ERDC
Engineer Research and Development Center

Incremental Sampling Methodology (ISM) for Metals: Number of Increments and Milling Necessity

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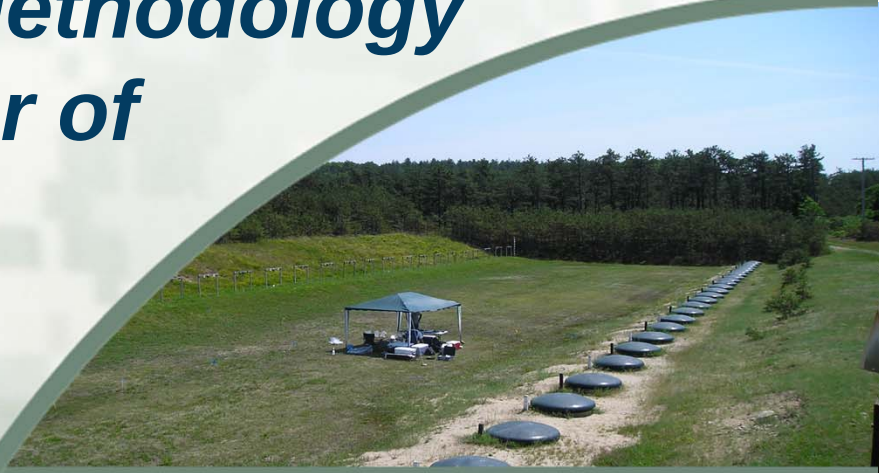


US Army Corps of Engineers®

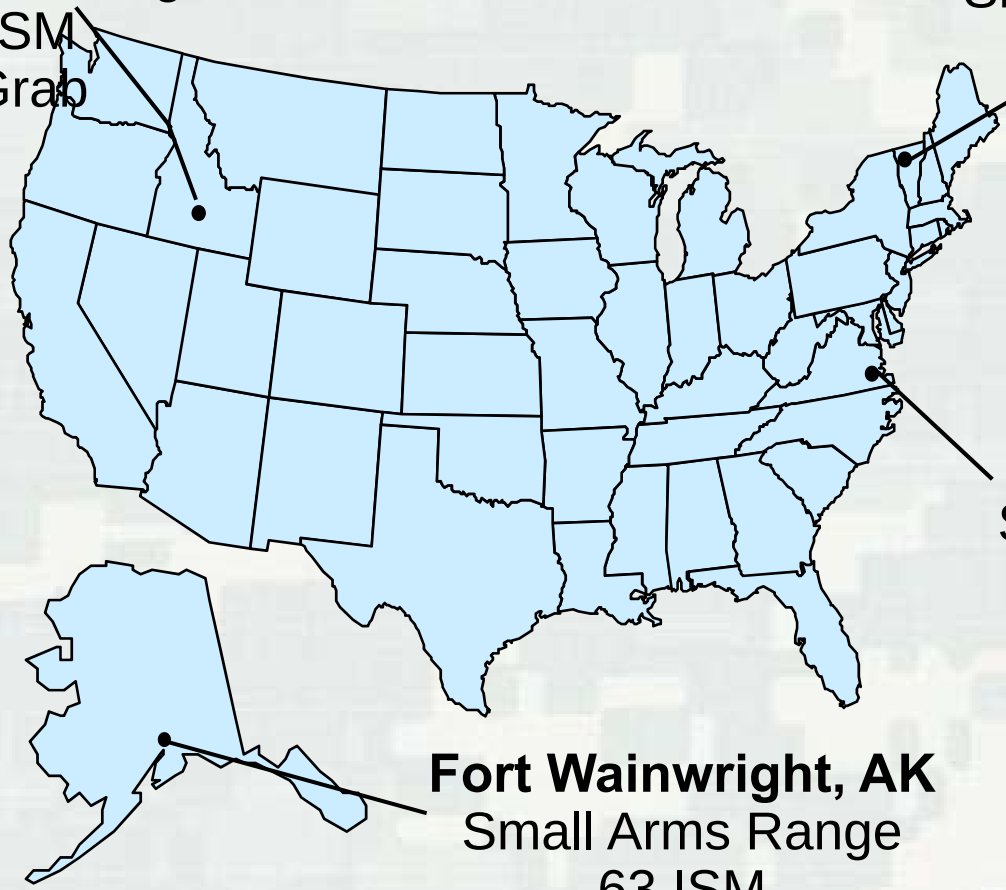
Soil Sampling Issues

- Unrepresentative results using conventional grab soil sampling at military ranges with energetic & metallic residues
- Poor precision for grabs (e.g., for duplicates)
- Large uncertainty at concentrations near decision limits
- Increasing State regulatory insistence for Incremental Sampling Methodology (ISM) for soils
- USEPA SW-846 work group to update Method 3050B to Method 3050C with ISM. By late 2015?

Demonstration Sites

Kimama, ID
Small Arms Range
21 ISM
30 Grab

Camp Ethan Allen, VT
Small Arms Range
43 ISM
36 Grab

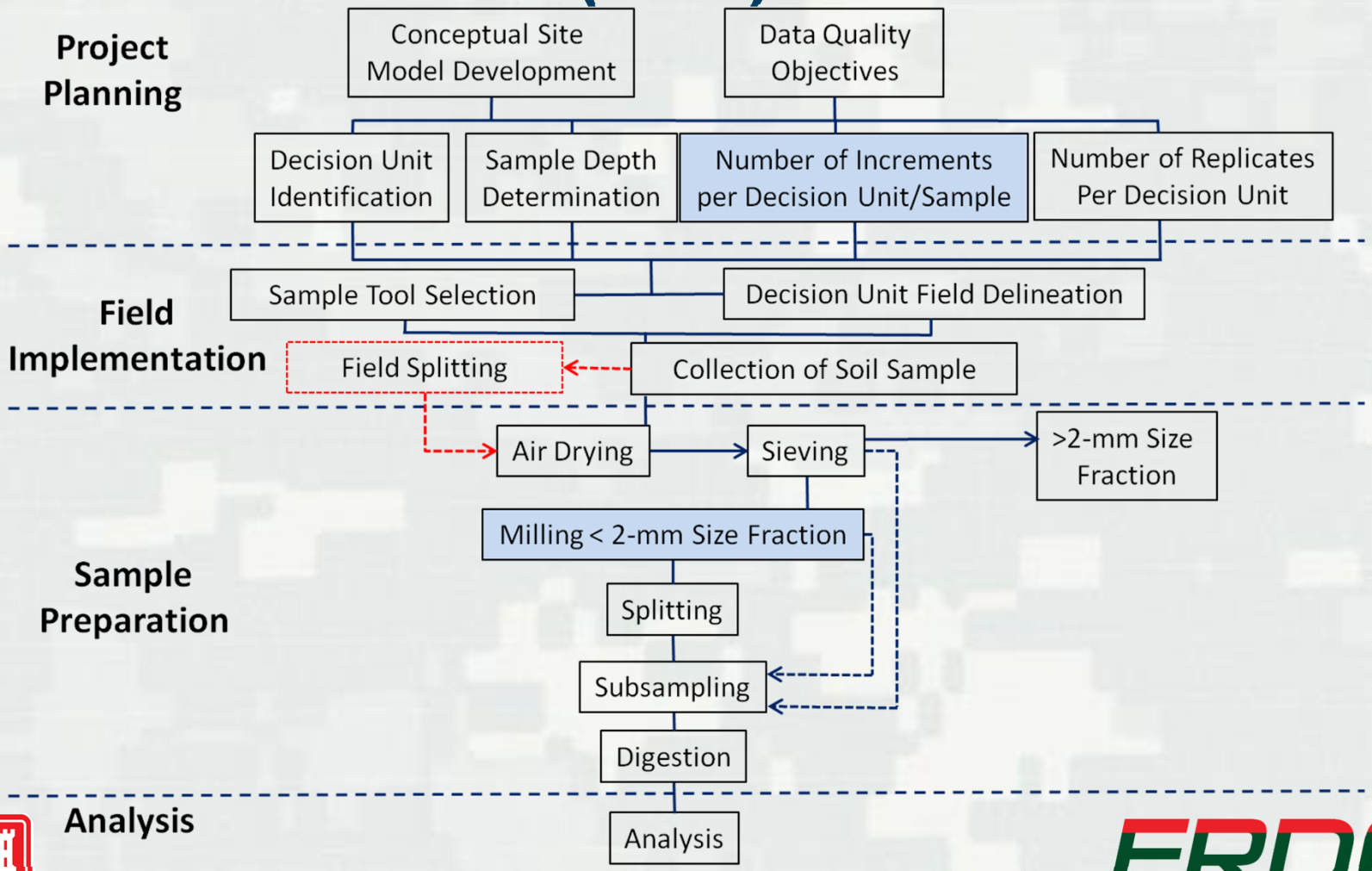


Fort Eustis, VA
Small Arms Range
27 ISM
33 Grab

Fort Wainwright, AK
Small Arms Range
63 ISM
52 Grab

Metallic Residues

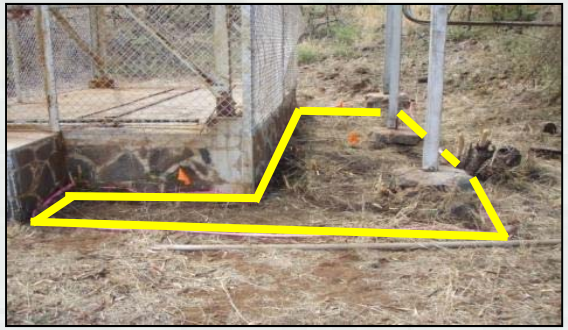
Incremental Sampling Methodology (ISM)



Decision Units (DUs)

Volume of soil to be sampled for which a decision needs to be made using the sample results.

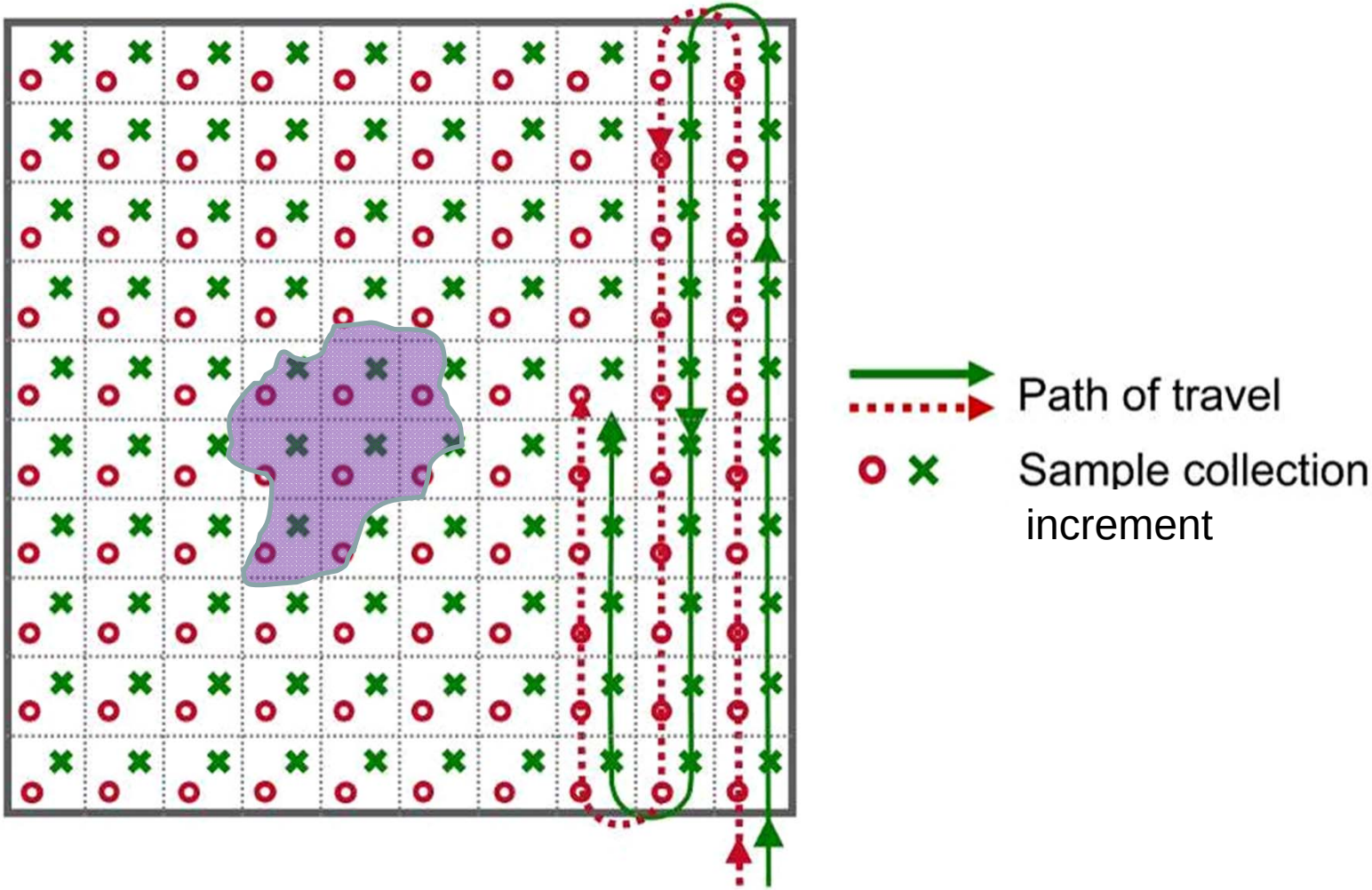
Source Areas



Exposure Areas

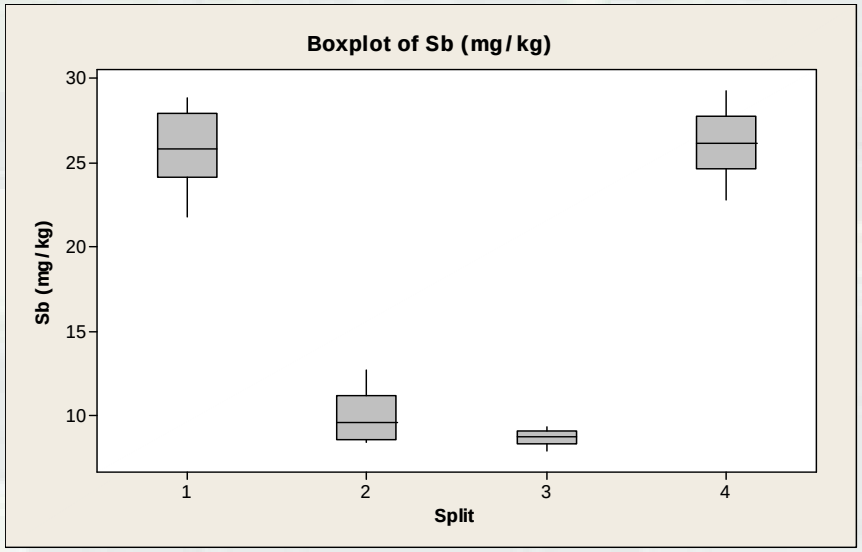
Size, shape, and type of DU determined during systematic planning and depend on Data Quality Objectives (DQOs).

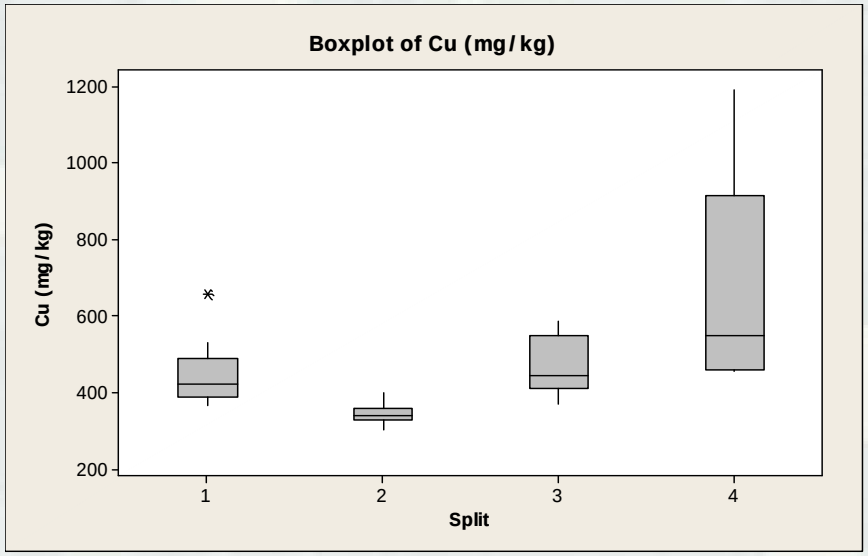
Incremental Sampling

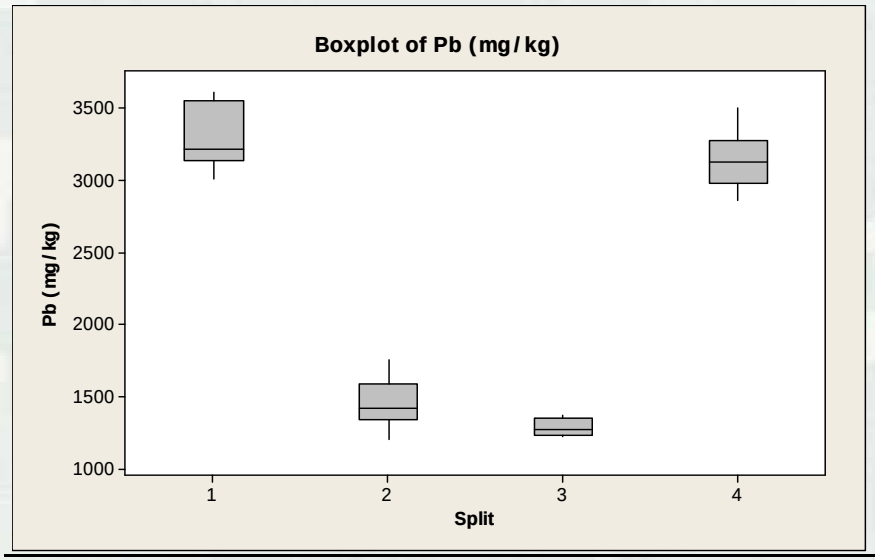


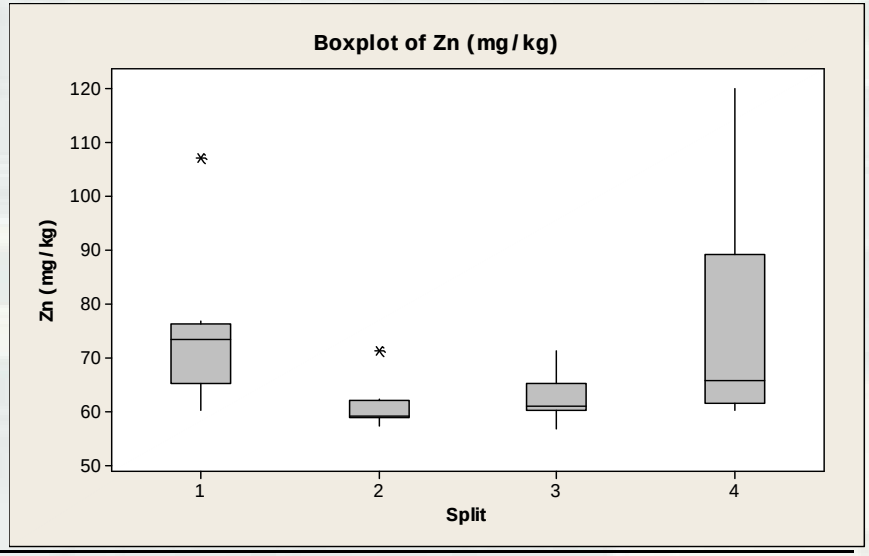
Can ISM samples be split in the field to reduce the mass that needs to be shipped to and processed in the laboratory?

Field Cone-and-Quartered Splits

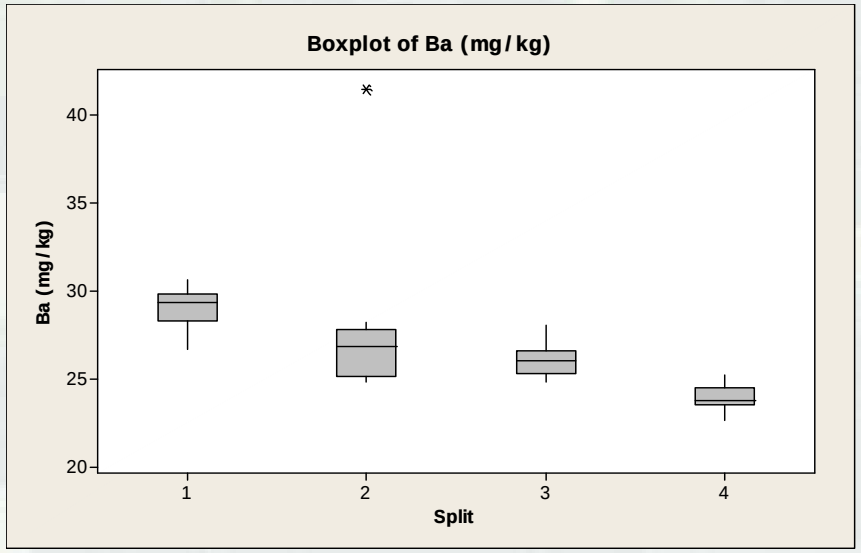


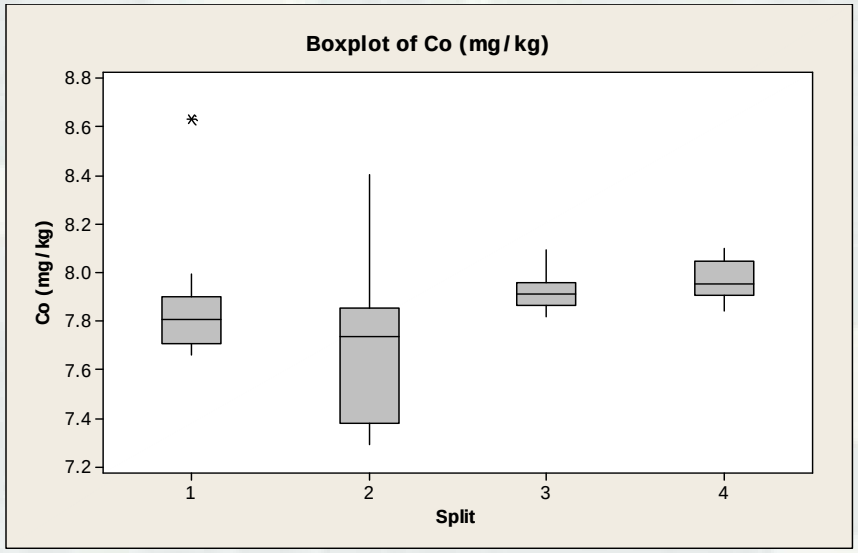


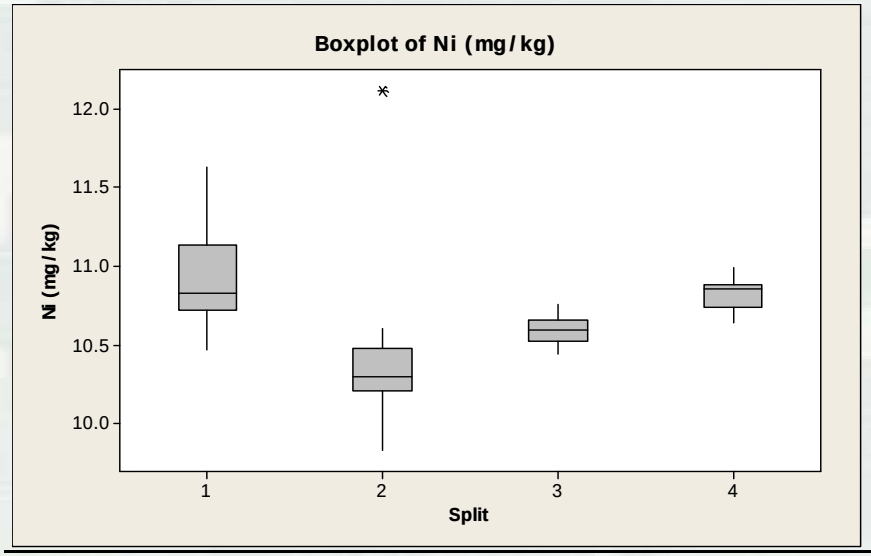


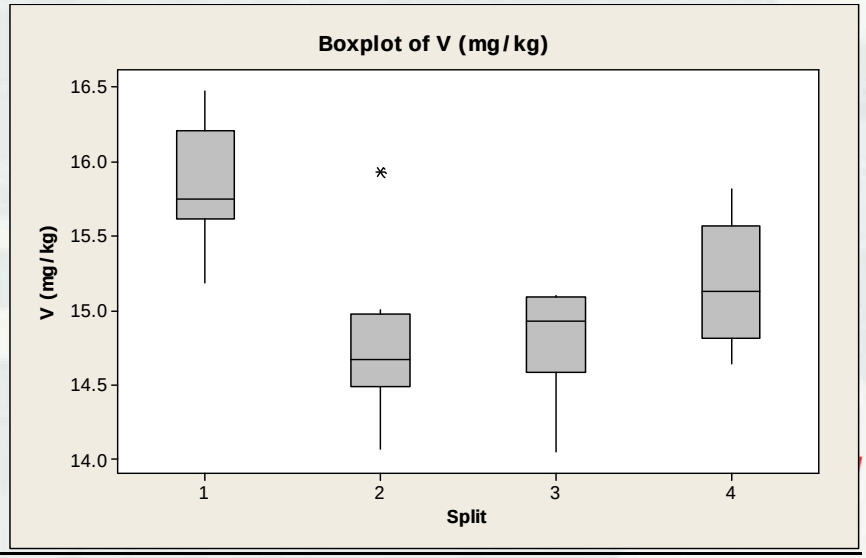


Background Metals









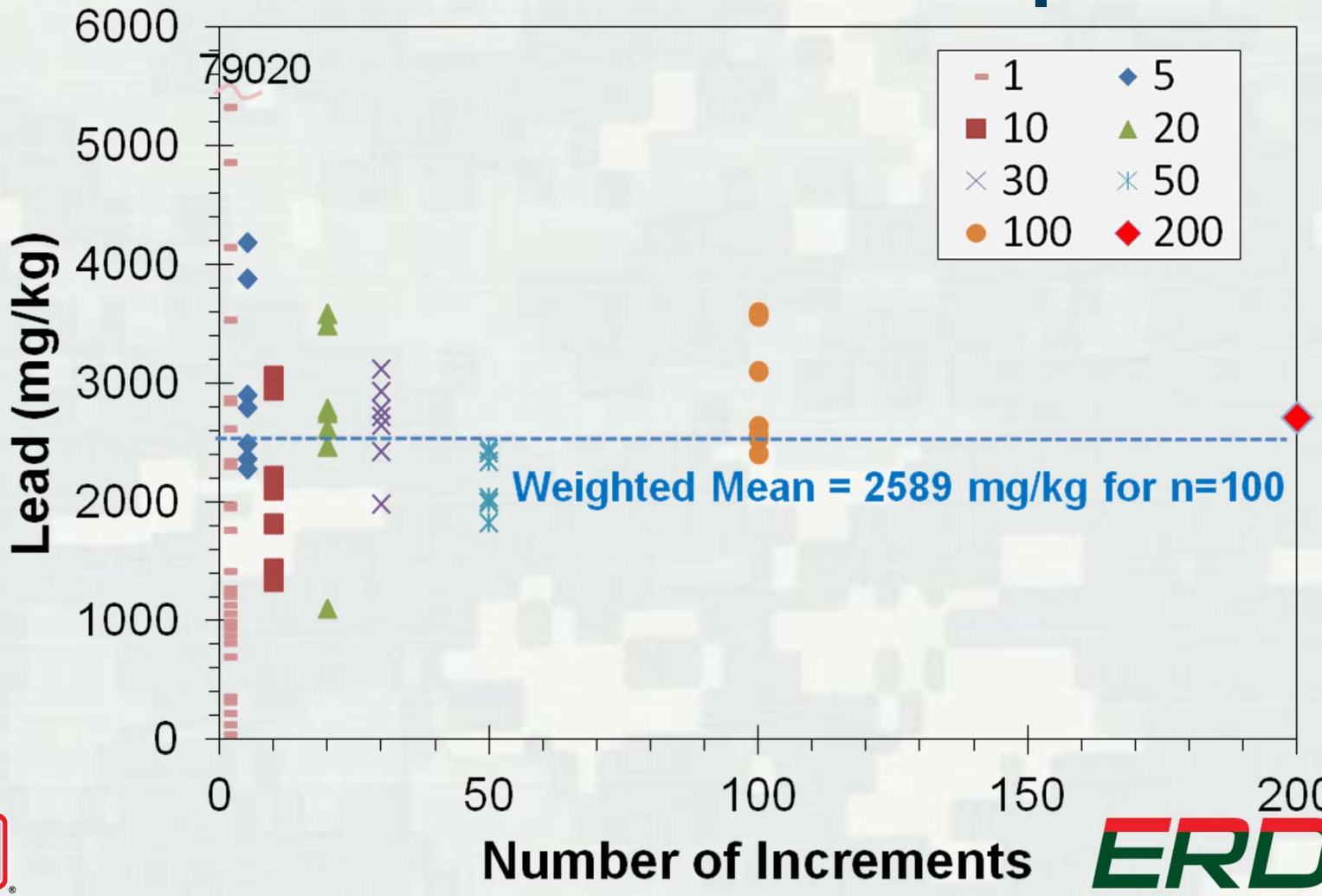
Number of Increments

Number of Increments vs. Relative Standard Deviation

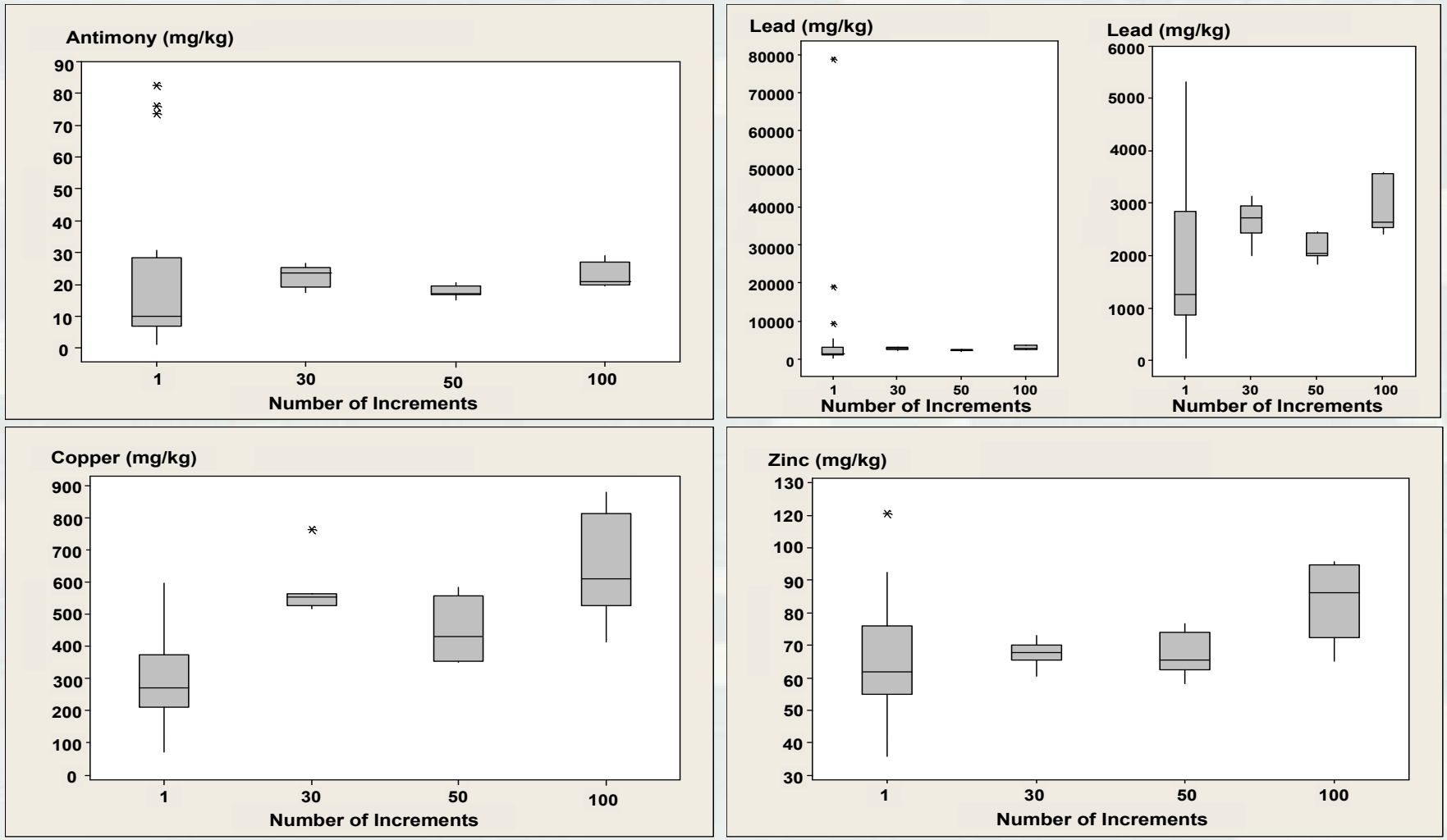
ISM	Percent Relative Standard Deviation (RSD)									
m	**Al**	**Cr**	**Cu**	**Fe**	**Mn**	**Ni**	**Pb**	**Sb**	**V**	**Zn**
5	3	10	**22**	4	4	3	**25**	**25**	6	**9**
10	8	6	**162**	4	4	4	**32**	**63**	5	**154**
20	27	121	**26**	22	18	26	**30**	**50**	32	**15**
30	3	7	**15**	10	4	3	**14**	**15**	6	**6**
50	3	15	**21**	10	2	4	**11**	**11**	6	**10**
100	3	7	**26**	4	2	2	**17**	**17**	3	**15**
200	6	3	**18**	4	5	2	**4**	**7**	1	**11**

m = number of increments per MI sample

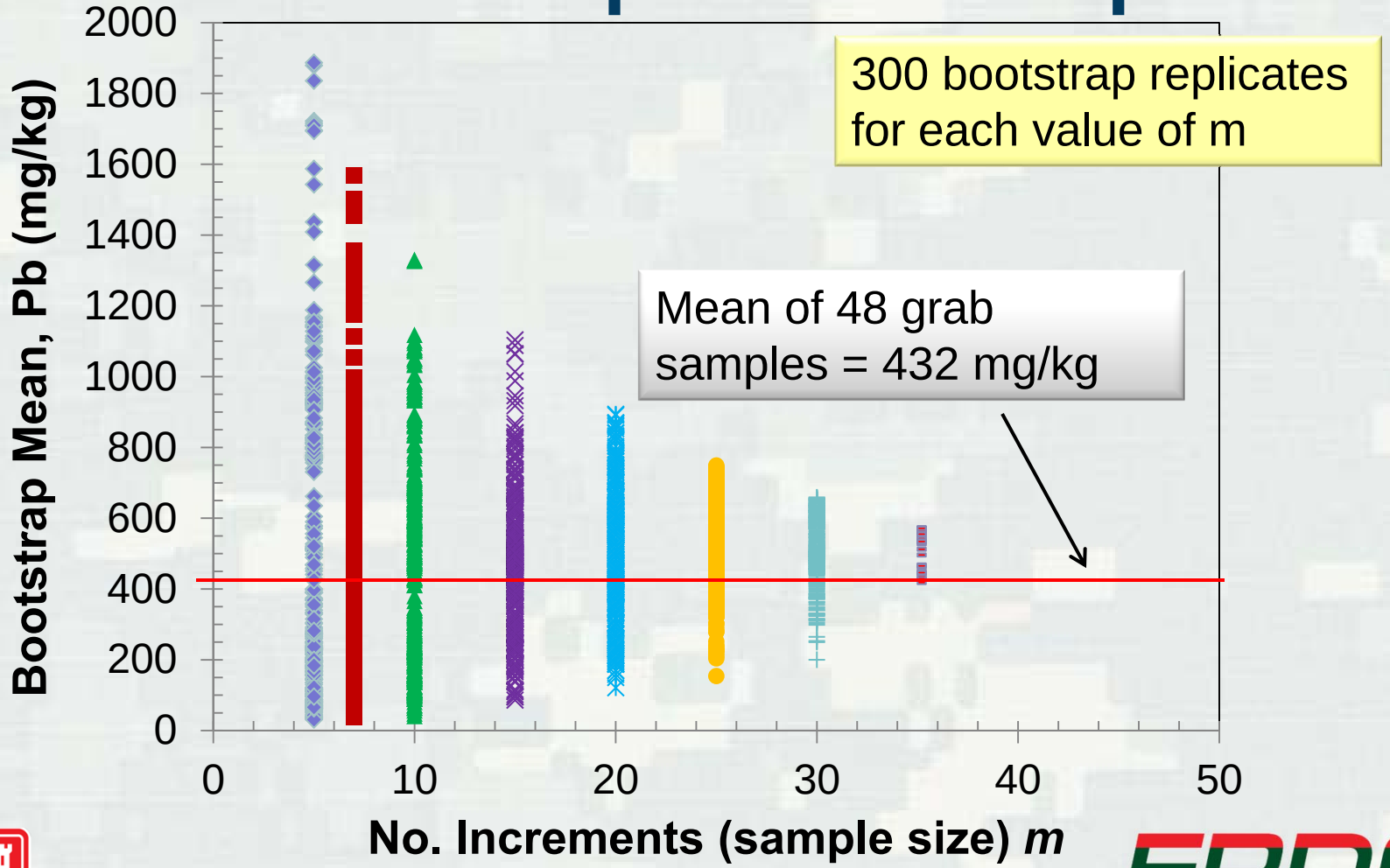
Lead Concentration vs. Number of Increments of ISM Field Replicates



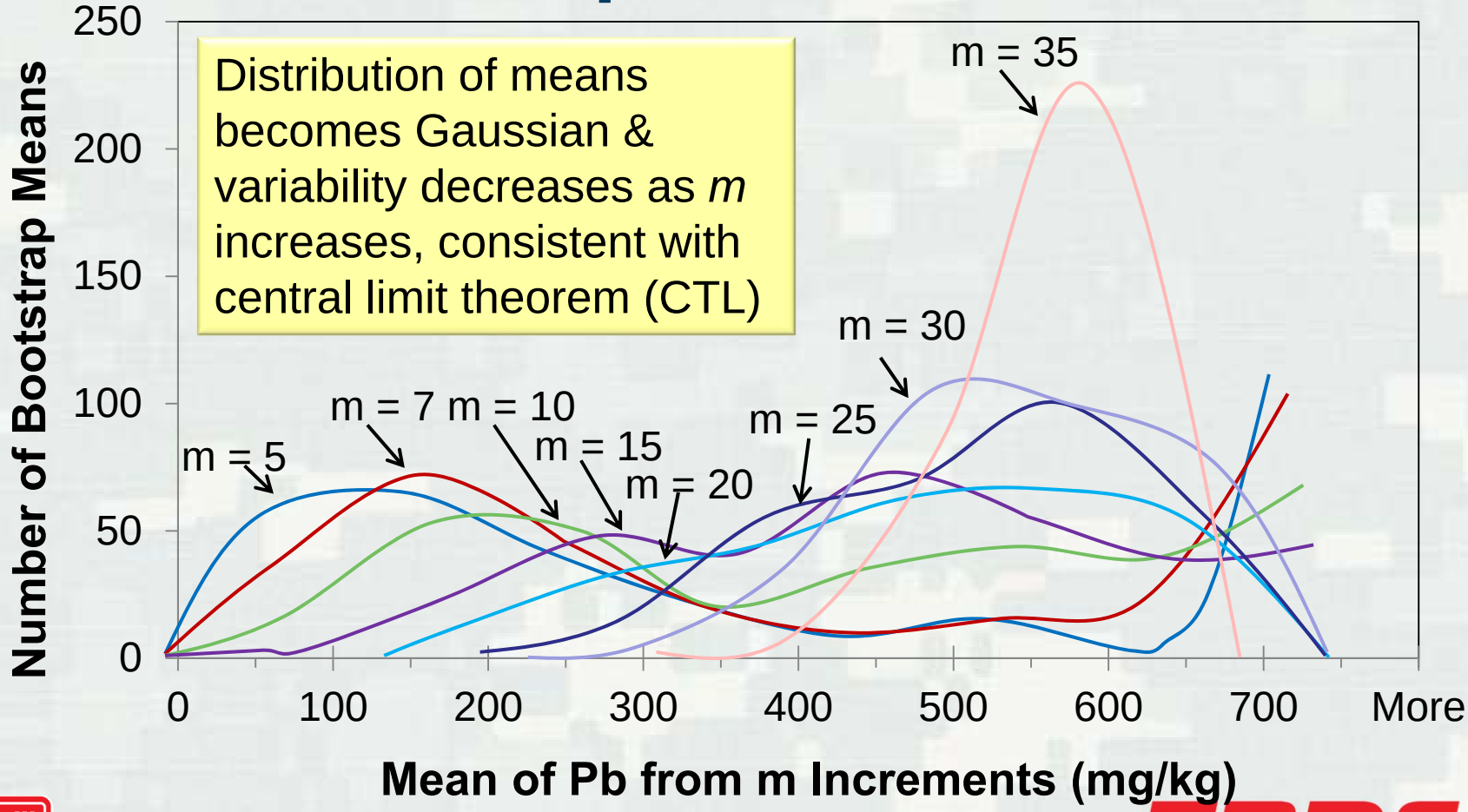
Number of Increments (*m*) for Small Arms Metals (Sb, Pb, Cu, & Zn)



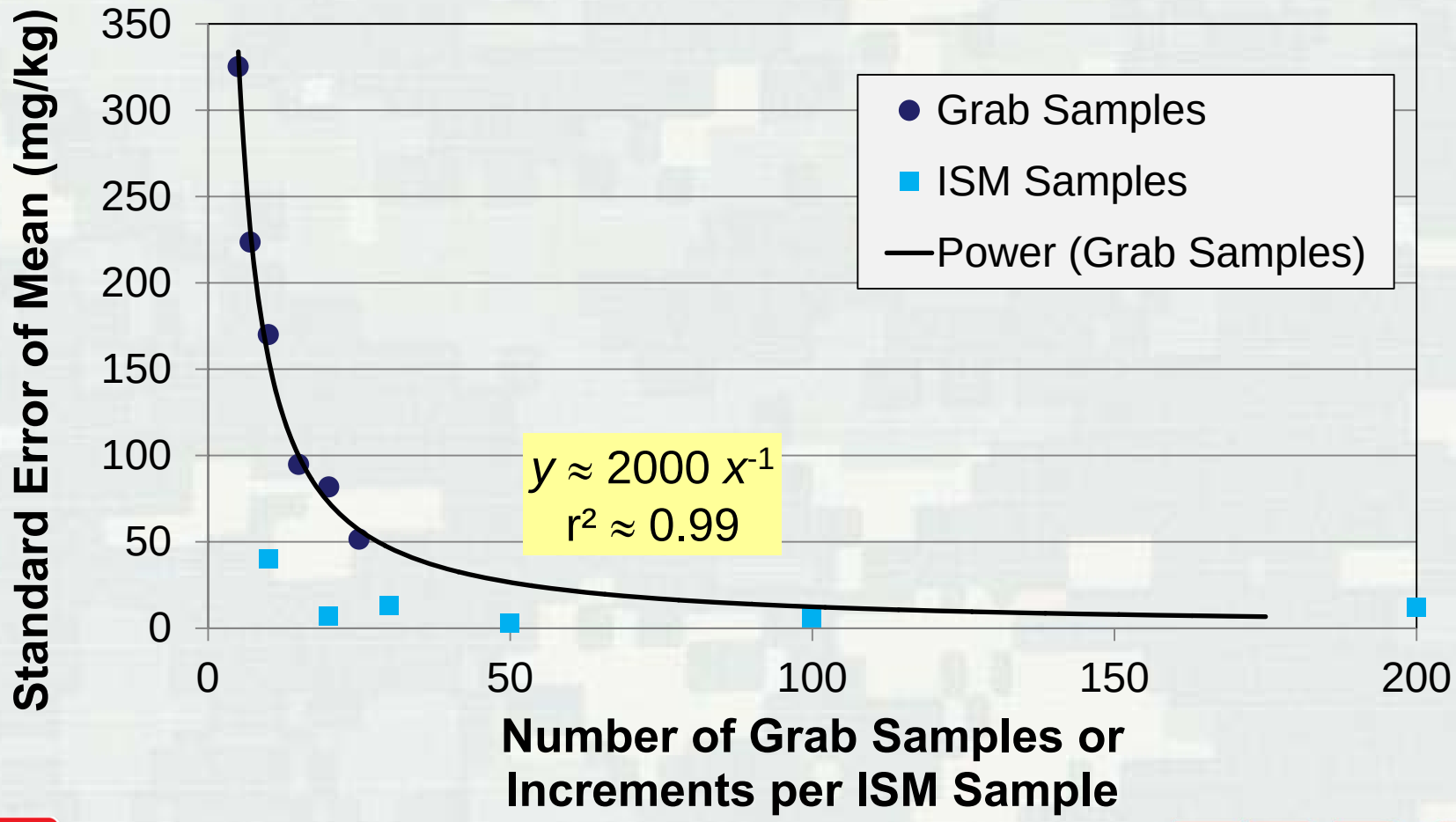
Bootstrap Simulation: Number of Increments per ISM Sample



Distributions of Simulated Bootstrap Lead Means



Bootstrap Standard Error of Mean Simulation



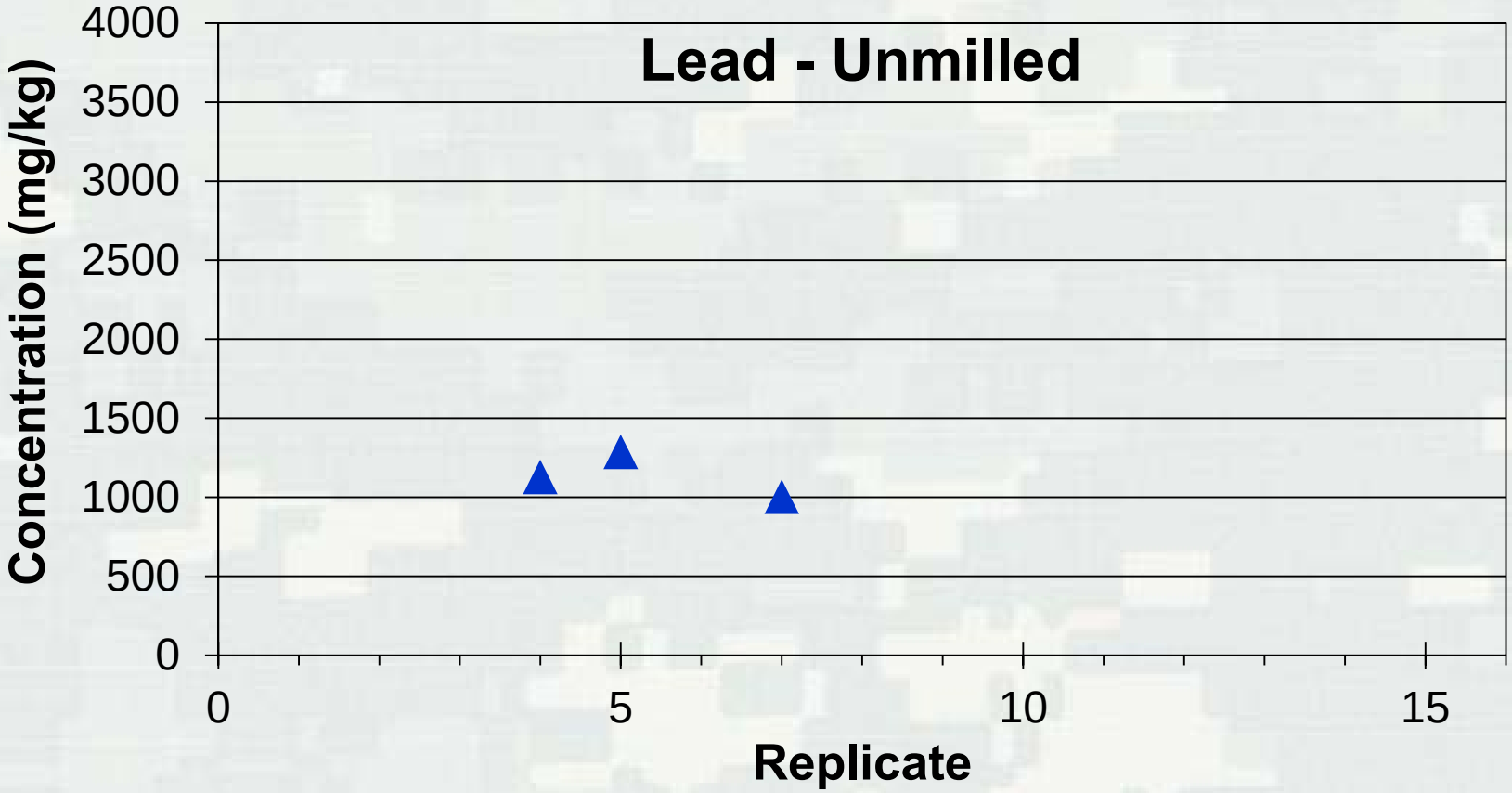
To mill or not mill: Whether ‘tis nobler in the mind to not mill and suffer the slings and arrows of outrageous fortune or to mill to obtain reproducible results…

Milled and Unmilled Comparison

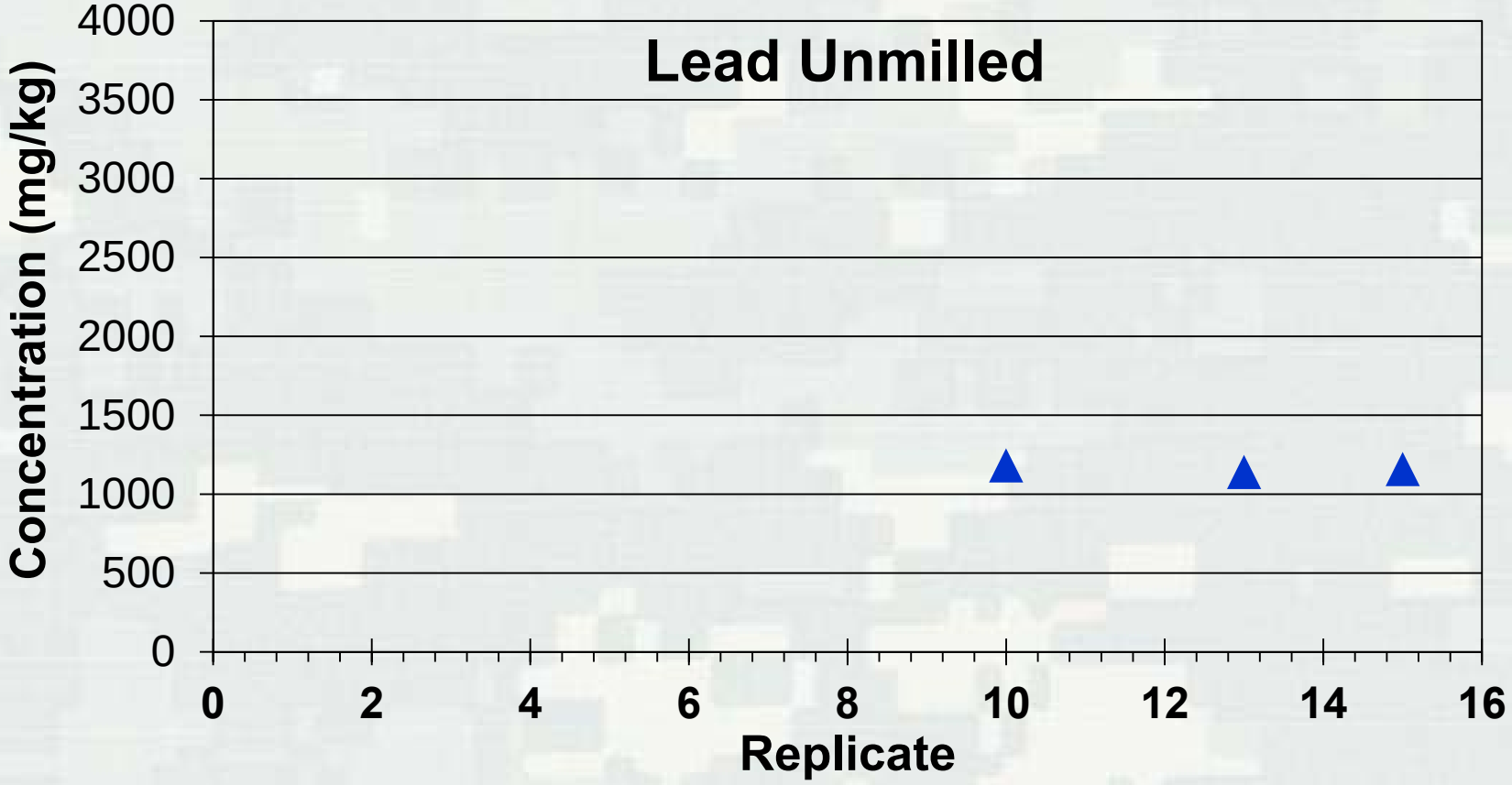
Subsample Replicates	Metals Conc. (mg/kg) Cu	Pb	Metals Conc. (mg/kg) Sb	Cu
1	**2,600**	360	5.5	99
2	110	330	5.0	90
3	300	920	7.6	87
4	**110**	300	4.3	99
5	130	280	4.3	**130**
6	140	**2,800**	**16**	90
7	860	1,600	12	88
8	540	330	4.6	99
9	1,200	850	4.2	83
10	130	1,500	4.5	98
11	1,900	380	4.9	99
12	120	330	4.3	110
13	130	**290**	**3.7**	**80**
14	120	300	4.1	87
15	110	820	8.2	84

Sample #1 **Sample #2**

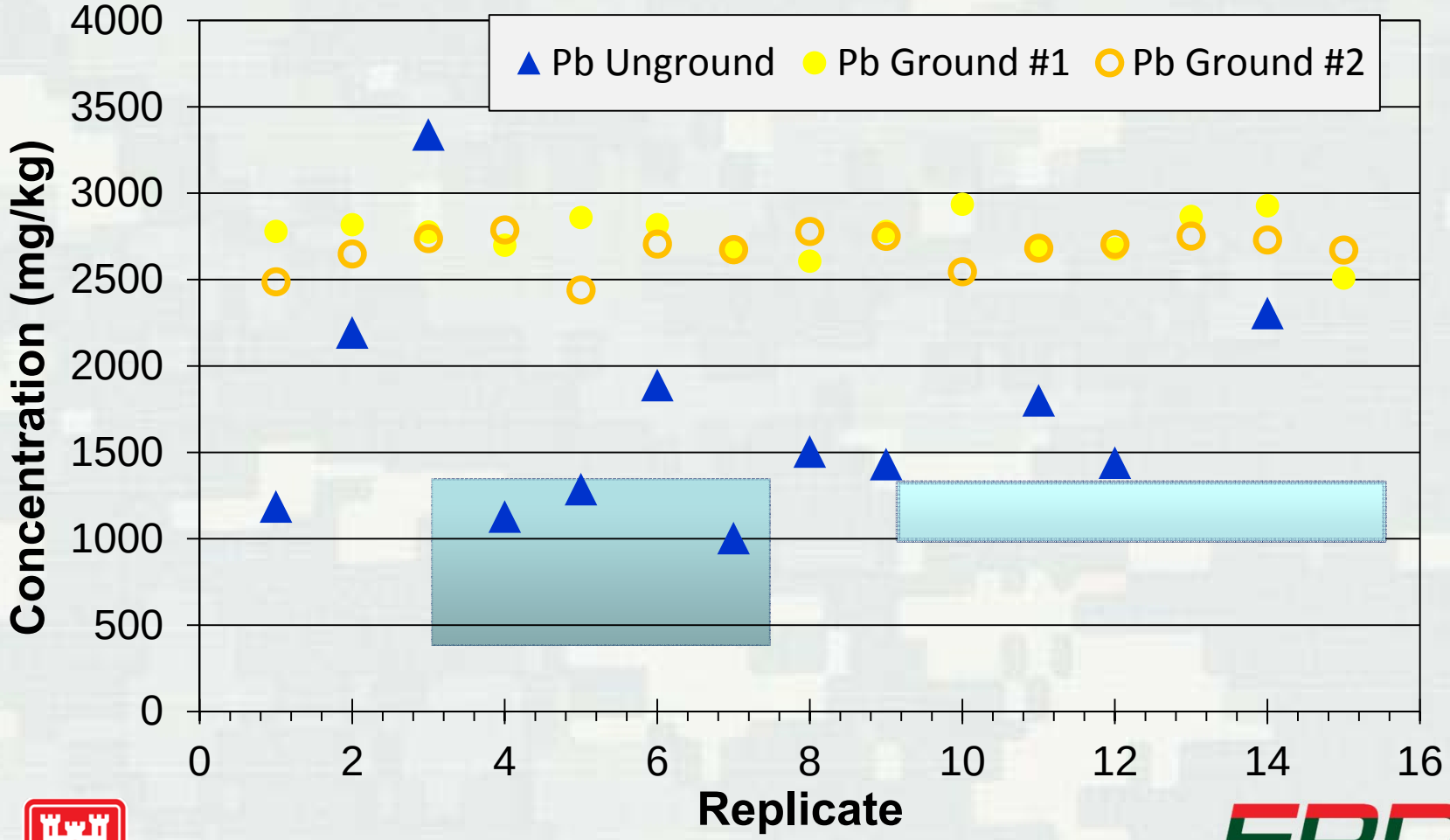
To Mill or Not To Mill (1)



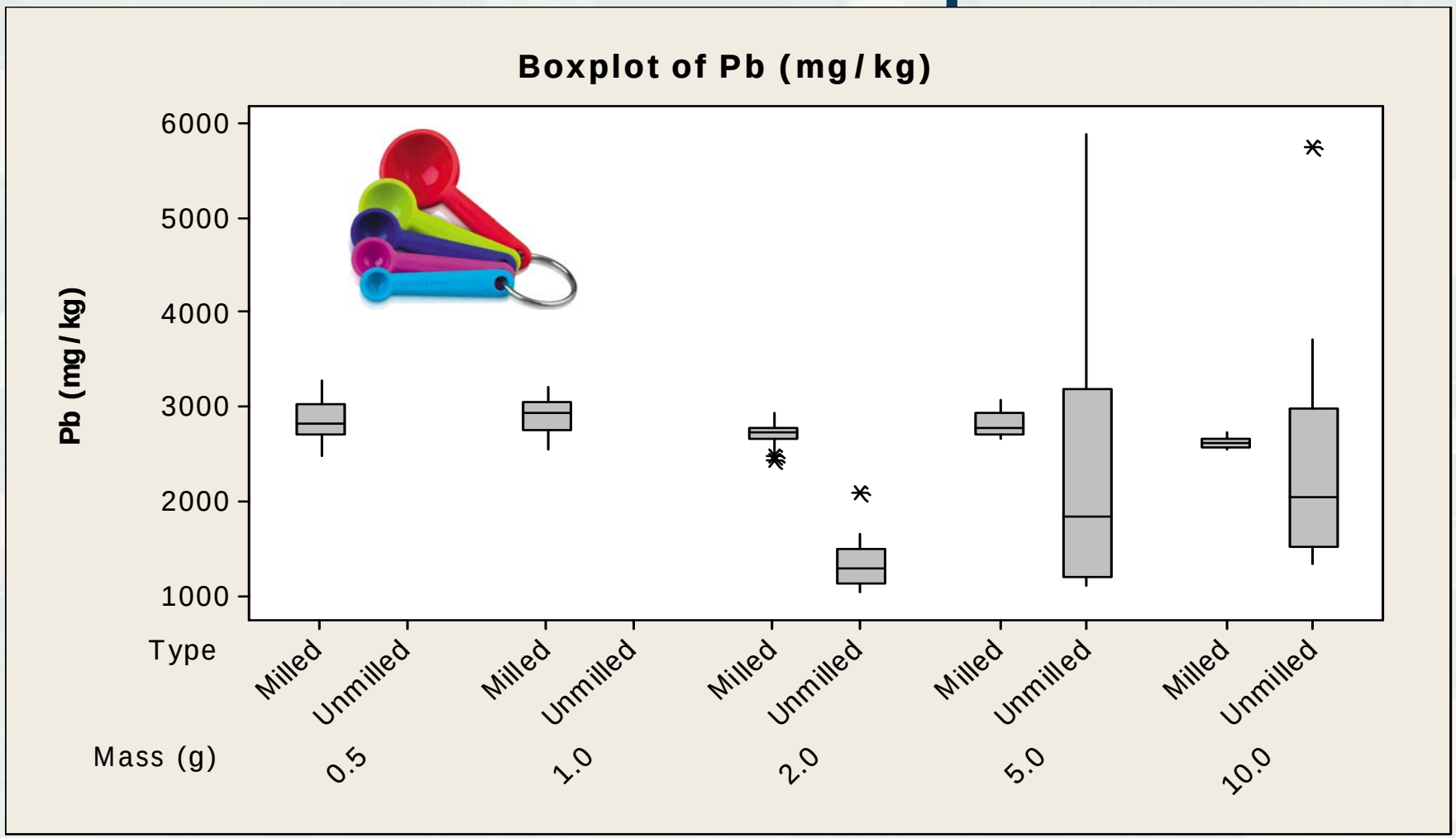
To Mill or Not To Mill (2)



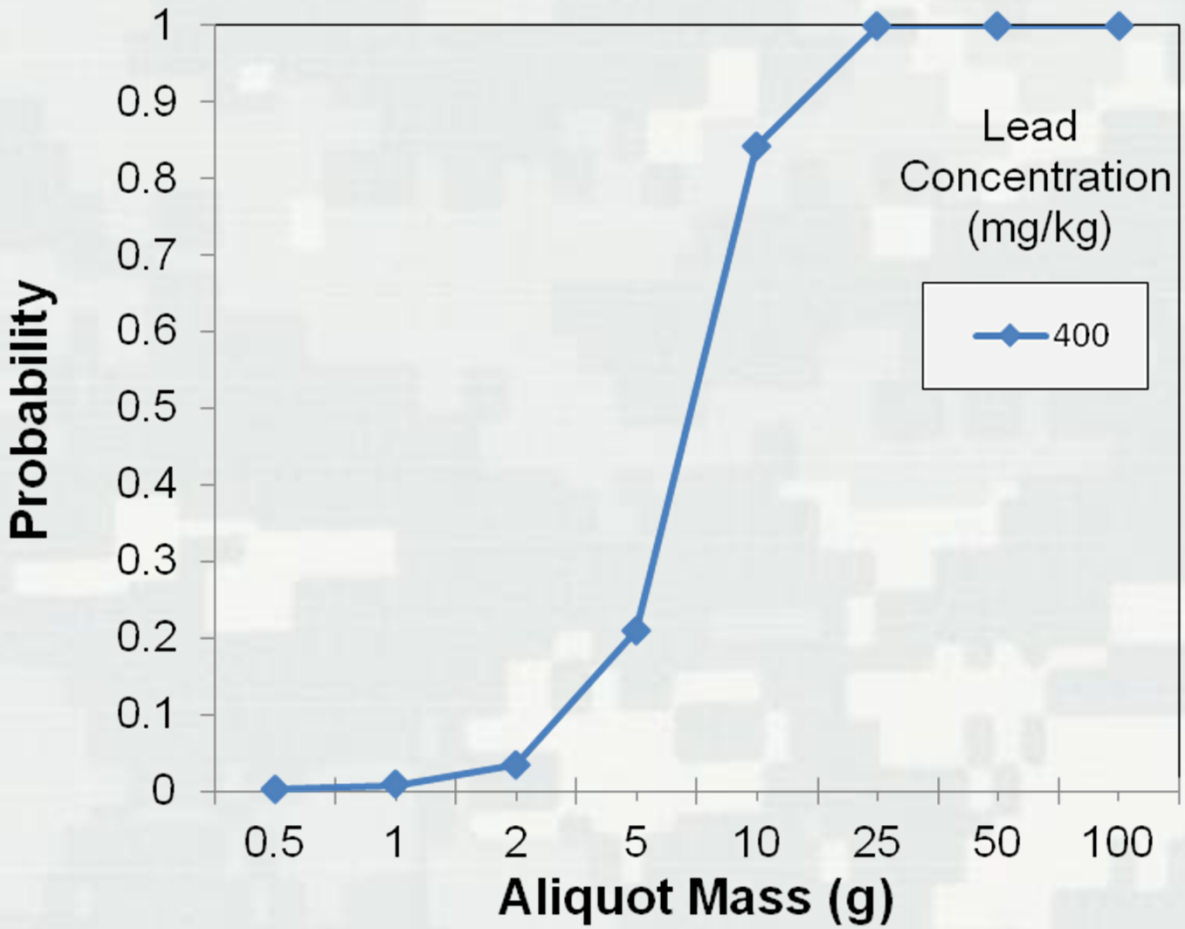
To Mill or Not To Mill (3)



Digestion Mass for Milled & Unmilled Samples



Probability of One Lead Particle by Aliquot Mass for Unmilled Soil



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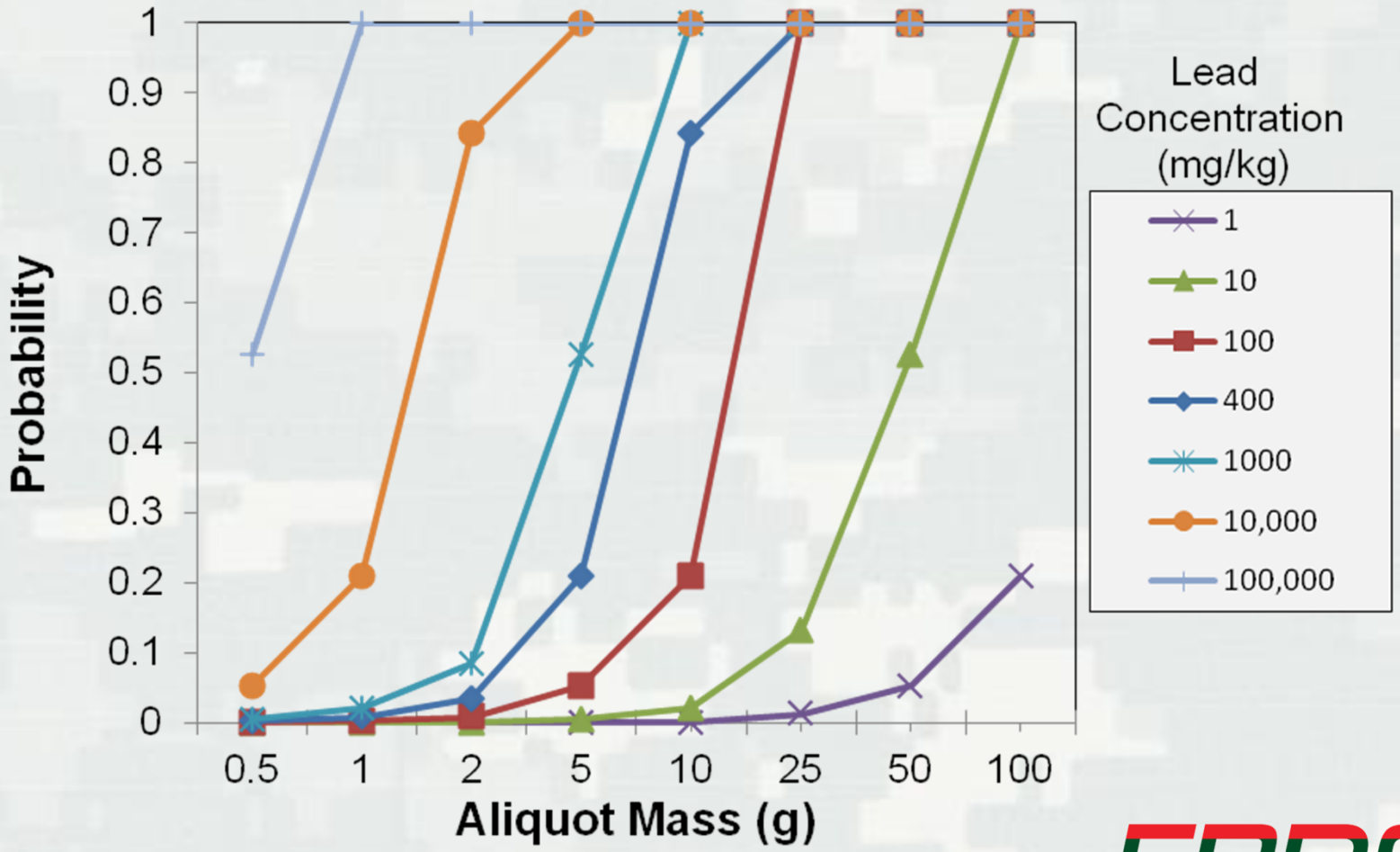
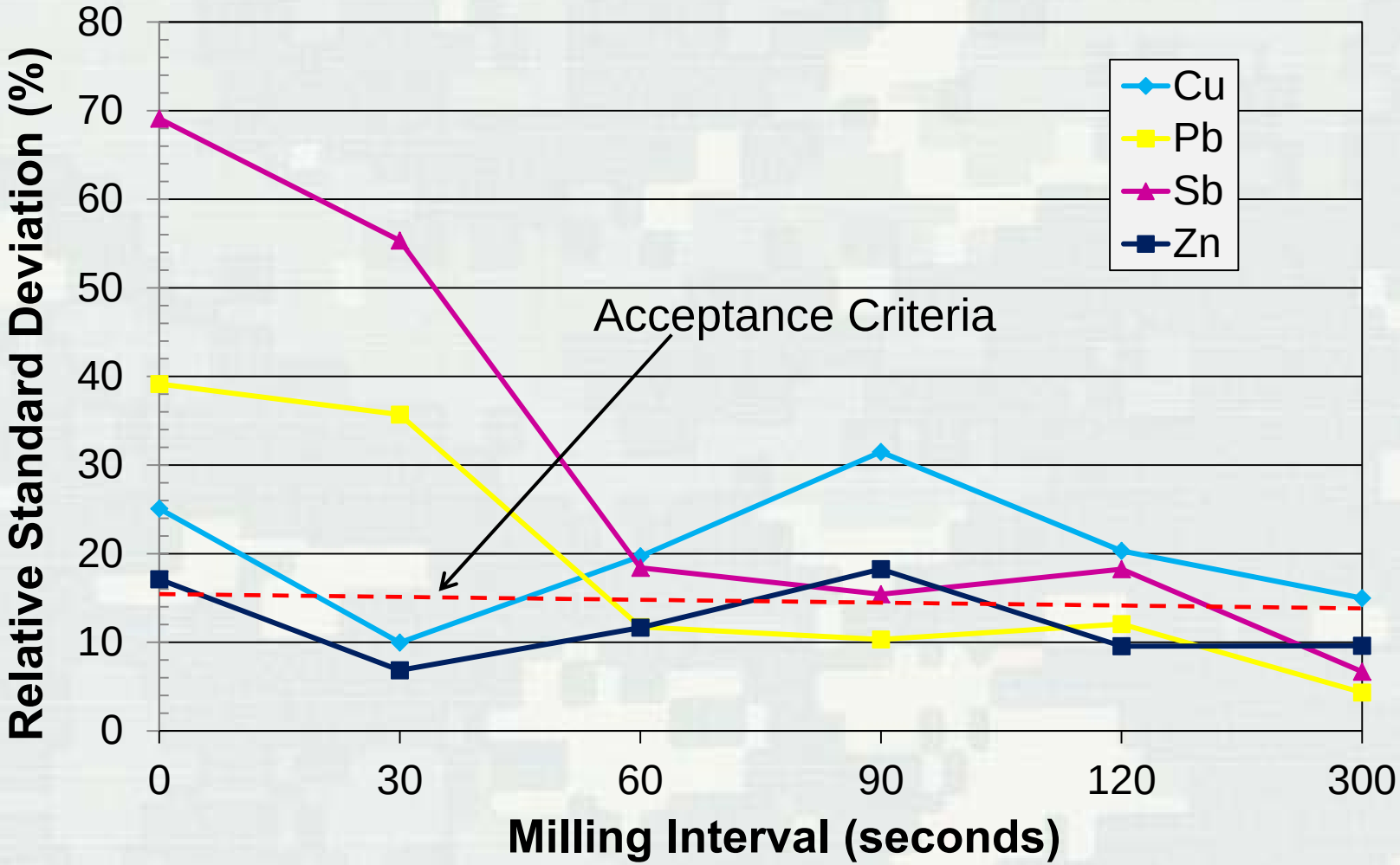
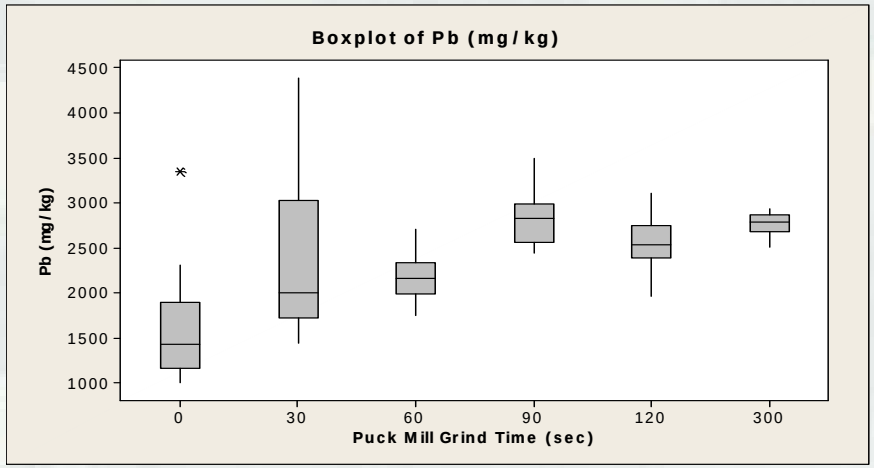
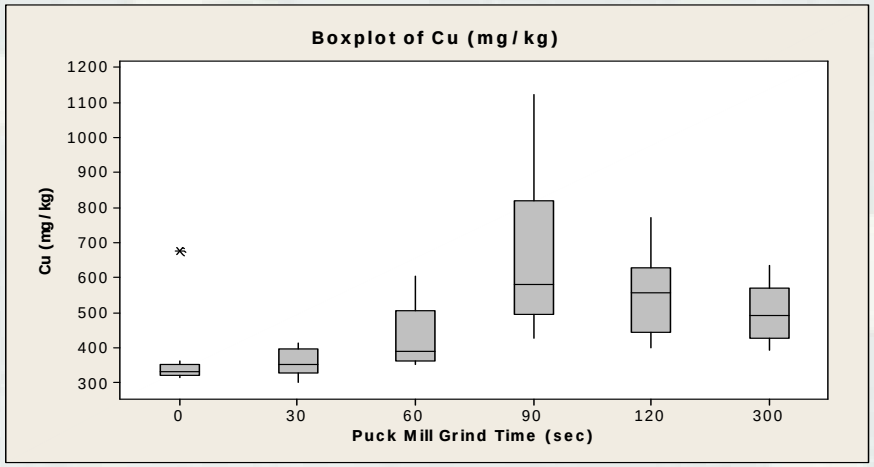


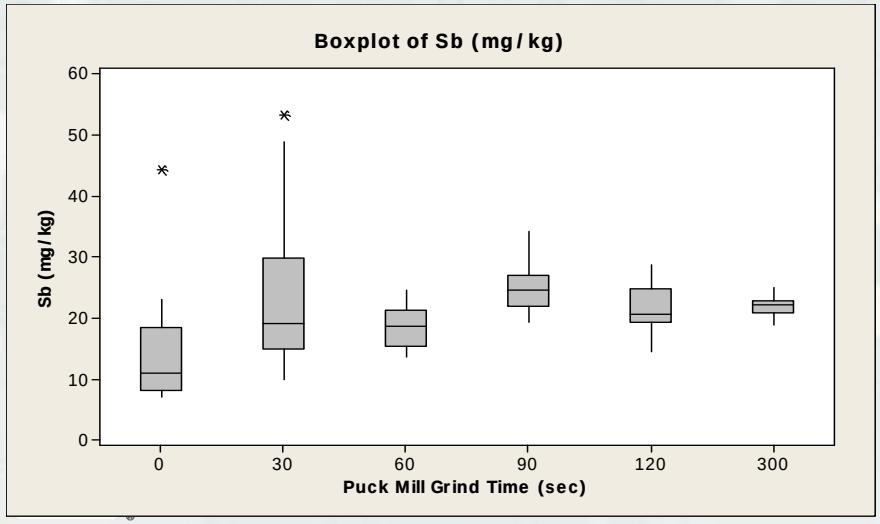
Puck Mill Precision

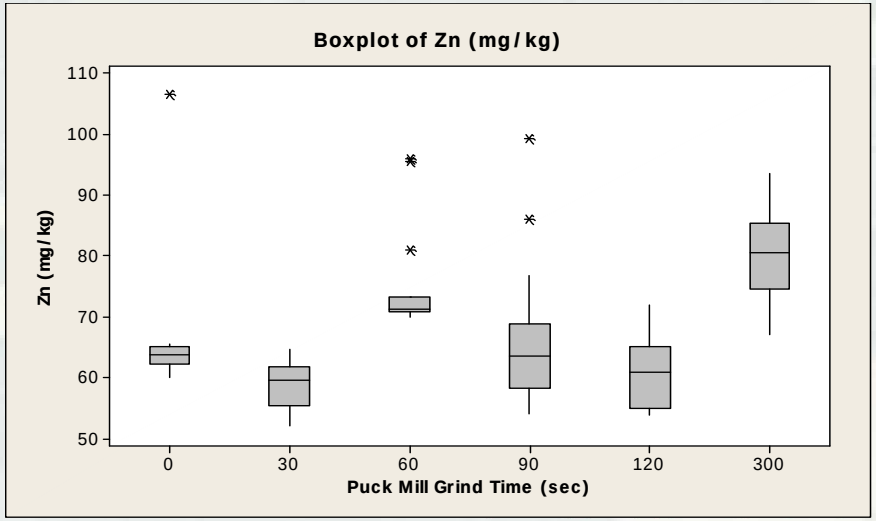


SAR Metal Concentration by Puck Mill Milling Time

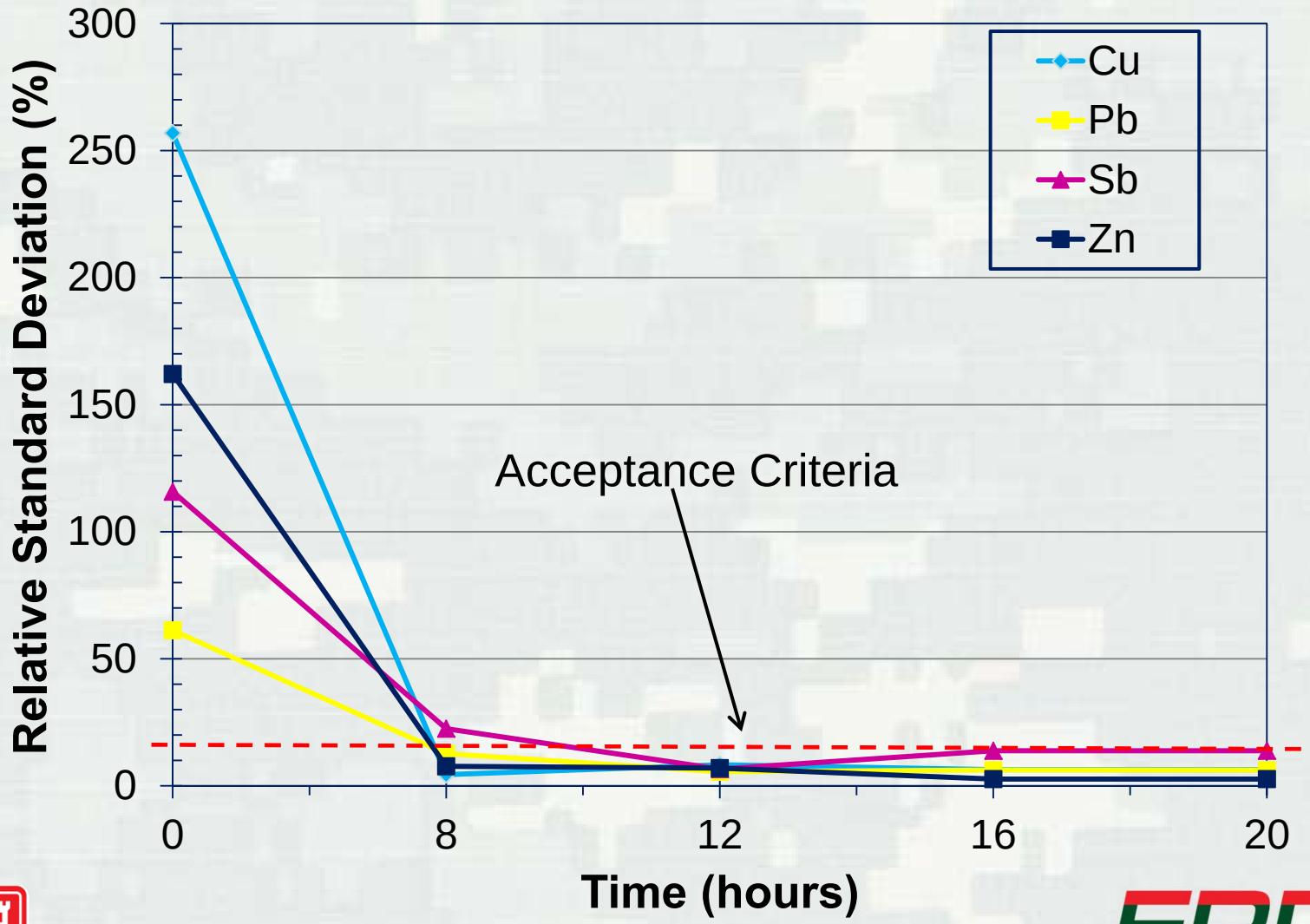




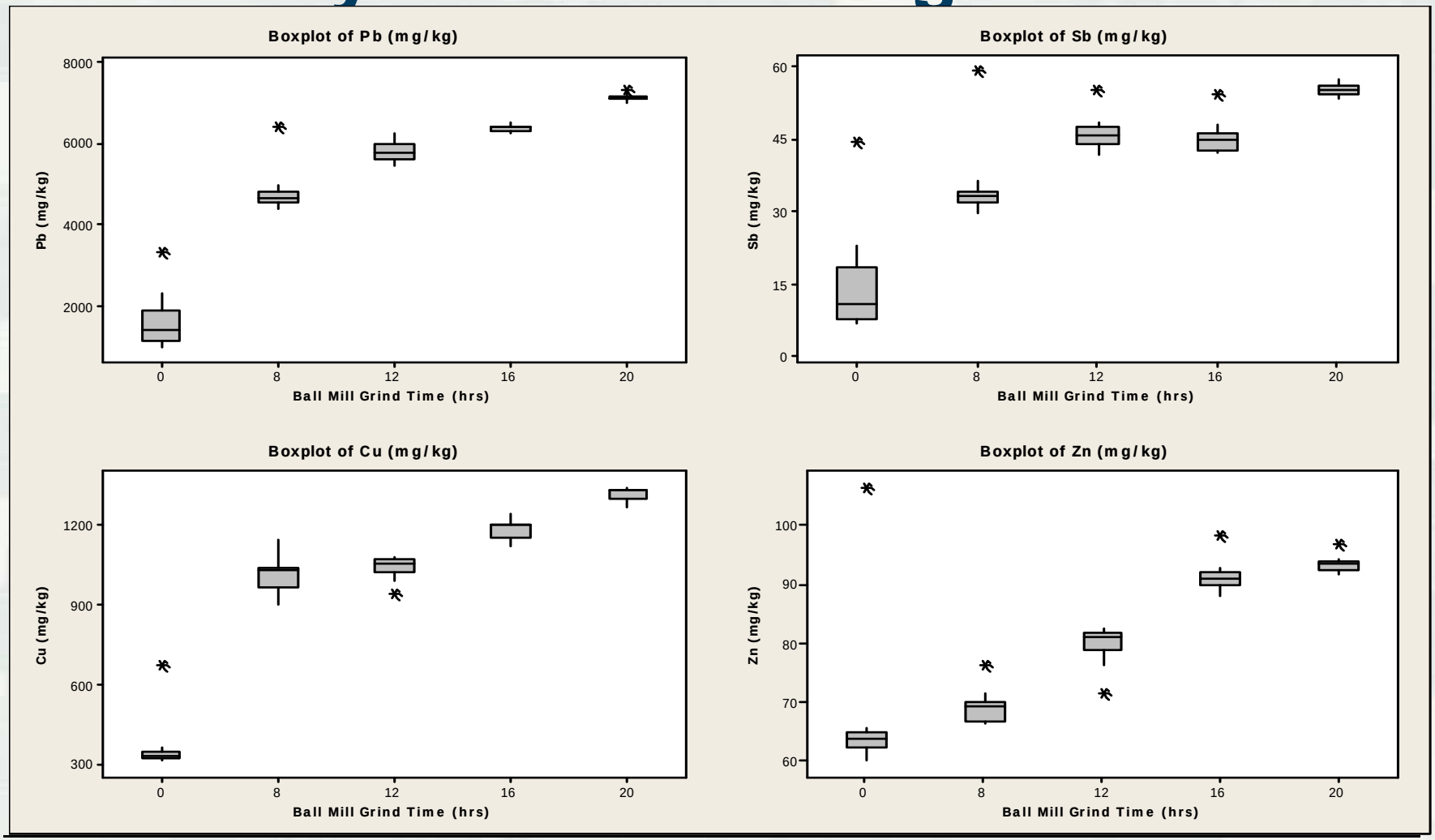




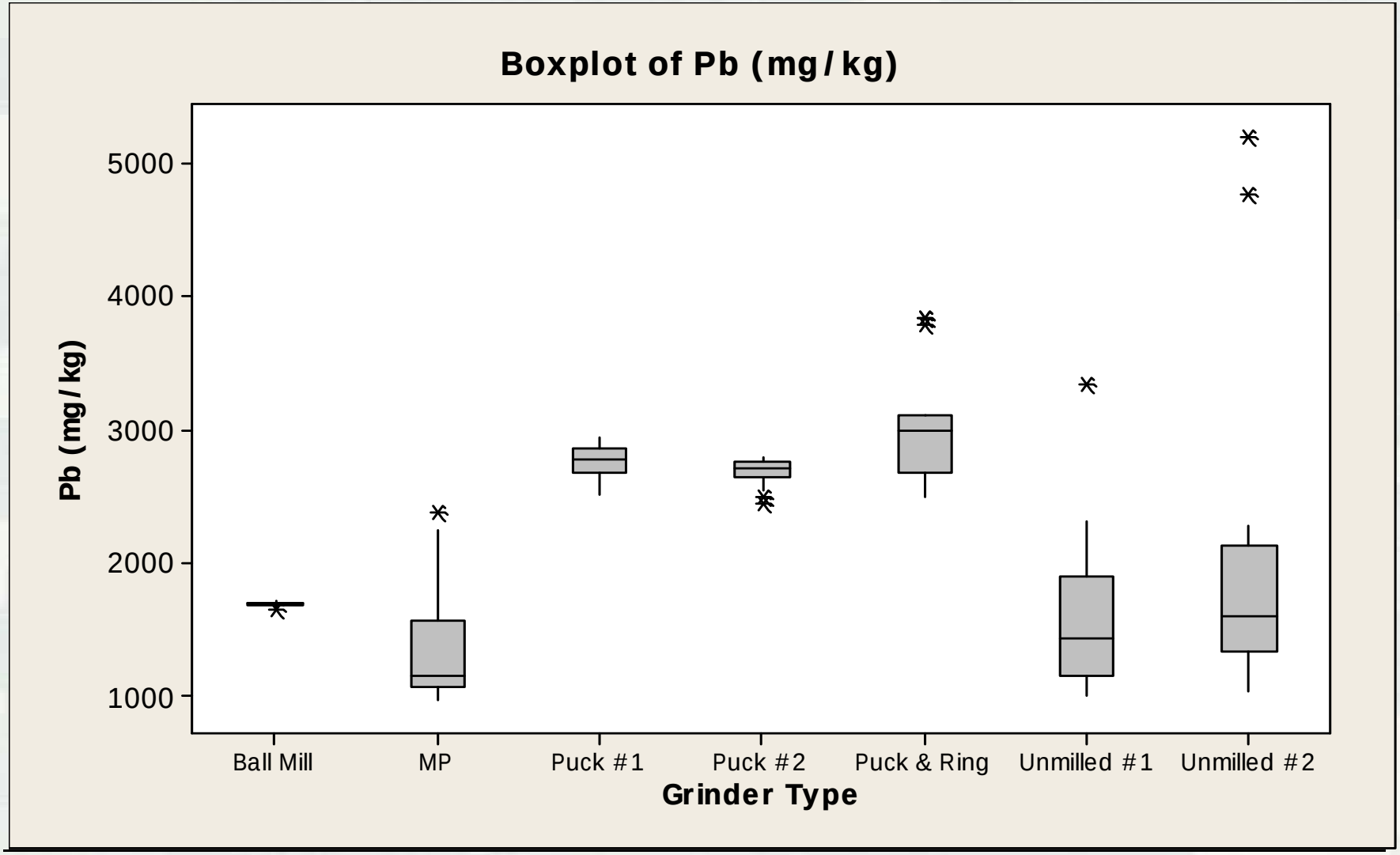
Ball Mill Precision



SAR Metal Concentration by Ball Mill Milling Time



Milling by Device Type



Performance Assessment

Activity	Yes	No
ISM, 30+ increments	√	
Grab Sampling		√
Field Splitting		√
Sieving	√	
Milling necessity	√	
Increased digestion mass	√	√
Increased digestion time		√
Subsampling	√	

No ISM-Lite

Cost Comparison

	Grab	ISM
Systematic Project Planning	$	$
Site Preparation	$$	$
Field Sampling	$$	$$$
Laboratory Sample Processing	$	$$$
Analysis	$$	$
Data Validation	$$	$
Per Sample Cost	**$-$$**	**$$-$$$**
Number of Samples	###	#
Total Project Cost	**$$-$$$**	**$-$$**

Conclusions

- Field splitting not recommended when metal particles expected
- Minimum of 30-increments per ISM sample
- Number of increments is a function of expected degree of heterogeneity
- Larger digestion aliquot in lieu of milling does not work
- Milling is necessary when metal particles are present
- Marginal improvements in data quality with larger digestion aliquots for milled samples
- Recommend 5 minutes milling with Puck and 8 hours with Roller Mills

References

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- Clausen, J. and N. Korte. 2009. The distribution of metals in soils and pore water at 3 U.S. military training facilities. *Soil Sed Cont J.* **18**(5):546-563. http://www.tandfonline.com/doi/abs/10.1080/15320380903085683