with a Thermo-Optic temperature control system, which incorporates a fiberoptic temperature probe connected to the MDS-2000 on-board computer for feedback control. (Note: An MDS-2100 equipped with a Thermo-Optic temperature control system is a viable substitute with slight microwave power parameter changes). The vessel set, which was developed for this work, was the CEM DV-50 high throughput vessel set. A Fisher Scientific Centrifuge model 225 Centrifuge which allowed for centrifugation of the diluted samples after digestion in the DV-50 vessel was employed. Analyses were carried out using a Thermo Jarell Ash 61E Trace ICP-AES (internally coupled plasma-atomic emission spectrometer).

The DV-50 Vessel set (Slide 4) was designed to allow one to thirty six samples to be processed simultaneously. It includes a turntable designed to accommodate the DV-50 vessel(s), and a fixture to support the fiberoptic temperature probe. The vessel is an open transparent, disposable fifty milliliter polypropylene tube molded in a conical shape. The vessels are equipped with a modified cap to allow gases to escape. The temperature control limit for this work was 115 °C. The fiber optic probe was inserted in a glass thermowell contained in one of the DV-50 vessels. The thermowell is designed such that the fiberoptic temperature probe tip was in the optimum location for temperature measurement..

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1 Lead-Based Paint Poisoning Act, 42 U.S.C. 4822 (d) (2) (A), 1971.
The following procedure (Slide 5) was developed as a safe high throughput preparatory technique for lead analysis in paint chips, soils and wipes. The samples were weighed directly into tared DV-50 vessels. A 0.1g sample weight was used for soils and paint chips. Concentrated nitric acid was then added to the vessels. Five milliliters of nitric acid was used for paint chips and ten milliliters was used for soils. Vessels were capped, positioned in the turntable and then placed in the microwave cavity. The fiberoptic temperature control probe was placed in a control vessel. Vessels were heated to 115 °C and held at that temperature. The time at temperature for paint chips was fifteen minutes and for soils thirty minutes. Samples were allowed to cool, then diluted to the fifty milliliter line on the DV-50 vessels with deionized water. Samples were then shaken to mix and centrifuged at ten G for approximately three minutes. The samples were aspirated directly into the ICP (or any desired piece of analytical instrumentation). [Note: Depending on the analytical range of the instrumentation some samples may require additional dilution before aspiration.]

(Slide 6) The TJA 61E Trace ICP-AES used for analyses was equipped with a Meinhard nebulizer, cyclone spray chamber, and a standard quartz torch. The wavelength of the lead line used was 220.353 nm. This system utilizes axial viewing of the plasma along with Crawford-Kunselman noise reduction to allow for GFAA like detection limits for lead, while maintaining the speed of ICP. The detection limit for this work was determined to be 1.02 ng/mL (ppb) [3 sigma]. The nebulizer gas flow rate was set at 0.6 L/min, the auxiliary gas flow was set at 0.5 L/min. and the ~RF power was set at 950 watts.

Three different standard reference material matrices were used in this study. (Slide 7) Paint chips and soils from ELPAT (Environmental Lead Proficiency Analytical Testing) and NIST (National Institute of Standards and Technology), and dust wipes from ELPAT.

The lead recoveries from ELPAT paint chips utilizing the microwave method and the certificate values were compared. (Slide 8) The results for all four proficiency samples showed the microwave lead recoveries fitting within the certificate range. The precision of the technique was expressed as a percentage relative to the standard deviation (%RSD). The results were all determined based on a population of thirty six (one DV-50 turntable) separate digestions.

Lead recoveries from NIST 1579a standard reference powdered paint chips were compared to the certified values. (Slide 9) The microwave data concerned us due to low lead recovery, well outside any acceptable range as compared to the certified value. The work was repeated with the same results obtained. We contacted NIST and were informed that many users of this material were coming up with low lead recoveries. We were informed there was a fraction in the sample which was insoluble in nitric acid. We were advised, in order to obtain complete lead recoveries, one must use hydrofluoric and perchloric acids. Because of the oxidizing properties of perchloric acid and the hazards associated with hydrofluoric acid, use of these acids in the DV-50 vessel system can be unsafe. Remembering our charge was to develop a safe, high throughput preparatory
procedure of lead analysis, we did not perform any digestions using hydrofluoric nor perchloric acids.

Utilizing an optimized procedure of ten milliliters of nitric acid and a thirty minute heating time, lead recoveries were obtained for ELPAT soil proficiency standards and compared to the certificate values. (Slide 10) The microwave lead recoveries were within the certificate ranges for all four soils. The %RSD’s showed good precision, lower than that of the proficiency material. All microwave statistics were based on a sample size of thirty six separate digestions.

We looked at lead recoveries from NIST standard reference soils; SRM #2709 (San Joaquin Soil), #2710 (Montana Soil I), and #2711 (Montana Soil II). NIST provides certificate values for total analytes and extractable analytes. Informational values for extractable analytes are based on the EPA method 3050, which is a hot acid leach. Lead is one of the extractables for which data is provided. Our developed method was also a hot acid leach therefore this informational data was an excellent comparison source. Shown are the recoveries compared to the 3050 leach reported values. (Slide 11) There was close agreement for the mean values, and the range of microwave results were within the certificate ranges. The mean microwave values were based on thirty six separate digestions.

Mixes were prepared of the NIST SRM’s (2709, 2710 and 2711) in order to test a more comprehensive analytical range. The three SRM’s (2709,2710, and 2711) combined with the two mixtures (1:1 2709:2711 mix and 1:1 2710:2711 mix) gave a concentration range between 13 and 5100 ppm. A graphic representation of the lead recoveries for these samples is presented to depict the comparison of the two preparatory techniques (Slide 12). The correlation coefficient of one (R²=1), shows that the method is applicable over the entire dynamic range.

The last sample matrix looked at was what are commonly called dust wipes. The dust wipe was a nine centimeter cellulose filter paper, obtained from the ELPAT program. Ten milliliters of nitric acid was used with a heating time of thirty minutes and a control temperature of 115 °C. The data demonstrated an agreement between the microwave DV-50 recovered value and the ELPAT certificate value for lead in dust wipes. (Slide 13)

(Slide 14) In conclusion, the microwave methods demonstrated a quick efficient sample preparation procedure for lead determination in paint chips, soils and dust wipes. Thirty six samples were acid digested in thirty minutes with a total preparation time of less than one hour. Microwave lead recoveries on standard reference materials compared very well with the certificate values. Dilution and centrifugation of the samples directly in the vessel eliminated the need for filtering as well as any additional sample handling which would introduce contamination. The added advantage of disposable vessels eliminated the need for vessel cleanup. Together, these characteristics combined into a safe, fast, inexpensive, accurate technique for lead determination in paint chips, soils and dust wipes.
Open Vessel Microwave Assisted Sample Preparation for Lead Analysis in Paint Chips, Wipes, and Soil

J. Doug Ferguson, Michael D. Miller, Robert Revesz

CEM Corporation
Overview

• Objective
• Equipment
• Procedure
• Results
• Conclusions
Objective

• Development of a sample preparation procedure for analysis of lead in paint chips and environmental samples

• Meet laboratory needs for increased throughput and turn around time

• Development of a safe, inexpensive, disposable, microwave vessel system
Equipment

• CEM MDS-2000/MDS-2100 microwave digestion systems with temperature control
• Fiberoptic temperature measurement
• CEM DV-50 High Throughput Vessel Set
• Fisher Scientific Centrific™ Centrifuge
• TJA 61E Trace® ICP
DV-50 Vessel Accessory Set Specifications

• Sample Capacity: 1-36 vessels
• Vessel: open disposable 50 mL centrifuge tube
• Operating temperature: 115 °C
Procedure

• Weigh sample directly into DV-50 tube*
• Add nitric acid**
• Heat at 115 °C for 15-30 min.***
• Allow to cool
• Dilute to the 50 mL mark with DI water
• Shake
• Centrifuge
• Aspirate directly into ICP or AA

* paint chips (0.1 g), soils (0.1 g)
** paint chips (5 mL), soils (10 mL)
*** paint chips (15 min.), soils (30 min.)
Analytical Instrumentation

- Thermo Jarrell Ash; TJA 61E Trace® Analyzer*

- Instrument Configuration:
  - Cyclone Spray Chamber
  - Meinhard® Nebulizer
  - Wavelength - 220.353

* Lead detection limit - 1.02 ng/mL
Sample Types

- **Paint Chips**
  - SRM 1579a*
  - ELPAT** Round 010

- **Soils**
  - SRM 2709, 2710, 2711*
  - ELPAT** Round 010

- **Wipes**
  - ELPAT** Round 010

* National Institute of Standards and Technology Reference Material
** Environmental Lead Proficiency Analytical Testing Reference Material
# ELPAT Paint Chip Lead Recoveries

## Microwave Lead Values

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Mean* (Wt. %)</th>
<th>% RSD</th>
<th>Low (Wt. %)</th>
<th>High (Wt. %)</th>
<th>% RSD</th>
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</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>0.1020</td>
<td>1.82</td>
<td>0.0875</td>
<td>0.1312</td>
<td>6.7</td>
</tr>
<tr>
<td>Sample 2</td>
<td>1.0089</td>
<td>4.25</td>
<td>0.7310</td>
<td>1.1843</td>
<td>7.9</td>
</tr>
<tr>
<td>Sample 3</td>
<td>3.4052</td>
<td>5.52</td>
<td>2.8367</td>
<td>4.3141</td>
<td>6.9</td>
</tr>
<tr>
<td>Sample 4</td>
<td>0.5449</td>
<td>4.43</td>
<td>0.4525</td>
<td>0.6730</td>
<td>6.5</td>
</tr>
</tbody>
</table>

* 36 digestions

## Certificate Lead Values

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Mean* (Wt. %)</th>
<th>% RSD</th>
<th>Low (Wt. %)</th>
<th>High (Wt. %)</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
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<td>0.0875</td>
<td>0.1312</td>
<td>6.7</td>
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<tr>
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<td>0.6730</td>
<td>6.5</td>
</tr>
</tbody>
</table>

* 36 digestions
# NIST 1579a Powdered Lead Based Paint Lead Recoveries

<table>
<thead>
<tr>
<th>Microwave Lead Values</th>
<th>Certificate Lead Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean* (Wt. %)</td>
<td>Mean (Wt. %)</td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>Uncertainty</td>
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<tr>
<td>11.222</td>
<td>11.995</td>
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<tr>
<td>0.246</td>
<td>0.031</td>
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</tbody>
</table>

* 36 digestions
## ELPAT Soil Lead Recoveries

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Mean* (ppm)</th>
<th>% RSD</th>
<th>Low (ppm)</th>
<th>High (ppm)</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>416.6</td>
<td>4.6</td>
<td>328.9</td>
<td>518.8</td>
<td>7.5</td>
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<tr>
<td>Sample 2</td>
<td>1493.9</td>
<td>4.1</td>
<td>1190</td>
<td>1845</td>
<td>7.2</td>
</tr>
<tr>
<td>Sample 3</td>
<td>192.3</td>
<td>5.2</td>
<td>151.1</td>
<td>240.8</td>
<td>7.6</td>
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<td>Sample 4</td>
<td>768.4</td>
<td>5.4</td>
<td>649.3</td>
<td>918.1</td>
<td>5.7</td>
</tr>
</tbody>
</table>

* 36 digestions
Lead Recovery Linearity Test for NIST Soils

**y = 0.9806 X + 1.0438**

Correlation coefficient = 1.00

* NIST SRM 2709, 1:1 2709:2711, 2711, 1:1 2710:2711, 2710
# ELPAT Wipe Lead Recoveries

## Microwave Lead Results

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Lead Value (ppm)</th>
<th>Certified Lead Value (ppm)</th>
<th>Range</th>
<th>Low</th>
<th>High</th>
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</thead>
<tbody>
<tr>
<td>Wipe 1</td>
<td>235</td>
<td>237</td>
<td>188</td>
<td>285</td>
<td></td>
</tr>
<tr>
<td>Wipe 2</td>
<td>60.8</td>
<td>69.5</td>
<td>47.8</td>
<td>91.2</td>
<td></td>
</tr>
<tr>
<td>Wipe 3</td>
<td>845</td>
<td>870</td>
<td>624</td>
<td>1115</td>
<td></td>
</tr>
<tr>
<td>Wipe 4</td>
<td>484</td>
<td>472</td>
<td>351</td>
<td>592</td>
<td></td>
</tr>
<tr>
<td>Wipe 5</td>
<td>103</td>
<td>108</td>
<td>84.0</td>
<td>133</td>
<td></td>
</tr>
<tr>
<td>Wipe 6</td>
<td>433</td>
<td>479</td>
<td>377</td>
<td>581</td>
<td></td>
</tr>
</tbody>
</table>
Conclusions

• The microwave methods provide a quick efficient sample preparation for lead determination in paint chips, wipes and soils.

• Digestion time for 36 samples is 30 min. or less.

• Total preparation time for 36 samples is less than one hour.

• Acceptable lead recoveries were obtained from standard reference materials.