

AAFCO PTP

2015 Minerals Program

A Targeted and Engineered Concentration Program

Approach:

- This quarterly program focuses on minerals of health and toxicological importance in the feed and food chain at significant concentration ranges.
- Minerals will be spiked into the feed or food materials to achieve the desired concentrations that are not available in the AAFCO monthly Proficiency Testing Program.

Target Elements		
Code	Element	Units
015	Aluminum	ppm
516	Arsenic	ppm
017	Boron	ppm
518	Cadmium	ppm
520	Chromium	ppm
021	Cobalt	ppm
022	Copper	ppm
023	Fluorine	ppm
024	Iodine	ppm
526	Lead	ppm
529	Mercury	ppb
038	Molybdenum	ppm
539	Nickel	ppm
034	Selenium	ppm
036	Sulfur	%
041	Vanadium	ppm

Minerals Program PT Materials

- Able Labs Inc. is spiking and producing materials according to USGS SOP.
 - o High confidence in procedure.
 - o Cost reasonable to AAFCO rates.



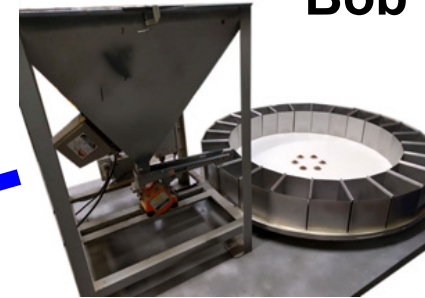
Materials Spiking Summary

- Spread unground material into thin layer on a non-metal table top or tray.
 - Aliquots of the different element solutions calculated to produce the target concentrations in the final blend are combined in a plastic spray bottle.
 - o Commercial elemental standard solutions (1 mg/mL or 10 mg/mL), when possible.
 - o In-house prepared solutions when concentration exceeding 10 mg/mL is needed.
 - Spray solution containing metal salts uniformly over the material from a distance of about 5 cm. When half of the volume is dispensed, mix the material to expose new surface area to the spraying procedure. Add about 2 mL of water to empty bottle, mix and apply the rinsate by spraying.
 - Dry.
 - Grind and split into portions using rotary splitter.
-

Minerals Program Sample Engineering

Base feed sourced from prior Check Samples.
Minerals sourced separately.

Able Laboratory Inc.
Bob Kieffer



Existing Minerals in base
feed established during
prior testing round.

- Added Minerals solubilized and sprayed over existing pre-ground base feed prior to blending and sample portioning.
- Spike concentrations calculated.



Homogenous Samples for distribution.

Target Values Based On:

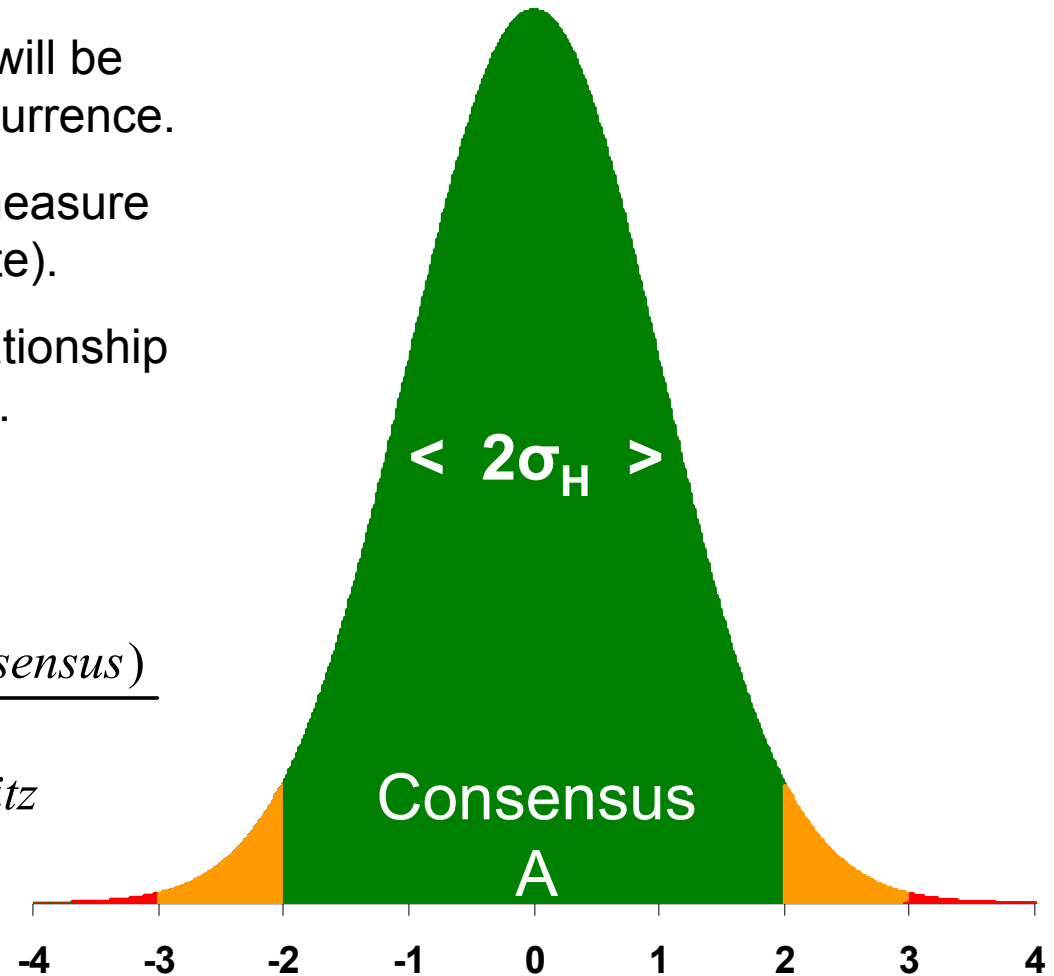
National Research Council Committee on Animal Nutrition,
Mineral Tolerance of Animals: Second Revised Edition (2005)

Element	Units	LOQ	Range	4 target Sample Concentrations			
Aluminum	ppm	20	20 - 2,000	200	2,000	1,000	450
Arsenic	ppm	0.2	0.2 - 60	2	60	30	10
Boron	ppm	15	15 - 300	30	300	150	50
Cadmium	ppm	0.05	0.05 - 20	1	20	10	3
Chromium	ppm	0.02	0.02 - 500	30	500	250	60
Cobalt	ppm	0.01	0.01 - 50	3	50	25	6
Copper	ppm	0.4	0.4 - 1,000	125	1,000	500	250
Fluorine	ppm	0.03	0.03 - 300	20	300	150	40
Iodine	ppm	0.01	0.01 - 800	50	800	400	100
Lead	ppm	0.5	0.5 - 200	10	200	100	30
Mercury	ppb	10	10 - 4,000	500	4,000	2,000	1,000
Molybdenum	ppm	0.02	0.02 - 300	5	300	150	40
Nickel	ppm	0.01	0.01 - 500	15	500	250	60
Selenium	ppm	0.01	0.01 - 10	1.25	10	5	2.5
Sulfur	%	0.005	0.005 - 1	0.125	1	0.5	0.25
Vanadium	ppm	0.005	0.005 - 100	5	100	50	10

Statistical Approach

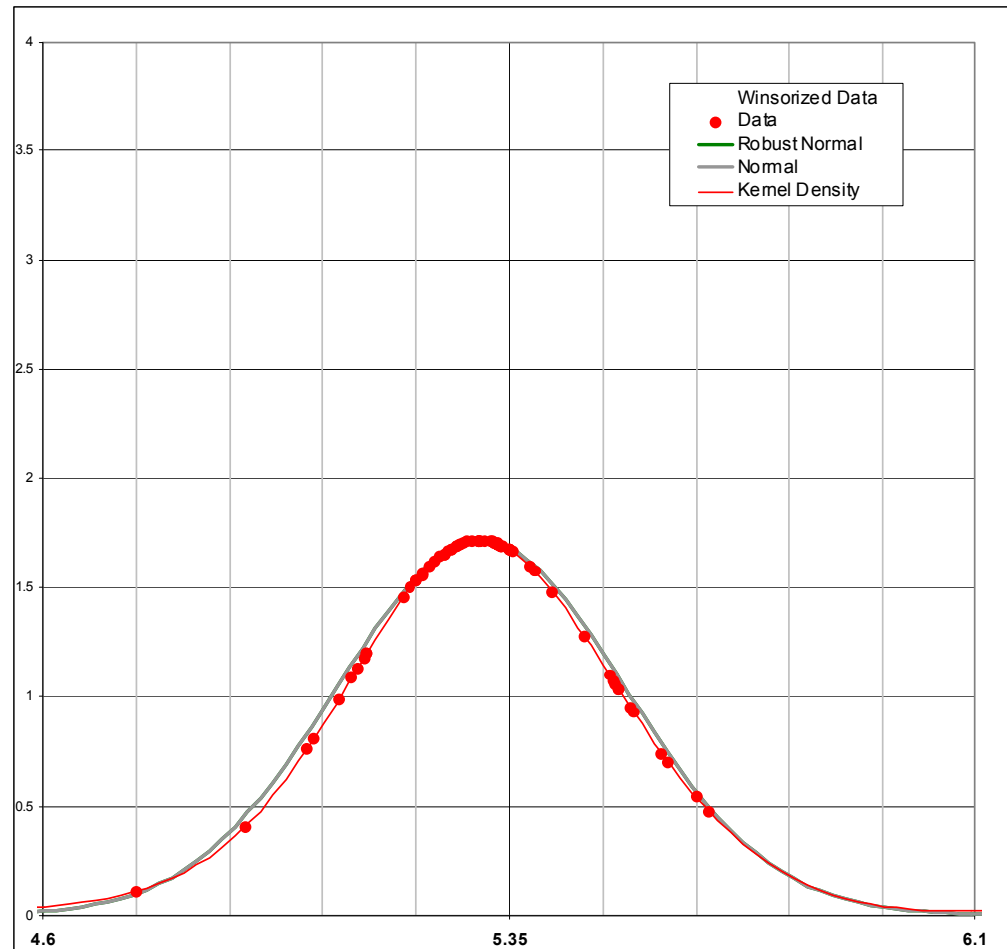
- Precise final concentration will be unknown due to natural occurrence.
- We will use a Consensus measure of Location (Robust Estimate).
- We will use the Horwitz relationship as a measure of Dispersion.

$$Z = \frac{X_{LAB} - X_{A(Consensus)}}{\sigma_{ModifiedHorwitz}}$$



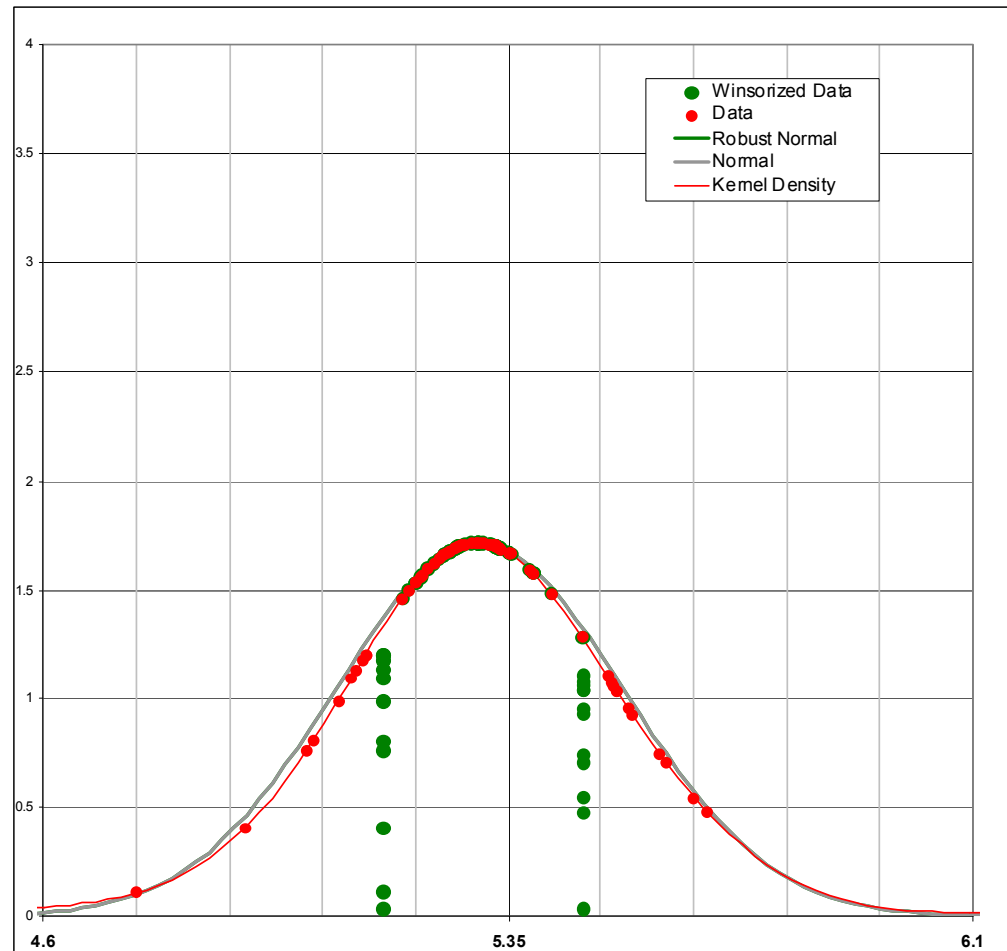
Robust Estimator Recap

- Data points (**Red**) on Kernel Density Envelope.
- Normal Curve (**Grey**), includes fat tails.



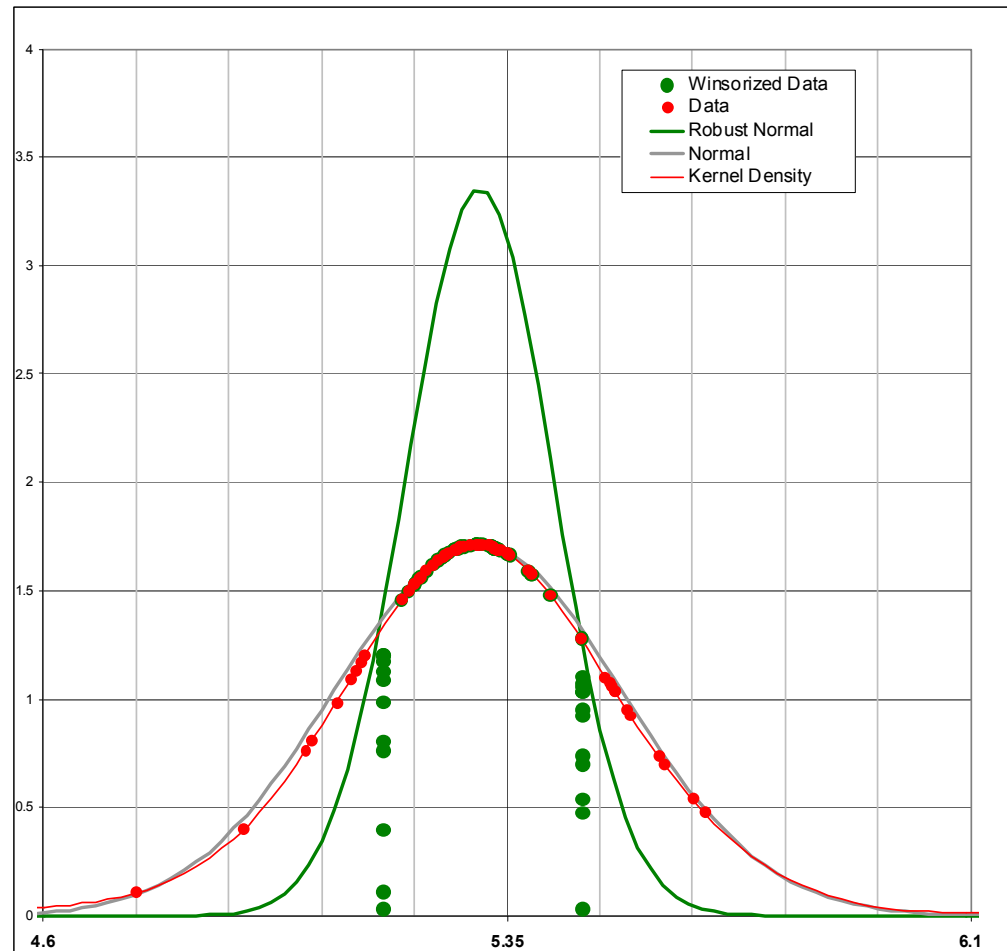
Robust Estimator Recap

- Data points (**Red**) on Kernel Density Envelope.
- Normal Curve (**Grey**), includes fat tails.
- Winsorizing Squeezes outer Data Points In (**Green Points**)



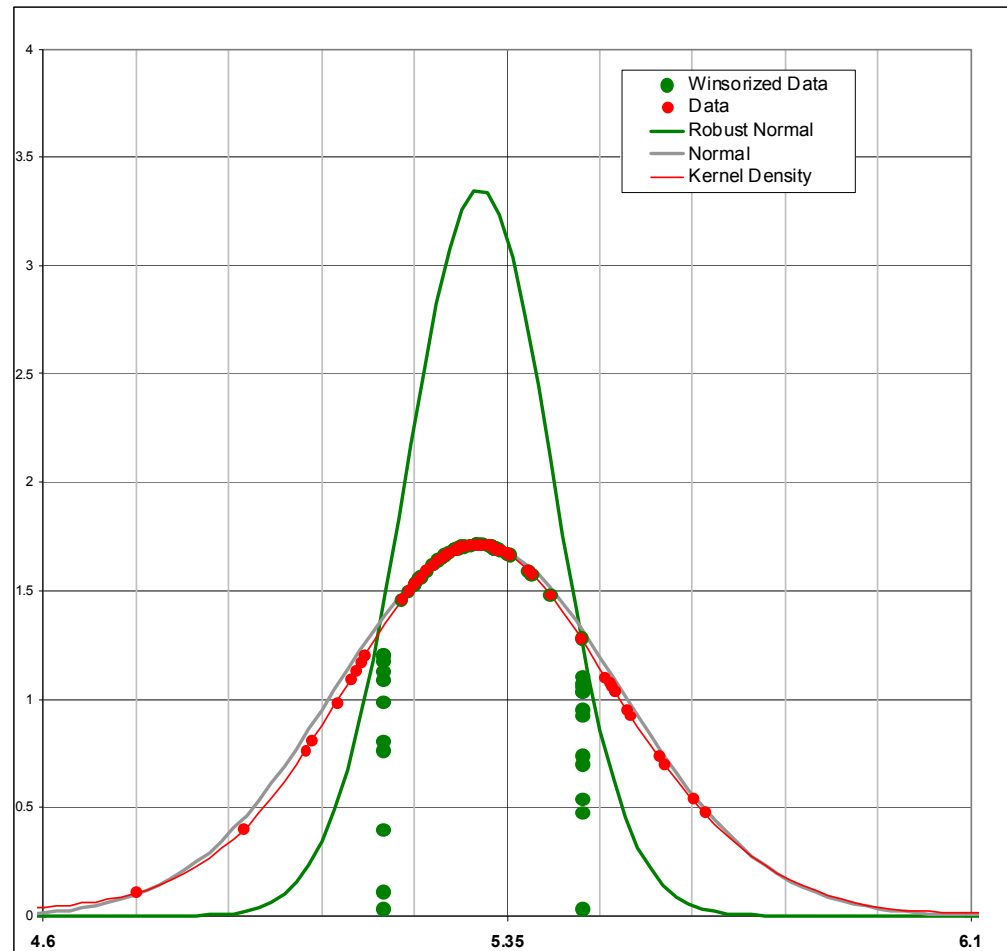
Robust Estimator Recap

- Data points (**Red**) on Kernel Density Envelope.
- Normal Curve (**Grey**), includes fat tails.
- Winsorizing Squeezes outer Data Points In (**Green Points**)
- A Robust Normal Is Calculated (**Green Curve**)
- The Robust curve provides a better estimate of the location of the mean.



Robust Estimator Recap

- Data points (**Red**) on Kernel Density Envelope.
- Normal Curve (**Grey**), includes fat tails.
- Winsorizing Squeezes outer Data Points In (**Green Points**)
- A Robust Normal Is Calculated (**Green Curve**)
- The Robust curve provides a better estimate of the location of the mean.
- Systematic reduction of the weighting of outer points.





Bill Horwitz

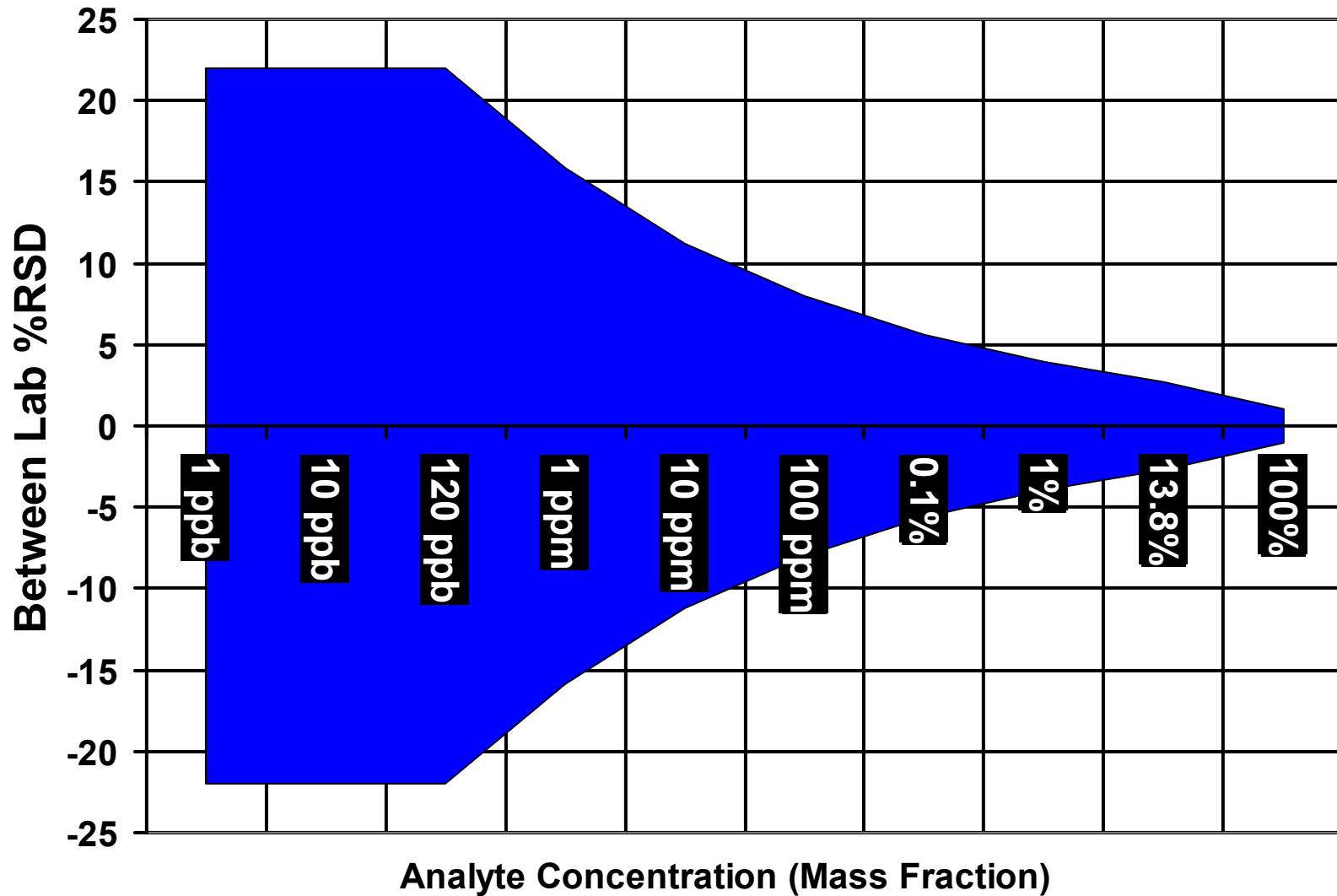
Observed very strong relationship between
Concentration and %RSD between labs.

$$\%RSD_H = 2 \times C_{mf}^{-0.1505}$$

C_{mf} (mass fraction)

IHP recommended and widely recognized among Proficiency testing providers as an appropriate measure of variance.

Modified Horwitz Trumpet



Calculating the Fit-For-Purpose Sigma

$$\sigma_{ffp} = X_A \times \frac{\%RSD_{Horwitz}}{100}$$

Measure of Dispersion For Proficiency Testing

All Targets Will be Above The LOQ

- If you Do NOT Detect it, then Do NOT Report it.
 - Report sufficient decimal places to include significant digits at the units requested.
 - Report raw data in the required units (40 ppm is 0.004 % not 0.00 %).
 - Do NOT report zero's.
 - I will purge raw data for blanks and 0's
 - All program participants do have access to the Statistical summary reports.
-



Minerals Program Assigned Value by Formulation



When there are too few participants to provide a robust Assigned Value.

- AV = Base Sample C from prior PT + Calculated Spike
- This AV used to calculate Horwitz Fit for Purpose SD.
- AV and SD Presented in **Blue**.
- Tentative Z Scores presented in **Grey**.

Method Group	Lamb Feed (201652) Analyte	Lab Code	Lab Data		Method Values				AAFCO CS Z Score	Your Method	Flag
			Value	Range	Rob X	Horwitz	R-bar	#			
023	Fluorine (mg / kg (ppm))	0619	35.300	1.8000	40.300	3.6960	1.2750	-1.35	023.01	0	
023	Fluorine (mg / kg (ppm))	0208	39.950	1.9000	40.300	3.6960	1.2750	-0.09	023.01	0	
023	Fluorine (mg / kg (ppm))	2033	43.100	0.0000	40.300	3.6960	1.2750	0.76	023.01	0	
023	Fluorine (mg / kg (ppm))	0563	60.600	1.4000	40.300	3.6960	1.2750	5.49	023.01	0	
024	Iodine (mg / kg (ppm))	0208	4.8850	0.8900	Base Conc. Not Available		0.44500	2	024.99	0	
024	Iodine (mg / kg (ppm))	2033	65.300	0.0000	Base Conc. Not Available		0.44500	2	024.53	0	
041	Vanadium (mg / kg (ppm))	0098	0.41350	0.0430	0.66600	0.11326	0.01400	-2.23	041.43	0	
041	Vanadium (mg / kg (ppm))	0047	0.42000	0.0000	0.66600	0.11326	0.01400	-2.17	041.52	0	
041	Vanadium (mg / kg (ppm))	0553	0.44950	0.0070	0.66600	0.11326	0.01400	-1.91	041.53	0	
041	Vanadium (mg / kg (ppm))	0278	0.71500	0.0100	0.66600	0.11326	0.01400	0.43	041.43	0	
041	Vanadium (mg / kg (ppm))	0563	0.83500	0.0100	0.66600	0.11326	0.01400	1.49	041.34	0	
041	Vanadium (mg / kg (ppm))	0619	0.00000	0.0000	0.66600	0.11326	0.01400	-5.88	041.41	4	