

Using PT to Assess Laboratory Subsampling of Soil



CALA
Trust, measured accurately

Acknowledgements

- A proficiency testing scheme to evaluate the effectiveness of laboratory sample reduction of a soil sample (Middlebrook K., Accred Qual Assur 24:137-142, 2019).
- Harold Malle, Lois Esler, Halka Klement of Environmental Science and Technology Laboratories, Environment and Climate Change Canada.

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Typical PT Schemes for Metals in Soil

- PT samples pre-dried and sieved.
- Thoroughly homogenized, between-bottle and within-bottle.
- Designed to test a laboratory's ability to digest and analyse ideal soil samples.
- Not designed to test their ability to obtain a representative sub-sample.

Purpose of the PT Challenge

- To evaluate ability of laboratory to obtain a representative sub-sample from a non-homogeneous soil sample.

Typical CALA Scheme Design

- Artificial soil matrix used for all samples.
 - 48.5% Silica sand
 - 11% sphagnum
 - 20% silica gel
 - 20% Kaolin clay
 - 0.5% calcium carbonate

Typical CALA Scheme Design

- Each batch spiked with metal solution and homogenized in a large capacity V-blender.



Typical CALA Scheme Design

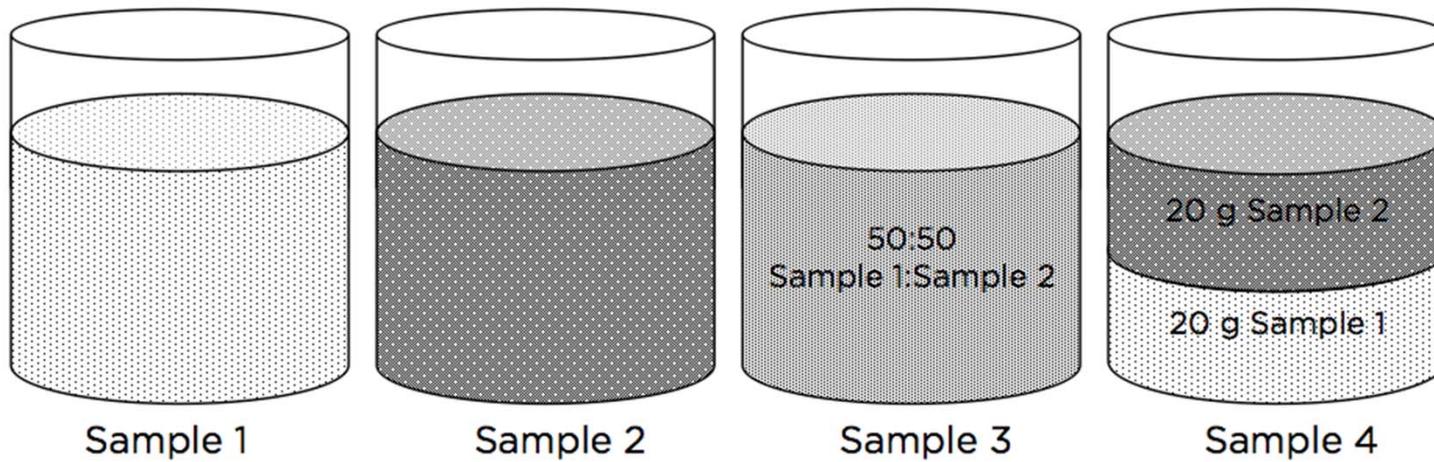
- Dispensed into glass ointment jars (~40 g).
- Each PT round consists of four different samples.



Modified CALA Scheme Design

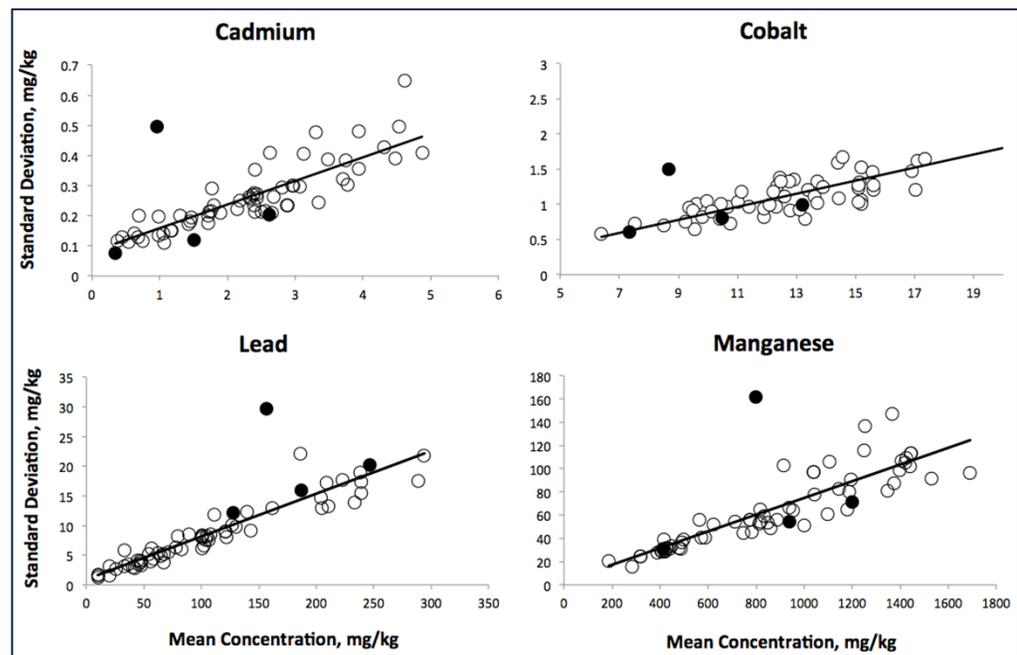
- Samples 1 and 2 prepared as normal but in a larger bulk quantity (~2x).
- Sample 3 prepared as 50:50 mix of Sample 1 and 2, homogenized before dispensing to jars.
- Sample 4 prepared by weighing 20 g of sample 1 into jars. 20 g of sample 2 was then weighed into same jars (i.e., 50:50 mix but not homogeneous).

Modified CALA Scheme Design



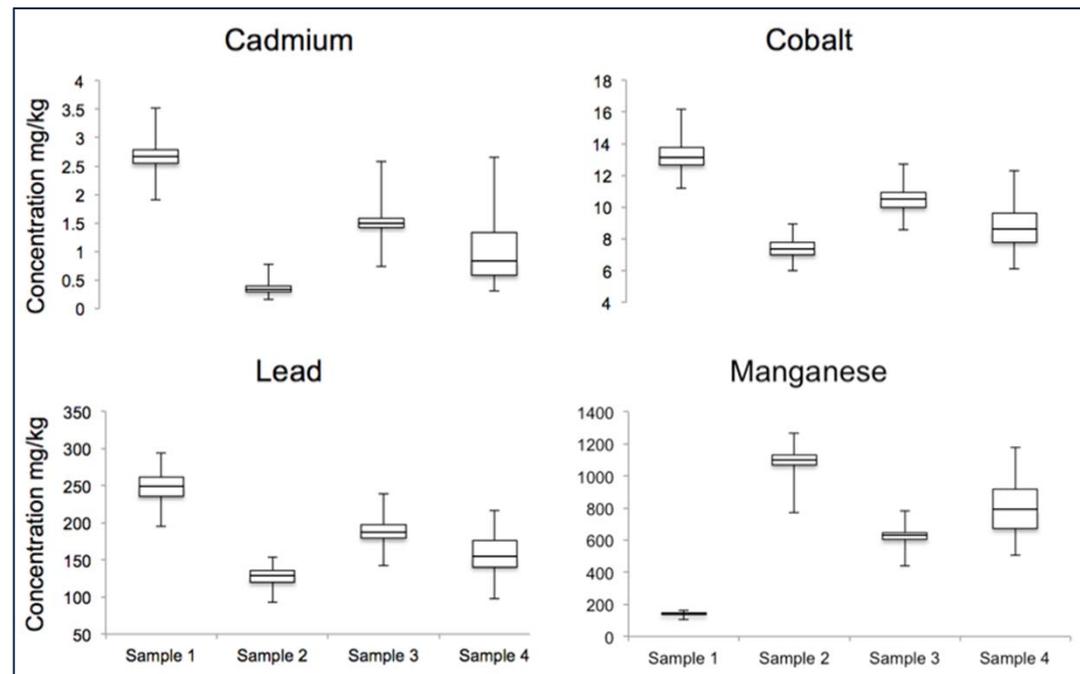
Results

- Robust Mean vs Stdev



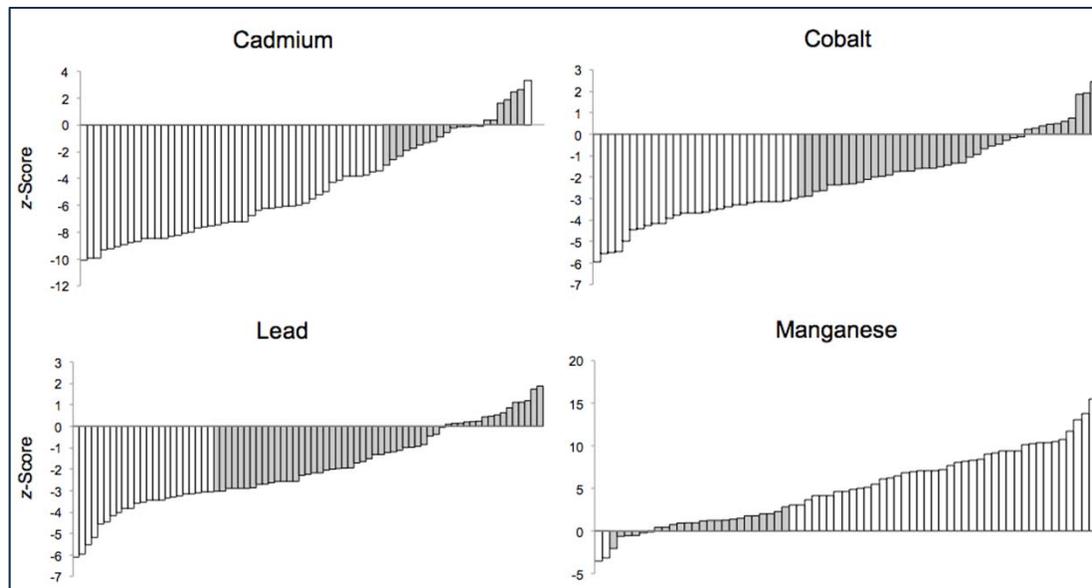
Results

- Data Distribution



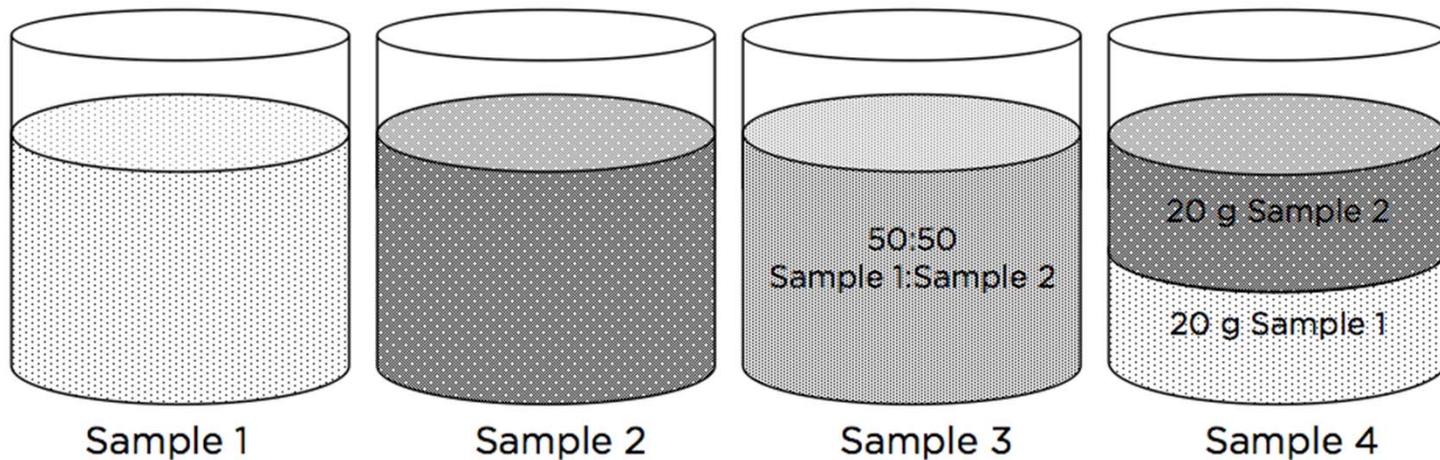
Results

- z-Scores for Challenge Sample



Conclusions

- Distribution of results suggest a bias towards the surface part of the sample.



Interpretation and Limitations

- Inadequate procedure for sub-sampling.
- Procedure for sub-sampling adequate but analyst not following it.
- Analysts instructed to assume PT samples are homogeneous.

Potential Risks

- e.g., Site Remediation
 - Risk to environment if concentration underestimated.
 - Increased remediation cost if site identified as hazardous due to overestimate of concentration.
- Both pose significant liability to laboratory.

QUESTIONS

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