



Lead in Soil: Comparison of the Field Extraction Kits and PDV6000PLUS Analysis with ICPOES Laboratory Extraction and Analysis

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When selecting a portable instrument for the analysis and evaluation of contaminated land, the most important consideration is how the instrument that is selected compares with the standard fixed laboratory methods and instrumentation. The most commonly used instrument in the fixed laboratory is the ICPOES (Inductively Coupled Plasma Optical Emission Spectrometer), and as such is the reference method for the evaluation of the PDV6000PLUS and the field extraction kits for lead (Pb) contaminated soil. A fixed laboratory that is certified in the United States must comply with the standard methods written for metals analysis in environmental samples. The methods used in this evaluation by the fixed laboratory for sample preparation are SW846 3050B¹, and for analysis is SW846 6010B².

The samples used in this evaluation were varied in composition. Any large pieces of debris (sticks, stones, and vegetation) were removed. The samples were homogenized and split into sub samples to be prepared by the field extraction procedure and the other portion was sent to the reference laboratory in the Midwestern USA.

The laboratory preparation of a soil sample for analysis is as follows. A 1-2g representative sample is weighed into the digestion vessel. 10mL of 1:1 HNO₃ is added to the digestion vessel, covered with a ribbed watch glass and refluxed at 95°C for 15 minutes without boiling. The sample is cooled to room temperature and then 5mL of concentrated HNO₃ is added. The cover is replaced and the sample refluxed for 2 hours or until the volume has been reduced to 5mL. The sample is again cooled and 3mL of 30% H₂O₂ is added to the digestion vessel. After heating and reaction of the H₂O₂, the sample is covered and again refluxed for 2 hours or until the sample volume is reduced to 5mL. 10mL of concentrated HCl is then added to the sample, and is again refluxed for 15 minutes. The sample is cooled and filtered into a 100mL dilution vessel and brought to volume and analyzed by 6010B². For a batch of 10 samples the total preparation time is approximately 6 hours.

The Pb in soil field extraction is performed as follows. 2g of soil is measured into a digestion bottle. 4mL of concentrated HNO₃ is added from the pre-measured acid pouch. The sample is mixed thoroughly, and any reaction of the sample with the acid is allowed to subside. 4mL of 30% H₂O₂ is then added in 1mL aliquots. After the reaction with the H₂O₂, 30mL of electrolyte is added to the bottle, mixed and the solids are allowed to settle. An aliquot of this liquid sample extract is analyzed by the PDV6000PLUS which uses ASV (Anodic Stripping Voltammetry). For a batch of 10 samples total preparation time is approximately 2 hours.

Table 1 Results from Field Extraction PDV6000PLUS (ASV) Analysis and Laboratory Extraction ICPOES Analysis³.

| Sample ID | ASV Result (mg/Kg) | ICPOES Result (mg/Kg) |
|-----------|--------------------|-----------------------|
| 1 | 7.1 | <10 |
| 2 | 14 | <10 |
| 3 | 52 | 44 |
| 4 | 67 | 75 |
| 5 | 84 | 58 |
| 6 | 84 | 130 |
| 7 | 93 | 120 |
| 8 | 102 | 120 |
| 9 | 127 | 86 |
| 10 | 134 | 120 |
| 11 | 260 | 210 |
| 12 | 270 | 250 |
| 13 | 346 | 360 |
| 14 | 362 | 350 |

Table 1 and Figure 1 display the results from the laboratory and the PDV6000PLUS field preparation and analysis of the same sample. The concentrations of lead in the samples range from low level amounts that are generally below the ICPOES quantitation limit provided by the laboratory, and rather contaminated soils with concentrations in the hundreds of ppm (mg/Kg).

For comparison between the 2 preparations and analysis methods, the results are plotted against each other and a linear regression performed to evaluate the correlation coefficient. Figure 2 displays the results of this plot, showing a correlation of 0.9366 for the field extraction and PDV 6000 analysis compared with the laboratory extraction and analysis.

When the field extract had been analyzed on the PDV6000PLUS, not all of the sample extract was consumed during the analysis. This extract was sent to the laboratory to be analysed. The analysis method in the laboratory was performed by liquid sample testing procedures. The extracts were filtered to remove any solids from the sample and digested on a hot block by SW846 3010A⁴. The results are also plotted against the results from the laboratory digestion and analysis of the original soil which can be interpreted as an extraction efficiency of the Field Kits. The correlation graph (Figure 3) shows that the Field extraction is as efficient at removing lead from the soil matrix

as the more involved and labour intensive laboratory extraction procedures. The correlation coefficient for this comparison is 0.9842.

Table 2 Results from Field Extraction ICPOES Liquid Analysis with Laboratory Extraction ICPOES Analysis

| Sample ID | ICP (mg/Kg) | Extract by ICP (mg/Kg) |
|-----------|-------------|------------------------|
| 1 | <10 | 5.7 |
| 2 | <10 | 6.4 |
| 3 | 44 | 42 |
| 4 | 58 | 48 |
| 5 | 75 | 46 |
| 6 | 86 | 93 |
| 7 | 210 | 210 |
| 8 | 270 | 240 |
| 9 | 250 | 240 |
| 10 | 360 | 320 |

The results from this study show good correlation with the laboratory in both extraction efficiency as well as final result. In general the correlation coefficient can be viewed as a percentage. Thus, the results derived by the Pb in soil extraction analysed on the PDV6000PLUS have a 93.66% correlation with the reference laboratory method. The field kit extraction efficiency displayed a 98.42% correlation with the reference laboratory result.

Results of Field Kit Extraction PDV 6000 (ASV) Analysis and Laboratory Extraction ICPOES Analysis for Lead in Soil

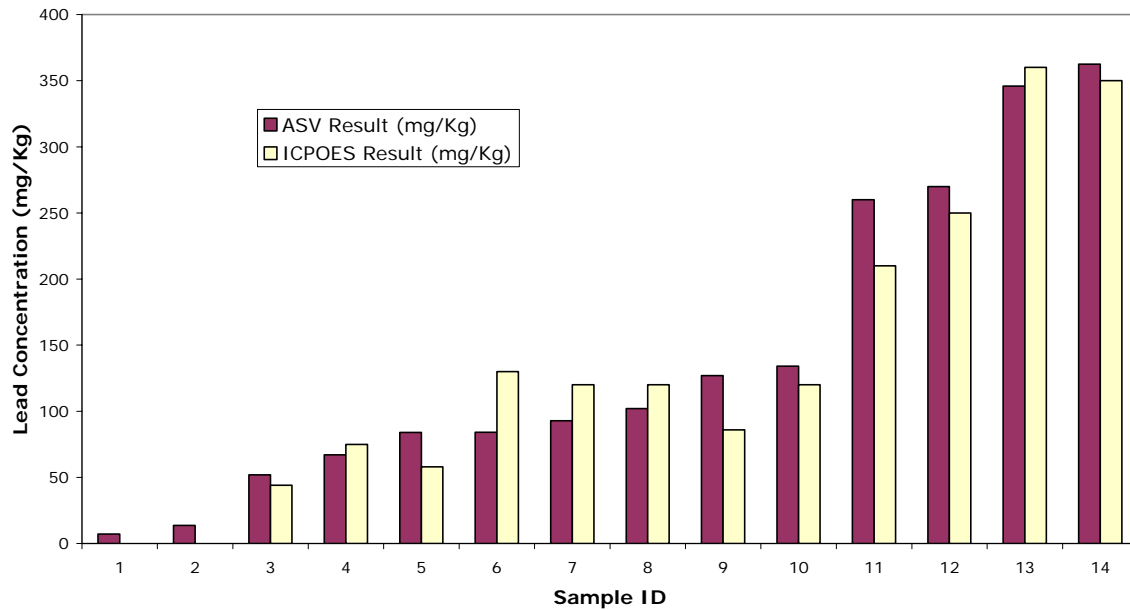


Figure 1 Data comparison for the Field Extraction PDV6000plus (ASV) Analysis with Laboratory Extraction ICPOES Analysis

Correlation between Field Extraction PDV 6000 (ASV) Analysis with Laboratory Extraction ICPOES Analysis for Lead in Soil

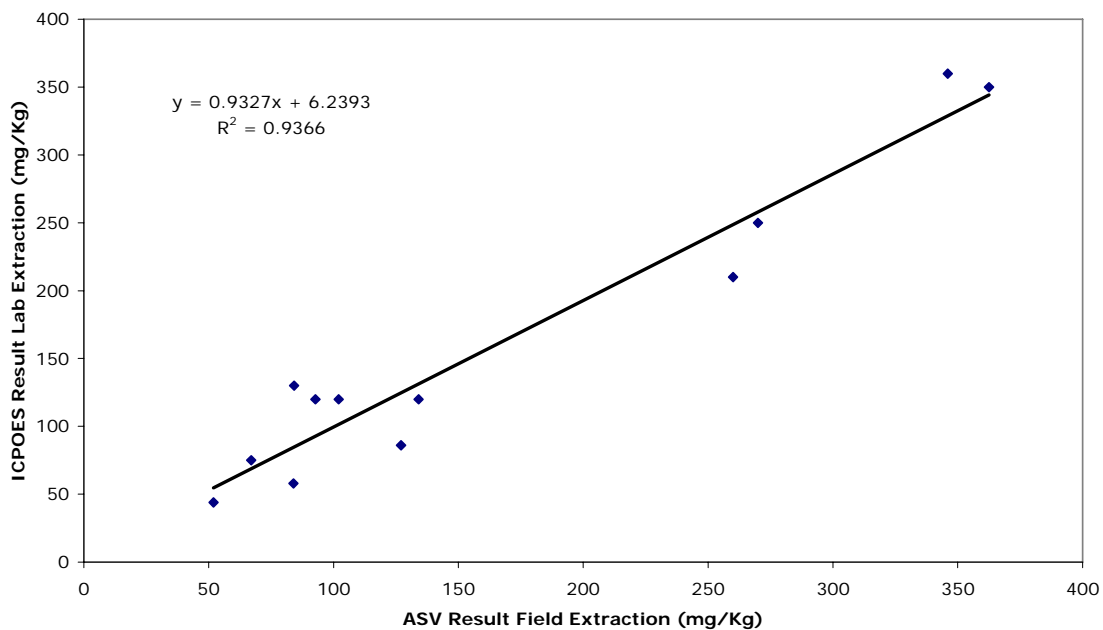


Figure 2 Correlation of the Field Extraction PDV6000plus (ASV) Analysis with Laboratory Extraction ICPOES Analysis

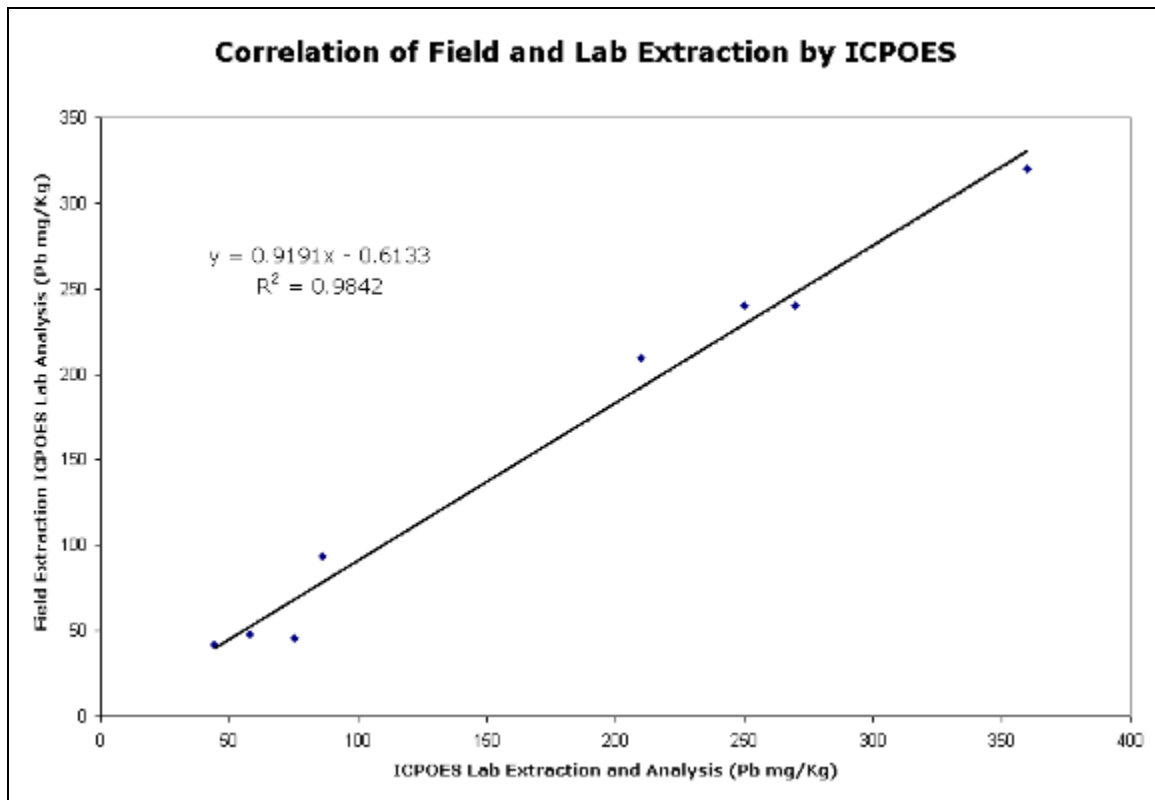


Figure 3 Correlation of the field extraction fluid analyzed by ICPOES and Laboratory extraction ICPOES analysis showing the extraction efficiency of the field method.

Appendix:

1. Method 3050B: Acid Digestion of Sediments, Sludges, and Soils; Revision 2, December 1996, pg. 3 – 5, Section 7.
2. Method 6010B: Inductively Coupled Plasma – Atomic Emission Spectrometry; Revision 2, December 1996, pg. 1, 2, Section 2 (Summary).
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4. Method 3010A: Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy; Revision 1, July 1992, pg. 2, 3, Section 7.