

AAFCO Check Sample 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 Check Sample Program.

Proposed Samples for 2015:

- Poultry Feed
- Rice
- Swine Feed or Dairy Feed
- Dog Food

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

- Solicited bids from private laboratories.
 - Cost prohibitive.
 - Little confidence in ability to produce.
- Located expert lab at USGS (makes NIST materials).
 - Success with prototype spiked dog food.
 - Developed SOP of spiking procedure.
 - High confidence in ability to produce at reasonable cost; but establishing a long-term contract uncertain (required approval at multiple federal agency levels). Our volumes too small for their operation.
 - Transferred SOP to Able Labs, who indicated prior experience spiking for other PT programs.
- Able is now spiking and producing materials according to USGS SOP.
 - High confidence in procedure.
 - Cost reasonable to AAFCO rates.





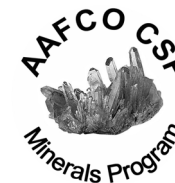
Materials Spiking Summary

- 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.
 - Commercial elemental standard solutions (1 mg/mL or 10 mg/mL), when possible.
 - 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 established procedure.
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Target Values Based On:

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

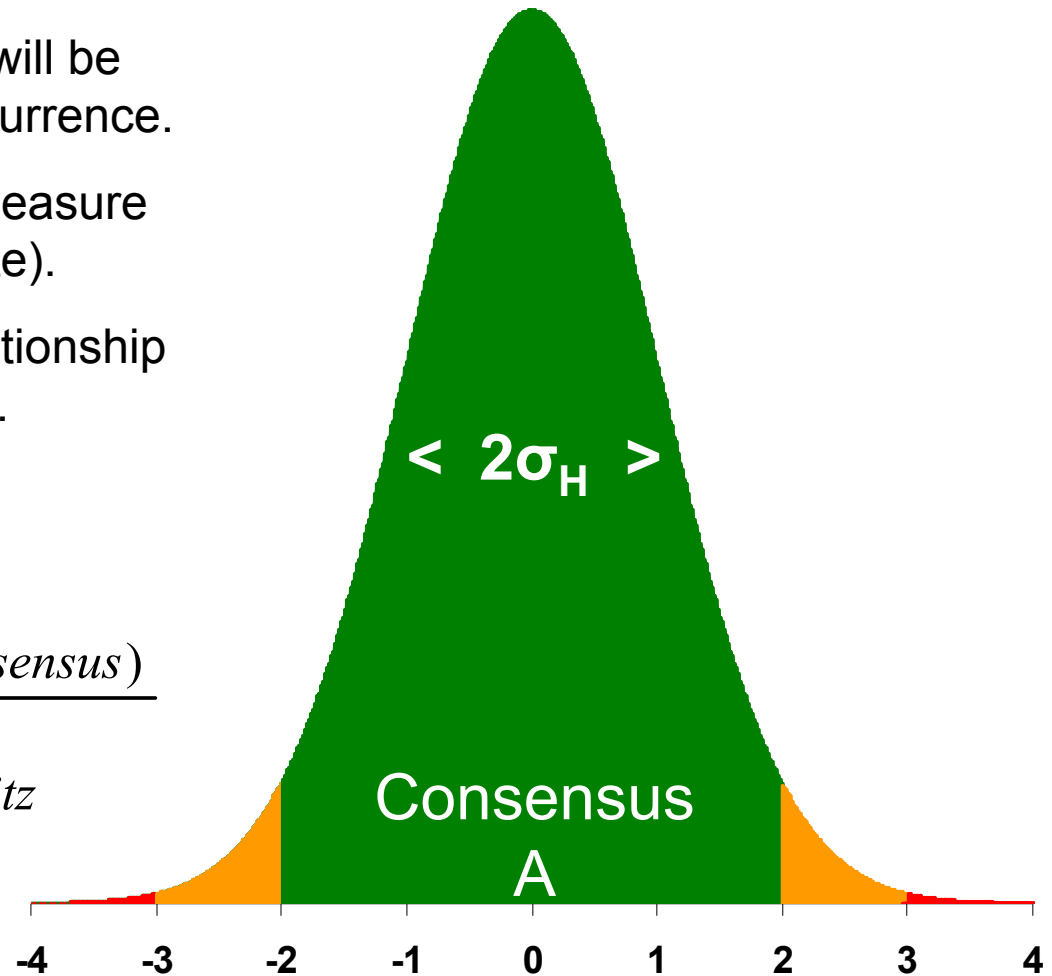


Element	Units	LOQ	Range	4 target Sample Concentrations			
Aluminum	ppm	20	20 - 2,000	200	2,000	1,000	450
Arsenic	ppm	0.2	0.2 - 60	2	60	30	10
Boron	ppm	15	15 - 300	30	300	150	50
Cadmium	ppm	0.05	0.05 - 20	1	20	10	3
Chromium	ppm	0.02	0.02 - 500	30	500	250	60
Cobalt	ppm	0.01	0.01 - 50	3	50	25	6
Copper	ppm	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

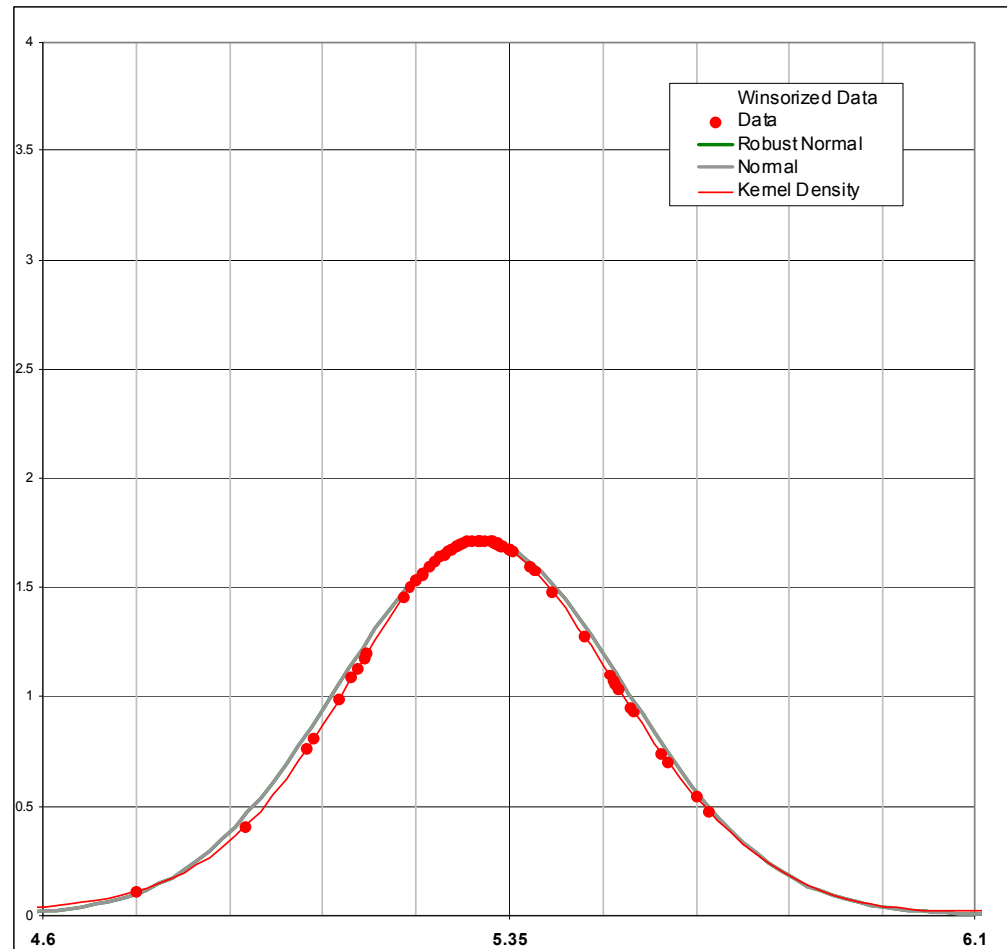
Statistical Approach

- 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.

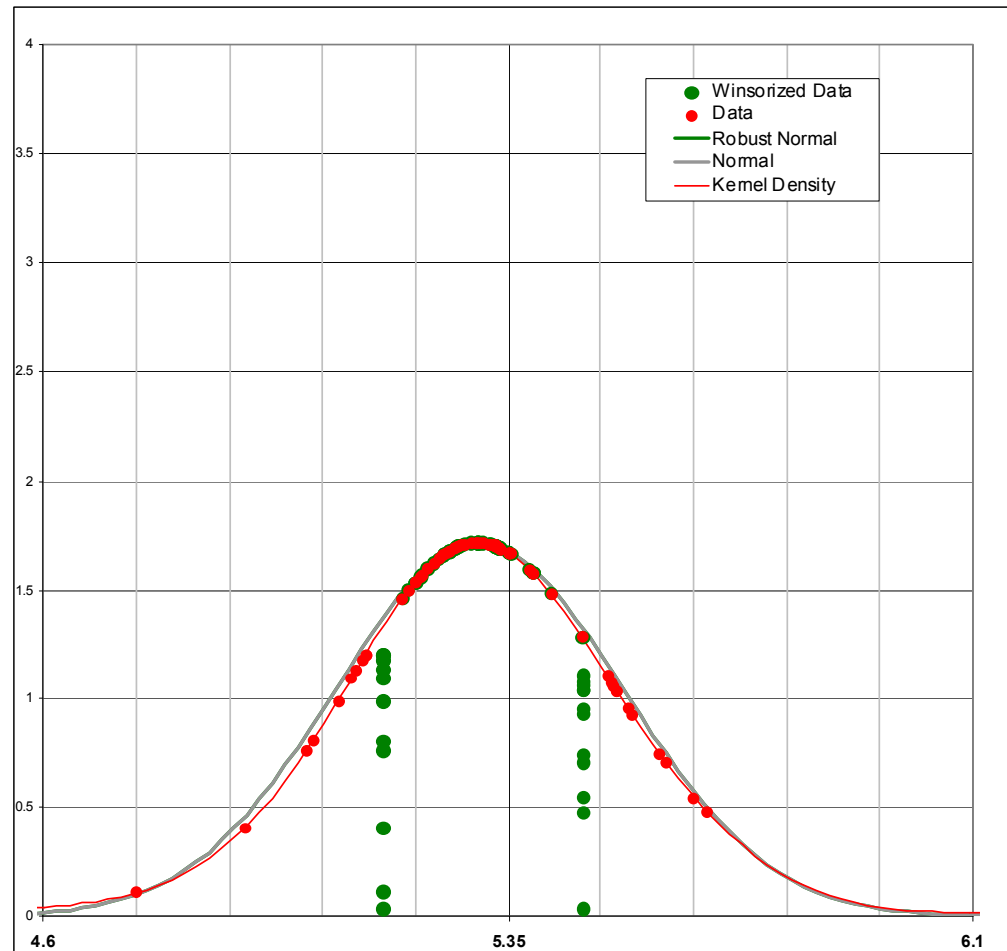
$$Z = \frac{X_{LAB} - X_{A(Consensus)}}{\sigma_{ModifiedHorwitz}}$$



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

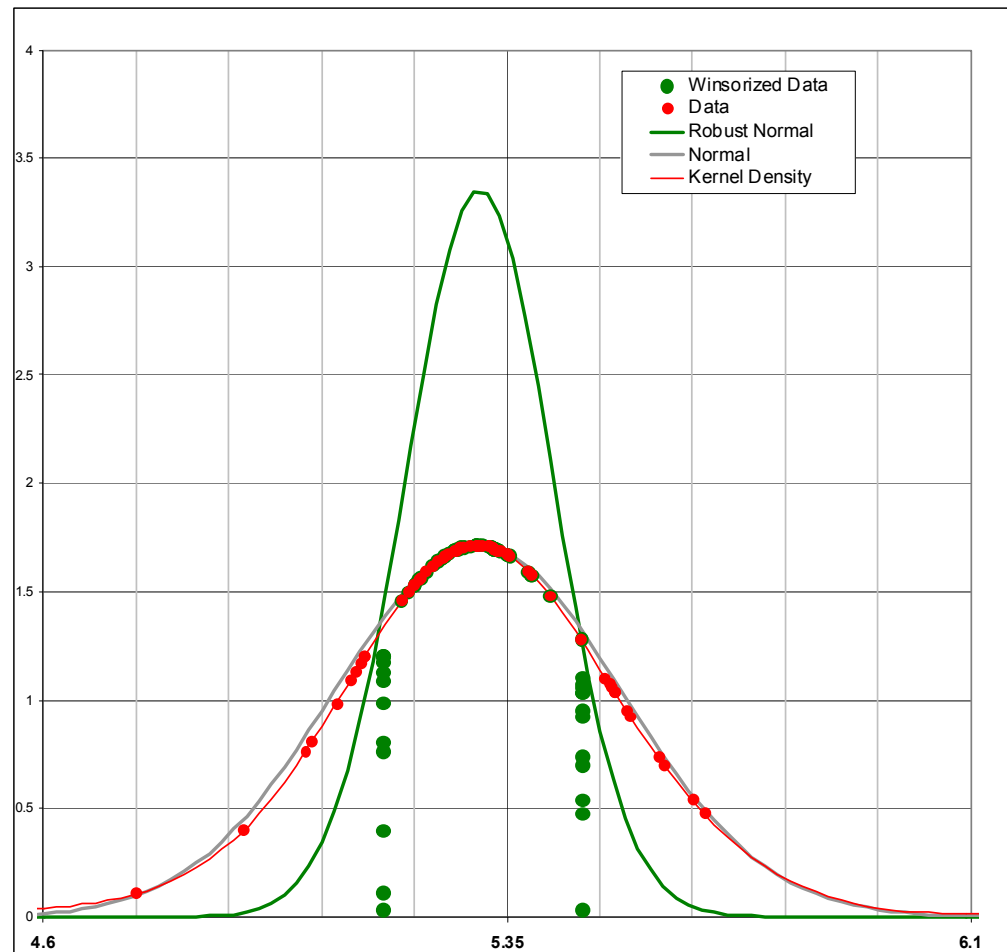


- Data points (**Red**) on Kernel Density Envelope.
- Normal Curve (**Grey**), includes fat tails.
- Winsorizing Squeezes outer Data Points In (**Green Points**)

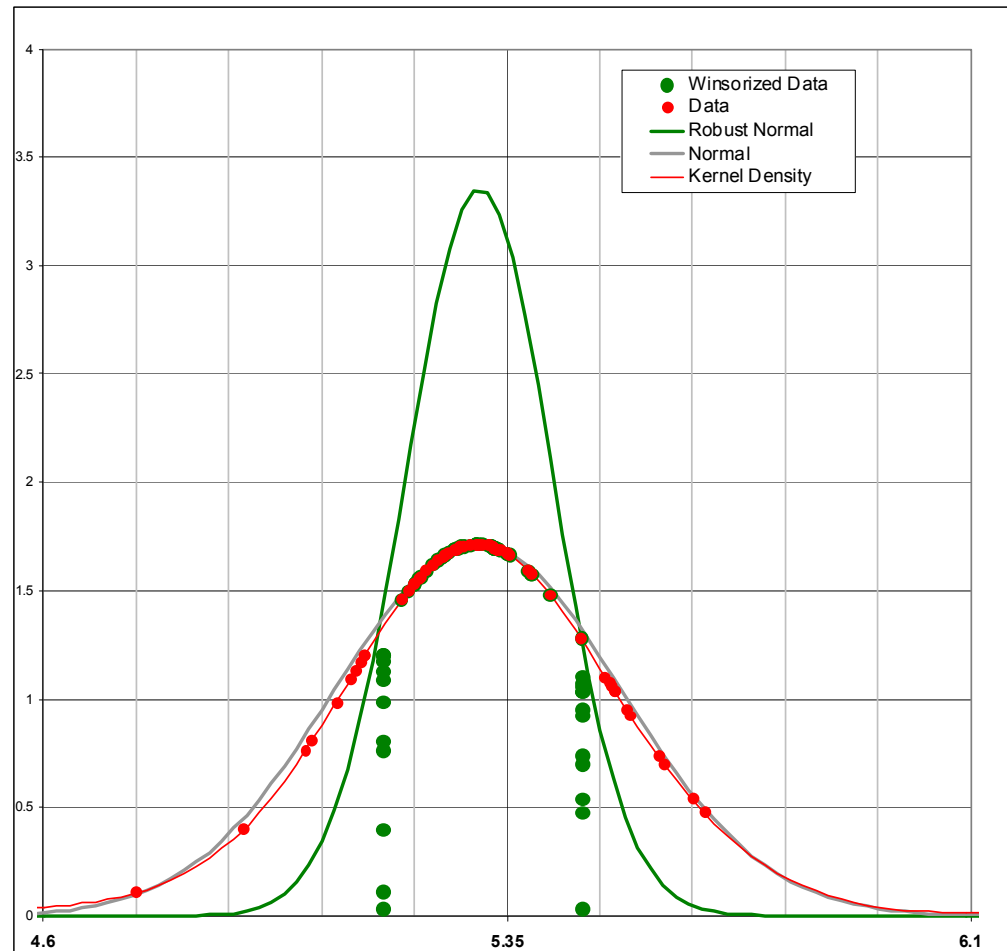


Robust Estimator Recap

- 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.



- 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

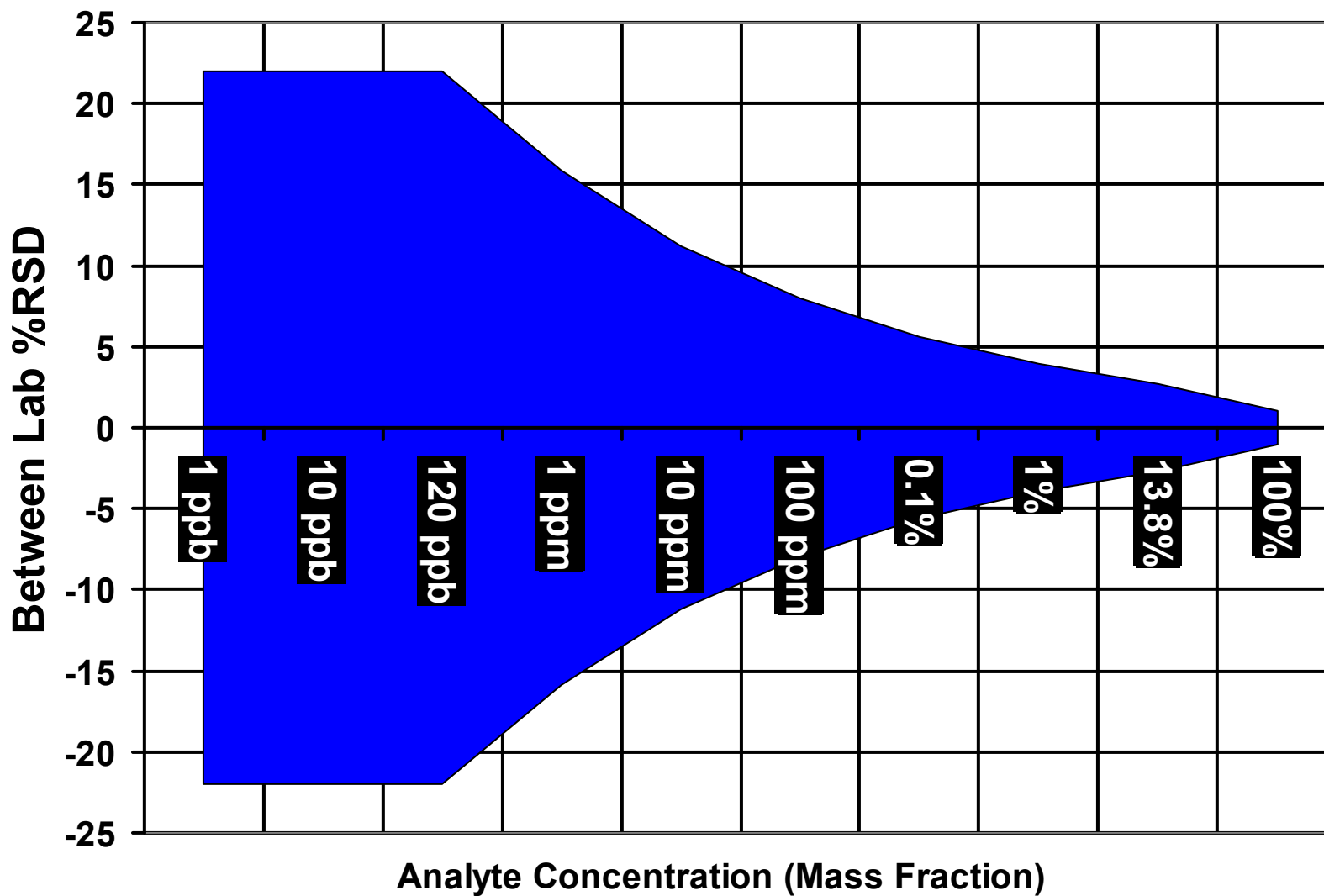
Observed very strong relationship between
Concentration and %RSD between labs.

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

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.
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Poultry Feed Is In Production

- Ship Date ~ 02/23/2015
- Data Due 04/10/2015
- Reports Available 04/30/2015





Thank You

AAFCO
Check Sample Program