

Update on ESTCP Project ER-0918: Field Sampling and Sample Processing for Metals on DoD Ranges

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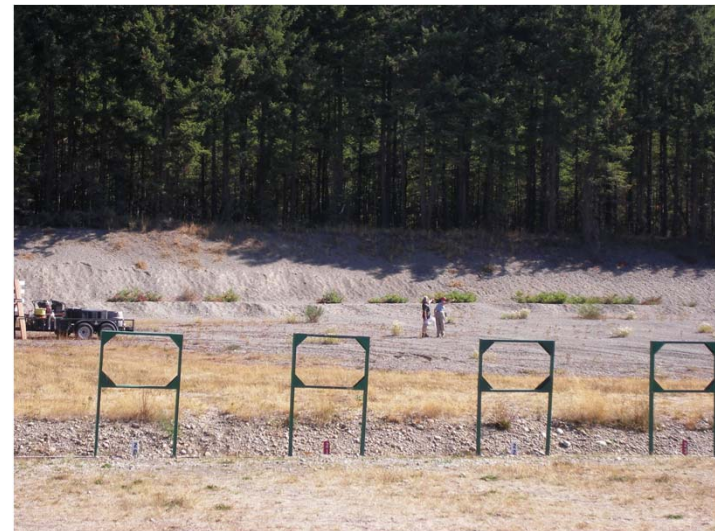
Project Team

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- Anthony Bednar: ERDC-EL
- Thomas Georgian: HNC@EMCX
- Larry Penfold: Test America
- Diane Anderson: APPL Laboratories



Technical Objectives

- Demonstrate improved data quality for metal constituents in surface soils on military training ranges by coupling multi-increment sampling with modifications to sample preparation and analysis methods such as:
 - Sample processing involving grinding
 - Sub-sampling to build the digestate aliquot
 - Digestion Issues (mass, acid ratio, time)
 - Laboratory processing protocol applicable to both metals and energetics



Experimental Design –Task 1

- Multi-increment versus grab samples
- Number of increments per decision unit
- Grinding necessity
- Digestion mass evaluation
- Digestion time
- Blank material identification and assessment
- Puck Mill metal carry over assessment (cross contamination)
- Grinder comparisons
- Puck Mill and Roller Mill optimum grinding interval
- Appropriateness of field splitting
- Subsampling for digestate preparation



Task 1 Activities
Task 4 Activities



Soil Test Material



- Site: Camp Ethan Allen, VT
- Range Type: Small Arms (Pistol, Rifle)
- Decision Unit: Berm Face – 3 by 30 m
- Soil Type: Silty sand, low CEC, low OM, pH~ 5
- Metal Content: 100's to low 1,000's ppm
- Samples Collected
 - Grab/discrete using grid-node approach – 30
 - Multi-increment using systematic random, 7 replicates of 5, 10, 20, 30, 50, 100 increments
 - One 200 increment sample ~ 25 kg

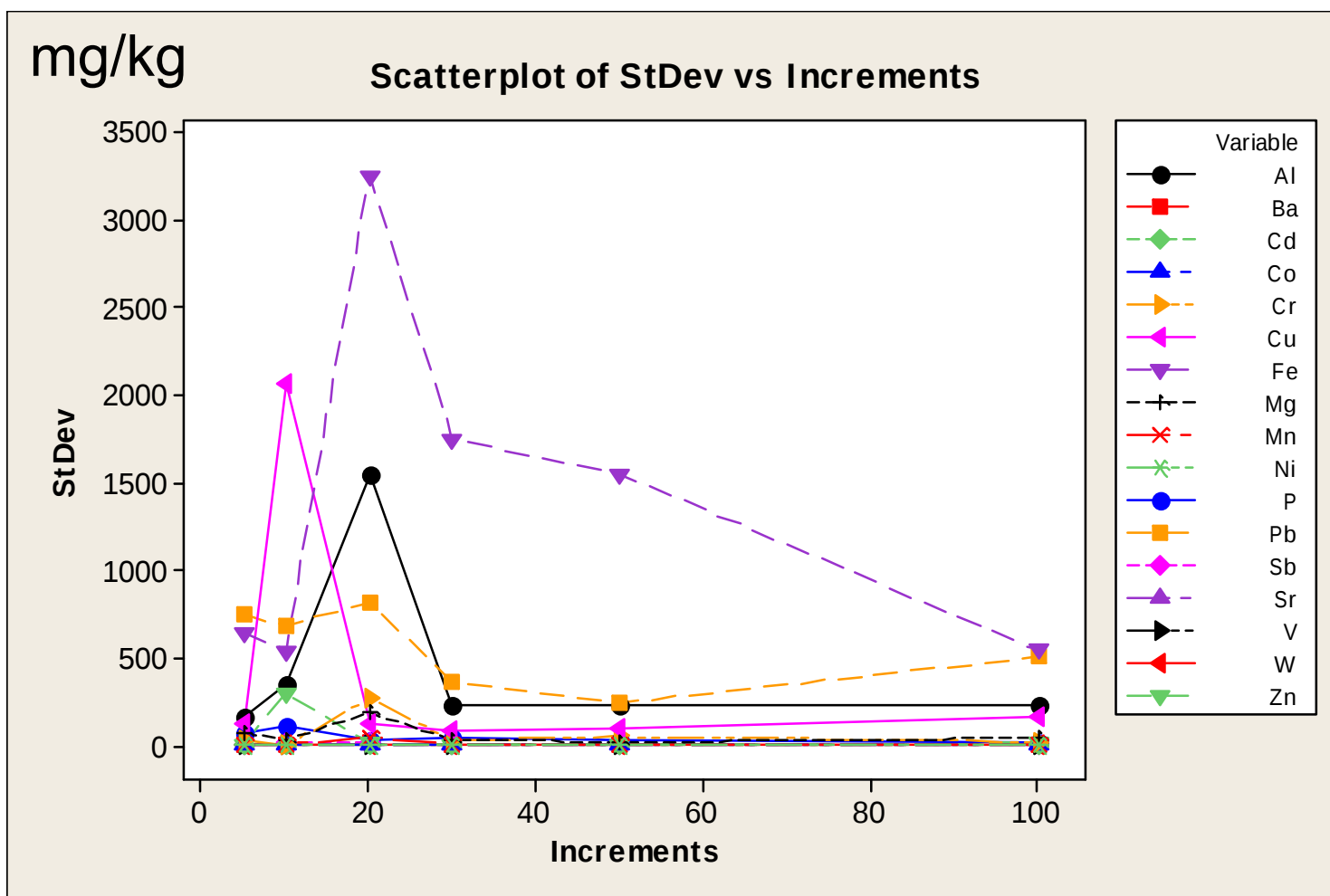
Soil Test Material



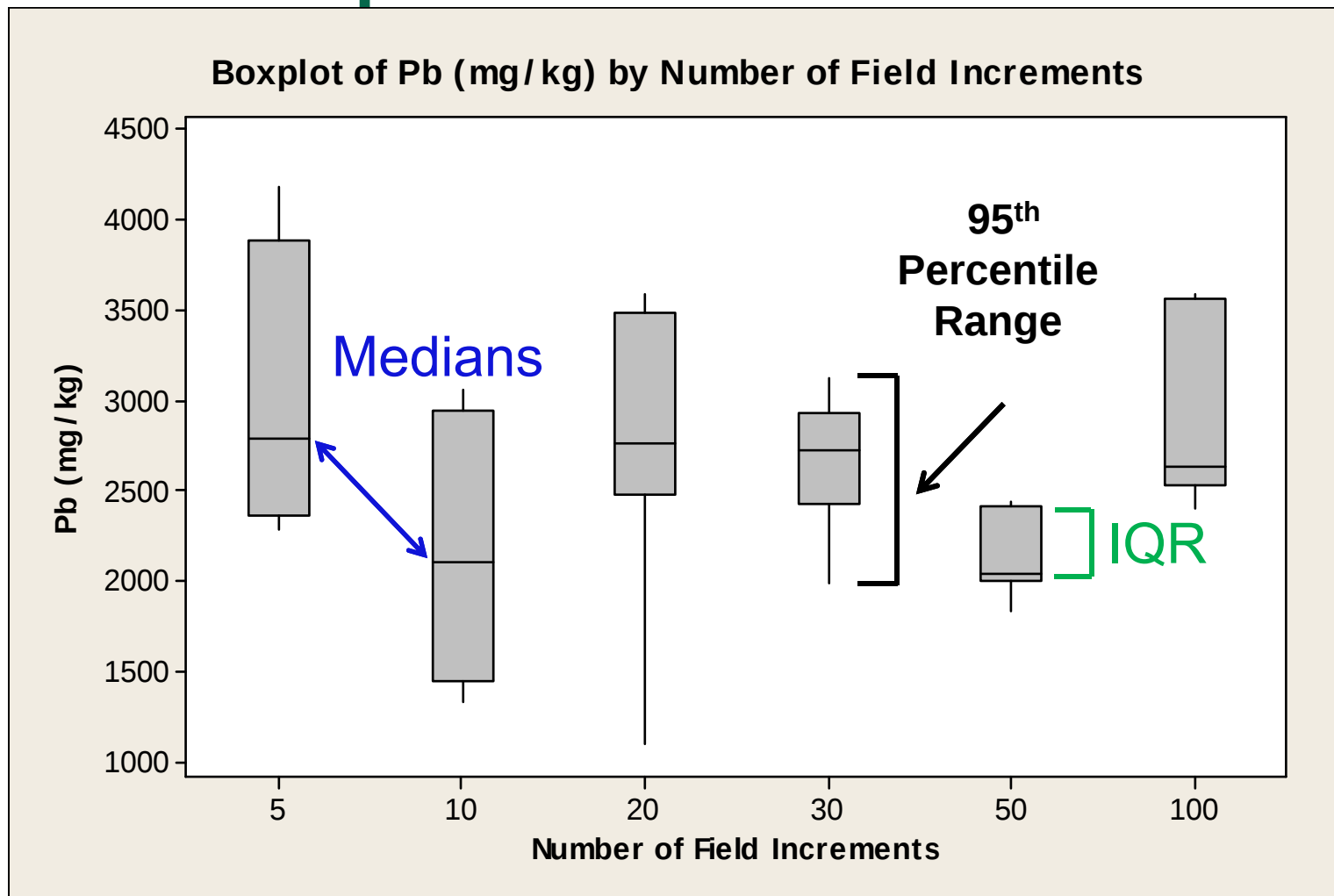
Multi-Increment vs Grab Samples

		Sb mg/kg	Cu mg/kg	Pb mg/kg	Zn mg/kg
Grab	Mean	88	300	5060	66
(n=30)	Std. Dev.	375	132	14,437	17.5
	RSD (%)	426	44	285	27
MI-30	Mean	23	573	2664	67
(n=7)	Std. Dev.	3.3	85	367	4.0
	RSD (%)	14	15	14	6
MI-50	Mean	17.6	457	2156	67
(n=7)	Std. Dev.	1.8	96	243	6.5
	RSD (%)	10	21	11	10

Number of Increments per Decision Unit



Number of Increments per Decision Unit



Grinding Necessity



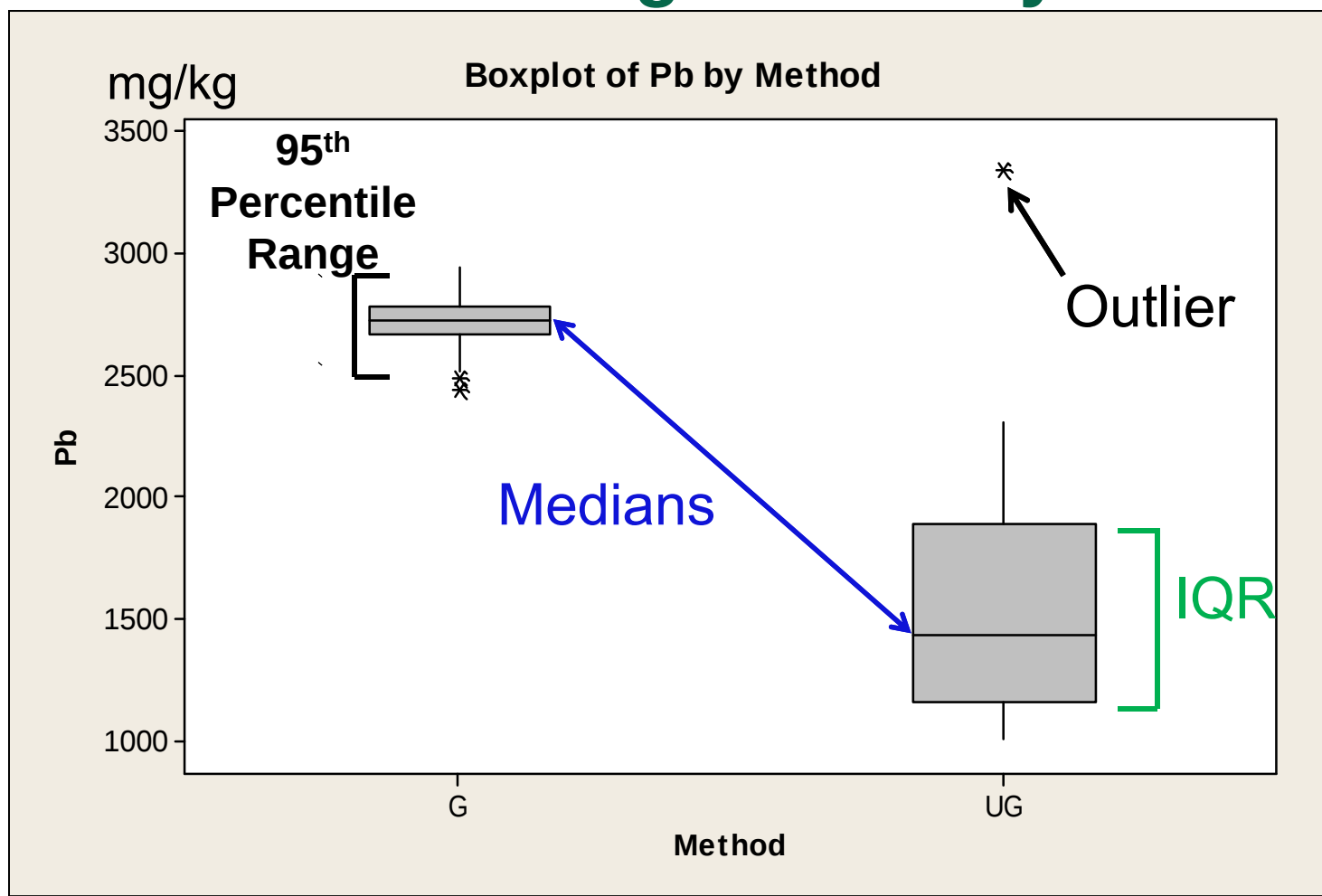
		Sb mg/kg	Cu mg/kg	Pb mg/kg	Zn mg/kg
Un-Ground	Mean	14	360	1600	66
(n=15)	Std. Dev.	10	90	630	11.3
	RSD (%)	71	25	39	17
Ground	Mean	23	550	2720	77
(n=15)	Std. Dev.	1.6	100	120	8.7
	RSD (%)	7.0	18	4.4	11

Performance criteria RSD < 15% for lab replicates (for concentrations > 100

Soil Post Grinding



Grinding Necessity



Performance Assessment – Sample Processing (Grinding) of Soil

Puck Mill

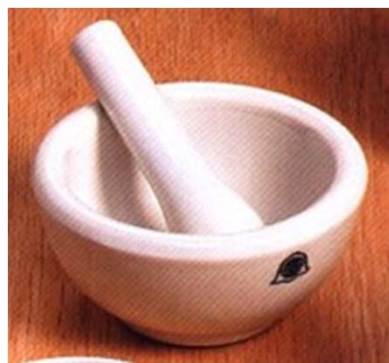


Fe, Mn, Cr, V

Roller Mill



Alumina cans polyethylene
Liner, ceramic balls



Mortar and Pestle
Ceramic

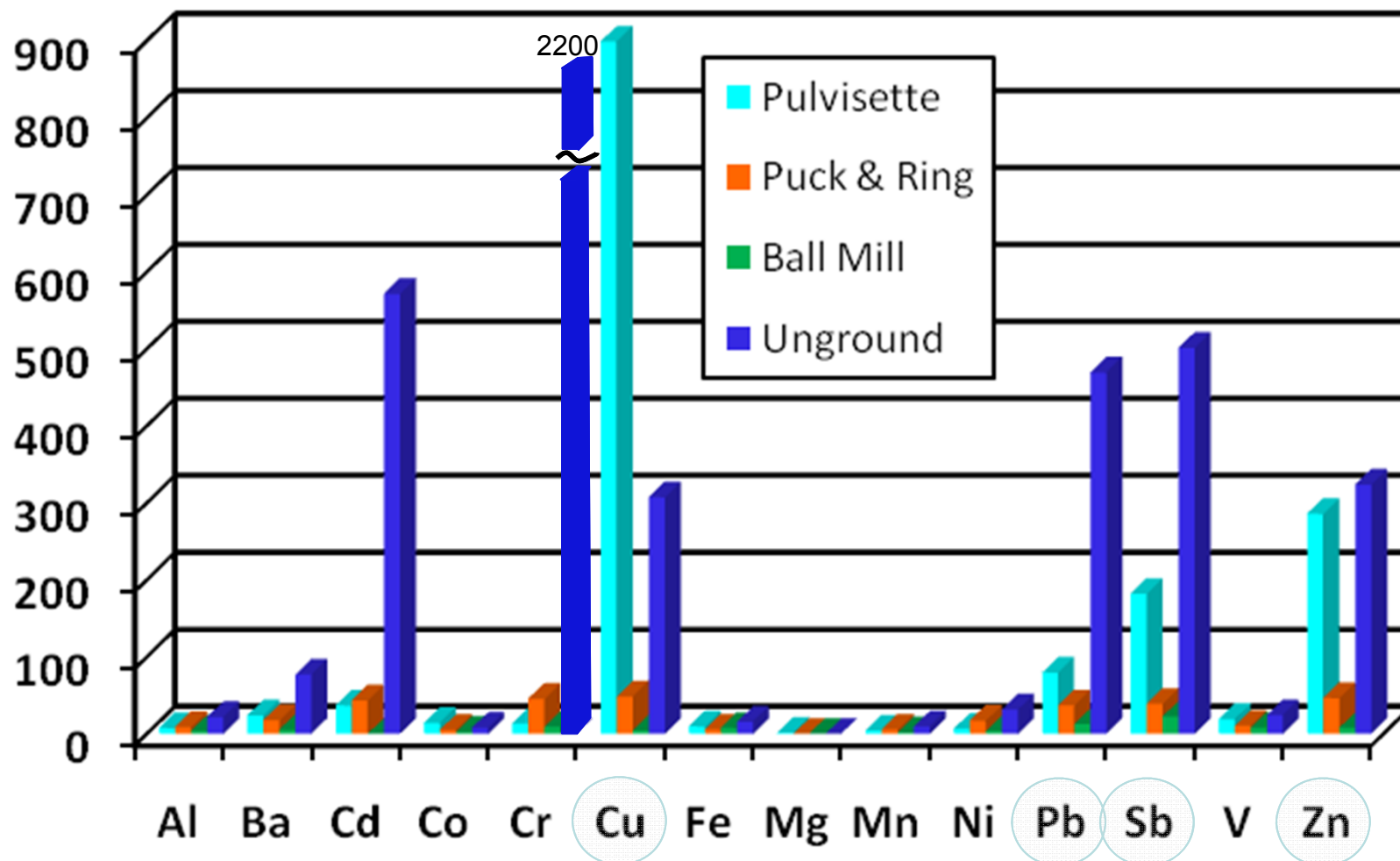
Pulvisette



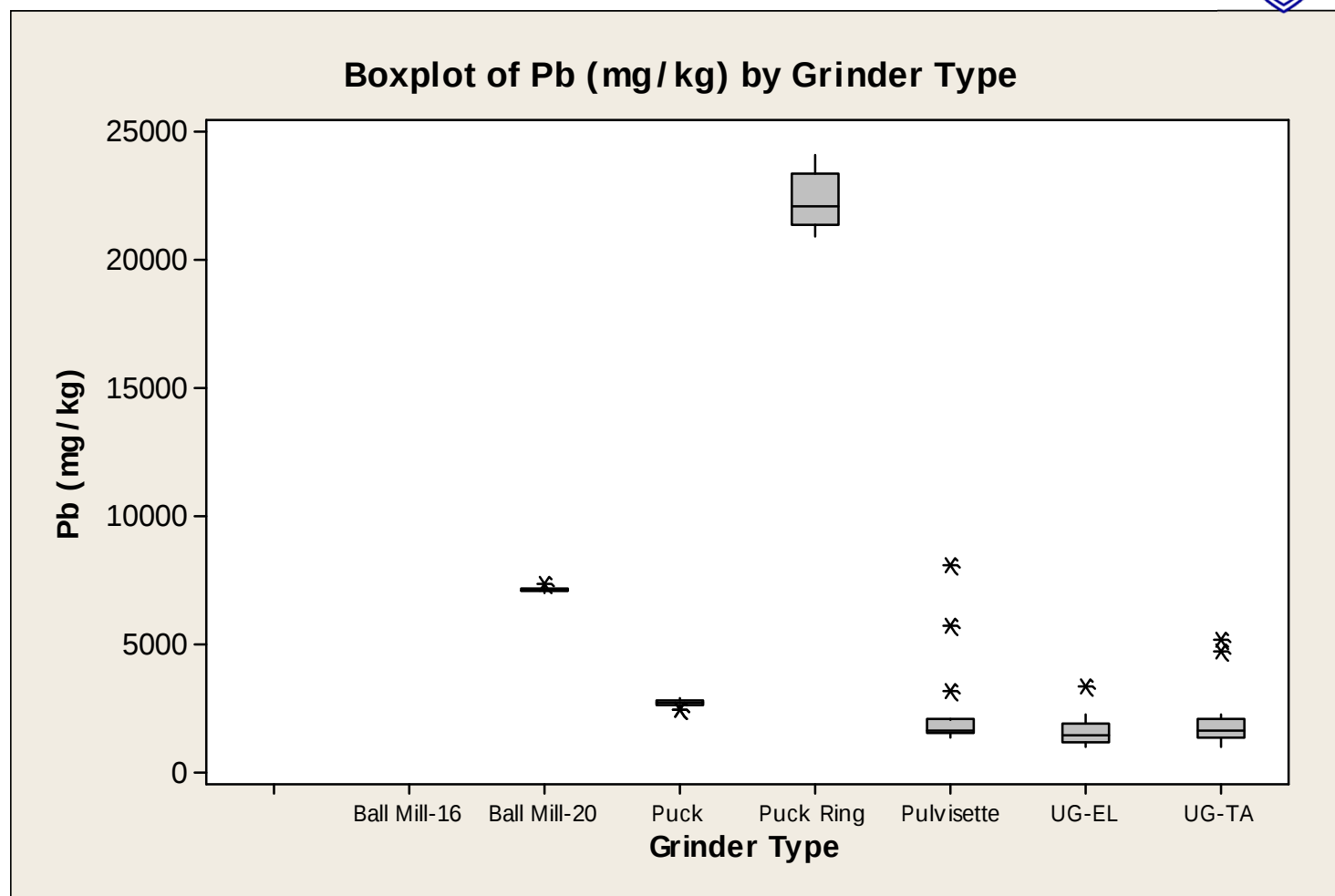
Agate balls

Grinder Comparisons

RSD (%)

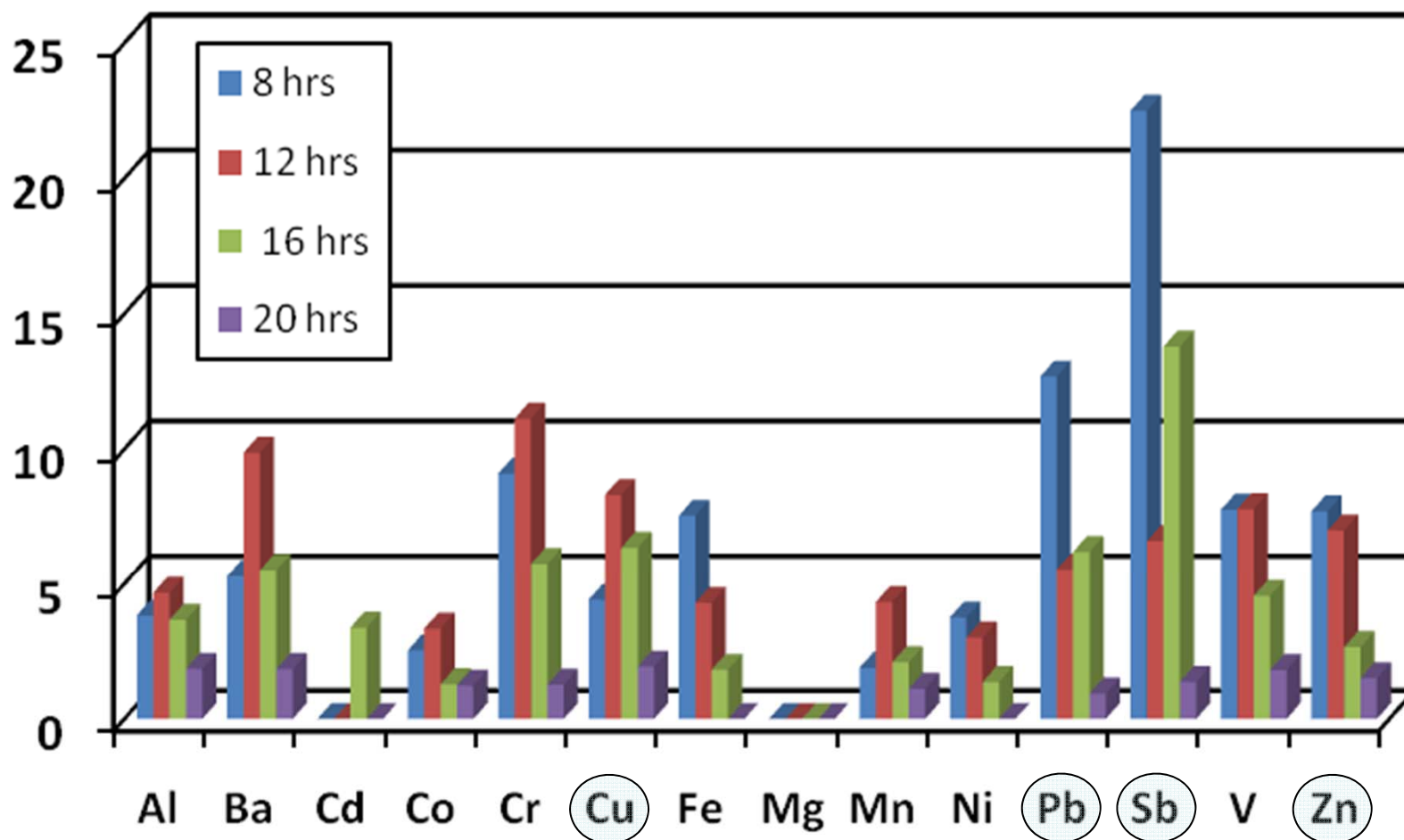


Grinder Comparisons

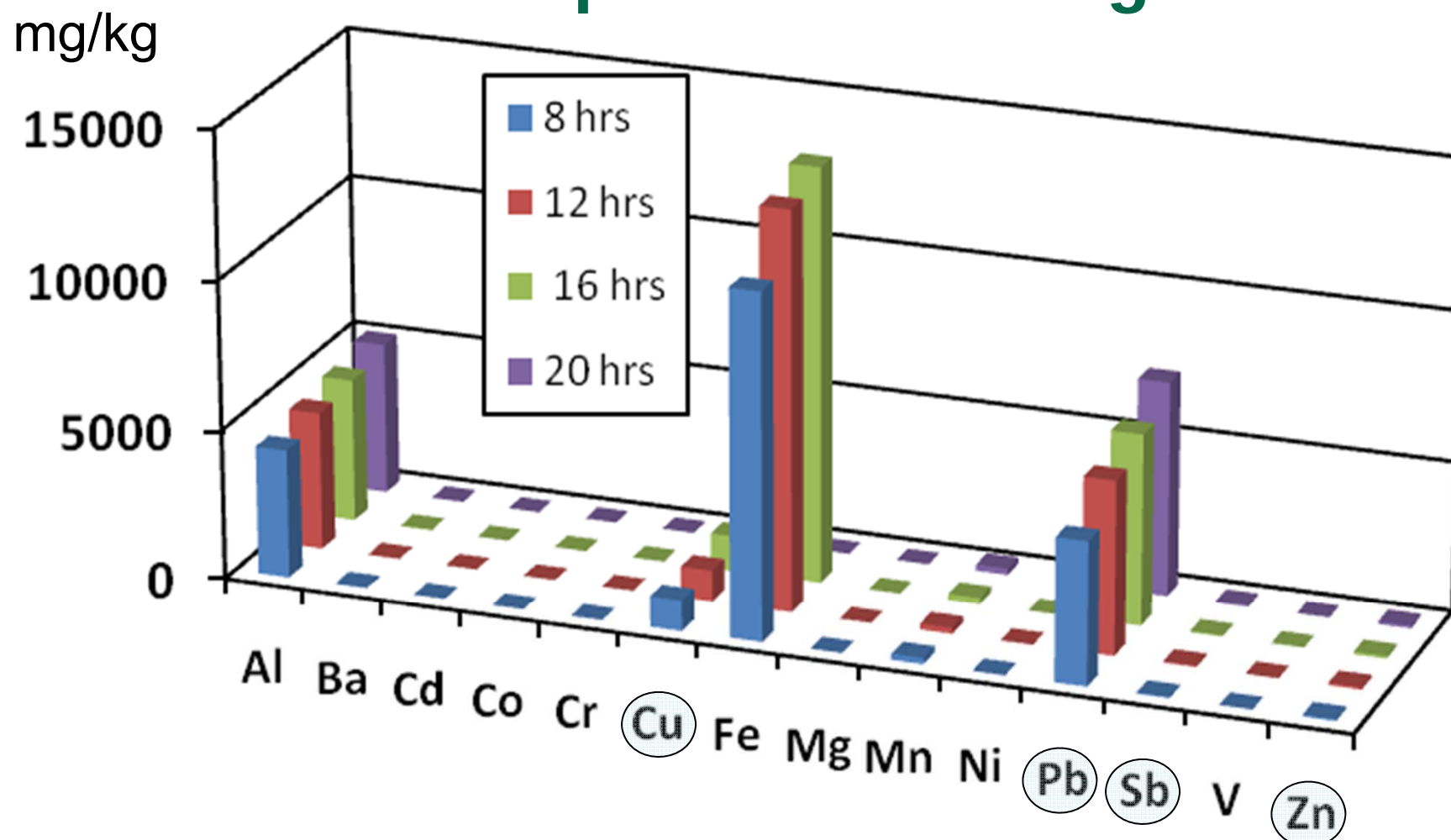


Roller Mill Optimum Grinding Interval

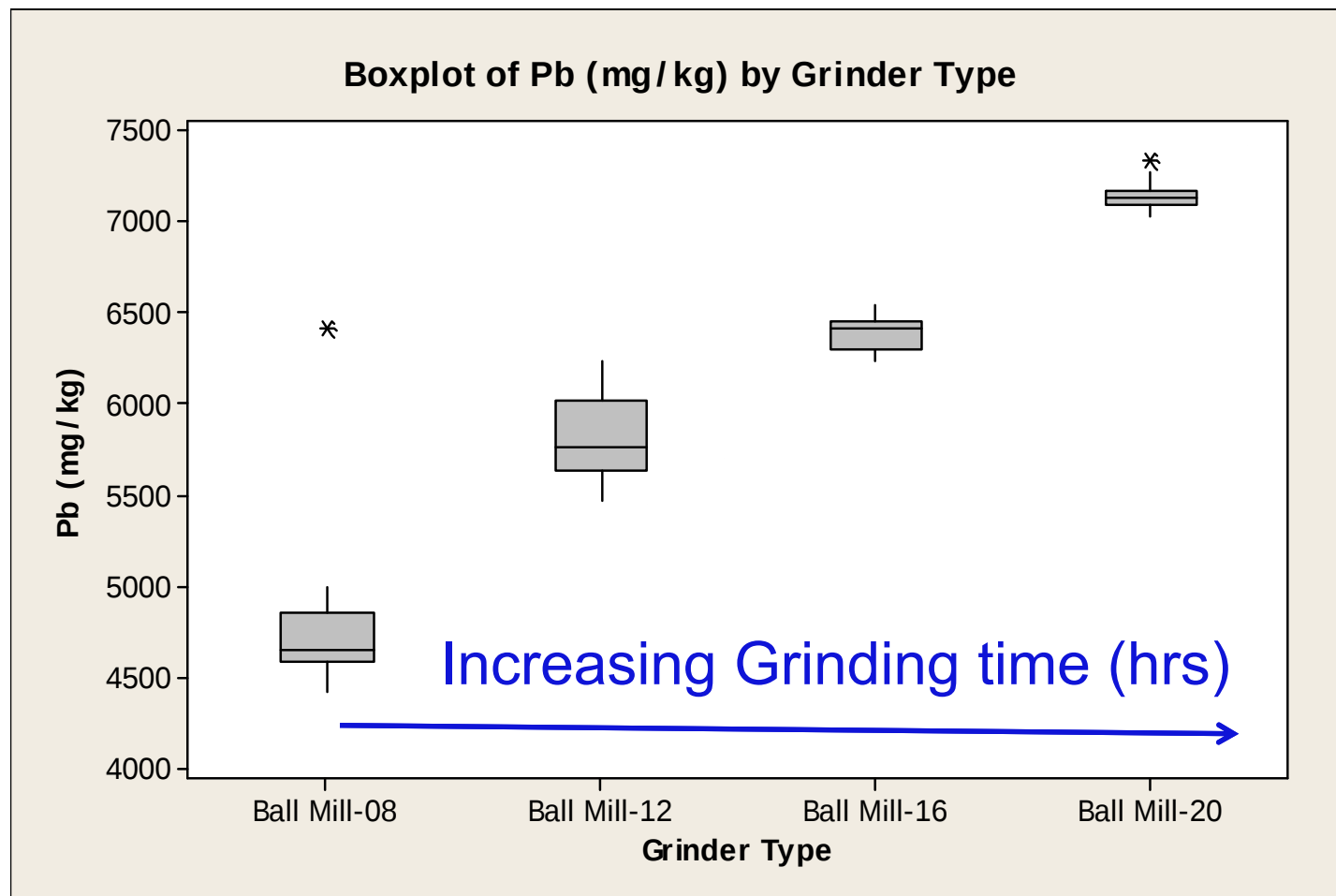
RSD (%)



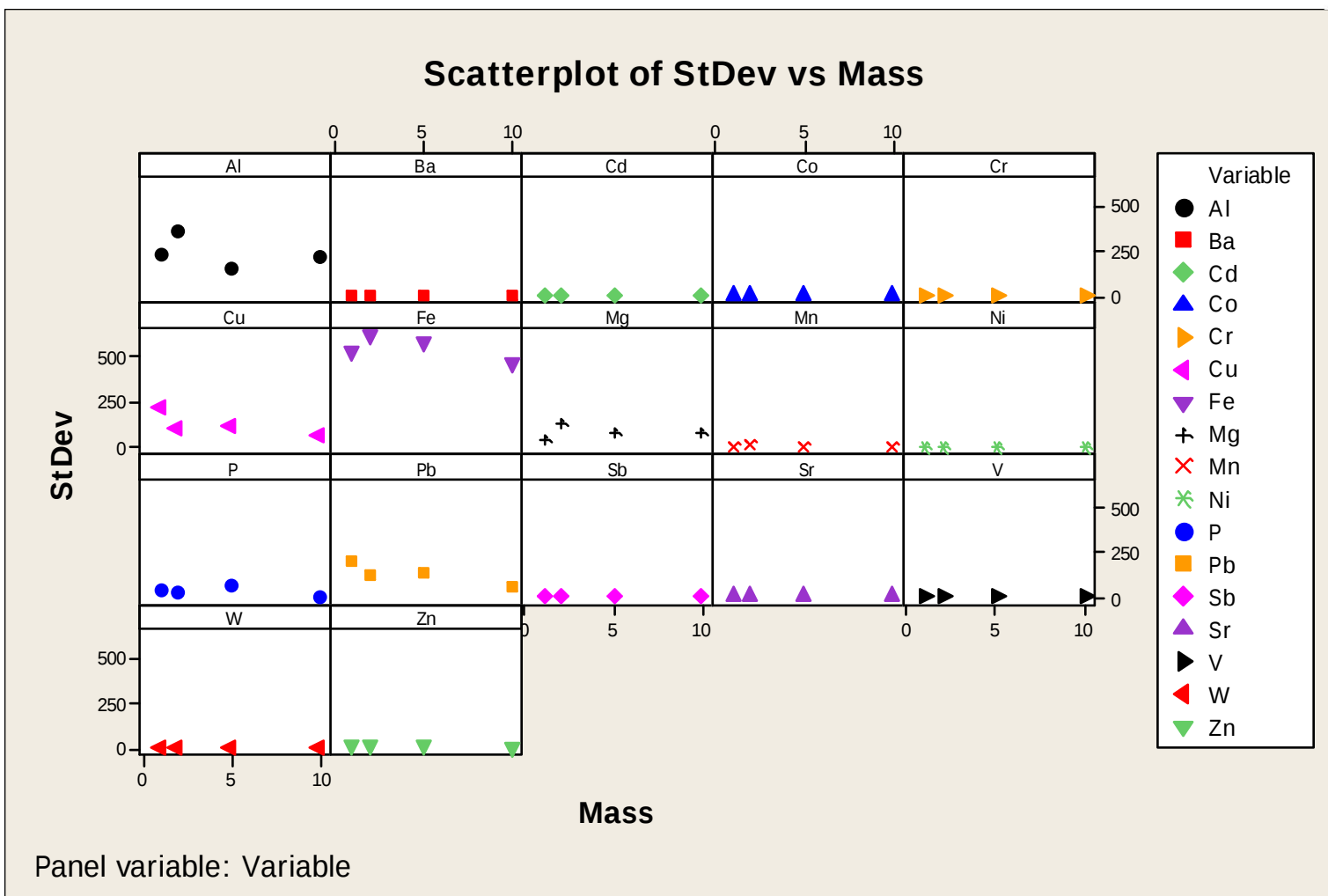
Roller Mill Optimum Grinding Interval



Roller Mill Optimum Grinding Interval



Digestion Mass



Digestion Time

Metal	M₂₄ (mg/kg)	M₄₈ (mg/kg)	Metal	M₂₄ (mg/kg)	M₄₈ (mg/kg)
Al	5678	6075	Mn	223.9	242.8
Ba	30.29	32.09	Ni	12.24	11.67
Cd	1.825	1.050	P	612.3	630.0
Co	8.60	8.935	Pb	2718	2893
Cr	221.2	242.1	Sb	22.61	20.59
Cu	542.5	498.2	Sr	21.51	23.80
Fe	16920	17293	V	15.14	16.32
Mg	2121	2259	Zn	75.80	79.88

M₂₄ , M₄₈ = Median 24- and 48-hr digestions, respectively

Issues

- Analysis error is still greater than expected between laboratories, believed associated with volume of acid used during digestion
- Considerable mass of metal remains in over size fraction (typically discarded)



- Ongoing question of impact of sample preparation method changes to risk determination
- Poor recovery of antimony is evident with conventional analysis; new digestion process needed