



Innovative X-ray Technologies

## Analysis of Lead and Arsenic in Soil Using Portable XRF

### Overview:

Field portable XRF has been utilized for decades to provide rapid, on-site measurements of metals in soil contamination for site assessment and remediation activities. In 1998, EPA incorporated Method 6200 for portable XRF into SWA846 as a standard method. Innov-X Systems has since provided the latest breakthrough by developing a handheld XRF analyzer that utilizes an x-ray tube instead of radioactive isotopes. The x-ray tube system eliminates most regulatory issues particularly for interstate travel – essential for the environmental assessment market where interstate travel is common. The “tube” also replaces multiple isotopes used in competing systems to offer simultaneous analysis of 20-25 metals including all 8 RCRA and Priority Pollutant metals.

### Arsenic and Lead Analysis:

Two of the most common metals requiring field analysis are lead (Pb) and arsenic (As). Arsenic analysis is one of the easiest elements to analyze with XRF. Interference-free detection limits (DLs) for both are shown in Table 1, and plots of results on laboratory-analyzed samples are shown in Fig. 1. Correlations are good in a variety of soil samples, exhibiting  $R^2$  values of 0.99 in each case. The XRF calibration – typically the Compton Normalization method described in EPA Method 6200 – provides good results without the need for site-specific calibration corrections.

Element	Detection Limit, ppm (Interference-Free, 2-minute Test Time)
Pb	13
As	7

\*As values represent latest (Aug. 2003) values.

While analyzing lead and arsenic with portable XRF is relatively straightforward, analyzing arsenic when lead is present, particularly in high concentrations, poses an additional challenge. Lead x-rays (from XRF) produce two strong spectral peaks at energies 10.5 and 12.6 keV. The most intense As spectral peak is also at 10.5 keV and is therefore strongly interfered by the presence of lead in the sample. Thus, the presence of lead in a sample has two main detrimental effects to the measurements of low levels of arsenic:

- ❑ Elevated As detection level.
- ❑ Moderately decreased precision for the same testing time on a sample with no lead.

### XRF Analysis for Arsenic in the Presence of Lead:

XRF analysis for soil is an easy process. The Innov-X handheld XRF automatically corrects the As data for the lead interference, and displays corrected results. However, the fact that an interference is present means that detection levels and precisions are not as good as for samples without lead present.

The impact on both As detection limit and precision can be determined. Results for the DL as a function of lead concentration is presented in Fig. 2. The As detection limit is expected to increase theoretically as the square root of the lead concentration, following the functional form in the



Pb and As Results - Portable XRF Analysis of Soil Samples

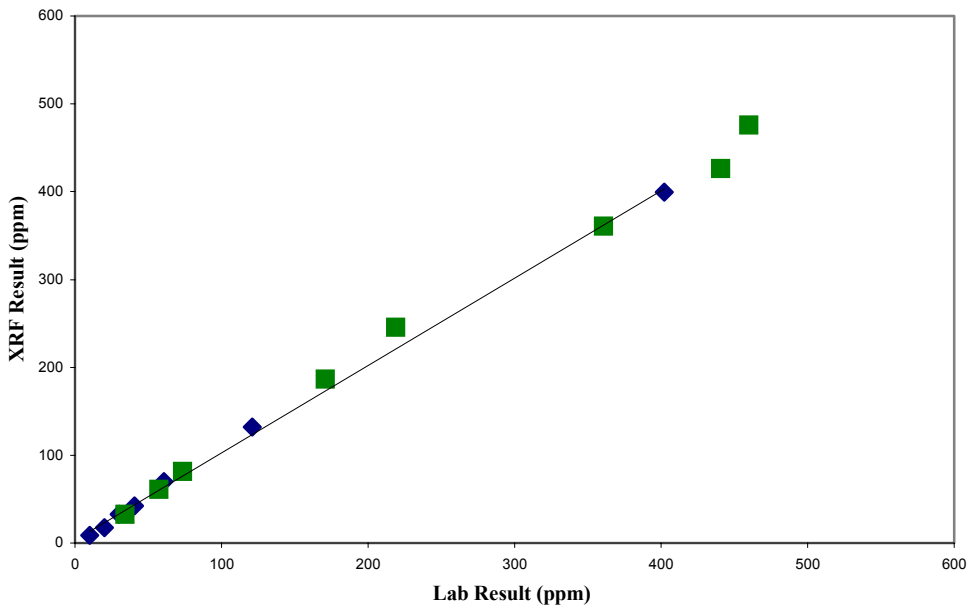


Fig. 1: Lead (green) and arsenic (blue) XRF results versus laboratory results.

equation below. Figure 2 shows both the calculated (solid line) and measured detection limit as a function of lead concentration. For example, for no detectable lead in the sample (< 15 ppm) the As detection limit is approximately 7 ppm. The As DL increases smoothly to a value of 15 for 100 ppm lead, and 31 for 1,000 ppm lead. Thus for a 10-fold increase in lead concentration (100 ppm to 1,000 ppm), the detection limit worsens by a factor of about two.

$$As|_{Pb} = As|_{noPb} + b\sqrt{Pb(ppm)} ;$$

b is a fitting coefficient, b=0.76

The agreement between the calculated and measured impact on As DL is good. The increase in DL is not a function of instrument performance. No XRF system, portable or stationary, can resolve the overlap between the lead La peak and the arsenic Ka peak, thus this effect will be present on any XRF system. The effect on precision of the As measurement will follow a similar trend, but is not shown in this paper.

**Summary:**

The latest x-ray tube technology offers faster, higher-precision measurements of important environmental metals in soil. Two of the most common elements analyzed are lead and arsenic. By themselves, both elements are excellent candidates for portable XRF analysis due to the high accuracy achievable, and the low detection limits. Measurement of low concentrations of As in the presence of high lead concentrations presents some unique challenges due to the large interference of the lead with the arsenic measurement. Innov-X has developed a handheld, point-and-shoot XRF that eliminates radioactive sources and provides good data quality on lead, arsenic, and the remaining RCRA metals. Studies have determined the effect of lead concentration on As detection limit – a common question from users of this type of equipment.



Innovative X-ray Technologies

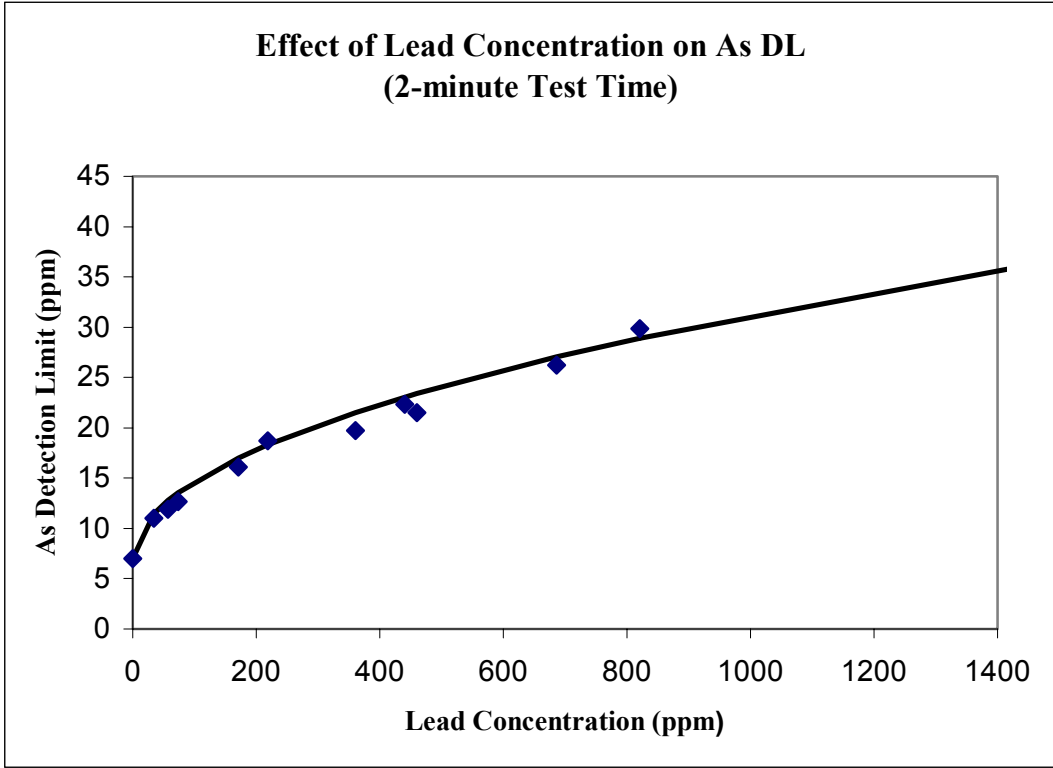


Figure 2. Effect of lead concentration on arsenic detection limit. Blue data points are measured values, solid curve is the expected value calculated from above formula (Pg. 2).