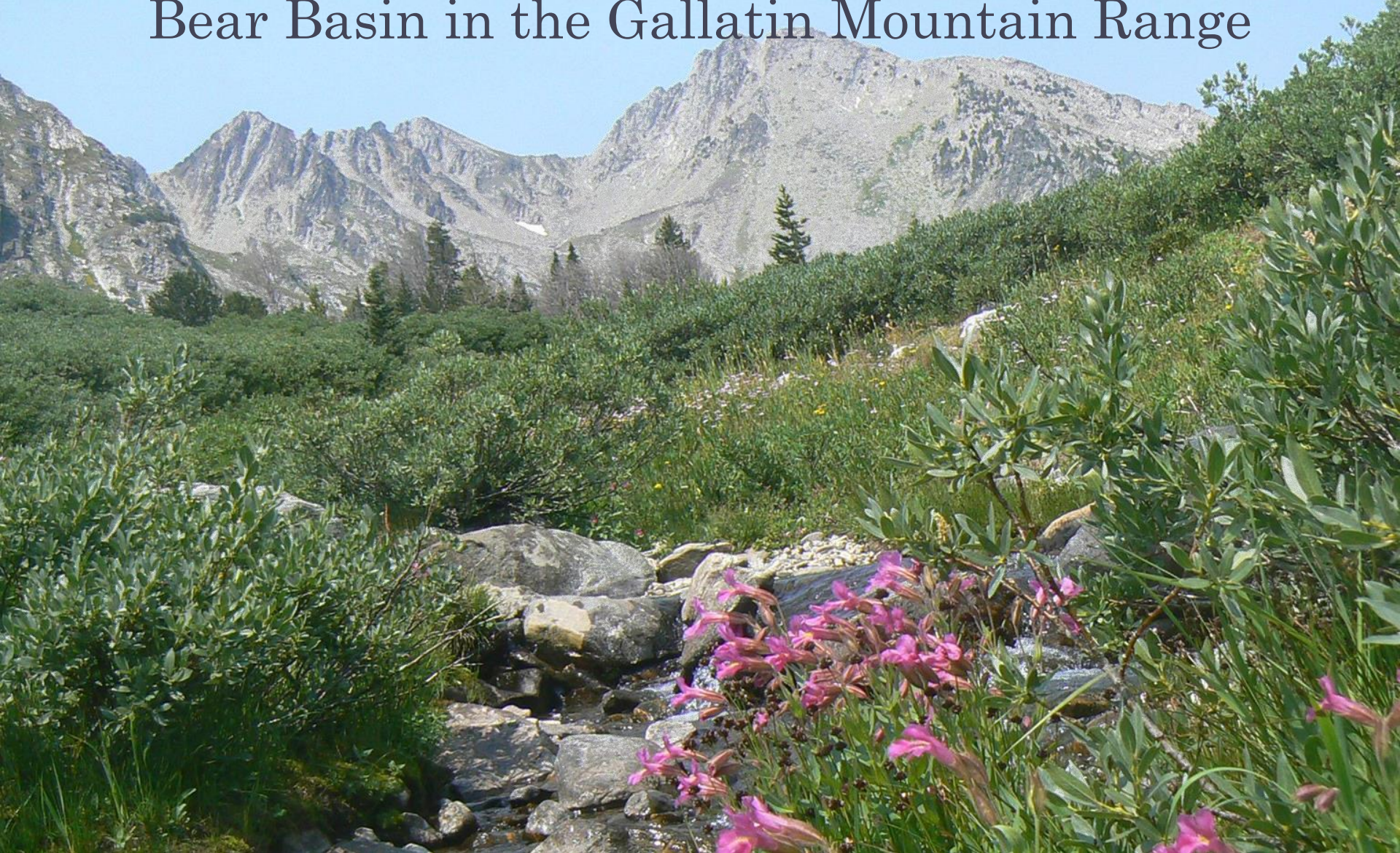
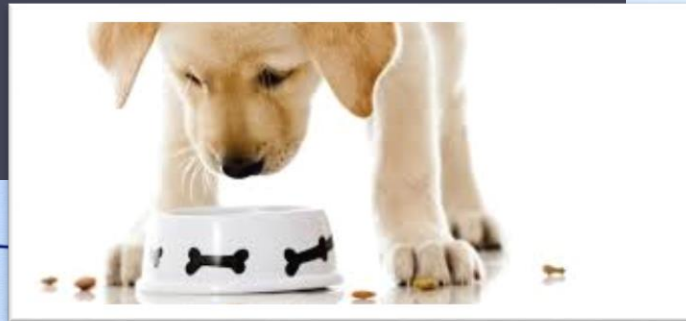


Bear Basin in the Gallatin Mountain Range



Crude Protein & Measurement Uncertainty



AAFCO Meeting
Louisville, KY 2016

ISO 17025 Requirements

- Result incomplete without a statement of uncertainty
- Reporting does not give wrong impression of uncertainty
- ISO requires that MU be estimated for your method
- **REQUIRED:** Procedure to estimate measurement uncertainty

ISO 17025 Requirements Cont.

- Understandable and relevant
- Need to put a number to the uncertainty
- You do not have to hire a statistician!
- Reasonable estimation based on method

Measurement Uncertainty (MU)

- Follow conventional methods
- Express uncertainty consistently and properly
- Others need to understand what we have done
- Traceable

Sources of Uncertainty

1. Analytical method
2. Sample PREP



ANALYTE	RESULT	SAMPLE #	DATE	ACODE	MATRIX
AAFCO QC 1229 Crude Protein	22.59	AB50802	08-Apr-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.64	AB50773	07-Apr-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.78	AB50701	30-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.67	AB50698	27-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.60	AB50690	27-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.60	AB50669	25-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.58	AB50655	23-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.70	AB50650	23-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.70	AB50648	20-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.65	AB50604	17-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.77	AB50603	17-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.72	AB50532	09-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.53	AB50523	06-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.56	AB50494	03-Mar-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.65	AB50411	20-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.60	AB50370	17-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.73	AB50359	11-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.60	AB50334	10-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.65	AB50314	09-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.67	AB50259	04-Feb-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.77	AB50230	30-Jan-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.74	AB50181	26-Jan-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.74	AB50120	16-Jan-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.70	AB50099	15-Jan-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.77	AB50019	07-Jan-15	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.69	AB43556	30-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.86	AB43551	30-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.49	AB43546	29-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.54	AB43489	19-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.75	AB43488	19-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.54	AB43419	12-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.40	AB43410	12-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.65	AB43357	05-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.64	AB43321	01-Dec-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.54	AB43240	20-Nov-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.60	AB43207	20-Nov-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.66	AB43179	18-Nov-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.62	AB43149	13-Nov-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.58	AB43068	03-Nov-14	ACPROT_FD	Feed
AAFCO QC 1229 Crude Protein	22.68	AB42335	27-Aug-14	ACPROT_FD	Feed

Average: 22.65
 Standard Deviation: 0.0915
 Instrument %RSD: 0.404

Method MU

- 40 data points from AAFCO 1229
- Collected over <1 year
- $\bar{x} = 22.65$
- SD = .0915
- RSD (%) = .404



Sample PREP MU

- 4 splits for 1 sample
- 3 replicates for each split
- Average of 3 replicates
- Average and SD of 4 splits
- Calculated %RSD
- Repeat for 4 samples
- Average %RSD of 4 samples
- %RSD= 0.88008

AB43425

sp1

	Results
Value 1	12.125
Value 2	12.124
Value 3	12.135
Average	12.128

AB43425

sp2

	Results
Value 1	11.848
Value 2	12.041
Value 3	12.082
Average	11.9903

AB43425

sp3

	Results
Value 1	11.86
Value 2	12.133
Value 3	12.042
Average	12.0117

AB43425

sp4

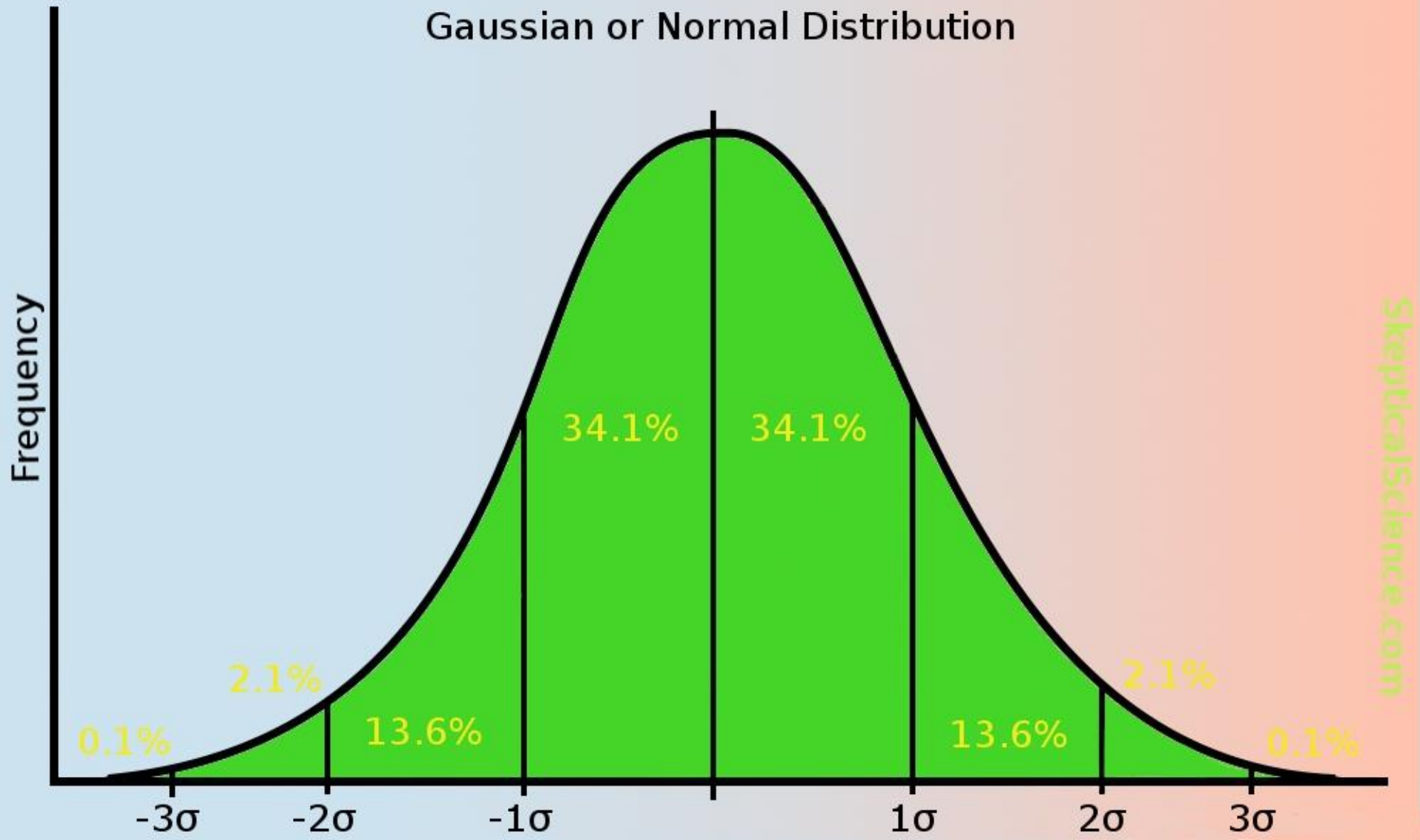
	Results
Value 1	11.928
Value 2	12.29
Value 3	12.22
Average	12.146

Mean: 12.069
STD DEV: 0.07934
%RSD: 0.6574

Crude Protein MU

- Add 2 uncertainties together
 - MU Sample PREP: 0.880
 - MU method: 0.404
- Use root sum square method (RSS)
- $$U_c = \sqrt{U_1^2 + U_2^2 + \dots + U_n^2}$$
- $$U_c = (.880^2 + .404^2)^{1/2} = .968$$
- Combined Uncertainties: .968 %

MT Example



Coverage factor K

- Numerical factor used as a multiplier
- Expand uncertainty
- Include more results at greater confidence
- Typically in the range of 2 to 3

Degrees of Freedom

- Degrees of freedom: number of measurements - 1
- For >20 values: $k = 2.0$, degrees of freedom = ∞
- Tabulated values of k shown below:

n -1	1	2	3	4	5	6	7	8	9	10
$k_{\text{(corr)}}$	12.71	4.30	3.18	2.78	2.57	2.45	2.37	2.31	2.26	2.23

n -1	11	12	13	14	15	16	17	18	19	20
$k_{\text{(corr)}}$	2.20	2.18	2.16	2.15	2.13	2.12	2.11	2.10	2.09	2.09

Crude Protein Measurement Uncertainty

- U₁: Sample PREP: 16 data points
- U₂: Method: 40 data points

Symbol	Source of Uncertainty	Value (%)	Distrb	Div	Uncert (1σ)	Degrees of Freedom
U ₁	Sample PREP	0.880	N	1	0.880	15
U ₂	Method	0.404	N	1	0.404	∞
U _c	Combined Uncertainty	0.968 %				
U	Expanded Uncertainty (k=2.13)	2.06 %				

- $U_c = (.880^2 + .404^2)^{1/2} = \mathbf{0.968}$
- $U = (2.13 \times .968) = \mathbf{2.06\%}$

Crude Protein MU

- Combined Uncertainty : **0.968 %**
- Expand Uncertainty: $.968 \% \times 2.13 = \mathbf{2.06 \%}$
- Sample: true value is **22.65 %** crude protein
- $2.06 \% \text{ of } 22.62 = \pm \mathbf{.467 \%}$
- **95%** confident result will be between **22.18 & 23.12 %**
- Expect 95% of results to be in this range

References

- International Standard, ISO/IEC 17025, Section 5.4 Test and calibration methods and method validation. Second edition 2005-05-15, Reference number ISO/IEC 17025:2005(E).
- Montana Department of Agriculture Quality Management System for ISO accredited methods
- A2LA Introduction to Measurement Uncertainty- Training Course 2013
- A Beginners guide to Uncertainty in Measurement , Stephanie Bell, NPL, Issue 2



*“A person who never made a mistake
never tried anything new.”*

Albert Einstein

