

## ABSTRACT

Bioaccessible Pb soil extractions are typically analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES). We propose using X-ray fluorescence (XRF) spectrometry instead because XRF is fast, simple, non-destructive, and cost-effective.

In this project, we

1. Developed an XRF method for measuring aqueous solutions of bioaccessible Pb extracted via EPA Method 1340.
  2. Constructed a simple linear regression model comparing bioaccessible Pb measurements from ICP-OES and XRF.
- We found that the XRF result is a significant predictor of the ICP-OES result at the 5% significance level, with a regression  $R^2$  of 98.4%.

## INTRODUCTION

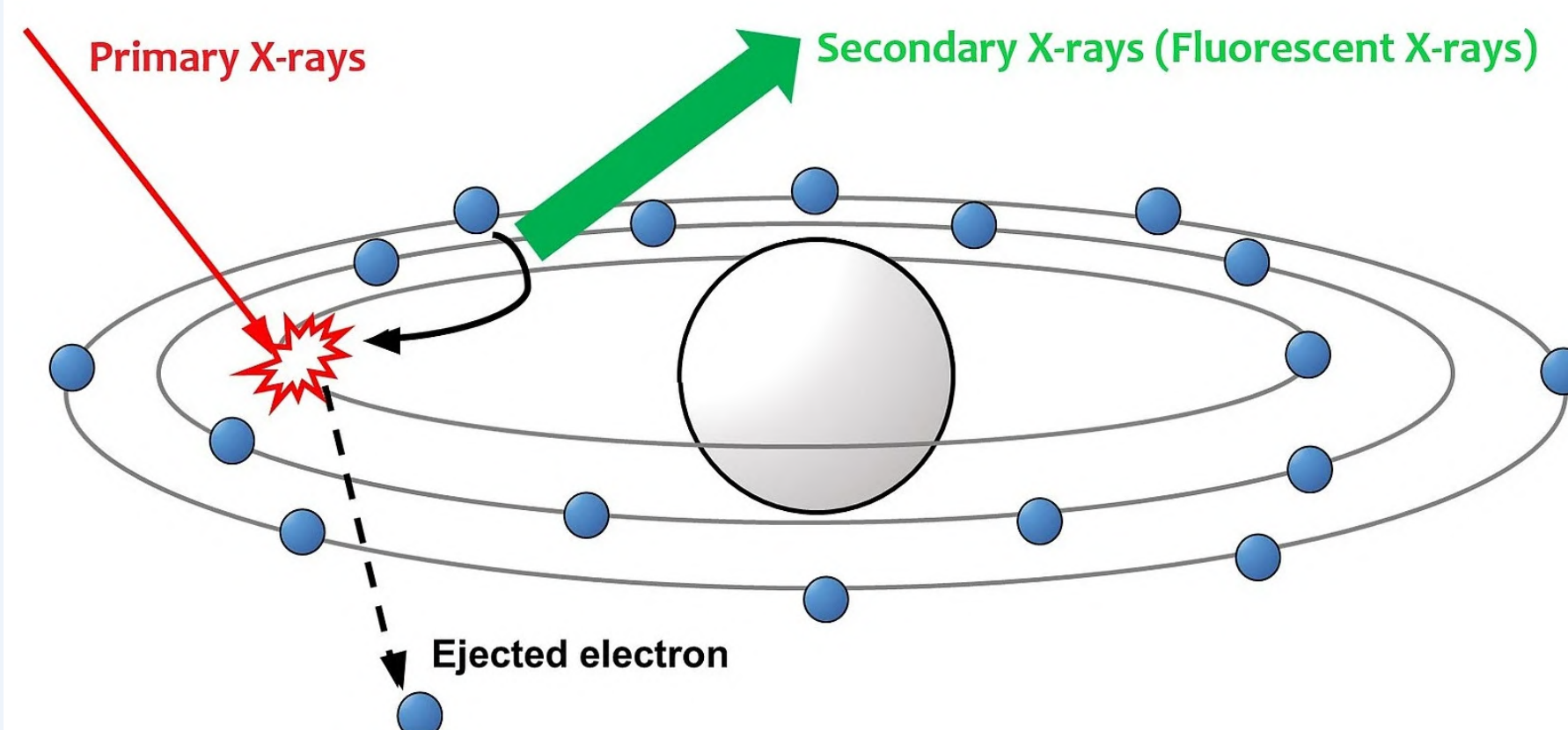


Figure 1. Diagram showing the process of X-ray fluorescence.<sup>2</sup>

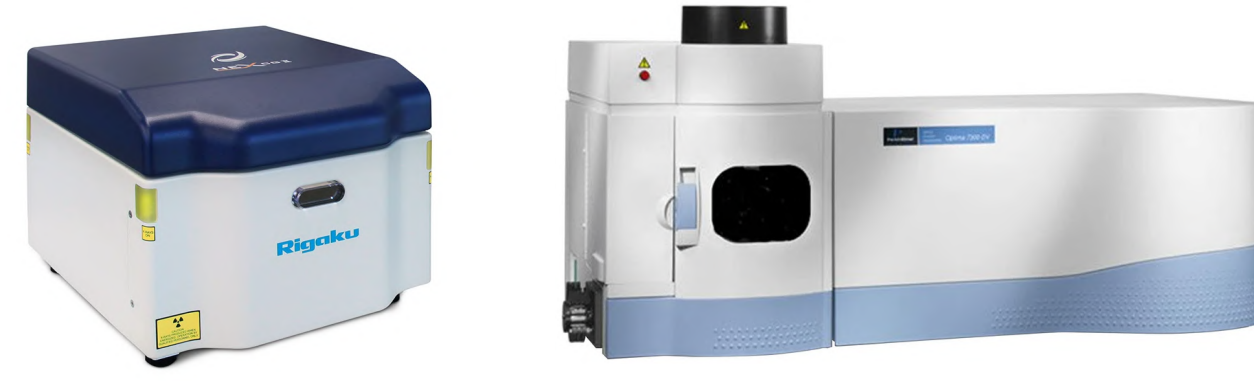
- One of the primary environmental sources of Pb exposure in urban areas is soil contamination.<sup>1</sup>
- In-vitro measurements of bioaccessible Pb tell us the portion of Pb dissolved into solution under conditions similar to those found in the human digestive tract.
- Analyzing bioaccessible Pb extractions typically requires the use of inductively coupled plasma optical emission spectroscopy (ICP-OES).
- In this project, we propose using X-ray fluorescence (XRF) spectrometry instead: a faster, simpler, non-destructive, and cost-effective method.
- XRF is commonly used for elemental analysis (Na to U) of solids.
- In liquids, lighter elements are not easily detected by XRF, as their weaker fluorescence does not escape the water matrix easily (Fig. 1).<sup>2</sup>
- **Because Pb is a heavy element with high energy fluorescence, we hypothesize that benchtop XRF can be used to provide results for aqueous in-vitro bioaccessible Pb extractions.**

## METHODS

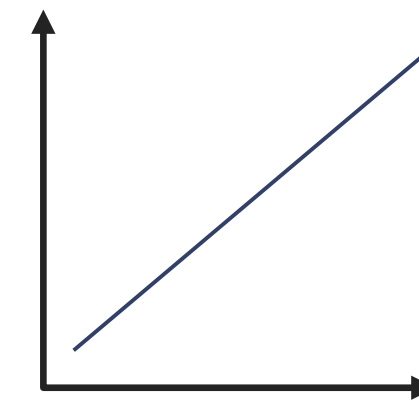
### 1. Sample preparation and bioaccessible Pb extraction



### 2. Elemental analysis by XRF and ICP



### 3. Regression analysis



### 1. Sample Preparation and IVBA Pb Extraction

Contaminated soils (52.9–662.2 ppm Pb) from Philadelphia, PA were air-dried, sieved to <2 mm, and analyzed for total Pb using EPA Method 3050B and ICP. Bioaccessible Pb was extracted following EPA Method 1340. NIST soil standard 2711a was included for quality control.

### 2. Elemental Analysis

Liquid samples (3 mL) were placed in plastic cups with x-ray transparent film. Each was analyzed three times using XRF in an air atmosphere, with results averaged. Detection and quantification limits were 0.170 ppm and 0.509 ppm, respectively. Background interference was minimized by subtracting a blank glycine spectrum and selecting Pb as the only analyte. Samples were also analyzed by ICP (EPA Method 6010B).

### 3. Statistical Analysis

Minitab software was used for all statistical analyses. One datum with a high Y value stood out as a possible outlier, but its removal did not significantly alter the model statistics, so we chose to retain it. Model validation was performed by dividing the dataset into ~80% training (30 pt.s) and ~20% validation (7 pt.s).

## RESULTS & CONCLUSIONS

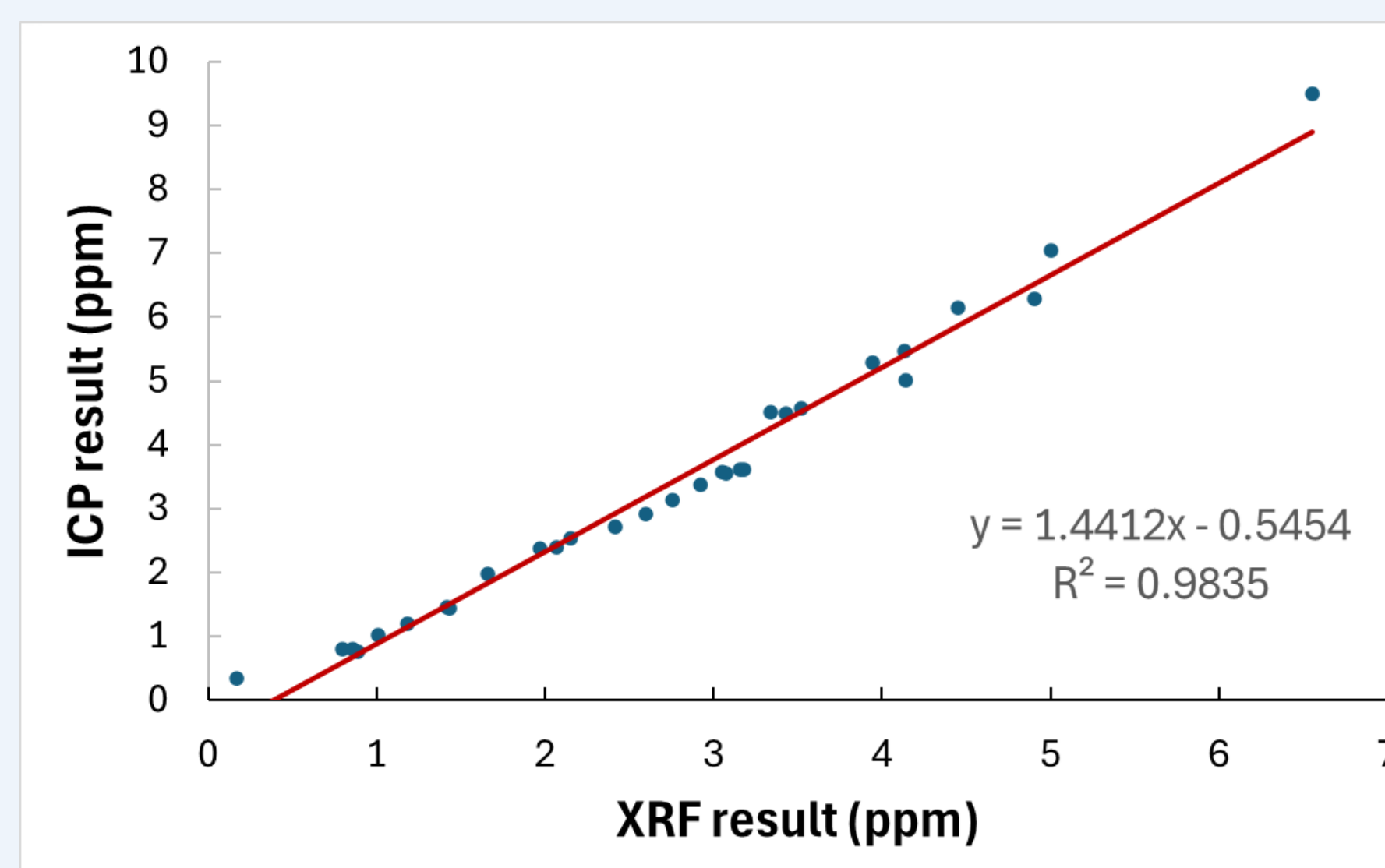


Figure 2. Regression line for the ICP vs. XRF model.

The regression model  $R^2$  value was 0.9835 (98.35% of the variation in ICP result is explained by XRF result). The t-test p-value was <0.001, meaning that at the 5% significance level, XRF result is a significant predictor of ICP result. The regression equation was  $\text{ICP result} = -0.5454 + 1.4412 \text{ XRF result}$  (Fig. 2).

For validation we entered the XRF data for our 7-sample validation set into the regression equation to obtain the expected ICP result, then plotted Expected vs. Actual ICP result. The data were highly correlated with an  $R^2$  of 99.57%, suggesting that the model has good predictive ability.

**In summary, our novel method of testing liquid bioaccessible Pb extractions by XRF was successful.** We expect that XRF users will be able to apply this method to estimate ICP results for EPA Method 1340 bioaccessible Pb assays. Users should note that the scope of this model is soils ranging from 52.9 to 662.2 ppm Pb, and it should not be used for soils containing amounts of Pb outside of that range. Future work should entail expanding the sample size and scope of the model to include soils of diverse types and a wider range of Pb levels, and the use of XRF with other solutions and analytes.

## ACKNOWLEDGEMENTS & REFERENCES

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