Stability Assessment from Laboratory Control Samples

Advantages and Limitations
Laboratory Control Samples

AAFCO PT Samples repeatedly analyzed over a period of time.

Sample storage ranges across:
Controlled Ambient – Refrigeration – Freezer – -15ºC.
These are not necessarily Shipping or Transportation conditions.

Elapsed time between Sample delivery and 1st LCS measurements sometimes lengthy. Particularly true of “Quality Reference Materials” purchases.

I received LCS Time vs Value and Time vs % Recovery data for several Samples from several labs. Thank you!

Analytes Included: Vet Drugs, Vitamin A, Minerals, Protein, Fat and Moisture. No alpha-tocopherol (Vit. E)!
What do we mean by STABILITY??

From the IHP:

“Materials distributed in proficiency tests must be sufficiently stable over the period in which the Assigned Value is to be valid.”

The Assigned Value is decided by Consensus over this period.

“Sufficiently Stable” means that Z scores are not significantly affected.

Do the Analyte values remain constant over a 12 week period?

Has the distribution unit “Sample” changed in any significant way?
STABILITY can be a Tricky Problem!

In the usual approach a sample is split and one half, the “Control” is analyzed. Then a period of time passes and the second half, the “Test” is analyzed.

Our sources of variance now must include; “Intermediate Variance”.

\[ \sigma_{SampleResults}^2 = \sigma_{Analytical}^2 + \sigma_{Labs}^2 + \sigma_{Sampling}^2 + \sigma_{Intermediate}^2 \]

We can control for:
- Analytical variance (\(\sigma_{Analytical}^2\)) by using a very precise method.
- Lab Bias (\(\sigma_{Labs}^2\)) by using one expert lab.
- Sampling (\(\sigma_{Sampling}^2\)) after a successful Homogeneity test.

Despite protestations, significant \(\sigma_{Intermediate}^2\) can be introduced over time. This may have nothing to do with Stability which is particularly true for “difficult” analytes.
Using Laboratory Control Samples to Assess Stability

From ISO 13528:2015 “B.5 Assessment criteria for a stability check”

\[
\left| \bar{y}_1 - \bar{y}_2 \right| \leq 0.3 \sigma_{pt}
\]

Where:
- \( \bar{y}_1 \) is mean of analyte values at 0 weeks.
- \( \bar{y}_2 \) is mean of analyte values at 12 weeks (in our case!).
- A statistical test is recommended to detect real differences.
- \( \sigma_{pt} \) is SD chosen for proficiency testing (Horwitz).

If repeatability is suspect check to see if: \( s_r > 0.5 \sigma_{pt} \)

Using 0.67 due to repeated sample handling!

Over time some intermediate variance will interfere with repeatability.
If \( s_r \) is greater than 2/3 \( \sigma_{pt} \) we have a repeatability problem.
Using Laboratory Control Samples to Assess Stability
Set up a **Linear Regression** of Time vs Analyte Value

- Calculate the **Slope** of the line.
- Calculate the significance of the Slope (P).
- $H_0$: Slope = 0 (null hypothesis)
- If $P \leq 0.05$, there is evidence of instability.
- If $P > 0.05$, there is no evidence of instability.
Remember!
We are only interested in the change over 12 weeks.

- If there is NO evidence of instability:
  - Slope = 0; in our best estimate no instability predicted.
- If there is evidence of instability (Slope <> 0):
  - Using the Slope calculate a difference over 12 weeks.
  - This is equivalent to the ISO recommendation:

\[
\left| \bar{Y}_{0\text{weeks}} - \bar{Y}_{12\text{weeks}} \right| \leq 0.3\sigma_{pt}
\]

Does the analytical variance swamp out \(\sigma_{pt}\)?
Check by looking for \(s_r > 0.67 \sigma_{pt}\)!
16 measurements of Amprolium (ppm) over 172 weeks!

- **Amprolium** 201326 Chicken Starter/Grower, Medicated ppm

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**Evidence of Instability over 172 weeks.**

- Significance of Slope: 0.0002
- 12 Week % Rel. Diff.: 0.48%
- Sigma PT (%RSD): 7.89% (Horwitz)
- Allowed Var, 33% σ PT (A): 2.919
- 12 Week Difference (B): 0.348

**Analytical %rsd** 1.26%
**Analytical Variance Flag** sr OK
Should be > 1 4.21

**Use SE of Y estimate**

- Weeks from End of Round: -3
9 measurements of Decoquinate (ppm) over 25 weeks!

Significance of Slope: 0.3040
No Evidence of Instability over 25 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 9.98% (Horwitz)
Allowed Var, 33%σ PT (A): 0.700
12 Week Difference (B): 0.000

Is B < A? YES, as NO difference seen.

Analytical %rSD: 2.40%
Analytical Variance Flag: sr OK
Should be > 1: 2.83

Use SD of 9 measurements

Weeks from End of Round: 16
18 measurements of Vitamin A (KU/kg) over 96 weeks!

Important for QRM’s but Not for 12 week window

Significance of Slope 0.0030
Evidence of Instability over 96 weeks.
12 Week % Rel. Diff. 4.78%

Sigma PT (%RSD) 18.56% (Participants)
Allowed Var, 33%σ PT (A) 19.595
12 Week Difference (B) 8.819

Analytical %rsd 9.73%
Analytical Variance Flag sr OK
Should be > 1 1.28
18 measurements of Crude Fat (%) over 43 weeks!

Crude Fat

201532 Distillers Dried Grains with Solubles %

< 45 Weeks >

Significance of Slope  0.3503
No Evidence of Instability over 43 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD)  3.28% (Horwitz)
Allowed Var , 33%σ PT (A)  0.038
12 Week Difference (B)  0.000

Analytical %rsd  1.83%
Analytical Variance Flag  sr OK
Should be > 1  2.35

Weeks from End of Round  45
18 measurements of Calcium (%) over 50 weeks!

No Evidence of Instability over 50 weeks.

Sigma PT (%RSD) 3.38% (Horwitz)
Allowed Var, 33% σ PT (A) 0.033
12 Week Difference (B) 0.000

Analytical %rsd 2.21%
Analytical Variance Flag sr OK
Should be > 1 1.30

Significance of Slope 0.0994

Weeks from End of Round 62
Using LCS to Assess Stability Data Summary

<table>
<thead>
<tr>
<th>Analyte</th>
<th># Data Sets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amprolium</td>
<td>4</td>
</tr>
<tr>
<td>Carbadox</td>
<td>1</td>
</tr>
<tr>
<td>Decoquinate</td>
<td>3</td>
</tr>
<tr>
<td>Lasalocid</td>
<td>2</td>
</tr>
<tr>
<td>Monensin</td>
<td>2</td>
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<tr>
<td>Vitamin A</td>
<td>10</td>
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<td>Calcium</td>
<td>5</td>
</tr>
<tr>
<td>Copper</td>
<td>1</td>
</tr>
<tr>
<td>Magnesium</td>
<td>4</td>
</tr>
<tr>
<td>Potassium</td>
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</tr>
<tr>
<td>Zinc</td>
<td>4</td>
</tr>
<tr>
<td>Crude Fat</td>
<td>2</td>
</tr>
<tr>
<td>Moj. Fat</td>
<td>1</td>
</tr>
<tr>
<td>Moisture</td>
<td>1</td>
</tr>
<tr>
<td>Protein</td>
<td>1</td>
</tr>
</tbody>
</table>

42 Data sets from 25 Samples Reviewed

- 4 Significant slopes \((P < 0.05)\)
  - 1 had a positive slope??
  - 2 Vitamin A
  - 1 Amprolium
  - 1 Decoquinate
- 6 Marginally significant slopes \((0.05 < P < 0.1)\)
- 38 show “No Evidence of Instability”
- 11 show “sr Suspect”
- 41 of 42 “PASSED” 12 week difference criteria.
- So 29 out of 41 “PASSED”, no decision on 13.
Using LCS to Assess Stability Data Summary

Some Observations:

- Does not account for period between grinding and analysis (12 week window).
- Does not account for fluctuating environmental conditions (temperature, humidity, etc.).
- Advantage of many measurements.
- Exposes long term stability for QRM’s.
- In many cases analytical variance not as precise as controlled study.
- Success in these studies requires excellent repeatability.
Using LCS to Assess Stability Data Summary

Some Thoughts:

- Unlike Homogeneity, Stability is extremely **Analyte specific**.
- The data suggests that this specificity may also be Sample specific.
- Identify Analytes of concern and direct studies at materials containing analytes of interest.

**My take on this:** From the reviewed data and many general observations of LCS, overall we are in good shape regarding PT sample **sufficient stability**.
Thank you! Can we review the additional data?
Amprolium 201326 Chicken Starter/Grower, Medicated ppm

Significance of Slope 0.0002
Evidence of Instability over 172 weeks.
12 Week % Rel. Diff. 0.48%

Sigma PT (%RSD) 7.89% (Horwitz)
Allowed Var , 33% σ PT (A) 2.919
12 Week Difference (B) 0.348

Analytical %rsd 1.26%
Analytical Variance Flag  sr OK
Should be > 1 4.21

Weeks from End of Round -3

LCS Values
Reported Value
Significance of Slope: 0.1155

No Evidence of Instability over 32 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 7.89% (Horwitz)

Allowed Var, 33% σ PT (A): 2.733

12 Week Difference (B): 0.000

Stability Decision, Is B < A?: PASS Allowed Var.

Analytical %rsd: 1.88%

Analytical Variance Flag: sr OK

Should be > 1: 4.04

Weeks from End of Round: 62
Amprolium 201527 Chick Starter, Medicated ppm

Significance of Slope 0.4492
No Evidence of Instability over 27 weeks.

12 Week % Rel. Diff. NA
Analysis Variance Flag sr OK

Sigma PT (%RSD) 8.01% (Horwitz)
Allowed Var, 33% σ PT (A) 2.620
12 Week Difference (B) 0.000

Analytical %rsd 1.69%

Weeks from End of Round 28
Amprolium 201623 Poultry Feed, Medicated %

Significance of Slope 0.6110
No Evidence of Instability over 13 weeks.
12 Week % Rel. Diff. NA

Analytical %rsd 5.32%
Analytical Variance Flag sr Suspect
Should be > 1 0.99

Sigma PT (%RSD) 7.56% (Horwitz)
Allowed Var , 33% σ PT (A) 0.000
12 Week Difference (B) 0.000


Weeks from End of Round 23


LCS Values
Reported Value
Calcium 201429 Equine Feed %

Significance of Slope 0.0994
No Evidence of Instability over 50 weeks.

Analytical %rsd 2.21%
Analytical Variance Flag sr OK
Should be > 1 1.30

Sigma PT (%RSD) 3.36% (Horwitz)
Allowed Var , 33% σ PT (A) 0.033
12 Week Difference (B) 0.000

weeks from End of Round 62
**Calcium 201427 Calf Starter / Grower, Medicated**

- **% Recovery**

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**Significance of Slope**: 0.0669

**No Evidence of Instability over 32 weeks.**

- **Analytical %rsd**: 3.10%
- **Analytical Variance Flag**: sr OK
  - Should be > 1
  - 1.20

**Sigma PT (%RSD)**: 5.19% (Participants)

- **Allowed Var, 33% σ PT (A)**: 0.019
- **12 Week Difference (B)**: 0.000

**Stability Decision, Is B < A?**: PASS Allowed Var.

**Weeks from End of Round**: 19
Calcium 201528 Dog Food % Recovery

Significance of Slope 0.5023
No Evidence of Instability over 29 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 2.81%
Analytical Variance Flag sr OK
Should be > 1 1.15

Sigma PT (%RSD) 4.92% (Participants)
Allowed Var, 33% σ PT (A) 0.023
12 Week Difference (B) 0.000


LCS Values
Robust Value

Weeks from End of Round 0
Calcium 201529 Calf Feed, Medicated % Recovery

Significance of Slope 0.1160
No Evidence of Instability over 31 weeks.

12 Week % Rel. Diff. NA Analytical %rsd 3.20%
Sigma PT (%RSD) 5.64% (Participants) Analytical Variance Flag sr OK

Allowed Var , 33% σ PT (A) 0.157 Should be > 1 1.23
12 Week Difference (B) 0.000

Weeks from End of Round 4
Calcium 201623 Poultry Feed, Medicated % Recovery

Significance of Slope 0.9966
No Evidence of Instability over 17 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 3.07%
Analytical Variance Flag sr OK
Should be > 1 1.16

Sigma PT (%RSD) 5.53% (Participants)
Allowed Var , 33% σ PT (A) 0.019
12 Week Difference (B) 0.000


Weeks from End of Round 2

LCS Values
Robust Value
Carbadox 201422 Swine Grower, Medicated ppm

Significance of Slope 0.1982
No Evidence of Instability over 106 weeks.

Analytical %rsd 6.45%
Analytical Variance Flag sr OK
Should be > 1 6.03

Sigma PT (%RSD) 9.02% (Horwitz)
Allowed Var 33% σ PT (A) 1.192
12 Week Difference (B) 0.000

Weeks from End of Round 4

LCS Values
Robust Value
Significance of Slope 0.1800

No Evidence of Instability over 50 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 0.68% (Horwitz)

Allowed Var , 33% σ PT (A) 0.434

12 Week Difference (B) 0.000


Analytical Variance Flag sr OK

Should be > 1 1.30

Analytical %rsd 3.51%

 Weeks from End of Round 63
**Crude Fat**

**201532**  
**Distillers Dried Grains with Solubles**  

- **Significance of Slope**: 0.3503
- **No Evidence of Instability over 43 weeks.**
- **Analytical %rstd**: 1.83%
- **Analytical Variance Flag**: sr OK
- **Should be > 1**: 2.35
- **Sigma PT (%RSD)**: 3.28%  
  Horwitz
- **Allowed Var., 33% σ PT (A)**: 0.038
- **12 Week Difference (B)**: 0.000
- **Stability Decision, Is B < A?**: PASS Allowed Var.
- **Weeks from End of Round**: 45
Significance of Slope: 0.5685
No Evidence of Instability over 2 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 3.07% (Horwitz)
Allowed Var, 33% σ PT (A): 0.054
12 Week Difference (B): 0.000

Stability Decision, Is B < A?: PASS Allowed Var.

Analytical %rsd: 1.36%
Analytical Variance Flag: sr OK
Should be > 1: 2.48

Weeks from End of Round: 29
Significance of Slope 0.0561
No Evidence of Instability over 36 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 9.46% (Horwitz)
Allowed Var , 33% σ PT (A) 1.050
12 Week Difference (B) 0.000


Analytical %rsd 8.77%
Analytical Variance Flag sr Suspect
Should be > 1 0.85

Sigma PT (%RSD) 9.46% (Horwitz)
Allowed Var , 33% σ PT (A) 1.050
12 Week Difference (B) 0.000


Weeks from End of Round 54
Decoquinate 201622 Lamb Feed, Medicated ppm

Significance of Slope 0.3040
No Evidence of Instability over 25 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 9.98% (Horwitz)
Allowed Var , 33% σ PT (A) 0.700
12 Week Difference (B) 0.000

Analytical %rsd 2.40%
Analytical Variance Flag sr OK
Should be > 1 2.83

Weeks from End of Round 16
Significance of Slope 0.0220
Evidence of Instability over 16 weeks.
12 Week % Rel. Diff. 14.62%
Sigma PT (%RSD) 10.03% (Horwitz)
Allowed Var , 33% σ PT (A) 0.704
12 Week Difference (B) 2.164

Analytical %rsd 3.97%
Analytical Variance Flag sr OK
Should be > 1 1.69

Weeks from End of Round 21
Lasalocid 201331 Cattle grower, Medicated ppm

Significance of Slope 0.6378
No Evidence of Instability over 37 weeks.

Analytical %rSD 5.83%
Analytical Variance Flag sr Suspect
Should be > 1 0.93

Sigma PT (%RSD) 8.76% (Horwitz)
Allowed Var, 33% σ PT (A) 1.614
12 Week Difference (B) 0.000

Weeks from End of Round 80
Significance of Slope 0.2173
No Evidence of Instability over 45 weeks.

12 Week % Rel. Diff.  NA
12 Week Difference (B) 0.000
Sigma PT (%RSD) 9.44% (Horwitz)
Allowed Var, 33% σ PT (A) 0.986
12 Week Difference (B) 0.000

LCS Values
Reported Value

Analytical %rsd 7.54%
Analytical Variance Flag sr OK
Should be > 1 1.07

Weeks from End of Round 35
Magnesium 201427 Calf Starter / Grower, Medicated % Recovery

Significance of Slope 0.7923
No Evidence of Instability over 32 weeks.
12 Week % Rel. Diff. NA

Sigma PT (%RSD) 4.12% (Participants)
Allowed Var, 33% σ PT (A) 0.004
12 Week Difference (B) 0.000

Analytical %rsd 3.48%
Analytical Variance Flag sr Suspect Should be > 1 0.76

Weeks from End of Round 19
**Magnesium 201429 Equine Feed**

**Significance of Slope**: 0.0555

**No Evidence of Instability over 50 weeks.**

**Analytical %rsd**: 3.16%

**Analytical Variance Flag**: sr OK

**Sigma PT (%RSD)**: 4.67% (Horwitz)

**Allowed Var, 33% σ PT (A)**: 0.005

**12 Week Difference (B)**: 0.000

**Stability Decision, Is B < A?** PASS Allowed Var.

**Weeks from End of Round**: 63
Significance of Slope 0.9166

**No Evidence of Instability over 29 weeks.**

12 Week % Rel. Diff. NA

Analytical %rsd 5.60%

Analytical Variance Flag sr Suspect

Should be > 1 0.67

Sigma PT (%RSD) 6.08% (Participants)

Allowed Var, 33% σ PT (A) 0.003

12 Week Difference (B) 0.000


Weeks from End of Round 0
Significance of Slope 0.1164

No Evidence of Instability over 19 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 3.15%

Analytical Variance Flag sr OK

Should be > 1 1.26

Sigma PT (%RSD) 5.11% (Participants)

Allowed Var, 33% σ PT (A) 0.006

12 Week Difference (B) 0.000


Weeks from End of Round 1
Moisture 201527 Chick Starter, Medicated % Recovery

<table>
<thead>
<tr>
<th>Date</th>
<th>LCS Values</th>
<th>Robust Value</th>
</tr>
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<tbody>
<tr>
<td>6/13/2015</td>
<td></td>
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<td>9/5/2015</td>
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<td></td>
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<tr>
<td>11/28/2015</td>
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</tbody>
</table>

Significance of Slope 0.9475

No Evidence of Instability over 9 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 0.46%

Analytical Variance Flag sr OK

Should be > 1 4.55

Sigma PT (%RSD) 3.31% (Participants)

Allowed Var , 33% σ PT (A) 0.104

12 Week Difference (B) 0.000


Weeks from End of Round 1
Moj. Fat 201225 Dry Cat Food %

Significance of Slope 0.7066

No Evidence of Instability over 34 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 2.80% (Horwitz)

Allowed Var , 33% σ PT (A) 0.094

12 Week Difference (B) 0.000


Weeks from End of Round 184

LCS Values

Robust Value
Significance of Slope

No Evidence of Instability over 56 weeks.

12 Week % Rel. Diff.  NA

Sigma PT (%RSD) 7.03% (Horwitz)
Allowed Var , 33% σ PT (A) 5.788
12 Week Difference (B) 0.000


Analytical %rSD 4.80%
Analytical Variance Flag sr Suspect
Should be > 1 0.97

Weeks from End of Round 135
Monensin 201524 Goat Feed, Medicated g/ton

Significance of Slope 0.0640
No Evidence of Instability over 65 weeks.

12 Week % Rel. Diff. NA

Analytical %rds 5.66%
Analytical Variance Flag sr OK
Should be > 1 1.11

Sigma PT (%RSD) 9.03% (Horwitz)
Allowed Var, 33% & PT (A) 0.611
12 Week Difference (B) 0.000


Weeks from End of Round 19
Significance of Slope 0.5903
No Evidence of Instability over 49 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 3.86% (Horwitz)
Allowed Var, 33% σ PT (A) 0.016
12 Week Difference (B) 0.000


Analytical %rsd 2.47%
Analytical Variance Flag sr OK
Should be > 1 1.48

Weeks from End of Round 63
Protein 201623 Poultry Feed, Medicated % Recovery

Significance of Slope 0.3302

No Evidence of Instability over 15 weeks.

Analytical %rsd 2.01%
Analytical Variance Flag sr Suspect
Should be > 1 0.57

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 1.71% (Participants)
Allowed Var , 33% σ PT (A) 0.106
12 Week Difference (B) 0.000


Weeks from End of Round 1
**Vitamin A 201491 Poultry Layer Feed KU/kg**

**Significance of Slope**: 0.0030

**Evidence of Instability over 96 weeks**.

12 Week % Rel. Diff. 4.78%

**Sigma PT (%RSD)** 18.56% (Participants)

Allowed Var, 33% σ PT (A) 19.595

12 Week Difference (B) 8.819

**Stability Decision, Is B < A?** PASS Allowed Var.

**Analytical %rsd** 9.73%

**Analytical Variance Flag** sr OK

**Should be > 1** 1.28

**Weeks from End of Round** 45
Significance of Slope: 0.9359

No Evidence of Instability over 11 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 13.67% (Participants)
Allowed Var, 33% σ PT (A): 0.975
12 Week Difference (B): 0.000


Analytical %rsd: 8.43%
Analytical Variance Flag: sr OK
Should be > 1: 1.02

Weeks from End of Round: 214
Significance of Slope 0.0271
Evidence of Instability over 119 weeks.
12 Week % Rel. Diff. 2.68%
Sigma PT (%RSD) 18.61% (Participants)
Allowed Var, 33% σ PT (A) 26.569
12 Week Difference (B) 6.199

Analytical %rsd 11.03%
Analytical Variance Flag sr OK
Should be > 1 1.13

Weeks from End of Round 80
Significance of Slope 0.7254
No Evidence of Instability over 103 weeks.
12 Week % Rel. Diff. NA
Analytical %rsd 20.58%
Analytical Variance Flag sr Suspect
Should be > 1 0.48
Sigma PT (%RSD) 15.00% (Participants)
Allowed Var, 33% σ PT (A) 1.461
12 Week Difference (B) 0.000
Weeks from End of Round 27
Vitamin A 201226 Dairy Herd & Beef Calf Milk Replacer, Me KU/kg

Significance of Slope 0.5265

No Evidence of Instability over 8 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 5.34%

Analytical Variance Flag sr OK

Should be > 1 1.88

Sigma PT (%RSD) 15.70% (Participants)

Allowed Var , 33% σ PT (A) 0.830

12 Week Difference (B) 0.000


Weeks from End of Round 1
Vitamin A 201228 Sheep and Goat Grower/Finisher, Medica KU/kg

Significance of Slope 0.5819
No Evidence of Instability over 180 weeks.
12 Week % Rel. Diff. NA
Analytical %rsd 11.73%
Analytical Variance Flag sr OK
Should be > 1 1.12
Sigma PT (%RSD) 20.00% (Participants)
Allowed Var, 33% σ PT (A) 0.347
12 Week Difference (B) 0.000

LCS Values
Robust Value

Weeks from End of Round 9
**Vitamin A 201523 Milk Replacer, Medicated**

- **Significance of Slope**: 0.0516
- **No Evidence of Instability over 27 weeks.**
- **Analytical %rsd**: 8.78%
- **Analytical Variance Flag**: sr OK
- **Should be > 1**: 1.65
- **Sigma PT (%RSD)**: 19.63% (Participants)
- **Allowed Var, 33% σ PT (A)**: 4506.246
- **12 Week Difference (B)**: 0.000
- **Stability Decision, Is B < A?**: PASS Allowed Var.
- **Weeks from End of Round**: 42

Graph showing LCS Values and Reported Value with data points from 3/12/2015 to 10/29/2016.
Vitamin A 201527 Chick Starter, Medicated U/kg

**Significance of Slope** 0.8357

**No Evidence of Instability over 36 weeks.**

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 24.23% (Participants)

Allowed Var, 33% σ PT (A) 1101.818

12 Week Difference (B) 0.000


**Analytical %rsd** 18.27%

**Analytical Variance Flag** sr Suspect

Should be > 1 0.83

**Weeks from End of Round** 17
Vitamin A 201591 Barley KU/kg

Significance of Slope 0.1916
No Evidence of Instability over 42 weeks.

Analytical %rsd 13.76%
Analytical Variance Flag sr OK
Should be > 1 1.51

Sigma PT (%RSD) 29.55% (Participants)
Allowed Var, 33% σ PT (A) 4.901
12 Week Difference (B) 0.000


LCS Values
Reported Value
Vitamin A 201626 Llama Feed U/kg

Significance of Slope 0.8716

No Evidence of Instability over 14 weeks.

12 Week % Rel. Diff. NA

Analytical %rsd 12.30%

Analytical Variance Flag sr Suspect
Should be > 1 0.77

Sigma PT (%RSD) 15.66% (Participants)

Allowed Var , 33% σ PT (A) 1813.001

12 Week Difference (B) 0.000


Weeks from End of Round 10

LCS Values
Reported Value
Significance of Slope 0.4504
No Evidence of Instability over 32 weeks.
12 Week % Rel. Diff. NA
Analytical %rsd 4.33%
Analytical Variance Flag sr Suspect
Should be > 1 0.99
Sigma PT (%RSD) 6.48% (Participants)
Allowed Var, 33% σ PT (A) 2.876
12 Week Difference (B) 0.000
Weeks from End of Round 19
Significance of Slope: 0.4195
No Evidence of Instability over 50 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 6.36% (Horwitz)
Allowed Var, 33% σ PT (A): 9.227
12 Week Difference (B): 0.000

Stability Decision, Is B < A?: PASS Allowed Var.

Analytical %rsd: 2.56%
Analytical Variance Flag: sr OK
Should be > 1: 1.66

Weeks from End of Round: 63
Significance of Slope: 0.5427

No Evidence of Instability over 29 weeks.

12 Week % Rel. Diff.: NA

Sigma PT (%RSD): 7.05% (Participants)

Allowed Var, 33% σ PT (A): 4.194

12 Week Difference (B): 0.000

Stability Decision, Is B < A?: PASS Allowed Var.

Analytical %rsd: 2.23%

Analytical Variance Flag: sr OK

Should be > 1: 2.07

Weeks from End of Round: 0
Significance of Slope 0.5960

No Evidence of Instability over 17 weeks.

12 Week % Rel. Diff. NA

Sigma PT (%RSD) 7.59% (Participants)
Allowed Var, 33% σ PT (A) 3.741
12 Week Difference (B) 0.000


Analytical %rsd 3.86%
Analytical Variance Flag sr OK
Should be > 1 1.27

Weeks from End of Round 2