



The UNIVERSITY of OKLAHOMA
Gallogly College of Engineering
School of Civil Engineering and Environmental Science



Center for Restoration of
Ecosystems and Watersheds
University of Oklahoma

A Comparison of Methods for Analyses of Soil Trace Metals in a Mining Impacted Agricultural Watershed

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School of Civil Engineering and Environmental Science

University of Oklahoma

June 5, 2018



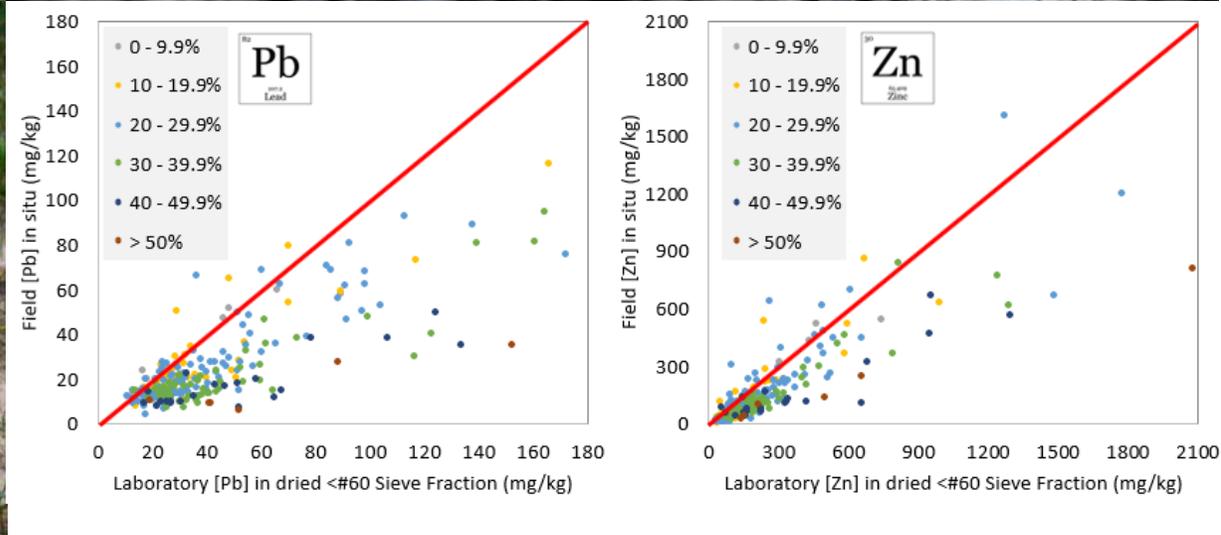
Introduction



Hypotheses



Methods & Locations

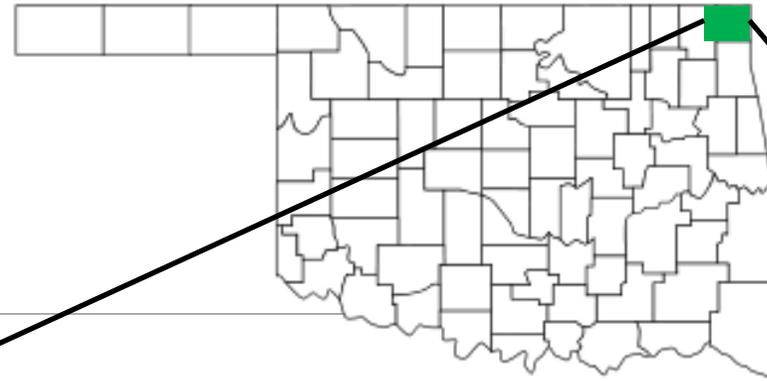


Results & Conclusions



Introduction

Neosho River Bottoms



- ~25,000 acre floodplain and upland area
- Significant restoration opportunities
 - Bottomland hardwood forest
 - Oxbow lakes
 - Scrub shrub wetland
 - Eastern tall grass prairie
- GRDA acquired 3,600 acres along the Neosho River

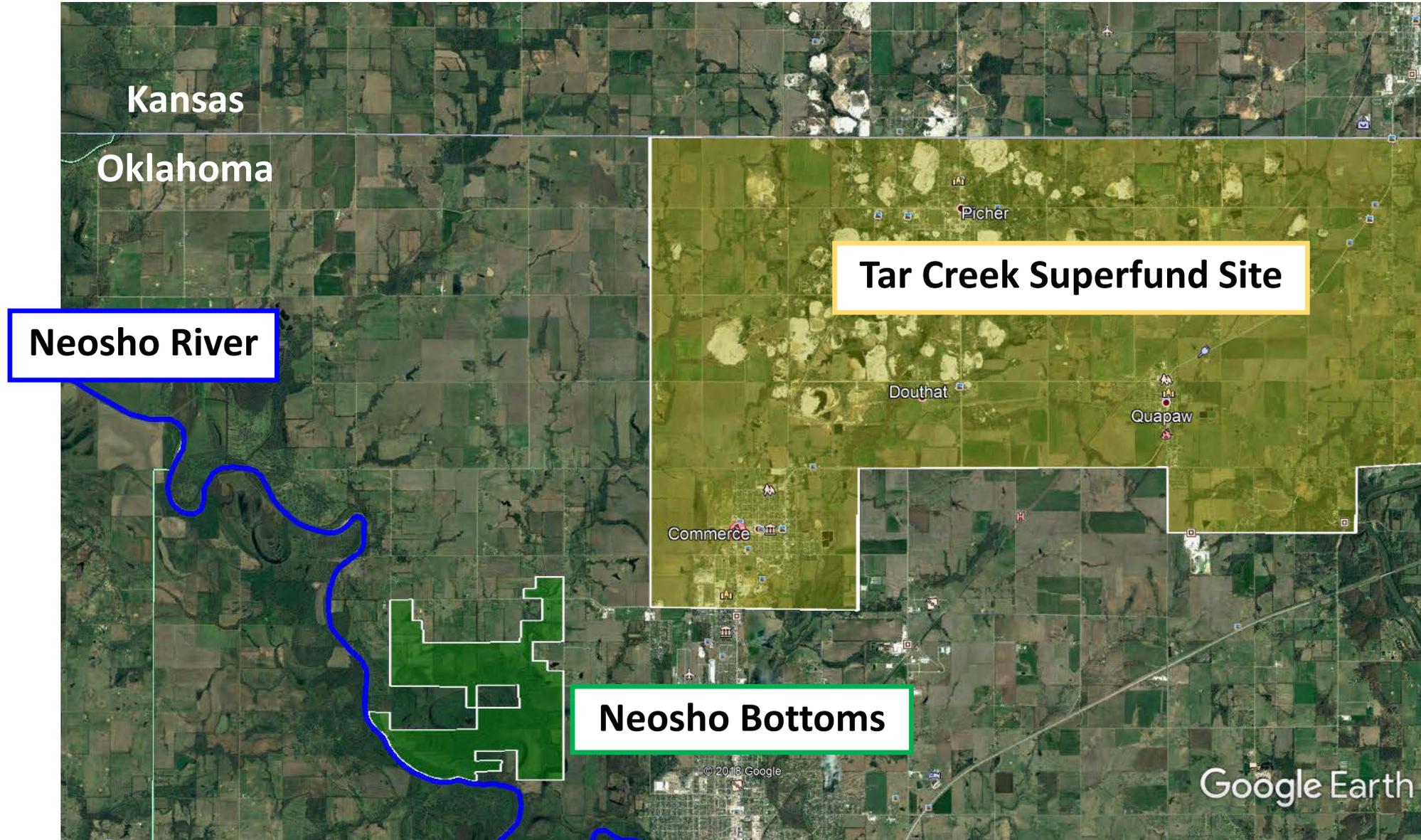


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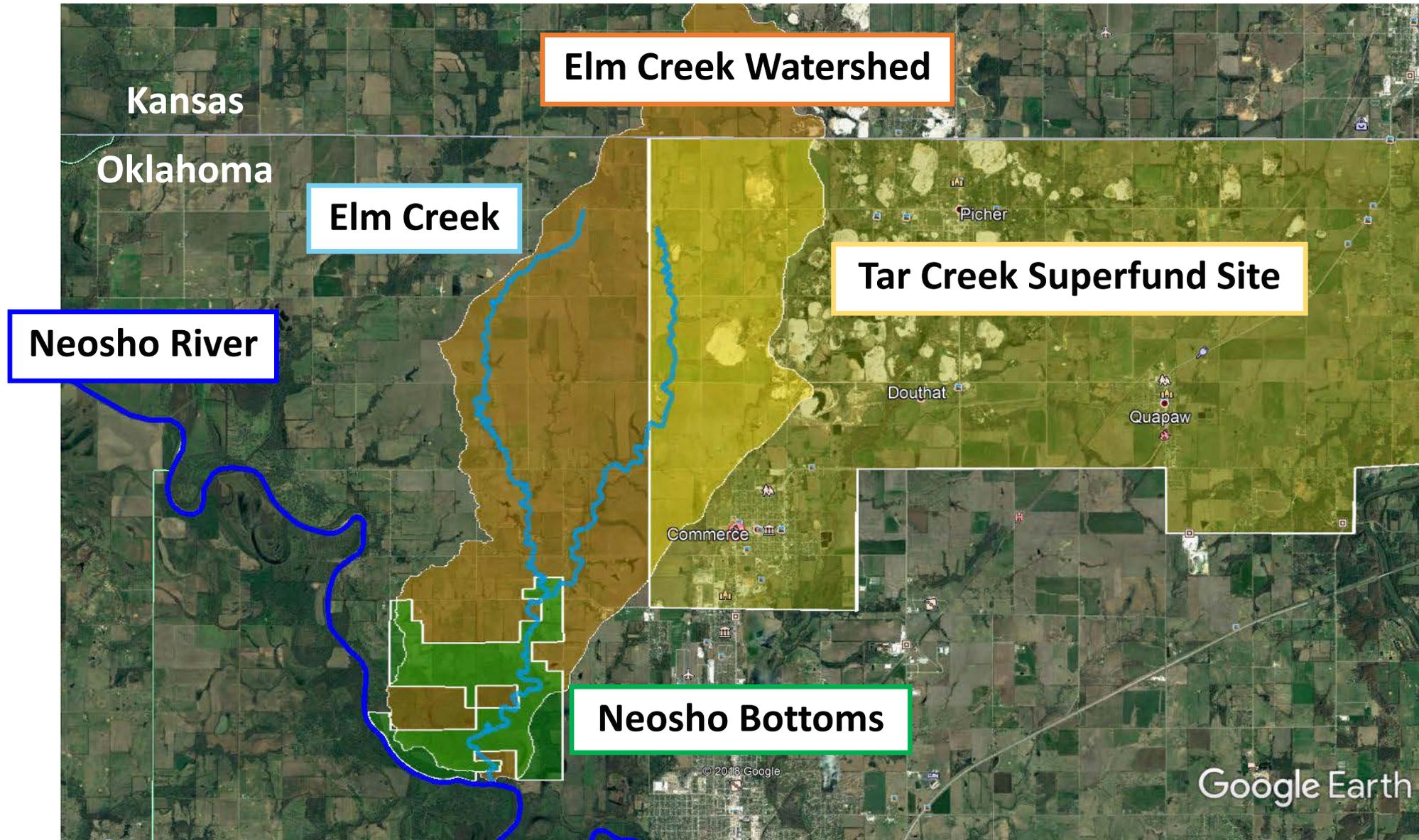
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Soil Trace Metals Detection

- Inductively coupled plasma optical emission spectrometry (ICP-OES)
- Inductively coupled plasma mass spectrometry (ICP-MS)
- X-ray fluorescence (XRF)
 - On-site fast screening method for soil metals
 - Cost effective when compared to ICP methods
 - Viewed by the environmental community as an acceptable analytical approach for field applications



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Methods & Locations



Thermo-Fisher Scientific
Niton XL3t GOLDD+ XRF

Methods & Locations

The soil metal concentration in the floodplain were determined three different ways

Method 1:

In Situ

Field Portable XRF Analyses (EPA 6200)
Bulk Sample



The soil metal concentration in the floodplain were determined three different ways

Method 1:

In Situ

Field Portable XRF Analyses (EPA 6200) Bulk Sample

- Soil samples were collected using stainless steel shovel
 - 13 cm X 13 cm X 10 cm cuttings
 - Sealed tightly in 3 mil or thicker plastic bag
- Sample locations were recorded with GPS
- Transported back to laboratory



The soil metal concentration in the floodplain were determined three different ways

Method 1:

In Situ

Field Portable XRF Analyses (EPA 6200)
Bulk Sample



Laboratory

Moisture content (ASTM D226-10)
Loss-on-ignition (Dean 1974)



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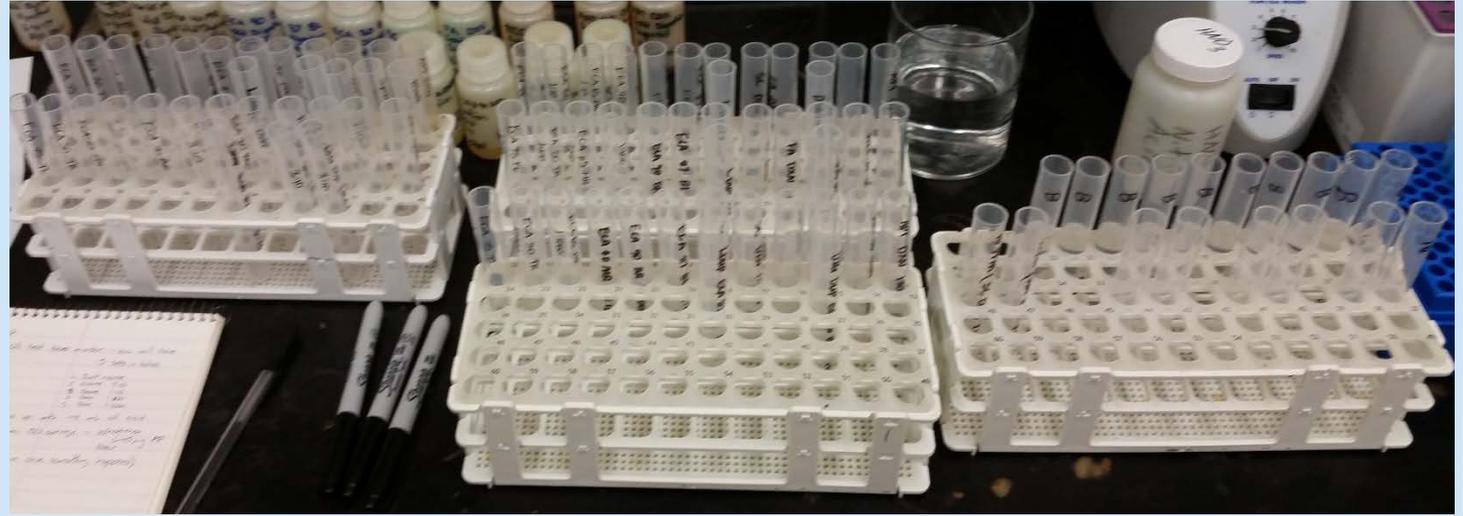
Method 2:

Laboratory

Field Portable XRF Analyses (EPA 6200)
Dried and < # 60 Sieve Fraction



The soil metal concentration in the floodplain were determined three different ways



Method 3:

Laboratory

Microwave HNO_3 digestion (EPA 3051)
Inductively Coupled Plasma-Optical Emission Spectrometry
(ICP-OES) Analyses (EPA 6010)
Dried and < # 60 Sieve Fraction



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Sampling Locations

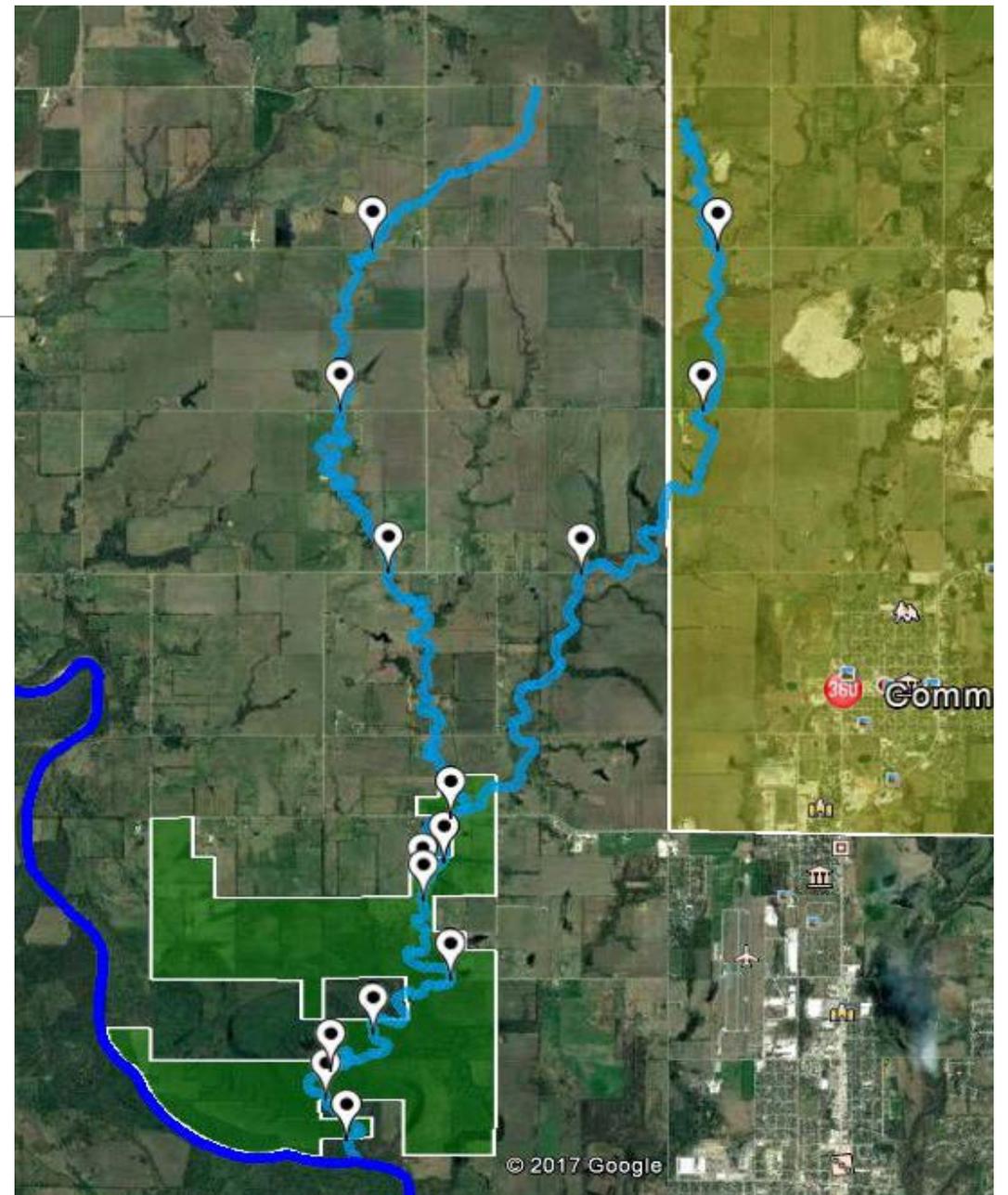
- Elm Creek riparian zone
 - Road crossings (intersecting the stream)
 - GRDA property
 - 106 soil samples

- Neosho Bottoms uplands
 - 278 soil samples



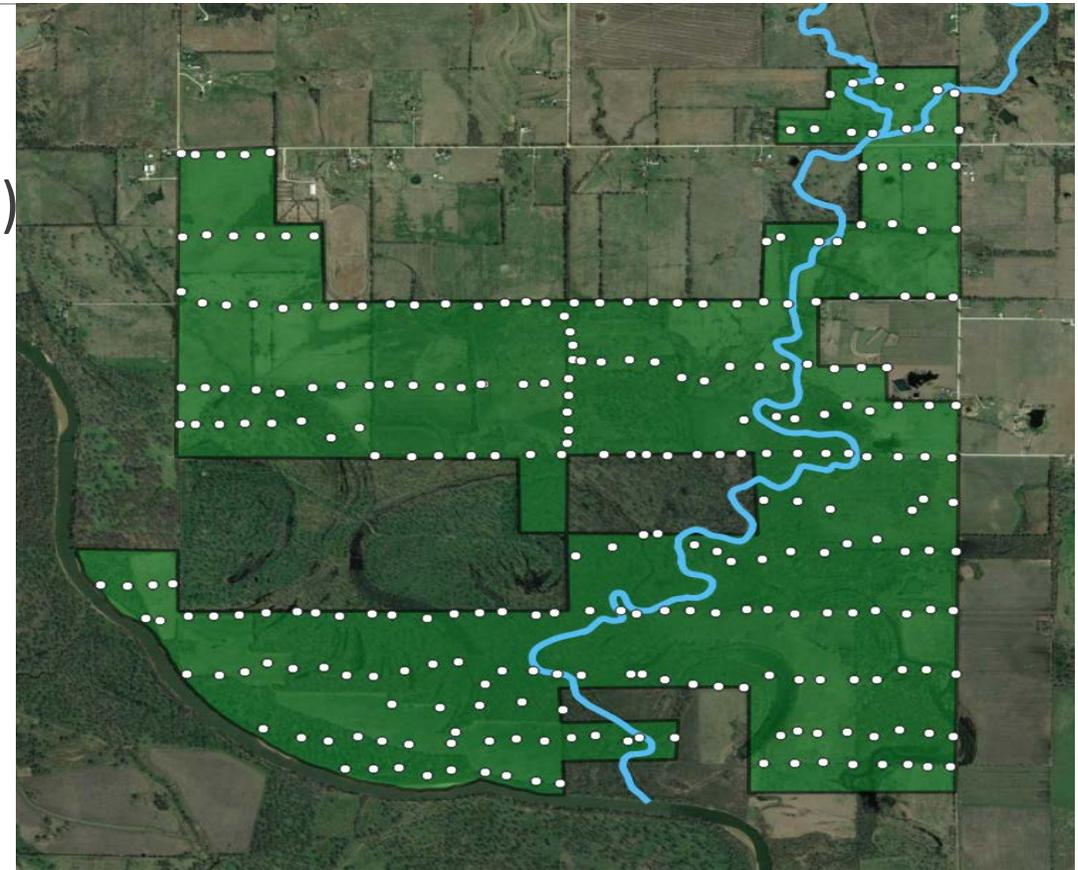
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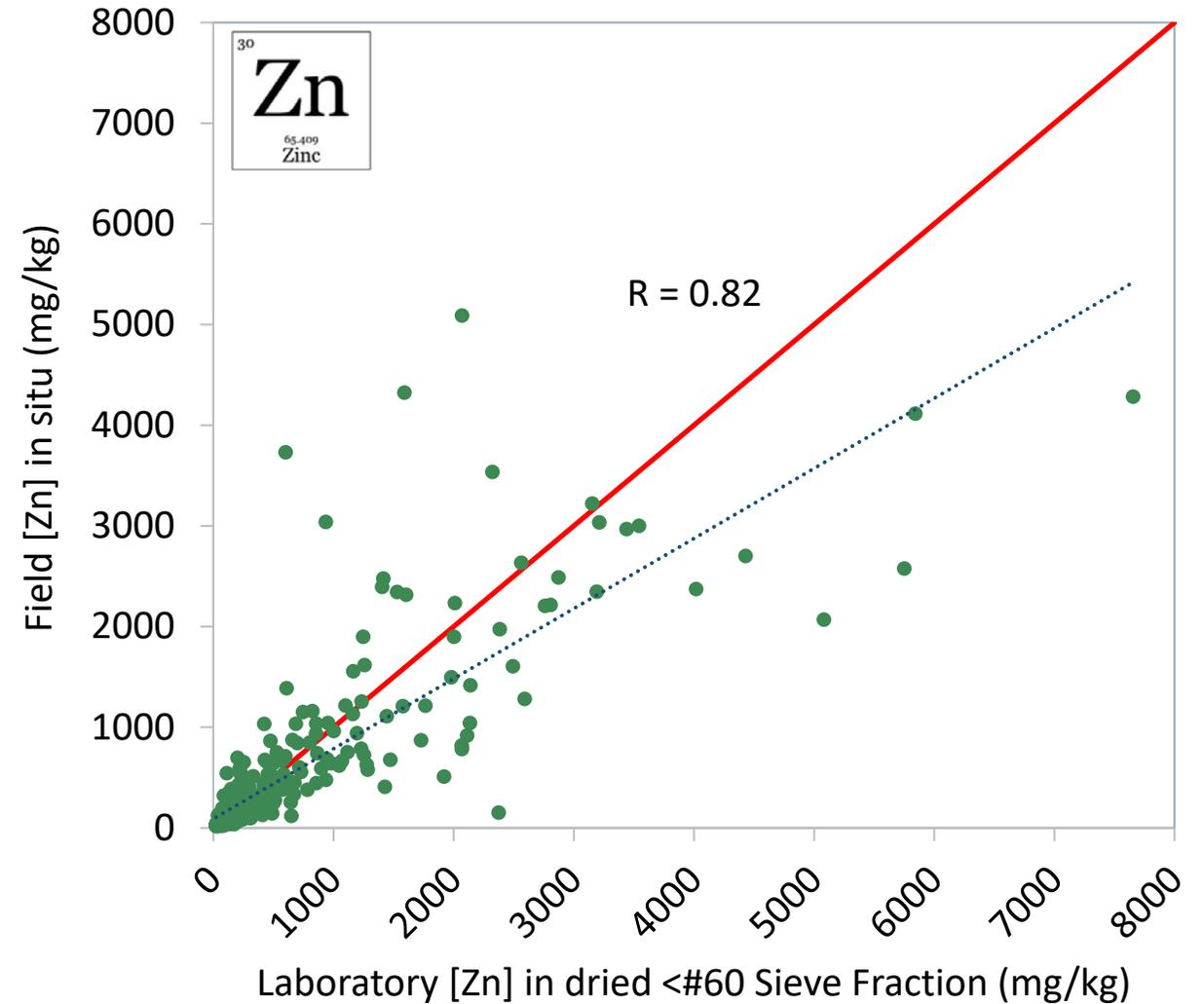
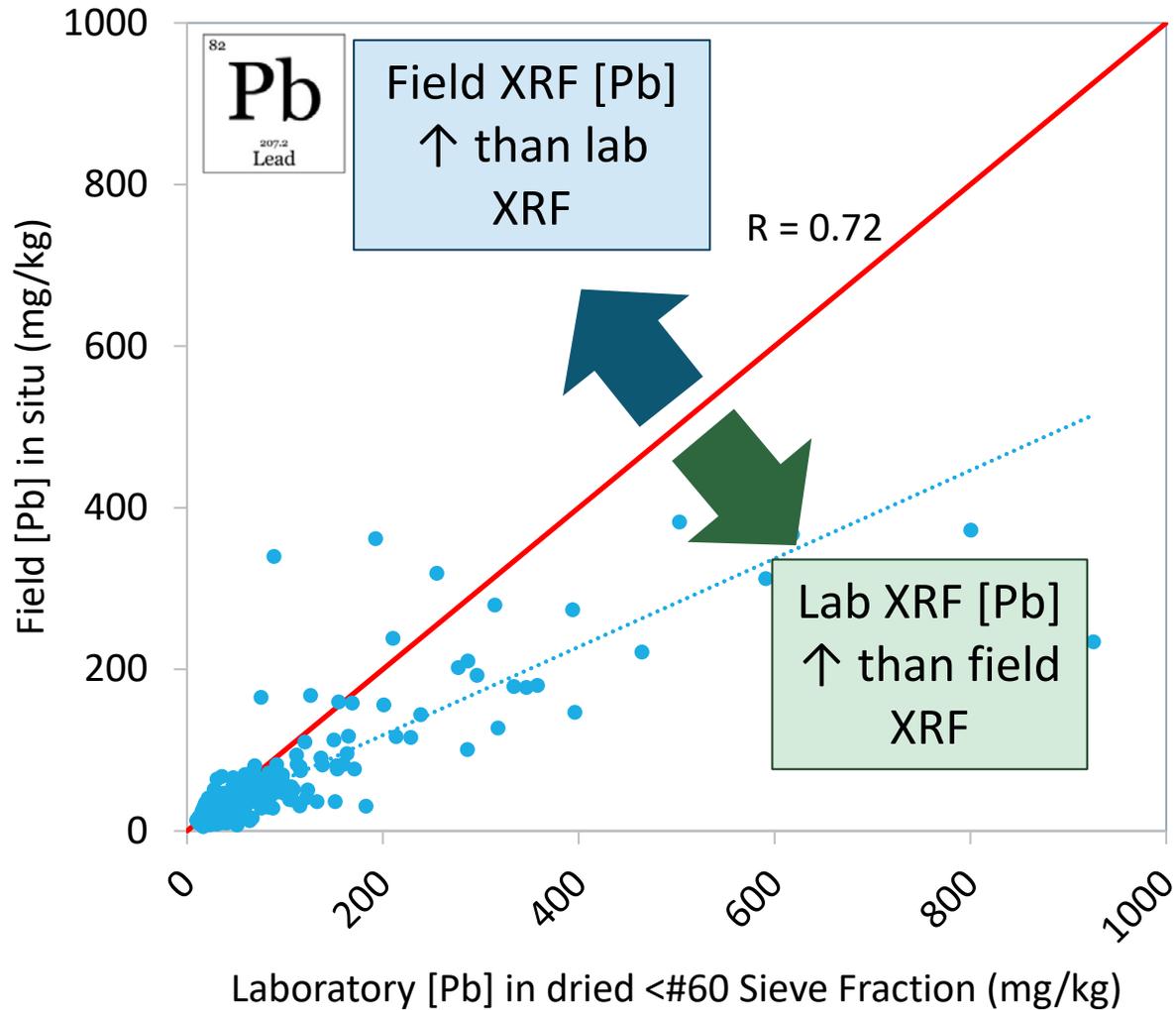
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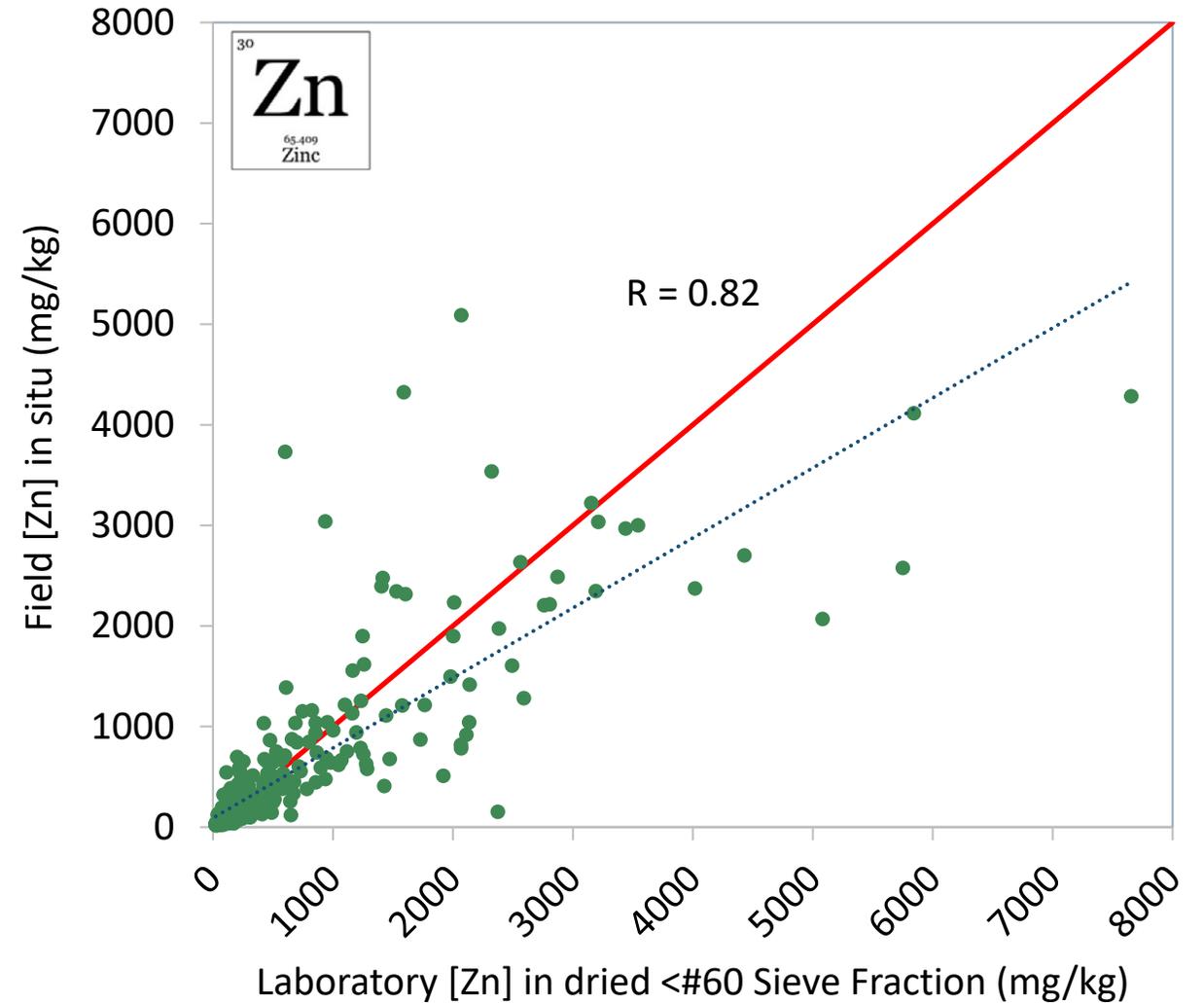
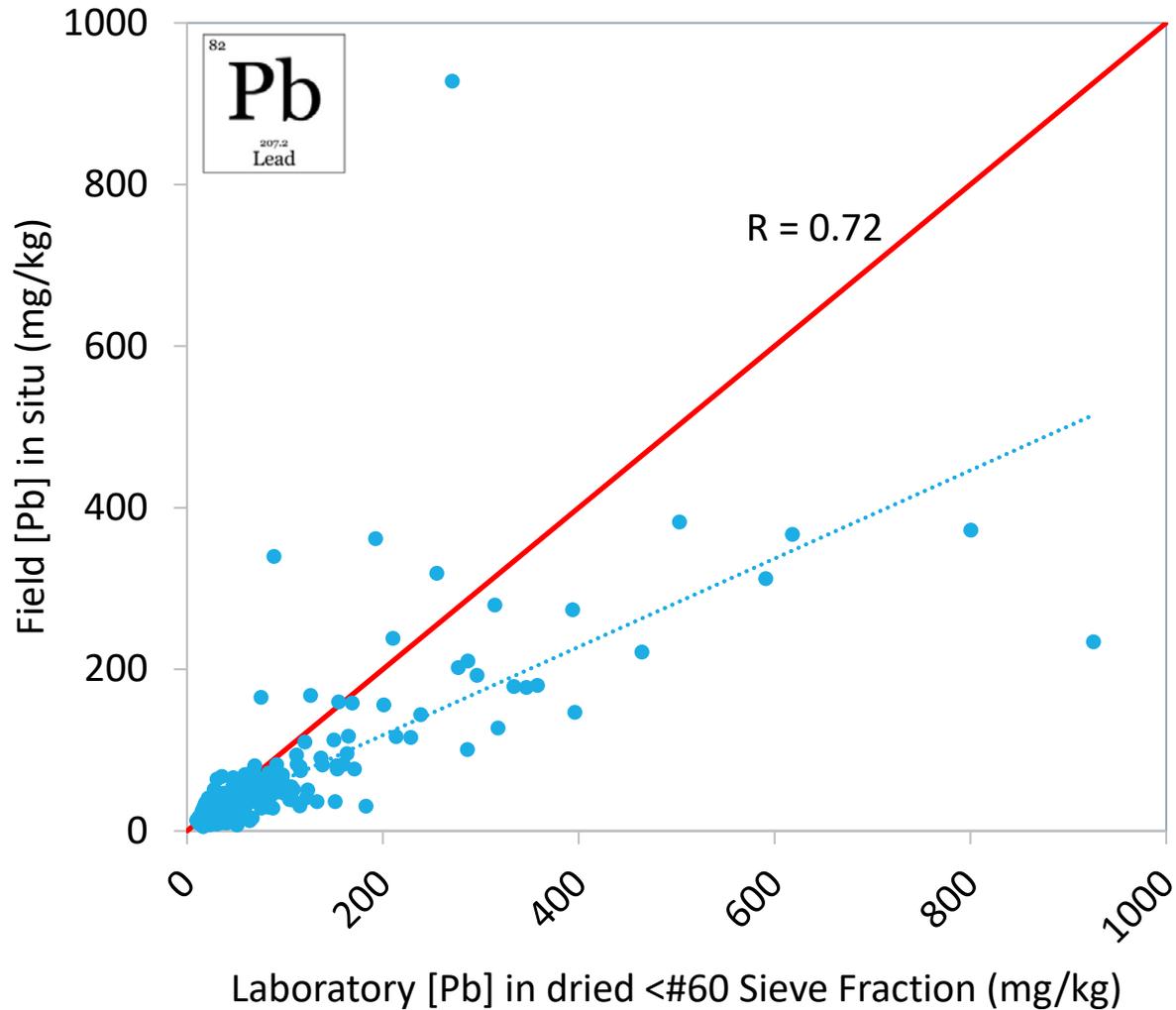


Results & Conclusions

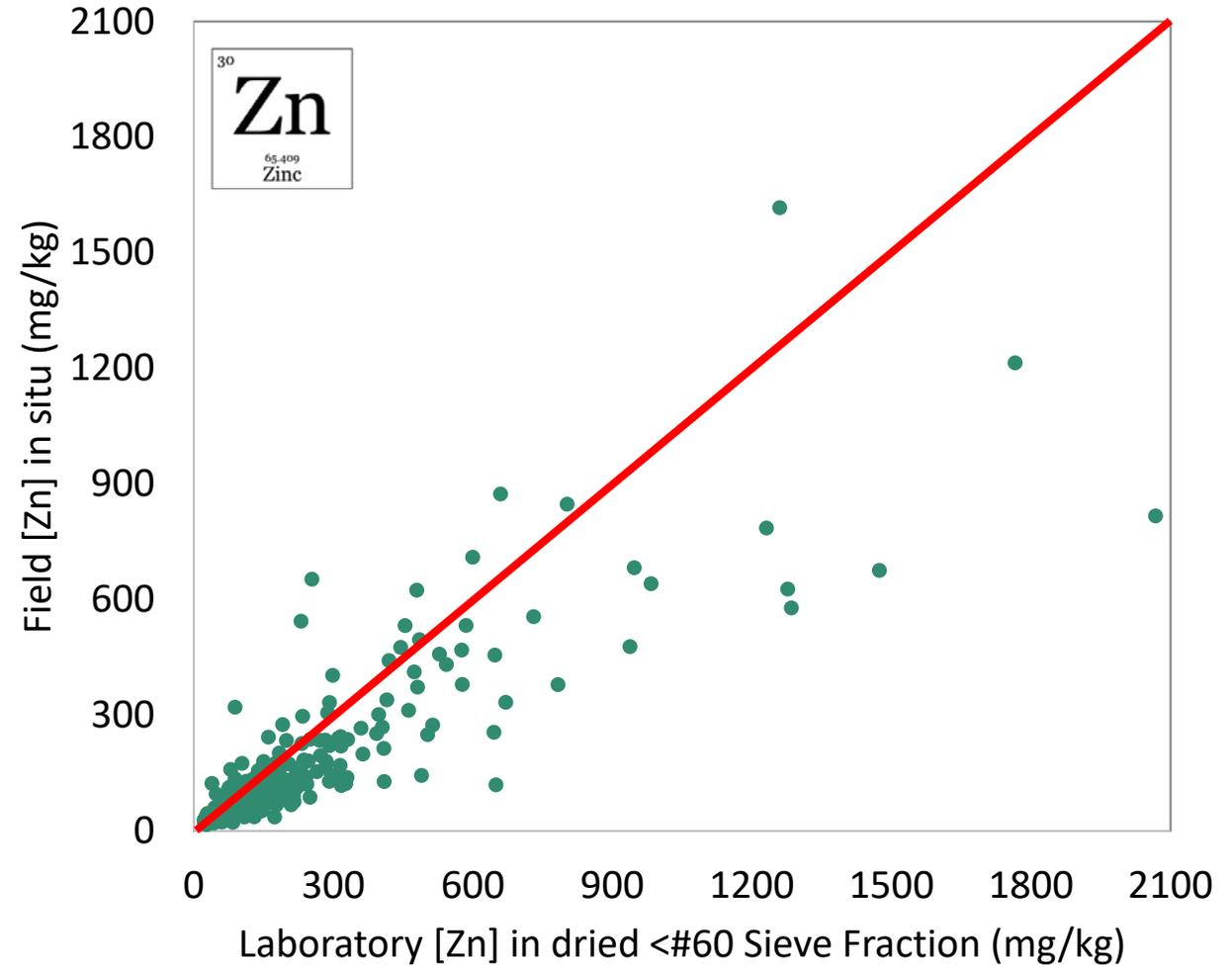
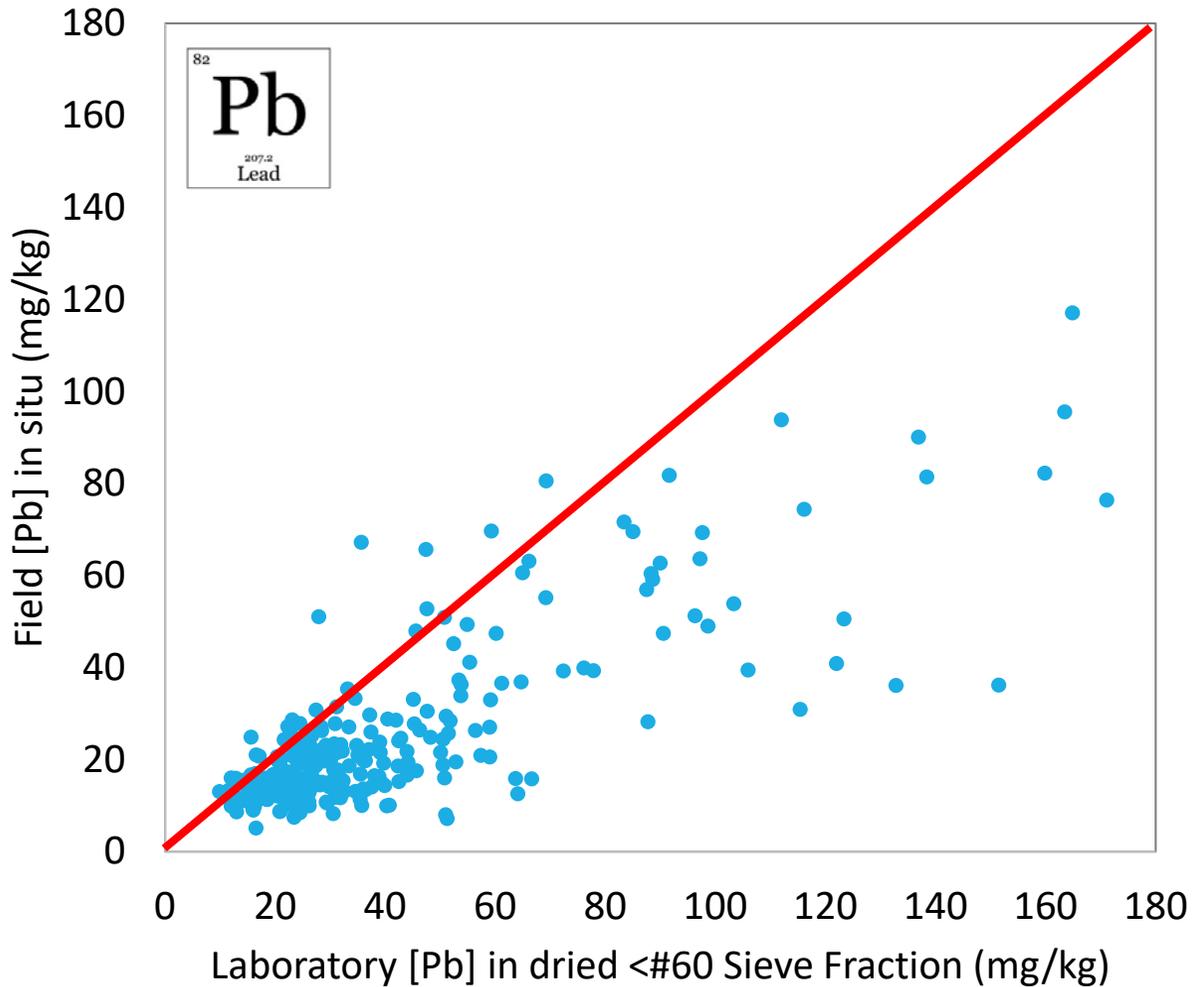
Relation Between Field and Laboratory XRF Concentrations



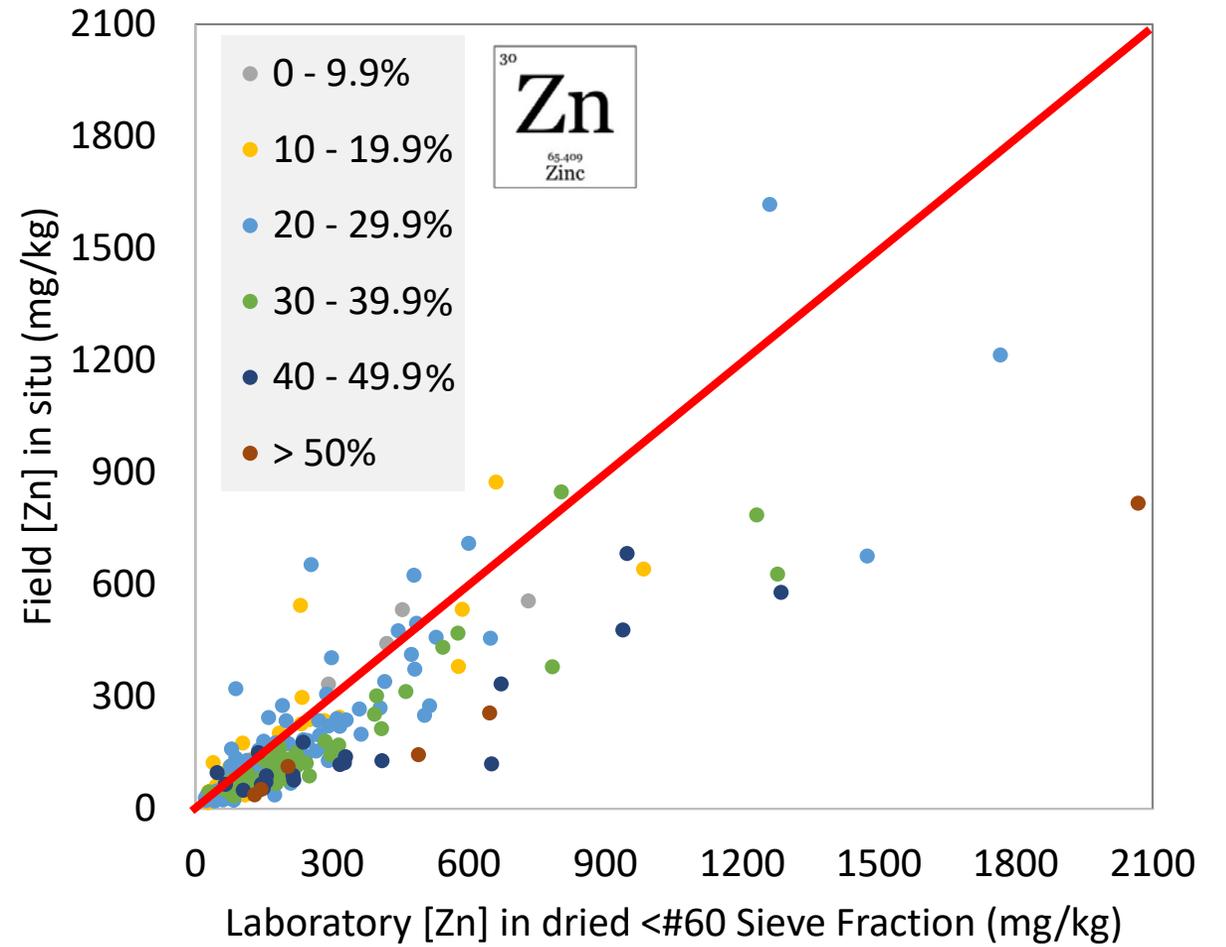
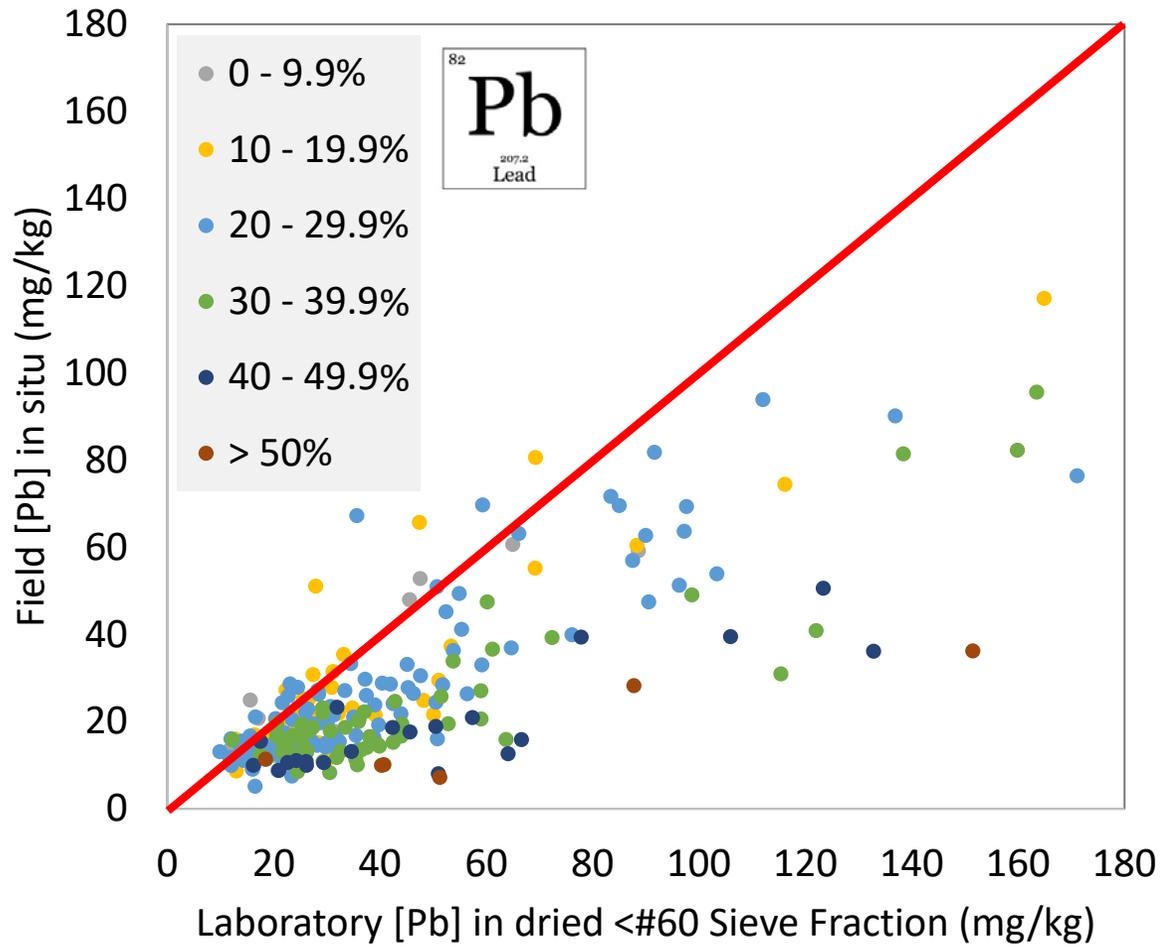
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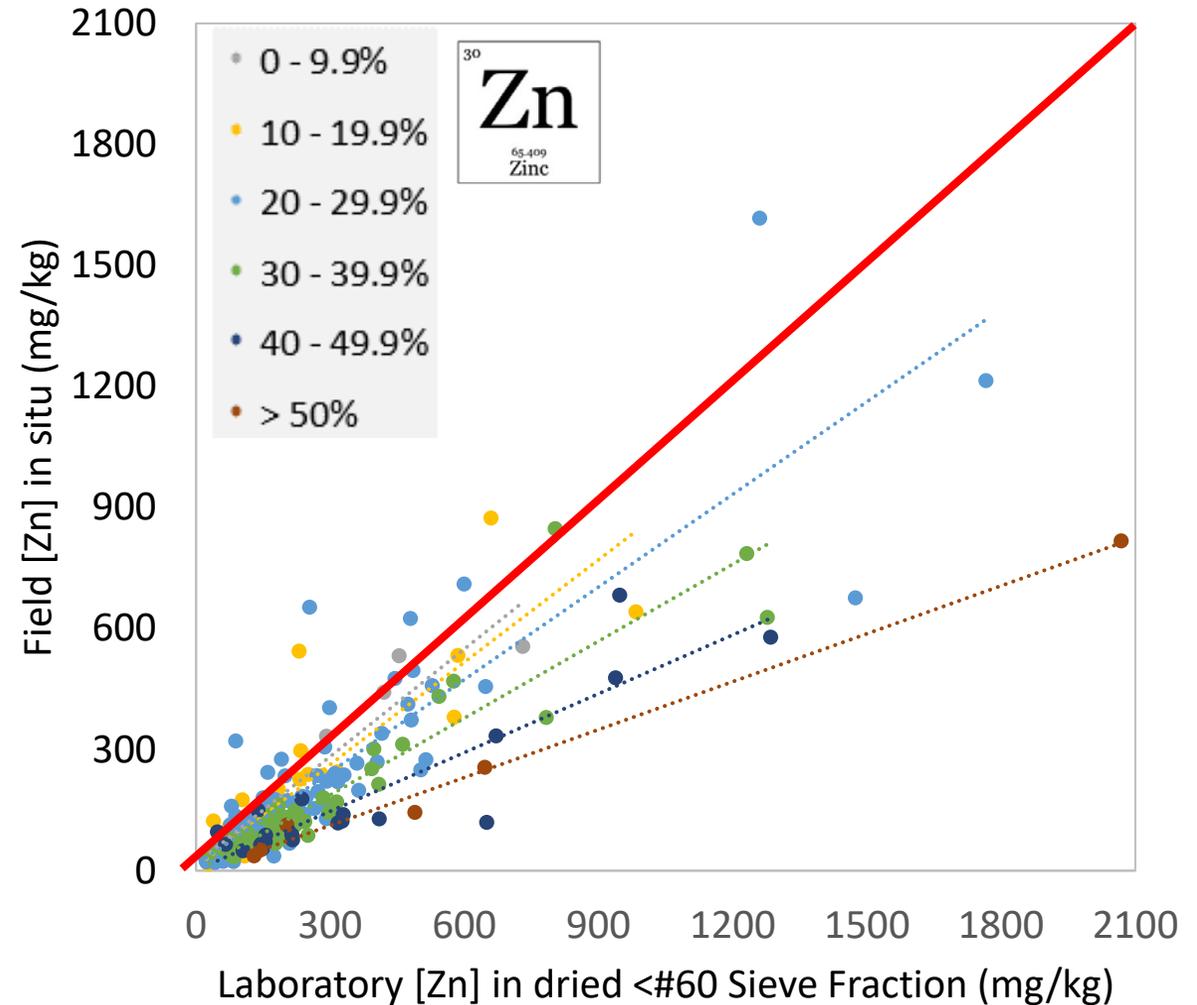
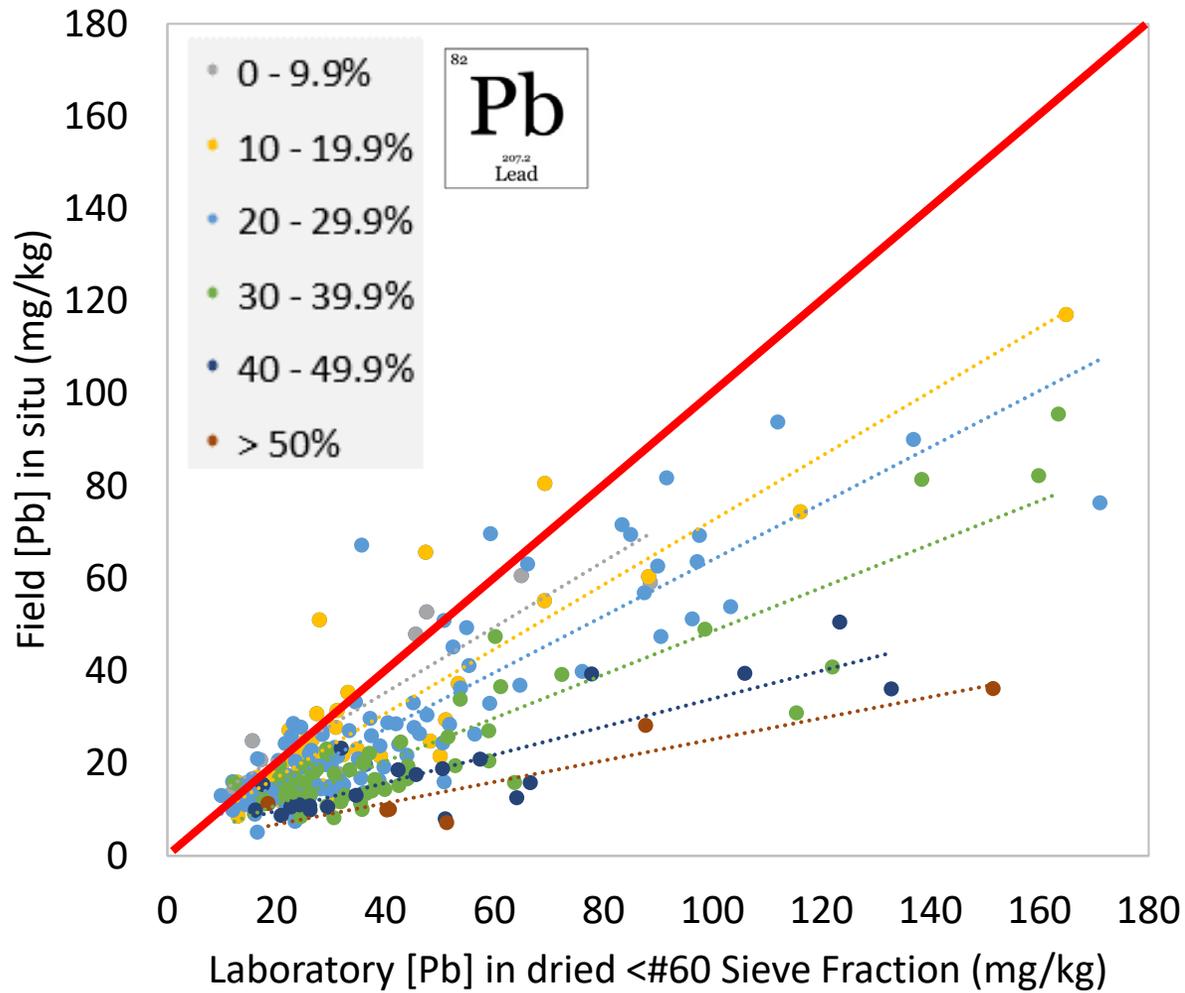
Relation Between Upland Field and Laboratory XRF Concentrations



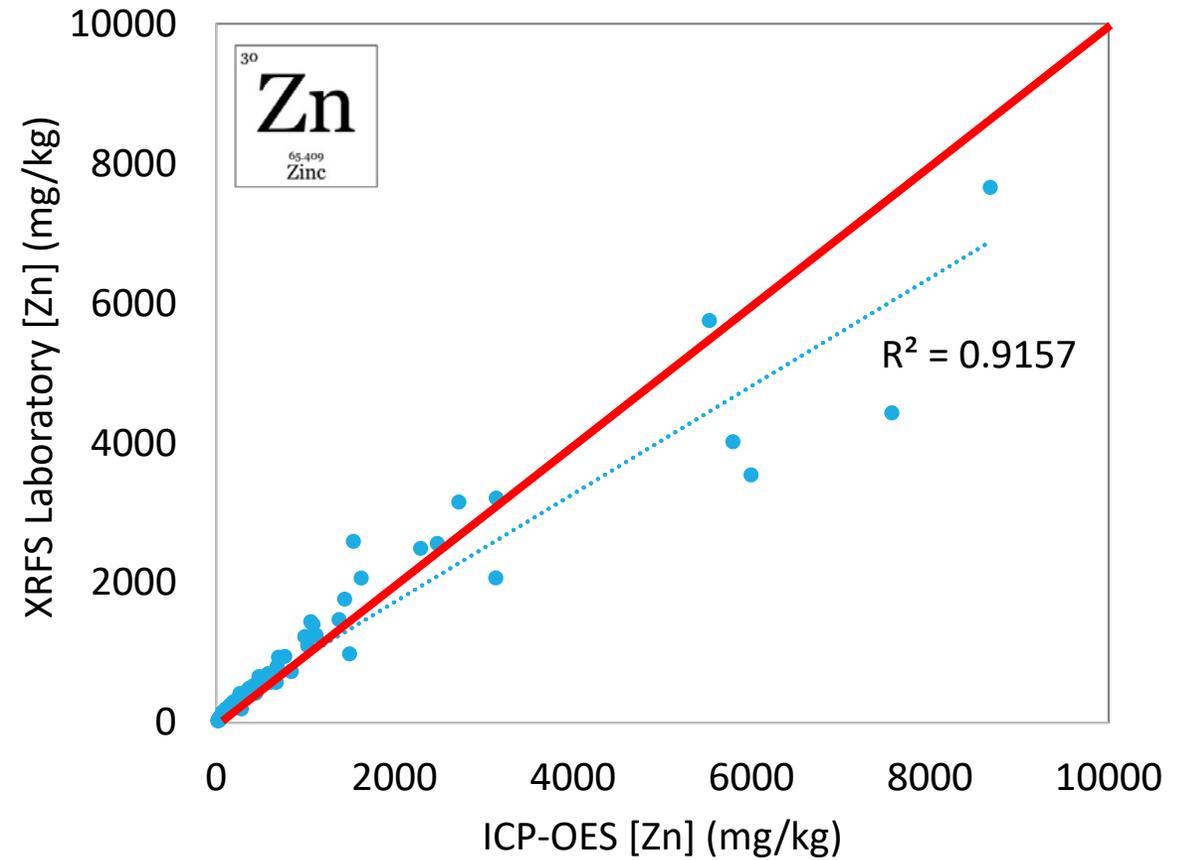
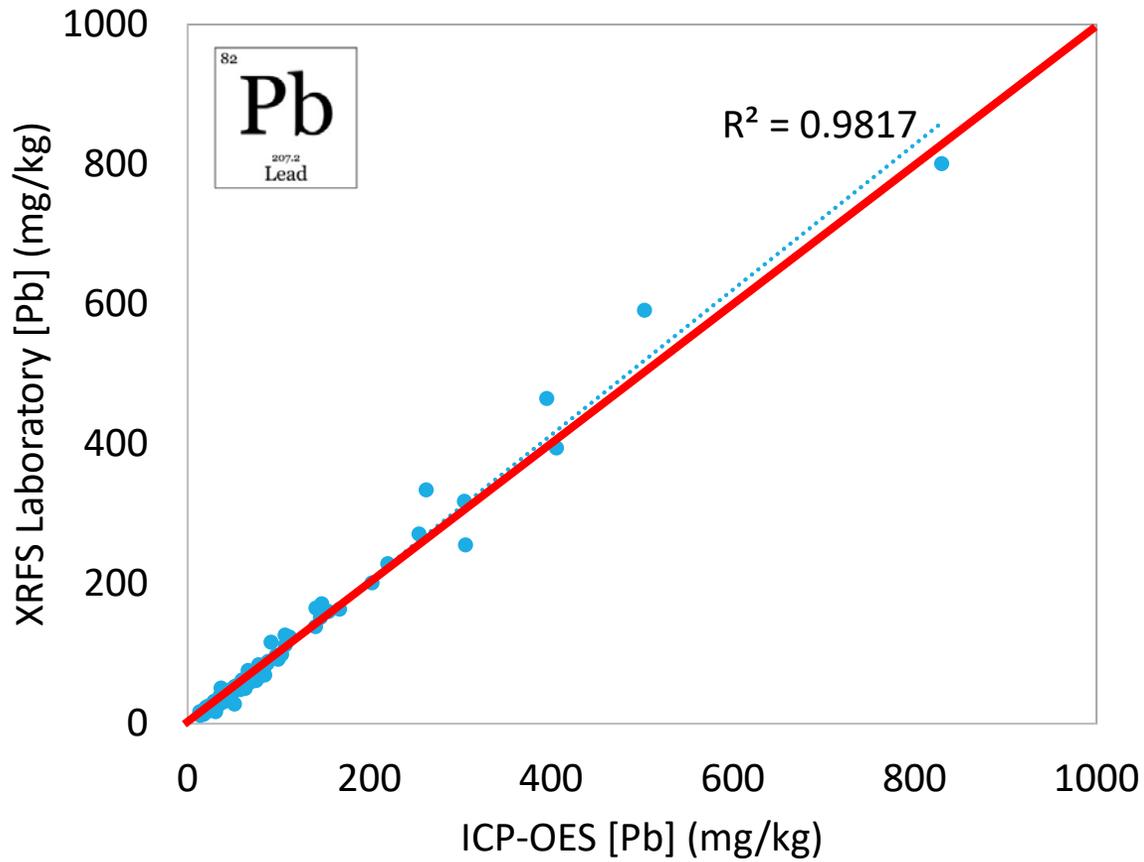
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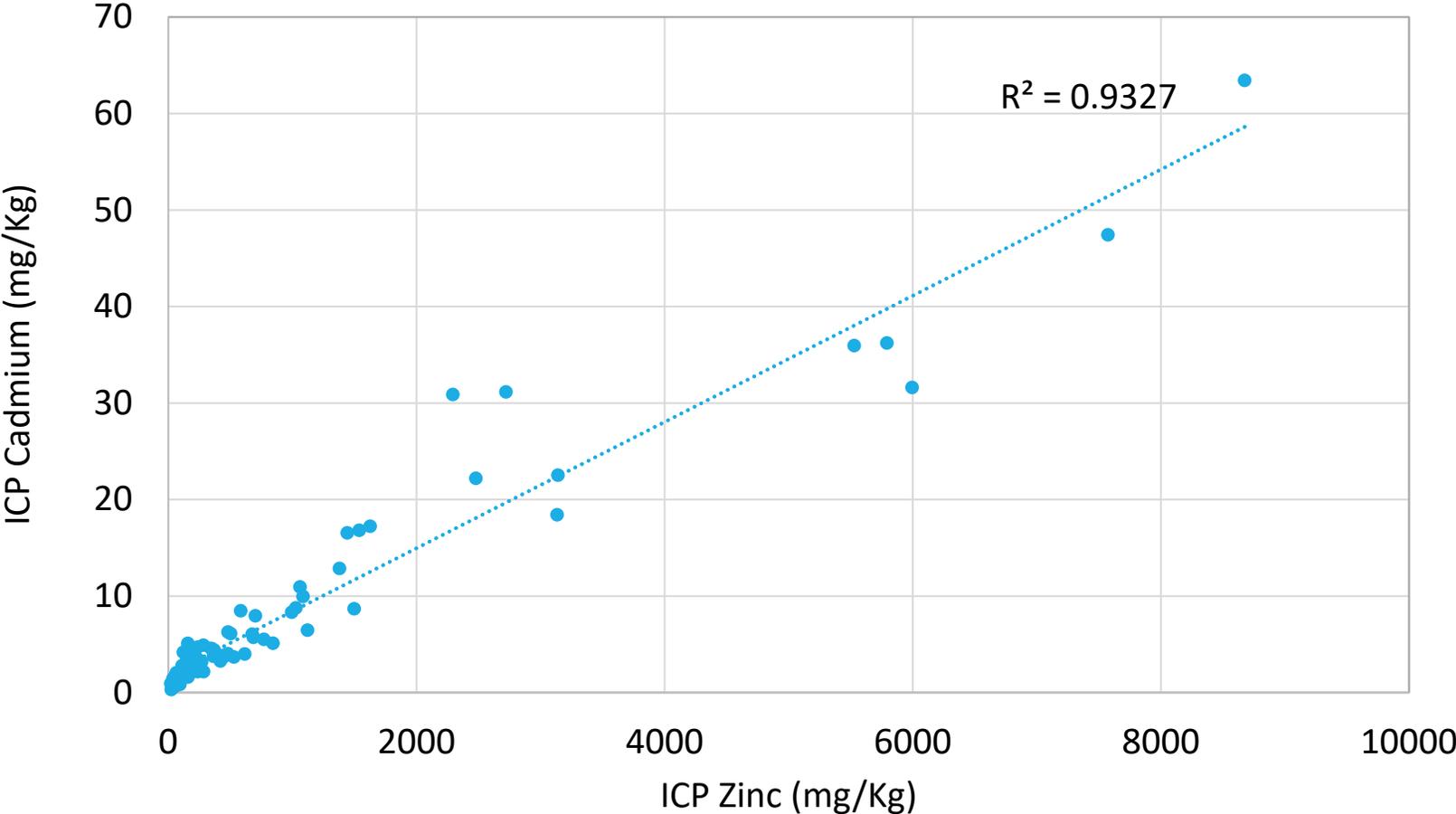
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Pb and Zn Laboratory XRFS v. ICP

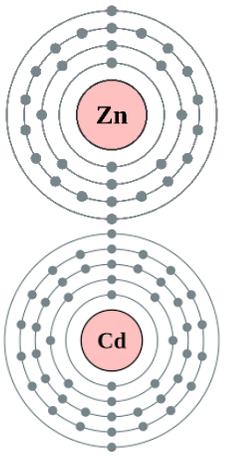


ICP Cadmium v. Zinc



30	2
Zn	18
Zinc	2
65.38	

48	2
Cd	18
Cadmium	18
112.411	2



Organic Matter Analysis

- Lead XRFS and ICP concentrations:
 - Statistically similar for OM >10%
 - Statistically different for OM <10%
- Zinc statistically different for both OM ranges



Conclusions

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- XRF may not be suited for *in situ* soil analysis due to variability in field conditions
- Sample preparation (drying and sieving), is necessary to generate reliable values
- XRF may only operate as a screening tool for zinc due to overreporting
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- OU CREW
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- Darren Shepherd
- Lane Maguire





Questions?