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Method Validation of Metals in Environmental Soil Samples

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Method Validation for Metals in Environmental Samples

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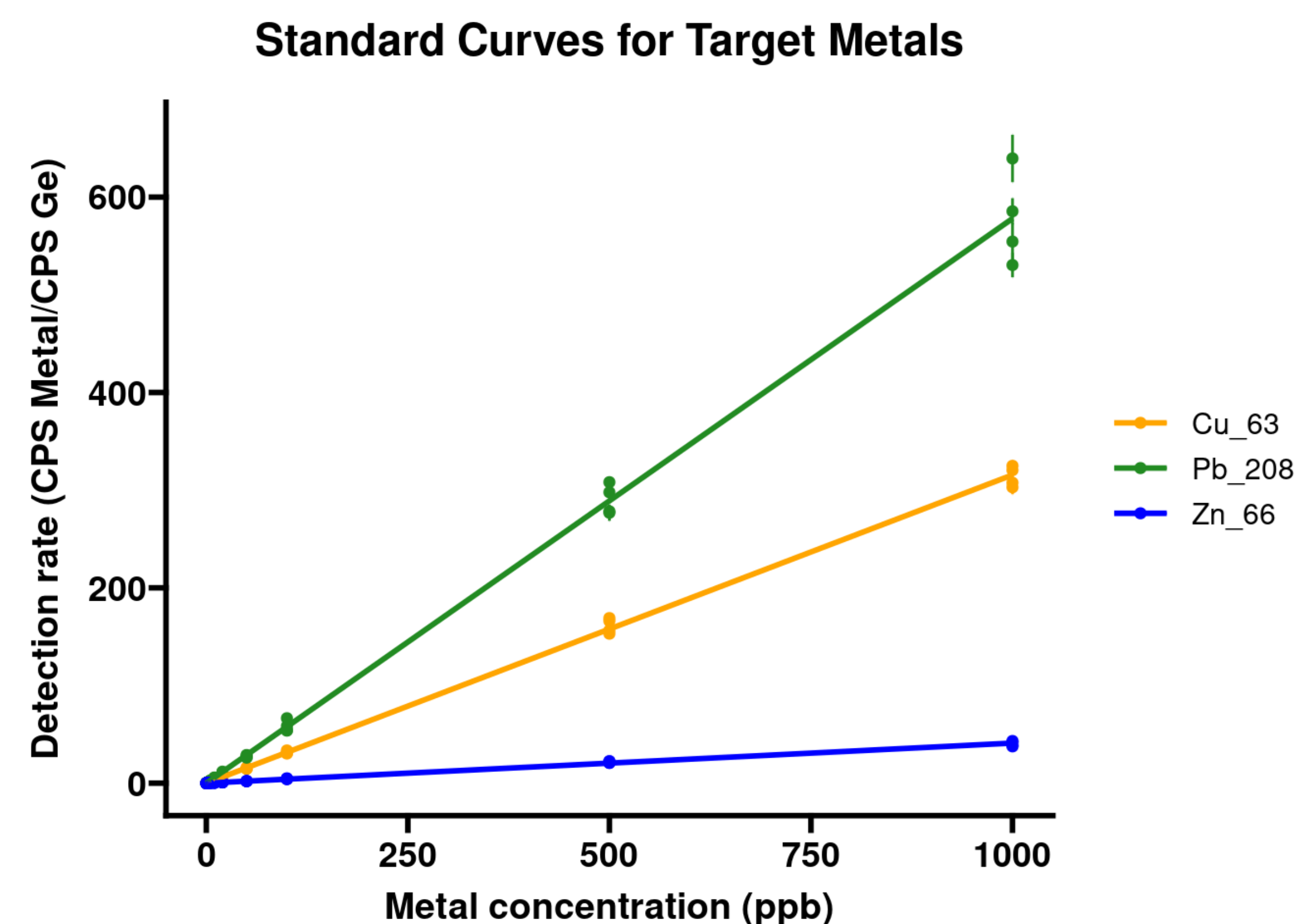
Introduction

Bioswales are a form of green infrastructure intended to reduce the amount of metal accumulation in runoff, although knowledge around their actual effectiveness is limited. Inductively coupled plasma mass spectrometry (ICP-MS) can be used to characterize environmental samples from bioswales but may also present multiple complications.

Driving Question:

How accurate is microwave-assisted digestion, followed by ICP-MS analysis, for the determination of trace metals in environmental samples? How can we optimize our methods?

Linear Range

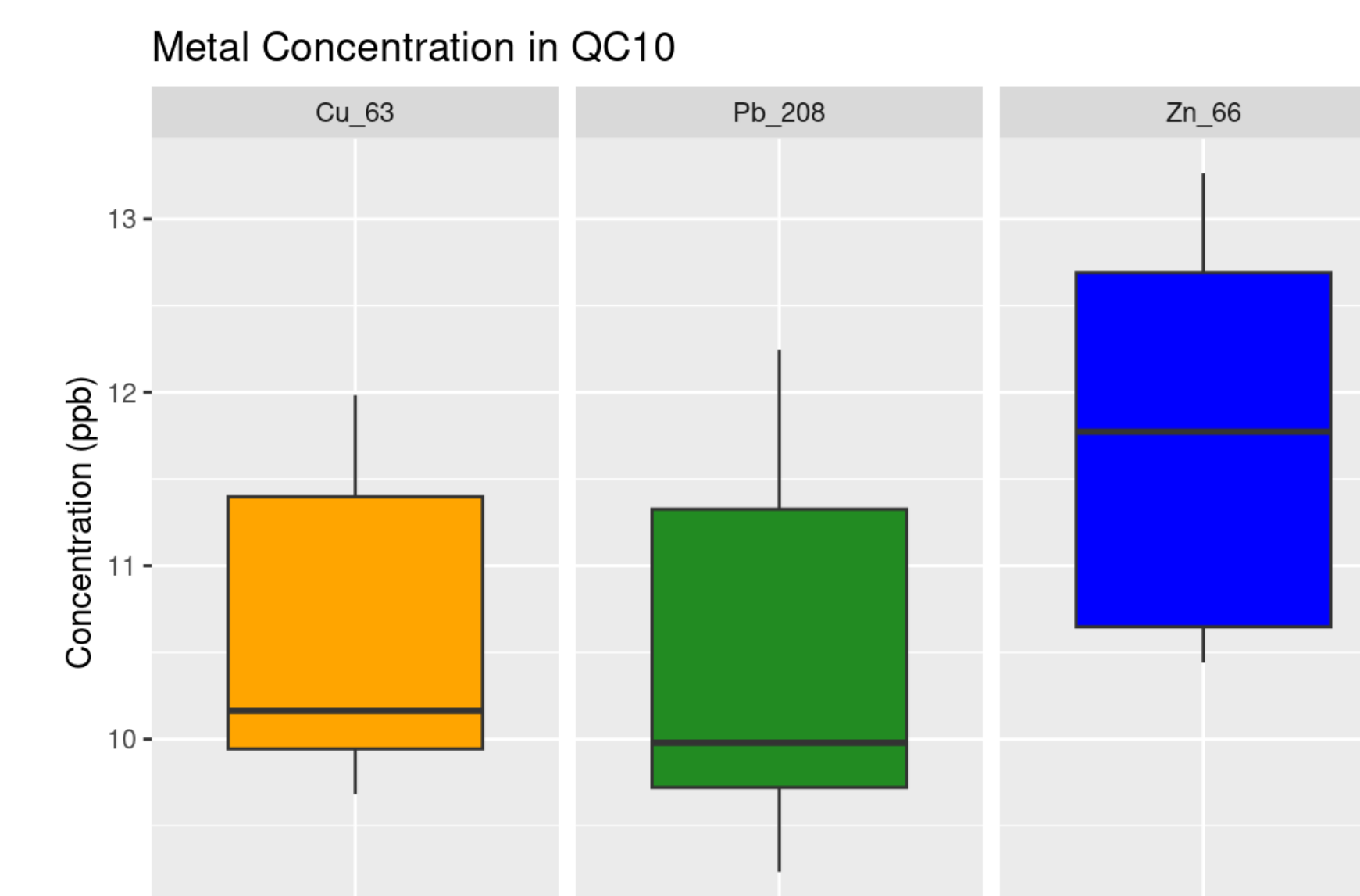


Copper: slope of $0.310 \pm 4 \times 10^{-3} \text{ ppb}^{-1}$, % Relative Uncertainty of 0.68%.

Lead: slope of $0.56 \pm 1 \times 10^{-2} \text{ ppb}^{-1}$, % Relative Uncertainty of 1.2%.

Zinc: slope of $0.0398 \pm 4 \times 10^{-4} \text{ ppb}^{-1}$, % Relative Uncertainty of 0.96%.

Precision



Values for Limit of Detection and Limit of Quantitation using

	Cu	Zn	Pb
Average (ppb)	10.6	12	10
SD (ppb)	±0.9	±1.	±1.
LOD (ppb)	3.	4.	4.
LOQ (ppb)	9.	11.	11.
% Relative Un	8.5 %	9.5 %	10.4 %

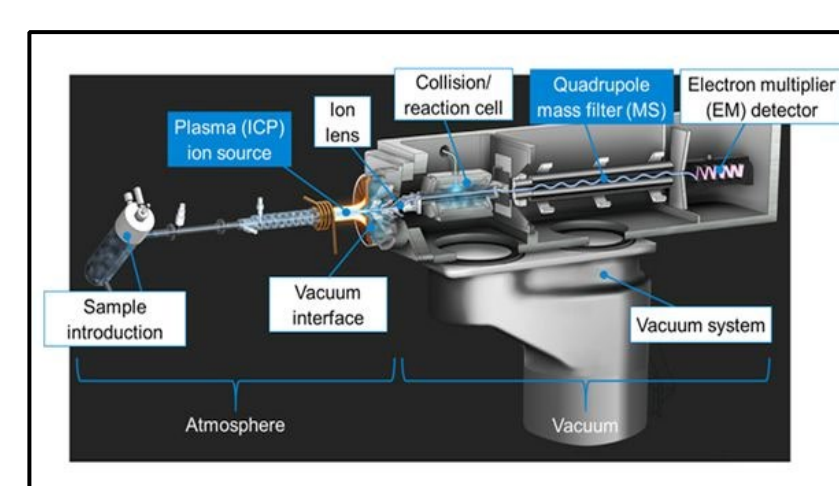
Method



Environmental Sample



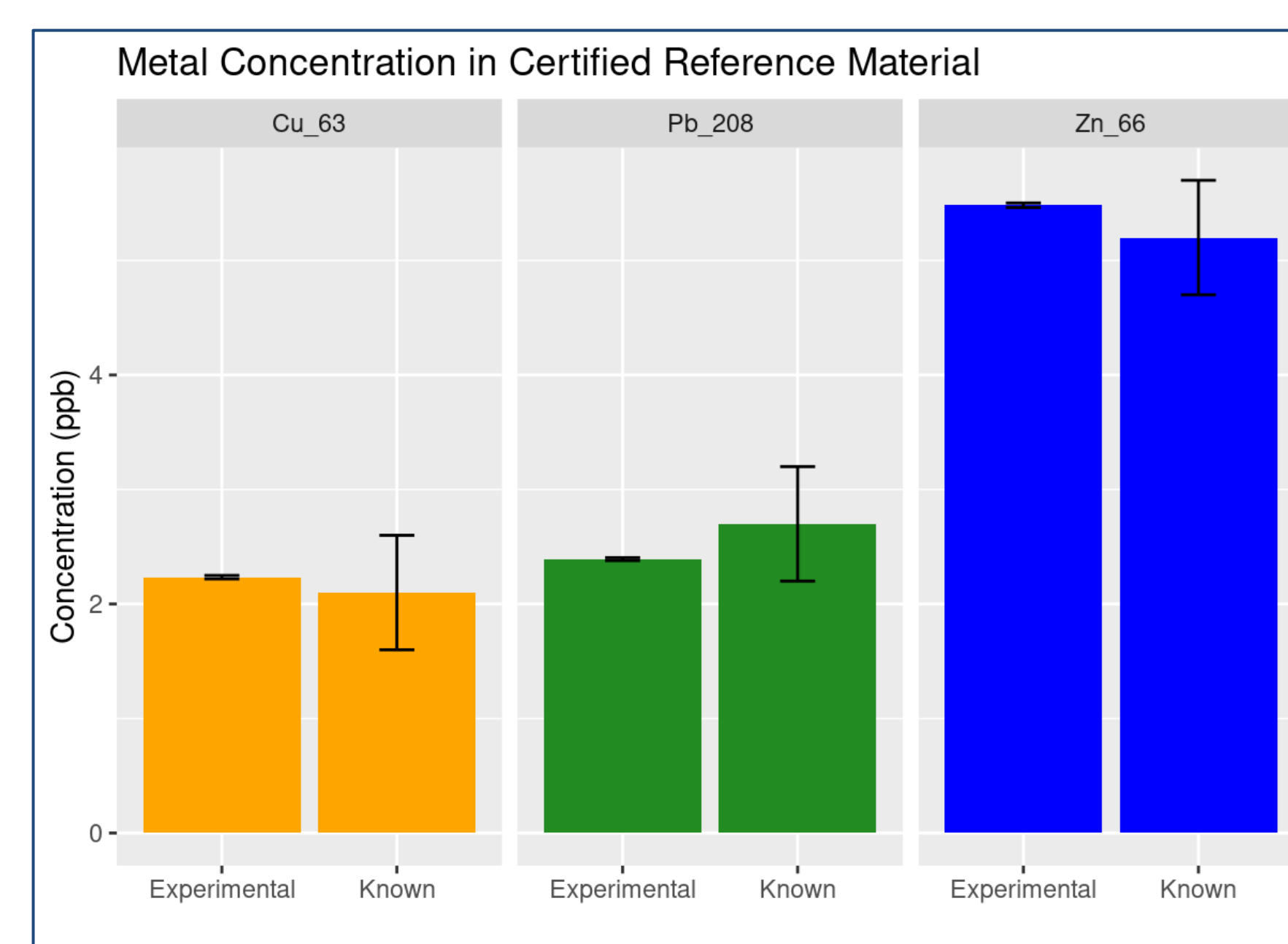
Microwave Digestion



ICP-MS Analysis

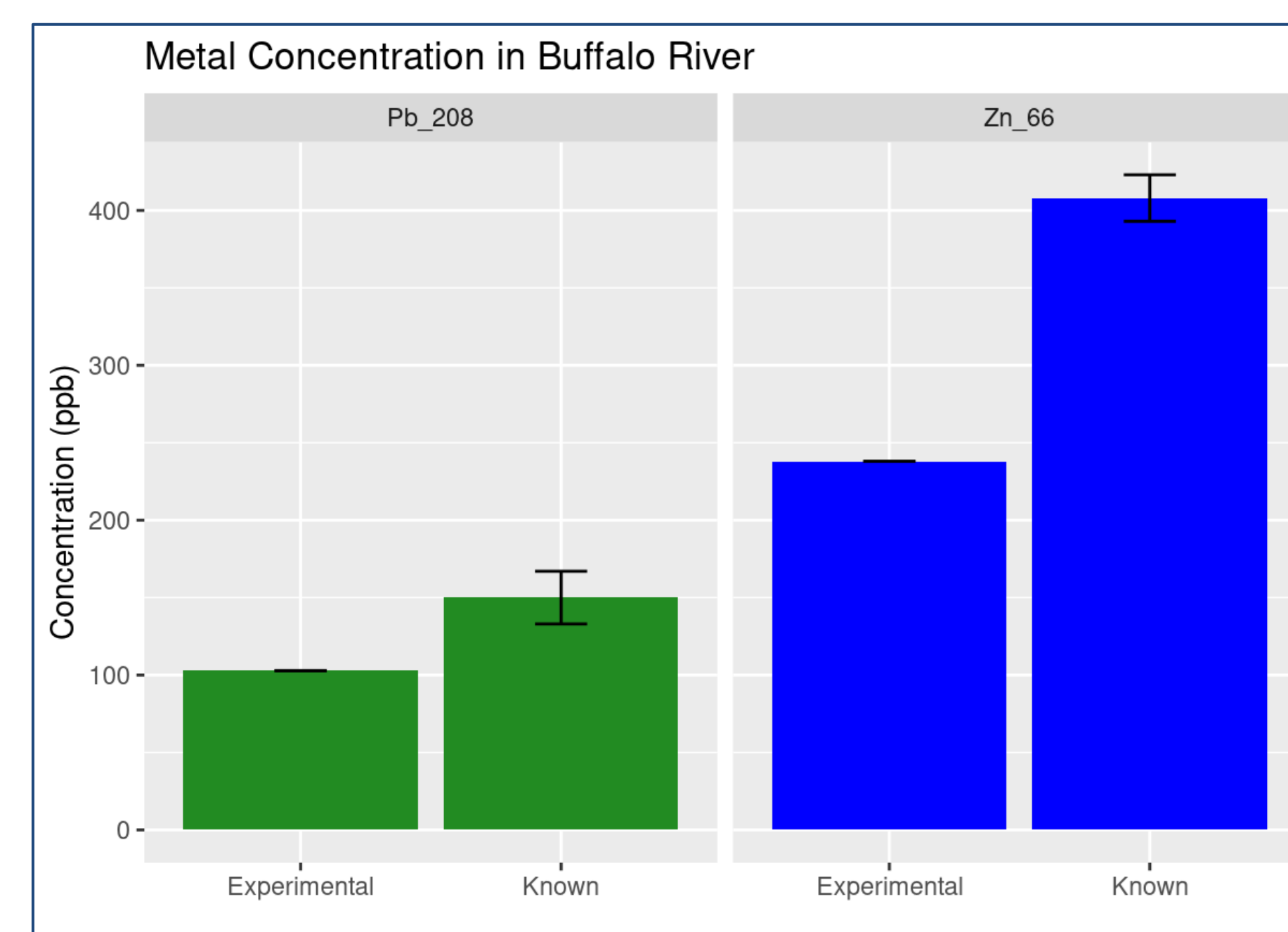
- Environmental Sample Control
 - NIST Buffalo River²
- Digestions in conc. HNO_3
 - organic matter $\rightarrow \text{CO}_2(\text{g}) + \text{H}_2\text{O}(\text{l}) + \text{NO}_x(\text{g})$
- ICP-MS Controls
 - TraceCert Periodic Table Mix 1 - Standards
 - Centipur MultiX (CRM)¹

Accuracy



ICP-MS resulted in no statistical difference between the experimental and expected results for the CRM¹

Digestion followed by ICP-MS statistically underestimated the true value of Pb and Zn in the Buffalo River Soil Samples².



Conclusion

Our digestion method is insufficient at liberating the target metals from the complex environmental samples, as supported by the underestimation of reported metal in the Buffalo River samples.

The ICP-MS is sufficient for reporting metal values of simple liquid samples, as supported by the metal concentration in the CRM.

Acknowledgments

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Literature Cited

- (1) Yildirim, Ayfer. *Certipur Certified Reference Material*. Frankfurter Str. 250, 64293 Darmstadt, Germany, Merck KGaA, 9 Nov. 2016.
- (2) Steven J. Choquette. *Reference Material 8704*. Gaithersburg, MD 20899, National Institute of Standards and Technology, 24 July 2018.